BOOK OF ABSTRACTS

43rd International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Session 1 Remote Sensing, Geophysical Prospection and Field archaeology

ORAL

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Contribution of geophysical measurements into the interdisciplinary project at the atypical Late Bronze Age settlement site at Březnice, South Bohemia

Křivánek, R.[1], Chvojka, O.[2], Kuna, M.[1], Menšík, P.[3], Šálková, T.[4]

[1]*Institute of Archaeology of the Czech Academy of Sciences, Prague, v.v.i., Department of Information Resources and Landscape Archaeology*
[2]*University of South Bohemia České Budějovice, Faculty of Arts, n University, Institute of Archaeology and South Bohemian Museum in České Budějovice*
[3]*University of West Bohemia in Pilsen, Faculty of Philosophy and Arts, Department of Archaeology*
[4]*University of South Bohemia České Budějovice, Faculty of Arts, n University, Institute of Archaeology and Faculty of Scinece, Laboratory of Archaeobotany and Palaeolecology*

Keywords: Late Bronze Age; trench-like feature; geophysical survey; settlement; archaeobotany

The Late Bronze Age site in Březnice near Bechyně represents unique mono-cultural settlement with atypical trench-like features and their fillings. Very similar situations were uncovered also in South and West Bohemia, Bavaria or Salzburg region. But in case of Březnice the extent of the site and number of proved or identified trench-like features does not have comparison with other known sites. The first rescue archaeological excavation of the University of South Bohemia took place in Březnice in 2005-2009. These excavations in combination with preliminary particular magnetometer surveys confirmed ten narrow and similary oriented (north-south) trench-like features and remains of other ploughed situations like storage pit, oven, sunken storage jars or pottery hoard. Uncovered shallow and between 4-7 m long trench-like sunken features contained specific composition often burnt daubts, ceramics, clay weights, other artefacts and also organic material remains. Therefore, these features have been analysed also for macrobotanical material. The fillings have a refuse character, rubbish was intentionally placed there and had come into contact with fire.
Unfortunately, the whole area is situated on elevated partly sloped plateau and shallow sunken features are continuously endangered by ploughing of fields. The all of new interdisciplinar results indicate that the whole site does not represent common settlement activity and the real extent of these activities is not known. The new interdisciplinary and interinstitutional work at Březnice has been supported by the project of the Grant Agency of the Czech Republic, no. 18-10747S (Weaving looms, intentional demolitions, burnt offerings…? A new look at rituals in the life of Bronze Age settlements). The project focuses on the study and analysis of a specific type of settlement trench-like features which could lead to probable interpretations of unusual activities at the Late Bronze Age settlement site. The full area magnetometer survey of all agricultural terrains confirmed at least 10 ha with settlement activity and identified over three dozens of trench-like features. Chosen linear magnetic anomalies were proved by new systematic archaeological trenches.These terrain activities were added also by particular detailed magnetic susceptibility measurements in situ and new sampling for macrobotanical analyses.
The sume and spatial arrangement of trench-like features together with character of fillings are for archaeological site near Březnice unique. The options of interpretation of such a site and features could contain more probable and still discussed variants (looms in houses or textile workshops, pottery production remains, cult activities, ritual burning of houses, etc.)

S1-O01.150

Roads of freedom, roads of power: Visualising historical paths of Mount Taygetos, Greece

Panagiotidis V., Valantou, V., Zacharias N.
Laboratory of Archaeometry, University of the Peloponnese; Old Camp, 24 133 Kalamata, Greece

Keywords: StoryMaps; Dentheliatis; Cultural Heritage Routes

Visualization has always played an important role in the presentation, interpretation and dissemination of data. Archaeological and historical information introduced from numerous aspects using visualization techniques from different scientific disciplines with applications using eg. GIS software have proved invaluable in the study of spatial and temporal relations of archaeological factors. Faster and easily accessible Internet connections in collaboration with versatile handheld computers and smartphones, introduce a different category of applications developed for a wide range of end-users, available for scientific study and presentation of results,

The mountainous area “Dentheliatis”, today under the name “Alagonia”, situated at the west part of Mount Taygetos, upon the Messenian Valley. Throughout this area are located, path routes connecting the districts of Messenia with Laconia that created a constant conflict between them (Luraghi, 2008), from Classical to Roman Times, in order to establish their respected borders. During Byzantine Times, this territory degraded and was depopulated from barbarian invasions until the arrival of Slavs in the 6th c. A.D., situating in the area and gradually unified with the local population. These routes and paths from Ancient Farai, Dentheliatis and Ancient Sparta to those travelled during the late Ottoman period from Kalamata and the villages of Alagonia to the city of Mistras embody important archaeological and historic information, telling stories of legends and kings, of generals and poets, testifying the constant presence and occupation of the area from the classical times up to the establishment of the Modern Greek State playing an important role during the Greek War of Independence.

This paper presents a visualization of this journey through storylines written on a map environment. Using opensource programming tools such as LeafletJS and StoryMapJS, an interactive story map (Panagiotidis et al, 2019) was developed to present the archaeological data of the area between Kalamata, Alagonia villages and Mistras as well as the evolution of the roads used to communicate between them demonstrating the continuous use of these routes and the need for recognition and protection. The map will be available online part of the “Kalamata 1821: Roads of Freedom” website.

Luraghi, N. (2008). The Ancient Messenians: Constructions of Ethnicity and Memory. Cambridge University Press

Panagiotidis V., Malaperdas G., Palamara E., Valantou V & Zacharias N. (2019). Information Technology, Smart Devices and Augmented Reality Applications for Cultural Heritage Enhancement: The Kalamata 1821 Project, 1st TMMCH, Athens. Springer (222-231).

S1-O02.533

Session 1 Remote Sensing, Geophysical Prospection and Field archaeology

POSTER

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Geophysical prospection of various enclosed sites in bohemia and evidence of risks of soil erosion

Křivánek, R.

Institute of Archaeology of the Czech Academy of Sciences, Prague, v.v.i., Department of Information Resources and Landscape Archaeology

Keywords: Late Bronze Age; trench-like feature; geophysical survey; settlement; archaeobotany

Study, separation or confirmation of different types of ditch enclosures or other systems of fortifications of hillforts is one from the long-time themes for cooperation of more non-destructive archaeological methods. Surface geophysical measurements are here combined with aerial prospection, remote sensing data, old maps and in some cases also with results of former archaeological excavations. The composition of these enclosed sites in agricultural lowland landscape is very variable. A full-are magnetometer prospection is very suitable method for identification of Neolithic roundels, Eneolithic or Bronze Age ditch enclosures, La Téne quadrangular enclosures, but also different prehistoric and early medieval hillforts. During the last two decades we can use these data also for monitoring and classification of level and intensity of landscape changes of sites very often connected with agricultural activities and soil erosions.

Changes of field conditions of enclosed areas were observed during earlier internal project of the Institute of Archaeology in Prague (Enclosed areas in prehistoric and early medieval times). At present time we repeatedly identify these soil erosion processes in a new archaeo-geophysical project supported by the programme of Regional Cooperation between the Regions and the Institutes of the Czech Academy of Sciences. (Non-destructive geophysical research of endangered sites by agricultural activity with different types of ditch enclosures).

Chosen examples of geophysical measurements documents different local, but sometimes also regional soil changes and erosions of different types of archaeological situations (rondels, ditch enclosures, outer baileys or internal structures of hillforts situated in different regions of Central, North or South Bohemia). Some of these changes of subsurface preservation of linear features we can observe also by other additional resistivity, radar or magnetic susceptibility measurements.

Archaeological sites situated on long-time agricultural terrains are changing including subsoil remains of various sunken features. These circumstances are unknown to neither archaeologists nor archaeological monument care. Soil degradation or erosion will play also important role for the future possibilities of identification of subsurface archaeological remains by non-destructive geophysical (or aerial) methods. Some of these new geophysical data were used in various archaeological projects and excavations, the other will be used as a base source for a new planned sub-project as a part of an international and interdisciplinary project COST Action SAGA (The Soil Science & Archaeo-geophysics Alliance: going beyond prospection, CA17131).

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Rural antiquities in changing landscapes: estimating land-use changes with the use of satellite imagery and GIS in the regional survey of northern Messenia, Greece

Vallianatou, E.[1], Zimi, E.[1], Malaperdas, G.[1]

[1]Department of History, Archaeology and Cultural Resources Management, University of the Peloponnese, Kalamata, Greece.

Keywords: Satellite imagery; GIS; rural archaeology; Messenia; archaeological prospection

Earth observation technologies are becoming more significant in the cultural heritage management and protection. Identifying changes in urban and rural areas caused by natural phenomena, abandonment and low or-non-maintenance, contributes to a better understanding of the contemporary landscapes and prompts their protection from further damage. Residential locations, such as villages and their rural environs, frequently accommodate structures of archaeological interest. Even though these historical structures may be well-known to the local community, they get worn out and destroyed due to the lack of use or adequate maintenance. Since the quality of satellite imagery and aerographic photography has vastly improved, high-resolution photographs are valuable for observing changes in the environment and the surrounds of inhabited sites. We can more effectively monitor the changes that occur in a larger research area with the help of GIS and the use of multilayer maps and data digitalization.

This paper focuses on the rural region of northern Messenia, where the fertile plain of Stenyclaros extended in antiquity. Its aim is to explore the use of remote sensing methods, such as satellite images and historical area photographs, in defining the impact a) of agriculture to antiquities - the case study chosen for this scope is a partly excavated Roman bath complex in the village of Polichni, b) of low-maintained or abandoned rural areas to the monuments located within their boundaries, examining selected monuments (e.g. the poorly preserved church of Hagios Athanasios) in the vicinity of the modern village of Stenyclaros.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Two electromagnetic surveys in the territory of Oliveira de Azemeis

De Man, A.[1], Tavares, J.T.[2]

[1]*United Arab Emirates University*
[2] *Municipality of Oliveira de Azemeis*

Keywords: Magnetic survey, fieldwork, methodology

Ossela and Ul are two hilltop sites in the municipality of Oliveira de Azemeis (Portugal). At both locations, preliminary fieldwork revealed long diachronic occupations, and they become relevant from a landscape archaeology perspective as well. They do stand out as territorial markers, the former as a chapel, and the latter as a partially adjacent village.

A major concern in applying electromagnetic surveys (in one of the cases combined with GPR), was to optimize existing knowledge and direct research towards currently available lots, in order to achieve a more integrated understanding of the sites. The equipment itself, as well as the topographic, geological, and even legal circumstances determined procedures and results.

Geophysical data were vital in targeting suitable fieldwork approaches; provisional outcomes became also significant for the refinement of methodological robustness. In short, they point to considerably complex structures, both funerary and domestic. As far as limitations are concerned, they need to be discussed in scientific, technical and budgetary terms.

Archaeological stratigraphy has been quite disturbed in both sites, a consequence of recent forestation, quarrying and building activity. Still, such disruptions hindered survey and excavation to different degrees. Especially at Ul, sections of the hill are better preserved, whereas the less extensive area at Ossela exposed what is basically a modern cemetery.

The results may be significant for current and future research, as they provide a practical, comparative reference in a rather challenging geographic setting.

S1-P04.273

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Georadar studies in Hagia Sofia, Istanbul (Turkey)

Barba, L.[1], Blancas, J.[1], Miriello, D.[2], Cura, M.[3], Crisci, G.[2], Cappa, M.[4], de Angelis, D.[4], Taranto, M.[2], Pecci, A.[1,2,5]

[1]Laboratorio de Prospección Arqueológica, Instituto de Investigaciones Antropológicas, Universidad Nacional Autónoma de México, 04510 México, DF, México
[2]Department of Biology, Ecology and Earth Sciences, University of Calabria, Arcavacata di Rende, CS, Italy
[3]Ankara Haci Bayram Veli University
[4]Restructura—Surveys for Cultural Heritage, Via Gerolamo De Rada n°60/i, 87100 Cosenza, CS, Italy
[5]Equip de Recerca Arqueològica i Arqueomètrica de la Universitat de Barcelona (ERAAUB), IA-UB, Departament d'Història i Arqueologia, Barcelona, Universitat de Barcelona, Spain

Keywords: Characterization, geophysics; constructive phases.

Within an interdisciplinary project to study Istanbul's Hagia Sofia, carried out as part of an agreement between the Hagia Sofia Museum, the University of Calabria and UNAM, and the participation of the University of Barcelona, a georadar survey was carried out in the central nave, in the upper floor and in some of the pillars and walls of the building using a Sir System 3000 with 400 MHz antenna.
The central nave was investigated to assess if the previously observed deformations of the structure could be related to a differential behavior of the subsoil caused by architectural remains underneath. The upper floor of the building was studied to understand the reasons of the breakage of the marble slabs of the floor and some discontinuities in the floor itself. Pillars and walls were studied to better understand the breakage and detach of the vertical marble slabs, and verify the building techniques of the pillars.
Part of this study faced an unusual challenge since it was necessary to study the space beneath the scaffolding placed for the restoration works of the building affecting the readings density in this part.
The survey of the central nave was successful and allowed the detection of remains of walls that probably formed the basement of a previous 18- by 22-m structure. In addition, another interesting feature was discovered 2 m below the marble mosaic in the southeastern part of the nave. Although it is not possible to define its function yet, it seems that there could be a suggestive relationship between this 2- by 3-m buried structure and the marble mosaic in the floor surface.
Although some structures were identified underneath the floor of Hagia Sofia, there are no major changes in the topography of the central nave floor.
The breaking of the marble slabs of the upper floors seems to be related to deformation of the structure and the construction process involving the use of un-pressed filling material, while the detachments of the vertical slabs is due to the deformation of the walls of the whole building.

S1-P05.380

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Preliminary results of the archaeological prospection in a central neighborhood in Teotihuacan (Mexico)

Barba, L.[1], Ortiz, A.[1], Blancas, J.[1], Moragas, N.[2], Pecci, A.[3,1]

[1]Laboratorio de Prospección Arqueológica, Instituto de Investigaciones Antropológicas, Universidad Nacional Autónoma de México, 04510 México, DF, México
[2]CINAF, Departament de Prehistòria i Arqueologia, Barcelona, Spain
[3]Equip de Recerca Arqueològica i Arqueomètrica, Universitat de Barcelona (ERAAUB), Departament de Prehistòria i Arqueologia, Barcelona, Spain

Keywords: Archaeological prospection, geophysics, Teotihuacan.

In 2017 we started an interdisciplinary project to study a neighborhood in the center of Teotihuacan (Mexico), in co direction between the UNAM (A. Ortiz, L. Barba) and the Universitat de Barcelona (A. Pecci, N. Moragas).
The project is aimed at investigating an area located in the center of Teotihuacan (The City of Gods), Mexico, between the river San Juan, the Sun Pyramid and the Street of the Dead, named by Millon “sectors N2E1 and N2E2”, through the application of geophysical prospection techniques (georradar, electrical and magnetic studies), topography, aerial photography with drone, the study of chemical residues on the surface and the registration of archaeological materials to identify structures and indicators of human activity. The data obtained until now in selected portions of the two sectors, are compared to the map proposed by Millon in 1973, and allow to suggest preliminary hypotheses about the presence of buildings, their distribution and depth.
The aim of the project it is to recognize the presence of a neighborhood, a typical unit in the organization of Teotihuacan, and its components such as residential areas, workshops, temples, squares.
We show here some examples of the preliminary results obtained.
The project is financied by the Spanish *Ministerio de Cultura y Deporte* (Ayudas a proyectos arqueológicos en el exterior 2017 and 2019), the Fundación Palarq (2018 y 2019), and it is part of the activities of the Laboratorio de Prospección Arqueológica of the UNAM, the groups ERAAUB (2017 SGR 1043) of the University of Barcelona and the contract RyC 2013- 13369 (MINECO).

S1-P06.523

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

UAVs in Cultural Heritage: The case of Geraki Lakonia, Greece

Kompoti A., Panagiotidis V.V., Zacharias N.

Laboratory of Archaeometry, University of the Peloponnese; Old Camp, 24 133 Kalamata, Greece

Keywords: UAVs; Geraki Lakonia; orthophotos; visualization archaeological sites; GIS

The archaeological site of Geraki in Lakonia is located on the south-western slopes of Mount Parnon in the SE Peloponnese, is comprised of the medieval castle and its settlement [3] and has recently been restored by the Greek Ministry of Culture. Geraki was built during Frankish rule in the Peloponnese, in the middle of the 13th century. The castle's location was crucial for controlling the road communication between Mystras and Monemvasia [1].

The aim of this study is the initial documentation of the current situation of the medieval settlement of Geraki applying innovative technology including unmanned aerial vehicles (UAVs). Following the developments in image capturing and processing, UAVs have become an alternative in cultural heritage domain and have successfully been used in different projects. Due to their ability to perform in high-risk situations (e.g. lack of accessibility, high slop remote areas and sites of high altitude) while the pilot maintains a safe distance UAVs can reach places where men cannot, making them a standard platform in the cultural heritage sector [2].

In this study, a UAV was used in order to visualize the archaeological site with surrounding buildings and structures through the capture of high resolution aerial photographs. Following processing the photos with photogrammetry software, digital orthophotos and digital surfaces models are produced. The orthophoto in turn is used with GIS software for the identification of the specific location of the archaeological site's buildings. Separated layers in GIS were created in order to include information about the topography, the architecture, the characterization and categorization of buildings, creating thus an important historical and archaeological database [4].

[1] Christodoulou, R., Simatou A, 1991. Observations in the medieval settlement of Geraki. Deltion of the Christian Archaelogical Society. Deltion XAE (1989-1990), Period D,Athens pp67-88

[2] Korumaz, A., Korumaz, M., Tucci, G., Borona, V. Niemeier, W., Riedl, B., 2012. UAV systems for documentation of cultural heritage. In: ICONARCH I-International Congress of Architecture-Innovative Approaches in Architecture and Planning, Konya, Turkey, Vol. ISBN: 978-975-448-206-5, pp419-430

[3] Ministry of Culture and Sports, Ephorate of Antiquities of Lakonia,2015. Organization, Improvement of Visitor Accessibility and Conservation of Monuments on the Archaeological Site of Geraki, Sparti

[4] Anagnostaki K., Petmezas S., Sarris Ap, 2014.Reading history on a map: The Venetian cadastre of Vonitsa, 3rd Symposium Arch_RNT- Archeological Research and New Technologies, Kalamata, 31-38.

S1-P07.1551

Field Surveying, Geostatistical and GIS methods in Archaeology. The Mycenaean Spercheios-Valley Archaeogeophysical Project (MY.SPE.AR. Project 2018-2022)

Malaperdas, G.[1], Maggidis, C.[2], Zacharias, N.[1]

[1]Department of History, Archaeology and Cultural Resources Management, University of the Peloponnese, 24 100 Kalamata, Greece
[2]Department of Archaeology, Dickinson College; President of the Mycenaean Foundation; Field Director, Mycenae Lower Town Excavation

Keywords: Mycenaean; GIS; Geostatistics; Field Archaeology; MY.SPE.AR.

An initial goal of the Mycenaean Spercheios-Valley Archaeological Project (MY.SPE.AR.) (2018-2022) is the systematic archaeogeophysical survey of the Spercheios Valley in central Greece. The extensive and intensive archaeological survey, aided by aerial reconnaissance, LIDAR, and geophysical survey, focuses on locating, documenting, mapping and analyzing remains of Mycenaean sites in combination with environmental features in the region.
In June 2018, the archaeogeophysical survey commenced in the Lamia municipality under the directorship and auspices of the local Ephorate of Antiquities, focusing initially on sites documented from publications and previous field reports and subsequently expanding to explore new sites.
The paper demonstrates the importance of the application of Geographical Information Systems (G.I.S.) and spatial analysis in archaeology. The idea lying behind this attempt is that the Mycenaeans would not have established their habitation sites randomly but according to decisive criteria, among which geomorphological, climatic and geological characteristics played an important role. G.I.S.-based geospatial analysis with a geo-cumulative approach, integrating archaeological, geomorphologic, geologic, geotectonic, paleo-environmental, hydrographic, and paleo-climatic variables, will help explore interaction between environment and site distribution; statistical, frequency, and spatial distribution models are applied to trace contact patterns and hierarchical dynamics among the sites, and to identify second order centers and possibly a first order administrative center in the region. The statistical approach we applied in this study can provide with important information on the location of sites of the Mycenaeans. Therefore, it can increase our understanding of the locations selected by Mycenaeans as well as their criteria for this selection.
Finally, the settlements under study sorted to the four tier settlement hierarchy schema established in the study of Mycenaean settlement patterns and distribution. It must be noted, however, that, in order to include each settlement in one or the other category, relevant studies usually combine estimated settlement size on other criteria, such as the existence of architectural remains and the types of existing nearby tombs. Based on the above, the present work will contribute to a better reading of the targeted area from an archaeological point of view and to posing new archeological questions.

S1-P08.565

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Storm risk monitoring in medieval fortifications by GIS and remote sensing

Moreno, M.[1], Ortiz, R.[1], Ortiz, P.[1]

[1]*Department of Physical, Chemical and Natural Systems, Pablo de Olavide University.*

Keywords: remote sensing; risk, medieval fortification; rammed earth, storm.

Torrential rains are one of the primary weather hazards in Europe. In the coming years, climate change is expected to modify precipitation patterns on a global scale, increasing the risk levels associated with heavy rainfall events. In this scenario historic and areological earth structures are particularly vulnerable. This is the case for medieval rammed earth fortifications, regarding which the infiltration of water into the walls causes erosion, loss of material and, in the worst-case scenarios, collapse. For this reason, ensuring their conservation entails understanding the factors involved in these risk situations. The purpose of this study is to investigate three past events in which rains caused damage and collapse to historic rammed earth fortifications in Andalusia in order to analyse whether it is possible to prevent similar situations from occurring in the future. The main objective is to identify the characteristics of force, intensity and duration of the rains that triggered the emergency, the degree of vulnerability presented by the structures and the climatic hazard of the environment through the use of GIS and satellite resources. For this, the use of the different modules of the Art-Risk methodology is proposed. The hazard presented by rain within this context has been obtained from *Art-Risk 3.0 GIS* a tool that identify the hazards from the environment according to a scale from 1-5. The vulnerability of the structures has been assessed with the *Art-Risk 1*, a model based on a Leopold matrix that includes a list of rammed earth wall pathologies and their magnitude or capacity to cause damage. To characterise the strength, duration and intensity of rainfall events, a workflow for the statistical use of *Global Precipitation Measurement* and *Global Satellite Mapping of Precipitation*, satellite resources, has been designed, validated and tested. The accuracy of the satellite data has been evaluated by comparing it with data from ground-based weather stations. As a result GSMaP precipitation data, with the high coefficients of determination observed (>0.85), is the most similar to data from ground-based weather stations, and therefore the most accurate. Regarding the three risk events analysed, although they occurred in areas with a torrential rain hazard, the damage was caused by non-intense rains that did not exceed 5 mm/hour. The continuation of the rains for several days and the poor state of conservation of the walls seem to be the factors that triggered the collapses that fundamentally affected restored areas.

S1-P09.3012

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Known methods - new approaches of interpretation, results of the geophysical prospection of the Roman Colonia Ulpia Traiana (Xanten)

Babucic, N.[1], Seifert, M.[1]

[1] *Hamburg University*

Keywords: geophysical prospection methods; magnetic mapping; gpr; site reconstruction

Geophysical prospections have been an integral part of archaeological fieldwork for decades and serve in many projects as a basis for the investigation of specific questions of urban infrastructure and economy. A desideratum is a set of analyses that go beyond the mere observation of prospection data and allow the reconstruction of large areas of entire sites. The Colonia Ulpia Traiana (Xanten), which has been excavated to about 15 % in the last hundred years, is the field of investigation to be presented. On the basis of the geophysical prospections carried out over the whole area by different scientific teams and the information exchange of different methods (magnetics, susceptibility measurement, GPR, aerial archaeology, processing of older excavation documentation and prospections) a standardized classification and validation of the survey results will be carried out, which will serve for a reconstruction of the whole Colonia.
At the invitation of the LVR-Archaeological Park Xanten, the Department of Classical Archaeology of the University of Hamburg has been conducting geophysical surveys with geomagnetics and georadar on the site of the CUT since 2013 and has meanwhile been able to map the entire accessible area of the CUT. The lecture will present the preliminary results of the presented approach and offer an outlook on possibilities of adaptation for further sites.

S1-P10.1570

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Non-invasive survey of ancient tin mining and metallurgical traces in Northwest Iberia

Meunier, E.[1], Fonte, J.[2], Dias, F.[3], Lima, A.[3], Figueiredo, E.[1]

[1]Cenimat/i3N, Faculdade de Ciências e Tecnologia, Universidade NOVA de Lisboa, Portugal
[2]Department of Archaeology, University of Exeter, UK & Institute of Heritage Sciences (Incipit), Spanish National Research Council (CSIC), Spain
[3]ICT, Faculdade de Ciências, Universidade do Porto, Portugal

Keywords: Airborne LiDAR; Aerial imagery; Mining Archaeology; Northwest Iberia, Tin.

Despite being considered one of the main areas where tin was exploited in antiquity, ancient tin mining and metallurgy in Northwest Iberia is still involved in uncertainty. The location of ancient tin mines and their chronology is still widely undefined. In the scope of the *IberianTin* project (PTDC/HAR-ARQ/32290/2017) we are developing an integrated, multiscale and interdisciplinary methodology to tackle this problem.
We will present and discuss some preliminary results from three mining areas in Northwest Iberia, surrounding the mountain ranges of Serra d'Arga, Serra do Larouco and Serra do Barroso, where ancient tin mining is known. These mines harnessed secondary deposits of cassiterite, and were exploited by a pre-industrial type of mining based on hydraulic systems. This implied the movement of high volumes of sediments over the entire mineralized area, transforming deeply the landscape. A comprehensive wide-ranging vision of the topography is then required to understand their organisation.
Thus, in the present work we integrate both airborne LiDAR and historic aerial imagery to locate and map tin mining vestiges from those three areas, revealing several phases of exploitation. With the combination of historic mining documentation and literature review, we can assess with further detail which areas were affected by modern mining and define which locations are the best preserved for archaeological purposes.
Ground-truthing involving field survey and geological sampling was performed in areas of interest for studies on mineralisation and for a better definition of the main features of the mining works. When available, metallurgical debris, as slags, have also been subjected to analytical studies, since these can confirm processing of cassiterite to produce tin in the close vicinity of the mine.
Based on the presented methodology, it will be possible to propose future invasive archaeological works in specific locations, targeting the recovery of archaeological material in stratigraphic context and precise dating evidences. With this study, we aim to contribute to the knowledge of the scale of mining activity and its evolution, as well as to define the type of metallurgical processes undergoing near the mine. This will finally lead to better understand the link between these activities and their socio-economic context.

S1-P11.670

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

How to evidence hypogeal tombs in pyroclastic and epiclastic rocks: the applicability of ground penetrating radar and resistivity tomography on the "Domus de Janas" of Luzzanas necropolis in Sardinia (Italy)

Testone, V., Longo, V., Mameli, P.

Department of Chemistry and Pharmacy, University of Sassari, Italy.

Keywords: Domus de Janas; electrical resistivity tomography; ground-penetrating radar; Luzzanas necropolis; Sardinia.

A GPR survey joined with ER tomographies was carried out in the "Domus de janas" hypogeic necropolis of Luzzanas (Benetutti), in central Sardinia (Italy), aiming to evaluate the applicability of the two methodologies in detecting voids related to tomb cells and to define the extent and shape of these particular archaeological structures.

The "Domus de janas" are typical Sardinian sepulchral structures of the Neolithic age represented by rock-cut tombs of various shapes. The name means “houses of the fairies” and they consist of several chambers tombs, which walls are often decorated with reliefs or etchings depicting magical and religious symbols such as spirals, zig-zag motifs and bull's horns.

The Luzzanas necropolis was excavated on two different lithotypes (1 epiclastic and 1 pyroclastic) but both characterized by an ashy/glassy groundmass, rich in neoformed clinoptilolite, and by crystals of plagioclase, augite and rare quartz.

The GPR survey was conducted using a monostatic GPR IDS model ‘RIS_MF_HiMod Duo’, consisting of a control unit working simultaneously with two transmitters operating at frequencies of 400 and 900 MHz, and two receivers. Two hitherto unexplored areas of 171 m^2 and 190 m^2 were investigated by regular GPR grids, with 50 cm lines spacing.

The ERT survey was performed by using a multielectrode system consisting of an Abem Terrameter SAS 1000 device combined with an ES10-64 electrode selector. For each 2D electrical line, 64 metal electrodes were deployed in a straight line with a constant spacing of 50 cm and a unit length of 31.5 m. Six ERT surveys were executed using a dipole–dipole array, which is sensitive to horizontal changes in resistivity and therefore suitable for detecting vertical structures such as cavities.

The ERT models have highlighted the resistivity contrast between the rocks of the area (ρ = 10 ÷ 70 Ωm) and the void cells (ρ = 80 ÷ 2000 Ωm).

The GPR survey provided fine detail of the shallower part of buried structures, but had strong limitations in defining the deeper and wetter levels, due to the presence of saturated soils and some flooded cells that determined a strong absorption of the electromagnetic signal.

Overall, this case study demonstrates the efficiency of the combined use of GPR and ERT acquisitions and shows how, in general, only the joint analysis of 2D data and in a 3D view can help the interpretation of the real distribution of the rock-cut tombs.

S1-P12.718

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Settlements of Chalcolithic Cucuteni Culture. Smart techniques for capitalization of buried archaeological heritage from NE Romania

Asăndulesei, A., Tencariu, F., Balaur, R., Asăndulesei, M.

"Alexandru Ioan Cuza" University from Iași, Romania,
Interdisciplinary Research Institute, Science Department, Arheoinvest Research Center

Keywords: Cucuteni culture; Settlements layout; Archaeo-geophysics; Remote sensing; Cultural heritage

Interdisciplinary scientific community from Romania focusing on archaeology and its sustainable management is devoting efforts to adopt, adjust and apply new tools to protect, manage and promote buried archaeological heritage, especially involving digital and cutting-edge technology borrowed from geosciences. Chalcolithic Cucuteni settlements represents one of the main cultural resources in the area, but rather underexploited and continuously facing the natural and anthropic destruction.

There are more than 1800 sites on Romanian territory, whether hilltop or lowland settlements, compact or scattered, seasonal or permanent, main or secondary, small, medium or large in size, clearly showing the extremely dynamic character of these communities.

By combining non-destructive and high-resolution ground-based geophysical (magnetic surveys) and airborne sensing techniques (aerial photogrammetry and LiDAR measurements), alongside minimum invasive soil sampling and analysis and targeted archaeological excavation we were able to start comprehend the complexity of internal spatial arrangement of Cucutenian sites from NE Romania.

A quite complex and novel site plan pattern was acknowledge documenting a so-called external habitation, organized outside of the enclosed core of the settlement. Different feature types (burnt dwellings, palisades, delineation or defensive ditches, pits, etc.) were also detected, interpreted as cultural anomalies and verified by targeted excavation (for the stratigraphy).

Identification of the exact site limits by archaeo-geophysical means and the precise GPS positioning will help the local community and other stakeholders to be more aware of this kind of cultural resource in order to better preserve the sites and exploit socio-economically.

S1-P13.731

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Surface characterization of vases and coffer seals of the Kha tomb in the TT8 project through RTI and normal map integration

Gargano, M[1]. Verni, E.[1], Grifoni, E.[1], Ferraris, E.[2], Cavaleri, T.[3]

[1]Dipartimento di Fisica A. Pontremoli, Università degli Studi di Milano, Via Celoria, 16, 20133, Milano, Italy
[2]Museo Egizio, Via Accademia delle Scienze, 6, 10123 Torino, Italy
[3]Fondazione Centro Conservazione e Restauro dei beni culturali La Venaria Reale, via XX settembre 18, Venaria Reale (TO), Italy

Keywords: RTI; Normal map integration; Profilometry; Hieroglyph Seals; Grave Goods.

Reflectance Transformation Imaging (RTI) is a computational imaging technique for visualizing a surface under a spatially variable source of illumination. A set of images of the object of interest is captured by keeping the camera position fixed and changing only the direction of the light source, which may be different depending on the case. The luminance dependence on the light direction provides information on the surface of the object obtaining a map of the object's normal. Through the integration of the normal map, it is possible to reconstruct the heights map coinciding with the object's profile.
Currently, to represent the profile of an object surface, high-resolution profilometers are the best methods. With this work, we propose an alternative and innovative technique, which is also non-invasive since it is not necessary to touch or move the object.
This research was realized in the framework of the TT8 project where different archaeometric analyses are being performed on the objects found in the Kha tomb, known as Theban Tomb 8 (TT8), one of few tombs of nobility to survive intact.
RTI and normal map integration were applied on clay oval seals with hieroglyphic writings applied on vases and wooden coffers found in the tomb as grave goods provided for Kha and his wife Merit.
With this technique it was possible to obtain the 3D reconstruction of the profile of the stamped seals allowing Egyptologists to better visualizing the hieroglyphics. The technique has proved to be particularly efficient showing details that otherwise would not be visible with other traditional techniques. The seals are in fact very small in size, 25 x 15 mm the largest, and placed in correspondence of the vases neck and then hardly accessible.
The extremely high spatial resolution of the 3D interactive models allowed furthermore to visualize the fingerprints of who placed, modelled and stamped the seal.
This new approach of implementing a well-established RTI technique to obtain high resolution surface profile models, enable scholars to study those objects under a new light, but also open to a wide audience the possibility to interact with rare artworks.

S1-P14.815

Geophysical approach for the study of open urban areas of the 3rd century BC in Celtic Europe: the example of La Peyrouse (Dordogne, France).

Hantrais, J.[1], Mathé, V.[2], Corfmat, P.[3], Chapoulie, R.[4], Chevillot, C.[5], Hiriart, E.[6]

[1]PhD student, Bordeaux-Montaigne University, France, IRAMAT-CRP2A
[2]Lecturer, La Rochelle University, France, LIENSs
[3]Master degree student, Bordeaux-Montaigne University, France
[4]Professor, Bordeaux-Montaigne University, France, IRAMAT-CRP2A
[5]Research associate, Ausonius
[6]Research Fellow CNRS, IRAMAT-CRP2A, Bordeaux-Montaigne University, France, IRAMAT-CRP2A

Keywords: geophysical prospections, open urban areas, 3rd century BC, proto-urbanization

In the Southwest of France, the development of open agglomeration in the 3rd century BC is a widely unknown and unexplored field. The region of Aquitania is a key area that revealed multiple sites enlightening the genesis of the urban phenomenon on the western rims of Celtic Europe. La Peyrouse is one of those.
La Peyrouse is located in Saint-Félix-de-Villadeix (Dordogne, France). The site was revealed by the discovery on ground surface of an abundant corpus of artefacts dating from the 3rd century BC, collected over an area of at least 30 hectares. Given the size of this urban site, the use of geophysical prospecting, an extensive and non-destructive method, was required in order to specify its extension and discern its internal organization.
Both magnetic and electrical geophysic detection methods were applied at La Peyrouse. The magnetic method is particularly well suited to the detection of hollow structures or combustion areas, most often associated with activities that amplify the physical properties of the soil. This method made it possible to quickly cover a large area of about 4.5 hectares. To complete an extensive first magnetic acquisition, electrical surveys (cartography and section) can point out particular elements, in particular a large quadrangular building visible on the magnetic anomaly map.
This first geophysical prospecting campaign at La Peyrouse made it possible to assess the archaeological potential of the site by revealing the first elements of its internal organization, using adapted and complementary geophysical methods. These first results allowed to focus future excavation operations that will complete and specify the nature of the perceived anomalies. These large-scale acquisitions thus initiate a global and exhaustive reflection on a type of site that is still poorly known.

S1-P15.845

The role of aerial prospection for monitoring and preserving the cultural heritage in northeastern Africa: the PERAIA project experience

Laguna-Palma, D.[1]

[1]*Department of Prehistory and Archaeology. University of Granada (Spain).*

Keywords: Remote sensing; Endangered heritage; Digital Archaeology; Open Data; Northeastern Africa.

Research in archaeology has experienced exponential growth over the last decades. This is mainly due to the influx of digital technologies and the merging of different theoretical schools. Quantitative approaches from Digital Humanities, and specifically from Digital Archaeology, have opened up new interpretative possibilities by combining techniques from other disciplines, such as Computer Science, and Social Sciences, which allow for the development of new research methods. Following this, the utilization of earth observation data through aerial photographs and satellite imagery has emerged as an unmatched tool in the exploration and (digital) preservation of endangered archaeological sites.

Supported by the Ministry of Universities of Spain and the Vice-Rectorate for Research and Knowledge Transfer of the University of Granada, the PERAIA project emerges as a joint effort between scholars from the University of Granada, the Autonomous University of Barcelona, the Institute of Archaeology of the University College London, and the Centre for Urban Networks Evolution (UrbNet). PERAIA aims at representing and analyzing the social and cultural impact of human-environment interactions along northeastern Africa (NE-Libya/NW-Egypt), from the Late Bronze to the Early Iron Age (c. 1500 – 700 BC).

Drawing on these aims, we pretend to deepen on the importance to implement these digital methods, at the same time it is also crucial to ensure the safety of archaeological and heritage sites in these vulnerable areas. As such, registering the archaeological heritage is also a way to counter the potential risk of destruction these historical sites are exposed to. This risk is mainly due to changes in coastlines, accelerated by climate change, urban development, and political instability.

To this end, we will present a comprehensive and detailed digital gazetteer that already contains over 2500 sites of archaeological and heritage interest located on coastlines, and adjacent areas available as Open Data through the project's geoportal. With this, we aim for both the scientific community and the local administrations, as well as any interested user, to access, review and use the data provided, fulfilling a commitment to seek social impact and dissemination.

Summing up, we pretend to show the importance of a critical assessment of digital technologies and, with this, we hope to show the implications and possibilities that open this approach in the research on field archaeology.

S1-P16.1563

Evaluating the Impact of Landscape Changes on GIS Viewshed Analysis of Copper Age Romanian Archaeological Sites

Tafani, A.[1], Covătaru, C.[2], Lazăr, C.[3], Tykot, R.H.[4]

[1]Department of Anthropology, University of South Florida, Tampa, FL 33620 USA
[2]Faculty of Geography, University of Bucharest, Romania
[3]ArchaeoScience Platform, The Research Institute of University of Bucharest, Romania
[4]Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: GIS viewshed analysis; Romania; Copper Age

Viewshed Analysis is one of the most straightforward GIS analyses to perform using commonly available GIS software; consequently, it is one of the most popular GIS tools used by archaeologists to attempt to understand the relationship between the geographical location of ancient communities and their environmental setting. However, given the scale of the changes in the landscape configuration during the last few decades, one may wonder whether performing this type of analysis on Digital Elevation Models (DEM) derived from contemporary or recent data may reveal some insightful views on the past situation. This study seeks to evaluate the impact of landscape changes on viewshed analyses performed with GIS software. In order to do so, we created several DEMs of the areas in the vicinity of several Romanian archaeological sites of the Copper Age period (4600-4000 BC). During this period, many sites were occupied over many generations and eventually constituted tells, artificial mounds that dominated the surrounding area. Consequently, comparing the results of Viewshed Analyses on such sites and on shorter-lived settlements should indicate whether the visual control of the neighboring territory influenced the location of the tells, or if other factors should be taken into consideration.

We were interested in examining any potential discrepancy between freely available Digital Surface Models (DSM), such as the Shuttle Radar Topography Mission (SRTM) and the Advanced Spaceborne Thermal Emission and Reflection Radiometer (Aster), in particular DSM that are produced with data obtained from older satellite images such as Corona and Hexagon, and Digital Terrain Models (DTM) based on Romanian topographic maps. We then applied the same kind of viewshed analyses on the different DEMs, and compared the results. By doing so, we were able to gain a better appreciation of the importance of the restitution of paleotopographic features for viewshed analysis of archaeological sites.

S1-P17.580

Session 2 Archaeochronometry (organic and inorganic materials)

ORAL

Chronological modeling of Egyptian Old Kingdom (MERYT)

Quiles, A.[1], Aubourg, E.[2], Beck, L.[3], Bellot-Gurlet, L.[4], Delque-Kolic, E.[3], Ferrant, M.[4], Invernon V.[5], Muller, S.[5]

[1]*Institut Français d'Archéologie Orientale, Pôle Archéométrie, Cairo, Egypt*
[2]*Laboratoire AstroParticule et Cosmologie, CNRS UMR 7164, Université de Paris, France*
[3]*Laboratoire de Mesure du Carbone 14/LSCE, CEA Saclay, Gif-sur-Yvette, France*
[4]*Sorbonne Université, CNRS, MONARIS (UMR 8233), France*
[5]*Institut de Systématique, Evolution, Biodiversité (ISYEB), MNHN, CNRS, Sorbonne Université, EPHE, Paris, France*

Keywords: Radiocarbon dating; Egyptian chronology; Calibration; Bayesian modelling

MERYT project aims to build an accurate, complex and multi-technical absolute chronological model for the Egyptian Old Kingdom (~3000-2400 BCE), through an integrated approach bringing together all the analytical criteria of Egyptology, Archaeology and Archaeometry. It addresses two major issues: 1) To develop a definitive chronological framework of the Old Kingdom, reign by reign, by building a statistical model reconciling Egyptological and analytical data; 2) To adapt the ^{14}C IntCal calibration curve considering the specific environmental conditions in Egypt in order to make the ^{14}C dating method more competitive for this geographical area. Four investigation axes are lead:

- ⇨ Historical: for each of the ca. 30 reigns, we are re-evaluating available chronometric evidence from archaeological, historical and textual sources to identify all reign certificates and assess their reliability. Confronted with recent archaeological fieldwork, different possible relative chronological schemes will be suggested.
- ⇨ Archaeometrical: more than hundred ^{14}C dates are currently carried out at the IFAO lab on samples from archaeological on-going excavation sites and collected in a closed context. Beyond "dating", the challenge is also to ensure the analysed sample is consistent with the associated archaeological event. We above all focus on sampling "good" specimens by clearly identifying the associated archaeological context, and mobilizing various archaeometrical techniques (FITR, Raman, GC-MS etc.).
- ⇨ Methodological: a major challenge is to check the applicability of the ^{14}C calibration curve to Egypt by determining possible regional offsets. They could be explained by seasonal variations in the $^{14}CO_2$ content in the atmosphere, linked to the particular environmental conditions caused by the Nile annual flooding before the construction of the High Dam. To do that, we are assessing the residual ^{14}C ratio of botanical specimens collected during the French military expedition in Egypt and conserved in the MNHN Herbarium, whose year and location of harvest are documented.
- ⇨ Statistics: heterogeneous constraints (relative and absolute) are finally combined in a chronological model based on a statistical formalism entirely produced by the MERYT consortium, which will simulate ages densities and uncertainties' estimates for each reign of the Old Kingdom.

This communication will sum-up first results of the MERYT project and discuss future perspectives.

S2-O01.97

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Radiocarbon dating of historical mortars: discrimination of contaminants for a more suitable dating procedure

Ricci, G.[1,2], Secco, M.[2,3], Caneve, L.[4], Marzaioli, F.[5], Passariello, I.[5], Terrasi, F.[5], Artioli, G. [1,2]

[1]University of Padova, Department of Geosciences, Padova, Italy
[2]Inter-departmental Research Center CIRCe, University of Padova, Padova, Italy
[3]University of Padova, Department of Cultural Heritage (DBC), Padova, Italy
[4]ENEA Technical Unit for the development of applications of radiations, CR Frascati, Italy
[5]Centre for Isotopic Research (CIRCE), University of 14 Campania "Luigi Vanvitelli", Caserta, Italy

Keywords: Radiocarbon dating; Mortars; Luminescence; Binder separation; Mortar characterization

Mortar dating is often requested to establish the chronology of masonry structures in historical contests. Radiocarbon dating is used to date mortars based on the atmospheric CO_2 signal absorbed during the hardening process by lime putty, during the carbonation process. The anthropogenic carbonate must be isolated from other carbonaceous sources, which can incorporate dead carbon overestimating the age of construction, as well as secondary alteration processes and delayed hydraulic reactions which could be responsible of an underestimation of the age. Despite the substantial efforts devoted to find efficient protocols and tests to isolate and measure the binder fraction, the problem is still at stake [1], [2].
This research focuses on the identification and elimination of non-anthropogenic contaminants. The adopted protocol employs a detailed characterization of the mineral phases by electron microscopy (SEM), X-Ray powder diffraction (XRPD), and laser induced fluorescence (LIF). The aim is to discriminate the geogenic and anthropogenic calcite exploiting the crystallographic and luminescence properties of carbonates [3], [4]. The method encompasses a multi-step separation of the binder fraction by gravimetric sedimentation and, eventually, by further purification treatments by thermal decomposition in order to eliminate the contribution of delayed hydraulic reactions. Representative cases studies showing the effectiveness of our procedures in radiocarbon dating are presented.

[1] J. Hale *et.al, Am. Sci.*, vol. 91, pp. 130–137, 2003.
[2] G. Ricci *et al.*, *Radiocarbon*, vol. 62, no. 3, 2020.
[3] M. B. Toffolo *et.al. Radiocarbon*, vol. 62, no. 3, pp. 545–564, 2020
[4] M. B. Toffolo *et.al. Sci. Rep.*, vol. 9, no. 1, 2019.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

OSL dating of lime and earth mortars: a review of seven-years dating historical buildings in NW Spain

Sanjurjo-Sánchez, J.[1], Blanco-Rotea, R.[2], Sánchez-Pardo, J.C.[2] García-Quintela, M.[2]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]Facultade de Historia, Universidade de Santiago de Compostela, Praza da Universidade, 1, 15703, Santiago de Compostela, Spain

Keywords:

Dating the construction of historical buildings is possible using historical written sources, building or decorative stylistic characteristic of different periods, or the relation with any archaeological or historical objects. However, they are not always available or clear enough. In such cases complementary or alternative dating techniques are needed to know the chronology of buildings. Often, materials such as bricks or timber were used for this purpose, but they did not always provide reliable results, because they can be re-used or substituted due to decay, providing ages that do not fit the period of construction. Therefore, mortars have caught the attention of researchers. Mortar making is contemporary to the construction moment of each specific area of the building. Both AMS radiocarbon and optically stimulated luminescence (OSL) have been used for dating mortars although routine procedures for dating still have not been established. Radiocarbon can be only used for lime mortars as the lime is used for dating, while OSL can be applied on other types of mortars, as the quartz of the aggregate is used.

The OSL signal increases during time, due to the exposure of the quartz grains to ionizing radiation. Such radiation comes from the radioisotopes (mainly from ^{238}U and ^{232}Th decay-chains and ^{40}K) present in the mortar and surrounding materials, as well as from cosmic radiation. But, OSL dating also requires that the optical geological signal of quartz grains is bleached during the mortar manufacture, due to daylight exposition. This usually occur after a few seconds of exposition. However, sometimes the daylight exposition is not enough to bleach de geological signal. In such cases, the mortar age can be overestimated. This implies the use of some strategies to get a reliable age although this is not always possible.

A research group from the University of A Coruña is deeply involved in OSL dating of mortars. In the last five years, the group have participated in archaeological projects carried out in the NW of Spain, with the purpose of getting an absolute chronology for a number of problematic and long-debated buildings by using OSL dating. Lime and earthen mortars have been dated as a result of such work. We review and detail here the key questions of the method and the results obtained for buildings during these projects.

S2-O03.341

Archaeomagnetic Synchronization of Biblical Military Campaigns

Vaknin, Y.[1,2], Shaar, R.[2], Lipschits, O.[1], Ben-Yosef, E.[1]

[1]*Institute of Archaeology, Tel Aviv University, Tel Aviv, Israel*
[2]*Institute of Earth Sciences, The Hebrew University of Jerusalem, Jerusalem, Israel*

Keywords: Archaeomagnetism; Chronology: Synchronization; Dating; Site-formation;

Near Eastern texts including the Hebrew Bible refer to Egyptian, Aramean, Assyrian and Babylonian military campaigns to the Southern Levant during the 10th-6th centuries BCE. Archaeological excavations in this region have indeed exposed many destruction layers dated to this period. A few of these destruction layers are securely linked to specific historical events and are therefore accepted as chronological anchors. However, debates regarding the dating of many other destruction layers pose dificulties in reconstructing the different military campaigns and question the historicity of the biblical narrative. Here we present archaeomagnetic synchronization of debated destruction layers and other artifacts with historically-dated chronological anchors. The synchronization is based on the intensity and/or direction of the ancient geomagnetic field recorded in 21 burnt destruction layers unearthed in 18 archaeological sites and in two ceramic assemblages. In the destruction layers the acquisition of the magnetic information had occurred at a discrete point in time, when sun-dried mud brick walls were burnt as a result of military campaigns. In some cases we used archaeomagnetism in order to reconstruct site-formation processes, securely linking the recording of the magnetic information to the destruction of structures. The historically based high resolution dating of the chronological anchors enabled us to use the magnetic data from these destruction layers as a skeleton for the Levantine archaeomagnetic curve for this period. The curve can now be used as a reliable dating tool. For example, the archaeomagnetic dating of the destruction of massive structures of public nature in Stratum S-1a at Tel Beth-Shean showed that they had been destroyed almost a century earlier than previously assumed. The archaeomagnetic dating using the new curve is particularly useful when dealing with the period after ~800 BCE, when a plateau in the radiocarbon calibration curve limits high resolution dating. Rapid changes and high intensity values characterized the magnetic field during the period in question in this region. This period included several spikes of more than twice the intensity of today's field. Our new data enabled us to chronologically constrain these short-term variations and resolve chronological debates regarding the violent campaigns against the kingdoms of Israel and Judah, the relationship between these kingdoms and their administrations.

S2-O04.1565

Luminescence and geochemistry applied to chronology and fill dynamic studies of negative archaeological structures

Rodrigues, A. L.[1], Dias, M.I.[1,2], Valera, A. C.[3,4], Rocha, F.[5,6], Prudêncio, M. I.[1,2], Marques, R.[1,2], Russo, D.[1,2]

[1]Centro de Ciências e Tecnologias Nucleares (C2TN), Instituto Superior Técnico, Univ. Lisboa, E.N.10 (km 139,7), 2695-066 Bobadela, Portugal
[2]Departamento de Engenharia e Ciências Nucleares (DECN), Instituto Superior Técnico, Univ. Lisboa, E.N.10 (km 139,7), 2695-066 Bobadela, Portugal
[3]ERA Arqueologia S.A., Calçada de Santa Catarina, 9C, 1495-705 Cruz Quebrada – Dafundo, Portugal
[4]Interdisciplinary Center for Archaeology and Evolution of Human Behavior (ICArHEB), Univ. Algarve, Campo de Gambelas, Faro, Portugal
[5]GeoBioTec, Univ. Aveiro, Campus Universitário de Santiago, 3810-193 Aveiro, Portugal
[6]Departamento de Geociências, Univ. Aveiro, Campus Universitário de Santiago, 3810-193 Aveiro, Portugal

Keywords: Luminescence dating; Geochemistry; Fill dynamic; Accumulation rate; Negative archaeological structures

The ditched enclosures comprise one of the most common and important architectures of recent Pre-history. These enclosed spaces, delimited by lines of excavated ditches and associated pits (negative archaeological structures), appear in different topographies and geological contexts, and have “life” time of occupation that can ranges from a few hundred to a thousand and a half years. Many of the them have particularities (sun orientation, topography, architecture, sequential fill with anthropogenic actions and selected materials, and a complex stratigraphy with events of opening, sealing and reopening) pointing to purposes related to symbolic and ceremonial practices. This work intends to show how an interdisciplinary approach is crucial for a better understanding of the dynamics of these structures. For this purpose, archaeological data together with dosimetry and absolute dating by luminescence, and compositional studies comprising chemical (neutron activation analyses and X-ray fluorescence) and mineralogical analyses (X-ray diffraction) was used. Negative archaeological structures of Pre-historic archaeological sites from southern Portugal were selected. Despite the intense work carried out on these archaeological sites, problematic questions still arise: difficulty in directly dating the opening of structures; rate of infill processes; existence of numerous negative structures, with unknown chronological sequence of the periods of construction, occupation and abandonment, as well as with archaeological evidence of re-excavation and reuse of spaces and materials. In this sense, new archaeological questions related to the nature and dynamics of the filling of these structures are arising. Stratigraphic detailed studies using the above-mentioned methods were applied to the fill materials and to the materials from corresponding geological contexts. The main results enhance the success of the used methodological approach, since clearly establish the nature and deposition rate of the fill materials, the definition of accumulation phases and chronology of the events, and infer about human/natural actions involved in each event/phase. Therefore, a contribution for a comprehensive discussion of the role of these structures in Southern Iberia Pre-historic times is achieved.

S2-O05.3002

Session 2 Archaeochronometry (organic and inorganic materials)

POSTER

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

An Archaeomagnetic investigation of Japanese baked clay artefacts.

Santos, Y.[1], Tema, E.[2,3], Trindade, R.[4], Hartmann, G.[5], Hatakeyama, T.[6], Matsumoto, N.[7], Mitsumoto, J.[8], Gulmini, M.[1]

[1]Dipartimento di Chimica, Università degli Studi di Torino, via Pietro Giuria 5, 10125 Torino, Italy
[2]Dipartimento di Scienze della Terra, Università degli Studi di Torino, via Valperga Caluso 35, 10125 Torino, Italy
[3]ALP-CIMaN Alpine Palaeomagnetic Laboratory, via Luigi Massa 6, 12016 Peveragno, Italy
[4]Departamento de Geofísica, Instituto de Astronomia, Geofísica e Ciências Atmosféricas, Universidade de São Paulo, Rua do Matão, 1226, 05508-090, São Paulo, Brazil
[5]Instituto de Geociências, Universidade Estadual de Campinas, Rua Carlos Gomes, 250, 13083-855, Campinas, Brazil
[6]Information Processing Center, Okayama University of Science, Ridaicho, 700-0005 Okayama, Japan
[7]Research Institute for the Dynamics of Civilizations, Okayama University, Kita-ku, Okayama 700-8530 Okayama, Japan
[8]Graduate School of Humanities and Social Sciences, Okayama University, Kita-ku, Okayama 700-8530 Okayama, Japan

Keywords: archaeointensity; baked clay; Japan; kofun; late yayoi

We present new archaeointensity data obtained from the analysis of baked clay artefacts from Okayama Prefecture (Japan), dated from 150 AD to 675AD, which refers to the Late Yayoi and Kofun periods. The studied samples are pottery, haniwa figures and coffin fragments from the archaeological sites of Tenguyama, Nima Ohtsuka, Tatetski, Tatezaka, Sada Higashizuka and Sada Nishizuka. Samples with higher magnetic stability, as shown in preliminary thermal demagnetization, were chosen for archeointensity analysis. Archaeointensities were determined using the Thellier-Thellier protocol as modified by Coe, including partial thermoremanent magnetization (pTRM) and pTRM-tail checks and corrections for anisotropy and cooling rate. To enrich the Japanese reference dataset and contribute to the reconstruction of the ancient geomagnetic field intensity in Japan during the Late Yayoi and Kofun periods, we obtained high quality results. Following strict quality selection criteria, from the 145 studied specimens, 99 (coming from 30 independent fragments) gave successful archaeointensity determinations, resulting in a success rate of 68%. The recently acquired data was compared to previously published archaeointensity records from Japan and to global magnetic field models predictions. Such comparison shows that the new data are characterized by much lower intensity values in respect to the previously available data, underlining the need for more high-quality data for the area. The new results are an important contribution to improve of our knowledge about the eld secular variation of the intensity of the geomagnetic field in Japan.

S2-P02.1537

^{14}C preparation protocols for archaeological samples at the LMC14, Saclay, France.

Dumoulin, J.P., Caffy, I., Delqué-Količ, E., Goulas, C., Messager, C., Moreau, C, Perron, M., Sieudat, M., Tellier, B., Beck, L.

Laboratoire de Mesure du Carbone 14 (LMC14), LSCE/IPSL, CEA-CNRS-UVSQ, Université Paris-Saclay, F-91191 Gif-sur-Yvette, France

Keywords: Radiocarbon dating; cellulose extraction; rock art; mortar; lead white

The LMC14 (Laboratoire de Mesure du Carbone 14) and its AMS Artemis is the national facility dedicated to high-precision radiocarbon measurement for five French institutions (CNRS, CEA, IRD, IRSN, Ministère de la Culture). Around 4000 samples are prepared and measured every year for different types of organic matters and $CaCO_3$ samples (Dumoulin et al 2017). The radiocarbon analysis of very small samples (below 0.2 mg of carbon) has also been explored (*Delqué-Kolic et al. 2013*) and modified graphitization procedures combined with a specific protocol of measurement are now employed when the carbon content is low (Moreau et al 2020). Samples like archaeological iron (*Leroy et al. 2015*) as well as water (*Dumoulin et al. 2013*) are also analyzed. For 2017, we have developed new protocols to widen our range of datable samples and take into account the expectations of new archaeological projects. The specific protocols developed for cellulose extraction (wood wiggle matching), oxalates (rock art), mortar (buildings) or lead white (cosmetics and paintings) dating will be detailed. The ^{14}C results obtained for artifacts and international standards containing various carbon contents will be presented for each type of samples.

Delqué-Količ E, Caffy I, Comby-Zerbino C, Dumoulin J P, Hain S, Massault M, Moreau C, et al. 2013. « Advances in Handling Small Radiocarbon Samples at the Laboratoire de Mesure du Carbone 14 in Saclay, France ». *Radiocarbon; Vol 55, No 2–3 (2013)*.

Dumoulin J P, Caffy I, Comby-Zerbino C, Delqué-Količ E, Hain S, Massault M, Moreau C, et al. 2013. « Development of a Line for Dissolved Inorganic Carbon Extraction at LMC14 Artemis Laboratory in Saclay, France ». *Radiocarbon; Vol 55, No 2–3 (2013)*.

Dumoulin JP, Comby-Zerbino C, Delqué-Količ E, Moreau C, Caffy I, Hain S, Perron M, Thellier B, Setti V, Berthier B and Beck L. (2017). Status report on sample preparation protocols developed at the LMC14 Laboratory, Saclay, France: from sample collection to 14C AMS measurement. Radiocarbon 59: 713-726.

Moreau C et al. (2020). ARTEMIS, the 14C AMS facility of the LMC14 National Laboratory: a status report on quality control and microsample procedures. Radiocarbon, 62(6), 1755-1770. doi:10.1017/RDC.2020.7

Leroy S, L'Héritier M, Delqué-Kolic E, Dumoulin J P, Moreau C, et Dillmann P. 2015. « Consolidation or initial design? Radiocarbon dating of ancient iron alloys sheds light on the reinforcements of French Gothic Cathedrals ». *Journal of Archaeological Science* 53 (2015): 190-201.

S2-P03.1538

Comparative Dating of Charcoal Rock Art Samples Using AMS Radiocarbon Analysis and Plasma-Chemical Oxidation

Peterkin, I.[1], Armitage, R.A.[1], Bonneau, A.[2], Pearce, D.[3], Southon, J.[4]

[1]Department of Chemistry, Eastern Michigan University, Ypsilanti, MI, USA
[2]Departments of Chemistry and History, Universite de Sherbrooke, Sherbrooke, QC, Canada
[3]Rock Art Research Institute, University of Witwatersrand, Johannesburg, South Africa
[4]Keck Carbon Cycle AMS, Department of Earth System Science, University of California, Irvine, CA, USA

Keywords: Rock art; Radiocarbon Dating; Plasma-Chemical Oxidation; AMS; South Africa

Generally, radiocarbon dating requires the complete combustion of an organic sample like charcoal to form carbon dioxide, which can then be further processed into graphite for analysis by accelerator mass spectrometry (AMS). Combustion is a destructive process that requires relatively large samples which must be treated with acid to remove carbonates and oxalates and leaves no material remaining for further analysis or dating. Plasma-chemical oxidation (PCO) is an alternative to combustion to form CO_2 via a low-temperature chemical reaction, which generates microgram samples of carbon as CO_2 for dating. Any organic matter, including contaminants from the soil such as humic acids or microbes, will react with the oxygen plasma and can contribute error to the measured age; thus, they must be removed prior to plasma oxidation. This project focuses on developing minimally-destructive pretreatments to remove these contaminants, and investigating their effect on the measured dates for previously dated materials. Comparing dates obtained with plasma-chemical oxidation sample preparation for rock paintings to measured ages from a more conventional combustion process is a significant step in the validation of these methods. We present here our results for plasma-chemical oxidation of rock paintings from RSA TYN2 in South Africa that were dated previously with a modified conventional pretreatment protocol. The samples were pretreated to remove humics in the same manner as before, and then plasma oxidized to yield carbon dioxide. Radiocarbon analysis by AMS is currently underway at the Keck CCAMS laboratory. This is the first time the same rock art samples have been independently prepared and dated by these two methods. Knowing the age of rock paintings is important for interpreting when they were created and to place them within the history of the populations who created them.

S2-P04.1577

OSL dating of the Lomba do Mouro Roman camp (Melgaço, northern Portugal)

Fonte, J.[1], Rodrigues, A.L.[2], Dias, M.I. [2,3], Russo, D [2,3], Oltean, I.[1]

[1]Department of Archaeology, University of Exeter
[2]Centro de Ciências e Tecnologias Nucleares (C2TN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal
[3]Departamento de Engenharia e Ciências Nucleares (DECN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal

Keywords: Lomba do Mouro; Roman camp; archaeological survey; luminescence dating.

Lomba do Mouro is a massive enclosure of c. 25 ha delimited by two stone walls. This site is in the Laboreiro Mountain and is divided by the border between Portugal (Castro Laboreiro, Melgaço) and Galicia (Verea, Ourense). An archaeological survey has taken place in this site in the summer of 2020 in the scope of the *Finisterrae* project (Marie Skłodowska-Curie Individual Fellowship, grant agreement 794048) and through a collaboration with the Melgaço Municipality, the Era-Arqueologia company, the Romanarmy.eu research collective and the Center for Nuclear Sciences and Technologies (C^2TN). The main objective was the archaeological characterization and the absolute dating of its defensive system. This survey allowed us to understand how the inner and outer stone walls were built and especially to recover sediment samples in contexts sealed by the walls. These were dated through optically stimulated luminescence (OSL), applying SAR protocol to coarse quartz grains (160-250 µm). Results point to a construction date of both walls around the 2nd century BC, albeit with some degree of uncertainty. The uncertainties are a consequence of absorbed dose dispersion motivated by an incomplete bleach of the quartz geological signal. This phenomenon is in accordance with a quick event of sun exposure of these sediments due to the construction of the ramparts in a brief period by a large military contingent. Its size, morphology, location, approximate dating and related historical written accounts are consistent with a late-Republican Roman military camp.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeomagnetic analysis of a burnt clay structure in ancient Philipopolis (Bulgaria)

Lesigyarski, D.[1], Kostadinova-Avramova, M.[1], Dimitrov, P.[1,2]

[1]National Institute of Geophysics, Geodesy and Geography – BAS, 3, Acad. G. Bonchev Str., 1113 Sofia, Bulgaria
[2]National Archaeological Institute with Museum – BAS, 2, Saborna Str., 1000 Sofia, Bulgaria

Keywords: archaeomagnetism; dating; clay; oven; firing temperatures

Although less popular than 14C dating, archaeomagnetic method allows acquiring of precise absolute dating intervals. One of its great advantages is that materials analysed are commonly found in significant amounts during archaeological excavations – structures made of baked clay, like ovens, burnt floors, plasters, bricks, ceramic fragments etc. Crucial for this analysis is determination of the intensity and direction (inclination and declination) of the ancient geomagnetic field recorded by the magnetic minerals contained in the baked clay. Besides the absolute dating, archaeomagnetic measurements can be applied as a reliable tool for the determination of maximum firing temperatures or even to establish possible displacement of the original in situ position of the structures in question. In this study a set of independently oriented samples from burnt adobe found in the forum place of ancient Philipopolis (nowadays city of Plovdiv) in Bulgaria is analysed. According to the archaeological evidence the adobe is dated to the 1st century CE, however its interpretation is hampered by a complex stratigraphy and finding of the structure in an unusual place and position. Laboratory measurements performed include various rock-magnetic analyses which allow making conclusions about the magnetic minerals contained in the specimens.
The measured inclinational and declination values undoubtedly indicate that that the studied structure has not cooled down in the position revealed by the archaeologists, i.e. it collapsed after its firing. This could explain its unusual place in the context of stratigraphy. On the other hand, the evident displacement of the structure do not allow any result for the direction of the ancient geomagnetic field to be obtained and used for archaeomagnetic dating. Thus, only the intensity values remain to determinate date interval. Using only one element of the field makes absolute dating difficult because it increases the number of possible date intervals. This confirms the importance of the simultaneous use of all three elements of the field for the purpose of archaeomagnetic dating. In this case, however, in the time interval suggested by archaeological observations, a very good coincidence is found between archaeological and archaeomagnetic dating.

Acknowledgements: This study is financed by the National Science Fund of Bulgaria under the contract No. КП-06-Н30/2.

S2-P10.317

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chronology and architecture of early medieval churches in Northwest Spain. Results of the EMCHAHE project

Sanjurjo-Sánchez, J.[1], Blanco-Rotea, R.[2], Sánchez-Pardo, J.C.[2]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]Facultade de Historia, Universidade de Santiago de Compostela, Praza da Universidade, 1, 15703, Santiago de Compostela, Spain

Keywords: OSL; ^{14}C; architecture; early medieval churches NW Spain

This presentation will show the main results of the Marie Curie-CIG project “Early Medieval Churches: History, Archaeology in Heritage” which has been running since 2014. The project focus on the early medieval churches of Galicia (Northwest Spain) and has carried out, for the first time, a global approach to the architecture of Christian buildings between 5th to 10th centuries in this region, by means of a combination of Archaeology of Architecture, Optically Stimulated Luminescence and radiocarbon mortar dating, as well as GIS compilation and analysis of all the documentary references to churches before the year 1000 AD and all the published decontextualized architectural elements of late antique and preromanesque churches in Galicia.

In a first stage of the project, a corpus of textual and material evidence of more than 800 early medieval churches in this region was created. Basing on this database, an extensive “architectural survey” was carried out in order to detect possible early medieval walls in current standing churches. Once completed the survey, twelve case study were selected for stratigraphical and chronotipological analysis and OSL and radiocarbon dating of their mortars, in addition to its characterization.

The results of this research, with a total of 20 intensive case study (including long-debated churches like Santa Comba de Bande) allow us not only to gain new knowledge about the chronology and typology of these churches but also about the dynamics of social power that lie behind their foundation in this period.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Minimally-destructive Plasma Oxidation and AMS Radiocarbon Dating of Archaeological Textile Fragments from the Seip Mound Complex, Ohio

Armitage, R.A.[1], Repaska, M.[1], Wilson, B.[1], Peterkin, I.[1], Jakes, K.[2], Southon, J.[3]

[1]Department of Chemistry, Eastern Michigan University, Ypsilanti, MI 48197 USA
[2]Professor Emerita, The Ohio State University, Columbus, OH 43210 USA
[3]Keck Carbon Cycle AMS, Department of Earth System Science, University of California, Irvine, CA 92697 USA

Keywords: Middle Woodland; Hopewell; textile; AMS radiocarbon; plasma oxidation

Ohio Hopewell archaeological sites, and the Seip Mound complex in particular, are well known for their preserved textiles. Though what remains today is mostly fragmentary, degraded or charred, these precious remnants are indicative of the complex craftsmanship of the Hopewell. Modern analytical and dating techniques allow for characterization and chronological studies of these materials that have not been possible in the past. Segments of what was thought to be a fabric canopy with repeating patterns of green coloration were recovered during early 20th-century excavations at Seip-Pricer. Subsequent investigations of smaller fragments sharing the same physical characteristics as the canopy fabric indicate that it is made from plant fibers, though it remains unclear how the material was decorated. Minute samples were collected, taken from small bits of yarn that appear to have fallen away from the larger fabric fragments. These samples provided a unique opportunity for chemical characterization and radiocarbon dating of textile materials from Seip. A variety of analytical methods were used to characterize the green coloration and to determine if a binding medium was present, in an effort to better understand the process used to decorate the textile. Minimally-destructive sample preparation with pH 8 phosphate buffer and plasma oxidation was carried out on fragments of the textile as well as on a small sample of a charred textile also from the Seip complex. Carbon dioxide produced by the plasma oxidation process was then subjected to graphitization and AMS radiocarbon analysis. An initial radiocarbon age for the green stained textile was found to be consistent with previous dates for Seip, though at the earliest edge of the measured ranges; the possible influence of the copper-carbonate pigment on the date will be discussed. The first measured age for the charred textile was not consistent with the expected range for the site, perhaps indicating that the cleaning protocol was insufficient or that it originated from a later intrusion. We will report our most recent dates for these objects herein. These results demonstrate how such miniscule fragments of ancient textiles can be utilized to understand Hopewell technologies and place these human-made objects into the chronology of the Seip Mound complex.

Ramped Pyroxidation Radiocarbon Dating of a Preservative Contaminated Early Medieval Irish Wooden (Alder) Bowl

Barrett, G.[1], Keaveney, E.[1], Reimer, P.J.[1], Mullarkey, P.[2], Seaver, M.[2]

[1] [14]CHRONO Centre for Climate, the Environment and Chronology, School of Natural and Built Environment, Queen's University Belfast

[2] National Museum of Ireland, Kildare Street, Dublin 2

Keywords:

Organic artefacts from museum or archaeological collections have often had preservatives applied that make it difficult or impossible to obtain accurate radiocarbon dates. Discovered in an Irish bog in the 1960s, the *Cuillard Bowl* is a triangular wooden (Alder - *Alnus Glutinosa*) dish that is of special interest chronologically on account of its unique geometry and style. However, due to the persistence of carbon-rich preservatives (polyethylene glycol, PEG), radiocarbon dating following conventional pre-treatment methods has produced problematic results.

A new ramped pyroxidation (RPO) facility has recently been constructed at the [14]CHRONO Centre. In RPO, samples are incrementally heated and CO_2 is produced as the sample undergoes thermal decomposition in the presence (oxidation) or absence (pyrolysis) of oxygen. The CO_2 evolved from different temperature fractions are collected cryogenically and radiocarbon dated. This method was applied to remaining pre-treated material from the Cuillard bowl to investigate if a preservative-free CO_2 fraction could be isolated for radiocarbon dating; this would work by taking advantage of the evolution of CO_2 from wood cellulose at lower temperatures than CO_2 evolves from PEG.

Radiocarbon dates produced on low temperature fractions of the wood produced statistically identical ages that calibrate to the mid-6th to mid-7th centuries AD, in agreement with stylistic interpretations. Higher temperature fractions were statistically different and older due to the removal of CO_2 derived from the contaminant preservative. Additional RPO and infrared-spectroscopy (FTIR) results on non-pretreated Cuillard bowl material, early medieval alder of known age (dendro dated), and modern alder are presented to highlight the reliability of low temperature fractions being free from contaminants and providing an accurate age for the wood from the bowl.

S2-P13.372

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Radiocarbon Dating Mortar: the identification of a lost medieval Irish round Tower using a multi-method inter-comparative approach

Barrett, G.[1], Donnelly, C.[2], Reimer, P.J.[1]

[1 14]CHRONO Centre for Climate, the Environment and Chronology, School of Natural and Built Environment, Queen's University Belfast

[2]Centre for Archaeological Fieldwork, School of Natural and Built Environment, Queen's University Belfast

Keywords:

The remains of a 17th century windmill in the city of Derry, N. Ireland, has long been suspected of being composed of the structure of a medieval Irish round tower. If true, this would be the only extant medieval building in Derry and the last remnant of what was a major Irish ecclesiastical centre.

The age of the building was recently examined through the application of radiocarbon dating of mortar to a sample retrieved from the structure during conservation work. Multiple sample preparation methods (cryo-breaking, mechanical, suspension) were used to isolate a series of different lime binder particle size fractions. Combined with sequential dissolution and subsequent radiocarbon dating this permitted the presence of multiple carbonate components to be identified. Aided by inter-comparison of the dissolution profiles from different methods and fractions sizes, those components and dates associated with the setting of the mortar could be separated. Furthermore, by assessing the efficacy of preparation methods through application of particle size analysis, x-ray diffraction, thermogravimetric analysis and Fourier transform infrared spectroscopy, interpretation of the results was further strengthened.

The results provide strong evidence that the building contains the remains of, at latest, a 13th-14th century Irish round tower. This would confirm it as the only medieval structure surviving from the city's monastic period and of great cultural and historical significance. The use of a multi-method mortar radiocarbon dating approach has been successfully applied and has the potential for broader applications to understanding building chronologies elsewhere.

S2-P14.373

AMS radiocarbon dating of lead white pigment

Beck, L.[1], Messager, C.[1], Caffy, I[1], Batur, K.[2], Degrigny, C.[3], Serneels, V.[4], Stols-Witlox, M.[5]

[1]Laboratoire de Mesure du Carbone 14 (LMC14), LSCE/IPSL, CEA-CNRS-UVSQ, Université Paris-Saclay, Gif-sur-Yvette, France
[2]University of Zadar, Department of Archaeology, 23 000 Zadar, Croatia
[3]Sarl Germolles, Château de Germolles, 100 place du 05 septembre 1944, 71640 Mellecey, France
[4]Université de Fribourg, Département de Géosciences, Chemin du Musée 6, 1700 Fribourg, Switzerland
[5]University of Amsterdam, Programme Conservation and Restoration of Cultural Heritage, Amsterdam, The Netherlands

Keywords: radiocarbon; lead white; cosmetic; painting; ^{14}C

The radiocarbon dating method is usually applied to organic materials - wood, charcoal, bone, etc. - which absorb radioactive carbon through photosynthesis or ingestion. However, a few studies have shown that radiocarbon dating can also reveal the age of inorganic manufactured materials. As early as the 1960s, ^{14}C dating was extended to mortar which absorbs atmospheric carbon dioxide as it hardens and more recently to iron, by combining chemical analysis and radiocarbon measurements.
Because of its opacity in oil, lead white was the main white pigment for house painters and artists from Antiquity to the 20th century. This mineral pigment contains two lead carbonates: cerussite $PbCO_3$ and hydrocerussite $2PbCO_3Pb(OH)_2$. Lead white was produced in a corrosion process involving metallic lead, vinegar and horse manure. Manure fermentation delivered heat needed to evaporate the vinegar, which reacted with the lead to form lead acetate, and released CO_2 required for the formation of lead carbonates from lead acetate. As CO_2 carries the atmospheric ^{14}C signature, we can assume that atmospheric ^{14}C is incorporated in the lead carbonates and that lead white can thus be dated by the radiocarbon method.
In this paper, we use lead white containing paints preserved in well-dated monuments, cerussite cosmetics and lead white pigments from known archaeological contexts to assess possibilities for radiocarbon dating. The results indicate that the methods can successfully date samples from objects as diverse as Greek cosmetics, Roman *collyria*, Medieval and Renaissance paintings and pigments, concerning a large scale of time ranging from the 4th century BC to the 20th century AD.

This study demonstrates that the radiocarbon dating method can be extended to inorganic materials such as lead white. This new application allows us to add important details to the investigation of the history of lead white production and provides a new tool for the authentication of cultural heritage objects.

S2-P17.383

Dating Early Iron Age sites in the SE Alpine region: first archaeomagnetic data for Slovenia

Harris, S.E.[1], Batt, C.M.[1], Črešnar, M.[2,3], Vinazza, M.[2]

[1]School of Archaeological and Forensic Sciences, University of Bradford, Bradford, UK
[2]Centre for Interdisciplinary Research in Archaeology, Department of Archaeology, Faculty of Arts, University of Ljubljana, Ljubljana, Slovenia
[3]Institute for the Protection of Cultural Heritage of Slovenia, Ljubljana, Slovenia

Keywords: Archaeomagnetism; Iron Age Europe; Dating

Archaeomagnetic studies offer the opportunity to date in situ fired archaeological structures (e.g. furnaces, kilns, domestic fires) based on their magnetic properties. The technique is a well-established scientific dating method utilised across numerous countries, particularly favoured as the dated event has a direct link to anthropogenic activity. This poster presents the first archaeomagnetic data for Slovenia.

In previous years, archaeomagnetic dating has been hindered in regions which lack data for calibration curves. Thanks to recent advances in global modelling of the geomagnetic field, it has become possible to use archaeomagnetic dating in countries with the lack of regional data, such as Slovenia. The overarching aim of this research was to use archaeomagnetic studies to date the occupation of two of Slovenia's most systematically researched Early Iron Age complexes. The Cvinger (near Dolenjske Toplice) archaeological complex is composed of a large hillfort with the largest until now discovered Early Iron Age iron smelting area in the region. The additional site sampled at Čreta (near Slivnica) shows little re-use after its prehistoric occupation with well-preserved stratigraphy. A total of 124 samples were taken across five fired archaeological features and subjected to a full suite of archaeomagnetic analyses to characterise the magnetic mineralogy and date the last use of each feature. Archaeomagnetic dating of these sites is considered an important step in dating these significant sites; especially as they lack typologically datable archaeological finds and calibration of radiocarbon determinations for the early Iron Age often falls on the Hallstatt plateau (resulting in wide calibrated date ranges).

The archaeomagnetic studies carried out for these two important early Iron Age sites have shown that the presented dating method has the potential to provide higher precision than radiocarbon dating. The clear anthropogenic link this dating tool offers allows a more nuanced understanding of these complexes. Furthermore, this research has produced the first archaeomagnetic data for the Early Iron Age in Slovenia.

S2-P18.387

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Lead isotopic (in)homogeneity of ancient copper alloys and reference materials in Archaeometry

Strnad, L.[1], Trubac J.[2], Vondrovicova L.[2], Danielisova A.[3]

[1]*Laboratories of The Geological Institutes, Charles University, Czechia*
[2]*Institute of Geochemistry, Mineralogy and Mineral resources, Charles University, Czechia*
[3]*Institute of Archaeology of the Czech Academy of Sciences, Czechia*

Keywords: bronzes; lead isotopes; reference materials; analytical error; MC-ICP-MS

Generally, ancient bronzes exhibit large variability in the compositions of the major (Cu, Sn, Pb) and minor elements (Zn, Ag, Fe, Ni, Co, As, Sb). The lead content usually varies substantially from almost zero to 30 wt% Pb. We tested the variability of the Pb isotope composition within single artefacts in several non-ferrous metal samples from the early Roman period in the Czech Republic. In addition, significant shifts and variations in the lead isotopic composition in one sample would have a major impact on the consistency and ultimately on the interpretation of the data obtained. It should be noted that many trends in lead isotope composition could be reflected in a larger RSD error during MC-ICP-MS measurements. This is especially true of the 206Pb/204Pb and 207Pb/204Pb ratios, whose relative analytical error (RSD ≤ 0.04 %) is up to one order of magnitude greater than for the 207Pb/206Pb ratios (RSD ≤ 0.005 %). This fact reflects the lowest naturally abundance of the 204Pb isotope. Therefore, it is important to depict the values in plots with error bars, making the information much clearer and assisting in the interpretation.
Many geological and archaeological applications require high-precision lead isotopic data measured by sensitive instrumentation with a magnetic field separator (e.g. MC-ICP-MS, TIMS). It is recommended that certified reference materials be used to verify the validity (e.g. accuracy) of the isotopic data and the entire analytical procedure. For archaeological materials, this could be complicated by insufficient availability in scientific communities. We performed homogeneity testing on 207Pb/206Pb, 208Pb/206Pb and 206Pb/204Pb ratios for the three geological CCRMP reference materials (CPB-2, CCU-1e and MP-1b) measured by MC-ICP-MS (with a bronze artefact-like matrix) and determined preliminary values for these reference materials, which are commercially available.

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A complex archaeometric approach to study a museum collection

Nikolova, A.

NIGGG BAS, Paleo- and Archaeomagnetic laboratory

Keywords: anchors; lead; Black sea; archaeometry; corrosion

The poster is presenting a current work on the museum collection “History of anchor”, Achtopol, Bulgaria. The principal aim of this study was to determine the reasons for the difference between the corrosion of antique anchor stocks with a central box surrounding a shank and other types of stocks made of lead. For the purpose the mineral composition of corrosion of twenty objects was analyzed with XRD (X-ray diffraction) and the chemical composition was determined with ICP-AES (Inductively coupled plasma atomic emission spectroscopy). The metallography was applied to reveal the microcomposition and a probable technological reason for the differences. The wood in a core of the anchor stocks type IIIC Haldane was examined - quercus sp. and viburnum sp. The key for the answer was hidden in the interactions between metal and hard woods in the construction of the anchor stocks type IIIC Haldane. Despite the collection is representative for almost every type of ancient anchor stocks made of lead, there are just four with central box which are only of type IIIC Haldane (with a wooden element inside). Many species of timber can produce small amount of volatile organic acids, particularly acetic and formic acids, harmful for lead. The humidity that a wooden core holds inside the stock construction increases the corrosion.

These conclusions played a crucial role for the choice of appropriate objects and protocol for two experiments with absolute dating techniques, based on the mass of the corrosion layer and its composition, despite some mistakes in the data obtained from the first XRD. The method published by Reich et al. 2003 and radiocarbon dating AMS 14C (wooden part and lead carbonates) were applied for two objects. A most precise second analyze of trace elements was performed to be combined with the isotopic signatures in searching of the origins of the metal. The goal of these experimental steps was to enlarge the knowledge for these artifacts which can be a good source for the dynamics of coastal life, economy and relations. Their informative potential is usually underestimated because the archaeological context is often missing and sometimes even their provenance is not certain.

An interdisciplinary team was involved in the project.

This study is partly supported by the Bilateral Project KP-06-Russia-10 granted from the Bulgarian National Science Fund.

S2-P22.611

AMS radiocarbon dating of Gallo-Roman collyria composed of metallic carbonates

Messager, C., Beck, L., Caffy, I., Mussard, S., Perron, M., Van Hove, C., Delqué-Količ, E., Dumoulin, J.-P., Moreau C.

Laboratoire de Mesure du Carbone 14 (LMC14), LCSE/IPSL, CEA-CNRS-UVSQ, Université Paris-Saclay, Gif-sur-Yvette, France

Keywords: radiocarbon dating; lead carbonates; cosmetics; Gallo-Roman *collyria*

Radiocarbon dating has recently been applied to two synthesized inorganic compounds, cerussite and phosgenite [1]. These lead carbonates were used as ingredients for cosmetics and remedies in Antiquity. Cerussite has been mainly synthesized since 400 BC by the corrosion of metallic lead in the presence of vinegar and a fermenting environment such as horse manure [2]. The phosgenite synthesis is older and is based on litharge and salt in water reactants. In both cases, it has been shown that the compounds incorporate the ^{14}C isotopic signature of the natural organic CO_2 source used for their manufacture [1]. Therefore, they can be absolutely dated by radiocarbon. Our laboratory has developed a protocol for extracting carbon from synthesized lead carbonates by thermal decomposition to achieve their radiocarbon dating [3]. Greek cerussite and Egyptian phosgenite cosmetics have been successfully dated by ^{14}C.

In this paper, we propose to date Gallo-Roman *collyria* found in the tomb of “La Favorite” in Lyon, France. These *collyria*, have been identified and chemically characterized by M. Aubin during her PhD (2016). They are composed of a complex mixture of metallic carbonates of lead, zinc, and copper [4, 5]. The recipes reported by the authors of Antiquity mentioned the synthesis of a green copper carbonate-based pigment similar to that of cerussite [6]. However, no mention is made about the synthesis of zinc carbonates. We have adapted our extraction protocol to these complex objects, adding a chemical pre-treatment to eliminate possible contaminations. Thanks to this combination, the Gallo-roman *collyria* have been successfully dated by radiocarbon, and the results are in perfect agreement with the archaeological data. In this poster, our approach will be explained in detail.

[1] L. Beck, et al., Absolute dating of lead carbonates in ancient cosmetics by radiocarbon, *Communications Chemistry*, 1(34), 1-7 (2018)

[2] Theophrastus. *'Liber de lapidibus'*. Transcribed in: Early R. Caley, John F.C. Richards, Theophrastus on stones. Introduction, Greek text, English translation, and commentary, (Columbus, Ohio, Ohio State University Press, 1956)

[3] L. Beck, et al., Thermal decomposition of lead carbonate to date lead white pigment, *Radiocarbon*, 61(5), 1345-1356 (2019)

[4] R. Boyer, et al., Les collyres, Découverte de la tombe d’un oculiste à Lyon (fin du IIe s. après J.-C.) Instruments et coffret avec collyres In: Gallia, tome 47, 1990, pp. 235-243

[5] M. Aubin, Révéler la chimie des préparations antiques, à usage cosmétique ou médical, impliquant des sels de métaux, PhD Thesis, Université Pierre et Marie Curie, Paris, 2016 (directors L. Bello-Gurlet and P. Walter)

[6] Pline, Natural History, 34, 26

S2-P23.612

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Assessing Portable Optically Stimulated Luminescence (pOSL) for the analysis of the formation of agricultural terraces and lynchets

Fallu, D.J.[1], Brown, A.G.[1,2]

[1]The Arctic University Museum of Norway, Tromsø
[2]Geography & Environmental Science, University of Southampton, UK

Keywords: Portable OSL, Stratigraphy, Agriculture, Geoarchaeology

Agricultural terraces and lynchets (unwalled terraces created through ploughing and erosion) are a conspicuous and nearly ubiquitous but poorly understood feature of human-altered landscapes. Bioturbation, ploughing, and re-use all contribute to the mixing of soil and the blurring of stratigraphic boundaries. Common dating methods (14C and OSL),in addition to being adversely affected by ploughing, are too costly to adequately cover the complex formational histories of terraces. Chemostratigraphy, the practice of discerning depositional units via elemental or molecular analysis, has proven useful in defining depositional histories, but is too sensitive to changes caused by soil formation, which may obscure sedimentological signatures. Pulsed Stimulated Luminescence, or Portable OSL, offers an inexpensive and reliable alternative to chemostratigraphy when dealing with complex or otherwise invisible depositional histories.

For this study (part of the ERC-funded TerrACE project), pOSL samples were taken from agricultural terraces and lynchets in England, Norway, and Sicily. The samples were taken using 2.5cm diameter aluminium tubes at 2.5 and 5 cm intervals. Samples were analysed using a SUERC Portable OSL reader to produce both OSL and IRSL signals. The same samples were then analysed for chemical composition using an Olympus Vanta M-Series Portable X-Ray Fluorescence (pXRF) Analyzer (GeoChem and Soil calibrations). OSL and IRSL results were then plotted over depth and compared to PCA and HCA analyses of the pXRF results for their ability to distinguish between the stratigraphic units identified in the field.

While pOSL does not provide calendar dates, the low cost of analysis allows for a near-continuous analysis of the terrace section, allowing for a high resolution relative dating of depositional events. In all cases, pOSL provided a more rapid method for verifying field-observed stratigraphy, as well as separating units which were less distinguishable in the field. In particular, PSL is less susceptible to the overprinting in chemostratigraphy caused by soil development. In the case of lynchets, gentle curves in the luminescence results may reflect gradual accumulation via ploughing, making pOSL an integral tool in discerning the use histories of agricultural landforms.

S2-P26.654

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Charcoal from different environmental conditions in context of pretreatment for ^{14}C measurements

Michalska, D., Szczepaniak, M.

[1]*Institute of Geology, Adam Mickiewicz University, ul. B. Krygowskiego 12, 61-680 Poznań, Poland*

Keywords: charcoal; pretreatment; 14C measurement; radiocarbon

Charcoal is one of the materials most frequently dated by radiocarbon method and relatively easy to prepare. However, the 14C measurements made on charcoal fragments sometimes give ages that are different than expected by archaeological or geological context. Experimental studies on selected group of samples from different sites and environmental conditions were shown. Each step of pretreatment was detailed monitored and weight loss was measured. Analyses were made on experimental samples of charcoals from mortars. Each of the pretreatment steps influence the groups of samples in different ways depending on the preservation state of charcoals and the environmental condition of their origin (Rebollo et al., 2008). The local geology has important meaning in context of preservation state of charcoal. Deposition of charcoals in favorable geological conditions, sands and gravels strongly differ from the organic-reach peat sands in context of their influence on sample.

Rebollo N.R., Cohen-Ofri I., Popovitz-Biro R., Bar-Yosef O., Meignen L., Goldberg P., Weiner S., Boaretto E. 2008: *Structural characterization of charcoal exposed to high and low pH: implications for 14C sample preparation and charcoal preservation*. Radiocarbon 50 (2): 289-307.

S2-P29.822

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archeology and geology: the traditional building materials of Braga, NW Iberian peninsula

Fragata, A.[1], Ribeiro, J.[2], Fontes, L.[2], Sanjurjo-Sánchez, J.[3], Fernández, A.[4],Rocha, F.[1]

[1]GeoBioTec, Department of Geosciences, University of Aveiro, Portugal
[2] Lab2Pt/IN2PAST, Institute of Social Sciences, University of Minho, Portugal
[3]Institute of Geology "Isidro Parga Pondal", University of Coruña, Spain
[4]Faculty of History, Department of Art, Geography and History, GEATT, University of Vigo, Spain

Keywords: Historic mortars, clayey ceramic materials, archaeology of construction, petrography techniques, OSL

Braga is a city located in the North of Portugal, in the NW Iberian Peninsula, with over 2000 years of history. Historic mortars and clayey ceramic materials have always assumed a fundamental role in the construction of the city in several contexts since its foundation, at the end of the first century BC, in Roman period (I-IV centuries), in the medieval period (V – XV centuries) and modern period (XVI-XVIII centuries). However, the construction technologies and the traditional building materials (i.e. mortars and clayey ceramic materials) adopted in those periods in Braga have not yet been systematically explored and studied.

This work presents an interdisciplinary project, with the collaboration of civil engineers, archaeologists and geologists, which aims to analyse the traditional building materials from relevant archaeological places, representative of the different historical periods and architectures, of the city and its territory, namely roman private and public buildings (houses, baths, necropolises, *villae*…), the city medieval wall/ towers, the Cathedral and the Convent of S. Francisco, *inter alia*, from petrographic, mineralogical and chemical point of view, supporting archaeological and historical research. The samples will be characterized employing archaeological tools and petrography techniques, X-ray power diffraction (XRD), X-ray fluorescence (XRF) and luminescence dating (OSL).

The final expected goal of this work, through the combination of all resulting data, is the establishment of the traditional building materials conservation state and provides adequate tools for future conservation works, in a time when sustainability and preservation of memory are becoming even more important. A comparative analysis will be made with other case studies from NW Iberian Peninsula. In relation to, exclusively, archaeological view, this kind of approach is surely a valuable tool as it gives more information about manufacturing processes, and generates chronological sequences, in order to interpret historical constructions.

S2-P31.878

Chuchuwaya: Man and Nature in Similkameen – Chronological modelling –

Quiles, A.[1], Allison M.[2], Clyburn A.[3], Delannoy J-J.[4], Jacquet, J.[4], Geneste J-M.[5], Gould, B.[6] Harris, M.[6], Marquet, P.[7], Rowley, S.[8]

[1]Institut Français d'Archéologie Orientale, Pôle Archéométrie, Cairo, Egypt
[2]Upper Similkameen Indian Band, Canada
[3]Independent researcher, Canada
[4] Université Savoie Mont-Blanc – Laboratoire EDYTEM- Bourget du Lac (France) et Institut Universitaire de France
[5]UMR 5199 CNRS Pacea. Universite de Bordeaux, France
[6]Similkameen Consulting, Canada
[7]RupArt productions, France
[8]Museum of Anthropology at the University of British Columbia, Vancouver, Canada

Keywords: Rock art; dating; British Columbia; integrated approach

Chuchuwayha rock shelter, located in southern British Columbia (Canada), is a sacred place within the territories of the Upper Similkameen Indian Band. Occupants of this rock shelter have left evidence of their presence in archaeological deposits and through the over 50 red, purple and black pictographs they created on its walls. None of these have yet been dated. The Chuchuwayha Project, initiated with the Upper Similkameen Indian band aims to develop a high-precision chronological model of both the identified cultural levels and also of the associated rock art. In addition, we are building an inter-disciplinary model drawing on Indigenous and archaeological knowledge of population movements in this important crossroads between the coast and the interior. Mainly focused on the excavation of the Chuchuwayha site, this study will be expanded to suggest a coherent regional framework of the dynamic human occupation in this region over time. Different analytical strategies are being used based on an integrated approach combining archaeology, archaeometry, geomorphology, ethnography, and oral history:

- sedimentological and granulometric analyses will enable the identification of climatic signatures explaining sediment dynamics over time and correlating archaeological phases with climatic phases
- ^{14}C dates on organic materials collected during excavation highlight a time depth of over 4000 years. These are being correlated to human occupation and the pictographs making
- a homogeneous compact ash level marks a major ancient catastrophic event in the region - this is Mt St Helens Yn event that occurred more than 3000 years ago. The precise timing of this event and the time frame within which people could reoccupy the region will be determined
- silico-calcium crusts surrounding red pictographs will be characterized prior to dating as termini ante/post quos to the drawings using ^{14}C/U-Th cross analyses
- the diversity of coloring materials has to be linked with identified potential sources, including an important ochre quarry located less than 60 kilometers away
- rock collapses which have detached localized wall sections sometimes linked with drawings making will be dated using cosmogenic dating (^{36}Cl, ^{10}Be)
- the comparative examination of archaeological material (fauna, flora, lithics) will be integrated with other archaeological and geomorphological work in the region to reconstruct paleoenvironments and anticipate population movements.

S2-P33.293

Session 3 Biological Materials and Bioarchaeology

ORAL

The Iberian Peninsula between the crescent moon and the cross: exploring Medieval dietary variation across social class, faith, and settlement contexts using stable isotope analysis

Ramallo, P.[1,2], Lorenzo-Lizalde, J.I.[3], Grandal-d´Anglade, A.[4], Alexander, M.[5], Fernández-Rodríguez, C.[6], Lareo-Porral, C.[7], Ayán-Vila, X.[8], Vigo-García, A.[9], Rivas-Nodar, M.A.[10], Staniewska, A.[11], Chivall, D.[12], Roberts, P.[13,14]

[1]Department of Archaeology, Max Planck Institute for the Science of Human History. Kahlaische Str. 10, 07745, Jena,Germany.
[2]Faculty of Medicine and Nursing, University of the Basque Country (UPV/EHU). C/ Pº Dr. Beguiristain 105, 20014, San Sebastián-Donostia, Spain.
[3]PPVE Grupo de investigación H-O7 Universidad de Zaragoza.
[4]Instituto Universitario de Xeoloxía, Universidade da Coruña (UDC). ESCI. 15071, A Coruña, Spain.
[5]Department of Archaeology, BioArCh, University of York
[6]Departamento de Historia, Universidad de León (ULE).
[7]Departamento de Humanidades, Universidade da Coruña (UDC).
[8]Portuguese Foundation for Science and Technology, Institute of Contemporary History, Universidade Nova de Lisboa.
[9]Arqueólogo Municipal- Ayuntamiento de Mondoñedo.
[10]Arqueólogo Municipal. Oficina Técnica de A Grela - Ayuntamiento de A Coruña.
[11]Institute of Ethnology and Cultural Anthropology, Adam Mickiewicz University (Poznań, Poland).
[12]Oxford Radiocarbon Accelerator Unit, Research Laboratory for Archaeology and the History of Art, University of Oxford, Oxford, OX13QY, United Kingdom.
[13]Department of Archaeology, Max Planck Institute for the Science of Human History. Kahlaische Str. 10, 07745, Jena, Germany.
[14]School of Social Sciences, University of Queensland, St Lucia QLD 4072, Brisbane, Australia

Keywords: Stable Isotope analysis, diet, Social Classes, Medieval Age, Iberian Peninsula.

The Iberian Peninsula was at the forefront of the religious, economic, and political changes that swept across Europe during the Medieval Period. Between the 8th-16th centuries AD, the Iberian Peninsula was characterised by the expansion of Christianity and new, associated political kingdoms following the disintegration of the Umayyad Caliphate and the eventual settlement and exploitation of the Americas for a growing European market. In order to understand the practical effects of these changes on the diets of different sectors of society, we applied stable carbon and nitrogen isotope analysis to bone and dentine collagen from human and fauna remains of 156 individuals (97 humans and 59 fauna), across the northern portion Spain, combining our novel data with that from the existing literature (663 human and 109 fauna, a total of 769 human and 168 fauna) to build the first large stable isotope dataset covering a variety of locations (n=45) across the Iberian Peninsula. This dataset allowed us to study the diets of the social elite, including bishops, the sons and daughters of kings, abbots, monks, priests, as well as lower class inhabitants of cities and rural locations. Moreover, we were able to explore dietary differences between individuals following Muslim or Christian faiths during their coexistence and changing fortunes in the Iberian Peninsula. We argue that our results demonstrate clear distinctions in terms of dietary access to meat, marine resources, and different types of crops between different sectors of society, with clear influences of geography, climate, and temporal context.

S3-O01.798

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Husbandry and crop strategies in Tell Humeida, a Chalcolithic site in the Middle Euphrates Valley

Grandal-d'Anglade, A.[1], García-Vázquez, A.[1], Moreno-García, M.[2], Peña-Chocarro, L.[2], Sanjurjo-Sánchez, J.[1], Montero-Fenollós, J.L.[3]

[1]Instituto Universitario de Xeoloxía, Universidade da Coruña. ESCI, Campus de Elviña s/n, 15071 A Coruña, Spain
[2]Instituto de Historia CCHS-CSIC, Albasanz 26-28, 28037 Madrid, Spain
[3]Facultade de Humanidades e Documentación, Universidade da Coruña, Campus de Esteiro 15403 Ferrol, Spain

Keywords: Middle Euphrates Valley; Late Chalcolithic; crop strategies; livestock management

A multidisciplinary study is presented that combines different techniques in archaeometry, applied in Tell Humeida, a site in the Middle Euphrates Valley (Syria).
Ceramic fragments, skeletal and carpological remains were recovered during the prospection of the site between 2009 and 2010. Subsequently, political circumstances prevented the continuation of excavation work, so that the set of samples for study is necessarily small. However, the application of different techniques has allowed us to obtain a solid set of data. Dating by OSL shows ages between 3000 and 4000 BP which places the levels studied in the regional Late Chalcolithic.
In addition to the classical morphological identification, and given the high fragmentation of bone remains, we performed peptide fingerprinting analysis (ZooMS), thanks to which we identified several species of ungulates (sheep, cattle, equids, gazelles, deer) and carnivores such as fox, leopard and caracal.
On these remains we performed isotopic analysis (δ13C, δ15N) of bone collagen, finding a gap between those that would be domestic herbivores (mainly sheep) and those that would be wild (mainly equids). High values of d15N in domestic herbivores suggest a specific management of livestock and crops, which would have been fertilized and irrigated abundantly. The isotopic study of a necessarily small number of carpological remains supports this interpretation. However, very low and homogeneous δ15N values in equids suggest that they may have been raised and fed specifically on vegetables of the Fabaceae family.

S3-O02.781

Searching for a white plague - osteological analysis of bones remains from three sites in Poland

Grzegorska, A.[1]

[1]*Interdisciplinary Doctoral School, Faculty of Archaeology, University of Warsaw*

Keywords: tuberculosis; infectious diseases; bioarchaeology; Early Modern Period, diagnosis

Till now, tuberculosis (TB) remains one of the most common causes of death in the world. Historical hospital records confirm high morbidity and mortality due to TB also in the past. However, a comparison of available data in Poland shows a disparity between expected and obtained bioarchaeological results.
Lesions caused by TB develop on the bones of 5% of infected, however not all of them are typical. While lytic lesions of the vertebrates are among 25 to 50% of infected, a severe form of angular kyphosis called Pott's disease develops only among 3% of them. The osteological diagnosis of TB is based mostly on Pott's disease due to its easily defined features. Other lesions that could suggest TB are e.g. ossified pleura, new bone formation or lytic lesions on the ribs.
For this project, 100 skeletons from Chełm (Poland, dated to 13th-18th century) were analysed. They were described using three independent diagnostic methods for TB: operational definitions of T. Waldron, identification based on typical lesions described in literature and differential diagnosis which included lesions like e.g. minor osseous lesions attributable to TB.
To modify BABAO protocol a system of coding of degenerative joint diseases was added, atypical lesions of the ribs, skull and vertebrae. The differential diagnosis was carried out for all skeletons with possible typical and atypical TB lesions. Samples from three individuals were later sent for DNA analysis to confirm results.
In total, 17 likely cases of TB in Chełm were diagnosed (6 probable out of which 3 were confirmed by DNA analysis, and 4 possible cases). Changes in the protocol did not influence time consigned to bone analysis, but they increased the number of recognized possible cases of tuberculosis. The most typical lesion - angular kyphosis was present in one case, small lytic lesions in vertebrae were present in three cases. The rest of the changes were less typical. Despite the lack of significant increase in probable cases, even a small increase in the number of recognized possible TB cases is important to supplement knowledge about TB.

S3-O03.1503

Activities in a sanctuary: pottery uses and ancient diet in Gravisca (VT, Italy)

Patrizi, G., Pennetta, A.

Laboratory of Analytical and Isotopic Mass Specrometry, University of Salento, campus Ecotekne, 73100 Lecce

Keywords: Organic residue analysis; Stable Isotope; Gas Chromatography; Mass Spectrometry; Gravisca

The site of Gravisca, situated about ten kilometres from Tarquinia (the ancient *Tarkna*), represents one of the most important harbours of antiquity. It was a crossroad of goods and people in the centre of the Mediterranean area between the end of the 7th century BC and the Roman period. It constitutes extraordinary evidence of an emporic sanctuary, similar to Naukratis in Egypt, created as a protected area where Greek merchants from the eastern Mediterranean Sea could exchange their products in the Etruscan territories, under the protection of divinities who were their guarantors.
In this complex framework, thanks to the accurate archaeological field work, during the archaeological campaign in 2013 has been investigated the well situated in the *sacellum* of of Adon, called well *23 δ*.
The ceramic finds found into the well, strictly related to the life of the sanctuary and hidden in this structure before the destruction of the site on 281 b.C., have been analysed by high Gas chromatography – mass spectrometry (GC-MS) and gas chromatography combustion isotope ratio mass spectrometry (GC-C-IRMS). The biomarkers preservation has been assessed using two differ extraction protocols and surviving residues from archaeological pots, both lipid and wine markers, have been characterized and identified. Their presence and amount have been essential to gather the original contents of the potteries and the intensity of their use. At last but not at least, the successful application of organic residue analysis (ORA) to the Gravisca potteries has permitted to connect the form of the vessels and their function. Thanks also to the stable isotope analysis combined with ORA results, have been possible starting to understand, although it was a small context, the diary in the sanctuary. The results obtained with this multidisciplinary approach will be described in the present communication.

S3-O04.461

Cinnabar and mercury exposure in prehistoric Iberia: biogenic and diagenetic pathways into human bone

Emslie, S.D., Alderman, A.E.

University of North Carolina, Department of Biology and Marine Biology, 601 S. College Road Wilmington, NC 28403 USA

Keywords: HgS; late Neolithic; Chalcolithic; burial rituals; mercury poisoning

Prehistoric and historic mining, processing, and use of cinnabar (HgS) in burial rituals and as a pigment and/or medicinal likely led to moderate to chronic exposure to mercury in individuals and cultures associated with this mineral. Late Neolithic and Chalcolithic populations in Iberia in particular show evidence for high usage of cinnabar with the Almadén mine in Spain as the largest and most likely prehistoric source for this mineral. Analyses of mercury levels in human bone from numerous sites throughout Iberia indicate that mercury poisoning was at least impacting individuals in life, if not the direct cause of their death. However, could some of the mercury in this bone have diagenetic sources, especially where cinnabar powder is associated with burials, or were biogenic pathways during life the primary source? Here, we present preliminary results on the first controlled experiments to test for diagenetic pathways of mercury from cinnabar into animal bone treated with cinnabar powder and two kinds of paint (yolk or linseed oil as a base mixed with cinnabar powder) compared to controls with no treatment. One experiment was conducted in a warm humid environment at the University of North Carolina campus, Wilmington (UNCW), using soil high in organic matter, while another experiment was conducted at an archaeological site (Perdigões) located in a warm, arid environment in Alentejo, Portugal, where moderate to high levels of mercury in archaeological human bone have been reported. Bones were left buried in the soils for 6-12 months between sampling. Total mercury (THg) concentrations in bones were measured in triplicate with a Nippon MA-3000 analyzer; selected bones also were analyzed with a scanning electron microscope and energy dispersive spectrometry (SEM EDS). Our results indicate that soils in humid environments with high organic matter do break down cinnabar allowing some mercury to absorb into associated bone matrix, especially in highly porous bone of juvenile animals (mean 26 – 36 ppm). Only trace concentrations of mercury were detected in the experimental animal bone at Perdigões (0.03 – 1.3 ppm) except for a slightly larger concentration (4.3 ppm) in the outermost layer of the bone cortex of bone treated with cinnabar powder. Significantly more mercury was found in bone treated with an oil-based vs yolk-based paint in the UNCW experiment. Ongoing experiments will continue to yield additional information on this diagenetic pathway.

S3-O05.092

On Drugs, Simple and Compound: the use of natural and synthetic minerals in 4th c BC preparations of simples and compound medicines, via Synchrotron techniques

Bots, J.[1], Cipiccia, S.[2], Hamilton, A.[1], Ignatiadou, D.[3] and Photos-Jones, E [4,5]

[1]Civil and Environmental Engineering, University of Strathclyde, Glasgow G1 1XQ, UK
[2]Diamond Light Source Ltd,,Harwell Science and Innovation Campus, Didcot, OX11 0DE, UK
[3]National Archaeological Museum, Athens 10682, Greece
[4]Archaeology, School of Humanities, University of Glasgow, Glasgow G12 8QQ, UK
[5]Analytical Services for Art and Archaeology (Ltd), Glasgow G12 8JD, UK

Keywords: Synchrotron, organic, inorganic, ancient medicines

The production of synthetic metallic minerals, in antiquity, is well-attested in the works of Theophrastus, Dioscorides, Pliny and others. Some of these reactions involved the use of metals like copper or lead and an acid, like acetic acid, for the production of, for example, cerussite/hydrocerussite (psimythion) and vergigris (ios) respectively. In their use as medicines, pharmaka, the above manufactured minerals were also be mixed, before or after heating, with organic materials like oils, resins, gums or beeswax. Understanding the nature of these minerals (morphology and composition) and the nature of the products of their reactions with organics, at the micro-crystalline level, can potentially provide considerable insight into their purported medicinal properties. Recent examination of the contents of small vials deriving from the burials of two doctors (as attested by associated medical/surgical equipment) dating to the 4th c BC and the burial of a third dating to the 2nd c AD, in N Greece, offer a unique insight into these exceedingly rare and hitherto little understood materials.

In examining these complex materials, we employ a novel approach by overlaying the data deriving from three different techniques

(i) nano-tomography (ptychography) which provides detailed morphological information, on surface and in depth,
(ii) 3D X-ray fluorescence imaging offering detailed information on the distribution of elements like Pb, Cu, Fe, Ca and potentially other trace elements in correlation with the observed morphology and
(iii) 3D XRD informing on the μ-crystalline phases, whether organic or inorganic, (e.g. Pb-carbonate or Pb-stearate, CuO or CuCl2(OH)3, beeswax, calcium formate, or mono hydro calcite, others), present as a function of the morphology and the chemical composition.

We suggest that the approach outlined above constitutes a powerful tool in the study of the interface between the organic and inorganic components of these complex materials which, based on documentary evidence, appear to have been used ‘regularly’ by the ancient world.

S3-O06.748

Session 3 Biological Materials and Bioarchaeology

POSTER

A multidisciplinary study of the enigmatic burial of the Iberian era in Cerro de Las Cabezas (Valdepeñas, Ciudad Real): taphonomy, forensic anthropology and stable isotopes

Grandal-d'Anglade, A.[1], Sarkic, N.[2], García-Vázquez, A.[1], Torres-González, T.[3], Herrerín-López, J.[2]

[1]Instituto Universitario de Xeoloxía, Universidade da Coruña. ESCI, Campus de Elviña s/n, 15071 A Coruña, Spain
[2]Facultad de Biología, Universidad Autónoma de Madrid. Campus de Cantoblanco, C/ Darwin 2. 28049 Madrid, Spain
[3]Grupo de Investigación del Cerro de las Cabezas GICC, Valdepeñas, Spain

Keywords: Iberian culture; unusual burial rite; stable isotopes; forensic anthropology

The Iberian oppidum of Cerro de Las Cabezas (Valdepeñas, Ciudad Real) is one of the great settlements that existed in the ancient territory of the Oretania. It is situated at 805 m a.s.l., in a strategic area of natural passage between the four cardinal points.
This settlement began in Late Bronze age and developed throughout the seventh and sixth centuries BC. In the fifth century BC there was an extension, building a wall surrounding a space of 130,000 m2 in which a complex urban framework was developed.
During the 2010 campaign, two human skeletons associated with the wall were found, which due to their stratigraphy would date from the end of the third century or the beginning of the second century BC. These are the anatomically connected remains of two individuals together with an ensemble of large deer antlers, in an unprecedented ritual in the Iberian Peninsula, but with some parallelism in other areas of Europe.
Since in Iberian culture the main funerary rite was incineration, it is worth asking about the meaning of this burial. The taphonomic study shows that the corpses were thrown post-mortem, without any care. The forensic anthropological study indicates that the individuals suffered traumatic death. The isotopic study of the bone collagen (C and N) and dental apatite (C, O and Sr) of both individuals, as well as a representative sample of local fauna, makes it possible to reconstruct a high trophic level and the local and foreign provenance of domestic animals and humans, respectively.

S3-P01.782

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Taíno Foodways: Molecular Evidence for the Original 'Barbecues'

Briggs, L. [1], Martínez Milantchí, M.M.[1], Cooper, J.[1], Samson, A.[2]

[1]British Museum, Americas Section, Great Russell Street, London WC1B 3DG, UK

[2]School of Archaeology and Ancient History, University of Leicester, University Road, Leicester, LE1 7RH, UK

Keywords: Caribbean archaeology; Iron nails; Archaeometallurgy

Our understanding of Taíno diet and foodways prior to European colonisation remains poorly understood. Ceramics form the most abundant class of archaeological material recovered from Taíno sites and can offer direct insights into subsistence strategies and culinary practices. Here, we report the results from the first molecular investigation of pottery vessel residues from Puerto Rico. Forty ceramic artefacts from Isla de Mona, Puerto Rico, including sherds from 25 Taíno vessels and 15 European vessels, were subjected to three lipid extraction methods to recover a wide variety of molecules that might shed light on the past contents of these pots. Gas chromatography-mass spectrometry (GC-MS) confirms that abundant lipids are preserved across all samples analysed, with sherds from the rims of Taíno vessels demonstrating the highest yield of lipid. The molecular profile in the Taíno and European vessels is substantially similar. While there is abundant zooarchaeological evidence for the consumption of fish in Isla de Mona, there is no molecular evidence of marine fats in either the Taíno vessels or the European vessels. Rather the vessels were used to prepare foods which may have supplemented the consumption of fish cooked using a different preparation method. These methods may have included spit roasting, pit roasting, or use of a 'barbecue' which is derived from the original Taíno word 'barbacoa' meaning a raised wooden grate on which to smoke food above a fire. Pit roasting remains a traditional way of preparing food in Puerto Rico to this day, which may represent an unbroken cultural practice with its roots in Taíno culinary traditions. Around 90% of the cultural remains of indigenous communities in the Caribbean consist of ceramic artefacts, and this important archaeological resource is now, for the first time, revealing the molecular evidence for lost cultural practices.

S3-P02.1499

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Molecular indicators of burning in the identification of past fire-related activities in cave sites from Polish Jura

Gryczewska, N.[1,2], Chmielewska, M.[2], Cyrek, K.[3], Kot, M.[1], Krajcarz, M.T.[4], Sudoł-Procyk, M.[3], Wilczyński, J.[5], Wojenka, M.[6], Suska-Malawska, M.[2]

[1]*Faculty of Archaeology, University of Warsaw, Warsaw, Poland*
[2]*Faculty of Biology, Biological and Chemical Research Centre, University of Warsaw, Warsaw, Poland*
[3] *Institute of Archaeology, Nicolaus Copernicus University, Toruń, Poland*
[4]*Institute of Geological Sciences, Polish Academy of Sciences, Warsaw, Poland*
[5]*Institute of Systematics and Evolution of Animals, Polish Academy of Sciences, Cracow, Poland*
[6]*Institute of Archaeology, Jagiellonian University, Cracow, Poland*

Keywords: Polycyclic Aromatic Hydrocarbons; elemental analysis; hearths; human activity; cave sediments

Polish Jura is a karstic region located in southern Poland with an archaeological record of long and variable use of caves. However, in some strata the record is enigmatic or fragmentary, which calls for new methods that would help gain unique insight into its complexity and focus on ancient human presence in caves.
Polycyclic aromatic hydrocarbons (PAHs) are one of the burning indicators, used as a proxy for quantification of combustion-originated organic carbon in soils and sediments that might have been affected by fire. Their analysis has been successfully applied in archaeological context, most notably in lacustrine contexts, but they were also applied to cave sediments. Presented preliminary results are a part of a broader research project (supported by National Science Centre, Poland grant no 2021/41/N/HS3/02369) focused on applying biochemical methods to sediments at cave sites and correlating them with archaeological data to understand past human settlement. Sediment samples from two caves with well-recognized Holocene sequences and confirmed traces of human occupation (Łokietka Cave and Sąspowska Zachodnia Cave) have been analysed with the use of Gas Chromatography with Mass Spectrometer. Additionally, six sediment samples connected to Middle Palaeolithic hearth were analysed for comparison. All samples have been analysed with regard to their carbon, nitrogen and phosphorus content.
Data on fire-related activity in selected caves acquired from macroscopic observations, charcoal presence in strata and elemental analysis only partially overlaps with results of PAHs analysis. Acquired results call for the discussion on PAHs usefulness in recognizing presence or absence of human-originated fire as complementary to data gained by archaeologists during excavations. Additional problem is using PAHs analysis for answering more specific questions about fire intensity throughout stratigraphic sequences.

Microbial identification for an effective biocleaning: the case study of the Santa Lucia alle Malve rupestrian church

Santacroce, M.[1], Baranek, J.[2], Adamski, Z.[3,4], Dabert, M.[5], Trzebny, A.[5], Bufo, S.A.[1], Scrano, L.[6]

[1]*Department of Sciences, University of Basilicata, Potenza, Italy*
[2]*Department of Microbiology, Institute of Experimental Biology, Adam Mickiewicz University in Poznań, Poznań, Poland.*
[3]*Department of Animal Physiology and Developmental Biology, Faculty of Biology, Adam Mickiewicz University in Poznań, Poznań, Poland*
[4]*Laboratory of Electron and Confocal Microscopy, Faculty of Biology, Adam Mickiewicz University in Poznań, Poznań, Poland*
[5]*Laboratory of Molecular Biology Techniques, Faculty of Biology, Adam Mickiewicz University in Poznań, Poznań, Poland*
[6]*Department of European Culture, University of Basilicata, Matera, Italy*

Keywords: Rupestrian church; *Bacillus; Firmicutes;* genotypic and phenotypic identification; bio-cleaning; *Solanum Nigrum* extracts

Identifying the microbial community that lives on the lithic cultural heritage through phenotypic and genotypic approaches is fundamental for understanding the contribution of colonisers in biodeterioration and selecting the best cleaning solutions with environmentally-friendly products. This was done with the cultivable microbiota present on the internal walls of the Santa Lucia alle Malve (SLM) rock church, recognised by UNESCO as a World Heritage Site. The SLM church is in the Sasso Caveoso area (Matera, South Italy). It is fully excavated in the limestone rock. Numerous frescoes cover the internal surfaces, but the bacterial and fungal colonisation spoils its beauty. The results of this study clearly showed the predominance of bacteria of the phylum *Firmicutes* and precisely of the genus *Bacillus,* whose prevalence may be due to the specific environmental conditions, very similar in all sampling sites of the church, and to their ability to produce endospores. These dormant forms allow bacteria to survive in unfavourable conditions. The genotypic approach showed that the various *Bacillus* species isolated on the surfaces of the experimented church are closely related and similar but show a different phenotypic profile. This diversity of physiological and morphological traits reflects the potential complexity of the metabolomes present in the bacterial communities of the internal walls of the SLM and confirms the need to identify the colonisers correctly and proceed with a non-temporary bio-cleaning. The results of bio-cleaning using *Solanum nigrum* extracts were effective, but the durability of the treatment has yet to be verified.

S3-P04.1540

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

‘Organic temper’ in southeast Europe Early Neolithic pottery becomes multilayered

Dzhanfezova, T.

University of Oxford, United Kingdom

Keywords: Early Neolithic, Southeast Europe, organic temper, subsistence practices

The Early Neolithic pottery in many European regions is characterised by the presence of ‘organic temper’, usually described as ‘chaff’. However, the type of organics, their role as actual functional temper and the various plant inclusion sources have not been investigated. Why was organic temper added? What were the plant materials that took place in pottery production? How to explain the diverse use of the ‘organic temper’?

The preliminary results of MINERVA, an Oxford based project that investigates over 20 Neolithic Balkan sites, has shown great variability in terms of plant inclusions types, concentrations, morphology, etc. – an observation valid within and between the sites. A detailed study of the plant inclusions not only reveals the interaction between two major Early Neolithic cycles – the agricultural and the technological (pottery production). It also allows to consider this integration in the correspondent local natural, cultural and social environment.

The research goal is to read the ‘organic temper’ technological component within holistic framework created by combining technological and agricultural landscapes. Archaeologically survived plants and phytoliths are investigated by optical microscopy, polarised and scanning electron microscopy in comination with experimental and ethnographic data. Such integrated methodology informs on essential details of various agricultural systems within the broader context of locally specific subsistence practices in a series of Early Neolithic Southeast European sites.

S3-P06.797

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Alcohol consumption in Iron Age Messapia: combining organic residue analysis, use-alteration traces and vessel morphology

Notarstefano, F.[1], Messa F.[2], Perrone, S.[2], Semeraro, G.[1]

[1]Dipartimento di Beni Culturali, Università del Salento
[2]Dipartimento di Scienze e Tecnologie Biologiche ed Ambientali (Di.S.Te.B.A.), Università del Salento

Keywords: Iron age, Organic residue analysis, Pottery, Alcoholic beverages, Iapygian culture

Alcohol consumption played an important role in feastings and social events in past agricultural societies, helping to establish and maintain social relationships. The study of the Messapian populations of southern Italy between the 8th and the 6th century BC is revealing interesting aspects related to the ceremonial sphere and its relevance in defining the social dynamics within local communities. An indirect evidence of ritual and social practices within the Iapygian culture can be found in the particular incidence of local pottery with a matt-painted decoration and the reoccurring set of drinking vessels: olla, jug, askos and biconical jar. The function of these vessels for the consumption of alcoholic beverages is suggested from morphological and organic residues analyses conducted on pottery recovered from two Iron Age settlements in southeastern Italy, Castello di Alceste and Castelluccio (Brindisi). Both are part of the Iapygian culture of the Salento peninsula, from which the archaeological evidence has suggested that in the 8th century the indigenous populations of this region were engaged in processes of settlement expansion, socio-economic differentiation and *elite* proliferation, as well as a growing exploitation of agricultural and pastoral resources, also confirmed by botanical and faunal data. The study of matt-painted pottery provides insights both into the emergence of social behaviors and into the development of commensal practices that possibly could have involved alcohol consumption. The pottery shapes and the use alteration traces on the interior of vessels show comparisons with those observed in ceramic vessels used for alcohol fermentation reported in many ethnoarchaeological studies. Organic residue analysis conducted by gas chromatography coupled to mass spectrometry (GC-MS) allowed the identification of *Pinaceae* products (resin/pitch), beeswax, plant waxes together with fermentation biomarkers in the samples recovered from different ceramic vessels found at the two archaeological sites. Based on integrated studies, we suggest that the vessels were used for the consumption of alcoholic beverages possibly derived from cereals or other plants. These preliminary results give the earliest direct evidence of the function of matt-painted pottery and contribute to our understanding of ceremonial and commensal practices among the indigenous communities of southern Italy in Iron Age.

S3-P07.1548

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Untangling intra-tooth isotopic data from dentine-collagen in high-crowned teeth: a new experimental study with modern sheep specimens

Díez-Canseco, C.[1,2], Aguilera, M.[3], Tornero, C [4,2]

[1]Àrea de Prehistoria, Universitat Rovira i Virgili (URV), Avinguda de Catalunya 35, 43002 Tarragona, Spain
[2]Institut Català de Paleoecologia Humana i Evolució Social (IPHES-CERCA), Campus Sescelades URV (Edifici W3), 43007 Tarragona, Spain
[3]Departament de Producció Vegetal i Ciència Forestal - AGROTECNIO Center, Universitat de Lleida, Avda. Rovira Roure 191, 25198, Lleida, Spain
[4]Department of Prehistory, Autonomous University of Barcelona (UAB), Edifici B Facultat de Filosofia i Lletres, 08193 Bellaterra, Spain

Keywords: sequential analysis; tooth dentine; carbon isotopes; nitrogen isotopes; serial sampling; modern reference data-set

During the last decades, intra-tooth stable isotopes analyses have become quite popular in zooarchaeological studies. Since dental tissues do not undergo remodeling once mineralized, they allow for obtaining isotopic data with a high temporal resolution, in some cases with a seasonal extent, a powerful approach for studying essential aspects of animal management such as reproduction, feeding habits, or mobility practices. Nowadays, intra-tooth stable isotopes analyses are widely implemented in enamel-bioapatite of hypsodont species; however, they have been scarcely performed in dentine-collagen, despite collagen constitutes a key biomolecule in paleodietary studies. Dentine develops following a secretion of layers oblique to the tooth vertical axis and this pattern makes hard to isolate dentine increments during sampling. The development of this analytical technique in high-crowned teeth has been limited by the shortage of experimental studies addressing methodological and technical issues to support its archaeological implementation and to guarantee a reliable interpretation of the isotopic data.

In this contribution, we test the resolution of a dentine sampling procedure to obtain intra-tooth isotopic variability of $\delta^{13}C_{collagen}$ and $\delta^{15}N_{collagen}$ values tied to diet shifts in caprines. A new experimental study with raised modern sheep breeds was conducted, covering a whole year and where carbon and nitrogen isotopic signatures of consumed plants were also analysed. Three diet periods with different isotopic compositions were alternated during sheep's life. Dentine was sequentially sampled in previously demineralized second and third mandibular sheep molars. Samples were treated with standard protocols of collagen purification and then subjected to carbon and nitrogen isotope analysis. In M2 and M3 isotopic sequences obtained, diet shifts are recorded along two progressive trends of increasing – decreasing isotopic values, but some affections of the sampling procedure are also present. We conclude that the dentine sequential sampling here implemented allows for obtaining reliable intra-tooth $\delta^{15}N_{collagen}$ and $\delta^{13}C_{collagen}$ values tied to dietary conditions in high-crowned teeth, although dentine growth patterns should be considered in detail in final interpretations. The integration of this high-resolution analysis in archaeological studies offers a new scope of possibilities for studying dietary patterns, seasonal dietary changes, and animal husbandry practices in past herds.

S3-P09.1555

Compositional Residue Analysis of "Perfume" Residues in a Collection of Double Unguentaria at the Michael C. Carlos Museum

Mahan, S.[1], Armitage, R.A.[1], Stein, R.[2], Faas, M.[2]

[1]Department of Chemistry, Eastern Michigan University College of Arts and Sciences, Ypsilanti, MI, 48197
[2] Michael C. Carlos Museum, Emory University, Atlanta, GA, 30322

Keywords: Residue, Unguentaria, Perfume, HS-SPME, DART-MS

Unguentaria are ancient glass or ceramic vessels commonly found in archaeological sites across the Mediterranean. The believed use of these vessels was the storage of oils, balms, ointments, cosmetics, and other unguents. The most common style of unguentaria is the simple single barrel variety, which have been widely studied. Double unguentaria are less common and there have been no studies connecting their morphology to their specific use. The two thin elongated barrels, which would reduce evaporation and their higher degree of adornment, implies a valued product suggesting that double unguentaria may have been used for the storage of perfumes. Historical texts, from notable figures such as Pliny the Elder, have given us the basic "recipe" for ancient perfume residues: a matrix or base component, such as a plant oil or animal fat, and an aroma element, commonly derived from a plant source. By characterizing the composition of residues found in double unguentaria, more information about how people in Greco-Roman times used double unguentaria. Gas chromatography-mass spectrometry (GC-MS) is the standard technique for archaeological residue analysis, and was used to identify organic components of the residues, focusing mainly on the matrix or base of the perfume. The volatile possible perfume components were extracted and concentrated by headspace solid-phase microextraction (HS-SPME) and characterized by GC-MS. Direct analysis in real time-mass spectrometry (DART-MS) was employed as a screening tool and used in conjunction with GC-MS and HS-SPME-GC-MS. Scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) was utilized to characterize inorganic substances that may have been used as a colorant. Our results have shown evidence of lipids consistent with plant oils and several biomarkers of aroma elements, which provides support to the theory that the residues in these double unguentaria were consistent with them having contained ancient perfumes.

S3-P10.1572

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Molecular Characterization of an Unusual Residue from an Egyptian Old Kingdom Mummy

Van Buskirk, C.[1], Armitage, R.A.[1], Stein, R.[2]

[1]Department of Chemistry, Eastern Michigan University College of Arts and Sciences, Ypsilanti, MI 48197
[2] Michael C. Carlos Museum, Emory University, Atlanta, GA, 30322

Keywords: DART-MS; GC-MS; Egypt; mummy; mummification balm

Mummification is the process by which a body is preserved either naturally, where the environment facilitates preservation through desiccation, or by deliberate intervention like embalming. It has been observed in Egypt as early as the prehistoric period (4500-3350 BC) and as late as the Copto-Byzantine period (395-645 AD). The Old Kingdom, which spanned from 2613-2181 BCE, was a period of innovation and experimentation with mummification practices. Even though the composition of mummification balms changed over time, typically some combination of salts, waxes, plant oils, animal fats, and resins were used. While very few surviving examples of Old Kingdom mummies exist and even fewer studies have been conducted on said mummies, compositional analysis is critical in order to understand what materials were available during that time period which can, in turn, also be used to examine the changes in mummification practices over time. The only Old Kingdom mummy in North America resides at the Michael C. Carlos Museum at Emory University. Study of this mummy presents a unique opportunity to understand what materials may have been used in mummification practices during that period. Characterization of the three residue samples, obtained from the mummy, was performed using gas chromatography mass spectrometry (GC-MS) and direct analysis in real time mass spectrometry (DART-MS). Analysis of the volatile components of the samples was done using headspace solid-phase microextraction (HS-SPME) paired with GC-MS and DART-MS. So far, these methods have shown the presence of fatty acids and their degradation products, which may be indicative of a drying oil or type of fat. Dehydroabietic acid was present in several of the samples, which is an oxidation product of resins obtained from conifer trees. These results are consistent with other studies of mummification balms and seem to indicate that a similar pool of ingredients were used in balms over thousands of years. Further study will provide insight into the mummification practices of the Egyptian Old Kingdom, including what other materials were used and how those materials may have been processed.

S3-P11.1571

A Method Distinguishing Types of Ash: Case Study at Qumran, Israel

Peters, I.[1], Langgut, D.[2], Boaretto, E.[3], Asscher, Y.[1]

[1]*Israel Antiquities Authority, Analytics Lab*
[2]*Tel Aviv University, Sonia and Marco Nadler Institute of Archaeology*
[3]*Weizmann Institute of Science, Scientific Archaeology Unit, D-REAMS Radiocarbon Dating Laboratory*

Keywords: Ash; anhydrite; gypsum; biogenic; geogenic

Ash is found at archaeological sites usually as calcium carbonate minerals, which can be differentiated from geogenic or biogenic sources via infrared spectroscopy. However, the white layer in between two charcoal-rich layers at Cave 49 in Qumran was anhydrite - a material from either rocks used for preparing gypsum plaster, like those found near the Dead Sea, or from tamarisk, a plant unusually rich in gypsum which also grows in the Dead Sea area. Anhydrite turns into gypsum upon hydration, rendering it difficult to identify tamarisk ash. In this study, we show it is possible to determine whether pyrogenic anhydrite originated from biogenic or geogenic sources, even if it has already hydrated to gypsum. Heating experiments of geogenic, synthetic and biogenic calcium sulfates of different structures, i.e. gypsum, anhydrite and bassanite, have been used to quantify the changes in chemical, morphological and structural disorder properties following heating. The atomic structural disorder was assessed using a newly analyzed infrared spectroscopic indicator that monitors the changes in ν4 and ν2 bands of anhydrites. A thick layer of ash in Qumran Cave 49 has been shown to contain anhydrites, associated with tamarisk ash, based on the disordered crystallinity, relatively high sodium content and irregular morphology. This layer of anhydrite from biogenic ash shows that there was an intense burning of tamarisk over a short period of time at Qumran, as seen by radiocarbon dating and this novel method.

S3-P12.3014

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Brachymetatarsia of the 1st toe as an indicator of dispersion and immigration of Greeks and Hellenic-born populations

Vounotrypidis, P.[1], Noutsou, P.[2]

[1]*Rheumatology department, 424 General Military Hospital, Thessaloniki, Greece*
[2]*BA(Hons) in Greek Culture*

Keywords: Osteomorphometry; Phylogenetic traits; Greek foot; Morton's toe; Metatarsus atavicus

Introduction: Among the methods that are used to identify the origins of a population and its historical background are phenotypical characters, including osteomorphometry. Brachymetatarsia of the 1st toe (BMT1), also termed Morton's toe or Greek foot in Classical Arts, is considered a congenital anomaly which may represent a phenotypical character of Greeks and subsequently a measure of their immigration.
Purpose: This study aims to investigate a possible relation of the unexplained so far origins of specific populations in Asia and North America with the worldwide distribution of the BMT1. The study uses medical, historical, sociological and geographical information to attain the answer in the question.
Methods: We performed a population-based study, in Thessaloniki and Athens, to estimate the prevalence of Morton's toe in Greece. We retrieved all the published data regarding the global dispersion of the specific feature. We conjugated Greek history with sociological and geographical information as well as current data to investigate an association of this specific feature with other populations.
Results: Our epidemiological study shows that Greek foot is present in half of the Greek population and that it is most prevalent in males (65%) than in females (35%), indicating an X-linked recessive inheritance, which means it is not a spontaneous anomaly, but it is transmitted by males. Worldwide, Greeks (Ionες) are found in second place bearing this genetic feature after Ainu (90%), which may represent an ethnic isolate, related to Greek immigration. They probably forced to further movement through the Bering strait and mix with Asian populations, in Alaska (Inuit) and North America (Ani-Yun-wiya), the later carry also, in anecdotal observations, this specific trait.
Conclusions: The present work outlines the results of a population-based study in Greece comparing them with the universally published results, conjugated with historical, cultural and geographical data, to describe the possible eastern expansion of Greeks across the continents, following the Hellenistic period.

S3-P13.116

Molecular approach of human mummies balms

Mezzatesta, E., Joliot, C., Vieillescazes, C., Mathe, C.

University of Avignon (France). UMR IMBE (Mediterranean Institute of marine and terrestrial Biodiversity and Ecology; CNRS 7263/IRD 237), Restoration Engineering of Natural and Cultural Heritage

Keywords: balms; gas chromatography; mass spectrometry

28 mummies and 45 heads of mummies from Ancient Egypt (from the twelfth dynasty to the Ptolemaic period), belonging to the Egyptian collection of the Confluences Museum in Lyon, have never been studied. This work is part of a scientific project called HELYCOM (Human Egyptian LYon COnfluences Mummies) within the framework of the Labex Archimede, led by an interdisciplinary research team. The aim is to characterize and identify the nature of the chemical substances present in the mummification balms.

Given the wide variety of natural substances that can be used in the formulation of balms, different analytical techniques, each providing complementary information, were used such as Fourier Transform Infrared (FT-IR) and Gas Chromatography Coupled to Mass Spectrometry (GC-MS).

The infrared spectroscopy study was carried out on 61 mummy balm samples to characterize the different chemical families present. Given the large number of samples studied, statistical analyses were carried out by Agglomerative Hierarchical Clustering (AHC) in order to group them into clusters and thus compare them with each other in terms of their chemical composition. Prior to the chromatographic analyses, an extraction step was developed using a conventional solvent. In addition, in order to perform molecular identification, the development of a new solid phase extraction protocol has been successfully developed and optimized.

The whole of this study permit to characterize different balm compositions associated with different formulations and states of alteration and/or degradation. Plant oil, animal fat, beeswax, di- and triterpene resins, bitumen, correspond to all the substances identified. An interpretation of the alteration and degradation processes of diterpenic molecules was carried out in Principal Component Analysis (PCA).

S3-P14.243

Diagenetic alterations model of heterogeneous archaeo-osteological tissues

Scaggion, C[1]., Dal Sasso, G.[2], Nodari, L.[3], Pagani, L.[4], Carrara, N.[5], Usai, D.[6], Gadioli, G.[5], Artioli, G[1].

[1]*Department of Geosciences, University of Padova, Padova, 35131, Italy*
[2]*Institute of Geosciences and Earth resources, Italian National Research Council-CNR, Padova, 35131, Italy*
[3]*Italian National Research Council-CNR, ICMATE, Padova, 35127, Italy*
[4]*Department of Biology, University of Padova, Padova, 35122, Italy*
[5]*Museum of Anthropology, University of Padova, Padova, 35121, Italy*
[6]*Sudanese and Sub-Saharan Studies Center (CSSeS), Treviso, Italy*

Keywords: diagenesis; FTIR; bone quality; collagen preservation; atomic disorder

Hard tissues such as bones and teeth are often the only direct proof remains of animals and humans of the past and hence represent valuable archives for paleogenomic, palaeoecology, palaeo-environment and palaeopathology studies. For these reasons, the osteological remains of historical, archaeological, medical-forensic and paleoanthropological interest are of fundamental importance due to their eminent research function. *Post-mortem* alterations of archaeological bone depend upon a number of various processes, collectively included in the concept of bone diagenesis, that can lead to degrade it rapidly due to extrinsic/intrinsic factors (i.e. burial environment, bony biochemistry and microstructure), which may make the specimen less suitable for the planned studies aiming at recovering pristine information on bone material. Here we intend to propose a broad model of diagenetic alteration, comparing the behavior of the organic and inorganic phase on a large and diversified set of bone samples characterized by different origins and chronological phases. From extremely altered archaeological specimens to modern, fresh bones, we evaluated the sensitivity and efficacy of the Infrared phosphate peak width including it as the most predictive parameter on the degree of alteration of the bone system. The work was extended to examine the modifications induced by diagenesis on the secondary structure of conserved collagen, evaluating their effects on bioapatite crystals. The results obtained prove that the width of the phosphate peak is advantageous for monitoring minimal changes in the structure and chemical properties of bioapatite as well as indirectly in collagen. This method could improve the selection process of bone samples for different fields of interest. Therefore, we will intend to proceed with this research idea by developing a minimally invasive and low-cost pre-screening method for molecular and isotope analysis.

S3-P15.3017

Lipids analysis and its impact on archaeological interpretation in a case of ritual features from north—eastern Poland

Klecha, A.[1], Manasterski, D.[2], Kałużna-Czaplińska, J.[3]

[1]Antiquity of Southeastern Europe Research Centre, University of Warsaw
[2]Institute of Archaeology, University of Warsaw
[3]The Faculty of Chemistry, Lodz University of Technology

Keywords: lipid analysis; Bell Beakers; ritual features; north-eastern Poland;

It has become a truism in archaeology to say that lipid analyses shed new light on the artefacts' interpretation. The example from north-eastern Poland can only confirm it. However, separated from the context, they can become a simple anecdote. The aim of the study is to present how interpretation is evolving from a simple characteristic of the ritual relics to a dynamic distinction of the rituals and symbolic culture of the people, who performed them. A number of pottery examples from four Bell Beakers features from Supraśl in north-eastern Poland were submitted for research. Among others, the lipid analysis proved the ritual functionality of the site in question. In view of the cultural background of north-eastern Poland in this period, the results are found very important. Petrographic research has shown that communities with such far-reaching provenance as the Bell Beakers have gone very far to the northeast. Although the reason for this journey can only be presumed, their activity has been confirmed. An example of this is the local ceramics, with a many stylistic components of this culture. The presence of the Bell Beakers was important in the cultural changes observed there, the question whether their ritualism was not equally significant.

S3-P16.740

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Late Neolithic amber ornaments from north-eastern Poland and their provenance using technological and raw material analyses

Cetwińska, A.[1], Manasterski, D.[2], Wagner-Wisiecka, E.[3], Kwiatkowska, K.[4],

[1]*Antiquity of Southeastern Europe Research Centre University of Warsaw, Krakowskie Przedmie- ście 26/28, 00-927 Warszawa, Poland*

[2] *Faculty of Archaeology University of Warsaw, Krakowskie Przedmieście 26/28, 00-927 Warszawa, Poland, e-mail: dmanasterski@uw.edu.pl*

[3]*Faculty of Chemistry, Department of Chemistry and Technology of Functional Materials, Gdańsk University of Technology, Narutowicza 11/12 80-233 Gdańsk, Poland, e-mail: ewa.wagner-wysiec- ka@pg.edu.pl*

[4]*Polish Academy of Sciences Museum of the Earth in Warsaw, Aleja Na Skarpie 20/26 i 27, 00-488 Warszawa, Poland*

Keywords: provenance studies; amber ornaments; Late Neolithic; north-eastern Poland; technological analysis and raw material analyses

Even if the area of north-eastern Poland was in Late Neolithic abundant in many (3rd millennium) amber workshops located in the Gulf of Gdańsk and in Żuławy Wiślane, there are only few finds of finished products found there. Among them are unique ornaments from sites: Ząbie 10, Supraśl 3 and Supraśl 6. The preliminary analysis showed that they differ from the products of local groups from territory of nowadays Poland. The selected artefacts were therefore multi dimensional stylistic analysis to identify their provenance, taking into consideration also the type of raw material used to their production by means of FTIR analysis. Stylistic analysis showed that several specimens have analogies in the amber beads known from the Bell Beaker phenomenon. However, most of these beads are unique and there are no analogy among Neolithic ornaments in Europe. In addition, it was found that in the specimens from Ząbie 10 and Supraśl 3 and 6 sites, holes were drilled with a metal drill, not a flint drill, which was previously unheard of in this part of Europe. The FTIR analysis revealed the use of local amber, including its different varieties i.e. succinite, gedano-succinite and gedanite.

S3-P18.738

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of glue recipes from Colonial America: further studies of repaired ceramics from Ferry Farm

Fraser, D.F.[1,2], Armitage, R.A.[2], Kaktins, M.[3], Marquis, M.[3]

[1]Department of Physical Science and Chemistry, Lourdes University, Sylvania, OH 43560 USA
[2]Department of Chemistry, Eastern Michigan University, Ypsilanti, MI 48197 USA
[3]George Washington Foundation, Fredericksburg, VA 22405 USA

Keywords: 18th-century; repaired ceramics; glue; ambient ionization mass spectrometry

During excavations at the 18th-century colonial American site of Ferry Farm, broken ceramics with traces of glue were discovered, indicating that the objects were repaired before ultimately being broken and discarded. This historic archaeological site is important as it was the boyhood home of George Washington, the first President of the United States. We present here our characterization of the organic content of these 18th-century glues with ambient ionization – direct analysis in real time and paper spray -- mass spectrometry and the mineral composition through Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy-energy dispersive X-ray spectroscopy. In particular, we will compare the results from replica glues prepared from historic recipes based on cheese and milk, rendered animal collagen, and pine resins with those from the 18th-century glue samples. This reference collection of historic glue recipes allows us to test how these homemade glues function and change during burial, which aids in the interpretation of our analyses of the historic glue residues. While proteins are expected to break down rapidly when combined with calcium oxide lime, we were able to identify with paper spray ionization and time-of-flight mass spectrometry peptide markers in the replica glues of the milk protein casein. Sample preparation to remove the mineral fraction plays an important role in obtaining reliable ambient ionzation MS results on the small molecule biomarkers like resin acids and beeswax esters. The results of our analyses of these glue residues will aid us in better understanding how and why the Washington family used household materials to repair their ceramic vessels.

S3-P24.231

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeobotanical Investigations on Waterlogged Plant Material from the Late Neolithic Austrian Lake Settlements Weyregg II (Attersee) and Mooswinkel (Mondsee) – Insights in Food and Farming Practices

Jakobitsch, T.[1], Heiss, A.G., Dworsky, C.[3], Pohl, H.[4], Leskovar, J.[5]

[1]*Austrian Academy of Sciences, Austrian Archaeological Institute, Vienna, Austria*
[2]*Austrian Academy of Sciences, Austrian Archaeological Institute, Vienna, Austria*
[3]*Kuratorium Pfahlbauten, Vienna, Austria*
[4]*Kuratorium Pfahlbauten, Attersee am Attersee, Austria*
[5]*Oberösterreichisches Landesmuseum, Linz, Austria*

Keywords: archaeobotany; Late Neolithic; lake settlements; agriculture; Austria

Within the framework of the research project “Zeitensprung” (Upper Austrian State Museum, Kuratorium Pfahlbauten) archaeobotanical investigations on Late Neolithic lake settlements at the Austrian lakes Attersee and Mondsee have been carried out. Core samples from the lake settlements of Weyregg II (Lake Attersee) and samples from interval sampling of cultural layers at Mooswinkel (Mondsee) are being analysed for their organic macroremains. The outstanding preservation of the mainly waterlogged material allows reconstructing subsistence strategies during the Late Neolithic, under the aspect of following research questions: 1. Did the inhabitants of the pile dwellings grow their cultivated crops on permanent fields, or rather in a shifting cultivation system with yearly changing fields by the use of fire? 2. How important was the gathering of wild fruit for the subsistence of the pile dwellers? Did fruit gathering imply the tending of hedges as a cultivation measure of woodland for securing high yields? 3. How was animal husbandry performed? Where did their cattle, sheep and goats graze? Was this grazing land cleared with the use of fire? And did the pile dwellers perform pollarding and coppicing – a form of woodland management – in order to feed their flocks during winter?

The quantitative analysis of subfossil and charred cereal remains, weed seeds and gathered fruit remains as well as dendrological analysis of thin twigs will generate data for a better understanding of the subsistence strategies of the Late Neolithic pile dwellings at Lake Attersee and Mondsee.

S3-P25.280

Wine economy in Picton territory (1st - 2nd Centuries AD) Study of organic residues preserved in the locally produced amphorae

Frugier, C.[1,2], Absalon, C.[3], Saint-Raymond, C.[2] Bégué, J.,[2] Lemaître, S.[1], Pianet, I.[2].

[1]HeRMA, Hellénisation et Romanisation dans le Monde Antique, UR 15071, Université de Poitiers, Hôtel Berthelot, 24 rue de la Chaîne, 86 000 Poitiers. France.
[2]Archéosciences Bordeaux, Matériaux, Temps, Images, Sociétés, UMR 6034, CNRS, Université Bordeaux Montaigne, Université de Bordeaux, Maison de l'archéologie, Esplanade des Antilles, 33607 Pessac Cedex. France.
[3]CESAMO, Institut des Sciences Moléculaires, UMR 5255, CNRS, Université de Bordeaux, Cours de la Libération, 33405 Talence Cedex, France.

Keywords: Organic residues, Wine Amphorae, GC-MS, py-GCMS.

Several studies have enlightened the existence of local workshops in Picton territory, western center of Gaul. Picton potters notably reproduced the forms of Pascual 1 and Dressel 2/4 amphorae imported from the north of Spain, and Gauloise 4 and 5 amphorae originating from the Narbonensis province.
The chemical study of the vessels was conducted to ascertain the usage of the amphorae. Pyrolysis of 1-2 mg of crushed ceramic was performed to volatilise the organic residues embedded in the vessels pores before they were analysed by gas chromatography coupled with mass spectrometry (GCMS). This analysis allowed us to obtain the global molecular print of the organic content preserved in the amphorae1, and to select extraction processes adapted to their specific molecular nature.
In the particular case of the amphorae samples studied, two successive extracts were performed. A dichloromethane-methanol solution was first used to extract the resinic acids and their identification and quantification were performed by GCMS. It gave information about the softwood specie preferentially selected to produce the pitch used to waterproof the vessels. The second extraction targeted small specific organic acids, considered as wine markers2. The GCMS analysis of the extracts permitted to identify and quantify the different species present in the sherds. The results acquired determined the presence of malic, fumaric, syringic, vanillic and lactic acid.
Herein, we present the results obtained on the amphorae excavated in two archaeological sites located inside the Picton capital Limonum ("Rue de la Marne" and "Îlot des Cordeliers"). Two other Picton sites are currently under study.

S3-P30.492

Greek toned photographs of the beginning of 20th century. Investigation of the materials, the structure and the process

Tsantiri, K., Kokla, V.

Dept. Conservation of Antiquities & Works of Art, University of West Attica, Agiou Spiridonos 28, 12243, Egaleo

Keywords: photography; toning; techniques; material; examination

Photography, one of the most important discoveries of modern times, allows us to copy reality mechanically. It appeared in a period of the 19th century, where art and technology were undergoing radical changes. Part of these changes was photography itself, which was developed thanks to the development of chemistry. Before 19th century, the first photographs were simple projections of images onto a surface using the camera obscura. That camera can be considered as the first camera, but it had one major drawback: it couldn't maintain the image. Nicéphore Niépce was the first who managed to capture a permanent image of the reality.

The purpose of the present study is to investigate the techniques were used for the production of photographs in Greece during the first five decades of 20th century. Especially, it examined photographs that had been manufactured using the photographic print toning process. Until now, our knowledge on the photographic techniques were used in Greece is deficient. The items under examination in this research were 23 photographs belong to the first half of the previous century.

The applied methods were the multispectral examination, macroscopic and microscopic, the ATR-FTIR and the scanning electron microscope in combination with energy dispersive X-ray analysis (SEM-EDS). Based on these methods important knowledge was obtained on the structure and materials of photographs, as well as, on the specific process used in their manufacture. The application of multispectral examination gave information on the underlying layers, overpaintings and the materials' characterization. The Fourier-Transform Infrared (FTIR) Spectrometry helped the investigation of organic components of photographs and photographic coatings. The scanning electron microscope (SEM) was being used to determine the ingredients present in the materials and their mapping. The using the energy dispersive X-ray analysis in conjunction with the scanning electron microscope yielded information on the structure of the photographic materials and its chemical composition.

S3-P31.502

Diet and mobility in Late Roman Christian community: isotopic data from the Catacombs of Santa Lucia, Syracuse (Sicily)

Tanasi, D.[1], Tykot, R.H.[2], Hassam, S.[1], I. Gradante[3], T. Ricciardi[4]

[1]Department of History, University of South Florida, USA
[2]Department of Anthropology, University of South Florida, USA
[3]Berlin-Brandenburgische Akademie der Wissenschaften, Germany
[4]Pontificia Commissione di Archeologia Sacra - Ispettorato delle Catacombe della Sicilia Orientale, Italy

Keywords: ancient diet; stable isotopes; Romans; Sicily

The catacombs of Santa Lucia were built in the 4th century for the burial of members of the Christian community. Tomb design and furniture indicate both wealthy and poorer individuals, and written sources testify to a large Christian community from Syria and the Levant in Syracuse at the time. This research was undertaken to evaluate individual life-histories of this Late Roman Christian community buried in the catacombs through stable isotope analysis and shed light on differential dietary habits between individuals of diverse status and place of origin to reassess the evidence from the written sources.
Stable carbon, nitrogen, and oxygen isotope analysis was conducted using well-established procedures on skeletal samples from 29 individuals from the catacombs of Santa Lucia. These include skeletal bone samples from 8 individuals that were analyzed successfully for both bone collagen and bone apatite; 16 other individuals for both tooth root and tooth enamel; and 5 for just tooth enamel. The bone isotope values represent the average long-term diet, with collagen representing primarily dietary protein and apatite the whole diet, while the isotope values for teeth present its age of formation.
The results suggest that a few individuals were non-locals, either as an adult, or as a child. The higher-than-average carbon isotope values for three individuals may be explained by the formation of the tooth during the age of breastfeeding. For one, however, both the higher carbon and very high nitrogen isotope values suggest some seafood in the diet at least as a child. The tooth enamel values for another individual strongly suggest a high amount of the C4 plant millet and perhaps origins as a child from the Near East or North Africa. The isotopic results for Santa Lucia are compared with those for other Roman and early Christian sites in the Mediterranean.

S3-P32.503

Multi-analytical study of organic materials in reference painting mixtures and real samples from wall paintings, icons and easel paintings

Argirova, M., Lumov, N., Anastassova, N., Stoyanov, S., Velcheva, E., Guncheva, M., Dimitrov, M., Yancheva, D., Stamboliyska, B.

Institute of Organic Chemistry with Centreof Phytochemistry, Bulgarian Academy of Sciences, Acad. G. Bonchev St., build. 9, 1113 Sofia (Bulgaria), email: margirova@orgchm.bas.bg

Keywords: multi-analytical approach; identification; organic materials; IR spectroscopy; ELISA assay

The physicochemical characterization of cultural heritage materials could provide valuable information regarding the composition, origin, changes due to fabrication process and natural aging, causes of the destruction of an object, detection of later interventions, technology of production etc. This information greatly enriches the information obtained from the study of historical documents and other written sources. It is also of primary importance for choosing the most appropriate methodology for their conservation and restoration. The analysis and identification of organic materials is a challenging process due to their smaller amount in comparison to the inorganic pigments and mortar and easier deterioration. Therefore, it requires combined analytical methods and a multi-step approach. Herein we report the analytical study of a series of reference mixtures of pigments and binders of proteinaceous and carbohydrate type, reference mixtures of several organic binding or varnishing materials, such as natural resins or drying oils. The reference materials were characterized by IR and Raman spectroscopy, thin layer chromatography, pyrolysis-gas chromatography/mass spectrometry, thermogravimetric methods, differential scanning calorimetry, and enzyme-linked immunosorbent assay. Along with the reference mixtures, a series of samples from original painting materials were studied by applying the same combined methods. The samples were collected from wall paintings, icons and easel paintings under restoration. The multi-analytical approach established by the analysis of the reference mixtures proved helpful and efficient in identifying the organic materials in the studied real samples.

Acknowledgement

This work has been financially supported by the National Science Fund of Bulgaria, Contract КП-06-ОПР 05/5. Equipment of INFRAMAT (Research Infrastructure from National roadmap of Bulgaria), supported by Contract D01-155/28.08.2018 with Bulgarian Ministry of Education and Science is used in a part of the present investigations.

S3-P33.526

Fluorosis in Roman vesuvian populations: Fluorine concentrations in human remains from Cumae

Orellana-Gonzalez, E.[1,2,3], Dubernet S.[2], Csepregi, A.[4], Dönczö, B.[4], Duday, H.[1], Castex, D.[1], Chapoulie, R.[2]

[1]*Université de Bordeaux, CNRS, MC, UMR 5199 PACEA (Pessac – France)*
[2]*Université Bordeaux Montaigne, CNRS, UMR 5060 IRAMAT-CRP2A (Pessac – France)*
[3]*Ecole française de Rome, MESRI (Rome – Italy)*
[4]*Institute for Nuclear Research, (Atomki, Debrecen – Hungary)*

Keywords: endemic fluorosis; Cumae; fluorine; ecotoxicology, ancient Rome

Fluorosis is an intoxication caused by the excessive ingestion of fluoride. The main signs of this disease are an increase in bone density and a hypomineralization of tooth enamel. The main source of intoxication is the consumption of water, especially groundwater in volcanic areas, that contain high doses of fluorine, often exceeding WHO recommendations (1, 5 mg/l). Nowadays, the Vesuvius area is considered as an endemic zone for this disease.
Archaeological excavations conducted in Cumae (Italy) have unearthed a great number of cremation burials from the Roman period. Anthropological observations indicate that several individuals present bone alterations related to fluorosis. Previous LIBS analyses have suggested a higher intensity of fluorine for these individuals. Nevertheless, quantitative results of fluorine concentrations could help us understand disease stages and progression.
To achieve the quantification of fluorine concentration, a sample of 28 human remains was studied with PIXE-PIGE, including individuals with suspected fluorosis as well as individuals with no trace of this disease. To consider the possible differences in nature and shape, different bones were analyzed and different kinds of sample preparation were used. Artificial pellets with known concentrations of fluorine allowed the results calibration.
Our results show a clear difference in fluorine concentrations between the individuals with suspected fluorosis and the ones without, proving that the Cumae inhabitants had indeed a higher concentration of fluorine in their bones. The correlation of the different fluorine concentrations among our sample, the type of bone and the osteological signs have given us clues to understand the evolution of the disease. Furthermore, studying the relation of the intoxication with the environment, health, and lifestyles of past and present populations, can provide a better understanding of the ecotoxicology.

This research is carried out within the framework of a Ph.D. grant from the Ecole française de Rome. Funding for PIXE-PIGE analyses was allotted by the IPERION CH project. We kindly thank Zita Szikszai, Zsófia Kertész and Enikő Furu for their support to achieve the analyses at the Institute for Nuclear Research (Atomki, Debrecen – Hungary). Special thanks to the Centre Jean Bérard (CNRS/EFR, USR 3133, Naples – Italy) especially to Priscila Munzi and Jean-Pierre Brun for giving us access to the materials.

S3-P35.597

Isotopic Analysis of Skeletal Remains from Richardson's Hammock (8Gu10) in Northwest Florida (USA)

Tykot, R.H.[1], White, N.M.[1], Tullos, R.B.[2]

[1]*Department of Anthropology, University of South Florida, Tampa, FL 33620 USA*
[2]*Environmental Research Group, Orlando, FL, USA*

Keywords: isotope analysis, prehistoric Florida, ancient diet and mobility

Richardson's Hammock (8GU10) is a prehistoric burial mound in northwest Florida on a small peninsula attached to the larger St. Joseph Peninsula that encloses St. Joseph Bay. The site has a Middle Woodland component (AD 350-700), and an even larger Fort Walton (Mississippi-period) component (AD 1000-1500). The large artifact assemblages from the mound include Middle Woodland fancy ceramics (Swift Creek Complicated-Stamped, Weeden Island Incised and Punctate, including bird-effigy vessels); Fort Walton materials (Fort Walton Incised, Cool Branch Incised pots, an engraved shell gorget, a copper plate fragment); and objects from either period such as ground-stone celts, various shell beads, and chert projectile points.
Radiocarbon dates obtained for excavated charcoal in two units in the village area are cal AD 890-1020 and cal AD 1280-1400. Human skeletal remains and high-status artifacts had been dug by collectors from the mound in the 1970s, and recently this private collection became available for scientific analysis. Study of the human skeletal remains representing at least 10 individuals identified age and sex, pathologies, and other features such as cranial modification. One skull was identified as that of an African-American male, much more recent than the prehistoric Native Americans. Stable isotope analysis of bone collagen, bone apatite, tooth enamel, and tooth roots was conducted to reconstruct the life histories of these individuals. The bone isotope values represent the average long-term diet, with collagen representing primarily dietary protein and apatite the whole diet, while the isotope values for teeth represent its age of formation. Few if any isotope studies previously done in northwest Florida included bone apatite analyses, which is essential for assessing the importance of C4 plant foods such as maize in the diet and distinguishing that from positive carbon isotope values related to seafood consumption. The individuals in this study represent a time period around when maize had been introduced to this region, and when there may have been greater mobility of individuals in these more complex societies.
Our research questions specifically address whether these individuals represent both Middle Woodland and Fort Walton time periods; whether they were native to the region or did the later people come from elsewhere; and if all had the same diet, including seafood on the bay and Gulf, or did the Fort Walton people visit seasonally but also cultivated maize and other crops inland.

S3-P38.719

Preliminary results of the project Roman amphorae and content analysis. (RACAMed I and II)

Pecci, A.[1], Mleto, S.[1], Bernal, D.[3], Vargas Girón, J.M.[3], Contino, A.[4], Ripoll, G.[1], Reynolds, P.[1,2]

[1]Equip de Recerca Arqueològica i Arqueomètrica, Universitat de Barcelona (ERAAUB), Departament d'Història i Arqueologia, Barcelona, IA-UB, Spain
[2]ICREA, Barcelona, Spain
[3]Universidad de Cádiz, Spain
[4]Ministero per i Beni e le Attività Culturali e per il Turismo

Keywords: Amphorae contents; organic residue analysis; wine

The study of organic residues absorbed in amphorae gives important information on their use and presence of organic coatings. Here we present the preliminary results of the project "Roman amphorae and content analysis".Racamed I and II. In this occasion we focus on the results obtained from the analysis of the amphorae recovered in ancient Gades (Cadiz), Pompeii and Ostia, the ancient port of Rome.
The analyzed amphorae from Cadiz are: Ovoid amphorae type 1 and 5 (as well as undetermined), Haltern 70 amphorae, and Dressel 1 amphorae, all recovered during the El Olivillo project conducted by the University of Cadiz. The amphorae recovered at Ostia are Ancient African amphorae.
The amphorae were sampled and analyzed following the methods proposed by Charters et al. (1993) and Correa-Ascencio and Evershed (2014) for the identification of lipids, and Pecci et al. (2013) for the identification of wine residues.
Many of the amphorae from Gades were coated with abundant resin or pitch from Pinaceae, which was used as a waterproofing agent and perhaps, in some occasions, also to give flavor and preserve the content of the amphorae. However, this practice was not yet as widespread as in later times, when practically all the studied amphorae show an internal coating. Few of the African amphorae were coated.
Most of the ovoid amphorae display wine (or other grape derivatives) residues, suggesting the production and distribution of these substances during the analyzed period. However, this is not the only content identified and possible plant oil and/or animal products appear to have been stored in the analyzed vessels. The African amphorae display compounds compatible with a mixture of substances, including plant oil.
The study is financed by the Spanish I+D Projects RACAMed I and II (HAR2017-84242-P and PID2020-113409GB-I00) and Garvm II (HAR2016-78691-P), the FRI-UB 2017, and it is part of the activities of the ERAAUB (2017 SGR 1043).

Correa-Ascencio, M., Evershed, R.P., 2014. High throughput screening of organic residues in archaeological potsherds using direct acidified methanol extraction. Anal. Methods 6, 1330.
Charters S, Evershed RP, Goad LJ, Leyden A, Blinkhorn PW, Denham V. 1993. Archaeometry, 35,2, 211–23.
Pecci A., Giorgi G., Salvini L., Cau M. A., 2013. Journal of Archaeological Science, 40, 109-115.

S3-P39.727

Isotope Analysis of Neolithic and Early Copper Age Human Skeletons from the Central Po River Valley, Northern Italy

Eck, C.J.[1], Tykot, R.H.[1], Vianello, A.[1], Sperduti, A.[2], Cavazzuti, C.[2]

[1]Department of Anthropology, University of South Florida, USA
[2]Museo delle Civiltà, Rome, Italy

Keywords: ancient diet, mobility, stable isotopes, Neolithic Po Valley

The fertile alluvial plain of the Po Valley in Northern Italy appears to have received the Neolithic package and undergone a commitment to agropastoral subsistence strategies nearly 1000 years after preliminary introduction in southeastern Italy of C3 plants such as emmer wheat and barley. During the early and middle Neolithic transition a major shift in ceramic production has been documented and described as the Bocca Quadrata (square-mouthed) culture which coincides with the arrival of new C3 cultivars not found in the south such as hulled and free-threshing wheat grasses. In addition to a sustained commitment to domesticate C3 grasses it is clear from contextual zooarchaeological evidence that animal management played a major role in Central Po Valley Neolithic life. The present study involves a collection of bioarchaeological materials excavated over a ten-year period from five sites in the Mantua Province and reconstruct the diets and mobility of these early agropastoral groups arriving in waves throughout the early and middle Neolithic. It is hypothesized that mobility increases into the late Neolithic and early Copper Age as short-range transhumance of domesticate animals emerges.

Over 100 human and faunal bone and teeth samples were prepared in the Laboratory for Archaeological Science & Technology at the University of South Florida, with analyses done using a Delta+XL continuous flow isotope ratio mass spectrometer equipped with a Carlo-Erba NA2500 elemental analyzer for carbon and nitrogen in bone and tooth root collagen, a ThermoFisher MAT253 isotope ratio mass spectrometer coupled to a GasBench-II with a continuous-flow interface for carbon and oxygen isotopes in bone apatite and tooth enamel, and a Thermo Neptune Plus multi-collector ICP mass spectrometer for strontium isotope analysis of tooth enamel and bone.

This research is critically important to understanding the earliest land-use and the intensity of agricultural production in the region, how the environment and biodiversity has changed, and the human sociocultural roles and identities that shaped and continue to affect the modern agricultural landscapes. Through isotopic ecology the earliest agricultural production, settlement patterns, and animal management lifeways inform contemporary sustainable cultivation and food production along the fertile alluvial plains that were the home of Ötzi the iceman in Northern Italy.

S3-P41.808

Multidisciplinary investigation of two Egyptian child mummies at the University of Tartu Art Museum, Estonia

Oras, E.[1,2], Anderson, J.[3], Tõrv, M.[2], Vahur, S.[1], Rammo, R.[2], Remmer, S.[4], Mölder, M.[4], Malve, M.[2], Saag, L.[5,6], Saage, R.[2], Teearu-Ojakäär, A.[1], Peets, P.[1], Tambets, K.[5], Metspalu, M.[5], Lees, D.C. [7], Barclay, M.V.L.[7], Hall, M.J.R.[7], Ikram, S.[8,9], Piombino-Mascali, D.[10]

[1]Institute of Chemistry, Faculty of Science and Technology, University of Tartu, Tartu, Estonia
[2]Institute of History and Archaeology, Faculty of Arts and Humanities, University of Tartu, Tartu, Estonia
[3]University of Tartu Museum, Tartu, Estonia
[4]Estonian Forensic Science Institute, Tallinn, Estonia
[5]Institute of Genomics, University of Tartu, Tartu, Estonia
[6]Institute of Molecular and Cell Biology, Faculty of Science and Technology, University of Tartu, Tartu, Estonia
[7]The Natural History Museum, London, United Kingdom
[8]Department of Sociology, Egyptology and Anthropology, American University in Cairo, New Cairo, Egypt
[9]Department of Ancient Studies, Stellenbosch University, Stellenbosch, South Africa
[10]Department of Anatomy, Histology and Anthropology, Institute of Biomedical Sciences, Faculty of Medicine, Vilnius University, Vilnius, Lithuania

Keywords: mummy studies; Ancient-Egypt; archaeological sciences; bioarchaeology

Two ancient child mummies curated at the University of Tartu Art Museum (Estonia) were, according to museum records, brought to Estonia from Egypt by the young Baltic-German scholar Otto Friedrich von Richter, during the early 19th century. However, a detailed investigation resolving their authenticity, provenance and exact date was missing. An interdisciplinary team of experts was summoned to study the remains using the most recent analytical methods. By combining osteology and archaeothanatology, radiological investigation, AMS radiocarbon dating, multi-instrumental chemical and textile analyses, 3D modelling, entomological as well as aDNA investigation, we present a full-scale and exhaustive analysis of these child mummies. We were able to establish that the remains are indeed of ancient Egyptian origin, belonging to the Graeco-Roman period. Furthermore, our results provide a detailed insight into and new information on the mummification processes in the Graeco-Roman world with special emphasis on subadult mummies. As a wider scientific contribution we highlight the main analytical approaches for establishing detailed biological profile of the mummified bodies, their spatiotemporal provenance as well as nuances of mummification and wider funerary practices.

S3-P43.869

Session 4 Technology/provenance - stone/pigments/plaster

ORAL

To compare red and red: How to correlate rock painting and colouring matter artefacts? The case of the schematic paintings of the Rocher du Château (Bessans, Haute Maurienne, France)

Chalmin, E.[1], Chassin de Kergommeaux, A.[1,2], Kergourlay, F.[3,4], Martinetto, P.[3], Blanc N.,[4] Boudet N.[4], Bellot-Gurlet, L.[5], Defrasne C.[6]

[1]Université Savoie Mont Blanc, CNRS, EDYTEM (UMR 5204), Le Bourget du Lac, France
[2]Univ Lyon, ENS de Lyon, Université Lyon 1, CNRS, UMR 5276 LGL-TPE, Lyon, France
[3]Institut Néel, CNRS/UGA UPR2940, Grenoble, France
[4]ESRF, Grenoble, France
[5]Sorbonne Université, CNRS, MONARIS (UMR 8233), Paris, France
[6]Aix Marseille Université, CNRS, Ministère de la Culture, LAMPEA (UMR 7269), Aix-en-Provence, France

Keywords: Rock art; Schematic painting; Colouring matter; Integrated study; Synchrotron radiation

Presence of colouring materials in an archaeological layer at the bottom of a painted wall is an important clue to enable the elaboration of a potential chronological framework of the realisation of the rock paintings. However, establishing with certainty the link between the matter used to prepare the paint and the fragment of raw matter or waste of preparation is today a conceptual and analytical challenge. Similar colour and similar chemical compositions are not enough to enable this correlation.

The integrated study of the colouring matter is crucial to raise this challenge by using combined non-invasive in situ methods (digital microscopy and Raman spectroscopy) and analytical studies of micro-samples and artefacts (SEM-EDX, µXRD based on synchrotron radiation, PIXE).

One of the limitations of such a study is due to the weathering process on the rock surface happening before and after the application of the paint layer. The presence of mineral accretions or crusts limits the access to the colouring matter during in situ analysis as well as during the analysis of the micro-sample. However, understanding these weathering processes makes it possible to distinguish the components of the painting material.

We conducted complementary physicochemical analyses at several scales of observation to identify the composition of the excavated pigments and pigmented materials coming from the Rocher du Château (Bessans, Savoie) one of the rock shelters of the western Alps with schematic rock paintings attributed to the Neolithic period.

This remarkable serpentinite rocky point in the Arc Valley presents the coexistence of many red schematic paintings and colouring and coloured materials found in archaeological surveys. Occupancy periods associated with the Middle and Late Neolithic were identified during the three survey campaigns organised by E. Thirault (1997, 2002, 2003). Some of the colouring materials proved to be anthropogenic blends combining hematite and charcoal of plant origin. This association has never been identified before in other prehistoric European archeological sites. Based on these data, the potential links between the pebbles, the production of pigments, and the rock paintings are discussed. Understanding these colouring materials also allows us to question the supply strategy for these raw materials as well as the technical level developed by the society of the Neolithic period.

S4-O01.823

Microbes and Microanalysis: Hunter-Gatherers Harvested and Heated Biogenic Oxides to Produce Rock Art Paint

MacDonald, B.L.[1], Stalla, D.[2], He, X.[2], Maschmann, M.[3], White, T.A.[2]

[1]Archaeometry Laboratory, University of Missouri Research Reactor
[2]Electron Microscopy Core, University of Missouri
[3]Mechanical and Aerospace Engineering, University of Missouri

Keywords: rock art; pigment; microanalysis; electron microscopy

Red pigments, often termed ochre, are the most commonly identified mineral pigments used throughout prehistory, and ochre use is increasingly recognized as fundamental component of traits associated with human evolutionary development, social interaction, and behavioral complexity. Ochre mineral deposits have been collected and prepared as pigment for use in rock art, personal adornment, and mortuary practices for millennia, yet little is known about early developments in mineral processing techniques in North America. Recent advances in microanalytical applications, including electron microscopy methods, Raman spectroscopy, and high-sensitivity magnetometry, have created new avenues for understanding rock art microenvironments and the preparation and use of mineral pigments in the past. We present the results of a multi-method investigation of rock art at Babine Lake, British Columbia (Canada). Our study has revealed a sophisticated use of iron oxide produced by the biomineralizing bacterium Leptothrix ochracea; a keystone species of chemolithotroph recognized in recent advances in the development of thermostable, colorfast biomaterial pigments. Here, we show evidence for the use of this bacterium, including nanostructural and magnetic properties evident of thermal enhancement, indicating that controlled use of pyrotechnology was a key feature of how biogenic iron oxides were prepared into paint. Our results demonstrate that the hunter-gatherers in this area of study prepared pigments by harvesting aquatic microbial iron mats dominated by iron-oxidizing bacteria, which were subsequently heated in large open hearths at a controlled range of 750°C to 850°C. This technical gesture was performed to enhance color properties, and increase colorfastness and resistance to degradation. This skilled production of highly thermostable and long-lasting rock art paint represents a specialized technological innovation. Our results contribute to a growing body of knowledge on historical-ecological resource use practices in the Pacific Northwest during the Late Holocene.

Shape, technological evolution, provenance and trade of the volcanic millstones in the Central-Western Mediterranean

Renzulli, A.[1], Santi, P.[1], Gambin, T.[2]

[1]*Dipartimento di Scienze Pure e Applicate, Università degli Studi di Urbino Carlo Bo, Urbino, Italy*
[2]*Department of Classics and Archaeology, University of Malta, Malta*

Keywords: Grinding technology; saddle quern; hopper-rubber millstone; rotary millstone; Central-Western Mediterranean volcanoes

Most of the volcanic millstones for grinding cereals are made of lava lithotypes, generally characterized by wear resistance and suitable abrasive properties, often supported by the rough vesicular surface. Since the Late Paleolithic Period, grinding stones occurred in a variety of shapes: oval/sub oval, rectangular, triangular and other irregular shapes which were primarily used as base-stones. A small, roughly squared hand-pebble of hard stone was then used to grind the cereals, causing many of the base-stones to have a smooth concave surface (saddle shapes). Saddle and flat querns became widespread throughout the Central-Western Mediterranean during the Bronze and Iron Ages. Starting from the 5th century BC, a great innovation took place somewhere in Greece with the development of the so-called hopper-rubber mills (Olynthian-style), operating on reciprocal motion, as did the querns, but with the upper stone widened with a cut into its circular cavity. Rectangular hopper-rubber varieties were also very widespread. In the same period, small rotary hand querns (operated by one person) probably originated in Spain. From the 4th–3th century BC, a fundamental technological advancement was made by the more efficient Morgantina-style rotary millstones which had to be operated by two persons because of their larger size, characterized by a base stone (*meta*) and an hourglass-shaped upper stone (*catillus)*. However, the largest, most impressive and best-known hourglass rotary millstones are certainly the Pompeian-style ones, often animal-driven (*mola asinaria*), whose invention, with perfectly reversible *catillus,* date back to the first century BC. The increase of public milling of grain and the development of baking facilities made the Pompeian-style millstones very widespread in the towns of the Imperial Age (e.g., *Pompeii* and *Ostia*).
Petrography and major-trace element geochemistry allowed to establish the provenance of the volcanic millstones found in several archaeological sites, from the Phoenician-Punic period to the Roman Empire, throughout the Central-Western Mediterranean and beyond the Strait of Gibraltar (i.e. Cádiz). The volcanic millstone trade was defined, starting from the exploited volcanoes in Sicily (Etna, Iblei, Ustica Island and Aeolian Archipelago), Sicily Channel (Pantelleria Island) and Central-Southern Italy (Vulsini Volcanic District, near Orvieto and Somma-Vesuvius volcano) up to the final destinations (archaeological sites) hundreds to 1500 km far away from the volcanic sources. Studies of well-preserved shipwrecks in Central-Western Mediterranean containing volcanic millstones (e.g. off the island of Gozo, Malta) strongly contributed to outline the volcanic millstone sea-trade.

S4-O03.135

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Mithras in Tróia (Portugal). Analysis of the Roman marble bas-relief with the banquet of the Gods Mithras and Hélios (3rd c CE)

Limão, F.[1], Lapuente, P.[2]

[1]Post-doctoral Fellow by the Portuguese Foundation for Science and Technology (FCT). Member of the History of Art Institute of the Faculty of Social Sciences and Humanities, Nova University, Lisbon (FCSH-NOVA)
[2]University of Zaragoza, UNIZAR Department of Earth Sciences

Keywords: Tróia (Portugal); Mithraic bas-relief; Marble decoration; Estremoz Anticline; Archaeometry

Tróia is the name of a sandy peninsula on the left bank of the river Sado in the southwestern Atlantic coast of Portugal. On the north tip of the peninsula close to the estuary of the river facing the Roman city of *Caetobriga* (currently Setúbal) in the mainland, a Roman industrial settlement based on the production of salt-fish goods evolved since the first century CE. In the mid forth century CE an early Christian basilica with frescoes was erected in an area previously covered by salt-fish vats. Life in the settlement of Tróia would cease during the fifth/ sixth centuries CE and the place would fall into oblivion until the sixteenth century.
When archaeological excavations in Tróia started in the turn of the twentieth century, five sculpted fragments of white marble were found in an area close to the Early Christian Basilica as archaeologist Marques da Costa later described (1934). When reassembled, the fragments composed a panel with the depiction of the banquet of the gods Mithras and Helios. Although incomplete, the panel made part of a larger composition, possibly a diptych or triptych, where the ritual of the killing of the bull - the *taurobolium* - might have been represented as well. Since its discovery, the bas-relief of Mithras has been considered as one of the finest of its genre as immediately stated by Franz Cumont. Currently, a replica of the bas-relief of Mithras is on display at the National Museum of Archaeology in Lisbon whereas the original is in private hands.
The goal of our work is to introduce the analysis carried out to determine the provenance of the marble of the Mithraic bas-relief of Tróia. A multi-method approach combining polarized-light microscopy, cathodoluminescence, X-ray powder diffraction, and stable C and O isotope analysis was applied to identify the marble provenance. The comparison of the results with the available databases confirms the use of a statuary white marble variety from the Lusitanian Estremoz Anticline, in the Alto Alentejo.
With this procedure it is expected to obtain a valuable complementary source of information for the study of the cult of Mithras in the westernmost part of the Roman province of Lusitania, a work still to be done. Moreover, this analysis is part of a wider context of research aiming at understanding the architecture and decoration in Tróia during Antiquity.

S4-O04.137

Marble provenance study in Delos Island architecture

Vettor, T.[1], Sautter, V.[1], Jolivet, L.[2], Moretti, J.-C.[3], Pont, S.[1]

[1]Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie (IMPMC), UMR CNRS 7590,Muséum National d'Histoire Naturelle, Sorbonne Université, 75005, Paris, France
[2]ISTeP, UMR CNRS 7193, Sorbonne Université, Paris, 75252, France
[3]CNRS, Institut de Recherche sur l'Architecture Antique, Université Lumière Lyon 2, MOM,Lyon, 69365, France)

Keywords: Marble; Quarry provenance; Antiquity period; Delos Island; Carbon and oxygen isotopes

This study presents for the first time geochemical data on the ancient (Antiquity period) marble quarries of Delos Island and architectural marbles from its famous and exceptionally well-preserved archaeological site. The study was conducted after Hadjidakis et al (2003) work describing for the first time Delos quarries at macroscopic scale. Delos geological substratum is mostly composed of granite including four decametric marble enclaves that were excavated during Antiquity. It has been shown that a large quantity of marble had to be imported from neighbouring Cycladic Islands and continental Greece but their provenance remains mostly unknown.

The present study reports two provenance methodologies applied to Delian marbles. The first one is non-invasive, based on a portable X-ray fluorescence (pXRF) analyser detecting major and trace elements, tested in association with Principal Component Analysis (PCA). The second one is micro-destructive, mainly based on Maximum Grain Size measurement and isotopic ratios of carbon and oxygen ($\delta^{13}C$, $\delta^{18}O$). A few thin sections and X-Ray Diffraction analyses were also used. The non-destructive approach brought preliminary promising results while the multi-technique approach obviously allowed to go further into our provenance diagnostics. Our results allow to decipher indigenous well characterized Delos marble from imported marble coming from well define quarries of neighbouring Cycladic Island.

S4-O05.1506

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Mortars of the Danube limes in Serbia. Building the Roman frontier

Nikolić, E.[1], Delić-Nikolić, I.[2], Miličić, Lj.[2], Jovičić, M.[1], Vučetić, S.[3], Ranogajec, J.[3]

[1]*Institute of Archaeology, Serbia*
[2] *Institute for Testing of Materials, Serbia*
[3] *Faculty of Technology Novi Sad, University of Novi Sad, Serbia*

Keywords: Roman mortars; Danube Limes; lime mortars; raw materials; pozzolanic properties

The project Mortar Design for Conservation - Danube Roman Frontier 2000 Years After (MoDeCo2000) explores Roman mortars of the former Danube Limes in Serbia. Its concept encompasses the comprehensive cross-disciplinary research into raw materials and technologies used for the creation of lime mortars, conducted with the aim of providing scientific information that could be used for the proper conservation of this unique serial monument which aims to be a part of the UNESCO WHS List.

The sampled buildings were built and used within the time span of the period from the 1st to the 6th century AD. Lime mortars originate from forty military and civilian buildings - legionary fortresses, auxiliary forts, smaller fortifications, and Trajan's bridge, baths, houses, and tombs. After the research done by archaeologists, architects, geologists, chemists, and engineers of materials, the mortars were examined using stereo optical and digital microscopy; spectrophotometry with colourimetry; analyses of physical and mechanical characteristics; thermal characterisation; and characterisation of samples, binders, and aggregates using XRF, XRD, FTIR, and Raman spectroscopy techniques. For the first time, some of the raw materials found in the region of the Danube in Serbia have been recognized as components of the Roman mortars used at this territory. At the same time, the completely unknown topic of the natural materials with pozzolanic properties used for the Roman mortars in the territory of today's Serbia has been illuminated, giving us the opportunity to analyze their provenance.

Construction and masonry techniques used in the Roman period at the territory of today Serbia followed principles of Roman architecture but used its varieties present in the eastern part of the Empire. They were rational, with the predominant use of local raw materials, but also with the use of hard to deliver materials for the most important military and public buildings, or for wealthy private investors. The research of mortars has confirmed this thesis and contributed to the knowledge of the economy, and trade routes used for the exploitation and transport of building materials, but also investors, masons, or inhabitants, offering insights into different aspects of life in a Roman period along the entire Danube frontier.

This research was supported by the Science Fund of the Republic of Serbia, PROMIS, #6067004, MoDeCo2000

S4-O06.1575

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Weathering of archaeological artefacts and archaeological interpretation. On the example of finds from north-eastern Poland.

Januszek, K.[1], Klecha, A.[2], Woronko, B.[3]

[1]Institute of Archaeology, University of Warsaw
[2]Antiquity of Southeastern Europe Research Centre, University of Warsaw
[3]Faculty of Geology, University of Warsaw

Keywords: weathering; scanning electron microscope; flint artefacts; Bell Beaker set; north-eastern Poland

Weathering can be understood in many ways. Usually, it is considered as destructive. However, this process, mainly concerning post depositional changes, can also be useful in interpreting artifacts and related behaviours. A unique example of this are the flint artefacts - they preserve on their surfaces not only technological and functional traces, but also changes resulting from the effects of natural events. They vary from one substrate to another, making it possible to trace these changes. A good example of this are flint artifacts from north-eastern Poland. Found in four ritual features and identified with Bell Beaker set, are an example of quite unique relics of heterogeneous provenance. Forty flint artefacts found there were examined using a scanning electron microscope. Analyses showed the presence of various weathering changes, which covered the examined artifacts. In order to examine their provenance, quartz grains from the direct vicinity of the finds were additionally analysed. The comparison made it possible to identify which changes are local and which are not. As the results of these analyses showed, the differences in the types of weathering are connected with a certain selection of flint objects. The reason for choosing them was probably an undefined ritualism, confirmed also by lipid analyses. They resulted in ritual deposits found in recent years. Apart from various exotic eco- and artifacts, they contained also this set of previously selected flint artifacts of various state of preservation. The aim of the research is to show that weathering can be understood not only as destructive, but also useful in archaeological interpretation.

S4-O07.806

Spectral imaging for stone surface investigation: the case of Horologion of Andronikos Kyrristos in Roman Agora, Athens, Greece

Alexopoulou, A.[1], Kaminari, A.A.[2], Tsoniotis, N.[3], Panou, E.[2]

[1]ARTICON Research Laboratory - Conservation and Promotion of Visual Arts, Books and Archival Material, Faculty of Applied Arts and Culture, University of West Attica, Campus 1, Aghiou Spiridonos 12, Egaleo 12243, Athens, Greece, athfrt@uniwa.gr
[2]Department of Conservation of Antiquities and Artworks, University of West Attica, Campus 1, Aghiou Spiridonos 12, Egaleo 12243, Athens, Greece
[3]Ephorate of Antiquities of Athens City, Ministry of Culture and Sport, Makriyanni 2-4, Athens 11742, Athens, Greece

Keywords: Horologion of Andronikos Kyrristos; spectral imaging, non-destructive testing, near infrared, stone

Horologion of Andronikos Kyrristos, also known as Tower of the Winds, is an octagonal marble building dating from the 1st century B.C., located on the Roman Agora in Athens, Greece. The monument is still in good condition today and attracts particular interest as one of the most important masterpieces of late Hellenistic architecture. It was a kind of planetarium of the time and it also served for timekeeping, thanks to multiple sundials and a hydraulic clock inside it. In the early Christian era, the monument was converted into a church, while in the 18th century it was used as an Islamic prayer site. After the liberation of Greece in 1828, it was considered part of the antiquities of Athens and was used as a temporary museum. Since 2013, it has been intensively and systematically studied from various angles - archaeological, physicochemical and astronomical - within the framework of a cultural heritage conservation project.

The present work refers to the application of modern diagnostic physicochemical methods in order to collect data useful for solving conservation problems and to document the different historical phases of the monument. The study focused on investigating the stone surface and the locally preserved decoration in visible and invisible radiation and on detecting color traces, engravings, graffiti and inscriptions associated with its historical phases and its use until more recent years. The study was carried out on all the interior surfaces of the building as well as on the exterior sculpture surfaces of the upper zone. Non-destructive testing was applied by means of imaging techniques in the visible and near infrared spectrum (Macro photography in normal and tangential illumination, Infrared Reflectography / SWIR Thermography, Hyperspectral Imaging (HSI) combined with False Colour Infrared (FCIR) and Spectal cube Spectroscopy).

It is noteworthy that these techniques, already widely used for painting diagnostics, have been for the first time successfully applied in a thorough and systematic manner to study large immobile stone surfaces. As a result, we were able to extract useful information regarding conservation status, surface morphology and archaeological history.

The successful implementation of this standard methodology on an immobile monument and the overcoming of the challenges associated with it expanded the capabilities of the methods, improved material observation and assisted in revealing and interpreting elements that are often valuable but invisible to the human eye. Consequently, this proved that modern non-destructive techniques are rightfully considered as state-of-the-art technology in the diagnostics of artworks and monuments.

S4-O08.768

New contributions to the analytical characterization of Asian lacquers: provenance and technology of an elaborate decoration

Pérez-Arantegui, J.[1], Tamburini, D.[2], Bonaduce, I.[3], Ribechini, E.[3], Gallego, C.[4]

[1]*Instituto de investigación en Ciencias Ambientales de Aragón (IUCA), Universidad de Zaragoza, 50009 Zaragoza, Spain*
[2]*Department of Scientific Research, The British Museum, London, UK*
[3]*Dipartimento di Chimica e Chimica Industriale, Università di Pisa, 56124 Pisa, Italy*
[4]*Museo de Zaragoza, 50001 Zaragoza, Spain*

Keywords: Pyrolysis-GC-MS; SEM; Provenance; Technology; Asian lacquers

Asian lacquer objects have always been very appreciated artworks, but they also involve important historical knowledge on production centres and trade routes and contacts. For instance, they were produced from the sap of three lacquer trees with different regional provenances: Rhus vernicifera (growing in China, Japan and Korea), Rhus succedanea (in Vietnam and Taiwan), and Melanorrhoea usitata (in Laos, Burma, Thailand and Cambodia), and with three different main chemical components of the product extracted: urushiol, laccol and thitsiol, respectively. Moreover, several pigments were also used to decorate oriental lacquers. Therefore, characterization of these artworks can add significant data to understand production, trade and influences of the Asian objects. Within this framework, a research project on the study of the production technology and the provenance of lacquers, including also European contacts, was considered.

Several objects (furniture, boxes, etc.), from different provenances and dated between the 16th and 20th centuries, were sampled and studied. They belong to the Asian art collection at the Museum of Zaragoza (Spain). Due to the material complexity and the micro-sampling requirements, pyrolysis coupled with gas chromatography and mass spectrometry (Py-GC MS) is the most suitable analytical approach for the chemical analysis of lacquers and for their identification in a sample. Moreover, cross-sections were examined by Scanning Electron Microscopy, coupled to Energy Dispersive X-ray Spectrometry (SEM-EDS), and other analyses by spectroscopy (IR and visible) were also carried out. The analytical results highlighted different provenances of these objects, from several geographical areas, and a complex preparation and treatment of the decorative surfaces, adding new insights into their technology of production.

S4-O09.568

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of multi-layered plaster samples from Neolithic Çatalhöyük

Güzel, A.[1], Meral-Akgül, Ç.[2]

[1]Middle East Technical University, Department of Archaeometry, Ph.D. Student, Ankara-Turkey
[1]Middle East Technical University, Central Laboratory, Ankara-Turkey
[2]Middle East Technical University, Department of Civil Engineering, Ankara-Turkey

Keywords: Çatalhöyük; Neolithic Plaster; XRF; BSE; FTIR; XRD; EPMA; SEM-EDS; Micro-CT

Çatalhöyük is a settlement in Central Anatolia, which is thought to host an average population of 3000 to 8000 people approximately 9000 years ago. Its residents lived in mudbrick houses clustered into an aggregate structure. Settlement composed of two mounds, the East Mound (the main mound) consisting of 18 archaeological levels, is dated to the Neolithic period, whereas the West Mound is dated to the Early Chalcolithic period. The settlement formed by filling the abandoned, old houses and building newer ones on top of them. The inner walls and floors of the mudbrick houses are covered with plaster.

The plastering technology has changed over the settlement's life span. The time of the technology transitions is not precise. In the early levels, fired-lime plasters and thick white marl plasters are found together. From about 6800 to 6400 BC, the technique changed to multi-layer plastering. This technique is based on frequent repetition (typically fifty to one hundred repetitions) of two-layered plaster prepared from two different types of sediment: the foundation layers from buff base clay and the finishing layers from magnesium-rich softlime. Over time, plastering with a single thick layer of white/off-white replaced the multi-layering technique. Finally, the emphasis on white plastering was faded, and buff marls were also used. The use of pebbles in the floor plasters was observed.

Within the scope of this study, multi-layered plaster samples taken from South and TPC areas of Çatalhöyük belonging to different buildings of the Neolithic period have been examined. Differences between the chemical (XRF, BSE), mineralogical (FTIR, XRD), and morphological (EPMA, SEM, Micro-CT) properties of the plasters are presented. Obtained results are used to evaluate the raw material characteristics utilized within different layers of the plasters.

S4-O10.445

Identification of the organic binding medium and fungi on wall paintings from the Victory Monument of Augustus in Nicopolis, using HPLC-FD, Culture Media Methods, Optical Microscopy and SEM

Tziamourani, E.[1], Facorellis, Y.[1], Karabotsos, A.[1], Zachos, K.L.[2]

[1]Laboratory for the Study and Conservation of Ancient and Contemporary Cultural Property, Faculty of Applied Arts and Culture, Department of Antiquities and Works of Art Conservation, University of West Attica
[2]Emeritus Curator of Antiquities, Hellenic Ministry of Culture

Keywords: Nicopolis, HPLC, Aspergillus, OM, SEM

The monument to the Actium victory raised by Octavian Augustus on the northern outskirts of Nicopolis, in the area where the Actian games were taking place, consists of an enormous podium built in ashlar masonry on the slopes of a hill sacred to Apollo. In the southern wall of the podium, which consisted the façade of the monument, 36 bronze rams from the captivated ships of Antony were displayed, which, according to the Latin inscription that adorned the upper part of the wall, were dedicated to Mars and Neptune. On the upper part of the terrace created by the podium a porticus triplex was surrounding a courtyard. The area of the courtyard was occupied by a monumental altar and by two pedestals carrying bronze statues of unknown figures. The walls of the porticus and of an annex on its north wing, were decorated with frescos, some fragments of which were preserved in situ, while a considerable number of other fragments were found scattered throughout the monument.

In this paper we present the results of the identification of the organic binding medium, as well as the isolation of microorganisms from the wall paintings fragments using HPLC-FD and classical microbiological isolation techniques (OM and SEM), respectively.

Ten samples were selected for HPLC-FD analysis and compared with two reference mixtures of egg yolk with gelatin 3:1 w/w and egg yolk with gelatin 5:1 w/w. Six of the ten samples gave proteinaceous profile results, yielding an amino acid profile similar to the mixture egg yolk/gelatin 3:1 w/w, which is relatively poor in gelatin content.

A total of 57 cultures (19 samples for each medium) were examined during a five days study period. The fungal species isolated were Aspergillus species (15.79%), Penicillium species (10.53%), unidentified strains of mold and fungi (68.42%) and Bacillus species (5.26%).

The purpose of this study is to assess the present day condition of the wall paintings for future investigation. It is well known that the metabolism of organic bindings in the substrate, like animal or plant glue lose their binding effect when they are contaminated or when they have been in use for an extended time and a consolidation is required. During microbial infestation, these organic bindings decompose thus losing their consolidation effect. Metabolism of organic bindings is not only restricted to natural organic bindings, but also affects synthetic polymers applied for consolidation purposes.

S4-O11.698

Ancient pozzolanicity revisited: new insights into raw materials, mix designs and reactive processes of Roman binders

Secco, M.[1,2], Ricci, G.[2,3], Dilaria, S.[1], Garbin, E.[2], Tamburini, S.[4], Asscher, Y.[5], Artioli, G.[2,3], Previato, C.[1], Bonetto, J.[1]

[1]*University of Padova, Department of Cultural Heritage (DBC)*
[2]*University of Padova, Inter-Departmental Research Center for the Study of Cement Materials and Hydraulic Binders (CIRCe)*
[3]*University of Padova, Department of Geosciences*
[4]*National Research Council, Institute of Condensed Matter Chemistry and Technologies for Energy (CNR-ICMATE)*
[5]*Israel Antiquities Authority (IAA)*

Keywords: pozzolanic reaction; *opus caementicium*; X-ray powder diffraction; nuclear magnetic resonance spectroscopy; scanning electron microscopy

The discovery of pozzolanic reaction through the employment of reactive materials represents the most relevant technological evolution in the field of inorganic binders since the beginning of pyrotechnology. Pozzolans are amorphous aluminosilicate compounds capable to react with lime and water and precipitate nanostructured insoluble phases with excellent mechanical, permeability and durability properties.
After the first applications by ancient Mediterranean societies of the Second and First Millennium b.C., the potentialities of pozzolanic binders were fully exploited by ancient Romans, which used them for large-scale structural purposes through the formulation and development of Roman concrete (opus caementicium) [1].
Driven by the optimization of production technology, the Roman use of pozzolanic binders systematically tested the most suitable geological sources of pyroclastic materials available in Centre-Southern Italy, as testified by written accounts such as Vitruvius' De Architectura. The following standardized use of the best materials led to relevant trades of raw materials throughout the Roman Empire. Nevertheless, there is evidence that established supplies were often overcome by the employment of local pozzolanic materials, both natural and anthropogenic, with obvious economic and social advantages.
In this study, a combined mineralogical-spectroscopic-microstructural analytical approach has been adopted for the characterization of the mineralogical and crystal-chemical features of Roman pozzolanic binders. Several materials collected from various archaeological sites in the Italian peninsula and around the Mediterranean region were investigated.
The study demonstrated that, besides the import of traditional pyroclastic compounds, several alternative materials were used by Roman craftsmen, including natural products such as volcanic breccias, microcrystalline sedimentary silicates, and artificial compounds such as ceramic by-products and combustion residues. The obtained information on microstructural features, reaction interfaces, short range atomic structure of hydration products, and extent of hydraulic reactions allowed a better understanding of the advanced levels of technical knowledge and the exceptional physical and engineering performances of the Roman structural materials. A new model for the reaction products of Roman pozzolanic materials is presented.
[1] Artioli G., Secco M., Addis A. The Vitruvian legacy: Mortars and binders before and after the Roman world. EMU Notes in Mineralogy, Vol. 20, Chpt. 4, 151-202, 2019

S4-O12.428

Pre-screening Hydraulic Lime-Binders for Disordered Calcite in Caesarea Maritima: characterizing the chemical environment using FTIR

Asscher, Y.[1], van Zuiden, A.[1], Elimelech, C.[1], Gendelman, P.[1], 'Ad, U.[1], Sharvit, J.[1], Secco, M.[2,3], Ricci, G.[2,4], Artioli, G.[2,4]

[1]*Israel Antiquities Authority, Israel*
[2]*Inter-Departmental Research Center for the Study of Cement Materials and Hydraulic Binders (CIRCe), University of Padova, Italy*
[3]*Department of Cultural Heritage (DBC), University of Padova, Italy*
[4]*Department of Geosciences, University of Padova, Italy*

Keywords: Hydraulic Plaster and Mortars, Pozzolana, FTIR Grinding Curves, pXRF, Caesarea Maritima

Hydraulic lime binders are considered a technological marvel that revolutionized construction techniques in antiquity. The core material is made of a binder that is a mixture of calcite and hydraulic phases, which are amorphous silicate compounds that nanostructurally-polymerize into insoluble phases that harden even underwater, formed during the reaction between lime and reactive silicates such as volcanic ash. These silicate phases are poorly ordered, which makes them hard to identify using X ray Diffraction (XRD) and thin sections analysis. The calcite fraction forms upon the incorporation of atmospheric carbon dioxide during the setting of the hydrated lime. Therefore, different characterization methods are being constantly developed for identifying and characterizing the different components of hydraulic lime-binders. In this work, we present a rapid characterization technique based on Fourier transform infrared spectroscopy (FTIR) that characterizes the atomic disorder and chemical environment of the carbonates and silicates fractions in the binder. The atomic disorder of the calcite crystallites was determined by the v2 and v4 vibrational modes, and the silicates were characterized by the main peak asymmetry and full width at half maximum (FWHM). Different hydraulic binders from Caesarea Maritima were examined, including Herodian mortars from the underwater breakwater and on-land plasters and mortars from the port's warehouse and vaults. Hydraulic binders, in which the calcite fraction in the binder shows atomic disorder that is comparable to modern plaster binders, was associated with silicates that have asymmetry and FWHM of clays and quartz. Interestingly, the atomic disorder of binders that underwent chemical alterations and recrystallization processes, are associated with reactive silicates aggregates such as volcanic ash (pozzolana). The binder is a mixture of calcite and hydraulic phases indiscriminately; therefore, the enhanced ionic exchanges of the hydraulic phases presumably promote the recrystallization processes in the calcitic fraction they are in contact with. These results were corroborated using a hydraulicity index based on chemical analysis of a portable X ray fluorescence spectrometer (pXRF) and XRD examinations. This is a new method to pre-screen materials for hydraulic products, recrystallization process and state of conservation, showing different hydraulicity between underwater and on-land structures in Caesarea Maritima.

Neolithic Obsidian Trade and Distribution to Northern Italy and Southern France

Tykot, R.H., Vianello, A.

Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: obsidian trade; Neolithic Italy; XRF

In the central Mediterranean, obsidian from four islands was acquired starting in the Early Neolithic (ca. 6000 BCE) and distributed long distances to sites in northern Italy. The nearest is the tiny island of Palmarola, several hundred km to the south, while Lipari, Monte Arci (Sardinia), and Pantelleria are even further away. Previous studies demonstrated that obsidian from all four reached this far north, raising questions about quantity and frequency from each and how they may have changed over three millenia. Our study used a non-destructive portable X-ray fluorescence spectrometer to conduct analyses within many different museums and storage facilities in Italy, with the results calibrated using a widely-shared set of 40 obsidian standards and assigned to specific sources and subsources by direct comparison with a large set of geological samples from the central Mediterranean sources which were analyzed with the same pXRF. This research significantly increased the number of archaeological sites and artifacts tested, allowing statistical comparisons between time periods and sites and provides some interpretations for the socioeconomic factors involved in this variation.

The sites tested in this study include Pescale (Prignano), just southwest of Modena, excavated by an early archaeologist and dating to the Middle-Late Neolithic. The vast majority of the 926 artifacts tested are small blades. The sites of Case Catena (56 artifacts), Pontetaro (66), and Guidorossi (47) are all near Parma, and date to the Early-to-Middle Neolithic. Along with mostly small blades, obsidian cores have been found at the first two of these sites, confirming the local production of the final tools used. More recent archaeological surveys and excavations have revealed obsidian artifacts on four of the northern Tyrrhennian islands, and we conducted analyses of artifacts from Capraia (17), Giannutri (97), and Pianosa (231). These data are combined with unpublished results from the first author's previous analysis of 63 obsidian artifacts from the island of Giglio. About 35 obsidian artifacts from several sites in northeastern-most Italy were also analyzed. The results obtained in this study show striking differences between the sites in the distribution patterns of obsidian from each source, as well as changes over time which may be related to increasingly complex socioeconomic patterns over the course of the Neolithic. The obsidian distribution patterns are used to propose potential transportation routes while assessment of the typo-technology of the artifacts addresses the involvement of lithic specialists in various stages of the *chaîne opératoire*.

S4-O14.456

^{13}C, ^{25}Mg and ^{43}Ca solid state NMR for dolomitic marbles provenance purpose

Pianet, I.[1], Gutiérrez Garcia-M., A.[2], Savin, M-C.[1], Trebosc, J.[3], Florian, P.[4], Cuchí, J.A.[5], Lapuente, P.[2,6]

[1]*IRAMAT-CRP2A, Maison de l'Archéologie, Esplanade des Antilles, 33600 Talence, France*
[2]Institut Català d'Arqueologia Clàssica (ICAC), Tarragona, España
[3]*Unité de Catalyse et Chimie du Solide, UMR 5181 université Lille II, France*
[4]*CEMTHI, CNRS, Orléans, France*
[5]*Ciencias Ambientales, Escuela Politécnica Superior (Huesca), Universidad de Zaragoza, España*
[6]*Petrología y Geoquímica, Departamiento de Ciencias de la Tierra, Uuniversidad de Zaragoza, Zaragoza, España*

Keywords: White Marbles; Dolomitic; NMR; Provenance

Dolomitic marble ($CaMg(CO_3)_2$) was widely used for fine arts during Classical Antiquity in the Greco-Roman world due to its high quality. While rather scarce, its resource is not limited to the Thassos Island in the Mediterranean Basin, as other quarries probably exploited since the Late Antiquity are also now known. This is the case of the quarries of Mijas-Coín located in the Málaga province, in the *Hispanic Baetica* Roman province or those of the quarry of Lez in the French Saint-Béat district, Haute Garonne province whose use for archaeologic artefacts is being investigated. To the extent of our knowledge, the distribution of the two first marbles during Roman times followed different routes: if the Greek marble is found throughout much of the Empire, including Hispania, the Hispanic one appears to be limited to Baetica and North Africa. To improve the knowledge about the origin of materials and the extent of their dispersion, multiple archaeometric studies were already performed including isotope analyses, petrography, cathodoluminescence, and elementary analyses. Yet the results are not always indisputable. Concerning the French quarry, its recent entrance in the scene as a possible source of ancient dolomitic marble, whose analytical characteristics resemble the Greek one, complicates the already complex panorama. Therefore, to this panel of complementary techniques, we propose to add Nuclear Magnetic Resonance (NMR) that permits to decipher the material at a molecular level by looking at different nuclei NMR sensitive: Carbon (^{13}C isotope), Magnesium (^{25}Mg isotope) and Calcium (^{43}Ca isotope).

The idea is to track differences at the molecular scale between the dolomitic varieties to be used as a complementary parameters for their discrimination. In this sense, the present contribution reports the progress made in the quarry samples as well as its application to some decorative architectural materials of the Roman theater of Zaragoza, and also to some sculptural artefacts of the Museum of Málaga.

S4-O15.488

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The marbles of Ascalon – another link in the Severan building projects

Prochaska, W.[1], Anevlavi, V.[2], Cenati, C.[2]

[1]Department of Geosciences and Geophysics, University of Leoben: Peter-Tunner-Straße, 8700 Leoben (Austria)
[2]Austrian Archaeological Institute, Austrian Academy of Sciences: Franz Klein-Gasse 1, 1190 Vienna (Austria)

Keywords: marble provenance, Israel, Severan period

This research presents the results of the investigations on the origin of the white marbles used for the architectural decoration of the Basilica of Ascalon, in Israel. The basilica is located in the civic centre of the Roman centre, and it is one of the relatively few known monumental buildings of Roman Palestine. The architectural elements that are preserved belong to the second phase of the basilica which dates back to the Severan time (beginning of the 3rd century AD).

The marbles used for the bases, the columns, the capitals and the decorated pillars were all imported since ancient Palestine had no sources of marble on its territory. The methods applied were petrographic investigations, chemical and isotopic analyses as well as chemical analyses of inclusion fluids and extractable salts (crush-leach analyses) of the marbles. The results show great import power and transregional trade.

The monumental size of the architectural elements in combination with the absence of a port in the ancient city of Ascalon provides important information for the study of the logistics, the transportation of the material as well as the general economy of Roman Palestine. Similar to the renowned Severan foundations or building programs like Leptis Magna, Perge, Ippos Susita etc. the quarries in the eastern provinces of the empire, such as Prokonnesos and Thasos are the main sources of building material for the construction program of the basilica of Ascalon. Of special interest is the prominent use of different marbles from the island of Lesbos in compliance with most of the Severan projects. In this respect the marble inventory of Ascalon is in good agreement with the well investigated marbles of Leptis Magna.

S4-O16.3003

Statuary qualities of Göktepe marble identified in several Roman case studies

Lapuente Mercadal, P.[1,2], Nogales-Basarrate, T.[3], Carvalho, A.[4]

[1]Petrology and Geochemistry. Earth Sciences Dept. Zaragoza University (UNIZAR), Spain
[2]Catalan Institut of Classical Archaeology (ICAC), Tarragona, Spain
[3]Roman Art National Museum (MNAR), Mérida, Spain
[4]National Museum Archaeology (MNA), Lisbon, Portugal

Keywords: Göktepe marble ; marble provenance ; Roman sculpture ; archaeometry

After its recent discovery, the marble from the Turkish quarries of Göktepe (Muğla province, Western Turkey) has aroused great interest on the part of the scientific community. The richness of the materials exploited in them with respect to the diversity of chromatic tones and their excellent properties for sculpturing, made them essential raw materials to carry out high-quality artistic works.

As part of a research program developed a decade ago concerning the identification of marble on sculptural pieces stored in the imperial Villa Adriana of Tivoli in Italy, different Göktepe qualities were recognized. Since the analytical parameters usually applied in provenance studies (petrography and C and O isotopes) do not properly discriminate certain white Göktepe from Luni - Carrara marble, different methods have been used in Göktepe quarry samples to contribute to their correct identification in archaeological pieces. Among others, they include the use of cathodoluminescence parameters, either qualitative or quantitative, electron paramagnetic resonance, trace elemental composition (Sr and Mn), 87Sr/86Sr isotopes and nuclear magnetic resonance attributes.

This contribution focuses on the role of the application of the most common techniques in combination with other more specific that have further contributed to the identification of the different statuary qualities in both white and black Göktepe. For this purpose, several Roman cases analyzed from Hispania are presented in comparison with those identified in Villa Adriana.

S4-O17.355

Session 4 Technology/provenance - stone/pigments/plaster

POSTER

Smartphone as an analytical tool for siliceous artefacts characterisation

Ramacciotti, M.[1,2], Gallello, G.[1], Columbu, S.[3], Díez Castillo, A.[1], Cervera, M.L.[2]

[1]Department of Prehistory, Archaeology and Ancient History, University of Valencia, Av. Blasco Ibáñez, 28, 46010 Valencia, Spain.
[2]Department of Analytical Chemistry, University of Valencia, Calle Dr. Moliner, 50, 46100 Burjassot, Valencia, Spain.
[3]Department of Chemical and Geological Sciences, University of Cagliari, Cittadella Universitaria di Monserrato, 09042 Monserrato, Italy.

Keywords: chert; quartzite; provenance; raw materials; imaging.

In the last few years, smartphones have become one of the most widespread electronic devices in the world and their use as analytical tools is nowadays a cutting-edge field in chemistry [1]. Nevertheless, the development of methods exploiting the potential of this commonly owned equipment is still an almost unexplored research area in archaeometry.
The present poster offers a first methodological proposal of smartphone use for the study of prehistoric siliceous artefacts tested on the lithic assemblage of La Calvera rock-shelter [2] (Cantabria, Spain). The samples include different kinds of cherts, rock crystal and quartzite unearthed during the excavation of the site, as well as chunks of raw material which outcrops in the same rock-shelter.
Colour features of each sample were characterised by imaging of smartphone pictures and data were compared with those obtained through a Vis-spectrophotometer, employed as a reference method. Furthermore, the samples were analysed by non-destructive techniques such as optical microscopy, X-ray fluorescence, Raman and diffuse reflectance infrared spectroscopy, in order to compare the smartphone imaging capability of discriminating among the different materials.
The obtained results suggest that imaging of smartphone pictures is a reliable approach to characterise colour features of siliceous rocks and that it can be employed as a valuable cheap, fast and non-destructive method as a previous step before carrying out more invasive analyses for provenance studies [3-4].

References

[1] Rezazadeh, M., Seidi, S., Lid, M., Pedersen-Bjergaard, S., & Yamini, Y. (2019). The modern role of smartphones in analytical chemistry. TrAC Trends in Analytical Chemistry, 118, 548-555.
[2] Díez Castillo, A. (1997). Utilización de los recursos en la Marina y Montaña cantábricas: una prehistoria ecológica de los valles del Deva y Nansa. Illunzar, 3, 23-190.
[3] Ramacciotti, M., Gallello, G., Pastor, A., Diez Castillo, A., & García Puchol, O. (2019). Chert nucleus and cortex characterization for archaeological provenance study tested in the Prebaetic system region (Valencian Community, Spain). Lithic Technology, 44(3), 166-180.
[4] Ramacciotti, M., García-Puchol, O., Cortell-Nicolau, A., Gallello, G., Morales-Rubio, A., & Pastor, A. Moving to the land: First archaeometric study of chert procurement at Cueva de la Cocina (Eastern Iberia). Geoarchaeology, in press.

S4-P01.1476

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Building a sanctuary: is a high diversity of stones the reflect of an international renown? The case study of Delphi, Greece

de Vals, M.[1], Moretti, I.[1], Perrier, A.[2]

[1]Sorbonne Université, CNRS-INSU, ISTeP, UMR 7193, 75005 Paris, France
[2]Université d'Orléans, IRAMAT, UMR 7065 Centre Ernest-Babelon, 45100 Orléans, France

Keywords: geology; building materials; quarries; Delphi; Greece

Delphi is one of the most renowned oracular sanctuary in Greece: diverse monuments were built around the Apollo Temple as offerings, dedicated by cities, families or illustrious figures from all the Mediterranean. It is also located in a calcareous mountainous region, at the foot of a large south dipping active fault bordering the Gulf of Corinth, therefore in a very hazardous and difficult setting. It was observed that various limestones were employed in construction and considered that Greek builders travelled with their own stones, but quantitative data were missing. Here we will focus on the building materials and the related historical consequences. What can we learn from the nature of the stones and their uses over time ?

This transdisciplinary project took into account the availability of resources (quarries, water sources), the building stones (nature, origin and volume), the building history (main construction program and planning) but also the global geological context (at the scale of the Gulf of Corinth). We used classical geological investigations: petrological descriptions on site, field works to identify quarries in the Corinthian area, geological mapping around Delphi.

Over 20 different types of stone were employed in the sanctuary, but around 60% of the volume is of local and regional origin. Thus, around 40% of the volume is imported, from the southern part of the Gulf mostly. This is exceptional in comparison to other archaeological sites, where one local stone represents usually more than 95% of the volume. However, earthworks were also extensive as the site developed on a 30° slope, waste materials could have been used to complete the production of defined quarries. It seems that early in the life of the sanctuary, high porosity allochthonous stones were favored: oolitic calcarenite, then sandstones, shelly limestones and travertine. This is attributable to the high density, fracture density and/or heterogeneity of local facies, which make them tough building material.

S4-P02.1483

Archaeometric study of mortars and plasters from Pompeii (Campania-Southern Italy)

Miriello, D.[1], Bloise, A.[1], Crisci, G.M.[1], De Luca, R. [1], De Nigris, B.[2], Martellone, A.[2], Osanna, M.[3], Pace, R.[4], Pecci, A.[5], Ruggieri, N.[6]

[1]Department of Biology, Ecology and Earth Sciences, University of Calabria, Arcavacata di Rende, CS, Italy
[2]Applied Research Laboratory of Archeological Park of Pompeii, Pompeii, NA, Italy
[3]General Director of Archeological Park of Pompeii, Pompeii, NA, Italy
[4]Laboratoire AOrOc Archéologie et Philologie d'Orient et d'Occident, Ecole Normale Supérieure, Paris, France
[5]Equip de Recerca Arqueològica i Arqueomètrica, Universitat de Barcelona (ERAAUB), Departament de Prehistòria i Arqueologia, Barcelona, Spain
[6]Soprintendenza Archeologia, Belle Arti e Paesaggio per le Province di Catanzaro, Cosenza e Crotone, Cosenza, Italy

Keywords: Characterization, production technology; constructive phases; multivariate statistical models

This work is part of the agreement signed in 2015 between the University of Calabria and the Applied Research Laboratory of the Archaeological Park of Pompeii. It concerns the archaeometric study of twenty-six samples coming from the archaeological site of Pompeii (Campania - Southern Italy). The samples were taken from materials with different constructive functions: joint mortars, floor mortars, filling mortars and plasters, belonging to different periods, ranging from the 2nd century BCE to the 18th century AD. Different areas of the archaeological site were sampled, in particular the Insula Occidentalis and the Mercury street of the Regio VI; the West Portico of the Forum, in front of the Basilica, and the Temple of Apollo; the northern terrace behind the Temple of Venus; Via Marina and the Amphitheatre alley.
All the samples were studied using a multi-analytical approach based on Optical Microscopy, X-Ray Powder Diffraction, X-ray Fluorescence, Electron Probe Micro Analysis and Raman Spectroscopy. A detailed image analysis was also carried out using the JMicroVision software to determine the percentages of binder, macropores and aggregate (crushed ceramic fragments - cocciopesto - rock fragments and monocrystals).
The compositional similarities and differences identified allowed to confirm or rebut the archaeological hypothesis on the dating of some samples. Moreover, new data on the production technology of the materials were provided. The study also provided the chemical and minero-petrographic characterization of the samples, which may be useful for preparing compatible repair mortars for future restoration works, creating recipes of mortars with the same composition as the ancient ones, using a mixing system based on optimization software.
In addition, the application of the discriminant analysis on several compositional variables allowed us to define the evolution in time of the materials and to build preliminary multivariate statistical models capable of discriminating mortars with different constructive functions and mortars belonging to different historical periods. Notwithstanding the discriminant analysis was carried out only on a limited number of samples, the results are very encouraging and can be improved by increasing the number of samples from known historical periods, to create statistical models that can be help to identify the various typologies of mortars used in the different historical periods.

S4-P03.103

The use of geochemistry to infer Upper Palaeolithic human mobility in the Pyrenees

Sánchez de la Torre, M.[1], Gratuze, B.[2], Le Bourdonnec, F.X.[3], Sacchi, D.[4], Mangado, X.[5]

[1]SERP-IAUB. Universitat de Barcelona (Spain)
[2]IRAMAT-CEB (UMR 5060). CNRS – Université d'Orléans (France)
[3]Archéosciences Bordeaux (UMR 6034). CNRS – Université Bordeaux Montaigne – Université de Bordeaux (France)
[4]TRACES (UMR 5608). CNRS – Université de Toulouse Jean Jaurès (France)

Keywords: chert; geochemistry; human mobility; Pyrenees; Magdalenian

The use of geochemistry to characterise chert outcrops and to relate chert tools recovered at hunter-gatherer sites with a specific geological formation has increased over the past decade, with examples around the world. Studies of this kind are particularly interesting for inferring the territorial behaviour of past societies and their mobility routes, especially regarding their strategies for acquiring and moving lithic raw materials. The analysis of the territories frequented by Palaeolithic hunter-gatherer groups is particularly interesting in areas with specific environmental and orographic conditions such as mountain regions.

In western Europe, the Pyrenean mountain range is one of these areas where analysing past human mobility is especially challenging, as this mountain chain naturally divides the Iberian Peninsula from the rest of continental Europe and was supposed to be a barrier for Palaeolithic communities. However, recent archaeological and environmental studies have demonstrated that this mountain chain was already frequented by past human groups during almost the last glaciation (c. 120.000 – 11.000 BP).

To infer the mobility strategies followed by human groups that frequented the Pyrenees during the Upper Palaeolithic, lithic artefacts recovered at three Magdalenian Pyrenean sequences have been analysed using geochemical tools. The archaeological materials that have been studied were recovered at the Magdalenian occupations from Parco Cave (Lleida, Spain) and Montlleó open-air site (Lleida, Spain), both in the southern slope of the Pyrenees, and Belvis Cave (Aude, France), in the northern part. The archaeological materials as well as geological samples from different sources have been first analysed macroscopically to define the texture and the micropalaeontological content and then petrographically. In some cases, these approaches do not give conclusive results, as several geological formations possess similar features than the archaeological artefacts. For this reason, geochemical characterisations were undertaken to determine the chemical content. Two main techniques were applied: energy dispersive X-ray fluorescence (ED-XRF) and laser-ablation inductively coupled plasma mass spectrometry (LA-ICP-MS).

Results have shown that geochemistry is a useful tool for establishing differences between geological sources and thus connecting archaeological samples with specific formations. This data has allowed us 1) defining the mobility strategies followed by human groups that settled the Pyrenees during the last glaciation and 2) demonstrating that the mountain chain was not just sporadically crossed by Magdalenian groups.

S4-P04.1490

Monitoring of fresco painting using portable X-ray fluorescence and statistical analysis

Khramchenkova, R.[1], Kaplan, P. [1], Sitdikov, A.[1,2]

[1]*Institute of Archaeology of Tatarstan Academy of Science*
[2]*Kazan Federal University*

Keywords: fresco 16-17 c.; PXRF; plaster; degradation; statistical analysis

The work is devoted to the results of studies of the wall painting of the 16-17th centuries of the Assumption Cathedral of Sviyazhsk Island (Russia) with portable XRF spectrometer Bruker S1-TURBO. A large array of analytical data was collected during the work in-situ. Analytical points of various colors and shades (from 8 to 20 points) were selected on each fresco and were scanned then by the spectrometer. Nineteen elements were identified: calcium, magnesium, silicon, iron, manganese, aluminium, sulfur, potassium, phosphorus, titanium, chromium, nickel, cobalt, copper, zinc, arsenic, lead, tin, antimony. The analysis resulted in a database containing more than 20,000 digital values of element concentration. Analytical data for each color was systematized by the statistical programs “Cluster 30” and “Statistics”.

The fresco base of all the cathedral's paintings is a stucco mortar obtained from dolomite flour. The ratio of concentrations of magnesium (MgO) and calcium (CaO) in the oxide form for the majority of the studied areas is approximately expressed as 2 : 3, which corresponds to the formula of dolomite $CaCO_3 \cdot MgCO_3$. Calcite plaster was detected only in a small part of the fresco paintings. For the most part, these areas correspond to restored regions.

Previous SEM studies have shown degradation processes in stucco. There was a spatial separation of magnesium and calcium in the structure of the fresco base in some areas. Elemental mapping of the studied fragments revealed an interesting feature of the distribution of magnesium, calcium and sulfur: calcium concentration is significantly lower in areas with high sulfur concentration. At the same time, restorers discovered a white fluffy coating on the frescoes, and analysis identified it as magnesium hexahydrite.

Studies by a non-destructive portable X-ray fluorescence method showed high efficiency not only for the determination of coloring materials. Statistical processing of the results on the chemical composition revealed areas with different ratios of magnesium, calcium and sulfur that allowed us to determine the degree of degradation of various sections of fresco painting. The most likely catalyst for degradation processes was sulfur, which is part of egg yolks.

S4-P05.106

Petroarchaeology of the tesserae in the Medusa mosaic of the Rio Verde Roman Villa, Marbella, Malaga (South Spain) as tool for the restoration process

Domínguez-Bella, S.[1], Durante Macías, A.[1], Ramírez Amador, J.L.[1], Moreno Prieto, C.[2], Vila Oblitas, M.[2], Becerra Martín, S.[3]

[1]UGEA-PHAM, Universidad de Cadiz. Puerto Real, Cadiz, Spain.
[2] Menia Restauración & Patrimonio Málaga, Spain.
[3]Grupo de Investigación HUM-440. IES Teba. Málaga, Spain.

Keywords: Roman mosaic; petroarchaeology; stone tesserae; Malaga

The Rio Verde villa is a rustic building of the Roman age, dated between the 1st and 2nd centuries A.D., which was discovered in the 1960s by Carlos Posac Mon and Fernando Alcalá Martín, the official chronicler of Marbella. It is a large maritime villa built by the sea and possibly related to the salting, garum and purple industries in this area of the coast. The excavations have documented a large villa with different rooms around a peristyle surrounded by twelve columns, all of them decorated with musivary pavements, highlighting some unique culinary themes. In one of these rooms, the so-called A, an exceptional mosaic was found with a central motif of a gorgon (Medusa) and framed with plant motifs and ducks, the presence of these birds is related to the proximity to the Rio Verde (green river). Many of the rooms, as well as the halls, are paved with a set of mosaics mainly bichrome. They present a great originality in their motifs, especially those related to Roman cuisine and make it irreplaceable in the western Mediterranean. The villa has been declared BIC (cultural interest site) since 2007.

After the unfortunate vandalism attack on the Roman mosaic of this villa in January 2016, restoration work has been undertaken in the affected areas. Within these actions, the petrological and geochemical characterization of the tesserae affected was carried out, in order to better understand their nature and possible source areas for the construction materials, in case it was necessary to replace any of the lost musivary elements.

During the restoration, we proceeded to take samples of broken tesserae in order to analyze and understand the process of mosaic construction.

The typology of the mosaic, construction technique and raw materials used are reviewed. Different stone tesserae or fragments have been analyzed by optical microscopy, preparing when possible small thin sections. Previously a study of the material by stereomicroscopy was conducted and finally they were analyzed by micro-fluorescence spectroscopy and X ray diffraction. Clear and dark colors tesserae (pale cream and dark grey-brown) present different types of limestone and marble lithologies.

S4-P07.112

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The conservation and preservation of historic plasterwork: study of the decorative pineapples of the Courtyard of the Maidens (the Royal Alcazar of Seville)

Torres-González, M.[1,2], Blasco-López, F.J.[2], Alejandre Sánchez, F.J.[2], Aducin-Ochoa, J.M.[2], Moya-Fierro, M.[3]

[1]Department of Civil Engineering, Architecture and Georesources, CERIS, Instituto Superior Técnico-University of Lisbon, Portugal.
[2]Department of Architectural Construction II, University of Seville, Seville, Spain.
[3]Universidad Austral de Chile – Valdivia, Chile.

Keywords: ancient plasterwork; execution techniques; anomalies; conservation; preservation;

The study of materials and construction systems for the conservation and preservation of architectural heritage is of great relevance in the case of the Royal Alcazar of Seville, declared a World Heritage Site by UNESCO in 1987. This palatial complex stands out for the decorations in its ceramic tiles, wood carpentry and ceilings, artistic ironworks, and plasterworks. The latter are the protagonists of the Courtyard of the Maidens (14th century) of the Palace of the King Peter I, a rectangular courtyard characterized by a perimeter gallery made up of arcades decorated with *sebka* surfaces, a decorative element of Almohad tradition in the form of an oblique grid, with an appearance of rhomboid geometric interlacing made with molded plasterwork pieces, supported by an internal brick structure. The different rhombuses that make up the set are composed by different decorative elements among which the atauriques, the veneras and the pineapples stand out.
At present, the detachment of the pineapples found in the *sebka* surfaces constitutes one of the most relevant risks for the preservation of these decorations, while the integrity of tourists who visit the monument is put at risk. Thus, this work focuses on organoleptic inspection of these decorative elements after carrying out a bibliographic review, a planimetric survey and a study of the environmental conditions of the Courtyard of the Maidens, which contextualizes the current state of conservation and the causes of deterioration. The study developed has allowed (i) to establish the hypothesis of the different fixation systems used over time, identifying the original fixation system and the compatibility with the base plaster mold; (ii) identify anomalies present in decorative pineapples; and (iii) analyze recent emergency actions undertaken in the pineapples to prevent their detachment.
The results obtained represent a novel information of historical, artistic, and constructive interest on these ornamental pieces in plaster, useful for future conservation and restoration interventions.

S4-P08.1509

Volcanic millstones found in the Late Bronze Age settlement of Monte Croce Guardia (Arcevia, Marche Region, Italy): searching for the extra-regional provenance

Santi, P.[1], Cardarelli, A.[2], Bettelli, M.[3], Di Renzoni, A.[3], Renzulli, A.[1]

[1]Dipartimento di Scienze Pure e Applicate, Università degli Studi di Urbino Carlo Bo, Urbino (Italia)
[2]Dipartimento di Scienze dell'Antichità, Università La Sapienza, Roma (Italia)
[3]Consiglio Nazionale delle Ricerche, Istituto di Scienze del Patrimonio Culturale (CNR -ISPC), Roma (Italia)

Keywords: volcanic rocks; millstones; material culture; Italian volcanic provinces; Late Bronze Age.

The Monte Croce Guardia settlement (Arcevia, Marche Region) is one of the most remarkable Late Bronze Age Italian sites. The morphology of the site consists of two hills linked by a col: Monte Guardia (666 m asl) and the plateau of Monte Croce (650 m asl). Being located on these high topographic reliefs, it was naturally defended and overlooked a wide portion of the surrounding territory, from the Adriatic coast to the Central Apennine ridge. The archaeological investigation allowed to understand the complex architecture of dwellings and other structures, along with the reconstruction of large portions of the settlement organization. The numerous archaeological remains can be dated between the 12th and 10th centuries BCE. The presence of specialized metallurgical activities and import goods such as amber and glass beads demonstrate the importance of the village and its connections in the network exchange. Among the imported stone materials there are also volcanic rocks artifacts, most likely fragments of millstones. According to the technological evolution of the grinding devices in antiquity, these fragments (up to 15 centimeters in size) should represent the upper and/or lower portions of saddle querns. They mainly consist of lavas with brownish or pale-dark grey color, low porphyricity and relatively high amount of vesicles (mostly elongated) up to few mm long. Few lithologies with micro-vesciculation are also present.
As the Central Apennine local lithologies are only represented by limestones, marls and sandstones, the volcanic stones, having better grinding capacity in comparison with the local sedimentary rocks of the Umbria-Marche Succession, were brought here as the result of commercial exchanges. In this way, to unravel the Late Bronze trade networks and human interactions, it will be of paramount importance to investigate the provenance of these volcanic stone artifacts through thin section modal mineralogy and petrography, coupled with major-trace elements chemical composition. Petrological footprints of the volcanic artifacts will be compared with the best candidates for the provenance, i.e. lavas of the Veneto Volcanic Province to the north (Lessini Mountains and Marostica- Berici- and Euganean-Hills) and the Roman Volcanic Province to the southwest (Tuscany-Northern Latium to Campania).

S4-P09.1512

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric investigations on Lengyel culture (Late Neolithic-Early Copper Age) polished stone tools from the iconic Lengyel site (SW Hungary)

Biró, K.T.[1], Szakmány, Gy.[2], Illés, L.[3], Kovács, Z.[2], Harsányi, I.[3], Szilágyi, V.[3]

[1]Hungarian National Museum, Budapest, Hungary
[2]Institute of Geography and Earth Sciences, Eötvös Loránd University, Budapest, Hungary
[3]Centre for Energy Research, Budapest, Hungary

Keywords: Lengyel culture, polished stone tools, provenance, petrography, chemical analysis

The iconic archaeological site at Lengyel (SW Hungary), excavated by Mór Wosinsky in 1882-1888, was the eponym site for the Late Neolithic-Early Copper Age Central European cultural unit, the Lengyel culture. According to the excavated material, an intensive handicraft activity (typical for Lengyel culture settlements; Kalicz 1986, Zalai-Gaál 1999) could be proved at the site involving products of chipped and polished stone tool industry. The stone tool finds were studied by archaeological methods in details (Wosinszky 1893, Tolnai-Dobosi 1968, Patay 1976, Bácskay and Biró 1984).

Wosinsky (1893) mentioned more than 150 polished stone tools, however, majority of the finds were lost during the last century. During the revision of the polished stone tool collection of the Hungarian National Museum, an assemblage containing 28 pieces was recovered from Lengyel. According to the record it was a gift from Mór Wosinsky to the Hungarian Geological Institute in 1904 and later transported to the Museum. An interesting aspect of the material that evidence on an earlier petrographic investigation (traces of cutting and thin sections) were found together with the axes, though not a report or publication on this research is known.

As the material represents only a small part of the originally excavated assemblage, the evaluation of the presently accessible finds is preliminary and incomplete. Achaeometric analysis of 21 axes of these polished stone tools was performed in the framework of an ongoing research project aiming to identify the raw materials of the axes and to connect them to source regions. A non-destructive methodology (macroscopic petrography, MS, PGAA, OS SEM-EDS) completed the reevaluation of the old thin sections. Based on our investigations, 12 stone tools proved to be made of local raw materials (alkaline igneous rocks, bituminous limestone) of the Mecsek Mts. In addition, regional (red sandstone and Tertiary basalt from NW Hungary) and long-distance import rock types indicating both northern (contact metabasite from the Northern Czech Massive, serpentinite from an unknown source), southern (‘whitestone’ from the W Balkans) and eastern (hornfels from Rusca Mts.) contacts of the inhabitants were also identified.

Project no. K-131814 has been implemented with the support provided by the Ministry of Innovation and Technology of Hungary from the National Research, Development and Innovation Fund.

S4-P10.1515

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Revealing the 19th centuries restorations of a well-known medieval tapestry ‘the Lady and the Unicorn’, using non-invasive methods.

Claisse, P.[1,3], de la Codre, H.[1], de Chancel-Bardelot, B.[2], Chapoulie, R.[1], Dallel, M.[3], Mounier, A.[1]

[1]Archéosciences Bordeaux (UMR CNRS 6034, UBM), Maison de l’archéologie, Esplanade des Antilles, 33607 PESSAC Cedex – France

[2]Musée de Cluny – Musée national du Moyen Âge, 28 rue du Sommerard, 75005 Paris – France

[3]Laboratoire de Recherche des Monuments Historiques (CRC, UAR 3224, MC-MNHN-CNRS), 29 rue de Paris, 77420 Champs-Sur-Marne – France

Keywords: The Lady and the Unicorn; restorations; natural dyes; hyperspectral imaging; optical microscopy.

The Lady and the Unicorn tapestry, made at the end of the 15th century, was acquired by the Musée de Cluny in 1882. The set consists of 6 tapestries representing an allegory of the five senses and another sixth sense which is still debated. Since their discovery, they have undergone more than a dozen restoration campaigns. These have been more or less documented in written sources. For example, the horizontal band at the bottom of the tapestries was made during the 1889 restoration by Jules André Lavaux, a tapestry weaver who worked both for the Gobelins manufactory and as an independent weaver. For its reweaving, scrap wool, dyed by the workshop of the Gobelins manufactory, would have been used. Nowadays, the fading of dyes results in a very visible restoration that stands out from the rest of the medieval tapestry.

The considerable state of degradation of this restoration has challenged us to study it with non- invasive and contactless methods (colorimetry, optical microscopy, hyperspectral imaging [VIS and SWIR] and fluorimetry). These methods have allowed the characterisation of the materials used during the restoration and have given rise to a discussion on the ethics of restoration in the 19th century by confronting these results with the written sources discovered in the archives. It has also been an opportunity to evaluate the capacity of our methods and data processing in the context of a heavily restored textile work.

The tapestry named "Mon seul désir" was studied during its exhibition at the Musée des Abattoirs in Toulouse (France) in November 2021. The analysis of the main colours (red, blue, yellow and green) has allowed the identification of natural dyes (madder, woad, weld). The entanglement of the wool threads has revealed unexpected restoration techniques. For example, for the red of the background, red and yellow fibres were used. For the blue tint, it was a weaving of blue and white wool thread that was chosen. The impact of this restoration technique can be seen with hyperspectral data on the acquired spectra. This is discussed as a new way for data processing. These preliminary results depend on our knowledge of this medieval masterpiece (e.g. materials used during its production) as well as its enigmatic restoration (revelation of the restoration methods and materials used).

S4-P11.1524

Study of classic Mayan mirrors and reflective artifacts using analytical techniques

Sandoval Molina, A.[1,2], Stern, B.[1], Barrientos Quezada, T.[2], Carías Portillo, T.[2]

[1]Archaeological and Forensic Sciences, University of Bradford, Bradford, West Yorkshire, BD7 1DP, UK.
[2]Centro de Investigaciones Arqueológicas y Antropológicas, Universidad del Valle de Guatemala. Guatemala City 01015, Guatemala.

Keywords: *Pyrite, iron ore, mirrors, La Corona, Cancuen.*

Iron ores and pyrite were used to craft a wide variety of artifacts in Pre-Columbian Mesoamerican cultures (e.g. mirrors, figurine eyes, beads, dental inlays); mirrors being the most studied. In the Mayan region, most of these raw materials have been identified with the naked eye. For that reason, the main objective of this research is to analyze the morphology and to identify the raw materials used to produce reflective objects in the Maya Lowlands zone, through samples from the archaeological sites of La Corona and Cancuen, located in Guatemala, Central America. The analyzed objects were tesserae from mosaic mirrors, beads, and potential raw material nodules. Selection was based on their “good state of preservation” and the abundance reported in each site. The techniques used to identify their composition were Scanning Electron Microscopy with EDS detector (SEM-EDS), X-ray Diffraction (XRD), Raman Spectroscopy and Magnetic Susceptibility. The study also includes the documentation of museum objects from The British Museum and Pitt Rivers Museum collections. The results indicate the presence of diverse materials such as pyrite (iron sulfide), goethite and hematite (iron oxides). This study aims to collect analytical evidence relating to the specific iron ore identification in the Maya region, the relation in between these minerals and some inferences about the shape of the raw materials. In this sense, this will contribute to further studies that aim to understand the usage and significance of these raw materials and their archaeological production in Pre-Columbian Mesoamerica.

S4-P12.1534

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A multi-method archaeometric approach to the provenance study of stone artefacts of Roman septempeda (Marche, Italy)

Taelman, D.[1], Antonelli, F.[2]

[1]Department of History, Archaeology, Arts, Philosophy and Ethics, Vrije Universiteit Brussel, Belgium, devi.taelman@vub.be
[2]LAMA – Laboratory for Analysing Materials of Ancient origin, Università IUAV di Venezia, Italy, fabrizio.antonelli@iuav.it

Keywords: Roman; Central Adriatic Italy; Septempeda; stone provenance; archaeometric analyses

The Late Republican and Early Imperial periods in Central Adriatic Italy were characterised by a growing urbanisation in the form of colony foundations and municipalisation of urban centres, as well as the embellishment of the centres with monumental buildings such as temples, *basilicae*, theatres and amphitheatres. These events resulted in an increased consumption market for goods, such as wine, olive oil, but also lithoid artefacts and high-quality decorative stones and marbles. Since the region is geologically characterised by few stone resources of sufficient quality to merit being moved around even regionally, most decorative and utilitarian stones had to be imported.

This contribution presents the results of a preliminary archaeometric study of the provenance of architectural white marbles (veneer slabs) and a volcanic millstone from the Roman town of *Septempeda* (San Severino Marche) in Central Adriatic Italy. Provenance determination was obtained through macroscopic examination, followed by thin section petrography, X-ray diffraction and stable isotopic analysis ($\delta^{18}O$ and $\delta^{13}C$) for marble samples; optical microscopy and geochemical analyses were performed for studying the volcanic millstone.

Provenance results indicate the presence of several varieties of white marble from the Eastern Mediterranean (Aegean Greece and Asia Minor) and confirm the use of tephri-phonolitic lavas from the Vulsini Volcanic District (Latium, Italy) for manufacturing millstones. The results for *Septempeda* are finally placed in the wider context of the stone trade in Central Adriatic Italy.

S4-P13.1539

The "Casa della Caccia Antica" (VII, 4, 48) mural paintings in Pompei: unveiling constituent materials and techniques details through non-invasive and micro-invasive analysis

Croveri, P.[1,2], Triolo, P.[1], Scarcella, A.[1], Appolonia, L.[1], Cardinali, M.[1], Elia, D.[3], Meirano V.[3]

[1]Centro Conservazione e Restauro "La Venaria Reale", XX Settembre 18 St.,Venaria Reale, Italy
[2]University of Turin, Department of Chemistry, P. Giuria 7 St., Turin (Italy)
[3]University of Turin, Department of Historical studies, Verdi 8 St., Turin (Italy)

Keywords: mural paintings; Pompei; XRF; SEM-EDX; Egiptian Blue; Imaging techniques

A recent research and didactical project carried out by the University of Turin and the Centro Restauro La Venaria Reale allowed to develop interdisciplinary researches aimed to deepen the archaeological, historical, technical and scientific knowledge on the "Casa della Caccia Antica" (House of Old Hunting), Regio VII, 4, 48 in Pompei Archaeological Park. Archaeologists, Chemists, Conservators, Biologists, Physicists, Geologists, Architects contribute to the project's outcomes.
The archaeological research and the scientific analyses performed on site and in laboratory on the original constituent materials, permitted to investigate the sequence of building construction phases of the House and the related executive techniques used for the decorative apparatus.
In fact, the Domus preserve, partially under covering shelters, remains of finest mural paintings. Nevertheless, the exposure to outdoor environmental conditions, old detaching and old restoration interventions affected negatively the decorated surfaces accelerating fading and deterioration processes, in some case hiding the signs of decorative techniques and making more difficult the analyse of the original materials.
Materials and techniques of mural paintings were investigated both with non-invasive and micro-invasive techniques. With multispectral imaging (VIS; UV, IR-FC and VIL, visible induced fluorescence) it was possible to enlighten the presence of pigments or binders also where there were present only as traces and hardly visible by eyes, helping to return a figurative idea of quite completed faded surfaces. Punctual analysis and mapping with XRF gave information on the pigments of the colour palette and the technique used, in some case the decorations are realised "a secco" (dry) with lime colours spread on fresco paintings.
In a second step it was possible to collect some samples and stratigraphic analyses were performed: cross sections observed by optical and electronic microscopy (MO, SEM-EDX) permitted to evidence the chemical and mineralogical characteristics of pigments and mortars, their layering, their granulometry and the technical solutions adopted to obtain the desired aesthetical effect.
All the archaeometric and archaeological data, linked to the collected information on the conservation state and the behaviour of ancient materials, are indeed fundamental to plan and define a suitable conservation intervention to preserve an important element of this unique World Heritage Site

S4-P14.1550

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Interdisciplinary approach to the material analysis of a 17th century banner

Kriznar, A.[1,3], Morón, A.G.[2], Gómez, A.G.[2], Respaldiza, M.A.[1,4]

[1]Centro nacional de Aceleradores – Universidad de Sevilla (CNA-US), Avda. Thomas A. Edison 7, 41092 Seville, Spain
[2]Instituto Andaluz de Patrimonio Histórico, Camino de los Descubrimientos, s/n, 41092 Seville, Spain
[3]Departamento de Escultura y teoría de las Artes Plásticas, Facultad de Bellas Artes de Sevilla, C/Laraña 3, 41003 Seville, Spain
[4]Departamento de Física Atómica, Molecular y Nuclear, Facultad de Física, Universidad de Sevilla, Avda./ Reina Mercedes s/n, 41012 Seville, Spain

Keywords: 17th century; banner; pigments; material analysis

Due to the organic nature of textile fibers applied in elaboration of banners, as well as to their use mostly outside, not many of such pieces are preserved. Therefore, their historic and artistic value is usually very high. In Sanlúcar (Cádiz, Spain), one of these historic banners has survived till today. Dated in the second half of the 17th century, it was used in the most solemn occasions, as religious festivities or the coronation of monarchs. The central shield on the red crimson damask textile represents the weapons of Carlos II on a golden background. Due to its antiquity and use, his conservation state was very deteriorated, which is why it was sent to the Instituto Andaluz de Patrimonio Histórico (IAPH) to be studied and restored. In order to obtain as much information as possible about its conservations state, degradation processes, materials applied and technique used, a team of restorers, conservators, art historians, physicists and chemists precisely studied the banner. The principal aim was to use the obtained data for a proper restoration of the piece.

Before and during its restoration, analyses have been carried out to characterize materials applied and their possible degradation. First, UV fluorescence has been used to observe old interventions. Next, small samples from the painted shield have been carefully extracted and once prepared as cross-sections, studied under optical microscope (OM) and with Scanning electron microscopy with Energy Dispersive X-ray spectroscopy (SEM-EDS). Also the non-destructive X-Ray Fluorescence (XRF) was applied to analyse materials in the areas where no samples could have been taken. The UV images revealed several textile patches added through time in attempt to repair the broken areas of the textile, as well as many retouched areas. SEM-EDX and XRF results identified pigments and the preparation used. The authors applied a gypsum preparation (characterised by Ca peaks), on top of which red bole (Fe) and gold (Au) were applied. The presence of silver (Ag), discovered in few points, is probably only product of the textile contamination by the banner's stick, around which the banner is rolled up. The pigments applied are lead white (Pb), yellow ochre (Fe) lead-tin yellow (Pb, Sn), red ochre (Fe), vermilion (Hg), minium (Pb), blue smalt (Co, Ni, As), azurite (Cu) and umber (Mn, Fe). The presence of Naples yellow (Pb, Sb) on one of the added patches comes from a later intervention.

S4-P15.147

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of historical pigments with SEM-Cathodoluminescence

Palamara, E.[1,2], Das, P.P.[3, 4], Nicolopoulos, S.[4], Tormo Cifuentes, L.[5]. Terlixi, A.[6], Kouloumpi, E.[6], Zacharias, N.[1]

[1]Laboratory of Archaeometry, University of the Peloponnese; Old Camp, 24 133 Kalamata, Greece; el.palamara@gmail.com
[2]Art-e Solutions PC, 18 Sfaktirias str., 24 133 Kalamata, Greece
[3]Electron Crystallography Solutions SL, Madrid, Calle Orense 8, 28020, Madrid
[4]NanoMEGAS SPRL, Rue Èmile Claus 49 bte 9, B-1050, Brussels, Belgium
[5]Museo Nacional de Ciencias Naturales, Calle de José Gutiérrez Abascal, 2, 28006, Madrid, Spain
[6]Laboratory of physicochemical research, Directorate of Conservation of Works of Art, National Gallery – Alexandros Soutzos Museum

Keywords: SEM-Cathodoluminescence; pigments; database; weathering; binding medium

The study of paintings, and in particular the identification of the painting layers and the determination of individual pigments, is a particularly complex process, due to various factors (e.g. complexity of materials used, alteration processes, etc.). The combination of Scanning Electron Microscopy (SEM) with Cathodoluminescence (CL) can serve as a powerful tool for the identification of individual pigments. SEM/CL has the potential of identifying both organic and inorganic pigments and can focus on a micrometer scale. The combination with Energy Dispersive Spectrometry (EDS) allows for robust, cross-checked, elemental and mineralogical characterization of pigments.

In order to apply SEM/CL in a routine-based way for the identification of pigments, it is necessary to have a robust, open-access database of characteristic CL spectra of pigments. A large project has been undertaken to create such a database, focusing primarily at the pigments, both organic and inorganic, which were most commonly used from antiquity until today. In the present project, the CL characterization of common pigments of various colours is presented. In most cases, the CL spectra present characteristic bands, which allow for a secure identification of the pigments.

Additionally, the effect of weathering on the CL spectra was evaluated, by comparison to naturally and artificially aged samples and to pigments identified on areas of two paintings, of the 19th and 20th c., respectively. Finally, the effect of binding media was also studied, using combinations of pigments with four common media: egg yolk, linseed, walnut and poppy oil. Overall, both weathering and binding media cause minor differences in the occurring spectra, without preventing the identification of pigments.

S4-P16.1562

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

"SPOSALIZIO DI S. CATERINA" of Adriaen Isenbrant: Diagnostic Analyses of the Painting with the Aid of Restoration

Zhou, Y.[1], Ridolfi, S.[1], Porzio, F.[2]

[1]Sapienza Università di Roma, Piazzale Aldo Moro 5, 00185 Rome, Italy
[2]Accademia Nazionale di San Luca, Piazza Accademia di S. Luca 77, 00187 Rome, Italy

Keywords: Flemish painting; Diagnostic analyses; Non-invasive techniques; Pigments; Restoration

This study investigates the deterioration, conservation status, previous intervention, executive techniques and pigments materials of the painting “SPOSALIZIO DI S. CATERINA”, attributed to Adriaen Isenbrant back to sixteenth century, on the basis of which, conservation strategies were proposed and implemented. The analysis was carried out based on a multidisciplinary combination of scientific examination and restoration exploration. The scientific examination includes both in-situ non-invasive techniques and laboratory micro-destructive techniques. Non-invasive examination consists of the application of ultraviolet fluorescence (UVF), infrared reflectography (IRR), infrared false color (IRFC) and energy dispersive X-ray fluorescence (ED-XRF), whereas micro-destructive analysis comprises optical microscope (OM). The obtained results revealed that the painting was in a relatively good condition with several times of previous restoration, art historically it was painted with typical Flemish painting techniques and materials in sixteenth century: a panel made of chestnut wood, a preparation layer composed of lead white colored in pale grey and brownish-orange, few of underdrawings and impressive pentimenti, with the palette composed of cinnabar and red ochre for red, verdigris or malachite for green, azurite for blue and yellow ocher for yellow. Moreover, this study provides an evidence that restoration, in particular during the cleaning of the painted surface and panel restoration process, is also acting as a very useful tool for painting investigation, being complementary with scientific examination, therefore it is of necessity to correctly record all these precious data in a timely manner.

S4-P18.163

Interdisciplinary research to African rock art preservation

Wu, Y.-L.[1], Villa, F. [2], Gallinaro, M. [3], Zerboni, A. [1]

[1]Dipartimento di Scienze della Terra "A. Desio", Università degli Studi di Milano, Milan, Italy
[2] Department of Food, Environmental and Nutritional Sciences, Università degli Studi di Milano, Milan, Italy
[3] Dipartimento di Scienze dell'Antichità, Sapienza Università di Roma, Rome, Italy

Keywords: Rock art; Rock weathering; Microscopy; Subarieal biofilm

African rock art is a fragile cultural heritage that requires conservation. This study from southern Ethiopia had recent archaeological investigation confirmed that the rock art galleries of the Yabelo region are of great cultural significance and require further research as well as preservation. Galleries are located on walls of rock shelters and suffer weathering from the natural environment, with reddish and whitish crusts formed on panels. The decay of rock art includes the deterioration of pigments and rock surface that involves complex interactions between minerals and microorganisms.

Preservation of rock art is understudied in the interface of rock surfaces and physical and chemical alteration as well as sub-aerial biofilms (SABs) formed by microorganisms that involve both of the above. The aim of this project is to observe and characterize the mineral rock surface and investigate the surface microbial communities, in hope of understanding more about weathering conditions with an interdisciplinary approach including archaeometry, archaeology and conservation science.

Rock samples from the rock art substrate were attained and analyzed with petrographic microscopy, Scanning Electronic Microscopy (SEM-EDS), and Confocal Laser Scanning Microscope (CLSM). Total genomic DNA of SABs inhabiting rock surfaces were extracted and a high-throughput sequencing of the 16S rRNA gene on the Illumina platform was performed.

Petrographic microscope shows a dark coating growing along the lithic surface and extending into cracks. SEM images reveal a continuous layer of inorganic coating that is 10-20 μm thick. EDS analysis suggests the coating is mainly silicate and clay materials. However, some thicker accumulations are detected with calcium phosphates, whose deposition could be related to biological processes. 3D reconstruction of biofilm images from CLSM show a dynamic biofilm that covers the surface of rock samples, with phototrophic and chemotrophic communities embedded in a continuous layer of self-produced polysaccharide matrix. The SAB communities are characterized by a set of abundant taxa (core microbiome) that performs most ecosystem functions.

Preliminary results reveal the complicated components in a deteriorated rock surface consisting weathering minerals, growing coatings and multifunctional bacterial communities. The interactions between agents of deterioration is a crucial issue to be further pursued for better preservation and management of the rock art.

S4-P19.167

Tracing Palaeolithic human routes through the geochemical characterization of chert tools from Caune de Belvis (Aude, France)

Sánchez de la Torre, M.[1], Sacchi, D.[2], Le Bourdonnec, F.X.[3], Gratuze, B.[4]

[1]SERP. Universitat de Barcelona (Spain)
[2]TRACES (UMR 5608). CNRS – Université Toulouse Jean Jaurès (France)
[3]IRAMAT-CRP2A (UMR 5060). CNRS – Université Bordeaux Montaigne (France)
[4]IRAMAT-CEB (UMR 5060). CNRS – Université d'Orléans (France)

Keywords: chert; ED-XRF, LA-ICP-MS, Magdalenian, Pyrenees

Caune de Belvis (Aude, France) is located in the northern slopes of the Eastern Pyrenees, in SE of France. The site was excavated during the last decades of the past century, identifying several human occupations from the Late Mousterian (Maroto, Sacchi & Ortega, 2015) and the Magdalenian periods (Sacchi, 1992). Archaeological remains are mostly composed of faunal bones and a rich osseous and lithic industry. In this paper we have focused on the analysis of lithic remains recovered in the Magdalenian levels.

The goals of this study have been to determine the territoriality of Magdalenian groups settled at Caune de Belvis and to identify their lithic procurement strategies. To achieve these goals, the recovered lithic remains from the Magdalenian levels as well as geological samples from the potentially used geological formations were analyzed using several analytical techniques. First, macroscopic and petrographic studies were developed to determine the textural, micropalaeontological and mineralogical content. After this, geochemical analyses were conducted to quantify the elemental chemical composition. Energy-dispersive X-ray Fluorescence (ED-XRF) was used to quantify major and minor elements and Laser-ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) was then employed to quantify trace elements.

During the macroscopic and petrographic analysis, it was attested that chert was the most used rock by Magdalenian groups, identifying until five different chert types. The geochemical analysis by ED-XRF and LA-ICP-MS allowed the establishment of differences between geological formations macroscopically identical and the connection between some archaeological chert types and specific geological formations.

Results have showed that different geological formations from the northern and southern Pyrenees were exploited by Magdalenian groups from Caune de Belvis, making evident the existent relation between both Pyrenean slopes during the Upper Palaeolithic and showing a great knowledge of the Pyrenean territory.

S4-P20.169

Neolithic plaster floors at Motza: Earliest case of burning dolomite for plaster?

Maor, Y., Khalaily, H., Vardi, J., Asscher, Y.

Israel Antiquities Authority, Rockefeller Museum, Post Office Box 586, Jerusalem, 91004

Keywords: Plaster; Neolithic; PPNB; Dolomite; Technology

Plaster production was a great technological development of the early Neolithic periods in the southern Levant. Previous research has reported the use of either calcite or gypsum as a binder with additions of local stone or sediment as aggregates. In cases where dolomite was present, it was used in its native form, unfired. Analysis of the Motza plasters showed that both calcite and dolomite were fired for plaster making.

Dolomite requires a lower firing temperature than calcite and a longer hydration period, but if done correctly it can produce plaster that is even stronger and more water resistant than calcitic plaster. In Motza we found evidence of separate burning pits for calcite and dolomite, suggesting the Neolithic population there had found how to identify the two stones and fire each to a suitable temperature.

Motza plasters and potential source materials were analyzed by FTIR, XRD, SEM and thin section microscopy. A new FTIR method was developed to assess the amount of dolomite and the degree of disorder in the dolomite, to show it has been decarbonized and reformed as plaster. In addition, experimental plaster was prepared, showing the segregation of magnesium rich areas like those seen in the Neolithic plaster and suggesting the mechanism of reforming dolomite in ancient dolomitic plasters.

The well-preserved plaster floors at Motza typically feature a preparation layer with a binder that is a mix of calcite and dolomite and a topcoat of pure white calcite. This shows the two plaster technologies were intentionally used for different purposes. There was consistent use of these same practices across the large site and over several occupation layers, all during the Pre-pottery Neolithic B period. The floor structure is similar to the plaster found at other PPNB sites in Israel, but the materials and technology were adapted to the local dolomite rich geology.

S4-P22.3015

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

XRF Ink analysis of selected fragments from the Herculaneum collection

Bonnerot, O.[1,2], Del Mastro, G.[3], Hammerstaedt, J.[4], Mocella, V.[5], Rabin, I.[1,2]

[1]Bundesanstalt für Materialforschung und -prüfung (BAM)
[2]Universität Hamburg - Centre for the Study of Manuscript Cultures
[3]Università della Campania „Luigi Vanvitelli“
[4]Institut für Altertumskunde - Universität zu Köln
[5]Consiglio Nazionale delle Ricerche (CNR)

Keywords: XRF; ink; Herculaneum; papyrus

Hundreds of papyrus rolls, carbonized during the 79CE eruption of Mount Vesuvius, were discovered in 1754 at Herculaneum. Sophisticated mechanical methods for unrolling the best-preserved scrolls have been applied, with varying success. However, such processes have been abandoned, to prevent risk from irremediable damage or loss and to preserve the integrity of the extremely fragile rolls. Following the development of X-ray based non-invasive techniques, attempts to virtually unroll the scrolls were made. The most common ink in Antiquity was carbon-based, and the main element of carbonized papyrus is carbon, making these investigations difficult. However, some attempts with synchrotron X-ray phase-contrast tomography (XPCT) were successful. Recently, the identification of antique inks containing metals raised hope that if some of the inks contain metal the rolls can be virtually unrolled using conventional CT- technique. We investigated the inks of a selection of partially unrolled fragments stored at the Biblioteca Nazionale di Napoli with X-ray fluorescence in order to select the best candidates for tomography. Despite the many difficulties (analysis of several layers sticking together, letters barely visible, difficulty to separate contribution from the ink and from the papyrus, inhomogeneity of the support, fragility of the fragments…), encouraging results were found, with a number of inks from Greek fragments found to contain additions to the soot (Fe, Pb, Cu P).

S4-P23.194

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The use of mortars in residential dacian buildings. A first

Ferencz, I.V.[1], Pușcaș, C.M.[2], Stremțan, C.C.[3], Tămaș, T.[4], Căsălean, A.[1], Roman, C.C.[5]

[1] Museum of Dacian and Roman Civilization, Deva, Romania
[2]Terra Analitic SRL, Alba Iulia, Romania
[3]Teledyne Photon Machines, Bozeman, MT, USA
[4]Babeș-Bolyai University, Dept. of Geology, Cluj-Napoca, Romania
[5]Independent Researcher, Hunedoara, Romania

Keywords: mortar; Dacia; XRD; petrography

In Dacia and especially in Southern/Western Transylvania a new type of settlements develops around the middle of the 1st century BC – the so-called "Dacian fortresses". Most of their characteristic traits reveal their social, economic, and symbolic functions, rather than the military ones. Present especially in the intra-Carpathian area (with some examples on the Eastern and Southern slopes of the Carpathians), they are set on heights and surrounded by an agricultural hinterland. The entire organization, vertical as well as horizontal, illustrates the fact that this type of habitat was created by a strongly hierarchized social structure and that it represents fortified aristocratic residences. The so-called *murus Dacicus* can be described as a special technique consisting of revetments made from sizeable stone blocks, sparsely connected by wooden beams, and the spaces in-between packed with rock and soil. In most cases, the technique is an adaptation of the one used in the Hellenistic world and the style was borrowed from the Mediterranean region, through master builders, probably of Greek origin.

In 2016, during a campaign on a Dacian "tower-house" type structure identified earlier on the Cetățuie Hill in Ardeu, Romania, an atypical material was noticed on the lower surface of a stone block sitting perpendicular to the revetment. Its appearance, of different colour and texture compared to the stone block it was attached to, led us to immediately assume it could be mortar. Subsequent campaigns on the site revealed several other examples of the same material.

Powder X-ray diffraction and petrographic investigations were used to definitively establish that the sample represents a synthetic compound, intended for use as a binder for building materials. Sand from the nearby river was also collected to test its mineralogy against that of the mortar samples and establish the source area. It is imperative to understand the use of mortar – as well as sourcing of the materials – in such constructions, especially since Dacians were not previously known to implement mortar in their residential buildings. Our aim is to raise awareness on this discovery, so that other sites will undergo similar scrutiny and the extent of mortar use by Dacian builders can be better constrained.

S4-P24.196

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The meroitic pottery from Napata (Karima, Sudan): preliminary results of the archaeometric study

Ursache, G.[1], Tesser, E.[2]*, Antonelli, F.[2], Ciampini, E.[1], Iannarilli, F.[1], Piovesan, R.[2], Maritan, L.[3]

[1]Università Ca' Foscari di Venezia., Dipartimento di Studi Umanistici. Palazzo Malcanton Marcorà, Dorsoduro 3484/D, I 30123 – Venezia
[2]LAMA - Laboratorio di Analisi dei Materiali Antichi, Università Iuav di Venezia. Palazzo Badoer, 30125 - Venezia
[3] Università di Padova, Dipartimento di Geoscienze. Via G. Gradenigo 6, 35131 Padova

Keywords: Sudan; Meroitic pottery; Archaeometric study; Petrography.

The ancient city of Napata (present Jebel Barkal, central Sudan) and its royal district were for centuries the hub for political and economic affairs of all the Nubian territory. During its history, the city played also the role of the Nubian capital and economic centre of the kingdom, attracting people and goods from Upper and Lower Egypt, even when the political capital changed to Meroe. The pottery, representing an important expression of the artistic and manufacturing workshops of Napata, is characterised by a unique style, since mixing African traditions with Hellenistic and Egyptian features. This ceramic attracted the interest of scholars only at the beginning of the last century when the complexity of its production was really evaluated and appreciated in detail. Forms and the main decorative motives of the Meroitic pottery produced between the 3rd century BC and the 3rd century AD have been studied and quite well outlined. However, one of the major issues about this manufacturing remains the identification of the production centres over the country. To answer this key question, in the last decades several archaeometric studies focused on this topic by considering possible production centres in the whole region, comparing local raw materials and composition of the production recipes. However, there are no archaeometric data currently available on the Meroitic pottery from the residential area of Napata investigated by the Italian Archaeological Mission. This research aims to cover this gap through the analysis of a set of samples, selected on a macroscopic base, which represent the ceramic repertoire from the palace of Natakamani (1st c. CE): storage and transport ware as well as painted, incised, impressed table ware (cups, bowls, goblets). The mineralogical-petrographic features and the firing temperature (i.e. information about the production technology) of the potsherds were studied by optical microscopy and X-ray powder diffraction analysis, and several ceramic pastes where distinguished. A comparison with the *in situ* raw materials – especially sandy deposits found in stratigraphic units coeval with respect to the archaeological pottery – indicated that the sand-sized inclusions of the pottery are generally consistent with the clastic deposit or, in one case, with the granitoids rocks outcropping at the nearby fourth cataract; this proved a local-regional production.

S4-P25.3018

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Pigments mineralogy, chemistry and stratigraphy of wall paintings from 16th century church in middle west Poland

Węcławska M.

Adam Mickiewicz University in Poznań

Keywords: pigments; thin section; wall paintings

During conservation work, in small church in middle west Poland (village Włościejewki), fragments of the original 16th century polychromies were exposed. This allowed to observe painting layers from different time periods: 16th, 16/17th and 19th century. This study is concentrating at wall paintings from the 16th century and also yunger painting layers.

The aim of this work is to recognize the mineralogy and chemistry of pigments used in paintings. An indirect goal is comprehensive analysis structure (plaster, whitewash) and stratigraphy of wall paintings in terms of the correlation of the time of their creation. In the years 2005-2010, the walls of the church were subjected to comprehensive restoration treatments, until now the only so-advanced. Stratigraphy of the painting layers showed that since 16th century they were repeatedly repainted in various techniques. There was no documentation regarding the original painting layers and the paintings have not been previously described for research conservation, nor subjected to physicochemical research.

Observation of the pigment layer and samples cross sections was carried out under the petrographic microscope. Documentation photos were taken in reflected and passing light. The chemical composition was analysed with scanning electron microscope, equipped with an EDS detector.

Obtained results show that in 16th century painting predominated pigments do not differ and are based on iron and iron oxides (red ocher, yellow ocher). The presence of delicately colored whitewash is common, carbon black is present. Samples dated as younger ones give ambiguous results, but iron pigments are also present.

S4-P26.213

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Technology and analytical characteristics of Roman floor mosaic substrates

Pachta, V.[1], Papayianni, I.[2]

[1]Dr. Architect, Conservator, Lab. of Building Materials, School of Civil Engineering, Aristotle University of Thessaloniki, PO Box:482, 54124, Thessaloniki, Greece
[2] Em. Professor, Lab. of Building Materials, School of Civil Engineering, Aristotle University of Thessaloniki, PO Box:482, 54124, Thessaloniki, Greece

Keywords: floor mosaics; substrate; mortar layers; properties; lime.

Mosaics, as a decoration mean of historic structures, firstly appeared in the 2nd millennium BC. The typology and structure of floor mosaics was formed during Classic Antiquity, concerning both the tesselation layer and their substrate, that was further exploited in Hellenistic times. In the Roman period, floor mosaics were systemized and wide spread around the Empire, decorating public buildings, private houses and open spaces (i.e. atriums). In all cases, their substrate had a key role for ensuring the stability and resistance to weathering and ageing. The mosaic substrate consisted of successive layers with specific characteristics for each of them. In the paper, four substrate samples from eminent Roman floor mosaics were analyzed, located in the Archaeological site of Dion and the Galerius Palace of Thessaloniki, Greece. The study concerns the classification of the layers through on site and macroscopic observation, microstructure examination of each layer, as well as determination of their physico-mechanical and chemical properties. The evaluation of the analysis results led to the identification of the characteristics of each layer, as well as of the substrate as a whole. Various aspects were assessed, including layers' type and thickness, binding system, origin and gradation of aggregates, Binder/Aggregate ratio, porosity and apparent specific gravity, compressive strength. According to the results, the substrates presented similar characteristics, including a stratigraphy of 3-4 very well compacted layers, supported in a substructure of pebbles (statumen), with a decreased thickness towards the surface. Mortars were based on lime, mixed with pozzolan, brick dust, natural and ceramic aggregates. The aggregates size and proportion was decreased in the upper layers, as well as porosity and mechanical strength. The adhesion of the layers was high in most cases, attributed to their good compaction and other technological aspects. Generally, the substrate of the mosaics was carefully assessed, following specific requirements in order to be durable and effective. This could be asserted by their preservation state that is usually good up to nowadays, although they have been subjected to sedimentation or ageing and constant deteriorating factors.

S4-P27.3019

Multidisciplinary techniques in the stratigraphic study of facade coatings and paints of architectural heritage

Robador González, M.D.[1], Arrighetti, A.[2], Magrini, D.[3], Fratini, F.[3]

[1]Universidad de Sevilla
[2]École Normale Supérieure - Université PSL
[3]CNR ISPC Institute of Heritage Sciences

Keywords: Coating stratigraphy; Paints; Colors; Multidisciplinary techniques; Architectural heritage

The church of San Bartolomé in Seville, of the late 18th century, is one of the most beautiful and characteristic temples of its time. The objective of the research was to carry out the architectural, scientific and archaeological study of the stratigraphy coatings deteriorated to obtain data for the restoration process.
The methodology used for interpreting the evolution of the plasters and paintings of the church took a stratigraphic approach, adopting the archaeology of architecture method, which allows the building to be broken down into Stratigraphic Units.
Micro-samples have been observed and documented under the microscope, and subsequently, cross-sections were obtained of the stratigraphy. ATR-FTIR analyses have been performed by a portable infrared spectrophotometer, equipped with a Globar IR source and the attenuated total reflection module with a diamond crystal. The infrared spectra have been acquired in the spectral range 4000–400 cm− 1 with a spectral resolution of 4 cm− 1 and 128 interferograms. FORS measurements were performed on the external layer of samples by an Ocean Optic spectrophotometer equipped with optical fibres and a tungsten lamp as the light source. A measurement head with illumination at 0° and signal collection at 45° allowed the acquisition of reflectance spectra from an area of approximately 2 mm^2.
The characterization of the mortars have been carried out through the determination of the principal mineralogical composition was performed through a PANalytical diffractometer X'PertPRO with radiation CuKα1 = 1,545 Å, operating at 40 KV, 30 mA, investigated range 2θ 3-70˚, equipped with X'Celerator multirevelator and High Score data acquisition and interpretation software. Optical microscopy in transmitted light was performed on thin sections with a polarised light microscope equipped with a camera and dedicated image analysis software.
It can be concluded that the stratigraphic sequences are complex and made up of even more than 20 layers. Combining the data that emerged from the archaeological reading of the walls with that of the specific reading of the plasters it emerges that the building began to be plastered following all the transformations visible from the stratigraphic reading of the walls. The analysis of the red paints points out the presence of iron-based pigments, highlighting the spectral bands at ca. 410, 498 and 612 cm-1 imputable to hematite pigment. On the yellowish paint fragments, iron hydroxides have been also identified. The mineralogical-petrographic investigation of the mortars constituting the plasters made it possible to recognize four different mixtures.

Acknowledgements: to the PID 2020-115786 GB-100 project, financed by MCIN/AEI/10.13039/501100011033

S4-P28.3020

Multi-technique approach on Middle and Upper Palaeolithic cherts from the Central Region of Mediterranean Iberia: preliminary results

Eixea, A.[1], Roldán, C.[2], Villaverde, V.[1], Dias, M. I.[3], Prudêncio, M.I.[3], Marques, R.[3], Russo, D.[3], Gméling, K.[4], Cavallo, G.[5], Murcia, S.[2]

[1]Departamento de Prehistoria, Arqueología e Historia Antigua. Universidad de Valencia. Spain.
[2]Instituto de Ciencia de Materiales (ICMUV). Universidad de Valencia. Spain.
[3]Centro de Ciências e Tecnologias Nucleares – C2TN. Instituto Superior Técnico, Univ. Lisboa. Portugal.
[4]Centre for Energy Research, Nuclear Analysis and Radiography Department. Budapest, Hungary.
[5]SUPSI. Istituto Materiali e Costruzioni. Switzerland

Keywords: Middle and Upper Palaeolithic; archaeological cherts; INAA; PLM-SEM-EDX

Forty chert fragments (flakes and chips resulting from stone-tool production) from seven Middle Palaeolithic (Cova Negra, Las Fuentes, Petxina, Pla de Palau, La Llacuna, Les Majones and La Llacuna-Majones) and two Upper Palaeolithic archaeological sites (Cova de les Malladetes and Cova de les Cendres), and cherts from geological outcrops located in the central area of the Mediterranean Iberian Peninsula were selected for macroscopic, petrographic, mineralogical and geochemical characterization.

Texture, grain size, impurities, cortex features, knapping aptitude and colour was carried out with a binocular microscope under 20x-40x magnification and using the Munsell Rock Colour Chart. Furthermore, the samples were analysed by polarising light microscopy (PLM) on polished thin sections and scanning electron microscopy with microanalysis (SEM-EDX). The chemical contents of twenty-seven elements (Na, K, Fe, Sc, Cr, Co, Zn, Ga, As, Br, Rb, Zr, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Yb, Lu, Hf, Ta, Th and U) were obtained on powdered aliquots from the core of the flint samples by instrumental neutron activation analysis (INAA). Multivariate statistical analysis by means of hierarchical cluster analysis (HCA) and principal components analysis (PCA) was applied to the chemical and macroscopic variables with the aim of identify clusters among the chert samples (archaeological artefacts and geological outcrops).

Geochemical data of the analysed samples were statically grouped into three clusters that show similarities between cherts found in different sites suggesting their provenance from local outcrops. However, these cherts cannot yet be assigned to specific quarries. The predominant macroscopic features are fine grain and texture, grey and orange colours, opaque transparency and good knappability. Thin sections have provided information on its elemental composition and its petrographic features indicated that samples mainly consist of homogeneous cherts exhibiting mudstone and wackestone microfacies. In addition, the microfauna detected indicates that the original carbonate rock formed in pelagic facies.

This work provides compelling source to artefacts correlations and will generate new information to analyse the strategies of raw materials catchment and management activities in the archaeological sites of this area. With this, identify the mobility patterns and the landscape uses by the Valencian Middle and Upper Palaeolithic populations.

S4-P29.3020

Egyptian blue pellets from the 1st c. BCE workshop of Kos (Greece): micro-analytical investigation by means of optical microscopy, SEM-EDS and micro-Raman spectroscopy

Kostomitsopoulou Marketou, A.[1], Andriulo, F.[2], Steindal, C.[2], Handberg, S.[3]

[1]MF-Norwegian School of Theology, Religion, and Society Gydas vei 4, 0302, Oslo, Norway
[2]Saving Oseberg Laboratory, Museum of Cultural History, University of Oslo, St.Olavs plass, Oslo, Norway
[3]Department of Archaeology, Conservation and History, University of Oslo, Blindernveien 11, Oslo, Norway

Keywords: Egyptian blue; pigment production; technology; micro-Raman; SEM-EDS

Egyptian blue is an artificial material, widely used as a pigment across the ancient Mediterranean world. However, archaeological evidence for its production is scarce. This paper aims to deepen our understanding of Egyptian blue production through the micro-analytical examination of material from the 1st century BCE pigment and metallurgical workshop of the island of Kos.

Egyptian blue is a multi-component material, with its blue colour attributed to the crystalline phase a copper-calcium tetrasilicate (cuprorivaite, $CaCuSi_4O_{10}$). Beyond cuprorivaite, the study of unreacted materials, high-temperature products and impurities, present in the final product can illustrate aspects of the production process and technological choices.

Eighteen Egyptian blue samples from the workshop are examined by means of optical microscopy and scanning electron microscopy (SEM-EDS), for the micro-morphological and elemental characterisation of the finds. Image analysis of the backscattered electron micrographs allows for the quantification of the various phases present in the material. Furthermore, the molecular characterisation of the various components is carried out through micro-Raman spectroscopy.

The obtained results show that the hue of the final product largely depends on the size of the cuprorivaite agglomerates. Moreover, firing conditions can be illustrated by the size variation of the cuprorivaite crystals, while the size and shape of the quartz particles can be used as a proxy for addressing further mechanical processing (grinding) of the pigment. Finally, the presence of metal impurities sheds light on the choice of raw materials, stressing the relationship between pigment production and metallurgy, both carried out at the same site.

S4-P30.256

Raman Micro-Spectroscopy and XRD applied to mineralogical characterization of local and regional Flint source-areas from Chalcolithic settlement of Zambujal (Torres Vedras, Portugal)

Jordão, P.[1,2,3], Guedes, A.[4], Pimentel, N.[2,3]

[1]*Faculdade de Ciências, ULisboa*
[2]*Instituto Dom Luiz (IDL), Faculdade de Ciências, ULisboa*
[3]*PhD student, Fundação para a Ciência e Tecnologia (FCT)*
[4] *ICT- Pólo Porto e DGAOT, Faculdade de Ciências, UPorto*

Keywords: Raman micro-spectroscopy; XRD; flint; source-areas; Zambujal

Flint provenance studies are important to know where these raw materials were available, but also and mainly to know where they were actually collected. The distance between primary and secondary host flint formations may be up to several tenths or hundredths of kilometres and therefore research on the distinction between primary and secondary sources is crucial.
When the artefacts do not have a weathering ring or cortex allowing to distinguish and characterize eroded flint, it is many times an unsolvable problem to choose between a primary and a secondary source for flint supply. This is usually the case in Neolithic/Chalcolithic specialized artefacts, and this study aims to contribute to the identification of eroded and non-eroded flint by testing, with different approaches and technologies, samples from the region of the Chalcolithic enclosure of Zambujal (Torres Vedras).
It is known that the elementary composition of flint is almost exclusively SiO_2 – α-Quartz – so we attempted to work on this aspect, measuring quartz crystallinity and quantifying its metastable phase (Moganite), as well as the presence of other minerals. Unlike XRD, Raman micro-spectroscopy technique has been used both in geological and archaeological samples, in polished thin-sections and in surfaces without preliminary preparation, from the same sample, in order to validate this non-destructive technique in future analysis.
Despite the small variations in Quartz crystallinity/Moganite proportion, it was noted that samples from Lisboa and Sintra Cenomanian flint have minor Moganite content than samples from Torres Vedras Cenomanian. The differences in Moganite between Cenomanian and Paleogene flint were most impressive in secondary Cenomanian samples. This conclusion confirms the reverse trend demonstrated by the Quartz Crystallinity Index higher in secondary than in primary Cenomanian samples, as presented in a previous study (Jordão *et al*., accept).
The Raman micro-spectroscopy has been also applied to some archaeological samples, previously correlated with the geological ones, and the results were mostly in accordance.
As a conclusion, it may be stated that a multi-analytical approach in the petro-archaeological characterization protocol of flint, must include Raman micro-spectroscopy, not only to complement petrographic results, but also to provide meaningful mineralogical information to help the differentiation of groups of raw materials from a specific provenance.

Acknowledgments: FCT- UID/GEO/50019/2019
Jordão, P., Guedes, A., Pimentel, N. (2022) - Raman Microspectroscopy applied to flint provenance at the Chalcolithic settlement of Zambujal (Torres Vedras, Portugal). *Archaeometry*. (Accep.)

S4-P36.319

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Deconstructing a Reconstruction. Marble analyses of the Domitian's facade in Ephesos

Anevlavi, V.[1], Ladstätter, S.[1], Prochaska, W.[2]

[1]Austrian Archaeological Institute – Austrian Academy of Sciences, Vienna, Austria
[2]Department of Geosciences and Geophysics, University of Leoben, Austria

Keywords: Marble provenance, Ephesos, Temple of Domitian

This research presents the results of the investigations on the origin and on the types of marbles used in the architecture of the façade of the so-called Domitian Temple in Ephesos, Asia Minor. The temple is located at the western end of the Upper Agora and was dedicated to the Flavian dynasty and erected under the reign of Domitian (89/90 AD). The façade is composed of three zones and decorated with reliefs, depicting barbarians and allegories. Shortly after the excavation in the 1960ies a partial re-erection (anastylosis) took place by using elements, which were attributed to the building without beforehand scientific proof. Recent archaeological investigations give rise to doubt the proposed composition.

One of the main questions referring to this construction program is if the building material was extracted from one main marble source or if other marble sources and quarries were used for that building. Especially high prestigious ventures like imperial projects were built from one hand and usually no spolia were used. There are many examples for this practice in Ephesos in the 1st and 2nd century. In the course of the current project practically all types of decorated architecture of the façade were investigated and samples were taken from decorated pillars, architraves, friezes, orthostates, doors, staircases, foundation blocks etc.

The methods applied the characterization of the different types of marbles and to pinpoint their origin were petrographic investigations, chemical and isotopic analyses and chemical analyses of inclusion fluids and extractable salts (crush-leach analyses) of the marbles. The results showed that different types of marbles can be detected in the present anastylosis. Most frequently Ephesos I marble was used, but there also occur, in a not systematic assemblage, Ephesos II and prokonnesian marbles. The marble analyses therefor reinforce the reasonable doubts on the correctness of the reconstruction.

S4-P37.323

Mineralogical interpretation of multispectral images in 2D and 3D: characterizing pigments of wall paintings in the Eastern Mediterranean

Asscher, Y.[1], Gasanova, S.[2], Abate, D.[2], Hermon, S.[2], Hallot, P.[3], Mathys, A.[3,4], Deiana, R.[5]

[1]*Israel Antiquities Authority, Israel*
[2]*The Science and Technology in Archaeology and Culture Research Center (STARC), The Cyprus Institute*
[3]*University of Liège, Belgium*
[4]*Royal Museum for Central Africa, Royal Belgian Institute of Natural Sciences*
[5]*Department of Cultural Heritage, University of Padova, Italy*

Keywords: Pigments; Frescoes; Multispectral imaging; Photogrammetry; XRF

Mural paintings are invaluable archives of materials, techniques, and artistic expressions of past civilizations, and the Roman art from mid-first century AD is an excellent example of such archives AD. Assessing wall paintings' state of preservation requires characterizing pigments on a large scale, to document alteration processes and determine conservation interventions, as some wall paintings degrade rapidly due to salt deposits and poor management. Here, we present a new methodology for large scale non-invasive characterization of murals that combines multispectral imaging with X ray fluorescence spectroscopy (XRF). We report the results of pigment analysis on the wall paintings from the Sarno Baths in Pompeii, in comparison with Roman Villas in the Eastern Mediterranean, including Paphos in Cyprus and Caesarea in Israel. The multispectral images are based on analyzing stacked photos that were registered through ultraviolet, visible, and infrared band-pass filters. The images are normalized to represent a relative reflected intensity at different band widths, and based on pigment-specific band ratios, we construct pigment maps in 2D. In addition, we also discuss a new method for analyzing multispectral 3D models that were taken using the different band-passes. The finds show the presence of iron in red pigments, suggesting the use of hematite or red ochre, lead that is associated with minium and green earth. Results confirm that the mural paintings at different sites were sharing similar pigments, that were found in Pompeii in 1st century AD.

S4-P38.346

Towards the provenance determination of lapis lazuli rocks processed at the Bronze Age archaeological site of Shahr-i-Sokhta (3rd millennium BCE)

Re, A.[1,2], Guidorzi, L.[1,2], Lo Giudice, A.[1,2], Borghi, A.[3], Es Sebar, L.[4,2], Frenez, D.[5], Vidale, M.[6]

[1]Dipartimento di Fisica, Università degli Studi di Torino, Via Pietro Giuria 1, 10125 Torino (Italy)
[2]INFN - Sezione di Torino, Via Pietro Giuria 1, 10125 Torino (Italy)
[3]Dipartimento di Scienze della Terra, Università degli Studi di Torino, Via Valperga Caluso 35, 10125 Torino (Italy)
[4]Dipartimento di Elettronica e Telecomunicazioni, Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129 Torino, (Italy)
[5]Dipartimento di Storia Culture Civiltà, Università di Bologna, Via San Vitale 28/30, 48121 Ravenna (Italy)
[6]Dipartimento dei Beni Culturali: Archeologia, Storia dell'Arte, del Cinema e della Musica – Università degli Studi di Padova, Piazza Capitaniato 7, 35139 Padova

Keywords: lapis lazuli; provenance; PIXE; Ionoluminescence; Shahr-i-Sokhta; Bronze Age

Shahr-i-Sokhta lapis lazuli working areas were discovered and investigated by archaeologists in the early 1970s. The site, located in eastern Iran and dated back to the 3rd millennium BCE, was a consumer of high quality lapis lazuli beads, and, in part, a hub for the trade routes of this semi-precious blue stone. Caravans linked the city and other towns on the Iranian Plateau to a widespread network of long-distance exchanges towards the western markets. A large amount of production waste fragments or partially carved rocks was retrieved during the archaeological excavation. Up to now, the examinations have been focused on the study of the manufacturing techniques [1].
The application of Ion Beam Analyses (IBA) can add more information on the provenance of the raw lapis lazuli that were carved at Shahr-i-Sokhta. In particular, the amount of trace elements present in diopside and pyrite, two of the main mineralogical phases contained in lapis lazuli rocks, can help in the discrimination between several known quarries [2]. Particle Induced X-rays Emission (PIXE) is employed for the elemental analysis and Ionoluminescence (IL) signal is simultaneously collected, providing spectra with characteristic shapes that can be used as well as provenance markers. The small dimensions of target minerals require a probe of micrometrical size: for this reason, all the analyses have been carried out at the microbeam line of the AN2000 accelerator at the INFN–LNL facility, using accelerated protons with an energy of 2 MeV.
For a first screening, we were able to analyse a total of 6 small samples: these were part of the manufacturing waste so it was possible to cut them to expose the core, untouched by weathering agents. The inner surfaces were characterized by optical microscopy, SEM-EDX and cold-cathodoluminescence. Moreover, preliminary IBA measurements were carried out on 13 diopside and 9 pyrite crystals. Results will be presented and discussed to attempt an attribution for the provenance of the raw material.

[1] Vidale, M. and Lazzari, A. *Lapis lazuli bead making at Shahr-i-Sokhta,* Antilia, Treviso, 2016
[2] Lo Giudice A *et. al.*, *Protocol for lapis lazuli provenance determination: evidence for an Afghan origin of the stones used for ancient carved artefacts kept at the Egyptian Museum of Florence (Italy)*, Archaeological and Anthropological Science 9 (2017) 637-651

S4-P39.352

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Hematite procurement and use in prehistoric Missouri (USA): A compositional analysis

Pierce, D.E.[1], Wright, P.J.[2], Popelka-Filcoff, R.S.[3]

[1]Université Bordeaux Montaigne
[2]University of Missouri- St. Louis
[3]Flinders University

Keywords:

Hematite has long been an important mineral for the production of a variety of items including its use as a pigment, a polishing tool, a medicine, and as a lithic tool. Worldwide, this Fe-oxide has been used in the form of ochre for hundreds of thousands of years. Though its use in prehistoric North America was commonplace, analyses of archaeological assemblages typically have focused upon ceramics and stone tools. Given the ubiquity and importance of hematite as a utilitarian as well as symbolically potent raw material, understanding its procurement and distribution can provide key insight into prehistoric lifeways. As the first geochemical study of archaeological hematite in the area, we recently analyzed a collection of 38 hematite artifacts (including plummets, preforms, and fragmented cobbles, from East Central Missouri, USA from mostly Late Archaic (3000 – 1000 BCE) and Middle Woodland (150 BCE – 400CE) contexts. The lack of available reference data precluded a full understanding of provenance. Yet, this study provided important information and direction for further research. Our results confirmed the ability to differentiate hematite sources based upon chemistry while establishing the most effective technique to control for the varying effect of iron in the compositional fingerprint. Through various multivariate statistical analyses, the archaeological specimens remained distinct from any available source data used as comparative reference. Nonetheless, results revealed at least two separate sources of hematite exploitation, yet the location of these sources remain unknown. As such, it is now necessary to expand our study to include a diversity of samples from other locations which may indicate trade and/or resource procurement. In our current follow-up study, we have augmented that initial assemblage with an additional 61 similarly dated artifacts from locations of suspected procurement and/or production. Twenty-two of these specimens were collected from the Verkamp Shelter in the Meramec River Valley (Missouri), a location of abundant Fe-ore deposits. The remaining sample includes small collections from two sites in Missouri, which are suspected to be the original source of hematite artifacts, and three sites from nearby Illinois. Here we use neutron activation analysis (NAA) to characterize the sample composition, and statistical analysis to compare to previously considered samples. Due to the paucity of hematite data for central United States, this study serves as a valuable contribution to our understanding of the distribution and use of this significantly understudied material, while providing a baseline for future studies in the region.

S4-P42.322

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Pietra ollare manufacts from the Medieval site of Nonantola (Modena, northern Italy): composition, provenance and use

Riccardi, M.P.[1], Santi, P.[2], Renzulli A. [2], Del Moro S.[3]

[1]*University of Pavia, Italy*
[2]*University of Urbino, Italy*
[3]*Spin Off Geo.In.Tech. srl, Italy*

Keywords: *pietra ollare*; manufacts; crucibles; glass; Middle Age

The archaeological excavations carried out at the medieval Abbey of San Silvestro (752 A.D.), in Nonantola (Northern Italy), gave back numerous fragments of artefacts produced with the so-called pietra ollare i.e. basic and ultrabasic metamorphic rocks belonging to the greenschist facies (e.g. soapstones). The investigated pietra ollare samples come from fragments of (i) containers probably used for domestic cooking (with signs of burning due to use on fire) and (ii) crucibles showing in the inner walls very thin layers of green transparent glass and therefore emphasizing a pyrotecnological use (glass production). Different temperatures attained by the manufacts during their use, will be tentatively defined on the basis of textural and mineralogical transformations suffered by the pietra ollare.

Mineralogical and petrographic study in thin section (Optical Microscopy), SEM-EDS analyses coupled with back-scattered electrons (BSE) images and XRD investigations concurred in identifying provenance of the fragments, at least for those less transformed by the pyrotecnological use. A provenance from Central-Western Alps (Valchiavenna lithotypes), referred to the Group D of pietra ollare as defined by the literature, was established very well for the best preserved, grey-greenish, chlorite-bearing magnesite talc-schists (soapstones stricto sensu).

Some fractured to high porosity, talc-bearing samples with anomalous grey-orange colour could also represent the same Group D of pietra ollare suffering higher temperature during use (absence of magnesite and occurrence of phases from chlorite breakdown). Other yellow-whitish fragments of pietra ollare show a modal mineralogy consisting of orthopyroxene and forsteritic olivine (± clinopyroxene), chlorite breakdown microstructures and lamellar exsolutions in the opaque grains, suggesting even higher temperature of anthropogenic heating, making difficult to establish their geological provenance. Concerning the crucible samples, they are represented by ochraceous, porous, very fractured and refractory rocks made of pyroxene, olivine and opaque minerals (and pseudomorphic phases after breakdown of talc+chlorite) covered, in the interior wall, by an anthropic glassy layer (up to 10 mm of thickness) having relatively high silica, calcium and alkalis. Within the 1 mm interface of the glass-rock contact of these crucibles, a clinopyroxene palisade of prismatic microlite crystals (up to 0.1 mm), perpendicular to the rock surface or around olivine nucleii is recognizable, as well as swallow-tailed crystals due to quench crystallization after an incipient partial melting of the pietra ollare lithotype during the pyrotechnological use.

S4-P43.412

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Inter-Instrumental Calibration and Data Comparison for XRF Analysis of Obsidian and other Archaeological Materials

Tykot, R.H.

Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: XRF data calibration; obsidian

The use of portable XRF instruments to conduct non-destructive elemental analysis of archaeological materials has increased tremendously in recent years, producing much data for each project. In some cases, however, no calibration of the raw data has been performed, while in many others different calibration software programs and standards have been used, limiting direct comparisons of numeric values between these studies. This is an issue not only between different XRF brands but also different models and even between batches or individual instruments. This study focuses on comparing both raw count data and calibrated values for analyses conducted over the past 13 years on the same obsidian artifacts and geological samples using four different Bruker Tracer models (III-V+, III-SD, Vi, Vg) - and different batches for three of these - while also comparing with published values from studies by other scholars.

One major issue with XRF data is the need for matrix-matching calibration, since the major elements in the sample composition affect the secondary X-rays reaching the detector. There also are significant differences between XRF spectrometer models in their X-ray generator, detector windows, filters, and detectors, and therefore the raw data which is produced. Measurement of low-Z elements varies a lot between instruments and whether a vacuum or helium flow is used. Calibration of each XRF instrument is accomplished by analyzing “standard” samples with known concentrations of elements of interest to create a calibration curve that relates those values to their raw counts.

A large number of geological obsidian samples from different sources (and hence variations in their concentration) has been analyzed by the University of Missouri using INAA, LA-ICP-MS, and XRF, and these standards and their official concentration values are available for others to analyze by pXRF. Theoretically, this would allow an archaeologist to make a direct comparison of the data from analysis of archaeological artifacts with the values for geological samples produced by others using similar or different instruments. Ideally, the combined data could be included in the same graphs and matches correctly made. This study specifically addresses how reliable this is by evaluating element concentrations, different element ratios, and multivariate statistical values using the data produced on the same samples from the multiple pXRF, INAA, LA-ICP-MS, and ED-XRF instruments.

S4-P45.455

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Colour degradation of 18th century Aubusson tapestries

de La Codre, H.[1], Servant, L.[2], Chapoulie, R.[1], Mounier, A.[1]

[1]Archéosciences Bordeaux (UMR 6034 CNRS / Université Bordeaux Montaigne) Maison de l'Archéologie 33 607 Pessac – France
[2]Institut des Sciences Moléculaires (UMR 5255 CNRS/Université de Bordeaux) 33405 Talence – France

Keywords: Tapestry; Dyes; Fading; Colour restitution

Aubusson, located in central France, is famous for its tapestry manufactory. In 2009, the knowledge of making Aubusson tapestries was added to UNESCO's List of Intangible Cultural Heritage of Humanity. In the 17th century, when the factory earned its title of Royal manufactory, the “Verdures fines” were born representing landscapes. These tapestries were made with specific techniques and materials to provide superior quality. However, the passage of time, environmental factors and especially exposure to light have damaged and faded the tapestries. The colours of the tapestries appear pastel and dull, but current research shows that, at the time, the shades were, in fact, bright and contrasting.

A project containing several steps was set up to identify the material used in the tapestries and understand the fading. The first consisted of creating a colour chart of 18th-century recipes varying textiles (silk or wool), parameters in the formulations of the mordants and dye baths. In a second step, reference spectra of these samples were recorded with several non-invasive analytical methods from ultraviolet to near-infrared range (HSI-VIS-NIR, FORS, LEDµSF…) to create a reference spectral database and finally compared to the data obtained on tapestries. In a final step, the laboratory's degradation protocol was set up to study the loss or change of colours exposed to light in the tapestries.

This study provided results concerning the lightfastness of several dyes. For example, indigo, known to be a light-resistant dye, appears to be very fleeting when dyed on silk but not on wool. This result allowed us to ensure that most of the yellow silk areas were, in fact, bright green when the tapestry was woven. These observations permitted the implementation of a methodology for the digital restitution of the original colours of the tapestry.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Lithic finds from the Neolithic settlement Veszprém, Jutasi street, W Hungary

Biró, K.T.[1], Regenye, J. [2]

[1] *Hungarian National Museum, Budapest*
[2] *Laczkó Dezső Museum, Veszprém*

Keywords: Lithic assemblage; Late Neolithic; Hungary; provenance

Veszprém-Jutasi út is one of the most important and certainly most extensively researched settlements of the Late Neolithic Lengyel Culture in Veszprém county. It was first excavated in the 1920-ies, and significant further efforts were made here in the 1970-ies of the last century.
Modern rescue excavations were performed here on large surface (8 000 m2) in 2003 by J. Regenye.
The lithic material of former excavations was studied by K. Biró in course of tracing distribution of 'Szentgál flint' and other Middle Jurassic radiolarites of the region. The lithic industry of the settlement unearthed by the 2003 excavation has been partly published, together with pottery and settlement features, in three subsequent part, most of them still in print.
Given the specific conditions of large scale rescue excavations, the recovery of the finds was mainly restricted to closed features (graves, pits, ditches). In spite of this, the number of lithic, especially with good and controlled context, is among the largest known from the period. Our current work aims at presenting the complete lithic inventory, with chipped stone artefacts, polished stone tools and a large variety of other stone utensils.
The basic method for the investigation of lithic industry was macroscopic analysis and comparing to existing reference collection of the Hungaian National Museum. For potential long distance elements, further non-destructive petrographical and geochemical techniques (especially PGAA and surface microscopy) were used.
Provenance study of the site revealed intensive local production as well as far reaching spatial contacts.

S4-P49.508

Titanclinohumite in serpentinite: an indicator of long-distance connection (from the W-Alps to NW-hungary)

Péterdi, B.[1], Kovács, Z.[2], Szakmány, Gy.[2], Kasztovszky, Zs.[3], T. Biró, K.[4]

[1]*Supervisory Authority of Regulatory Affairs*
[2]*Department of Petrology and Geochemistry, Eötvös Loránd University*
[3]*Centre for Energy Research*
[4]*Hungarian National Museum*

Keywords: titanclinohumite, serpentinite; polished stone tool; Western-Alps; Hungary

Serpentinite was a widely used raw material for polished stone tools in the Carpathian Basin especially in the western and north-western territories. In the other parts of the Basin only few serpentinite artefacts were found on a particular site. In the environs of the Carpathian Basin there are some serpentinite sources on the surface: e.g. the Penninic tectonic windows of the Eastern Alps (Austria), Lower Silesia (Poland), the Slovak Ore Mountains (Slovakia), the Maros Valley (Romania), the Vardar Zone in the Dinarides (Serbia), the Rhodopes (Bulgaria-Greece) etc. Due to the similar mineral and chemical composition of these “harzburgitic” serpentinites it is difficult to distinguish the potential geological sources. However, some characteristic mineral-chemical features may help to identify some raw material sources.

One of the aims of our recent research is the detailed petrographic and geochemical characterization of the serpentinite artefacts found on Hungarian sites, using primarily non-destructive methods: PGAA and SEM-EDX: the “original surface investigation” method. In the case of fragmented artefacts we are using classical thin-section petrography and SEM-EDX as supplementary methods.

In the course of our investigations we found a special mineral phase: titanclinohumite in the serpentinite raw material of an artefact from the Miháldy-Collection (an old surface collection of the Laczkó Dezső Museum, Veszprém, Hungary). The presence of titanclinohumite in a serpentinite rock indicates high pressure conditions at the formation of the rock. In our case titanclinohumite exist in association with clinopyroxenes (diopside, augite), magnetite, chromium-spinel, Cr-bearing chlorite, olivine and pentlandite with serpentine minerals. Such serpentinite can not be found in the vicinity of the Carpathian Basin, so the source of this raw material must have originated from a longer distance: most probably from the Aosta Valley (Italy) in the Western Alps. The Voltri Massif (Italy) or the Malenco Valley (Italy) can also be considered for the provenance of this axe.

The authors would like to thank the National Research, Development and Innovation Fund (NKFIA) Grant No. K 131814 for financial support.

S4-P50.509

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Iron-rich raw material in Middle and Upper Palaeolithic caves in Belgium: characterization, transformation and sourcing

Chassin de Kergommeaux, A.[1,2], Chalmin, E.[2], Goovaerts, R.[3], Jadin I.[4], Leduc T.[3], Lemasson Q.[5,6], Salomon, H.[2], Goemaere, E.[3]

[1]Univ Lyon, ENS de Lyon, Université Lyon 1, CNRS, UMR 5276 LGL-TPE, F-69364, Lyon, France
[2]EDYTEM (UMR 5204), Univ. Savoie Mont-Blanc - CNRS, 5 bd de la mer Caspienne, 73376, Le Bourget du Lac Cedex, France & Université Savoie Mont Blanc, CNRS, Ministère de la Culture, France
[3]Geological Survey of Belgium, Royal Belgian Institute of Natural Sciences, rue Vautierstraat, 29, 1000, Brussels, Belgium
[4]Anthropology & Prehistory, Quaternary, OD Earth and History of Life, Royal Belgian Institute of Natural Sciences, rue Vautierstraat, 29, 1000, Brussels, Belgium
[5]Centre de Recherche et de Restauration des Musées de France (C2RMF), Ministère de la Culture, Palais du Louvre, 14 quai F. Mitterrand, 75001, Paris, France
[6]New AGLAE FR 3506 – Chimie-Paristech/Ministère de la Culture et de la Communication, Palais du Louvre,14 quai François Mitterand, 75001 Paris, France

Keywords: Haematite; oolihic ironstones; sourcing; caves; Upper Palaeolithic

Earliest archaeological occurrences of red matter in Belgium comes from Middle and Upper Palaeolithic layers of several caves (Spy Cave, Goyet Cave, Trou Magrite, Walou Cave...) developed along the Meuse river and its main tributaries. These caves were intensively excavated since the end of the 19th century and are major milestones in the history of science and in the piecing together of clues regarding the life of Neandertals. They are formed in Middle Devonian and Dinantian limestones from the Ardennan Allochton and the Brabant Parautochton.

Red matter is divided in two groups: red Clinton-type oolithic ironstones (OIS) and non oolithic iron-rich or iron-stained sedimentary rocks (sandstones, siltstones, alterites). Characterization of artefacts was achieved through a) macroscopic to microscopic observations (Optical Microscopy in thin-section and Scanning Electronic Microscopy), b) elemental analyses by Energy Dispersive X-Ray Spectrometry (EDS), Particule Induced X-Ray Emission (PIXE) and Laser Ablasion Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS); c) mineral identification by X-Ray Diffraction and d) Magnetic Susceptibility, to make possible the identification of fingerprints to differentiate the different geological and geographical sources. Several stratigraphically distinct OIS levels have been reported from Lower Devonian (Lochkovian), Middle Devonian (Eifelian), Upper Devonian (Frasnian and Famennian) for Red-Clinton-type OIS, and Middle Jurassic (Aalenian and Bajocian) for Minette-type OIS.

The discovered oolithic material can be divided into two main groups: the red OIS and the carbonated OIS. The differences lie in the nature of the binding material between ooids (haematitic terrigenous matrix *versus* carbonated cement) but also in the size and shape (undeformed to strongly deformed) of the ooids and their fossiliferous content, indicating that they come from different geological settings (paleoenvironments of deposit) and distinct stratigraphic levels. The criteria of selection and the short/long distance transport of these kinds of matter will be discussed to draw up an overview of the exploitation, transformation and supply of iron-rich material during Belgian Middle and Upper Palaeolithic. Finally, a comparative approach will be attempted with iron-rich artefacts of Early Neolithic from Belgium (Hesbaye area).

S4-P54.576

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

New Data from Obsidian Analyses Reveal Trade Networks in Prehistoric Yellowstone National Park (Northwestern USA)

Vianello, A., Tykot, R.H.

Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: pollen; pXRF; soil; Peru

An analytical study of nearly 700 obsidian artifacts from Yellowstone National Park using a Bruker 5i pXRF and encompassing several Native American prehistoric sites (ca. 10,000-1,000 BP) is revealing new patterns of source acquisition and trade of obsidian. In particular, the use of raw material from Obsidian Cliff is mapped across the park. The use of the park area in antiquity was mostly seasonal or periodic, largely due to the coldness of winters and abundance of snow, and it was shared among several tribes, as it is today. As a result, specific patterns of consumption are highly variable, but it is possible to track local procurement of obsidian vs. access to more distant sources.

Obsidian Cliff was undoubtedly a major source for obsidian, and it was traded significant distances, including to Ohio and Maine. Yellowstone Park was inserted in existing trade networks as demonstrated by the presence of different sources, and it was a place for different tribes to meet, given the periodic occupation of the land and the vast spaces available. Tracking and mapping the movement of obsidian in different areas is of great value to identify the major routes in ancient exchange systems and identify areas possibly used by different tribes, such as the Shoshone and Black Feet that are known to have frequented the area before European contact. It is also an area of great significance for insights on craft specialization among mobile Native Americans and the development of very long-distance trade networks across America.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multi-analytical investigation of historic mortars technology from water cisterns at Vryokastro, Kythnos, Greece

Papoutsaki, A.[1], Facorellis, Y.[1], Karatasios, I.[2], Boyatzis, S.[1], Stefanis, A.[1], Mazarakis- Ainian, A.[3]

[1]Department of Conservation of Antiquities and Art Works, School of Applied Arts and Culture, University of West Attica AgiouSpyridonos, 122 43 Aegaleo, Greece
[2]Institute of Nanoscience and Nanotechnology, National Center for SientificReaserch"Demokritos", AgiaParaskevi, 15310 Athens, Greece
[3] Department of History, Archeology and Social Anthropology, University of Thessaly, Argonauton& Filellhnon,38221 Volos, Greece

Keywords: historic mortars; cisterns; Vryokastro; analytical techniques

The archaeological site of Vryokastro, the ancient capital of the island of Kythnos, is located on the northwest side of the Cycladic island of Kythnos, between the bay of Merichas and the bay of Apokrousi (37° 24´ 28´´ N, 24° 23´ 42´´E).Since 2002 the site is being excavated by the Department of History, Archeology and Social Anthropology of the University of Thessaly in collaboration with the Ephorate of Antiquities of the Cyclades. According to the archaeological finds, the site was inhabitedbetweenthe 10thcentury BCandthe 7thcentury AD.

In this paper we investigate the technology of mortars originating from the main cistern(ca 5th-4th BC)found next to the sanctuary of Asclepios and Aphrodite of the Upper Town (3 samples)in comparison with other mortars sampled from another cistern, which is still in use (1 sample), and the main fountain-house (3 samples)in the middleof the ancient city.

Powdered, freshly fractured and polished sections were analyzedin order to determine their chemical and mineralogical composition using variousanalytical techniques, namely: Optical Microscopy (OM), Scanning Electron Microscopy with micro-analyzer (SEM/EDS) and X-rays diffraction (XRD). Furthermore, grain size analysis, Differencial Thermal Analysis (TG/DTA), Fourier Transform Infrared Spectroscopy (FTIR) and Mercury Intrusion Porosimetry (MIP) were also conducted.

FTIR results indicated the existence of calcium carbonates, silicates, quartz and nitrates (biodeterioration products). One sample from the main cistern also present hydrated gypsum (CaSO4.2H2O). The size of aggregates was found to be <8 mm in all samples except for two samples obtainedfrom the interior of the fountain whose size is <4mm.Their rounded shape and the presence of seashellssuggest that sand from a local beach was addedin the mortars. Halite (NaCl) crystals were detected in the samples and their presence may be due to the proximity of the site to the sea. The low pore size of the mortars (<3%) is in agreement with the purpose of their use.TG/DTA analysis showed that a loss of chemical bound water between 3.8-5% occurred between 200-600 oC (endothermic peaks). In conclusion, it concerns of lime mortars containing calcitic (pure and dolomitic), quartz, feldspars (KAlSi3O8), Fe oxides and in some samples seashells and charcoal aggregates.

Analysis and state of conservation of Prehistoric pigment in open-air schematic rock art panels in post-Paleolithic rock art sites in Iberian Peninsula: the case of Juanita shelter and Bercialejo shelter.

Rosina, P.[1], Collado, H.[2], Garcês, S.[1], Gomes, H.[3], Eftekhari, N.[5], Leis, M.[5], Nicoli, M.[4], Vaccaro, C.[5]

[1]Instituto Politécnico de Tomar; Centro de Geociência de Coimbra, Portugal, prosina@ipt.pt
[2]Consejería Educación y Cultura; Junta de Extremadura, Spain; Centro de Geociência de Coimbra, Portugal
[3]Centro de Geociência de Coimbra, Portugal;
[4]Department of Human Studies, University of Ferrara (Ferrara, Italy)
[5]Department of Physics and Earth Sciences, University of Ferrara (Ferrara, Italy)

Keywords: Geosciences; Pigments; Prehistoric Rock Art; SEM –EDS; Micro-Raman spectroscopy

In recent years, excellent contexts of the open-air rock art of post-Paleolithic sites in quartzite lithologies of Spain have been discovered. Extremadura (Spanish region) is one of the principal regions in the Iberian Peninsula with more presence of pre-historic rock art sites, with Badajoz province corresponding to more than 565 sites itself, which constitutes more than half of the total sites in Extremadura. Badajoz province exhibits a rich heritage of archaeological as well as cultural value and forms a hallmark for many territories that goes hand in hand enhancing the landscape as well as environmental value. These testimonies of rock art have high artistic value and have survived despite their high exposure to rainfall and thermoclastism as quartzites (low-grade metamorphic rocks) being free of feldspars and micas does not produce neogenesis of minerals associated with lichen activity, so they have limited biodegradation and high resistance to weathering. This work has analyzed the rock shelter of Abrigo Juanita, located in the quarzitic crest of Sierra del Conde and Cueva Bercialejo, located on the eastern side of the province of Badajoz in the municipality of Zarza Capilla and belongs to the mountain range of Sierra de la Cabras. A set of different archaeometry techniques has been used in both sites. The pigment samples from Abrigo Juanita were observed and analysed through SEM/EDS technique. The results confirm the quarzitic composition of the rocks, the absence of clay minerals due to weathering and reveal the presence of an initial lichen colonization, invisible to the naked eye, on the painted surface. The SEM observations and EDS microanalyses have been also used to characterize the morphology of pigments and their composition. Micro-Raman spectroscopy contributed to characterize the main mineral component of the paintings. The results show the presence of hematite in the red pigment and small black particles of amorphous carbon has also been detected on its surface. On the base of this last result the small black particles may correspond to the lichens observed under the optical microscope and SEM. In Bercialejo shelter, Energy-Dispersive x-ray micro-fluorescence (EDxrf) registered iron and calcium are the main components of the samples. Calcium can be attributed to the substrate or to mineral accretion over the pigment, while iron is compatible with the presence of pigment. Unfortunately, it was not possible to acquire good Raman spectra of the samples because of the bad signal detected. This probably depends on sample composition and can be due to the presence of salts, clay minerals (such as kaolinite) or organic matter.

S4-P65.635

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Provenance of the pumice manuports from archaeological sites at the Vitim river, East Siberia, Russia

Demonterova E.[1], Tetenkin A.[2], Ivanov A.[1], Lebedev V.[3]

[1]Institute of the Earth crust SB RAS, Irkutsk, Russian Federation
[2]Irkutsk National Research Technical University, Irkutsk, Russian Federation
[3]Institute of the Geology of Ore Deposits, Petrography, Mineralogy, and Geochemistry, Moscow, Russian Federation

Keywords: pumice manuport, East Siberia, elemental analysis, K-Ar dating method

The migration of prehistoric people along large rivers is considered to be one of the major factors of cultural communications in North-East Asia. In this presentation we will talk about a pumice manuport, which was excavated at archaeological sites: Ust'-Karenga XVI (8-7 kyrs. cal BP), the upper part of the Vitim River and Kovrizhka III (~11 kyrs. cal BP), the lower part of the Vitim River, East Siberia, Russia. Pumice material is fragile and could not have been transported by water flowing to these archaeological sites and had to be carried out by people from where it was collected. There are two volcanic fields, namely Vitim and Udokan, which could be sources of the pumice manuports, both are ~300 km away from the Ust'-Karenga XVI site. These volcanic fields are 520 and 800 km away from the Kovrizhka III site. The chemical composition of the pumice manuport from the Kovrizhka III site points its origins towards the distant Udokan volcanic field. Whereas the chemical composition of the pumice manuport from the Ust'-Karenga XVI site is ambivalent and can be matched to pumice from any of the two volcanic fields. To solve the question, where the pumice manuports were collected, we dated the pumice samples by the K-Ar dating method. The manuports from the Kavrizhka III and Ust'-Karenga XVI sites yielded an age of 3.30 □ 0.12 million years and 2.63 □ 0.11 million years, respectively. Using published K-Ar ages of eruptions within the Udokan and Vitim volcanic fields we unambitiously match the manuport from the Ust'-Karenga XVI site to the Udokan volcanic field. The manuport from the Kovrizhka III site can be matched by the K-Ar age to either of the two volcanic fields. However, the chemistry of rocks from the Udokan volcanic field also points to the manuport from the Kovrizhka III site. Thus, this is an additional proof of the migration of Siberian people in the late Pleistocene – Early Holocene covering distances of up to 300-600 km.

We acknowledge the Centre for Geodynamics and Geochronology at the Institute of the Earth's Crust SB RAS for the access to the analytical equipment and grants of the Russian Science Foundation (19-78-10084) and Russian Foundation for Basic Research (18-59-22003\18) for the financial support of the analytical and archaeological researches, respectively.

S4-P67.640

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

TT8 Project: decoding the materiality of the intact grave goods of Kha (Egypt, ca 1400 a.C.)

Ferraris, E.[1], Facchetti, F.[1], Turina V.[1], Borla, M.[2], Cavaleri, T.[3], Gargano, M.[4], Festa, G.[5], Romano, F.P.[6]

[1]Museo Egizio, Via Accademia delle Scienze 6, 10134, Torino (TO), Italy
[2]Soprintendenza ABAP-TO, Piazza San Giovanni 1, 10134, Torino (TO), Italy
[3]Fondazione Centro Conservazione e Restauro La Venaria Reale, Via XX Settembre 18, 10078, Venaria Reale (TO), Italy
[4]Dipartimento di Fisica A. Pontremoli, Università degli Studi di Milano, Via Celoria 16, 20133, Milano (MI), Italy
[5]Museo Storico della Fisica e Centro Studi e Ricerche "Enrico Fermi", Piazza del Viminale 1, 00184, Roma (RM), Italy
[6]ISPC-CNR, Via Biblioteca 4, 95124, Catania (CT), Italy

Keywords: Multispectral imaging; MA-XRF; Neutron and Gamma Techniques; RTI; Museums;

On February 15th, 1906, in a valley next to the village of Deir el-Medina, after a month of hard work with more than 250 workers to remove vast deposits of debris, the Italian archaeological mission discovered the shaft of an underground tomb, which had been sealed by a landslide. This shaft led down to passage at the end of which, after removing a sturdy wooden door, the diggers found themselves in a chamber with the intact grave goods of two high-ranking individuals, the “director of works” Kha and his wife Merit, who lived during the XVIII Dyn. (around 1400 B.C.). Schiaparelli was granted permission to bring all the 440 objects (painted wood and pottery, metal bowls and tools, textiles, oils and compounds, food and human remains) found in the tomb to Turin, leaving only a few objects in Cairo, and since then the grave goods of Kha and Merit is exhibited at the Museo Egizio.

After 90 years and few studies focused on individual objects or groups of objects, TT8 (funerary chapel, tomb, and grave goods) still lacks a comprehensive research and publication embracing archaeological, historical, cultural and conservative perspectives.

Egyptological interests could benefit from the diagnostic investigations that precede the restoration process; indeed, the latter is more and more an opportunity to investigate the nature, the origin and alterations of the materials the objects are made of, as well as shedding new light on the production techniques and the conservative history of an ancient artifact.

In 2017, due to the relevance of TT8 for the discipline and for the scientific history of Museo Egizio, it has been started a plan of archaeometric analysis as part of a research programme titled “TT8 Project” to investigate all the 460 intact objects from the tomb; the project is developed in collaboration with a growing international community of scholars and institutions (heritage scientists, Egyptologists, restorers, museum curators, archivists, archaeologists) and aims to publish a full study of TT8 for the bicentennial of the Museo Egizio collection, in 2024.

The paper will present the scientific and museological framework of TT8 project and a summary of the preliminary results obtained in this first 2 years of activities that provided invaluable information about the characterization of materials employed in the production of an ancient Egyptian grave goods dating back 3400 years.

S4-P68.641

Multi-technique analytical approach for the study of Romanesque wall paintings from "Santuario della Consolata" (Turin, Italy)

Cavaleri, T.[1,2], Borghi, A.[3], Gambino, F.[3], Barberis, E.[4], Marengo, E.[4], Manfredi, M.[5], Poretti, G.[6], Fedi, M.[7], Liccioli, L.[7], Barone, S.[8], Cardinali, M.[1], Malizia, I.[1], Manchinu, P.[1], Moratti, V.[9], Fantone, M.[9]

[1]Fondazione Centro Conservazione e Restauro La Venaria Reale, Via XX Settembre 18, Venaria Reale (TO), Italy
[2]Dipartimento di Economia, Ingegneria, Società e Impresa, Università della Tuscia, Via del Paradiso, 47, 01100 Viterbo, Italy
[3]Dipartimento di Scienze della Terra, Università degli Studi di Torino, Via Valperga Caluso 35, Torino, Italy
[4]Dipartimento di Scienze e Innovazione Tecnologica, Università del Piemonte Orientale, Viale Teresa Michel 11, Alessandria
[5]Department of Translational Medicine, Center for Translational Research on Autoimmune & Allergic Diseases – CAAD, Università del Piemonte Orientale, Corso Trieste 15, 28100, Novara, Italy
[6]Santuario della B.V. Consolata, Diocesi di Torino, Via Maria Adelaide 2, Torino, Italy
[7]INFN Sezione di Firenze, via Sansone 1, 50019 Sesto Fiorentino (FI), Italy
[8]Dipartimento di Chimica Ugo Schiff, Università di Firenze, via della Lastruccia 3, Sesto Fiorentino (FI), Italy
[9]Soprintendenza archeologia, belle arti e paesaggio per la città metropolitana di Torino, Ministero dei beni e delle attività culturali e del turismo, Musei Reali di Torino, Piazzetta Reale, 1, Torino (Italy)

Keywords: Romanesque wall paintings; Archaeometry; minero-petrographic analysis; protein binder; mortar radiocarbon dating

An archeometric study was conducted on the Romanesque wall paintings belonging to “Santuario della Consolata”, well-known as a masterpiece of the Piedmont’s Baroque in Turin. The actual sanctuary baroque body is the result of numerous expansions, from the first nucleus of the Romanesque Sant’Andrea’s church to the last massive renovation projects realized by famous architects as Guarini, Juvarra and Ceppi. Recently a conservation intervention in one of the church chapels had brought some wall paintings of the ancient Sant’Andrea church to light, that were preserved under a multiple plaster layers. It is an extraordinary discovery for the community and even more for the city of Turin preserving very few testimonials of medieval architecture. The aim of the study is to characterize the paintings material and the technique used. A multidisciplinary team of scientists, art historians and conservators approached to the site in order to guide the conservation intervention and to analyse the most interesting aspects of the paintings, such as chemical composition, dating and provenience of the ingredients used and state of preservation. Multispectral imaging and XRF spectrometry led to the first reconnaissance of the palette. Cross section samples coming from the North and South walls of the chapel were analysed with optical microscopy and SEM-EDS to document the refined pictorial technique: an expert use of coloured backgrounds and pigment mixtures to render depth effects were detected while the presence of lapis lazuli suggested the use of a different organic binder. In addition to traditional FT-IR spectroscopy, a new technique of non-invasive extraction of proteins with EVA strips was applied to characterize the binder. The analyses performed by Nano LC-MS/MS allowed us to identify two ancient proteins from animal glue (Bovine). Moreover, thin section samples were prepared to study the recipes of the mortars, the composition of the inorganic binders and the provenience of the aggregate: a new protocol based on minero-petrographic approach with optical microscope and SEM-EDS analysis was applied. The information collected from the petrographic analyses also allowed us to study the feasibility of dating mortars by radiocarbon. The collected information enabled us to investigate the raw materials used by the artists and to make a correlation between the two walls.

S4-P71.669

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric analysis of former Bazzani Mill in the Municipality of San Possidonio (Modena)

Lattao, V.[1,2,3], Marrocchino, E.[4], Eftekhari, N.[5], Mariotti, G.[5], Rosina, P.[2,6], Vaccaro, C.[4]

[1]University of Coimbra (Polo II), Faculty of Sciences and Technology, Department of Earth Sciences and Geosciences Center, Portugal
[2]Geosciences Centre, University of Coimbra (u. ID73 – FCT), Portugal
[3]Foundation for Science and Technology, Portugal
[4]University of Ferrara - Department of Environmental and Prevention Sciences, Italy
[5]University of Ferrara – Department of Physic and Earth Sciences, Italy
6Polytechnic Institute of Tomar, Portugal

Keywords: Artificial stone; mortar; cement; plaster; brick

At the end of the XIX century the use of artificial stone spread for the creation of high-quality decorative surfaces which previously lacked decorations due to the high cost of ornamental natural stone materials. The production of artificial stone consists in a reuse of secondary materials derived from the waste material created during the processing of ornamental rocks. A perfect example of this high-quality decorative surfaces is the Mulino Bazzani, in the municipality of San Possidonio (Modena). This mill was built at the beginning of the 20th century, with steam plants and worked until the post-war period. This work gave rise to the relationship of the post seismic redevelopment plan, according to the protection constraints defined by the Superintendency for Monumental Heritage. The project involves the architectural recovery of the façade and the enhancement of the building and also consists in the relocation of the Varini theater. For this work samples of mortars, cements, plaster and some bricks (both from the façade and the inner parts) were taken.
Chemical (WD-XRF, SEM-EDS), petrographical (Stereomicroscope, Optical microscope) and mineralogical (XRD) analyses were carried out to define the nature of the materials and also, in order to identify the presence of any salts or other types of degradation.
A mixture of cement and gypsum is applied in lateral bodies on masonry brick walls and in the central body on elements in artificial stone.
The cement mortars of the elements in artificial stone were made with Portland cement added to about 5% of gypsum and fine sand as aggregate. This aggregate from the petrographic study consist of local sand with 5% of finely grounded brick fragments. An exception is the covering of the west façade which has undergone important alterations and restructuring works and which presents salt efflorescences.
The internal mortars are all aerial mortars with added plaster. Inside the structure there are several layers of plaster characterized by a different composition based on the purpose they took. The chemical analysis allowed us to identify the use of local clays not belonging to the Po but to the Reno and Panaro rivers. The presence of calcium sulphate (gypsum) also emerged from the chemical analysis, probably due to the mobilization of the gypsum in the wall faces affected by capillary rise.

S4-P72.674

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Black or blue? The backgrounds of Greek 16th Century wall paintings)

Mastrotheodoros, G.P.[1,2], Filippaki, E.[2], Beltsios, K.G.[3], Papadopoulou, V.[4], Bassiakos, Y.[2]

[1]*Conservation of Antiquities & Works of Art Department, West Attika University, Aegaleo, Greece.*
[2]*Institute of Nanoscience and Nanotechnology, NCSR "Demokritos", Agia Paraskevi, Greece.*
[3]*School of Chemical Engineering, National Technical University of Athens, Zografou, Greece.*
[4]*Ephorate of Antiquities of Arta, Arta, Greece.*

Keywords: fresco-secco-charcoal-azurite-smalt

The majority of the Greek territories remained under Ottoman rule from circa mid-15th up to mid-19th century. Nevertheless, most of the inhabitants retained their Christian Orthodox faith and continued practicing their liturgical duties, and, hence, the religious painting flourished throughout this extended period of time. Craftsmen of that era created thousands of portable panel paintings ("icons") and decorated the walls of hundreds of churches with mural paintings, many of which survive today. The high artistic and spiritual value of these paintings has been greatly appreciated by recent scholars
and a significant effort to study and preserve this artistic creation has emerged.
Thus, many important 16th century monuments located in the Epirus territory (NW Greece) underwent recently restoration treatment. In this framework, the materials and techniques employed in the wall paintings of six churches were investigated by means of various analytical techniques. In detail, corresponding micro-samples were thoroughly examined through optical microscopy, SEM-EDX, XRD, μ-XRF and μ-Raman, paying special attention on the samples that stem from the backgrounds of the paintings that nowadays appear rather black.
The investigation of the relevant samples showed that in all cases the backgrounds have been indeed rendered by a carbon black and lime mixture applied on a wet plaster. Nevertheless, in several instances we have located at the background surfaces small amounts of blue pigments that can be best interpreted as remnants of corresponding top layers originally applied via a secco technique. Consequently, 16th c. wall paintings backgrounds that appear black today were, often if not always, originally deep blue ones as a result of superimposition of azurite or smalt on the blackish (: charcoal + lime) substrate. The near complete loss of the blue top material should be attributed to the gradual deterioration and eventual failure of an unidentified organic medium / binder. Later blue overpaintings, also occasionally surviving today, are compatible with the will to restore the visual impact of the original
backgrounds. On this basis one shall reassess these paintings, bearing in mind that their contemporary appearance deviates substantially from their original one.

S4-P74.692

Mineralogical and chemical diversity of mineral and glass beads from the area of the one of the biggest Polish early medieval necropolis in Dziekanowice, Greater Poland, Poland

Szczepaniak, M.[1], Wrzesińska, A.[2], Wrzesiński, J.[2]

[1]Institute of Geology, Adam Mickiewicz University, ul. B. Krygowskiego 12, 61-680 Poznań, Poland
[2]Museum of the First Piasts in Lednica, Poland

Keywords: mineral and glass beads; necropolis; Middle Ages, SEM; Poland;

Dziekanowice village, site 22, is one of the largest cemeteries from the Middle Ages in Poland. A total of 1665 graves and 1730 bodies at various ages have been identified so far (Wrzesiński, 2016). In addition, many others artifacts have been identified in individual graves. They contained coins, knives, bone products, stone objects and various types and shapes of beads that are part of the jewellery.
The work presents the results of microscopic and chemical analyses of selected 38 various mineral and glass beads from the necropolis in Dziekanowice. Laboratory analyses were conducted using EPMA (Electron Probe Micro Analysis) and SEM-EDS (Scanning Electron Microscopy - Energy Dispersive Spectroscopy).
Quartz beads constituted the largest group of the beads. The second most numerous group were beads made of orange chalcedony - carnelian. Among other beads, objects made of amber, flint, white chalcedony, fluorite and lead silicate glass with variable chemical composition were identified.
The material from which individual beads were made does not occur in the area of Dziekanowice and Greater Poland. With the exception of flint, it was certainly entirely imported material. Due to the monotonous geological structure of the area of central Poland (mainly alluvial and postglacial sediments) and the lack of the medieval glass workshops in this area, after identifying the composition of the beads, a problem of their provenance appeared. Therefore, the goal of the analysis has become not only the identification of the beads themselves, but also determining of their origin. However, due to the lack of a comparative base determining its provenance is difficult.
However, looking at the similarity to archaeological material from the Early Middle Ages sites of Lower Silesia (both in composition and appearance), it can be assumed that this region of Poland could become the source of Dziekanowice beads. In the middle of the 10th century, the importance of the export of mineral material and glass products from Lower Silesian workshop (mainly from the regions of Niemcza, Wrocław and Opole; Lisowska, 2013) also increased gradually.

WRZESIŃSKI J. 2016: Nummus Bonum Fragile Est. Groby z monetami wczesnośredniowiecznego cmentarzyska w Dziekanowicach. Biblioteka Studiów Lednickich Fontes 7:1, Lednica.
LISOWSKA E. 2013: Wydobycie i dystrybucja surowców kamiennych we wczesnym średniowieczu na Dolnym Śląsku. Uniwersytet Wrocławski, Instytut Archeologii, Wrocław.

S4-P76.700

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of Roman mortars from Bracara Augusta (NW Portugal)

Fragata, A.[1], Rocha, F.[1]

[1]*GeoBioTec, Universidade de Aveiro, Portugal*

Keywords: Roman mortars, Bracara Augusta, mortar characterization, XRD, XRF

Bracara Augusta is one of the three urban centres of the Iberian peninsula northwest region created in the context of the administrative organization carried out by Emperor Cesar Augustus.
This work will focus on roman mortars from mosaic floor base layers as well as render and plasters from walls of “domus” showing different states of conservation and from different sites of Bracara Augusta. The mortars from the mosaic layers of the imperial domus integrated in Regional Museum of Archeology D. Diogo de Sousa - the oldest roman dwelling testimonies found in Bracara Augusta - will be deeply studied.
Despite its sites archaeological relevance, no literature data concerning the mortars characterization is available. This study intends to fill this gap by mineralogical and chemical characterization of mortar samples, mainly by means of X-ray diffractometry (XRD) and X-ray fluorescence (XRF) analyses.
This work intends to be a first approach to mortars characterization of Bracara Augusta, providing valuable data on the construction material and techniques used on ancient Roman building materials and contributing to understand mortars state of conservation based on the composition, execution techniques and surrounding conditions. At the end, a correlation between the analyzed characteristics of the studied mortars and the results of ancient Roman mortars from other archaeological sites will be established. In the future the results from this research aims at furnishing a useful reference for conservation actions in roman sites of Bracara Augusta.

S4-P79.734

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Between sea and mountains, local rocks as a marker of mobility and exchange of Corsican protohistoric peoples.

Leck, A., Le Bourdonnec, F.-X., Chapoulie, R.

Institut de Recherches sur les Archéomatériaux - Centre de Recherche en Physique Appliquée à l'Archéologie (IRAMAT-CRP2A) – Univ. Bordeaux Montaigne, CNRS : UMR5060 – Maison de l'Archéologie, Esplanade des Antilles, 33607 Pessac, France

Keywords: Neolithic, Corsica, Rhyolite, EDXRF, Provenance Study

During pre- and protohistory, the first inhabitants of Corsica imported many lithic materials to make their tools, in particular obsidian and flint from Sardinia, two materials of good quality but absent from the Corsican landscape. These exogenous rocks have been at the heart of most of the typotechnological and provenance studies carried out on the lithic series unearthed in Corsica, highlighting the existence of long-distance diffusion networks. Endogenous rocks have also been used, notably rhyolite, a fine-grained siliceous volcanic rock widely distributed in Corsica, but they have received much less attention, notably due to a lack of methodological tools. Indeed, provenance studies of flint and obsidian have been carried out for several decades in many places in the world and multiple methodological developments have already been realized, which has not been the case for rhyolites. However, this material seems to have circulated at different levels within the island, from local to regional, and is thus potentially a carrier of information on mobility, exchange networks or territoriality, inaccessible from the study of exogenous materials.

After setting up an effective and non-destructive analytical protocol to determine the origin of Corsican rhyolites, mostly based on EDXRF method, and built a geological referential based on field prospections, we analysed several archaeological series, from the Early Neolithic to the Middle Bronze Age. We present here the results of a 4-year research project that allow us to get a first glimpse of unsuspected intra-island diffusion networks, to shed light on the relationships between coastal and mountain sites and to inform us of the ways in which these peoples used their environment.

Building Roman Britons: Sourcing the Stone and Ceramic Building Materials of Roman Bath

Kearn, O., Welham, K., Pitman, D., Brisbane, M.

Bournemouth University

Keywords: Ceramic Building Material; Stone; Roman; Provenance; pXRF

The Roman baths and temple at Bath, UK are a UNESCO World Heritage Site exploiting the only hot springs in the British Isles. Famous during the Roman period, its popularity continues to the present day with the site receiving 1.3 million visitors annually. Despite its fame, the sources of the stone and ceramic building material (CBM) used in Roman Bath are poorly understood.

Previous research at the baths and temple established the complex phases of building at this site throughout the Roman period. This work, however, focussed on the narrative of the development of the baths, but did not investigate the range, importance or sources of the building materials present. While commercial work in the city has improved our understanding of the CBM used in the Roman town, significant questions of provenance remain. Even with the increase in developer-funded archaeology in Bath, understanding of the Roman stone in the city remains rudimentary. Furthermore, the allocation of CBM and building stone to different specialists has worked against any holistic understanding of the movement of building material into the town or its part in regional exchange networks.

This project investigates the stone and ceramic building materials from Roman Bath together. These materials are being analysed at a range of scales using techniques of scientific analysis, with the results integrated to better understand the provenance, movement and conceptualisation of building materials in the wider region of Roman Bath. This will be done by combining traditional methods, such as thin-section petrography, with modern techniques of compositional analysis, such as portable x-ray fluorescence (pXRF) and SEM-EDS, to characterise and match samples of stone and CBM from Roman Bath with samples from quarries, clay deposits and kiln sites. Results so far include pilot studies using pXRF on limestone and brick and tile from the baths to explore the extent of surface contamination and the implications for characterisation and provenancing using non-invasive compositional techniques.

While this project will source the building materials of Roman Bath, it will also produce conclusions that go beyond the materials studied. This novel holistic approach will provide insights into how these materials were conceptualised, and the roles that these building materials and industries played in the development of Romano-British identities in the region of Bath.

S4-P83.754

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric analysis of clay building materials from the Iron Age of the NW of Portugal: a comparative study

Oliveira, N.[1], Gonçalves, L.[2], Bettencourt, A.M.S.[3]

[1]Landscapes, Heritage and Territory Laboratory (LAB2PT): FCT PhD fellowship University of Minho, Portugal
[2]Earth Sciences Centre (CCT/UM), Earth Sciences Department, University of Minho, Portugal
[3]Landscapes, Heritage and Territory Laboratory (LAB2PT), Department of History, University of Minho, Portugal

Keywords: NW Portugal, Iron Age, plaster/pavement, constructive characteristics, archaeometric analysis.

Archaeometric analyses have been widely applied in studies of Iron Age building materials.
Clayed plasters and pavements are rarely found in Iron Age settlements from Northwest Portugal, why few archeometric studies were done, to the date, on this type of building materials.
This research aims to present the results from microanalysis of clayed plasters and pavements from three Iron Age archaeological sites located in NW Portugal, namely São João de Rei, Castro Máximo and São Paio settlements and the ceremonial site (?) of Frijão. The materials collected for analysis comprise a chronology from the fourth to the second centuries BCE and from the second BCE to the first AD centuries, which corresponds to the Early and Later Iron Age of the region.
The study was done by means of mineralogical petrographic analysis, X-ray powder diffraction (XRD) and Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM-EDS). The main components of the studied materials are micas, particularly muscovite, feldspar and quartz, although small amounts of phyllosilicates, such as kaolinite and illite were also identified. Some of the pavements show signs of having being fired. The mineralogical data indicates that firing was done at temperatures lower than 950º C. The minerals from these materials came from the surroundings of the archaeological sites as they are commonly found in the granitic soils and weathered granitic overburden that characterize the region. This investigation offers fresh insight into proto-historic building materials from the Iron Age context of NW Portugal and provides useful knowledge of the constructive characteristics and skills of this chronology.

S4-P85.767

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Stone and decay characterization of the Palmavera nuragic complex, Sardinia (Italy)

Mameli P.[1], Puccini A.[1], Tomassetti P.[2], Cuccuru S.[1]

[1]*Dipartimento di Chimica e Farmacia, Università di Sassari, Italy.*
[2]*Segretariato regionale del Ministero per i beni e le attività culturali e per il turismo per la Sardegna, Cagliari, Italy.*

Keywords: Nuragic complex; stone characterization; decay characterization; Palmavera; Sardinia.

The Palmavera nuragic complex is an archeological site characterized by three nuragic towers surrounded by a village, whose first building phase is dated to the XV century BC. Unicum of this settlement is the occurrence, inside a “meeting hut”, of a “nuraghe” stony model, which allows to reconstruct the original shape of the towers, including the roof structures.
The present work shows the results of the study carried out during a recent excavation and restoration intervention, in order to i) characterize the ashlars and identify their provenance, ii) identify and classify the stones decay and iii) evaluate the efficiency of a consolidant in order to limit the degradation of two lithotypes.
The Palmavera complex lies in Nurra, a sub-region of north-western Sardinia (Italy). The area is geologically characterized by Mesozoic limestone/dolostone and Cenozoic volcanites. These lithotypes form also the clasts of Quaternary breccias and toghether siliciclasts form the grains of an aeolian sandstone that outcrops close to the monument.
To better understand the building stone diversity and the different deterioration features, the single ashlars and the relative alteration/degradation phenomena were mapped; in addition, samples from unweathered and weathered rocks were collected from differently exposed parts of the "nuraghe". Minero-petrographic techniques such as polarizing microscopy on thin sections and X-ray powder diffraction were performed, paying particular attention to the severe sandstone decay. In fact, despite the deterioration phenomena are differently distributed in the monument, and are characterized by almost all the different forms of alteration and degradation described by NorMaL1/88 (CNR-ICR), they are particularly evident in the sandstone, stratified or not, which was the most used building material.
A complete physico-mechanical characterization (including dry density, water absorption, capillarity absorption, open porosity, ultrasonic velocity and uniaxial compressive strength) was also carried out, following the respective UNI-EN standards, on sandstones and breccias pre and post a consolidating treatment with Nanorestore Plus©. After a seasonal cycle the treatment didn’t achieved the expected result limiting its use in conservative restoration.

S4-P90.802

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The microfossils in archaeological perspective of stone economy of Nea Paphos (Cyprus)

Dzwoniarek-Konieczna, M.

Institute of Geology, Adam Mickiewicz University in Poznań

Keywords: foraminifera; limestone; micropaleontology; Nea Paphos; stone artefacts

Microfossils are extremely important in geological studies. Their applicability in archeology is most often used for paleoclimatic studies, landscape changes (especially on the coast) or in paleobotanical analysis. In petrography of sedimentary rocks, micropaleontological research is a standard, but in research on archaeological stone objects it is less common. However, knowledge of micropaleontology has a great importance for studying the provenance of imported objects. The presence of groups of microfossils alongside other distinctive microstructure characteristics in thin section is often sufficient to relate stone artefact to specific deposits of raw materials.

This work presents the use of microfossills in research on the provenance of stone artefacts from Polish excavations in Hellenistic-Roman city of Nea Paphos, located on the southwest coast of Cyprus.

During more than 50 years of work of the Polish archaeological mission within Paphos, extensive stone material, including building and decorative materials (i.e., facing slabs, mosaics or architectural detail); utility materials, such as quern, mortaria, bowls, trays; and also sculptures, incense burners, casting moulds for coin flans making or ballistic balls; has been collected.

The material of the vast majority of these artifacts are carbonate rocks, rich in biogenic skeleton. In the course of petrographic research of these objects a number of fossils have been distinguished, e.g. algae or foraminifera and even of small fragments of mammals. Thin section researches, supported by SEM-imaging analysis, showed a differentiation in the taxonomic image of the biogenic skeleton, including foraminifera. Micropaleontological analysis of forams (e.g., Morozovella sp. or Lepidocyclinidae sp.) allowed for the diversification of raw rock materials from artifacts with similar structural and texture features, which by years were archaeologically identified as a one source of raw material. It was also possible to identify material imported from outside of Cyprus, e.g., the presence of large lens-shaped benthonic Nummulites sp. allowed the exclusion of local (Cypriot) provenance of some mortaria.

S4-P92.814

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The marble trade in the east Hellenistic-Roman Mediterranean basin: new data from the analysis of marble from Nea Paphos (Cyprus)

Dzwoniarek-Konieczna, M.

Institute of Geology, Adam Mickiewicz University in Poznań

Keywords: geochemical analysis; Cyprus; marble; petrography; provenance

The Hellenistic-Roman city of Nea Paphos is located on the south-eastern coast of the Mediterranean. It was located at the junction of zones of influence of political, economic and cultural of the contemporary world and meet a very important strategic and administrative functions. The city was a kind of gateway for Levant contacts with the world of the western Mediterranean. Despite the high economic position (copper trade), we do not know much about its place in the distribution system of stone raw materials in ancient times.

Research on the use and provenance of marbles focused on objects discovered by the Polish archaeological mission in the residential part of city - Maloutena (included e.g., Villa of Theseus). Marble artifacts involved decorative material (e.g., facing slabs, mosaics, capitals, fragments of scupltures) and utilitarian objects (e.g., vessels, mortaria or querns).

Archaeometric analysis based on petrography techniques, both macroscopically in situ and in thin section (e.g., MGS and GBS parameters description). Results were supported by SEM-EDS analysis, and also oxygen and carbon stable isotopic ratio determination and micro-chemical on XRF analysis and cathodoluminescence imaging studies.

The results indicate that marbles were imported, e.g., from the area of present-day Greece and Turkey. Many types of marble were identified from very fine (to coarse) crystalline calcite or dolomitic white marbles (e.g., Thasos Aliki or Naxos), through coarse crystalline gray or with gray streaks (e.g., Proconessos marble), and green (e.g., Verde antico from Larissa) or foliated (e.g., cipollino verde of Euboea) to the red varieties and breccia marble (e.g., breccia corallina) or Pavonazzetto, probably from Afyon.

S4-P93.816

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The red colour at the Salão Árabe (Palácio da Bolsa, Porto, Portugal)

Mestre M. Carvalho, P.[1], Rocha, F.[2], Coroado, J.[3]

[1]Geobiotec, Dep. Geociências, Univ. Aveiro, Campo de Santiago, 3810-192 Aveiro, Portugal
[2]Geobiotec, Dep. Geociências, Univ. Aveiro, Campo de Santiago, 3810-192 Aveir, Portugal
[3]Technology, Restoration and Arts Enhancement Center (Techn&Art), Instituto Politécnico de Tomar, Campus de Tomar, 3200-313 Tomar.Portugal

Keywords: Vermilion; Chrome Red; Pigments; Plaster

Polychrome plasterwork has played an important role in the decorative arts in Portugal, either in religious or in civil buildings, particularly in the north part of the country, reaching its apogee along the eighteenth and nineteenth centuries.
Several materials and techniques are used in this artistic technique in order to colour to the surface of the gypsum plaster ornaments.
The Salão Árabe (Arab Room in english) of Palácio da Bolsa, in Porto (North of Portugal), built between 1862 and 1880 is an emblematic symbol of the city due to its intense decoration, particularly the polychromatic and gilded plasterwork of its walls and ceilings. Throughout its existence, this 135 m2 room has experienced several restoration interventions, some of them without any adequate scientific supervision or technical support, apart from the latest one, in 2009.
The present study focus on the characterization by PLM-OM, SEM-EDS and XRD of four samples (removed before the latest conservation intervention) of the pigments originally used in order to provide the red colour on the wall and ceiling ornaments of the Salão Árabe, especially synthetic mercury (II) sulfide, also known as 'wet' process vermilion (HgS) and lead chromate (VI) oxide, commonly known as Chrome Red (PbCrO4.PbO).

S4-P95.826

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization and Provenance of Nelsonite Beads from the Tairona and Nahuange Cultures, Sierra Nevada de Santa Marta, Northern Colombia.

Betancur, S.[1], Weber, M.[1], Acevedo, N.[1], Villanova-de-Benavent, C.[2], Proenza, J.[2]

[1]Facultad de Minas, Universidad Nacional de Colombia, Sede Medellín, Colombia
[2]Departament de Mineralogia, Petrologia i Geologia Aplicada, Universitat de Barcelona, Spain

Keywords: Nelsonite; Tairona; Nahuange; provenance, textures.

Nelsonite beads of the precolumbian Nahuange (A.D. 100 – 900) and Tairona (A.D. 1000 – 1600) cultures were studied with Electron Microprobe Analysis, X-Ray Diffraction and Scanning Electron Microscope. Nelsonites are rare ilmenite-apatite rocks spatially linked to Proterozoic massif-type anorthosites emplaced during the Grenville orogeny (~1.2-1.0 Ga) [1]. Their black luster, high magnetism and weight distinguish them from other dark-colored stones. Archaeological nelsonite beads were identified in different important Colombian collections such as the Museo del Oro of the Banco de la República, the Instituto Colombiano de Antropología e Historia (ICANH) in Bogotá, the Mapuka Museum of the Universidad del Norte in Barranquilla, the Museum of the Universidad de Antioquia and the Anthropology Laboratory in the Universidad del Magdalena in Santa Marta. The majority of the pieces are cylindrical perforated or non-perforated collar beads of 5 cm length in average, with diameters of up to 2 cm. Nevertheless, other artifacts with carvings are present. Our results indicate that the source of the nelsonite for the Nahuange and Tairona artifact production is local, and lies in the Sierra Nevada de Santa Marta, Colombia, in the El Hierro creek that contains abundant pebbles of this material. Typical exsolution textures were observed between the different Fe-Ti oxide minerals, where a patchy, worm-like texture is present both in beads and in pebbles. EMPA results indicate a solid solution between ilmenite and titaniferous hematite (Ilm-Hemss) responsible for the patchy exsolutions, and other mineral phases such as rutile and hercynite. This is the first report of nelsonite used for archaeological production in South America. Until now, to our knowledge, Mexico is the only place where nelsonite has been reported for production of archaeological artifacts, most of them multi-perforated pieces in archaeological excavations from the Olmec culture [2], [3].

[1] Charlier, B., Namur, O., Bolle, O., Latypov, R., & Duchesne, J.C. (2015). Fe-Ti-V-P ore deposits associated with Proterozoic massif-type anorthosites and related rocks. Earth-Science Reviews, 141, 56-81.
[2] Alva-Valdivia, L.M., Cyphers, A., De La Luz Rivas-Sánchez, M., Agarwal, A., Zurita-Noguera, J., & Urrutia-Fucugauchi, J. (2017). Mineralogical and magnetic characterization of Olmec ilmenite multi-perforated artifacts and inferences on source provenance. European Journal of Mineralogy, 29(5), 851-860.
[3] Jones, S.E., Jones, S.T., & Jones, D.E. (1997). Archaeometry Applied to Olmec Iron Ore Beads. BYU Studies, 37(4), 129-143.

S4-P98.839

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The Pigmentotheque: presentation of a set of geological standards

Chalmin, E.[1], Salomon, H.[1], Chanteraud, C.[1], Develle, A.L.[1], Chassin de Kergommeaux, A.[1,2], Rossi, M.[1], Lemasson, Q.[3,4], Pichon, L.[3,4], Goemaere, E.[5]

[1]Université Savoie Mont Blanc, CNRS, EDYTEM (UMR 5204), Le Bourget du Lac, France
[2]Univ Lyon, ENS de Lyon, Université Lyon 1, CNRS, UMR 5276 LGL-TPE, Lyon, France
[3]New AGLAE FR 3506 – Chimie-Paristech/Ministère de la Culture et de la Communication, Palais du Louvre, Paris, France
[4]Centre de Recherche et de Restauration des Musées de France (C2RMF), Ministère de la Culture, Palais du Louvre, 14 quai F. Mitterrand, 75001, Paris, France
[5]Geological Survey of Belgium, Royal Belgian Institute of Natural Sciences, Rue Vautier, 29, 1000, Brussels, Belgium

Keywords: Colouring matter; Iron-rich rocks; Prehistory; Provenance

The iron oxi(hydroxi)de-rich rocks used during the Prehistory are very various and require a robust geological, structural and geochemical characterization in order to discriminate them and to be able to make relationship with specific geological formation, or even precise outcrops.
The issue of the creation of the « pigmentotheque » is based on the capacity to establish an ID card, specific for each geological deposit or group of deposits. The ferruginous rocks, associated to various geological formations and evolution contexts, present distinctive features at various scales of observation and analysis. To obtain a specific identification for each raw material, a rigorous methodology is required starting from the sampling in the field to the use of complementary and multi-scale methods. According to the nature of the rocks and the archaeological remains, the fingerprints could be then obtained thanks to petrography (at macro-, meso- or micro-scale), mineralogy (nature and association of minerals, crystallinity) and geochemistry (content in major, minor and trace elements).
The most challenging issue of this research is to be able to compare, with the same criteria and comparable results, geological references (without constraints) to divers kinds of archaeological remains (solid rocks and applied pigments). Due to conservational considerations in this case, only little quantities and often weathered are available for analysis. To overcome these methodological limits, we develop a combination of physicochemical measures using our own geological standards (iron-rich rocks). This set of reference samples is currently shared with other teams of research and is increasing with certified geological standards in order to cover the large variability of petrographic nature and geological origin. This set of references, already analyzed with other methods, is used both to fix the analytical parameters and to quantify with high precision the limits of detection. Consequently, this will increase the robustness of the protocol (measurements and sample preparation) and enable the comparison between experiments (pXRF, petrography, PIXE and ICPAES, ICPMS).

S4-P99.840

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Assessing the raw materials utilized in lime production: a case study of plasters and mortars from Kalavasos-Ayios Dhimitrios, Cyprus

Herrick, H.[1], Berna, F.[1]

[1]*Department of Archaeology, Simon Fraser University*

Keywords: lime production; plasters; thin-section petrography; FTIR microscopy; experimental archaeology

The study of ancient lime production has the potential to help better understand the creation of early synthetic materials, as well the beginnings of human impact on environmental processes. However, a consistent protocol for the analysis of lime production and its products has yet to be formally applied. One critical objective in analyzing lime in the archaeological record is the determination of the raw material(s) utilized in its production. The particular consistency and chemistry of the parent material used in lime production may be highly variable, and these factors have the potential to impact the thermodynamic processes of lime production itself. Lime was extensively used in the production of plasters and mortars at the Late Bronze Age (1200 BCE - 500 BCE) archaeological site Kalavasos-Ayios Dhimitrios (K-AD) in southern Cyprus. The materials from K-AD act as a suitable case study to assess the proposed methodology for the analysis of archaeological lime production. Raw materials analysis of a series of these archaeological lime plasters and mortars using a proposed analytical protocol via thin-section petrography and FTIR microscopy will be examined. Further, the role of these results in the assessment of the energetics analysis to predict the scale of lime production at K-AD will be explored.

S4-P101.851

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeological and archaeometric investigation of the marble sculptures of the so-called “Villa of Poppea” (Villa A) at Oplontis (Torre Annunziata, Naples)

Antonelli, F.[1], Lazzarini, L.[1], Pensabene, P.[2]

[1]LAMA - Laboratory for Analysing Materials of Ancient origin, Iuav University of Venice, Italy
[2]Department of Ancient World Studies, Sapienza University of Rome, Italy

Keywords:

The goals of this research is to (i) identify the geographical origin of the white marbles used for the sculptures and for some architectural elements of the Villa A of Oplontis (Torre Annunziata, Naples); (ii) clarify the activity of a Campanian workshop in the villa; (iii) contribute to understand the function of the villa after the earthquake of 62 A.D., when many statues, ninety percent of its marble revetments, and all marble capitals and columns were stripped and stored.
Through standard archaeometric analyses (optical microscopy on thin section, powder X-Ray diffraction and mass-spectrometric determination of the C & O stable isotopic ratios) we identified the different white marbles used for all the sculptures and for four Corinthian capitals as from Carrara, Mount Penteli and the island of Paros. On both this result and their stylistic study, we have hypothesized that some sculptures were imported from Athens and finished (in situ?) by a Campanian workshop, some others were instead fully carved in Campania and were acquired through local mercatores.
After the 62 A.D. earthquake all the architectural decoration and statuary were restored, but at that time not all these items were completed: almost any of them was replaced at the moment of the Vesuvius’ eruption.

S4-P111.3008

Characterization, procurement and transformation of iron-rich raw material during Early Neolithic in North-western Europe

Goemaere, E.[1], Chassin de Kergommeaux, A.[2, 4], Blouet, V.[3], Chalmin, C.[4], Goffioul, C.[5], Goovaerts, T.[1], Jadin J.[6], Jungels, C.[7], Leduc T.[1], Lemasson Q.[8,9], Salomon, H.[4], Tromme, F.[10]

[1]Geological Survey of Belgium, Royal Belgian Institute of Natural Sciences, Rue Vautier, 29, 1000, Brussels, Belgium
[2]Univ Lyon, ENS de Lyon, Université Lyon 1, CNRS, UMR 5276 LGL-TPE, F-69364, Lyon, France
[3]SRA Grand Est - Service régional de l'archéologie du Grand Est, France
[4]EDYTEM (UMR 5204), Univ. Savoie Mont Blanc - CNRS, 5 bd de la mer Caspienne, 73376, Le Bourget du Lac Cedex, France
[5]Service de l'Archéologie de la Direction de Liège, AWAP, Avenue des tilleuls, 62, 4000 Liège, Belgium
[6]Anthropology & Prehistory, Royal Belgian Institute of Natural Sciences
[7]Préhistomuseum, Rue de la Grotte, 128, 4400 Flémalle, Belgium
[8]Centre de Recherche et de Restauration des Musées de France (C2RMF), Ministère de la Culture, Palais du Louvre, 14 quai F. Mitterrand, 75001, Paris, France
[9]New AGLAE FR 3506 – Chimie-Paristech/Ministère de la Culture et de la Communication, Palais du Louvre,14 quai François Mitterand, 75001 Paris, France
[10]CETREP, Les Chercheurs de la Wallonie, Flémalle, Belgium

Keywords: haematite; oolithic ironstones; Early Neolithic; sourcing

Haematite crystal and (red to black) haematite rich stones (sedimentary, metamorphic or weathered rocks), with high red colouring strength have been intensively used throughout prehistoric times. In North-western Europe, at the beginning of the Neolithic, oolithic ironstones (OIS) were commonly used. Even if, red OIS are absent in all the LBK (Linear Band Keramik) settlements of the French Lorraine (Moselle valley and tributaries), this early exploitation is documented specially in Belgium (Hesbaye and Dendre springs areas), the Netherlands (Maastricht area) and France (Normandy). First results dedicated to the LBK OIS from Hesbaye, the Netherlands and Normandy were published earlier as part of a crossborder research project launched in 2010. But a (wide) variety of non-oolithic black-red stones are also found besides the OIS and/or including fired iron hydroxides and burned carbonate concretions, often in lower quantities. The « red » or black-red material frequently shows wear traces of abrasion (flattened surfaces and facetted pieces showing sometimes striations). In all sites, smaller angular fragments with no clear evidence of use are also found. Some of the iron-rich material from Belgium and Lorraine has a common geological origin. In some places, grindstone, which are reused broken millstones made out of various sandstones, were found covered by a thin layer of red powder. This research is focused on a) the characterization of all types of red artefacts found in Hesbaye and French Lorraine through studies at different scales from macroscopic to elemental thanks to petrological and physico-chemical analyses (Optical Microscopy in thin-sections, Scanning Electronic Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS) and Particule Induced X-Ray Emission (PIXE), Magnetic Susceptibility); b) the relationship between the red powder sampled from the grindstones and the artefacts ; c) the determination of the geological and geographical origin of the haematitic items through the identification of fingerprints ; d) the methods of red powder production, especially the firing of iron-sulphides, goethite-rich sediments and iron-carbonates concretions and e) the common material between the Hesbaye and Lorraine areas. The conclusion will draw a synthesis of our current knowledge on the exploitation, transformation and supply strategies of iron-rich material during Ancient Neolithic in North-western Europe.

S4-P113.564

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Session 5 Technology/provenance - ceramics/vitreous/glass

ORAL

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Continuity and change in indigenous ceramic recipes in the Lesser Antilles across the Historical Divide

Stienaers, A.[1], Degryse, P.[1], Hofman, C.[2]

[1]KU Leuven and University of Leiden, Celestijnenlaan 200E, 3001 Leuven and Einsteinweg 2, 2333 Leiden
[2]University of Leiden, Einsteinweg 2, 2333 Leiden

Keywords: Ceramics; Lesser Antilles; Petrography; Geochemistry

Few region-wide archaeometric studies on indigenous Caribbean ceramics have been published to date. This study aids in bridging this knowledge gap by considering ceramic and clay samples from eleven islands all across the Lesser Antilles from both the pre-colonial and early colonial period. 351 samples of indigenous ceramics from Trinidad, Barbados, Grenada, St. Vincent, St. Lucia, Guadeloupe, St. Kitts, Antigua, Saba, St. Eustatius and St. Martin from both the pre-colonial and the early colonial period and 242 local clay samples are characterised using a combination of thin section petrography and geochemistry (XRF and ICP-OES). Despite great optical and chemical similarities resulting from a similar local geology, the authors have succeeded in isolating 25 meaningful fabric groups for which provenancing hypotheses have subsequently been postulated with varying degrees of certainty. The results not only show that important exchange networks existed between many of these islands but also that the indigenous paste recipes display a surprising consistency through time, surviving both the Kalinago and Colonial ‘migration waves’.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Using elemental chemistry and strontium isotopes as tracers for fresh-water shell-tempered ceramics

Renson, V.[1], Peacock, E.[2], Kirkland, B.[3], Sherman, S.P.III [4]

[1]Archaeometry Laboratory, Research Reactor Center, University of Missouri, Columbia, MO 65211, USA.
[2]Department of Anthropology and Middle Eastern Cultures, Mississippi State University, Mississippi State, MS 39762.
[3]Department of Geosciences, Mississippi State University, Mississippi State, MS 39762.
[4]Department of Earth Sciences, University of Memphis, TN 38152

Keywords: shells; ceramics; strontium isotopes; elemental chemistry

The present research consists of an evaluation of elemental and strontium isotope analysis as tracers to identify shell-tempered ceramic circulation and exchange. Recent pilot studies showed that freshwater mussel shells found at various archaeological sites along different drainages have different elemental compositions. Here, elemental analyses are combined with strontium isotopic analyses. Both methods are applied to whole freshwater shells and shell temper fragments extracted from shell—tempered ceramics. A total of 240 samples were selected from twelve sites located in or adjacent to the Yazoo Basin (Mississippi, USA). The sites represent multiple locations in multiple drainages and date from the Late Woodland and/or the Mississippian period. Both methodological approach and results are presented, and the importance of evaluating the material for diagenesis prior to interpretation of geochemical data is discussed. We then evaluate which elements are discriminants in terms of provenance. We present the variability of elemental chemistry and strontium isotopic signature between the different sites across the Yazoo Basin and adjacent regions, and the comparison of geochemical signatures of shell temper and whole shells. We finally present variations of elemental chemistry between whole shells and shell-temper from the same site and discuss the possibility of chemical exchanges between the clay and the shell temper.

S5-O02.238

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A local or imported production? Study of the Ychsma ceramic from the site of Armatambo, Lima Peru

Dante, P.[1,2,3], Javier, I.[2], Rémy, Ch.[1], Luisa, D.[3], Gorka, A.[4], Ayed, B.[1], Nadia, C.[1]

[1]IRAMAT-CRP2A, UMR 5060 CNRS - Université Bordeaux Montaigne, Maison de l'Archéologie, Domaine Universitaire, Esplanade des Antilles, 33607 Pessac Cedex, France
[2]GPAC, C. I. Micaela Portilla, Universidad del País Vasco (UPV/EHU), c/ Justo Vélez de Elorriaga, 1, Vitoria-Gasteiz 01006, País Vasco – ESPAÑA
[3]Grupo de Investigación Sociedades Prehispanicas del Litoral–Yungas, Departamento Académico de Arqueología, Universidad Nacional Mayor de San Marcos, Facultad de Ciencias Sociales, Avenida Carlos Germán Amezaga 375 Cercado de Lima, PERU
[4]IBeA dpt. Química Analítica, Facultad de Ciencia y Tecnología, Leioa, Universidad del País Vasco (UPV/EHU)

Keywords: Ceramic; Ychsma; ICP_MS; Petrography; Geology; Peru

The Ychsma society developed in the Central Coast of Peru from 900 AD to 1532 AD, occupying the area corresponding to the lower region of the Rimac Valley and the Lurin Valley, in the current department of Lima (Peru). This society was organised into chiefdoms that controlled large agricultural areas. This social and economic system came to an end with the arrival of the Spanish in 1532.

Our project addresses the study of the provenance of the raw material, the archaeological site of Armatambo, one of the main sites associated with the Ychsma society. This site was inhabited between the Middle Ychsma period (1250-1350 AD) and Late Ychsma (1350-1530 AD). This major archaeological site has constituted a geographical and cultural space of communication and exchange between the various coastal sites associated with the Ychsma society. Unfortunately, nowadays the rapid urban development in the area has vastly affected this important archaeological site.

Our research seeks to determine whether there was a local production of pottery at Armatambo or whether this site was a recipient of vessels from other production centres. For this purpose, we have worked with 62 samples of ceramics corresponding to three typologies: jars, pots and pitchers, from funerary contexts. During our work, we adopted a multi-analytical approach using mineralogical and chemical techniques. The petrographic study of Ychsma ceramics allowed us to identify four petrographic groups in the Armatambo area. On the other hand, the chemical analysis of the ceramic samples by ICP-MS, in addition to the local clays, allow us to determine the relationship of the ceramic productions of the samples. The results obtained from petrography and ICP-MS were contrasted with archaeological and geological data from the region, allowing us to propose two possible areas of raw material supply during the Middle Ychsma and Late Ychsma. This allows us to propose as a hypothesis that there was a local production at Armatambo, however, this site received vessels from other areas of the Rímac Valley.

S5-O03.547

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

See through glass: Iron Age and Roman glass in northwestern Portugal

Wade, M.[1], Dussubieux, L.[2]

[1]Department of Anthropology - University of Texas at Austin
[2]Elemental Analysis Facility – Chicago Field Museum

Keywords: Glass; Interactions; Iron Age; Roman

The analytical opacity glass from primary workshops brings to the analysis of glass products from secondary workshops renders interpretation of place of manufacture of glass objects and trade connections difficult. Previous analysis of fifty-three glass fragments from archaeological excavations and subsurface collections, conducted by the Elemental Analysis Facility at the Chicago Field Museum of using Laser ablation - Inductively Coupled Plasma-Mass Spectrometry, revealed three compositional groups and some overlap between groups. Those results obtained from glass materials from the Roman town of *Bracara Augusta*, Roman villas as well as from five Iron Age hillforts, all archaeological sites in northwestern Portugal. The number of samples from the hillforts was considerably smaller than that from *Bracara Augusta*. This paper discusses the analytical results from a larger set of samples obtained from the same Iron Age hillforts as well as from three other hillforts and considers the present results in relation to the earlier analytical work.
The aim of this study is to explore the interactions between hillforts in northwestern Portugal and possible recycling practices.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric study of glass working indicators of altare (Northern Italy) and other Ligurian contexts in the Middle Ages

Bagnasco M.[1], Ferrero D.[2], Varaldo C.[3], Riccardi M.P.[4,5]

[1]Archeologist (collaborator of University of Genoa)
[2]Historian (collaborator of University of Genoa)
[3]DAFIST - University of Genoa, Via Balbi 2, 16126 Genoa, Italy
[4]DiSTA - University of Pavia, via Ferrata 9, I27100 Pavia, Italy;
[5]Arvedi Laboratory, University of Pavia, via Ferrata 9, I27100 Pavia, Italy

Keywords: Altare; Monte Lecco; Savona; medieval glass; chemical and geochemical composition.

The archaeological research carried out in Altare (Lascito Balestra, years 2017/2018) has allowed to identify a stratification from today to the Middle Ages. The archaeological levels concerning the various periods have highlighted a large number of indicators of glass processing such as fragments of crucibles with and without glass, drops, collars, clippings and fragments of finished glass. This archaeometric study was conducted with the aim of defining the chemical and geochemical composition of “Altarese glass” in the Middle Ages and to compare its composition with other contemporary ligurian sites of glass production (Monte Lecco glasshouse) and contexts of use, such as the city of Savona.
Findings have been studied at a macroscopic level to describe their shape and, when possible, their function. Some archaeological finds have been prepared for analysis following standard metallographic procedures for microstructural and microchemical examination. The microstructural and chemical analyses were performed using the petrographic optical microscope (OM) and the scanning electron microscope (SEM) equipped with EDX system. Geochemical data (trace and minor element) were performed by LA ICP MS.
In the Altarese production context, different types of glass were processed: Na-Ca glass and K-Ca glass. The fluxes were mainly derived from the ashes of the plants. The chemical and geochemical composition of the glass produced in Altare overlaps with that of the glass working indicators of Monte Lecco glasshouse. Even the minor and traces elements do not distinguish the two productions, except for the contents of Rb and Sr. Based on the content of Rb and Sr, the glass finds from Savona seem more similar to the glass of the Altarese production context than to the production of the Monte Lecco glasshouse. The mobility of the Altaresi glassmakers makes this reseach really significant for the entire European panorama of glass studies.

S5-O05.549

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Petrographic investigation on exotic, imitation and traditional ceramic productions from the Forum of Cumae (southern Italy)

Izzo, F.[1,2], Guarino, V.[1], Ciotola, A.[3], Verde, M.[1], De Bonis, V.[1,2], Morra, V.[1,2]

[1]DiSTAR, University of Naples Federico II, Via Cintia, 21, 80126 Napoli, Italy
[2]Center of Research on Archaeometry and Conservation Science, Via Cintia, 21, 80126 Napoli, Italy
[3]Dipartimento di Studi Umanistici, University of Naples Federico II, Via Porta di Massa, 1, 80133 Napoli, Italy

Keywords: Internal Red-Slip; African imitations; Aegean pottery; Cumae; volcanic sands.

Fifty ceramic sherds of Roman Coarse Ware collected from the Forum of Cumae have been characterized from a mineralogical and petrographic point of views in order to define the provenance of geological raw materials and the production technologies.
As a whole, the examined samples can be gathered in several petrographic groups referred both to exotic and local ceramic productions.
In this frame, Internal Red-Slip cooking ware (also known as *Pompeian red ware*) can be considered one of the most representative local ceramic production since manufactured using a low-CaO clays (generally ≤ 6%wt.) mixed to tempers compatible with the volcanic sands from the Bay of Naples. Nevertheless, some differences could be noticed as a function of their chronology. In particular, the older samples (dated between the end of the 1st century BC and the begin of the 1st century AD) show in thin section a birefringent matrix and bimodal distributed grains mainly formed by crystals of clinopyroxenes, alkali feldspars and leucite/plagioclase-bearing scoriae fragments along with minor chamotte, rare crystals of garnet, amphibole and very rare olivine. The occurrence of hematite and the presence of residual phyllosilicates suggest for this potteries a firing temperature probably above 800°C but not over the 950°C, in oxidizing atmosphere. On the other hand, the a-plastic inclusions of the late samples (dated from the 1st to the 4th centuries AD) appear enriched in quartz and feldspars in the place of volcanic components, along with carbonate fragments thermally decomposed (calcite *"ghosts"*). Raman and FTIR analyses performed on the red coatings reveal the presence of hematite and phyllosilicates.
A significant number of table and cooking wares also displays the typical volcanic grains of local geological source even if mixed to not negligible sedimentary intakes from the near Volturno river.
It's also noteworthy the occurrence of common wares imitating African potteries but produced using temper locally supplied.
Lastly, part of the analyzed samples was classified as outliers namely with peculiar textural and compositional features. In some cases, the presence of granitic fragments or rhyolitic glasses define them as exotic (probably Aegean).
These results shed new light about production and circulation of Coarse Wares in Cumae from the 2nd century BC to the late Antiquity, highlighting the important role of this city in pottery production during the past.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometry of Medieval Pottery from Basilicata

Annunziata, E.M.[1], Di Leo, P.[2,3], Sogliani, F[2], Medici, L.[3], Lettino, A.[3]

[1]PhD fellow, DiCEM – Department of European and Mediterranean Cultures, University of Basilicata, Matera, Italy
[2]School of Specialization in Archaeological Heritage, SSBA DiCEM – Department of European and Mediterranean Cultures, University of Basilicata, Matera, Italy
[3]Institute of Methodologies for Environmental Analysis - National Research Council, Tito Scalo (PZ), Italy

Keywords: medieval pottery; archaeometry; Basilicata;

The present work is aimed to delineate the production and circulation system of pottery products in Basilicata during the Middle Age. The research is an integral part of the CHORA project - Archeology Laboratories in Basilicata, funded to the School of Specialization in Archaeological Heritage, University of Basilicata, Matera (MT). By understanding the economic systems through the analysis of raw material/finished products circulation and integrating this information with climate and landscape evolution, it will be possible to decipher dynamics of lucanian medieval sites in the diachrony. The present study focuses on archaeometric analyses of the medieval pottery from the excavation contexts of S. Maria d'Anglona, Moliterno and Satrianum. Samples of natural clays outcropping in the area surrounding the archaeological sites were also analysed and subjected to experimental *firing tests*. The work systematically addresses the issue of the production and circulation of Middle Ages ceramic artefacts in the regional context of Basilicata using an interdisciplinary approach that implies the integration of the archaeometry to the study of archaeological finds. The systematic study was conducted on about 100 fragments of pottery from three distinct medieval Lucanian archaeological sites, *i.e.* Satrianum, the Castle of Moliterno and Santa Maria d'Anglona. Tempers and coatings of the artifacts were compositionally characterized using conventional and destructive/non-destructive analytical techniques and compared with clay rich deposits outcropping in Basilicata - as possible raw materials - and with firing tests. The archaeometric approach has made it possible to return an articulated picture of the presence of productions in the regional area, that during the Middle Ages circulated on a geographical scale, including extra-regional ones, thanks to a terrestrial and river communication network. The integration of the results of the archaeometric analyzes, of the firing tests, the geolocation of the sampled outcrops and the identification of the geographical relationships with the archaeological sites made it possible to circumscribe any local productions, define the circulation of raw materials and know-how in the diachrony in a territory, such as that of Lucania, rich in archaeological evidence, as well as densely man-made from prehistoric times to the Middle Ages.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Movement of people and goods: Production and circulation of amphorae in Pre-Roman Adriatic

Mise, M.[1], Quinn, S. P.[1], Maritan, L.[2]

[1]UCL Institute of Archaeology
[2]Department of Geosciences, University of Padova

Keywords: Amphorae; compositional analysis; Optical Microscopy; NAA; Adriatic

During the recent years the studies of economy of the Adriatic region during the Pre-Roman period, via the distribution of amphorae and trade in wine and olive oil were focus of our investigation. The compositional characterization of transport amphorae from 15 different sites along the Eastern Adriatic coast was conducted with think section optical microscopy at the UCL Institute of Archaeology and Neutron activation Analysis at the Missouri University Research Reactor. The analysis include the most common types of amphorae that circulated in the Adriatic-Ionian region; Corinthian A' and type B, Graeco-Italic and Lamboglia 2 amphorae from Greek and Iron Age settlements, and shipwrecks. Our study was focused on the period between the establishments of Greek settlements in the 4th c. BC on the Eastern Adriatic coast until the consolidation of Roman power toward the end of the last millennium BC in the region. The aim of our research was to identify the movement of amphorae from productions sites via ships to consumptions sites and to reconstruct the trade routes and patterns of the ancient Adriatic economy.

The result of comprehensive project have shown not only trade patterns between western and eastern Adriatic coast and Ionian, but also the changes in the trade patterns due to different economic policies in the last centuries BC, the diversity of the market of the Hellenistic states and rise of the single market with the Roman political and economic expansion in the region.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Rethinking technological choices in industrial ceramic production: comparing sugar pots manufacture in the western Mediterranean in the medieval and post-medieval periods

Mentesana, R.[1], Buxeda i Garrigòs, J.[1], Kilikoglou, V.[2], Hein, A.[2], Madrid i Fernàndez, M.[1]

[1]Dept. d'Història i Arqueologia, Facultat de Geografia i Història, Universitat de Barcelona, Spain
[2]Institute of Nanoscience and Nanotechnology, N.C.S.R. "Demokritos", Greece

Keywords: ceramics; technology; Sicily; Spain

What impinges craft production, especially when it is aimed at the industry? What creates a manufacturing standard, a model to follow, and when does it happen? How the standard is followed, transmitted and transformed across time and space? These are the questions addressed during this paper, taking as a case study the manufacture of *sugar pots* in the 11th-16th centuries in Sicily and Spain.

Sugar pots are specialized ceramic vessels, used in the last phase of sugar production when the liquid syrup is left to crystallize. These vessels require to be produced in high quantity and following specific technical requirements in terms of shape, volume and thermal and mechanical features to absolve. Across the western Mediterranean regions and over phases, there are glimpses of following some standards in these vessels' manufacture but there has not been further study assessing these features in order to see technical knowledge movement, transfer or change.

For this paper, technological and morphological features of sugar pots from several sites in Sicily (Palermo, Partinico, Himera) and Spain (Gandia, Oliva, Paterna, Barcelona) will be shown. 200 samples from these sites have been assessed aided by instrumental analysis (Petrography, SEM, XRF, XRD). Sugar pots manufacturing strategies from these sites are compared in order to see their continuities or change across sites and phases.

Preliminary results show that there seem to be general ideas about sugar pots manufacture circulating across the western Mediterranean, probably linked to the movement of people connected to the sugar industry as well as some expectations from sugar producers. However, this was far from following manufacturing standards from both technological and morphological points of view; rather, in each context we observe a rather local re-interpretation of common and generic requirements. This study will offer the opportunity to reflect on how local craftspeople adapt or transformed their ceramic making traditions in order to face a *global* product demands, in this case from the sugar production industry in the Mediterranean.

S5-O09.789

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Glass beads, rings and pendants. Archaeometric insights into an 18th century commercial context in downtown Lisbon

Coutinho, I.[1,2], C. Lopes, L.[3], Filipe, V.[4], Henriques, J.P.[4], Casimiro, T.M.[5]

[1]Department of Conservation and Restoration, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[2]Research Unit VICARTE, Vidro e Cerâmica para as Artes, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[3]C2TN, Instituto Superior Técnico, Universidade de Lisboa, 2695-066 Bodadela LRS, Portugal
[4]COTA80.86 (Archaeology Company), Rua Alves Torgo 16b Lisboa, Portugal
[5] IHC/AIP NOVA FCSH Avenida de Berna 26 C 1969-061 Lisbon, Portugal

Keywords: Glass Beads and Rings; Archaeometry; Portugal; Modern Period; Trading relations

Beads can be considered one of the oldest traded materials in the world. Considering the European case, it was in the 17th and 18th centuries when the trade of glass beads gained considerable importance. In the present work a set of glass beads and rings dated to the 18th century and unearthed in Lisbon were studied and chemically characterized by µ-PIXE.

An archaeological excavation in the corner of Rua de São Paulo and Rua Nova do Carvalho in downtown Lisbon uncovered a unique archaeological context, dated from mid 18th century and possible destroyed in 1755. Thousands of glass beads from different sizes and colours, glass rings and pendants were found, in association with large quantities of other objects such as flintlock stones and metal buckles. The excavation of the site revealed that these artefacts were kept inside bags and wooden boxes. The location of the site, close to the riverfront and the storage of these artefacts, suggests that this site was most likely a warehouse, although archaeology has not been able to confirm if these artefacts were entering or leaving the city.

It is historically known that Venice and the Bohemian region were among the main producers of beads that were being traded across the Atlantic and Pacific oceans. Moreover, at this time, the variety of sizes, shapes and decorations were immense. This is the first systematic study of a set of beads and rings that were found inside a possible storage in Lisbon. The first approach to study the beads set was to categorize them according to their sizes, shapes and decorations. Two different production methods were identified among the beads under study: drawn beads and lamp-wound beads. After this first contact with the objects, a selection of 22 beads and 6 rings was characterized by µ-PIXE in order to try to attribute a provenance. The study of this set is of utmost importance, since, as far as the authors now, no glass bead production was ever found or even reported in the literature in Portugal, which leaves up the following questions and hypotheses: have the beads just arrived from out sea or were these ready to leave Portugal? If the beads were leaving the country, what was their provenance and where were they heading to? Finally, if the beads were entering Portugal, besides its provenance, was this their final destination?

The studies on glass beads from this chronology have experienced a growing interest mainly because these represent trading practices from the European countries with their colonies in a period of European mass expansion.

To sum up, the particular time capsule situation of the archaeological context from where the glass beads and rings were retrieved makes the study of this set of great importance to bring more data on the Portuguese commercial relations with European and colonial countries.

S5-O10.239

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of glass beads from Nima Ohtsuka Kofun (Okayama Prefecture): a contribution to the scientific knowledge of Japanese archaeological glass

Gulmini, M.[1], Davit, P.[1], Croveri, P.[1], Poli, T.[1], Re, A.[2,3], Guidorzi, L.[2,3], Lo Giudice, A.[2,3], Angelici, D.[4], Nozaka, T.[5], Matsumoto, N.[6]

[1]Department of Chemistry – University of Torino – via Giuria, 7 – 10125 Torino, Italy
[2]Department of Physics - University of Torino via Giuria, 1 – 10125 Torino, Italy
[3]INFN – Sezione di Torino – via Giuria, 1 – 10125 Torino, Italy
[4]Tecnart s.r.l – via Modena, 58 – 10124 Torino, Italy
[5]Department of Earth Sciences - Okayama University – 3-1-1 Tsushima-naka, Kita-ku, Okayama 700-8530, Japan
[6]Department of Archaeology - Okayama University – 3-1-1 Tsushima-naka, Kita-ku, Okayama 700-8530, Japan

Keywords: Glass; Beads; Kofun; Japan

Nima Ohtsuka kofun is a keyhole-shape burial mound (kofun) in Kurashiki area (Okayama prefecture, Japan). The kofun dates back to the mid-6th Century CE and extends for 38 m in length and some 5.5 m in height. The soil building up the mound covers a stone structure which leads to the burial chamber (some 4.70 m long, 2.50 m wide and 2.50 m high) through a passage opening on the South side of the round portion of the mound [1]. The several fragments found on the mound testify the original presence of haniwa (pottery cylinders and figures) and Sue pottery, whereas numerous beads and other artefacts were found in the stone chamber. Beads were made of silver, jasper, crystalline quartz, amber, soapstone, clay and, as the large majority, glass. Glass beads included five eye-beads (tombodama), two round beads (about 16 mm in diameter) and 1,103 small beads (about 4 mm in diameter) coloured red, orange, yellow, green (two shades) and blue (two shades) [1].

Sixty-eight glass beads have been considered in this work for non-invasive investigation with optical microscopy (OM), Fiber Optics Reflectance Spectroscopy (FORS) and X-ray fluorescence spectroscopy (XRF) in order to recognize glass type, colourants, opacifiers and production techniques. In addition, nine micro-fragments (mounted as polished sections) were analysed by SEM-EDS to obtain information on the composition of the glass and details on opacifiers.

OM showed that the beads were mainly obtained by cutting a glass tube, with evident stripes of opacifier in red and green beads. Data from the non-invasive investigation indicated that the blue colour is related to Co(II) in the tombodama, whereas both Cu(II) and Fe(II) play a role in the final colour of other blue and green beads. Lead stannate gives colour (and opacity) to the yellow beads and Cu and/or Cu2O accounts for the red colour of the few red items. SEM-EDS revealed that the considered samples are “high magnesium glass” (HMG). Opaque green and yellow glass also contain small (< 10 micron) particles of lead stannate, quartz, feldspar and zircon. The overall results are considered in this work within the frame of already published data on glass beads from the Yayoi and Kofun periods [2 and refs therein].

[1] I. Niiro and N. Miura (Eds) Nima Ohtsuka Kofun. Excavation of a 6th century burial mound in Okayama Prefecture, Excavation team of Nima Ohtsuka Kofun, Okayama, 2018
[2] T. Tamura, K. Oga (2016). Microchem. J., 126, 7-17.

S5-O11.262

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Analytical evidence for a very early glassmaking workshop in Egypt

Shortland, A.J.[1], Brownscombe, W.[2], Kemp, T.[1]

[1]Cranfield University, Defence Academy of the UK, Shrivenham, Wilts, UK
[2]Natural History Museum, Exhibition Road, London, UK.

Keywords: Glass; Egypt; Late Bronze Age; Amenhotep II; LA-ICPMS

Glassmaking workshops are rare in the archaeological record and especially rare in the Late Bronze Age when glass was first regularly produced. The earliest glassmaking sites known are both in Egypt, at Amarna and Malkalta. The city of Amarna can be closely dated, as it is recorded that building commenced there in the 5th year of the reign of the Egyptian King Akhenaten, probably 1347 BC. Malkata was the Theban residence of his father, Amenhotep III (1390-1352 BC), so might date to as much as 40 years or so earlier. However, significant finds of glass have been recovered in Egypt associated with at least the three previous kings: Tuthmosis III, Amenhotep II and Tuthmosis IV. Where this glass might have been made is not known, but it has often been linked to gifts from Near Eastern rulers – so the glass might have been an import from Mesopotamia.

This paper presents evidence from the LA-ICPMS analysis of glass securely dated to the reign of Amenhotep II - either associated with his cartouche or with his tomb, KV35, in the Valley of the Kings. Previous work has shown that LBA glass made in Egypt and Mesopotamia are compositionally distinct, and combining existing and new LA-ICPMS data, along with some new interpretations of longstanding EPMA data, shows that this early glass comes from more than one workshop. In addition, the glass from KV35 is stylistically unusual and the paper draws on art historical interpretations as well as ancient texts to interpret the results. It shows that it is very likely that there was a significant production of glass in Egypt, in a range of colours, at least as early as the reigns of Tuthmosis III and Amenhotep II, i.e. the last quarter of the fifteenth century BC. In addition, glass of Mesopotamian composition was also present, and very likely being brought to the Egyptian court, perhaps in connection with Royal gifts to the King. Thus this early period shows a very interesting case where individual objects might be able to be interpreted as royal gifts brought from abroad to the Egyptian court.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Tracking glass production in India: elemental and isotopic analysis of raw materials

Dussubieux, L.[1], Fenn, T.R.[2], Abraham, S.A.[3], Kanungo, A.K.[4]

[1]Field Museum, Chicago, USA
[2]University of Oklahoma, USA
[3]Saint Lawrence University, USA
[4]Indian Institute of Technology Gandhinagar, India

Keywords: Glass; India; raw material; isotope

The scarcity or absence of written documents dealing with ancient glass technology and of archaeological evidence of primary glass manufacturing workshops in India create difficulties in understanding the organization of the ancient glass industries in India that produced, starting around the mid-1st millennium BCE, huge quantities of small drawn beads that were traded locally, but also all over the Indian Ocean and beyond as indicated by the recent discovery of Indian drawn beads in Merovingian sepultures in France. Elemental compositions conducted on glass beads found in India or on Indian beads found elsewhere show a great variability that could be linked in some cases to different production regions (Northeastern, South and Western India); however, a more precise provenance attribution and the distinction of several possible production centers in each regions was not possible without additional research. In India, ethnographic evidence shows that glass was often produced from one single ingredient, called generally reh, containing immature sand with high alumina concentrations and moderate lime and potash quantities and a sodic efflorescence able to produce a vitreous material when heated in a traditional furnace. Assuming that each glass center would procure such an ingredient from a nearby source, the diversity of the geology of India is providing the possibility of characterizing each production center with a unique isotopic signature that will be identical in the raw materials and the finished glass. We collected reh samples from different regions in India for which evidence of glass making in the recent past, and/or in the more distant past was available. This presentation will report on the elemental composition obtained using laser ablation – inductively coupled plasma - mass spectrometry and the isotopic composition (Pb, Sr and Nd) of these reh samples and the results will be compared to data obtained from glass. Our results are showing that some regions are more likely than other to have been places of glass production in ancient time.

S5-O13.495

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

XRF application on faience cylinder seals towards unveiling the flow of materials and ideas

Karydas, A.G.[1], Kaparou, M.[1], Asvestas, A.[2], Tsampa, K.[1], Kladouri, N.K.[1,3], Anagnostopoulos, D.[2], Tsouparopoulou, Chr.[4,5]

[1]*Institute of Nuclear and Particle Physics, NCSR Demokritos, P. Grigoriou and Neapoleos, Agia Paraskevi, Attiki, Greece*
[2]*Department of Materials Science and Engineering, School of Engineering, University of Ioannina,45110, Greece*
[3]*Department of History, Archaeology and Cultural Resources Management, University of the Peloponnese, 24133, Kalamata, Greece*
[4]*McDonald Institute for Archaeological Research, University of Cambridge, UK*
[5]Academy of Sciences, Warsaw, Poland

Keywords: vitreous materials; faience; cylinder seals; micro- XRF; handheld XRF; LBA

The study of commonplace cylinder seals of Late Bronze Age Western Eurasia can offer new insights into non-élite populations, rendering possible to map the flows of materials and ideas in the Late Bronze Age. Literature review pinpointed the occurrence of such faience cylinder seals of a Syro-Mesopotamian style in different archaeological contexts in the Greek mainland. Thus, samples from the Archaeological Museums of Lamia (3), Thiva (4), Chalkis (2), Volos (2), Nemea (2), Patras (4), Isthmia (1) and the National Archaeological Museum (1) were investigated. For reasons of comparison and in an attempt to extract valuable information regarding likely provenance and technology, 4 scarabs of the Late Bronze Age and of similar archaeological contexts from the Nemea Archaeological Museum, registered as faience in the archaeological record, were also incorporated in the analytical campaign.

Towards the aim of the study and with the restrictions posed by the in-situ and non- destructive character of the study, two portable X-ray Fluorescence (XRF) spectrometers were employed, a micro-XRF spectrometer and a handheld-XRF analyzer. The analysis of the spectra was conducted through PyMCA open source software, whereas the quantification was conducted by custom developed calibration methodologies adapted to suit best for the specifics of both instruments. The weathering state of the artifacts, in cases manifested as severe degradation, was a factor that compromised the validity of the quantitative results in some cases. Thus, the combination of quantitative, qualitative, macroscopic and archaeological data offered important insights into technological aspects regarding the glass network towards the formation of the desired glaze, colorants and opacifiers. Specific artistic choices were also revealed with a number of samples having been most probably covered with golden leafs, as suggested both by microphotography and chemical results. Interestingly, for the samples (scarabs) from the Nemea assemblage the analytical results clearly suggested that they were made of steatite with the superimposition of a glazed layer.

The current study aspires to shed light on important aspects of our understanding with regard to the technology and likely movement of a ‘low cost’ material, accessible by all and widely circulated fully charged with cultural ideas.

References: Ameri**,** M., Kielt Costello, S., Jamison, G. Jarmer Scott, S. 2018. Seals and Sealing in the Ancient World, Case Studies from the Near East, Egypt, the Aegean, and South Asia, Cambridge University Press.DOI: https://doi.org/10.1017/9781108160186.028,

Acknowledgements: Research was funded by the EC project PLOMAT Grant nos.748293 and no. 951328. We thank the Archaeological Museums of Lamia, Thiva, Chalkis, Volos, Nemea, Patras, Isthmia and the National Archaeological Museum, as well as Dr Kaza-Papageorgiou

S5-O14.1543

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A new isotope tracer for natron glass

Barfod, G.H.[1,2], Freestone, I.C.[3], Lesher, C.E.[1,2], Lichtenberger, A.[4], Raja, R.[2,5]

[1]Aarhus Geochemistry and Isotope Research (AGiR) Platform, Geoscience, Aarhus University, Aarhus, Denmark
[2]The Danish National Research Foundation's Centre of Excellence for Urban Network Evolutions, Aarhus University, Denmark
[3]Institute of Archaeology, London's Global University UCL, London, United Kingdom
[4]Institut für Klassische Archäologie und Christliche Archäologie, Münster University, Münster, Germany
[5]School of Culture and Society, Aarhus University, Aarhus, Denmark

Keywords: natron glass, new isotope system, Egypt, Levant

During Roman times, colourless glass was produced at factories in the coastal regions of Syro-Palestine by addition of manganese, whereas the production site of the more desirable antimony-decolourized glass has remained an enigma. Antimony-decoloured glasses share many chemical features with the Egyptian natron glass type Foy 2.1 implying an Egyptian origin, while at the same time the relatively low titanium content points to use of sands from Syro-Palestine in their production. Provenance studies using strontium (Sr) and neodymium (Nd) isotopes place their production along the coast of the eastern Mediterranean, but do not distinguish unambiguously between glasses from the two regions. Here, we present the results of applying the hafnium (Hf) isotope system to natron glass types recovered from the ancient city of Gerasa in Northern Jordan. The results place antimony-decolorized glass production in Egypt and show distinctly different isotope compositions for Egyptian versus Levantine natron glass types. We attribute this to systematic sorting of zircons within the Nile sediments used in glass production during longshore drift and aeolian transport along the south-eastern Mediterranean coast. These results suggest that the addition of Hf to the current isotopic repertoire significantly extends provenancing capabilities and should find wide application in the archaeometric investigation of silicate-based melts and materials.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The invention of lead glass in al-Andalus

Schibille, N.[1], De Juan Ares, J.[1], Casal García, M.T.[2], Zamorano, A.M.[3], Blet-Lemarquand, M.[1], Tereygeol, F.[4]

[1]IRAMAT-CEB, CNRS, Université d'Orléans, 3D rue de la Ferollerie, Orléans 45071, France
[2]CCHS, CSIC, Albasanz 26 – 28, Madrid 28037, Spain
[3]Universidad de Sevilla, Doña María de Padilla, S/N, 41004 - Sevilla
[4]IRAMAT-LMC, LAPA, NIMBE, CEA, CNRS, Université Paris-Saclay, CEA Saclay, Gif-sur-Yvette 91191, France

Keywords: soda-ash lead glass; lead isotopes; chlorine; Islamic glass

Different types of high lead glasses appeared in the ninth to tenth century CE across Europe, including lead silica glasses in the strict sense with very high lead (PbO ≥ 60%) and silica as the main constituents, as well as potash lead (several variants) and soda-ash lead glasses with lower lead oxide contents (PbO typically between about 20% and 60%) and considerable amounts of alkali and alkaline earth elements.* Since their identification (Duckworth *et al.*, 2015), Islamic lead and soda-ash lead glasses have turned up in the archaeological record throughout the Iberian Peninsula, dating to the eighth to twelfth century CE. They constitute for instance 70% of the tenth-century glass assemblage from Madinat al-Zahra (Cordoba) . One of the most striking features of these glasses are the exceptionally high chlorine concentrations (up to 2.5%). Recently, we have identified a second type of high lead glass among the eighth- to early ninth-century finds from the suburb of Saqunda (Cordoba) that appear to be a precursor of soda-ash lead glasses.
The chemical compositions of the two types of lead glasses were determined by LA-ICP-MS, and lead isotope analyses were performed on a subset of samples by thermal ionization mass spectrometry (TIMS). The results revealed that the lead raw material was likely sourced from mining districts in al-Andalus, and identified significant differences in the nature and processing of the raw materials. The trace element characteristics of the high lead glasses from Saqunda suggest the use of lead slag, whereas a purer source of lead underlies the soda-ash lead glasses from Madinat al-Zahra. Replication experiments based on Theophilus' *De diversis artibus* yielded clues about the production processes and possible source of the high chlorine concentrations in these soda-ash lead glasses. Based on our analytical, isotopic and experimental results, we advance a model of production for the high lead and soda-ash lead glasses in early Islamic al-Andalus.

De Juan Ares & Schibille, 2017. Glass import and production in Hispania during the early medieval period: The glass from Ciudad de Vascos (Toledo), *PLoS One* 12, e0182129.

Duckworth *et al.*, 2015. Electron microprobe analysis of 9th - 12th century Islamic glass from Córdoba, Spain, *Archaeometry* 57, 27-50.

Gratuze *et al.*, 2017. Les galets de verre au plomb carolingiens issus des scories de Melle : élaboration et distribution, *Mine, métal monnaie, Melle*.

Mecking, 2013. Medieval lead glass in central Europe, *Archaeometry* 55, 640-662.

Wedepohl *et al.*, 1995. Medieval lead glass from northwestern Europe, *Journal of Glass Studies*, 65-82.

S5-O16.679

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Ceramic “Sugar Jars” from Aveiro, Lisbon and Madeira (Portugal): archaeometric assessment

Rocha, F.[1], Morgado, P.[1], Costa, C.[2, 1], Moutinho, S.[3], Terroso, D.[1], Sequeira, C.[1]

[1]Geobiotec, Geosciences Dept, University of Aveiro, 3810-193 Aveiro. Portugal)
[2]Technological Center of Ceramics and Glass, 3040-540 Antanhol (Coimbra), Portugal
[3]RISCO, Civil Engineering Dept, University of Aveiro, 3810-193 Aveiro. Portugal)

Keywords: Ancient ceramics; Sugar Jars; Archeometrics

Sugar jars were conic ceramic forms having a hole at the bottom, being used specifically for the stage of the purge of the sugar cake. These pieces played a paramount role in sugar production cycle, being used for the maturation of the sugar, and since the 15th until the beginning of the 19th centuries, the old pottery centres from Aveiro and Lisbon regions, produced heavily these “formas de açúcar” (“sugar forms”) which were exported to sugar production areas, at places as diverse as Madeira, Canaries, Cape Verde, Cuba and Brazil. Recent archaeological findings in Aveiro, Lisbon and Madeira are giving opportunities to characterize different typologies of production. The conservation and restoration intervention intended to prepare implies the knowledge of the present state of the support material, in this case terracotta. Possible local and regional clayey raw materials were also studied.

Our main goal is to obtain information allowing typological definition, group discrimination, raw materials characterization and provenance of the studied materials. Mineralogical analysis by x-ray powder diffraction was carried out on bulk samples. Chemical composition was assessed by X-Ray fluorescence and provided the major and trace elements chemical data. Thermogravimetric analyses (TGA) and Compressive Strenght (Rc) were also assessed.

Multivariate statistical methods were applied, namely Cluster, Principal Components and Discriminant Analysis.

The obtained results of composition have given important information about the provenance of the studied materials, as well as about their raw materials. Mineralogical and chemical data obtained in samples from Aveiro point to a local production, using the upper Cretaceous marly (dolomitic) clays (Maastrichian) and clayey sands as main raw materials. Ceramics from Barreiro (Lisbon) are in general more silicated and less carbonated, composition close to the Tagus Miocene Basin clays.

The higher iron content of Aveiro clays favours the glazing of ceramic paste at lower temperatures, giving them better mechanical resistance values which can justify "their best quality", as referred to in ancient documents.

Furthermore, the analysis showed that the chemical composition of ceramic vessels found in Machico city (Madeira), where sugar production was very important on XV and XVI centuries, is closer to the chemical composition of Aveiro clays, which can indicate the place of production of those ceramics.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Post-Byzantine glass from sites of the Peloponnese: Investigating the complex glass network of southern Greece

Palamara, E., Zacharias, N.

Laboratory of Archaeometry, University of the Peloponnese, Old Camp, 24133 Kalamata, Greece

Keywords: Post-Byzantine glass; Messenia; SEM/EDS; Raman spectroscopy; provenance

The current project focuses on the chemical analysis of utilitarian glass objects of the post-Byzantine period, from two sites in mainland Messenia, Peloponnese, southern Greece: Androusa and Alagonia. The assemblage consists of 46 glass fragments of thick-walled vessels and glasses. The vast majority of the samples are colourless or naturally coloured, although there are also a few blue, green and amber samples; few samples present decoration, primarily with engraved and enamel patterns. Their exact date is uncertain; they are placed between the 15th and 18th c.

Based on the chemical and mineralogical analysis (carried out via SEM/EDS and Raman) a complex image emerges, suggesting the use of varied raw materials (Na- or K-rich plant ash, potash, saltpetre as the alkali source; manganese and arsenic as decolourants; tin and antimony as opacifiers). Given that during this period there was a high diversity in the manufacturing technologies used in glass workshops from different areas, it is possible to identify glasses originating from both the eastern regions of the Ottoman Empire (following the Islamic tradition of glass making) and from Europe (e.g. Venetian or 'façon de Venise' glass, Bohemian crystal, forest glass from central Europe etc.).

The determination of technology and provenance of the assemblage, and the comparison between the traits of each site, provided with valuable new information regarding the production and trade of glass in mainland Greece during the post-Byzantine period. Overall, an especially complex trading network is suggested, which is even more intriguing given that neither of these sites had a port and therefore direct access to the significant marine trading networks of the period.

This project was implemented within the scope of the “Exceptional Laboratory Practices in Cultural Heritage: Upgrading Infrastructure and Extending Research Perspectives of the Laboratory of Archaeometry”, a co-financed by Greece and the European Union project under the auspices of the program “Competitiveness, Entrepreneurship and Innovation” NSRF 2014-2020.

S5-O18.1561

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

LA-ICP-MS analyzes to determine provenances of Na-based fluxes and Co-based pigments used in early Ottoman pottery

Burlot, J.[1,2], Waksman, S.Y.[2], Higgins, B.[1]

[1]Archaeometry Laboratory, Research Reactor Center, University of Missouri, 1513 Research Park Drive, Columbia, MO 65211, USA
[2]Laboratoire ArAr. Archéologie et Archéométrie - CNRS UMR 5138, Maison de l'Orient et de la Méditerranée - Jean Pouilloux, 7 rue Raulin, 69365 Lyon, France

Keywords: LA-ICP-MS analyzes; Ottoman pottery; glaze's fluxes; Co-based pigment

Previous studies using LA-ICP-MS analyzes enable us to trace the origin of fluxes and pigments used in the manufacture of ceramic decorations. For instance, researchers demonstrated through such analyses that Ottoman potters used a soda-rich evaporite from western Anatolian hot spring waters as the source of the soda flux to produce the glazes of the famous Iznik Ware. Furthermore, we can now clearly identify the origin of some cobalt-based ores from Germany or Iran.

Based on these references, we carried out a series of LA-ICP-MS analyzes to determine the origin of the fluxes and cobalt-blue pigments used in the glazes of one of the first Ottoman ceramics productions: the Miletus Ware. The SEM-EDS analyzes we carried out so far on the Miletus Ware glazes revealed compositions whose ratios of some elements suggest similarities with the soda-based minerals from western Anatolian hot spring waters. Moreover, the blue glazes of Miletus Ware feature a Co-Ni-Fe-Cu association linked to the colorant, which might be an indicator of origin and/or processing. LA-ICP-MS analyzes were used to measure the contents of trace elements that could be provenances' and techniques' signatures, such as Li and B for the sodium flux, and As, Zn and In for the cobalt ores, respectively.

We will present the new data resulting from the LA-ICP-MS analyzes, which will enable us to deepen this study on Miletus Ware. We will use these results to better define production techniques of early Ottoman ceramic decorations and to suggest new commercial relations in the pottery industry of Western Anatolia, which will enrich our knowledge on the history of techniques of Ottoman ceramics.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The (Silk) Road not taken? Reconsidering the role of Central Asia in the making of Islamic glazes during the 9th to 13th centuries CE

Ting, C.

McDonald Institute for Archaeological Research, University of Cambridge

Keywords: glaze technology; late medieval ceramics; Islamic archaeology; Central Asia; Silk Road

Islamic glazes are considered to be a revolution in artistic representation and ceramic technology that occurred during the late medieval times, which would have sparked a series of technological and social changes with lasting impacts. The extant understanding of this important episode in the development of ceramic technology is built on the evidence from the Middle East and Iberian Peninsula, suggesting that Islamic glazes were most likely originated from the Middle East and spread from east to west through the Arab conquest. What seems to have been overlooked in the current narrative is the role played by local productions in other areas that were also within the realm of Islamic rule. Thus, this study represents the first, systematic effort to explore how local glaze technologies developed in Central Asia, where glaze production did not begin until after the Arab conquest. Special attention is paid on investigating how the region's long-standing involvement in trans-Eurasian Silk Road trade might have facilitated the transfer and exchange of technical knowledge in the making of Islamic glazes.

Glazed ware assemblages from production sites in Bukhara, Samarkand, and Merv – which were major oasis cities participating in Silk Road trade – are the focus of this study. These assemblages comprise a wide variety of ware types, including monochrome or polychrome painted, monochrome glazed, and sgraffito, dated to the 9th to 13th centuries CE, the period when Central Asia was under the direct or indirect Islamic rule. Thin-section petrography and scanning electron microscopy energy dispersive spectrometry were used to reconstruct the range of technical practices characteristic of the local productions in Central Asia. The resultant data are then compared with the published ones on Chinese, Byzantine, and pre-Islamic Mesopotamian traditions to highlight the potential connections between the Central Asian technologies and established glaze traditions that can be found along the Silk Road. The new evidence from Central Asia is expected to contribute to redefining what Islamic glazes truly entail, both in terms of technological repertoire and craft organisation, and delineating the nature and extent of social processes and cultural interactions that stimulated technological changes.

S5-O20.548

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

New Insights about 16th and 17th century Pick-up decorated Glass: comparison between finds in Portugal and on the Gnalić shipwreck

Pulido Valente, F.[1,2], Coutinho, I.[1,2], Medici, T. [2], Gratuze, B.[3], Alves, L. C.[4], Bonneau, A.[5], Varela Gomes, R.[6], Varela Gomes, M.[6], Radić Rossi, I.[7], Batur, K.[7], Vilarigues, M.[1,2]

[1]*Dep. of Conservation and Restoration, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal*
[2]*Research Unit VICARTE - "Vidro e Cerâmica para as Artes", Campus Caparica, 2829-516 Caparica, Portugal*
[3]*IRAMAT-CEB, CNRS/Univ. Orléans, Orléans, France*
[4]*C2TN, Instituto Superior Técnico, Universidade de Lisboa, Bobadela LRS 2695-066, Portugal*
[5]*Archaeometry research group and CELAT, Laval University, Canada*
[6]*Dep. of History, Faculdade de Ciências Sociais e Humanas, Universidade Nova de Lisboa, Av. Berna, 26C, 2780-343 Lisboa*
[7]*University of Zadar, Mihovila Pavlinovića 1, 23000 Zadar, Croatia*

Keywords: 16th and 17th Centuries, Archaeology, Pick-up Glass, Portugal, Gnalić shipwreck

Pick-up is a complex glass decorative technique that has two different morphologies: *millefiori* and *blobbing*. In the *Millefiori* (literally “thousand flowers”) embedded *murrine* (thin sliced glass canes with colorful concentric patterns in cross-section) fused into the surface of the glassware. *Blobbing* has several colored dots produced by fusing sliced colored glasses without any decorative pattern into the body glass.

Although Pick-up has been considered one of the most representative technique of Venetian glassmaking during the 16th and the 17th centuries, this type of glassware seems to be rare and only few specimens belong to museums and private collections or have been documented in archaeological excavations spread throughout Europe. Given that the documentation is of paramount importance to preserve and give value to cultural heritage, a survey that combines a morphological observation with the chemical characterisation has begun, in order to enrich our knowledge about the history of this glass. This survey resorts both to Portuguese finds and to Gnalić rescued cargo. Gnalić was a merchant ship of the end of the 16th century that sunk in Croatia with all its cargo on board: glass beads and glassware decorated in Venetian style. Also noticed that Portugal and Croatia were well-known shipping routes in this period and, for this reason, the glass composition comparison between both can bring new insights about glass trade through Europe.

In terms of dample analysis, Stereoscopy and optical microscopy were used for the morphological study. The chemical characterization was performed by the following techniques: particle induced X-ray emission (μ-PIXE) mapping to visualize how the oxides are distributed through the different layers and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) to obtain the major, minor, trace and rare earth elements composition. To observe, in cross-section, the morphology of the stratigraphic layers, opacifying agents and inclusions, scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) was performed. Raman spectroscopy was used to study the opacifiers, and UV-Visible reflectance spectroscopy to assess the chromophores present in the glass.

The work developed so far allowed us to understand that new decorative motifs were found in Portuguese contexts and it was possible to determine a large variation in the materials and manufacture quality. In addition, the main chemical composition of the Portuguese glass fragments is not compatible with the fragments found in the Gnalić shipwreck, Venetian glasses, or with other European known production centres, opening the possibility for a local production.

S5-O21.772

Session 5 Technology/provenance - ceramics/vitreous/glass

POSTER

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Investigation of the Bantu arrival in southern Mozambique in the light of geophysical prospection and archaeometric pottery analysis

Stempfle, S.[1], Linstädter, J.[2], Seifert, M.[1], Babucic, N.[1]

[1]Hamburg University, Institute for Archaeology and Cultural History of the Ancient Mediterranean Region, Edmund- Siemers-Allee 1 (West), 20146 Hamburg, Germany
[2]German Archaeological Institute, Commission for Archaeology of Non-European Cultures, Dürenstr. 35-37, 53173 Bonn, Germany

Keywords: Bantu expansion; Archaeometry; Geophysical prospection; Pottery; Early Farming Communities

In southern Africa, the appearance of pottery was first recognized in the context of Early Farming Communities (EFC) about 2000 years ago, which are called the Bantu speakers. Recent research challenges this model, since distinct pottery was found in archaeological contexts of hunter-gatherer or hunter-herder communities. Furthermore, the EFC pottery in Mozambique, called Matola pottery, has been dated a few hundred years earlier, query the previous attribution to Early Farming Communities.
In 2016, a research cooperation between the Eduardo Mondlane University and the German Archaeological Institute was started. Since then, this cooperation performed various surveys and geomagnetic prospection and developed with Hamburg University a dedicated research project focusing on geophysical prospection and archaeometric pottery analysis, which this contribution introduces.
In 2018, a first geomagnetic prospection was performed at an open-air site near the famous Daimane shelter. The detected 18 magnetic anomalies reveal two round anomalies interpreted as possible huts or kilns. The general lack of comparative studies causes a verification by other geophysical methods and archaeological excavation. Comprehensive geophysical surveys may help to record magnetic variations and electromagnetic signals in order to identify further pit-houses, pottery fields and kilns in the context of EFC. The results will reveal further excavation sites, which in return allow confirming the geophysical results.
The geophysical surveys will link the archaeometric pottery analysis with the aim to study the Matola pottery, to test the Bantu model and to investigate the beginning of pottery production in southern Africa regarding dating, the process and the diversity in raw material, techniques and use. The absolute ages will be re-evaluated by radiocarbon dating, thermoluminescence dating and compound specific lipid dating. The diversity in raw material, technique and use within the classified pottery unit will be determined using polarized light microscopy, lipid analysis, X-ray fluorescence analysis and infrared spectroscopy.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chemical and petrographic characterization of à chardón vases from south-western Iberia

Krueger, M.[1], Moreno, V.[2], Brandherm, D.[3], Dunne, J.[4], Krueger, M.[1], Michniewicz, J.[5], Michalska, D.[5] Węcławska M.[5]

[1]Uniwersytet im. Adama Mickiewicza w Poznaniu, Wydział Archeologii
[2]Universidad de Sevilla, Departamento de Prehistoria y Arqueología
[3]Queen's University Belfast. School of Natural and Built Environment
[4]University of Bristol, Organic Geochemistry Unit
[5]Uniwersytet im. Adama Mickiewicza w Poznaniu, Instytut Geologii

Keywords: pottery; Iberia; Iron Age

There are plenty of archaeological reports presenting new findings from excavated Orientalizing sites in south-western Iberia. However, only few of them have looked at paste properties and the contents of the vessels. These issues are crucial to answer questions about the production and function of pottery. Vases of *à chardón* type are one of the most intriguing forms of pottery vessel from the Iberian Early Iron Age. In comparison to other types of ceramic urn vessels from archaeological sites of the Orientalizing period in Andalusia, *à chardón* vases are relatively common, and are generally recognized as the early type of vessel within the Tartessian sequence. Many *à chardón* vessels were found in funerary context, although this type is also present in settlements. Their first appearance is synchronous with the introduction of new ceramic technologies introduced from the Eastern Mediterranean by the Phoenicians. Thirty samples of *à chardón* vases from four archaeological sites located in western Andalusia were analysed through thin-section petrography, X-ray Fluorescence Spectrometry, Scanning Electron Microscopy, Energy-Dispersive X-ray Spectroscopy and Gas Chromatography-Mass Spectrometry in order to determine their chemical composition, technological properties and the contents of the vessels. The results indicate that *à chardón* vases constitute a heterogeneous group displaying a diverse range of petrographic and chemical characteristics. Organic Residue Analysis identified beeswax in six hand-made vases, and also in one wheel-thrown vessel.

S5-P02.784

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The impact of manganese on the colour of opaque turquoise glass in the Islamic period

Richards, L., Freestone, I.

UCL Institute of Archaeology, London WC1H 0PY, UK

Keywords: Glass; Colourant Technology; Opaque Turquoise; Islamic period; EPMA; Experiment

Opaque turquoise glass is very common in Late Bronze Age glass, but appears to be restricted to specific base glass compositions in Roman glass and is rare in the Islamic period. The value attached to the gemstone turquoise and the abundance of turquoise glazed tiles suggests that this was not a matter of taste, but that some other reason should be sought. New analyses of opaque turquoise glass vessels from Tunisia, Iran, and Egypt indicate all are plant ash glass with considerable concentrations of lead oxide, coloured blue by copper and opacified by tin. All of the glasses have low manganese oxide, at less than 0.05%, which is interpreted as the background manganese from the glassmaking sand and ash; MnO contents this low are unusual in Islamic plant ash glass. Based on composition, it appears likely that the ability to make opaque turquoise was limited by the method of manufacture of primary glass, which typically included manganese as a decolouriser. The reddish colour imparted by the Mn^{3+} cation had a detrimental impact on the pale blue due to copper, and it is this which is likely to explain the limited occurrence of opaque turquoise glass. To test this, a series of simplified plant ash glass compositions were made up using standard laboratory grade reagents. The results demonstrate that the presence of Mn^{3+} in the glass is not conducive to the generation of a light copper-blue colour, such as that seen in opaque turquoise glasses.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Revisiting Early Bronze Age ceramic assemblages: a pilot analytical programme in the Insular East Aegean, Greece

Menelaou, S.

Department of History and Archaeology, Archaeological Research Unit, University of Cyprus, 20537 CY - 1678 Nicosia, Cyprus

Keywords: Early Bronze Age; East Aegean islands; Ceramic Petrography; Provenance

The eastern Aegean, which includes the offshore islands and the opposite Anatolian coast, has been overwhelmingly neglected within prehistoric Mediterranean Archaeology, in contrast with other culturally- or geographically-defined areas in the wider region. Nevertheless, according to previous synthetic works and more recent data, the eastern Aegean region forms a significant interface between the central Aegean basin and the Anatolian plateau, itself linked through long distance exchange networks with the early complex societies across the Eastern Mediterranean throughout the 3rd millennium BC (Early Bronze Age/EBA). Integrated research in the area has until recently been hindered by the lack of analytical work and by discrepancies in archaeological practice across the Greek-Turkish political border. In addition to that, the insular eastern Aegean has long been considered as peripheral or marginal between two different, neighbouring 'worlds' or as passive recipient in developments channelled from the East.

With the aim to bridge this research gap, a large-scale programme with the acronym BORDER (*Borderlands as areas of mobility and connectivity during the third millennium BC: Examining regional ceramic technologies between the east Aegean islands, western Anatolia and Cyprus*) was initiated in 2020, developing an analytical strategy for the EBA ceramic technologies of the proto-urban island settlements at Poliochni on Lemnos, Thermi on Lesbos, Emporio on Chios, and Heraion on Samos. The overall project addressed four themes of cross-regional significance: 1. Ancient craft technology, 2. Mobility and connectivity, 3. The definition of cultural and geographical borders, and 4. The interdisciplinary study of old ceramic material through a combination of traditional and modern analytical techniques.

This paper presents preliminary results from a pilot compositional and technological study of pottery from these sites, using a combined assessment of morphostylistic and fabric (macroscopic and petrographic) characteristics. Pottery is hereby used as a proxy for adding new information in our knowledge of regional exchange networks and connectivity between the different islands, western Anatolia, and the central/west Aegean. Revisiting analytically old ceramic collections, currently published and stored in museums, holds the potential of understanding anew the active role of island societies through the determination of provenance, shifting away from solely typo-chronological identifications.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

INGOT-EL: what can Iron Age beads from Etruria and Latium tell about glass production and circulation?

Yatsuk, O.[1], Iaia, C.[2], Gulmini, M.[1]

[1]Department of Chemistry, University of Turin, Via Giuria, 7 – 10125 Torino (Italy)
[2]Department of Historical studies, University of Turin, Via Sant'Ottavio, 20 – 10124 Torino (Italy)

Keywords: glass; technology; provenance; beads; Etruscan.

The research project "INvestigation of Glass Origin and Technology in the Etruscan Lands" (INGOT-EL) has the primary objective of shedding light on the circulation of glass as a material in the lands under the Etruscan influence (nowadays, Central Italy along the Tyrrhenian coast). The project is organized into three main stages: 1) comprehensive documentation, including optical microscopy examination; 2) compositional and morphological screening of a broad selection of glass objects, mostly glass beads, using non-invasive approaches with portable FORS and XRF instruments; 3) after the evaluation of information from stage 1 & 2, the selection of a representative set of objects was done and this set was subjected to in-depth laboratory analyses with SEM-EDS, μ-Raman, μ-XRD and LA-ICP-MS. Complementing techniques provide a robust ground for conclusions.

The archaeometric work was supported by the coordinated effort of several Italian research centers and Museums. In this way, we could gather information at the level of micro-structure of the material and of trace elements concentrations, which are one of the few proxies for the provenance determination. The main body of this work, which is the most comprehensive ever attempted on Etruscan materials of the same kind, includes 288 glass objects originating from 14 archaeological localities and representing collections of thousands of pieces from two important museums in Rome: the National Etruscan Museum of Villa Giulia and the Museo delle Civiltà. These artefacts belong to one of the most interesting periods of glass making history - 10th-5th centuries BCE, which saw the emergence of multiple urban centers and a complex exchange network on a Mediterranean scale. The archaeometric approach is related here with questions on glass technology, including but not limited to the selection of raw materials, furnace conditions, color manipulation, forming techniques and, subsequently, raw glass provenance.

S5-P05.1523

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Preliminary macroscopic and petrographic analysis of Early Bronze Age Pottery from Southeastern Iberia site: El Cerro de la Virgen (Granada)

Pinillos de la Granja, P.

[1]*Departamento de Prehistoria y Arqueología, Universidad de Granada, Campus Universitario de Cartuja s/n, 18071 Granada, Spain*

Keywords: SE Iberia; Ceramic technology; Petrographic analyses; Early Bronze Age

This paper presents the preliminary results of the technological and petrographic analysis of Early Bronze Age pottery (2150-1900/1900-1750 BC). Pottery comes from the stratigraphic levels 27/28 carried out in the 1986 archaeological excavation as part of the "Millares Project".
Technology is a social phenomenon that determine the decision-making process, from the procurement of raw materials to the finished products and their use. Thus, the methodological strategy implemented for the study aims to analyse the continuities and changes in the pottery production sequence during the transitional phase of the Bell-Beaker and Argaric culture in the southeast of Iberia.
In this work, a macroscopic analysis was carried out on 39 samples using the Leica Zoom 2000 stereoscopic loupe (10.5 X magnification) for the description of the ceramic fragments, analysis of the external surfaces and of the matrices. The next phase consisted of a petrography study of ten thin sections in order to expand the information from the textural analysis of the ceramic pastes. The thin section were observed using a petrographic microscope with plane polarised light and BMS 100-series crossed nicols with T2 adapter for Canon EOS cameras. The description uses the modified system of Whitbread (1995). The grain size distribution and orientation of the components is calculated following the guidelines of Bullock (1985) and applies the frequency category according to Matthew (1997).
The study sheds new light on how technological tradition participates in phenomena of change, stability or even resistance and the discussion of social complexity in the southeastern Iberian. The general uniformity of the raw materials suggests a local provenance. However, variations are detected in the percentage use of certain minerals - quartz, limestone, microfossils, schist- and specific characteristics in the ceramic manufacturing patterns of large vessels. Finally, the data also allow us to conclude that in the late 3rd millennium BC there was an integration of production processes in the traditions of the southeast of the Iberian Peninsula and a population continuity, which contrasts with recent proposals based on genetic studies.

S5-P06.1525

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Pottery raw material sources at the multi-period archaeological site of Jagodnjak – Krčevine, Croatia

Neral, N.[1], Kudelić, A.[1], Maričić, A.[2], Mileusnić, M.[2]

[1]*Institute of archaeology, Jurjevska street 15, 10000 Zagreb, CROATIA*
[2]*University of Zagreb, Faculty of Mining, Geology and Petroleum Engineering, Pierottijeva 6, 10000 Zagreb, CROATIA*

Keywords: pottery; archaeometry; raw material; provenance studies; Croatia

The tradition of studying archaeological pottery in Croatia is deeply rooted in the cultural-historical approach, especially when it comes to prehistoric pottery. However, turning the approach by studying ceramics throughout its life cycle, from the selection of raw material, through the various stages of production, to distribution and use, can provide valuable research contributions to a topic that has not yet been fully explored. Therefore, a five-year project focusing on the study of prehistoric pottery traditions in Croatia was launched in 2021. A large-scale systematic archaeometric research programme will be combined with macroscopic physical forms of investigation of previously excavated pottery.

The paper presents the preliminary results of a case study focused on the multi-period (Neolithic, Bronze Age, Iron Age, Roman, Mediaeval) archaeological site of Jagodnjak – Krčevine. Ceramic petrography, X-ray diffraction analysis and field sampling is used to identify the characteristics of archaeological ceramics and to determine the provenance, availability, and types of raw material used for the production of ceramics in the different periods of the past. In order to obtain an optimal data set related to the provenance of raw materials, a field survey was conducted. Field sampling was established according to zones with different distances from the archaeological site and in accordance with the characteristics of the lowland landscape. Comparison between XRD data of clayey material and pottery, and data obtained by optical microscopy suggests the location of the raw material source.

Preliminary results of the archaeometric analysis indicated that raw material was probably collected in the vicinity of the settlement, during all periods of the past recorded at the site. However, certain preferences were observed, concerning not only the tempering material used, but also the choice of raw material sources and clay processing, that match different communities. Such diversity in the choice of raw materials indicates not only technological but also different patterns of landscape use over time.

S5-P07.1526

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

An archaeometric approach to the Late Roman coarse ware from the rural settlement at Podšilo bay (Lopar, Rab Island, Croatia)

Nowacki, B.[1], Trzciński, J.[2, 3], Konestra, A.[4], Welc, F.[3], Waligóra, P.[3]

[1]*Faculty of "Artes Liberales", University of Warsaw*
[2]*Faculty of Geology, University of Warsaw*
[3]*Institute of Archaeology of the Cardinal Stefan Wyszynski University in Warsaw*
[4]*Institute of Archaeology, Zagreb*

Keywords: coarse wares; 3D reconstruction; fabric; SEM-EDS; chemical composition

Among the pottery excavated from the late Roman layers at Classe (Italy), the presence of coarse ware Classe type was highlighted by Gelichi (1983). From that time such coarse ware has been recognized in several upper Adriatic sites. Within the *"Archaeological Topography of the Island of Rab"* project, the site of a Roman rural settlement is being excavated in Podšilo Bay, in N Lopar. In late antique layers (6th c.) notable quantities of coarse wares have been found. Initial analysis of this pottery has led to a hypothesis that some items could have been imported from Italy and might belong to pots of the Classe type.
One of the research problems of the project is determining the origin of coarse ware. The aim of the study is a typological characteristics of the best preserved Classe type pot from Podšilo and an archaeometric analysis of its fabric. The aim was achieved by analysis of shape, dimensions and decorations. 3D reconstruction was based on photographs and drawings processed with Blender software. Analysis of the fabric was accomplished by optical and SEM-EDS microscopy.
The pot fragment with a red tint on the fracture (Munsell 7.5R 6/6) contains 5–10% white grains which are 2–0.2 mm in size, sharp-edged, with an isometric shape, a platy habit and a glassy lustre. The grains concentrate on the both surfaces. Horizontal throwing marks can be noted on the interior, whereas the pot is black-red on the exterior (Munsell 7.5R 2/1).
The ceramic is composed of a medium homogenized clay mass (matrix) containing mineral grains. The matrix is poorly oriented, in some places with a turbulent arrangement and low porosity. The grains contain Ca and C, and small amounts of Si, Al, Mg, K and Na. The matrix comprises Si and Al, with the admixture of Ca, Fe and Ti, and small amounts of K, Mg and Na.
The ceramic material is composed of a raw material (sheet aluminosilicates – clay minerals) which have been multiply subject to high temperature, and of a temper, calcium carbonate(?) grains (probably calcite). Detailed analysis with application of thin sections and advanced techniques will be required for a exact determination of the composition and technological characteristics of the ceramic.
This should allow for a comparison with similar vessels from western and upper Adriatic sites while further analysis of the coarse wares from Podšilo will bring forth more detailed characterisation of its composition and technological features, evidence the scale and origin of imports, and the local preferences in cooking wares in a timeframe of profound socio-economic changes.

S5-P08.1527

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Luxury ceramic vessels from the Dacian Kingdom, SW Transylvania

Pușcaș, C.M.[1], Stremțan, C.C.[2], Ferencz, I.V.[3], Roman, C.C.[4], Tămaș, T.[5]

[1]*Terra Analitic SRL, Alba Iulia, Romania*
[2]*Teledyne Photon Machines, Bozeman, MT, USA*
[3]*Museum of Dacian and Roman Civilization, Deva, Romania*
[4]Independent researcher, Hunedoara, Romania
[5]Babeș-Bolyai University, Dept. of Geology, Cluj-Napoca, Romania

Keywords: ceramics; Dacia; hill forts; XRD; petrography

The first century of the Christian Era was a time of prosperity for the Dacian Kingdom in South-western Transylvania. Seignorial residences, with pretentious buildings that held luxury inventory items, became frequent in the area. Along with these, settlements from the Mureș Valley, the most important waterway in the area, prospered as well owing to commerce. Examples of such sites are the Ardeu - Cetățuie and Hunedoara - Dealul Sânpetru hill forts, as well as the Tărtăria - Pietroșița settlement. These three settlements are representative for the Dacian habitat typology in the area, such that the analysed ceramic material was selected from their archaeological inventory. Pottery – manufactured from a fine paste and painted with geometric motifs and ribbons of colour – is a noteworthy example of luxury goods.

A set of samples from such luxury ceramic objects were analysed by X-ray diffraction (XRD) and petrographic microscopy in order to identify their mineralogical composition (for raw material sourcing) and level of production technology (technique, colour, surface treatments, tempering material, firing temperature etc.). The aim was to differentiate among different compositions of the clay and temper, in order to try and identify potential sources for local or distant raw materials, according to each of the three sites where the ceramic fragments were discovered.

The combined results from the analytical methods we implemented for this study show significant variations concerning the locally produced ceramic goods (regarding the quality of the raw materials, the manufacturing and firing techniques, attention to detail etc.) relative to those which we consider to be imports. Aside from different raw materials, the techniques identified for ceramic manufacturing vary as well, further supporting the hypothesis that the ceramic goods were produced locally as well as imported.

In addition to producing important data that supplement the available information on an exceptional category of Dacian pottery, the results offer a new research perspective on the pottery from that era. Extending this type of in-depth analytical research to a statistically significant set of samples will facilitate the understanding of economic and social phenomena that were specific to the Dacian Kingdom. We also hope that our contribution will help to set improved criteria for future inter-site comparisons.

S5-P09.197

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Agios Petros and the Neolithic Pottery Making Traditions of the Deserted Islands, Northern Sporades, Greece

Barouda, A.[1], Quinn, P.[1], Efstratiou, N.[2]

[1]Institute of Archaeology, University College London
[2]School of History and Archaeology, Aristotle University of Thessaloniki

Keywords: Neolithic Aegean; Pottery; Provenance; Ancient Technology; Archaeometry

The prehistoric site of Agios Petros on the island of Kyra Panagia in the Deserted Islands of the northern Sporades, Greece is one of the earliest Neolithic settlements of the late 7th millennium BC northern Aegean. Its excavation between 1967–1981 revealed a rich assemblage of decorated ceramics that exhibit stylistic links with other regions, as well material with a unique local character, such as canvas like red-on-white painted wares that also occur at the Cyclops Cave on the neighbouring island of Yioura. Previous analysis of contemporaneous ceramics from this seasonally occupied cave have suggested that the people of the northern Sporades were well connected to the rest of the Aegean and may have participated in long distant exchange networks that included obsidian and pottery. In order to further investigate these links and characterise in more detail the nature of local pottery production on the Deserted Islands, we have analysed in a selection of ceramics from the assemblage of Agios Petros, which was a permanent settlement in Neolithic times. Ceramics and raw material samples from Kyra Panagia have been characterised via ceramic petrography, neutron activation analysis, scanning electron microscopy and x-ray powder diffraction, and compared directly to material from the Cyclops Cave. Our results suggest that the pottery from these islands was mostly locally produced. A well-developed tradition of ceramic production seems to have been brought to the northern Sporades some time in the late 7th millennium BC. It is characterised by a high level of technological know-how and remains essentially unchanged for over 1000 years, until the islands were deserted in the 5th millennium BC. Agios Petros appears to be the main pottery production centre in the area with ceramics being transported to the Cyclops Cave during sea travels or fishing expeditions. We have not detected long distant exchange on ceramics at Agios Petros compared to the neighbouring Cyclops Cave, suggesting that the settled Neolithic communities on these far flung outposts of the Sporades chain had a more stable long-lasting local ceramic tradition than previously thought.

S5-P10.1530

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Production and use of 'bevelled rim bowls' in the Middle Syrian Euphrates during the Late Chalcolithic period: an archaeometry study

Sanjurjo-Sánchez, J.[1], Montero Fenollós, J.L.[2]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]Departamento de Humanidades, University of A Coruña, Campus de Esteiro, 15403 Ferrol, Spain

.

Keywords: bevelled rim bowls; BRB; Uruk; pottery; Middle Euphrates

The so-called "bevelled rim bowls" (BRB) are a "fossil" director of the material culture of Uruk, that can be found almost everywhere in the Near East in archaeological sites of the Late Chalcolithic 4-5 period (also called Middle and Late Uruk). However, its production process, trade and use are still unknown. In the PAMES Project (Archaeological Project of the Middle Syrian Euphrates) a special effort has been devoted to know these issues in detail, through geochemical analyses of BRB samples from various sites, analyses of organic compounds found in BRB and the use of experimental archaeology to reproduce its production process. Although most of the samples studied come from the Middle Euphrates of Syria, it has been possible to contrast the data obtained with other sites in Iraq and Iran. The study of the mineral and elemental composition of BRB samples from the Middle Syrian Euphrates revealed minimal geochemical differences between sites, so that two possible hypotheses are considered: (a) a local production due to the high homogeneity between the sources of raw materials (fluvial clays), or (b) a production and distribution center in the area. The BRB samples studied from deposits Iraq and Iran clearly show a different composition.

Over the last few decades different hypotheses have been proposed for the production process of the BRB, ranging from moulding in holes dug into the ground to moulding by hand. We have been contrasted these with the use of moulds. This proposal is based on a possible mould found in Tell Qannas (north of Syria). A manufacturing experiment has shown that the mobile mould would be the form of manufacture that best fits the morphology of the BRBs.

Finally, with regard to its use, several hypotheses have also been raised, ranging from cooking and producing yoghurt or bread to religious uses. To get more light on this, we have analysed the organic matter contained in the BRB matrix by pyrolysis coupled to gas chromatography and mass spectrometry (Py-GC/MS). The study reveals that the hypothesis compatible with the analytical results is a cereal-based content, which coincides with its use to make leavened bread. This study has yet to be concluded with the analysis of the DNA contained in the bowls.

S5-P11.336

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Petrographic and chemical analyses of the Late Neolithic Period pottery from the Lushanmao site in Shaanxi, China

Nani Liu.[1], Mingzhi Ma.[2], Kunlong Chen.[1]

[1]*Institute for Cultural Heritage and History of Science &Technology, University of Science and Technology Beijing*
[2]*Shaanxi Academy of Archaeology*

Keywords Lushanmao site, petrography, chemical analysis, pottery

This paper presents the results of petrographic and geochemical analysis of pottery sherds discovered from the Late Neolithic Lushanmao site in Shaanxi Province, China. The discovery of high-level buildings and jade and tiles at Lushanmao indicates its position as the core settlement in the region. We applied optical microscopy (OM) and X-ray fluorescence (XRF) on some selected samples to analyze the tiles and vessels in order to understand the production status for different purposes, and then analyze the pottery production models. Chemical analysis shows that pottery raw materials are common meltable clay, CaO content in most vessels is less than 3%, while in most tiles is more than 3%. Petrographic analysis divides all samples into five groups: fine-grain quartz, coarse -grain mudstone, coarse-grain granite, coarse-grain gneiss and plant. The first three groups are found in tiles, and vessels covers all groups. Furthermore, different types of fabrics also appear in the same type of tiles or vessels. The selection of materials for the tiles is not specific, but given the geographical context, it is likely that all samples was produced locally and in a variety of tradition of production technology. The aforementioned materials and technical characteristics imply that the pottery production at Lushanmao was not highly specialized, multiple small production units may product tiles and vessels to satisfy the needs of people in nucleated settlement.

S5-P12.1536

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Characterization of pigments in ceramic productions in the Iron Age settlement of Sierra Boyera (Belmez, Córdoba, Spain)

Cristo-Ropero, A., Dorado Alejos, A., Coria Noguera, J.C., González-Zambrano, P., Pérez-L'Huilliere, D., Adroher Auroux, A.M.

Universidad de Granada

Keywords: pottery; pigments; Iron Age; Iberians, archaeometry

The Iron Age settlement of Sierra Boyera is located in the norther part of Cordoba province (Spain), in a region between the Guadalquivir River valley and the basin of the Guadiana River, which are connected thanks to tributaries such as the Guadiato and the Zújar. Archaeological excavations carried out in 2021 documented a pottery production area dated in the IV-III centuries BC. A Phoenician-Punic inspired pottery kiln with a central pillar, failed ceramic products and other pottery production elements where recorded in this place.

A fact of this settlement is its location next to Sierra Boyera reservoir, which entails the flooding of the site except in dry seasons. That situation affects the conservation status of archaeological evidence. One of the most exposed materials are pots, which have been severely deteriorated and lost the major part of the pigments.

This study aims to realice a preliminary approach to ceramic production from the Iron Age hillfort of Sierra Boyera, particulary those with pigments remains. For this porpuse, 23 painted ceramic samples, which shows red, white and black colors, were selected in order to carry out three archaeometric analysis. Firsly, a macroscopic ware analysis have been utiliced to study the ceramic matrix and surface characteristics. Along with this initial data, we examinated the sherd surfaces with the Dtstrech program, which shows imperceptible colorations for the human eye. Finally, as a mean to determinate the raw material and chemical composition of the pigments we fulfilled FTIR analysis.

This investigation sheds some light on ceramic production of the Protohistoric communities settled in the central part of Sierra Morena system. In addition, archaeometric data provides a much necesary dataset for pottery analysis of Iron Age ceramics in this part of the Iberian peninsula.

S5-P14.1569

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The GLASSGEM project: a PIXE/PIGE campaign to disclose composition of glass-gem samples from the National Archaeological Museum in Aquileia

Gagetti, E.[1], Di Martino, D.[2], Marcucci, G.[2], Lemasson, Q.[3,4], Riccardi, M.P.[5,6]

[1]Dipartimento di Beni Culturali e Ambientali Università degli Studi di Milano 'La Statale'", Milan, Italy
[2]Dipartimento di Fisica "G. Occhialini", University of Milano-Bicocca, Milan, Italy
[3]Centre de recherche et de restauration des musées de France, Palais du Louvre, 14 quai François Mitterrand, 75001 Paris, France
[4]New AGLAE FR 3506 – Chimie-Paristech/Ministère de la Culture, Palais du Louvre, 14 quai François Mitterrand, 75001 Paris, France
[5]DiSTA-University of Pavia, via Ferrata 9, I27100 Pavia, Italy;
[6]Arvedi Laboratory, University of Pavia, via Ferrata 9, I27100 Pavia, Italy

Keywords: Glass-gem; PIXE; PIGE; Roman glass, non-destructive techniques

Artistic glassmaking has ancient origins: glassmakers of different historical periods and places used different raw materials and manufacturing techniques [1]. Generally, glass classification follows elemental composition as for major (for example silica), minor (for example soda, ashes or potash) and trace components (for example B, F, Ti, Fe, but also Sb, Sn and Pb elements). Glasses were produced also for imitation purposes, for example in gemology [2], and in some cases there are doubts about the glassy or crystalline nature of the specimens. The whole composition of the specimens thus reflects the progress reached by the manufacturer's workshop and it can be used as an archaeological marker of production times and geographical area. A set of more of 100 specimens were at our disposal from the National Archeological Museum in Aquileia (Italy), selected among the wide collection of the museum (more than 8000 gems).
Glass-gems were studied in the past mainly by glyptic and iconographic researches. We wanted to address the glass composition and derive hints on its fabrication route.
More than 150 samples of finished and semi-finished artefacts were analysed, for a total of about 400 measurements collected during an experimental campaign by particle induced X-ray and gamma emission studies. PIXE and PIGE data, after a first raw data analysis, returned the composition of all specimens. Our results were compared with typical main glass groups and with Roman glass composition (like Na content), trace elements related to raw materials (like Ti or other "marker" elements); minor or trace elements connected to colouring and decolouring agents (like Fe, Cu, Mn, Sb) [3-5].
The glass gems of the Aquileia Museum were made of different types of glass. The largest nucleus shows a natron glass composition, but also Na-Ca, Na-K-Ca and K-Ca glass, referable to the use of fluxes derived from sodium, mixed alkali and potassium ash are well represented.
We will present these first results in order to understand their significance in relationship with technology, history and commercial trades in the Aquileia area.

References: [1] Weyl, W. A., 1959, Coloured glasses, Society of Glass Technology, Sheffield. [2] Sena Chiesa, G., 2010, Pallas 83, 225. [3] Jackson, C.M. 2005 Archaeometry 47, 763. [4] Shortland et al., 2006. Journal of Archaeological Science 33, 521–30. [5] Calligaro et al., 2008. X-Ray spectrometry 37, 169-177.

S5-P15.1574

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Identification of colouring agents of surface finishing using raman spectroscopy

Kolářová M.[1], Kloužková, A.[1], Dvořáková, P.[1], Šefců R.[2]

[1]University of Chemistry and Technology, Prague, Technická 5, 166 28 Prague, Czech Republic
[2]National Gallery Prague, Staroměstské nám. 12, 110 15 Prague, Czech Republic

Keywords: Infrared spectroscopy; Raman microscopy; pigments; dyes; surface finishing

Several analytical techniques including Raman spectroscopy are widely used for the identification of pigments, raw materials and ceramics in culture heritage. Archaeological and historical glazed ceramic fragment are often characterised by a combination of analyses to obtain an overall identification of the sample. Vibrational spectroscopy techniques mainly Raman microscopy, FT-Raman spectroscopy and ATR were applied as non-destructive techniques, which could provide the information about pigments, dyes and surface coatings. The main aim of this work was to identify different types of surface finishing of archaeological ceramics by Raman techniques. Four types of laser wavenumbers (532, 623, 785 and 1064 nm) were used for measurements of pigments, dyes and glazes, which have been partially covered by corrosion products. The appropriate choice of laser has an important impact on experimental capabilities as sensitivity and spatial resolution. Sample behaviour has the essential effect on the optimisation of measurement technique. The data collected proved great efficiency of surface layer identification by Raman spectroscopy. Two specific types of carbon coating of different origin were identified within the surface of tile prehistoric ceramic fragments from Hallstatt period and Renaissance container. Moreover, the Raman spectra of green and yellow glazes of Renaissance ceramics proved that both colours were made using similar raw materials.

This research has been supported by the grant NAKI II DG18P02OVV028

S5-P17.1552

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Jun ware glaze colours: An X-ray Absorption Spectroscopy study

Yuan, M.[1], Hou, J.[2], Gorni, G.[3], Crespo, D.[1], Li, Y.[2], Pradell, T.[1]

[1]Physics Department and Barcelona Research Centre in Multiscale Science and Engineering, UPC BarcelonaTech, Campus Diagonal-Besòs, c) Eduard Maristany 16 08930 Barcelona, Spain
[2]The Palace Museum and Key Scientific Research Base of Ancient Ceramics, State Administration of Cultural Heritage, Beijing, China
[3]CELLS-ALBA Synchrotron, Carrer de la Llum 2-26, 08290 Cerdanyola del Vallès, Barcelona, Spain

Keywords: Fe K-pre-edge fitting; Metallic copper nanoparticles; Iron oxidation state; Absorption and scattering; Jun ware glaze

Jun ware is high-fired grey-bodied stoneware created in the late Northern Song dynasty (12th century) with a subtle blue glaze combining transparent-blue and whitish-opaque submillimetric areas.
The glaze has a glass nanostructure with lime-rich droplets in a silica-rich matrix resulting from a high temperature liquid-liquid phase separation. Calcium rich opaque and calcium poor transparent areas are combined. Iron is more oxidised in the calcium rich areas (≈17-20% Fe^{2+}) than in the calcium poor areas (≈60-70% Fe^{2+}) of the glaze. This implies that iron is predominantly oxidised in the lime-rich droplets and reduced in the silica-rich matrix. The *sky*-like appearance of the glaze is due to the combination of the light absorption in the transparent-dark-blue Fe^{2+} rich areas and light scattering in the white-yellowish Fe^{3+} rich areas.
Copper appears mainly as Cu^{+} and Cu^{2+} in the glaze but in the red areas of the glaze a few small metallic copper nanoparticles are also present. Iron appears more oxidised in the red glaze than in the blue glaze. This is more pronounced in the calcium poor areas (Fe^{2+} richer) than in the calcium rich areas (Fe^{2+} poorer). The results obtained point out to the simultaneous reduction of copper and oxidation of iron.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Bronze Age domestic ceramics from Sardinia (Italy): A social and technological overview

Gradoli M. G.

Geologist, MA and PhD in Archaeology

Keywords: Bronze Age, domestic ceramics, technology and social networks

This research focused on the relationship between ceramic technology and social organization, studying 488 ceramics from eight Bronze Age settlements (1800-750 BC) in south-central Sardinia. The approach proposed – analyzing ceramic fabric variability among selected common vessel forms (cooking, storage and drinking vessels) in close connection with domestic architecture – represented an innovation with respect to the previous studies of pottery in Sardinia, that have mainly focused on stylistic attributes and their use in assessing a chronological typology.

The principal research question was whether a technological study of a selected group of domestic ceramics could shed new light on the pattern of pottery production, consumption, and exchange in the region studied. Ceramics Petrography, using the comprehensive and systematic method proposed by Whitbread, permitted me to characterize thin sections into seven Fabric Groups and 38 Fabric Classes, highlighting specific technological (rock tempering) and social choices (grog addition) preferred by potters; the detailed raw materials sampling and regional geological knowledge, allowed identification of the clays used in the vessels manufacture and possible production centers; while the study of the 'chaîne opératoire' and the experimental archaeology helped in reconstructing potters' technological and social behavior. The study was combined with organic residue analyses from selected cooking vessels, which illuminated on some specific culinary practices (some of which still present in Sardinia) and vessels multifunctional use.

The realization that local domestic plain vessels could have been circulating among these settlements as exchanged items throughout the Bronze Age and even the pre-nuragic times, was demonstrated on petrographic and geological basis. This result was complemented by identification, in a part of the region studied, of several pottery traditions that might reflect patterns of co-residence between groups of people having different cultural affiliation.

Considering fabric heterogeneity and vessel shapes through time, this research also demonstrated how potters had a widespread appreciation of similar raw materials, hand-made pottery production and firing conditions. This indicates not only a shared technological tradition but also wider community interactions.

The 'nuragic grey/black burnished ware' table-production, typical of one of the largest nuraghi of Sardinia, the Nuraghe Arrubiu at Orroli, was recently recognized by the writer, under the petrographic microscope, comparing these fabrics to those of five small black bowls from three votive pits at the Hala Sultan Tekke cemetery (Cyprus) which confirmed, once again, extra-insular contacts between Sardinia and the Eastern Mediterranean.

S5-P19.566

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Study of manufacturing technology in historical materials bymeans of reproductions: the case of the mediaeval *carreaux de pavement* from the Iberian Peninsula

Ruiz–Ardanaz, I., Gil-Fernández, M., Arias-López, V., Araiz-González, S., Lasheras, E., Durán, A.

University of Navarra, Chemistry Department (C/ Irunlarrea, 1, Edificio de Investigación, 31008 Pamplona, Navarra)

Keywords: *carreaux de pavement*; experimental archaeology; mediaeval pottery; archaeometry; multivariate modelling

Tiebas Castle (Navarra, Spain) was built in the mid-13th century as a sign of ostentation and power for the newly crowned Teobaldo II of Navarre. The castle was designed following the canons and luxuries of the French court to which Teobaldo II belonged as Count of Champagne. One of these luxuries was tiling the castle floors with decorated glazed ceramics, called *carreaux de pavement*. This type of mediaeval tiles reached its maximum diffusion in France and England between the 12th and 14th centuries.

The manufacturing technique of these tiles has been unknown until now. Through experimental archaeology, replicas were made and simultaneously analysed together with the original archaeological samples found in the Castle. To achieve compatibility with the elemental and mineral composition of the original samples, a local decalcification clay was used as raw material for the replicas. Additionally, they were constructed using similar thicknesses (1 cm and 2,5 cm) as the archaeological samples. The replicas were fired at different temperatures (700, 800, 850, 900, 950, 1000 and 1100 ºC) and times (1, 3, 6, 24, 48, 72 hours). The analytical techniques used were the following: acid digestion, colorimetry, compressive tests, Fourier transform infrared spectroscopy (FT-IR), X-ray fluorescence (XRF) and X-ray diffraction (XRD).

One of the key variables used to follow the changes produced during the firing processes was the hematite crystal size. Sintering and internal reduction of porosity during firing led to resistance to attacks by aqua regia (3 HCl + HNO_3) to generally soluble elements at low pH, e.g. Fe. The results of hematite crystal size and %SiO_2 and %Fe_2O_3 of the solid residue (after the acid attack) were used to build an empirical model that determined the firing time and temperature of the archaeological samples. Some mineralogical phases were also useful to confirm the firing temperature range like the absence of mullite and cristobalite, high temperature phases (≥1000 ºC) or illite, low temperature phase (≤850 ºC).

Our study concluded that the firing time of the archaeological samples was higher than 24 hours and the firing temperature range was between 875 and 950 ºC. The results not only allow us to estimate a narrow range of firing temperatures, but also serve as a basis for future investigations on the manufacturing technology of the upper layers (engobe and glaze) of these types of ceramics.

**The reported study was funded by the Dirección General de Cultura-Institución Príncipe de Viana (Navarre Government) under the projects “Thibalt. Caracterización arqueométrica de *Carreaux de Pavement* procedentes del Castillo de Tiebas (Navarra)” and “Aplicación del arqueomagnetismo y otras técnicas

S5-P20.3013

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Medieval shipyard of Seville in Spain: an archaeometry approach of the material characterization of brick and mortar masonry

Debenedictis, D.[1], Pérez-Rodríguez, J.L.[2], Pagliuca, A.[3], Robador González, M.D.[1]

[1]*Universidad de Sevilla*
[2]*Instituto de Ciencia de Materiales de Sevilla, CSIC-Universidad de Sevilla*
[3]*Università degli Studi della Basilicata*

Keywords: Medieval masonry; Ceramic Brick; Lime Mortar; Shipyard; Seville

The *Atarazanas* of Seville, built by Alfonso X "*el Sabio*" from 1252, undoubtedly represented at the time the most powerful and largest shipyard in the Mediterranean, with its 17 naves (or aisles) perpendicular to the Guadalquivir River, occupying an area of about 15,000 sqm. Several uses and changes have occurred after the cessation of their main function, some naves have been transformed and others, unfortunately, have been demolished in the last century. But, luckily, there is still a legacy that helps us to explore its history and magnificence.

In this study the chemical, physical and mineralogical characterization of mortars and bricks used in the walls of the ancient shipyard of Seville have been investigated to analyze the technical aspects of the structure and its materials, trying to determine the production process adopted, the dimension and distribution of the aggregates, the origin of the clays and the aggregates, the firing temperature of the bricks, etc. The characterization of the mortars has been carried out through petrographic, granulometric and aggregate distribution analyses, while the characterization of the bricks by X-ray diffraction (XRD), mercury porosimetry and petrographic observations.

The purpose of the scientific analysis has been to identify the very origin of the material, in order to use bricks and mortars with the same characteristics in a restoration intervention.

Through the results of the analysis, it can be concluded that the constitution of the mortars is heterogeneous. However, the main mortars are mainly hydraulic constituted by calcite, aggregates and gypsum of contamination. The aggregates, mainly responsible for the hydraulic character, obtained by acid treatment of the mortars are constituted by a high percentage of quartz and mica, albite, anorthite, and chlorite. Restauration mortars have been scarcely used constituted by calcite, quartz and portlandite. The bricks used as masonry units or aggregate in the mortars were manufactured from calcium-rich clay, characteristic of Guadalquivir river valley applied in ceramic from antiquity till actuality. The phases present in the bricks show different firing temperatures: 1) fired over 900 ºC, which have anorthite and gehlenite and do not have illite, 2) fired just below 900 ºC with illite, gehlenite and wollastonite and 3) fired just below 800 ºC temperature without neoformed phases been illite present. Monocrystalline and polycrystalline calcite has been detected in mortars and bricks, respectively. Biological activity and salts have been detected on the surface of the walls.

Acknowledgements: To the *Fundación para la Investigación y Difusión de la Arquitectura en Sevilla* for the "*Beca FIDAS 2021 a la Investigación*" and to the PID 2020-115786 GB-100 project, financed by MCIN/AEI/10.13039/501100011033

S5-P21.3021

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Studies of medieval ceramics treated with acids for the removal of calcareous deposits

Sáenz-Martínez, A.[1], Estébanez, M.P.[1], Andrés, M.S.[1], Alvarez de Buergo, M.[2], Fort, R.[2]

[1]Departamento de Pintura y Conservación-Restauración, Facultad de Bellas Artes, Universidad Complutense de Madrid (UCM). C/ Pintor el Greco 2, 28040, Madrid, España
[2]Instituto de Geociencias IGEO (Consejo Superior de Investigaciones Científicas y Universidad Complutense de Madrid). C/ Severo Ochoa 7, 28040, Madrid, España

Keywords: archaeological ceramics; insoluble salts removal; calcium carbonate deposits; acid treatments; scientific analyses

The presence of insoluble salts deposits on archaeological ceramics, which is very common, can imply several damages, from aesthetics to physical ones, passing through information loss, provided by decorations, inscriptions or stamped seals (Orton et al, 1997). Since the 70s, this kind of alteration has been mostly treated with acid and basic solutions, without carrying out studies about their effectivennes or the effects on the ceramic materials (Pearson, 1987; Berducou, 1990; Buys and Oakley, 1993; Fernández, 2003; Crisci et al, 2010; Casaletto et al., 2008; Johnson et al, 2011; Coladonato et al, 2013).
This research arises from the need of analysing the effects of several chemical treatments based on acid products applied to remove calcium carbonate deposits from archaeological ceramic surfaces. With this aim, three medieval sherds form the archaeological site of Guarrazar (Guadamur, Toledo, Spain) were selected and their compositions were characterized with different techniques: X-ray fluorescence (XRF), powder X-ray diffraction (P-XRD), Fourier-transform infrared spectroscopy with attenuated total reflection (FTIR-ATR), and thermal analyses were performed to quantify the content in calcium carbonate (TG-DSC). P-XRD results allowed to determine the equivalent firing temperatures between 800 and 1000ºC. Besides, the calcium carbonate content detected by TG-DSC defined the clay matrix as non calcareous (<6%).
Furthermore, their physical properties were established by non-destructive techniques (NDT). Open porosity and density were set by the saturation test (UNE-EN 1936:2007), as well as colour measurements (UNE-EN 15886:2001) and optical micro-roughness measurements were conducted.
The ceramic specimens were characterized with these techniques before and after the acid cleaning treatments tested. Consequently, it has been possible to study not only the effectiveness of the treatments, but also the changes suffered by the ceramic sherds. In this way, the results obtained will be later applied on ceramics with similar characteristics, from the archaeological site of Guarrazar, and from other sites of similar chronologies.

S5-P22.183

X-ray fluorescence methods for an elemental characterization of archaeological ceramics from Baikal Siberia

Maltsev, A.[1,2], Chubarov, V.[3], Pashkova G.[1,4], Demonterova E.[1], Amosova A.[3], Shergin D.[4]

[1]*Institute of the Earth's Crust, Siberian Branch of Russian Academy of Sciences, 128 Lermontov st., Irkutsk, Russian Federation.*
[2]*Kazan National Research Technological University. 68 K. Marx st., Kazan, Russian Federation*
[3] *Vinogradov Institute of Geochemistry, Siberian Branch of Russian Academy of Sciences, 1A Favorsky st., Irkutsk, Russian Federation*
[4] *Irkutsk State University, 1 Karl Marx St., Irkutsk, Russian Federation*

Keywords: X-Ray fluorescence, ceramic, elemental analysis, Baikal Siberia, chemometrics

Elemental analysis of archaeological artefacts is a key for the assessment of transportation routes and cultural interconnections between people were living in different regions. The involvement of modern instrumental analytical methods expands the potential of understanding a migration, economic and sign-symbolic activities of the ancient population. The study of ceramics from Baikal Siberia located in Upper Lena river (archaeological sites of Popovsky Meadow, Makarovo 1-2, Shishkino, Ust-Yamny) and Vitim river (archaeological sites of Gingerbread IV, Ust-Karenga I-XVI) have been done. A research of ceramics using natural science methods is widespread in world practice, but in this case, the studies for the Baikal Siberia region was performed for the first time.

Two original quantitative techniques have been developed for the analysis of ceramics by WDXRF and TXRF. New WDXRF technique enables to use only 110 mg of calcined sample at 950 °C for a fusion with lithium methaborate, and less than 300 mg for pressed pellets. Therefore, less than 500 mg is required for quantitative WDXRF analysis. Samples were prepared as pressed pellets for a minor elements (V, Cr, Co, Ni, Cu, Zn, Sr, Zr, Y, Ba, Pb, As) determination, and as fused beads with 10 mm diameter for a major compounds (Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, CaO, TiO_2, MnO, Fe_2O_3) determination.

TXRF technique were developed for very small parts of found ceramics with the aim to save historically important material. Small amounts of samples (less than 10 mg) were digested using an aqua regia solution and then were spiked with ultrapure water and internal standard. However, the digestion process cannot be fully completed for silicate part of samples, so we used addition sample preparation technique – water suspension. TXRF technique enables to determine major elements (Mg, Al, Si, P, K, Ca, Ti, Mn, Fe), and trace (V, Cr, Ni, Cu, Zn, Rb, Sr, Y, La, Ce, Pr, Nd, Sm, Pb) elements.

Data on elemental composition obtained by WDXRF and TXRF methods for the ceramic samples from Baikal Siberia have been compared and processed using principal component analysis (PCA). This statistical approach enables to classify samples to similar groups, and this is the way to assume different provenance places of ceramics.

The reported study was funded by Russian science foundation, project number 19-78-10084. Measurements were performed using the equipment of the joint use centers (Geodynamics and Geochronology Center and Isotope-geochemical Research Center).

S5-P23.599

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Roman bricks in Northern Italy: Manufacturing technologies over time

Pérez-Monserrat, E.M.[1], Maritan, L.[1], Causarano, M.A.[2], Urbanova, P. [2,3], Chavarria, A.[2]

[1]Geosciences Department, Padua University, Italy
[2]Cultural Heritage Department, Padua University, Italy
[3]IRAMAT-CRPAA, CNRS-University Bordeaux Montaigne, France

Keywords: Interdisciplinary studies; gehlenite formation; knowledge transfer; built heritage conservation

Building bricks entail a traditional material meaningfully linked with cultural identity of peoples and represent a sustainable solution for the built heritage conservation. Through an interdisciplinary approach based on archaeometric methodologies, the bricks of the Basilica of Santa Justina of Padua (Northern Italy) were studied. The city of Padova is located in the Eastern side of the Pianura Padana, characterized by the abundance of clayey materials, mostly corresponding to Quaternary alluvial deposits. The late antiquity (5-7th centuries), when the production of bricks in the city were made accordingly the Roman techniques, and medieval (12-13th centuries) areas of the basilica were selected.
The main objectives addressed were the analysis of bricks manufacturing by means of a multianalytical study (Spectophotometry, XRF, XRDP, POM, FESEM-EDS) and the data transfer to current ceramic industry in order to afford more sustainable production, ensuring at the same time the bricks quality and aesthetics.
The studied bricks shown a quite good conservation state. Bricks with a yellowish colour and bricks with an orange hue were observed on the two areas selected. Compositional data stand out the important presence of gehelenite, diopside and anorthite, and hematite and calcite to a lesser extent. This indicates the use of mixtures of clays, illitic-kaolinitic and very calcareous, and firing temperatures of 900–1000 °C at oxidizing conditions. The significative presence of high-temperature mineral phases provided an important durability to bricks and the yellowing of the pastes was mainly due to gehlenite formation.
On the one side, these results might suggest that the knowledge regarding to the manufacturing technologies used could be transmitted over the centuries along the construction of Santa Justina, as well as that late antiquity bricks were reused during medieval times. On the other, they pointed out the important skills achieved at those times to reach such high temperatures. Nevertheless, the mixtures and the firing conditions were not properly controlled, so the formation of the high-temperature phases largely depended on where the bricks were placed inside the furnaces and for how long were remained in.
The data attained could allow the new production of a very high durability bricks from clayey materials outcropped nearby Padua that preserved the aesthetical values of the city. Such new bricks would be especially suitable for the proper conservation of the built heritage of Nothern Italy shaped by bricks made according to Roman techniques.

S5-P27.710

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The clay of the Yucatan Peninsula as a target of archaeometric study of the Mayan ceramics

Guasch-Ferré, N.[1], Doménech-Carbó, Mª.T.[2], Prada Pérez, J.L.[3], Vázquez de Ágredos Pascual, Mª.L.[4], Osete Cortina, L.[2]

[1]Departament d'Arts i Conservació-Restauració, Facultat de Belles Arts, C/ Pau Gargallo, 4, 08028 Barcelona, Catalonia, Spain
[2]Institut Universitari de Restauració del Patrimoni. Universitat Politècnica de València. Camí de Vera s/n, 46022. València, Spain
[3]Grup de Recerca Aplicada al Patrimoni Cultural (GRAPAC). Universitat Autònoma de Barcelona. Carrer dels Til·lers, Facultat de Ciències, Departament de Geologia, Campus de la UAB, 08193. Bellaterra, Catalonia, Spain
[4]Departament d'Història de l'Art. Universitat de València. Adv. Blasco Ibánez 28, 46010. València, Spain

Keywords: clay, ceramic, multi-technical, mineralogy, Mayan area

This contribution presents a multi-technical methodology enabling the characterization the samples of clayey sediments coming from the north of Yucatan Peninsula, from three of the localities where the most recognized outcrops of palygorskite-type clays appear in pre-Hispanic times and today. According to historical and ethnographic evidence, these locations are: *Sak lu'um* cave (white earth), from the community of *Sak lu'um*; cave in the village of Muna, and *Actun Hi* cave, in the locality of *Ticul*.

The study is based on a macromorphological description of the hand samples and a mineralogical characterization of the sediments, as the aim of the study is not the description of the soil or its formation processes in question, but the determination of the mineralogy of the clays and its geological context. For such purpose, the multi-technique approach proposed includes instrumental techniques for characterizing inorganic materials such as optical microscopy (OM), Fourier transform infrared spectroscopy (FT-IR), x-ray diffraction (XRD), and scanning electron microscopy-x-ray microanalysis (SEM-EDX).

The samples from clay sediments extracted from *Sak lu'um* cave are mainly composed by palygorskite together with dolomite and montmorillonite-chlorite. These results show that these samples are part of the primary clays made by direct crystallization group, which is the generic type of clays found in Yucatan Peninsula, composed by talc, chlorite, palygorskite-sepiolite and kaolinite-montmorillonite mixed layers. The samples from clay sediments extracted from different caves from *Muna* and *Ticul* differ from the previous ones and are composed mainly by montmorillonite-chlorite, and in less proportion, by dolomite and palygorskite, respectively. These results indicate that these samples from clay sediments are part of the detritical clays made by the detritus sedimentation, mostly restricted in *polje* mining areas scattered around *Sierra de Ticul*, and composed by smectite clays associated to detrital quartz, iron oxides and, in less proportion, kaolinite.

These results are consistent with the line of research that indicates, on the one hand, the importance of this clay as an ingredient in pre-Columbian culture for the manufacture of pigments in mural painting, sculpture and ceramics and, on the other hand, that these clays are restricted to the northeast of the Yucatan Peninsula, located in sinkholes or *cenotes*.

S5-P28.3022

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

ICP-MS analysis of "Losanga-style" imitations ceramics from South-western Iberia

Beltrame M.[1], Barrulas P.[1], Soria V.[2], Arruda A.M.[2], Mirão J.[1], Schiavon N.[1]

[1]HERCULES Laboratory, University of Évora, Largo Marquês de Marialva, 8, 7000, Évora, Portugal.
[2]UNIARQ, Centre of Archaeology, University of Lisbon, Alameda da Universidade, 1600-214 Lisbon, Portugal

Keywords: ICP-MS; Losanga-style; Campaniense; Ceramics; South Western Iberia; Roman

Within the Italic black gloss tableware production (i.e. Ceramica Campana, Céramica Campaniense or "Ceramique Campanienne" according to its geographical distribution) the so called "Losanga-style" typology is a special grey ceramic production of high socio-historical significance [1]. This pottery typology dates back to the IV and I century BC mainly in the Italic Peninsula. The development of imitations in provincial contexts, especially during the late Republican period, is a consequence of processes driving imports of exogenous goods [2] as well as of local Italic black gloss ceramic productions (imitations). Furthermore, the Roman presence in South-Western Iberia stimulated people mobility and technological transfer with all the Roman empire: the diffusion of "Losanga style" ceramics provides clues to monitor these important archaeological aspects.

Recent archaeometrical analytical using results obtained from the analysis of Italic Late Republican (2nd- 1rst century BC) imitations of black gloss tableware from Portuguese and Spanish (Andalusia) archaeological sites in SW Iberia [3] did not completely clarify a debate, still very much alive, regarding the production of "Campaniense" pottery in SW Iberia. The fairly homogeneous nature of the geological substrate and of the raw materials available in the Portuguese and Spanish sites investigated made it hard to draw unequivocal conclusions supporting a common local production of the potsherds.

In order to clarify the existence of possible imitation production centres of Losanga style ceramics in south-western Iberia and/or to identify possible trade routes, a set of archaeological samples [3], have been analysed by ICP-MS together with several fluvial sediment samples. The sediments samples have been collected from the quaternary alluvial deposits of the river Tagus, left side, in the vicinity of the city of Santarém (Portugal). In this case rare earth elements analysis by ICP-MS is used to assess similarities or differences of the local river sediments with the archaeological samples [3].

[1] PEDRONI, L. (2001): Ceramica calena a vernice nera. Produzione e diffusione. Petruzzi, editore Naples

[2] SÁEZ, R. A. M. (2008). La producción cerámica en Gadir en época tardopúnica siglos III. Oxford, England: J. and E. Hedges.

[3] N. Schiavon, V. Soria, A. M. Arruda, M. Beltrame, J. Mirão, Losanga-style" Imitations of Italic Late Republican black gloss tableware from South-Western Iberia: a multianalytical/microchemical characterization, Microchemical Journal 124 (2016) 712–718

S5-P30.396

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The Performance of Portable X-ray Fluorescence (pXRF) in Investigating Bronze Age Pottery from Kuwait and Bahrain

Ashkanani, H.

Kuwait University

Keywords: Kuwait, pXRF, Dilmun, Arabian Gulf, pottery

Pottery assemblages from the site of Al-Khidr on Failaka Island, Kuwait, and Barbar Temples in Bahrain were analysed to reconstruct the chemical composition of Bronze Age wares and to build a mineralogical database of Bronze Age pottery dated from Failaka Periods 1–3B and Barbar IIb-c (2000–1650 BCE). A total of 145 ceramic sherds from Al-Khidr and Barbar, as well as reference groups, were analysed by non-destructive portable X-ray fluorescence (pXRF) spectrometry. Preliminarily petrographic thin-section analysis was applied to 12 samples to reconstruct possible clay paste recipes and to identify raw materials. The results indicate that performance of pXRF analysis can successfully distinguish subgroups within a typological category of ceramic assemblages non-destructively. The results identified two subgroups within the Al-Khidr typological category: the Dilmun Barbar tradition and the Mesopotamian tradition. By using Bahrian samples, further comparative analysis utilized confirmed the effectiveness of the pXRF result to build reference groups for the Arabian Gulf ceramic studies. The result of this study was also used as a reference, in turn, to further investigation for fingerprinting the assemblage from Kuwait of Late Neolithic Age, known as Ubaid culture in the 6th millennium BC. The performance of pXRF were also compared to ICP-MS to test the reliability of the instrument and suggested to be utilized in the Arabian-Persian Gulf pottery studies. Further compositional studies can be conducted to explore other aspects of craft specialisation, such as ceramic technological choices and possibly the influence of sociopolitical units.

S5-P31.3023

Roman glass from Viminacium and Egeta (Serbia) demonstrates change of glass-trading patterns on Iron Gates during Late Antiquity

Balvanović, R.[1], Šmit, Ž[2], Stojanović, M.M.[3], Spasić-Đurić, D.[4], Špehar, P.[5], Milović, O.[4]

[1]*Laboratory of Physics, Vinča Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, Serbia*
[2]*Jožef Stefan Institute and Faculty of Mathematics and Physics, University of Ljubljana, Slovenia*
[3]*National Museum Belgrade, Serbia*
[4]*National Museum Požarevac, Serbia*
[5]*University of Belgrade, Faculty of Philosophy – Department of Archaeology, Serbia*

Keywords: Glass; Late Roman; Viminacium; Serbia; PIXE-PIGE

We are reporting the composition of thirty-eight Late Roman glass fragments (3rd – 4th c. CE) from Viminacium, the capital of Moesia Province, and Egeta, the fort controlling Iron Gates Gorge on the Roman Danube Limes. The glasses are measured using simultaneous Particle-Induced X-ray Emission (PIXE) and Particle-Induced Gamma-Ray Emission (PIGE).
The compositional analysis shows that sixteen glasses belong to the Roman glass decolourized by antimony, nine to the Roman glass decolourized by manganese, one is recycled using these two types. Five glasses belong to the Foy série 3.2, two to HIMT, one to Jalame-type with manganese. One glass belongs the rare plant-ash type P-1, produced in Egypt. This glass is for the first time reported from the continental Europe.
The comparison of the finds with the contemporary glass from Serbia and the wider Balkans indicates that a marked shift in glass trading patterns occurred between the High Empire and the Late Antiquity. During the High Empire, glass seems to be imported to the central and eastern Balkans mainly from the west, over the Roman road Aquileia-Viminacium and over Adriatic ports. In the Late Antiquity, import was from east to west, over the Danube, Via Militaris or Aegean ports. The change in trading patterns of such a valued commodity as glass may indicate a change of trading patterns of other commodities as well, by implication reflecting not only commercial but also political transformations that occurred in this period.
Another find is the richness of the glass market in Viminacium, indicating that the capital of Moesia province was a cosmopolitan city. This find questions earlier notion of Danubian Limes of the central Balkans being a backward area on the remote and restless Roman border.

S5-P33.3024

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

LA-ICP-MS analysis on late Iron Age glass vessel fragments discovered at the Dacian fortress from Ardeu, Hunedoara County, Romania

Căsălean, C.A.[1], Stremțan, C.C.[2], Pușcaș, C.M.[3], Šelih, V.S.[4]

[1]Museum of Dacian and Roman Civilization, Deva, Romania
[2]Teledyne Photon Machines, Bozeman, MT, USA
[3]Terra Analitic SRL, Alba Iulia, Romania
[4]National Institute of Chemistry, Ljubljana, Slovenia

Keywords: LA-ICP-MS; glass vessels; Dacian; fortress; La Tène

The archaeological literature concerning the Dacian horizon has long neglected the analysis of glass vessels due to their rarity and poor state of conservation. Some bibliographic incursions that addressed the subject analysed the archaeological material through common archaeological methods by framing it typologically, chronologically, and by discussing particularities given by the context of discovery. Whilst analysing only the identifiable fragments of vessels with the help of well-known glass typology studies, most of the authors viewed glass vessels as luxury imported goods originating from the Mediterranean Basin, generally ranging from the 2nd century BC until the beginning of the 2nd century AD, when Dacia became a Roman province.
In this context the main goal of the present study is to give new perspectives regarding 14 glass vessel fragments discovered in late iron age contexts at the Dacian fortress from Ardeu, Romania. We combine the analysis of the archaeological material through the aforementioned archaeological methods with chemical analysis using laser ablation inductively coupled plasma mass spectrometry. The goal is to establish the origin of the raw materials from which the vessels were made, as well as the area where these vessels were manufactured, and details regarding the production technique.
Elemental concentrations (major, minor, and trace elements; 55 elements measured in total) indicate that the samples fall into three chemically distinct categories, potentially connected to different source areas for the raw material utilised in producing the glass; it is however possible that some of the samples were made from recycled glass.
Such detailed analysis opens new perspectives regarding the interactions of the community from Ardeu with other areas, their material preferences, patterns of consumption, supply routes, as well as the purpose and functionality of each analysed artefact within the community.

S5-P36.178

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

What kind of batch recipes have been used in medieval forest glass huts without finished product relicts?

Wilke, D.[1], Stephan, H.-G. [2]

[1]Dr. Wilke Management & Consulting GmbH, 30974 Wennigsen, Germany
[2]Institut fuer Kunstgeschichte und Archaeologien Europas, Archaeologie des Mittelalters und der Neuzeit, Martin-Luther-Universitaet Halle-Wittenberg, 06108 Halle/Saale, Germany

Keywords: wood ash glass; drops drippings blobs; low-Z elements; non-destructive XRF

Archaeologists generally consider find contexts with kiln remains, glass pot fragments (crucibles) and vitreous drops and drippings as glass workshops, regardless whether finished glass relicts have been conserved in an analytically adequate quality. This is a frequent archaeological situation for medieval forest glass huts in Germany dating from the 9th/12th to the 14th century, where the glass composition often did not allow for finished product conservation under humid, acid soil conditions. However, experimental archaeological studies suggest that vitreous blobs in wood-fired furnaces derive from the partial liquefaction of kiln furniture dropping into the ash pit rather than representing actual batch recipes applied in the glass pots. This is a serious limitation for the study and pattern allocation of medieval forest glass huts until the high Ca/low K wood ash glasses predominated from the 15th century onwards. We have thoroughly analysed the waster assemblages of several workshops in the upper Weser region dating from the early 15th to the late 17th century with non-destructive XRF spectrometry under Helium flush using a low-Z sensitive graphene detector device, and must confirm that drops and drippings indeed do not represent the high Ca/low K pattern of the finished glass type. Rather they cluster together with the waster material recovered in the medieval forest glass huts characterized by high K/low Ca ratios. Though the blobs belong to the most significant, often exclusive indicators of early forest huts, the results support the notion that they represent unintentional “artificial” by-products of the high-temperature process, but without indexical value for the thus still unproven glass making process practiced in these medieval workshops.

S5-P39.266

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

(En)Lighting the Past: The Oil Lamps from São João de Tarouca Monastery

Fernandes, A.[1,2], Villamariz, C.[1,2], Cerqueira Alves, L.[3], Wiley, R.[1,2], Vilarigues, M.[1,2], Coutinho, I.[1,2]

[1]Department of Conservation and Restoration, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[2]Research Unit VICARTE, Vidro e Cerâmica para as Artes, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[3]C2TN, Instituto Superior Técnico, Universidade de Lisboa, 2695-066 Bodadela LRS, Portugal

Keywords: Cistercian Monastery, 17th Century, Oil Lamps, Glass, Archaeometry, Production Technology

São João de Tarouca Monastery, of the Cistercian order, was built in 1154, in Lamego (located in the north of Portugal). It houses a Latin cross church with nave and aisles and an echeloned east end. The fact that the church does not have a clerestory means that the lighting of the central nave is not direct, hence the importance of using artificial lighting. This lighting was once performed with the use of oil lamps, which were suspended (together or individually), thus providing the desired lighting for the space.

These lamps were later replaced by other lighting sources, and then buried with other objects that were no longer useful. Along the several performed archaeological excavations that took place in the São João de Tarouca Monastery, more than 90 fragmented suspension lamps dated to the 17th century were recovered. As far as the authors know, this is the largest set of oil lamps from this chronological period retrieved from a Cistercian monastery.

The first approach to the set will be to study the shape and colour variances and create groups according to the differences and similarities observed among the lamps. Some of the shapes will be reproduced in order to study the production methodology of this type of object, which will allow for the discussion about the degree of difficulty during its manufacturing process. Finally, from the previous defined groups, fragments will be selected to be characterize by µ-PIXE and determine the glass chemical composition.

To sum up, the aim of this work is to study the oil lamps, its importance in the monastery structure and daily-life, bringing new data on the lighting habits during the 17th century and focusing on religious buildings. Moreover, the variance in shapes and colours of the glass will be approached, together with the glass chemical composition, in order to relate this set with coeval glass found in Portugal and abroad to finally propose a provenance for these objects.

S5-P40.313

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric study on the production technology of opaque red glass sectilia from the 2nd century AD Lucius Verus villa in Rome

Bandiera, M.[1,2], Verità, M.[3], Lehuédé, P.[4], Cerqueira, L.A.[5], Pinto, J.[6], Saguì, L.[7], Vilarigues, M.[1,2]

[1]VICARTE, Research Unit Vidro e Ceramica para as Artes, FCT/UNL, Campus Caparica, 2829-516 Caparica, Portugal
[2]Dep. de Conservacao e Restauro, FCT/UNL, Campus Caparica, 2829-516 Caparica, Portugal
[3]LAMA laboratory, Iuav University, Venice (Italy)
[4]C2RMF, Centre de Reserch e de Restauration do Musee de France, Palais du Louvre – Porte des Lions 14, quai François Mitterrand 75001 Paris
[5]Centro de Ciencias e Tecnologias Nucleares, C2TN, Campus Tecnologico e Nuclear, Instituto Superiore Tecnico, Universidade de Lisboa, EN10 km 139, 2685-066 Bobadela, Portugal
[6]CENIMAT/i3N—Centro de Investigação em Materiais, Departamento de Ciência dos Materiais FCT/UNL, 2829-516 Caparica, Portugal
7 Sapienza Università di Roma, Rome (Italy)

Keywords: Roman glass – copper-based opacifier– opus sectile – archaeometry

Although the production of opaque red glass date back to the beginning of glassmaking technology, many technical aspects concerning the achievement of the colour are still unclear. A set of 40 opaque red glass *sectilia* was selected from the opus sectile decoration of *Lucius Verus* Villa (2nd century AD) in Rome, in order to investigate the origin of the different red hues, from orange to brown, by a multi analytical approach. This decoration is one of the oldest examples of glass *sectilia* used in the *opus sectile* technique and belong to a period characterized by a paucity of archaeometric analyses. Thus, the investigation of this assemblage is a rare opportunity to study the Roman glassmaking production during the 2nd century AD.
The colorimetric measurements and FORS analyses allowed to identify five different red hues, which were characterized by quantitative chemical analysis (EPMA and PIXE). The nature of the colouring agents and their features were studied with the combination of FEG-SEM, μ-Raman and X-ray diffraction.
The multi-analytical approach allowed to establish differences in composition of the five red groups, while metallic copper nanoparticles and nanometric crystals of cuprite were identified as the main colouring and opacifying agents. A strict correlation between the red groups and the number and size of the colouring agents was evidenced by FEG-SEM examination.
These results provide new information on the sophisticated technology of production of opaque red glass, during the 2nd century AD and underlined the extraordinary skills of Roman glassmakers to control the formation of colour in this tricking glass.

S5-P43.545

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Roman nanotechnology: a new dichroic glass fragment found in Hungary

Fórizs, I.[1], Dévai, K.[2], Mráv, Zs[3], Kovács Kis, V.[4]

[1]*Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Budapest, Hungary*
[2]*MTA - ELTE Research Group for Interdisciplinary Archaeology, Budapest, Hungary*
[3]*Hungarian National Museum, Budapest, Hungary*
[4]*Institute for Technical Physics and Materials Science, Centre for Energy Research, Budapest, Hungary, and Institute of Environmental Sciences, University of Pannonia, Veszprém, Hungary*

Keywords: Roman time, dichroic glass, cage cup, Pannonia, crystallites

The glass making in the Roman Empire was well organized and in a certain level standardized. As usually in big empires supposedly the best artisans were settled down in the capital and worked for the emperor producing very high quality and many times unique objects. Beside the fabulous gold and silver items beautiful, luxurious and unique glass items were produced as well. Among these luxurious glass pieces the most well-known is the cage cup or *vas diatretum*. The cage glass consists of an inner beaker and an outer cage or shell of decoration (usually coloured) that stands out from the body of the cup, to which it is attached by short stems, this way rendering a very spectacular appearance to the cup. In parallel with the cage cup, the even rarer dichroic glass appeared. This glass, which is also called two-colour glass, has got a very special optical character: its colour is pale greenish-bluish in reflected light, while its colour in transmitted light is orange-red.

A new fragment of dichroic glass cage cup came to light from the late Roman high-status villa at Nagyharsány, which is situated in the territory of Pannonia Valeria, near (about 30 km) from the capital of the province (Sopianae). Beside the well-known attributions of the dichroic glasses this piece has got a distinctive feature; optically it consists of three layers parallel to the walls: in the middle there is an orange layer of about 0.2 mm width, while the two outer layers are pale blue in normal light. The three-layered dichroic fragment was found in a banquet hall along with fragments of other luxurious glass vessels fragments (e.g. cage cups, golden glass), so these pieces could have formed part of a luxury tableware set.

A question arises that what is the role of this inner layer, if it has any, in producing the optical properties of dichroic glass? Several methods have been applied for determining physical and chemical characters of the layers. Micro X-ray diffractometry was applied first time for dichroic glass. Long acquisition time (8 to 48 hours) diffractograms proved the existence of Ag and/or Au crystallites in all the three layers. The chemical composition and size of one crystallite was determined by TEM. The type of chemical composition of the bulk glass has been determined as HIMT glass by SEM-EDS. Further trace element and optical measurements are scheduled. The supposed role of the three layers will be discussed in details in the presentation.

S5-P46.630

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Investigating the homogeneity of the chemical composition of a glass bead cargo: the case study of the Gnalić shipwreck, 1583, Croatia

Bonneau, A.[1], Gratuze, B.[2], Batur K.[3], Radić Rossi, I.[3], Auger, R.[1], Delmas V.[4]

[1]*Archaeometry research group and CELAT, Laval University, Quebec City, Canada*
[2]*IRAMAT-CEB, CNRS/Univ. Orléans, Orléans, France*
[3]*University of Zadar, Zadar, Croatia*
[4]*Institut de Recherche en Histoire Maritime et Archéologie Subaquatique, Montreal, Canada*

Keywords: glass beads, Gnalić shipwreck, trace elements homogeneity, LA-ICP-MS, Raman spectroscopy

When investigating the provenance of glass, the chemical composition and the quantification of trace elements are crucial. Compared with data from glass workshops, they enable us to reconstruct trade routes. However, when studying glass bead trade networks during modern times (16th-19th centuries), one data is missing: were beads from a single type, made in a specific workshop homogeneous in term of elemental (trace elements) and molecular composition? This information is crucial to set what can be considered as separate groups or not, especially considering the paucity of archaeological information regarding glass beads making in Europe, from the 16th to the 19th century.

In early November 1583, the Venetian merchant ship known as *Gagliana Grossa* sank close to the islet of Gnalić, Croatia. Departing from Venice, it carried various types of precious objects for the construction of the old sultan palace in Constantinople. Among its cargo, a large number of glass artefacts has been recovered, including windowpanes, fine venetian drinking glasses, and more than 12kg of glass beads. This collection offers a rare possibility to study glass chemical composition from Venice at the end of the 16th century from a single cargo, and assumed to have been made in a short time span.

The glass beads exhibit ten main colors: white, red, black, cobalt blue, turquoise, black, colorless, vine rose and green, as layers of glass or as decorating stripes, which represent 78 different types (according to the Kidd and Kidd typology 1970). From 2 to 5 beads of each type have been analyzed using microscope, Raman spectroscopy and LA-ICP-MS, in order to estimate the homogeneity of the elemental and molecular composition depending on the color of the glass and its use in the making of glass beads (layer of glass or decorating stripes). They were compared to 15 fragments of colored and colorless glass sampled from glass objects recovered from in the Gnalić shipwreck.

Results enable us to start a discussion on the possibility of distinguishing glass provenance during modern times from elemental and molecular composition data.

Bibliography:

Kidd, K.E., Kidd, M.A., 1970. *A classification system for glass beads for the use of field archaeologists*, Canadian Historic Sites: Occasional Papers in Archaeology and History 1

S5-P53.804

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multiproxy microanalysis as a useful tool for reconstruction of the faience manufacturing in Ancient Egypt. A case study of ptolemaic decorated bowl from Athribis (Tell Atrib)

Zaremba, M.[1], Trzciński, J.[2], Welc, F.[1], Południkiewicz, A.[3], Rogulska, M[4], Kaproń, G.[2]

[1]Institute of Archaeology, Cardinal Stefan Wyszynski University, Wóycickiego 1/3, 01-938 Warsaw, Poland
[2]Faculty of Geology, University of Warsaw, Żwirki i Wigury 93, 02-089 Warsaw, Poland
[3]Polish Centre of Mediterranean Archaeology, University of Warsaw, Nowy Świat 4, 00-497 Warsaw, Poland
[4]Faculty of Chemistry, Maria Curie Skłodowska University in Lublin, pl. Marii Curie-Skłodowskiej 2, 20-031 Lublin, Poland

Keywords: light microscope (LM); Scanning Electron Microscope (SEM) with EDS; X-ray diffraction (XRD); Thermal Analysis (TG–DSC–FTIR–MS)

Due to the advanced production technology, in order to obtain a product different from traditional ceramics, Egyptian faience is considered the first high-tech ceramics in the history of mankind. Ptolemaic relief-decorated vessels are one of the most technologically advanced forms made of faience. Previous studies have been mostly conducted using individual research methods. Such approach may causes in errors in the interpretation of the results.

The purpose of this research was to develop a new, multiproxy methodology for analysing faience objects, which would allow for comprehensive reconstruction of their production technology. The use of several complementary research methods gives the possibility of more precise interpretation of the various steps of faience manufacturing. To achieve the goal, detailed archeometric research was undertaken.

A Ptolemaic, relief-decorated bowl, collected during the Polish-Egyptian Archaeological Mission (1985-1995) in the Tell Atrib archaeological site, the central part of the Nile Delta, was selected for analyses.

The following laboratory techniques were used: light microscope, scanning electron microscope, qualitative and quantitative image analyses, mineralogical, chemical and thermal analyses. The investigations were financially supported by the Polish National Science Centre, project “Production technology of faience pottery in Ptolemaic Egypt in the light of archaeological and archaeometric investigations”, grant no. 2017/27/N/HS3/02464.

Selected aspects of procedures of making faience objects were determined, from obtaining the raw material to firing of the item. Quantitative parameters of particle size and pore space of faience body determined with image analysis have been specified by qualitative observations and have supplied numerous data on the processes of faience manufacturing. Raw material was ground to a very fine powder with usage of stone tools with a hardness exceeding that of quartz. The analysed bowl was formed by compressing in two-part mould. The results obtained from XRD confirm and complement the results from EDS. Analyses have shown that in addition to quartz, the faience body has also trace amounts of feldspars and cristobalite. High quality raw materials were used for the preparation of the silica paste. Gas present in the glaze pores was identified as CO_2 and the temperature at which it began to evolve were determined. The analyse faience bowl is characterized by finest quality, pointing to the high competence of the workshops at each production stage. The items were produced in highly specialized workshops with good technical appliances.

S5-P55.214

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Imported ceramics in a Swahili context from the Ibo Island (Northern Mozambique)

Agua, F.[1], Madiquida, H.[2], Fernández, V.M.[3], Villegas, M.A.[1], Ruiz-Gálvez, M.L.[3], García-Heras, M.[2]

[1]Institute of History, Spanish National Research Council (CSIC), Madrid, Spain.
[2]Department of Archaeology and Anthropology, Eduardo Mondlane University, Maputo, Mozambique.
[3]Department of Prehistory, Ancient History and Archaeology, Complutense University, Madrid, Spain

Keywords: Ceramics; Mozambique; Ibo Island; Swahili trade

This contribution introduces the results derived from a characterization study carried out on a set of imported ceramics provided by recent archaeological fieldworks (2015-2017) undertaken in a Swahili occupation of the Ibo Island (Quirimbas archipelago). The present work is being developed in the framework of a Mozambican-Spanish research project on Swahili trade in Northern Mozambique. The main research goals of the study were to contrast some insights into the technology and probable provenance of a set of imported ceramics, mostly glazed, to help for tracing the trade dynamics of this Swahili settlement in the context of the African east coast which is preliminary dated between late 10th and 12th centuries AD. For this purpose selected ceramic samples, attending mainly their glaze colour and characteristics, were characterized by binocular glass, thin-section petrographic observations by polarized light microscopy, X-ray diffractometry (XRD), X-ray fluorescence spectrometry (XRF), field emission scanning electron microscopy (FESEM) equipped with an energy dispersive X-ray (EDS) spectrometer, and UV-Vis spectrophotometry.
Resulting data indicated the presence of two main groups of ceramic materials. One of them composed of very calcareous ceramics (around 20 wt. % of CaO) which were fired at an equivalent temperature higher than 1100 °C due to the presence of anorthite and pyroxene, and the second one composed of less calcareous ceramics (around 7 wt. % of CaO) which were fired approximately at the same temperature for a prolonged period of time that favoured the continuous crystallization of anorthite. The two groups showed, on the contrary, very different glazes in terms of chemical composition. The very calcareous one showed a plant-ash alkaline glaze with, when present, very low lead concentration (lower than 2 wt. %), while the less calcareous ceramics showed a lead-rich or plumbic glaze with contents of PbO between 30 and 48 wt. %. Firing technology and chemical composition of the glazes can be generally compatible with a Middle East provenance and, particularly, with the so-called “Samarra” glazes from Iran, which are known since the 10th century and onwards. The results have thereby served to deepen into the Swahili trade networks in which the Ibo Island operated within the general trends of the African east coast and are in conjunction with provenance of other traded materials present in Ibo such as glass beads.

S5-P58.442

Islamic glazed ceramic from the Iberia South-West: the case study of Mértola (Portugal)

Beltrame M.[1,2], Martínez S.G.[3,4], Schiavon N.[1], Mirão J.[1,5]

[1]*Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Budapest, Hungary*
[2]*MTA - ELTE Research Group for Interdisciplinary Archaeology, Budapest, Hungary*
[3]*Hungarian National Museum, Budapest, Hungary*
[4]*Institute for Technical Physics and Materials Science, Centre for Energy Research, Budapest, Hungary, and Institute of Environmental Sciences, University of Pannonia, Veszprém, Hungary*

Keywords: Mértola; Islamic Ceramic; Glaze Technology; Bone

This study presents the results of an analytical research program on Islamic polychromatic glazed ceramics recovered in the town of "Mértola" (Portugal), located in the Iberia South-West (*Garb al-Andaluz* during Islamic time). It provides the analysis of green and brown, partial *corda seca*, and honey and black glazed ceramics recovered in different areas of the old Islamic town, in addition to different kiln bars with traces of glaze on top. Glazed decorated ceramics have been classified as bowls and jugs, with zoomorphic, epigraphic, naturalist, or geometric glazed decorations, and they are chronologically dated between the second half of the 10th century and the first half of 13th century. Kiln rods were recovered in an Islamic workshop from the middle of the 13th century.
Until recent times, the lack of information regarding the production of Islamic glazed ceramics in the Iberia South-West suggested that they were mainly imported from Sothern Iberia or from the North of Africa. Nevertheless, recent studies have shown that polychromatic glazed ceramics were also produced at Mértola during the Islamic period (Beltrame, 2022), while other were imported. Thus, the final goal of the study is to understand the glaze technology applied at Mértola during the Islamic period. Glazed decorations have been characterized by SEM-EDS and µRaman spectroscopy.
In all cases decorations were applied on a biscuit fired ceramic body using different silica-lead glaze mixtures with a variable content of alkali and alkaline earth metals. Cu, Mn, Fe and Sb oxides were utilized on colored decoration. Sn oxide was utilized as opacificant in most cases, in combination (or not) with unmelted quartz grains.
In many cases, several Ca and P rich particles were observed. Raman spectroscopy confirmed they were small bone fragments. Actually during the microstructural analysis of the ceramic paste many frit fragments were observed. They were enriched on Ca, P, Si, and Pb. In some case bone structures were particularly evident. Observations indicate that bones were deliberately added for frit preparation, and subsequently utilized in the glaze as opacificant.

Beltrame, M. (2022). Caliph: Comprehensive Archaeological and laboratory investigation of Islamic pottery in Portuguese History. PhD thesis in History, Évora University.

S5-P65.749

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multidisciplinary study of early medieval *sparse glazed ware* from Donoratico (Italy)

Fornacelli, C.[1], Briano, A.[2], Chiarantini, L.[3], Volpi, V.[4], Bianchi, G.[2], Giamello, M.[1]

[1]Università degli Studi di Siena, Dipartimento di Scienze Fisiche della Terra e dell'Ambiente, via Laterina 8, 53100 Siena.
[2]Università degli Studi di Siena, Dipartimento di Scienze Storiche e dei Beni Culturali, via Roma 56, 53100 Siena.
[3]Università degli Studi di Firenze, MEMA (Centro Servizi di Microscopia Elettronica e Microanalisi), Dipartimento di Scienze della Terra, Via Gino Capponi 3,50121 Firenze.
[4]Università degli Studi di Siena, Dipartimento di Biotecnologie, Chimica e Farmacia, via Aldo Moro 2, 53100 Siena.

Keywords: Sparse glazed ware, lead isotopes, SEM-EDS, p-XRF, cassiterite, Tuscany

Between Late Antiquity and early Middle Ages, an extensive circulation of lead-glazed pottery was supported in Italy by the intensification of inter-regional trades (via medium-to-long range sea routes) and the increase of regional production centers. In this context, a sub-regional and low-cost mass production, characterized by highly standardized forms and a restricted use of the coating, diffused in Rome and in central-northern Italy (Paroli 1992).
The excavations conduced so far within the Italian peninsula returned a limited number of fragments of this so-called *sparse glaze ware* and the resulting archaeometric investigations consisted of few and isolated case studies (Fortina et al. 2008, Maltoni 2012). In this framework, the castle of Donoratico (Livorno, Italy) returned an exceptional repertory, consisting of more than three thousand fragments belonging to several hundred artifacts.
The mineralogical and chemical evidence suggested the application of a lead compound flux to an unfired non-calcareous ceramic body, in accordance with late Antique and early-Medieval traditions. Portable XRF and SEM-EDS analysis of the ceramic bodies confirmed the use of local clays via the identification of high levels of chrome and the presence of secondary products from the dismantling of the ophiolitic outcrops of the Monti Livornesi (Tassi 2018).
The isotopic investigation of the glazes suggested different sources for PbO, that was frequently applied as a mix of different metal batches. The Pb isotopic record identified the Colline Metallifere District (CMD), southern Tuscany, and a source from the northern districts of Central Europe as the most reliable sources. Tin impurities observed via SEM-EDS in most of the coatings in the form of secondary cassiterite agglomerates were related to the presence of cassiterite veins in the CMD lead sulphide deposits and represented an important marker for the exploitation of CMD lead.
The comparison with other contexts (Arezzo, Lucca, Cosa, Monteverdi and Rome) suggested a sub-regional circulation of knowledges, raw materials and/or finished products. In particular, the identification of cassiterite in some of the samples from Arezzo and Rome opened new perspectives on the exploitation of southern Tuscany poly-metallic deposits at the turn of the millennium.

Fortina, C., et al., 2008, *Archaeometry*, **50**(1), 30–47.
Maltoni, S., et al., 2012, *Journal of Archaeological Science*, **39**(7), 2071–8.
Paroli, L., 1992, *La ceramica invetriata tardoantica e altomedievale in Italia: atti del seminario,* , Ed. all'Insegna del Giglio.
Tassi, E. et al., 2018, *Environmental Science: Processes and Impacts*, **20**(6), 965–76

S5-P66.762

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Technological characterisation of 2nd century AD tripods from the Alpine region, Austria

Borgers, B.[1], Auer, M.[2], Daszkiewicz M.[3]

[1]*University of Vienna, Department of Classical Archaeology*
[2]*University of Innsbruck, Institute of Archaeology*
[3]*ARCHEA, Warszawa, Poland*

Keywords: Tripods; Pottery Technology; WD-XRF; MGR; OM

Sherds of grey tripods with horizontal rim are very common on 2nd century AD Roman sites in the Alpine and wider region of Austria (known as *Noricum*). The tripods measure c. 20-25 cm in diameter, and were wheel thrown. They were used for cooking during the Imperial Roman era. Due to their abundance and homogeneous appearance all over *Noricum*, these tripods hold important potential for studying the technological setting and production organisation during the 2nd century AD, and, in particular, the trade networks between the communities that produced and used them.

A total of 31 sherds have been selected from five sites in *Noricum*, comprising Dölsach (*Aguntum)*, Magdalensberg, St. Peter in Holz *(Teurnia),* Moosham (*Immurium*) and Klagenfurt (*Virunum*). The sites were situated in the Alpine region, and interconnected by roads and waterways. These five settlements had differing histories during the time span considered. More specifically, the site at the Magdalensberg flourished between 50 BC – 50 AD, and ceased to exist when *Virunum* became the capital of *Noricum*. In turn, *Aguntum* and *Teurnia* became cities, while *Immurium* remained a Roman small town.

As part of a larger project, which focuses on pottery regions, we have analysed the 31 sherds of grey tripods compositionally, with the aim of identifying the raw materials and reconstructing the production technology. These grey tripods show a high level of macroscopic homogeneity. Therefore, we have examined their chemical and mineralogical composition, using WD-XRF, MGR, and OM.

Our results indicate that there are several compositional groups among the samples analysed, most of which are confined to one particular site. However, there is also one well-defined group, which occurs on four out of the five sites studied. This suggests that pottery production was organised at a local scale, and in different workshops. However, one workshop seems to have established a lively trade of its tripods in Alpine *Noricum* during the 2nd century AD.

Acknowledgements: the detailed compositional study was financed by the *Aktion D. Swarowski* at the University of Innsbruck.

S5-P67.096

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Regional common wares from the Muslim period at the city of Pollentia (Mallorca, Balearic Islands): recent advances from archaeometric analysis

Fantuzzi, L.[1,2], Cau Ontiveros, M.A.[2,3,4], Mas Florit, C.[2], Tsantini, E.[2], Chávez-Álvarez, E.[5]

[1]Universidad de Cádiz, Spain
[2]ERAAUB, Universitat de Barcelona, Facultat de Geografia i Història, Departament d'Història i Arqueologia, c/ Montalegre 6-8, 08001 Barcelona, Spain.
[3]ICREA, Pg. Lluís Companys 23, 08010 Barcelona, Spain
[4]Chercheur associé, Centre Camille Jullian, MMSH, CNRS/Université Aix-Marseille, France
[5]Universidad de La Laguna (ULL), Facultad de Humanidades, Departamento de Geografía e Historia, Campus de Guajara s/n, Apart. 456, 38200. La Laguna (Tenerife), Spain

Keywords: pottery; Islamic period; WD-XRF; petrography; Balearic Islands

The Roman city of Pollentia is located in the northern part of the island of Mallorca (Balearic Islands, Spain). After having been largely destroyed by a fire at the end of the 3rd century AD, the city survived through the Vandal and the Byzantine periods. At the beginning of the 10th century the Balearics were conquered by Muslim forces. Very little is known of the occupation of Pollentia from this moment. However, since the early years of archaeological investigation, numerous findings of ceramics dated to the Muslim period were attested in different areas of the city.

Recent excavations at the forum area, particularly in the zone occupied by the Late Roman fortification, have uncovered stratigraphic layers with large quantities of common or utilitarian wares dated to the Muslim period. The majority of these consisted of water jars, sometimes with painted decoration, and usually showing an approximately similar form (Type 3A of Rosselló Bordoy). A first preliminary analysis on a few of these jars had suggested the hypothesis of a local/regional provenance, but also pointed to the existence of more than one fabric. In order to better understand the provenance, technology and diversity of these ceramic products, sampling has been recently expanded to include a much larger number and broader range of materials for archaeometric characterization. As a result, in this contribution we present the analysis carried out on 59 ceramic samples found in the Muslim contexts of the site. The aim is to further investigate their provenance, as well as to shed new light on some aspects of their technology of production. For this purpose, an archaeometric characterization was applied using a combination of techniques. These included optical microscopy by thin-section analysis for the mineralogical and petrographic characterization, X-ray diffraction (XRD) for further information on the mineralogical composition, and wavelength dispersive X-ray fluorescence (WD-XRF) for the chemical characterization of the materials.

The results showed the existence of two main compositional groups, along with a series of less common fabrics, which might be associated with a local or regional provenance. Given the scarcity of other similar archaeometric studies on Mallorcan pottery from the Muslim period, this study is an important step towards our understanding of ceramic production and consumption in the island during this historical moment.

S5-P70.209

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Mortaria from Tel Akko in the light of petrography research

Węcławska M., Michniewicz J.

Adam Mickiewicz University in Poznań

Keywords: mortarium; petrography; Tel Akko

Tel Akko is also called Tell el-Fukhar which means ‘‘mound of potsherds’’. Among many types of ceramic vessels, researchers pay attention to mortaria. They are shallow open bowls, with characteristic thick rim, and diameter of about 30 cm. In the southern Levant the earliest mortaria appeared at the end of 8th century BC and has spread out along the southern Phoenician coast becaming more common finds in the layers dated on 7th and 6th century BC. In this research we present result of examination of 21 ceramic sherds from excavations in Tel Akko, of which 13 are dated 7/6 century BC. In that time, Acco was an important trade Phoenician city.

The poster is focusing on the current stage of research which is thin section petrography analysis. Main goal of whole study is to determine provenance of vessels. Additional goal is to document the diversity of mortarium fabric

Thin sections petrography analysis and photographic documentation were made in transmitted light with parallel and crossed polarizers. Support for recognition mineral phases was scanning electron microscope with an EDS detector.

The undertaken research allowed to distinguish at least five fabric groups, of which two note our special attention. The first group (6 samples) has components associated with the areas of occurrence of mafic and ultramafic rocks (serpentinite, olivine, pyroxene, gabbro, peridotite fragments). Such composition of mortaria fabric is attributed to the ophiolite complexes. Previous research indicates the Cypriot origin of this type of pottery. The second group (5 samples) is distinguished by microfossils and silty rendzina soil balls that suggesting a Phoenician trace.

S5-P72.215

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multidisciplinary investigation of ceramic pedestals from Tatetsuki Proto-historic burial mound (Okayama prefecture, Japan)

Gulmini, M.[1], Davit, P.[1], La Macchia, A.[1], Re, A.[2,3], Guidorzi, L.[2,3], Lo Giudice, A.[2,3], Giustetto, R.[4], Tema, E.[4], Angelici, D.[5], Fantino, F.[5], Ferrara, E.[6], Hatakeyama, T.[7], Nozaka, T.[8], Mitsumoto, J.[9], Yamaguchi, R.[9], Seike, A.[9], Matsumoto, N.[9]

[1]Department of Chemistry – University of Torino – via Giuria, 7 – 10125 Torino, Italy
[2]Department of Physics - University of Torino & [3]INFN – Sezione di Torino – via Giuria, 1 – 10125 Torino, Italy
[4]Department of Earth Science - University of Torino – Via Valperga Caluso, 35 – 10125 Torino, Italy
[5]Tecnart s.r.l – via Modena, 58 – 10124 Torino, Italy
[6]Istituto Nazionale di Ricerca Metrologica – Str. delle Cacce, 91 – 10135 Torino, Italy
[7]Information Processing Center – Okayama University of Science – Ridaicho – 700-0005 Okayama, Japan
[8]Department of Earth Sciences - Okayama University – 3-1-1 Tsushima-naka, Kita-ku, Okayama 700-8530, Japan
[9]Department of Archaeology - Okayama University – 3-1-1 Tsushima-naka, Kita-ku, Okayama 700-8530, Japan

Keywords: Late Yayoi; Burial mound; Pottery; Japan;

Burial mounds with various shapes, albeit with distinctive local variations, were built in Japan during the Late Yayoi period (50 - 250 CE). They are called funkyubo and provide evidence of the relevance of the buried individuals. The different local features of the funkyubo evolved then in the keyhole-shape mound during the following kofun period (250 - 646 CE).
Peculiar ceramic pedestals (kidai) and jars (tsubo) were found over several funkyubo in the Okayama region and they are considered as the prototype of haniwa, the pottery figures that were placed over kofun mounds in the subsequent period.
This study considers shards of kidai found on the Tatetsuki mound (Kurashiki City, Okayama Prefecture, Japan), which is one of the most significant examples of Late Yayoi megalithic tomb. This mound dates back to the 2nd century CE and has a unique circular architecture with two square-shaped protrusions of about 83 meters in length. According to archaeological evidence and some preliminary thin sections analysis, the fabric of the fragmentary pedestals suggests that different raw clays were used for their manufacture.
The topic is further considered in this work by a large international and multidisciplinary team within the activity of the MSCA-RISE project BE-ARCHAEO (grant No. 823826). The investigation considers both the issue of (possibly) identifying the location of the raw materials used to manufacture the kidai, and of investigating production techniques. Ten ceramic shards, representing various macroscopic fabrics, were subjected to a comprehensive investigation including stereomicroscopy, XRF, thin-sections microscopic studies, SEM-EDS, SEM-WDS, XRD and rock magnetic analyses. Thermoluminescence dating is envisaged on four fragments.
Elemental analyses indicated that all fragments are low calcareous; within this general frame (at least) two groups can be distinguished, as two shards show higher Al2O3/SiO2 levels and lower K2O content with respect to the other shards. The fabrics consist of 40 - 50 vol% of sub-angular mineral inclusions (0.1 - 2.0 mm in size) set in a brownish clay-rich matrix. The inclusions are dominantly quartz and feldspar with minor amounts of green amphibole. In addition, dark-brown clay inclusions are conspicuous in the group with higher Al2O3/SiO2 and lower K2O contents. Microscopic analyses and SEM-WDS investigation evidenced the development of liquid phases during firing, spreading into the porosity of the clay body.

S5-P73.261

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

First study on Roman handmade pottery at Tamuda (Tetouan, Morocco): new insights into their provenance and production technology

Fantuzzi, L.[1,2], Bernal-Casasola, D.[1], Cau Ontiveros, M.A.[2,3,4], Bustamante-Álvarez, M.[5]

[1]*Universidad de Cádiz, Spain*
[2]*Equip de Recerca Arqueològica i Arqueomètrica de la Universitat de Barcelona (ERAAUB), Universitat de Barcelona, Spain*
[3]*Institució Catalana de Recerca i Estudis Avançats (ICREA), Barcelona, Spain*
[4]*Chercheur associé, Centre Camille Jullian, MMSH, CNRS/Université Aix-Marseille*
[5] *Universidad de Granada, Spain*

Keywords: petrography; handmade pottery; cooking wares; Roman pottery; Morocco

This study presents the analysis of a Roman assemblage of handmade cooking wares, found at the ancient settlement of *Tamuda* (Tetouan, Morocco). Excavations conducted in recent years at this archaeological site uncovered large quantities of handmade pottery in ceramic contexts dated to the 2nd century AD. Findings of handmade ceramics dated to the Roman Empire period are unusually rare in the western Mediterranean, compared to the higher abundance of such vessels observed in the Late Roman period, therefore this study provides an unique opportunity to gain insight into a poorly known aspect of ceramic production and consumption at the Roman *Mauretania Tingitana*.

The macroscopic characteristics of these ceramics suggest that they were produced locally or regionally. However, no studies were carried out on handmade wares from the site in the past, resulting in an absence of published reference materials for comparison. In order to carry out a first characterization of the materials, 33 ceramic individuals were sampled for archaeometric analysis. This paper focuses on the petrographic analysis of these vessels, performed by means of thin-section optical microscopy. The selected samples are mostly cooking pots, usually fired in a reducing-oxidizing atmosphere, and in some cases with applied or incised decoration. Based on the analysis of petrographic fabrics, the aim of this research is to examine the hypothesis of a local or regional provenance and to shed light on some technological features in the production of these handmade wares.

The petrographic study indicates the existence of a diversity of fabrics and products, higher than the one suggested by the macroscopic analysis. For some of the main fabrics a local production seems plausible, however other fabrics might be associated with a broader regional provenance. This research is a significant contribution towards a better understanding of the handmade pottery that was produced and consumed in the northern Moroccan Rif in the Roman period.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometry of Medieval Pottery from Basilicata

Annunziata, E.M.[1], Di Leo, P.[2,3], Sogliani, F.[2], Medici, L.[3], Lettino, A.[3]

[1]PhD fellow, DiCEM – Department of european and mediterranean Cultures, University of Basilicata, Matera, Italy
[2]School of Specialization in Archaeological Heritage, SSBA DiCEM – Department of European and Mediterranean Cultures, University of Basilicata, Matera, Italy
[3]Institute of Methodologies for Environmental Analysis - National Research Council, Tito Scalo (PZ), Italy

Keywords: medieval pottery; archaeometry; Basilicata

The present work is aimed to delineate the production and circulation system of pottery products in Basilicata during the Middle Age. The research is an integral part of the CHORA project - Archeology Laboratories in Basilicata, funded to the School of Specialization in Archaeological Heritage, University of Basilicata, Matera (MT). By understanding the economic systems through the analysis of raw material/finished products circulation and integrating this information with climate and landscape evolution, it will be possible to decipher dynamics of lucanian medieval sites in the diachrony. The present study focuses on archaeometric analyses of the medieval pottery from the excavation contexts of S. Maria d'Anglona, Moliterno and Satrianum. Samples of natural clays outcropping in the area surrounding the archaeological sites were also analysed and subjected to experimental firing tests. The work systematically addresses the issue of the production and circulation of Middle Ages ceramic artefacts in the regional context of Basilicata using an interdisciplinary approach that implies the integration of the archaeometry to the study of archaeological finds. The systematic study was conducted on about 100 fragments of pottery from three distinct medieval Lucanian archaeological sites, i.e. Satrianum, the Castle of Moliterno and Santa Maria d'Anglona. Tempers and coatings of the artifacts were compositionally characterized using conventional and destructive/non-destructive analytical techniques and compared with clay rich deposits outcropping in Basilicata - as possible raw materials - and with firing tests. The archaeometric approach has made it possible to return an articulated picture of the presence of productions in the regional area, that during the Middle Ages circulated on a geographical scale, including extra-regional ones, thanks to a terrestrial and river communication network. The integration of the results of the archaeometric analyzes, of the firing tests, the geolocation of the sampled outcrops and the identification of the geographical relationships with the archaeological sites made it possible to circumscribe any local productions, define the circulation of raw materials and know-how in the diachrony in a territory, such as that of Lucania, rich in archaeological evidence, as well as densely man-made from prehistoric times to the Middle Ages.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Fibre Optics Reflectance Spectroscopy for the differentiation of clays from ceramics. The case study of Kastrouli, Greece

Kaminari, A.[1], Liritzis, I.[2], Alexopoulou, A.[1]

[1]*University of West Attica*
[2]*University of the Aegean*

Keywords: Fibre Optics Reflectance Spectroscopy; ceramics; visible near-infrared spectra

The present research refers to the spectroscopic analysis of 105 ceramic fragments from the excavation site in Kastrouli, in Greece. Kastrouli, a contemporary name of the location, is situated 11km away from Delphi, in Fokis and it is a Mycenaean settlement in which every summer excavations are held in order to promote archaeological and archaeometrical expertise. A stone built chamber was excavated to the west part of the fortified area contained a burial of at least 15 people. The main pottery shape is the arm vase and small findings include phis- (Phi) and psis- (Psi) shaped figurines, steatite beads and gold leaf fragments.
The ceramic fragments and figurines bear colour marks and decoration. Fibre optics reflectance spectroscopy (FORS) was employed in order to study mainly the different colours of the clays rather than the colours on the fragments. The purpose of the study was to search whether and to what extent this spectroscopic technique could help researchers distinct and categorize clays of ceramics and pottery. This might be used as an indication to help archaeologist link the type of clay to the geographical origin of ceramic findings, vessels and figurines.
FORS uses fibre optics to obtain both spectroscopic and colourimetric information: through specific calibrations, spectra in the visible and near infrared region (400-1000nm) can be obtained as well as tristimulous values pertaining to any colourimetric system the researcher wants to choose. Apart from being non-destructive and non-invasive, this technique has the advantage of examining a tiny area, ensuring that data come only from the selected part of the object without background interferences. Ceramic fragments and figurines were examined regarding the colour of the clay and the presence of pigments on their surface. Both spectra from visible to near infrared region and CIE Lab measurements were obtained. Even though examination with naked eye could differentiate the clays, colorimetric measurements were inconclusive, with data spreading illogically all over the 3-D Lab axes. No patterns or groups could be located. However, spectroscopy helped immensely in categorizing the clays, as the morphology of the spectra could indicate the general group in which the clay of each object belonged and consequently to form sub-categories depending on the lightness or darkness of the clay. The spectra of pigments were also recorded for a potential database as they were few and could not be categorized.

S5-P77.465

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Wine economy in Picton Territory (1st - 2nd Centuries AD) Study of the locally produced amphorae

Frugier, C.[1,2], Lemaître, S.[1], Pianet, I.[2], Cantin, N.[2]

[1]HeRMA, Hellénisation et Romanisation dans le Monde Antique, UR 15071, Université de Poitiers, Hôtel Berthelot, 24 rue de la Chaîne, 86 000 Poitiers.
[2] Archéosciences Bordeaux, Matériaux, Temps, Images, Sociétés, UMR 6034, CNRS, Université Bordeaux Montaigne, Université de Bordeaux, Maison de l'archéologie, Esplanade des Antilles, 33607. Pessac Cedex

Keywords: Roman Antiquity; Picton amphorae; provenance study; petrography; WDXRF

In Picton territory, western centre of Gaul, amphorae were mainly imported from Italy, north-eastern Spain, or – to a lesser extent – from the Middle East between the 1st and the 2nd Century AD.

However, in several Picton archaeological sites, the presence of locally produced amphorae was evidenced. Furthermore, the existence of regional workshops, imitating winery amphorae originating from the Tarraconensis province (Dressel 2/4, Pascual 1), and from the Narbonensis province (Gauloise 4 and 5), is also well attested in Picton territory and its surroundings[1,2].
To deepen our understanding of the past wine economy in the region, we studied the productions of two Picton workshops: Gourgé – Le-Bourg-est (Deux-Sèvres) and Naintré – Les Groseilliers (Vienne). Geochemical analyses (WDXRF), macroscopic and petrographic observations were carried out on the amphorae they produced, as well as on a wide selection of common wares.
To appreciate the economic weight of these two workshops, we attempted to determine the provenance of the local amphorae distributed and consumed throughout Picton territory. Using the methodology described above, we examined the regional vessels coming from four archaeological
sites: « rue de la Marne » and « îlot des Cordeliers », Poitiers (Vienne), *Rauranum,* Rom (Deux-Sèvres) and « le Gué-de-Sciaux », Antigny (Vienne). We present here some of the results we already obtained.
In addition to this work, we intended to verify if the Picton amphorae contained wine as their typology suggests. We implemented a methodology to analyse the organic residues preserved in the vessels pores and we applied it on the amphorae originating from Poitiers. The results are exhibited in a second poster.

[1] Brunie, D., 2008. *Gourgé, le bourg-est : fouille d'une agglomération secondaire des Pictons*. Série fouilles préventives. INRAP - GSO, Poitiers.

[2] Durquety, M., Thirion-Merle, V., Schmitt, A., 2012. Production de céramiques du Haut-Empire dans l'Est du territoire picton : les exemples des ateliers de Gourgé (Deux-Sèvres) et de Naintré (Vienne). *Echanges et société en Gaule : les céramiques en territoire picton (IIe s. av. J.-C.-VIe s. apr. J.-C.), Actes du congrès de Poitiers, (17-20 mai 2012)*. Société Française d'Etude de la Céramique Antique en Gaule (SFECAG), 105-134

S5-P78.487

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometry studies on an Early Mycenaean pottery assemblage from Thebes, Greece

Valantou, V.[1], Zacharias, N.[1], Fappas, I.[2], Aravantinos, V.[2]

[1]Laboratory of Archaeometry, Department of History, Archaeology and Cultural Resources Management, University of the Peloponnese, 24100 Kalamata, Greece
[2]Ephorate of Antiquities of Boeotia, Threpsiadou str. 1, 32200, Thebes, Greece

Keywords: pottery; technology; provenance; Thebes; Mycenaean archaeology

Pottery fragments as the commonest finding in archaeological excavations play an important role as respond to questions about human activities and various aspects of culture. Further, by employing archaeometry procedures valuable information is resulted about technological and provenance issues. The city of Thebes, located in Boeotia, Greece, was an important Mycenaean centre during the 14th and 13th century B.C. (Aravantinos, 2012). Continuous excavations within the city walls have revealed evidence of the Mycenaean occupation in an extensive area now covered by the contemporary city of Thebes. Architectural features of a large Mycenaean palace were unearthed including large amount of pottery fragments. During these excavations a large amount of vessels and buried material of the 17th century B.C., were found in a waster pit on the top of Kadmeia, palace area. Probably the pottery was used for symposium, as all the shapes belong to tableware pottery with few storage vessels. This pit also included bone fragments of domestic animals and incised fragments of antlers, evidence of workshops wastes. As the pit was centrally situated above the top hill, probably was used as a discharge area of various human activities.
Here we present the results of the chemical and technological analysis of an assemblage of 73 samples, most of them fineware vessels (cups, goblets, plates, amphorae etc.) some bearing painted decoration and a small amount of more courseware samples (jugs and jars), using a combination of optical microscopy (OM), petrography analyses (PA), portable X-ray Fluorescence (p-XRF) and Scanning Electron Microscopy coupled with Energy Dispersion X-ray Analysis (SEM/EDS) techniques. Resulted data provide evidence that the majority of the samples show a local provenance according to the area's geology and compositional differentiations are observed within statistical variations. Finally, all fragments show a good quality wheel-made pottery.

Aravantinos, V. and Fappas, I., 2012. "A new Mycenaean palatial building from the Kadmeia acropolis of Thebes"), in: A. Mazarakis-Ainian (ed.), Proceedings of the 3rd Archaeological Conference on the Archaeological Work in Thessaly and Central Greece, Volos, March 12-15, 2009. Volos, 929-943 (in greek: «Νέομυκηναϊκό ανακτορικό κτήριο από την Καδμεία (Θήβα)»).

S5-P80.557

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Pottery as an indicator of economic integration of the Greek countryside: The case of the Mazi Plain in the Late Roman and Post-Roman periods (Northern Attica)

Liard, F.[1], Kondyli, F.[2]

[1] *IRAMAT-CRP2A, UMR5060, Université Bordeaux Montaigne, Pessac, France*
[2]*McIntire Department of Art, University of Virginia, Charlottesville, USA*

Keywords: Ceramic petrology; Latin Greece; Thebes; Chalkida; glaze analysis

In Greek archaeology, questions pertaining to social and economic exchanges mostly focus on cities as regional centers of political power, economic growth and administration. The role of rural settlements has been given scant attention, especially where archaeometric approaches to the provenance of goods and commodities are concerned. However, rural sites represent important consumers and suppliers of foodstuff, raw materials and artisanal products. After the Fourth Crusade in 1204, several formerly Byzantine territories of Greece passed in the hands of various Latin groups. Feudal norms and traditions were introduced in Greece, and this political change is often seen as reinforcing the economic reliance between a city and its hinterland.

This article explores the socioeconomic organization of hamlets and villages, their sustainability and their relationship to urban centers during the late years of the Byzantine domination (12th c.) and under the Latins (14th c.). The chosen approach is a provenance analysis of plain utilitarian pottery and lead-glazed tablewares discovered in the Mazi Plain, a fertile mountain valley located 25 km south of Thebes (Boeotia) and 70 km northeast of Corinth (Peloponnese). The region came under Latin control in 1204, with the Frankish Duchy of Athens and Thebes covering the modern demes of Boeotia and Attica. The Mazi Plain is part of the Duchy. It is located on a land route of commerce that linked Constantinople with Thessaloniki, Thebes, Elefsina, and eventually Corinth and the Peloponnese. There is archaeological and historical evidence for artisanal production at Mazi in the late medieval period.

Pottery provenance is examined through the petrographic analysis of 82 samples collected during surface prospection in the Mazi Plain and complemented with SEM-EDS analysis of the glazed decoration. Petrographic comparisons are proposed with pottery discovered during excavation at Thebes and Corinth, and with clay-rich sediments collected in Boeotia, Attica and the Peloponnese. We identify strong reliance on pottery imports at Mazi, with lead-glazed tablewares originating from the Corinthia, Euboea and Boeotia, and utilitarian pots imported from a thriving pottery industry at the city of Chalkida (Euboea). Pottery products from Italy and the Latin East remain rare. These supply strategies recall those identified at Thebes, although some specificities do exist and highlight the relative independency of Mazi from the urban center.

S5-P82.574

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometrical investigation of figurines from the ancient city of Akanthos, in Chalkidiki

Trakosopoulou-Salakidou, E.[1], Vivdenko, S.[2], Michalopoulou, V.[3], Vasileiadou, A.[4], Nasioka, O.[5]

[1]Greek Ministry of Culture, Emeritus Director of the former 16th Ephorate of Prehistoric and Classical Antiquities
[2]Greek Ministry of Culture, Ephorate of Antiquities of Thessaloniki Region
[3]Greek Ministry of Culture, Archaeological Museum of Thessaloniki
[4]Aristotle University of Thessaloniki, Department of Chemistry, Laboratory of Analytical Chemistry
[5]Greek Ministry of Culture, Ephorate of Antiquities of Chalcidice and Mount Athos

Keywords: Clay figurines; (PLM); SEM-EDX; m-FTIR; HPLC

In this work, the long-standing archaeometric research on figurines, which were discovered during the excavations of ancient Akanthus cemetery in Chalkidiki, is summarized. The excavations carried out under the supervision of the former 16th Ephorate of Prehistoric & Classical Antiquities.
Specifically, the materials and techniques used for the manufacture of the figurines are investigated in detail: the clay of the body is examined, the firing conditions are described, and the basic components of the preparation and the applied pigments are identified. The applied characterization techniques include: the visual observation of the collected samples with a Stereomicroscope and the examination of thin cross-sections with a Polarized Light Microscope (PLM).
At first for the identification of the colourant pigments (the minerals responsible for the colour) their optical properties, colour, pleochroism, relief, refractive index, birefringence, polarization colour, extinction etc., were examined using PLM. Additionally, the physico-chemical analysis of the above-mentioned samples was applied by means of m-XRF, SEM-EDX, m-FTIR, m-Raman, XRD and HPLC.
The results of the complementary analyzes on the figurines are compared with the archaeometric data from figurines and paintings of monuments of various sites in Macedonia (North Greece).
Conclusively the following inorganic pigments were identified: Calcite-Chalk, Red ochre- hematite, Vermilion cinnabar, Yellow ochre-goethit, coal, Egyptian blue. On the rose organic colourants the Rubia peregrina L. (wild madder) was identified. Furthermore, difference between clays derived not only from different typological group but also from the same, was defined.

S5-P83.586

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Analytical investigations of archaeological pipes from Nditam (Centre Cameroon, West central Africa)

Epossi Ntah, Z.L.[1], Cultrone, G.[2], Mache, J.R.[3]

[1]University of Yaounde 1, Department of Arts and Archaeology
[2]Uinversity of Granada, Department of Mineralogy and Petrology
[3]Uinversity of Yaounde1, Department of Inorganic Chemistry / University of Liege, Department of Geology

Keywords: Archaeological pipes; XRF; XRD; TG; Technology

Nditam is located in the Tikar area, central part of Cameroon. It is an important archaeological site because Tikar people produced many artefacts (mainly ceramics and metals) and archaeologists are interested in their production techniques. This research is part of the first archaeometric project in the Tikar area and is focussed on the technological aspects of the pipes uncovered in Nditam (2000-3000 BP). Mineralogical, thermal, chemical and physical analyses of eleven fragments of pipes have been carried out by using X-ray Diffraction (XRD), X-ray Fluorescence spectrometry (XRF), thermogravimetric analyses (TG) and colorimetry. Three mineralogical groups were distinguished. From the mineralogical point of view, group 1 is composed of seven pipes and contains quartz, kaolinite, biotite, K-feldspar and plagioclase. Group 2 (three samples) is characterized by quartz, biotite, K-feldspar and plagioclase Group 3 (one sample) presents kaolinite, quartz and K-feldspar. Hornblende and brookite were also detected as accessory minerals in some samples. The chemical composition revealed a quite homogeneous composition with the exception of the sample from group 3. TG analyses showed the decomposition of kaolinite with an important weight loss between 400 and 500 °C for some pipes and between 500 and 600 °C for other samples. The estimated firing temperature seems not exceed 500 ºC or 600 °C for the pipes containing kaolinite, whereas a range of 600 ºC to 900 °C was assigned to the pipes from group 2.The existence of oxidising and reducing atmospheres is deduced due to the presence of the red core in seven samples and black cores in others.

S5-P84.590

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Documenting the Pot: 3D recording techniques on 16th century ceramics from Alcazar de Najera (La Rioja, Spain)

Lopetegi Galarraga, A.[1], Valle Melón, J.M.[1], Ortega Bretón, J.D.[1], Rodríguez Miranda, A.[1], Ceniceros, J.[2], Iñañez, J.G.[3]

[1]LDGP, Faculty of Engineering, University of the Basque Country (UPV/EHU), Vitoria-Gasteiz, Spain
[2] Museo Histórico Arqueológico Najerillense, Nájera, Spain
[3]GPAC, Built Heritage Research Group, Faculty of Arts, University of the Basque Country (UPV/EHU), Vitoria-Gasteiz,Spain

Keywords: 3D, laser scanning, pottery

A substantial part of the knowledge about pottery items is tied to their geometry (size, shape, thickness…) and visual appearance (color, coating, decoration…). Actually, this information plays a key role when it comes to define and understand aspects such as production processes, typologies and contexts of use.
In recent years, significant technological advancements with regards to the methodologies for recording and representing the geometry of the objects have given way to new possibilities concerning the resolution of the three-dimension models, their geometric accuracy, execution time or printing options. In parallel, the new requirements regarding the distributed used of information are stressing the importance of the preservation, cataloguing, dissemination and re-use of the 3D models.
In this text, these points are developed in the context of the modeling of a collection of pottery items from the Museo Najerillense (La Rioja, Spain). This collection is formed by the relevant ceramic set unearthed during the archaeological excavations carried out on the Alcázar of Najera, a 16th century fortress and palace that belong to the duke of Najera. This 16th century ceramic set includes mainly tableware, which would have been presumably used by the noble families that were settled in the fortified palace during that period. Three-dimensional models were generated by means of a combination of photogrammetric techniques and the use of different types of laser scanners, the method of recording was determined by the characteristics of each piece, such as: dimensions, shape, color, roughness, reflectivity, etc. Finally, the text goes through the benefits given by these models to the proposed research and the establishment of a 3D database that can be accessible to the scientific community.

S5-P92.722

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Production and Distribution of Prehistoric Ceramics along the Apalachicola River in Northwest Florida (USA): Non-Destructive Analyses by pXRF

Tykot, R.H., White, N.M., Lockman, M.

Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: ceramics trade, prehistoric Florida, pXRF analysis

Elemental analysis using a portable X-ray fluorescence (pXRF) spectrometer was performed on Native American ceramics from eight Middle Woodland burial mound sites along the Apalachicola and Chipola Rivers in northwest Florida. The Middle Woodland period (ca. 1-650 CE) saw the height of burial mound ceremonialism in the Apalachicola region. Pottery samples selected for analysis were of both Swift Creek and Weeden Island-series types, which occur mostly together in the region. More than 60 sherds from the burial mound at Richardson's Hammock (8Gu10) were tested, plus 95 were selected for comparative purposes from the mounds at Chipola Cutoff (8Gu5, 10), Gotier Hammock (8Gu2, 10), Jackson (8Fr15, 12), Michaux Landing (8Li6, 10), Pierce (8Fr14, 23), Poplar Spring (8Ja138, 19), and Porter's Bar (8Fr1, 11).
These non-painted wares were analyzed on both inside and outside surfaces to test for any heterogeneity. A Bruker Tracer Vi was used with settings of 50 kV, 35 A, and 30 seconds, and a filter to reduce mid-range background and enhance the results for trace elements Rb, Sr, Y, Zr, Nb, Ba, and Th as well as major/minor elements including Fe. Quantitative values were produced using widely shared calibration software for these elements. Principal components analysis of the multi-element data show that the ceramics fall into multiple group ranges, with most of the ceramic samples tested most likely coming from clay sources along the river near each site, but many examples of non-local ceramics were also identified. The analysis of such a large number of artifacts from 8 different sites in northwest Florida allows us to determine the range of compositional data along the Apalachicola and Chipola Rivers. The ability to assign outliers from some sites with the mainstream composition for other sites allows the identification of prehistoric routes of trade and/or transport, as well as what must be ritual exchange, and thus the socioeconomic and potentially religious practices within this region, which extends at least 100 river miles from the interior to the Gulf Coast. Further investigation assesses whether there are patterns based on ceramic type and decoration, and the contexts in which these artifacts have been found. Swift Creek and Weeden Island have come to be used as terms representing archaeological "cultures," but this research helps demonstrate that they are simply temporally overlapping ceramic styles within the wider Middle Woodland "interaction sphere" that stressed elaborate and unusual artifact forms in both burial contexts and daily life.

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Crafting choices, mobility and pottery circulation in the Aegean Middle Bronze Age (2000-1750 bc): the case of Heraion on Samos

Kouka, O., Menelaou, S.

Department of History and Archaeology, Archaeological Research Unit, University of Cyprus, 20537 CY - 1678 Nicosia, Cyprus

Keywords: Pottery; Provenance; Petrography; Samos-East Aegean; Middle Bronze Age

Archaeological investigations conducted by the German Archaeological Institute and the University of Cyprus at the sanctuary of Hera on Samos have revealed the hitherto unknown prehistoric past of the island. More particularly, excavations carried out between the 1950s and 1980s, as well as renewed fieldwork undertaken by the University of Cyprus in 2009-2013 have brought to light a rich architectural sequence that demonstrates a continuous habitation at the site from the Chalcolithic (4500-3000 BC) through the Early (3000-2000 BC) and the Middle Bronze Ages (2000-1750 BC).

The multi-level study and analysis of pottery from the earlier prehistoric strata at Heraion, as part of 'The Prehistoric Settlement at Heraion on Samos (Sacred Road)' project between 2009 and 2018 within the framework of an integrated programme of research, has led to the development of a holistic understanding of how the production, consumption, and circulation of pottery during the early history of the insular settlement has evolved through time, bringing to light evidence for the mobility of finished products, knowledge, and people.

Building on previous archaeological and archaeometric observations, we aim to extend that project chronologically to include also the analysis of the pottery assemblage dating to the Middle Bronze Age using a combination of macroscopic and petrographic examination of a large number of ceramics. Ultimately, this paper considers the ceramic materials as markers of human mobility in the East Aegean during a period of intense communication networks in the eastern Mediterranean and the Aegean basin in particular. Focusing on a single island site more meaningful and well-constructed arguments can be reached regarding the definition and re-definition of how things move. Islands, as well-defined spaces provide useful units of study in order to understand the changing nature of insularity and connectivity. Based on preliminary results of this project and the determination of provenance a number of imported vessels have been identified, reflecting the Heraion's interactions with a number of micro-regions through maritime travel and exchange of ceramic products and presumably their contents with the western Anatolian littoral, Mainland Greece, the Cyclades, and Crete. The advantageous location of Samos opposite the Maeander Delta, and on natural sea-routes that linked the eastern with the western and souther Aegean, would explain its role as an important node of maritime trade for the emerging 'Minoan Thalassocracy' of the Cretan Old Palaces (1900-1750 BC).

S5-P95.785

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Inference of composition and production technology of archaeological ceramic artifacts based on a multi-analytical approach

Kazakou T.[1], Pavlidou E.[1], Chrissafis K.[1], Kambanis P.[2]

[1]Section of Solid State Physics, Department of Physics, Faculty of Sciences, Aristotle University of Thessaloniki, 541 24, Thessaloniki, Greece

[2]Museum of Byzantine Culture, 2 Stratou Avenue, 546 40, Thessaloniki, Greece

Keywords: ceramics; SEM; XRD; FTIR; TG/DTG-DTA

The present study is a multi-analytical approach on the characterization of different archaeological ceramic artifacts that belong to the Museum of Byzantine Culture in Thessaloniki, Greece, and date from the early Byzantine era to the time of the Ottoman Empire.

In particular, the supplementary techniques of Optical Microscopy, Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR), were used complementary for the determination of the morphological, elemental and mineralogical characteristics of the ceramics, in order to define the nature of the raw material and the technology of the ceramic production (firing temperature and atmosphere). The information derived on their composition and production conditions was validated by SEM observations concerning the extent of vitrification. The minimum concentration of $CaCO_3$ in the original ceramic paste and the highest firing temperature were estimated by Thermogravimetric Analysis (TG/DTG-DTA).

As expected, the preliminary results indicated that the studied objects, half of which date back to the 6th century A.D. and the other half to the 17th century A.D., exhibit diversity in the composition of the raw materials, as well as the firing process applied for their manufacture. The firing temperatures were estimated in the range from 600º to 850º.

S5-P100.848

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Stability of archaeological ceramic finds in CaO-SiO2-Al2O3 system

Dvořáková, P.[1], Kloužková, A.[1], Kolářová, M.[1], Kohoutková, M.[2]

[1]Department of Glass and Ceramics, University of Chemistry and Technology, Prague, Technická 5, 166 28 Prague, Czech Republic
[2]Central Laboratories, University of Chemistry and Technology, Prague, Technická 5, 166 28 Prague, Czech Republic

Keywords: CaO-SiO_2-Al_2O_3 ceramic system; rehydration/rehydroxylation; anorthite; vibrational spectroscopy; X-ray analytical techniques

Archaeological ceramics made of clay-rich raw materials are often exposed to rehydration/rehydroxylation processes due to influence of humid environment and consequently moisture expansion of ceramic bodies occurs. The extent of irreversible moisture expansion is mainly affected by the type of clay minerals in a plastic component of a ceramic body. The aim of this paper is to evaluate the influence of a Ca-component on stability of archaeological ceramic materials exposed to humid environment.

Faience objects were selected from archaeological finds from the locality of Hradčany district in Prague. These faiences include imports from Bayreuth, Nuremberg and Italy and also local Czech production. Model ceramic bodies from limestone, feldspars and various clay materials were prepared on the basis of parameters of archaeological ceramic bodies (especially bulk density, weight absorption, apparent porosity and apparent density evaluated according to standardized method) and results of X-ray analyses (XRF, XRD) of archaeological bodies. The ratio of CaO, Al_2O_3 and SiO_2, influences formation of secondary phases (anorthite, gehlenite, mullite or wollastonite). Fired model ceramic samples were exposed to hydrothermal treatment to simulate aging of ceramic bodies due to the effect of humid environment. The rate of rehydration/rehydroxylation was determined by the combination of X-ray analyses (XRD, XRF), thermal analyses (dilatometry and STA) and infrared spectroscopy (ATR, DRIFTS).

The results of model samples showed significant influence of mineralogical composition on their final mechanical properties. It was proved that the major influence on properties, especially strength and shrinkage, had the presence of anorthite in a ceramic body. The calcium component in the form of anorthite leads to critical suppression of expansion of a ceramic body and to the increase of its stability to humid environment.

This work has been financially supported by a project of the Ministry of Culture Czech Republic DG18P02OVV028 “Technology of Treatment and Identification of Degradation Processes of Ceramic Finds from Hradčany Palaces – Methods of Restoration and Conservation of Porous and Dense Ceramics and Porcelain”.

S5-P101.858

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric analysis of the ornamentation of a Late Bronze Age vessel (13th/12th century BC)

Jankovits, K.[1], Kovács I.[2], Szőkefalvi-Nagy, Z.[2]

[1]Pázmány Péter Catholic University, Egyetem u. 1, 2087 Piliscsaba, Hungary
[2]Wigner Research Centre for Physics, Konkoly -Thege M. út 29-33, 1121 Budapest, Hungary

Keywords: Late Bronze Age, Urnfield culture; decorated mug; white bronze, PIXE

The finds from a settlement of the early Urnfield culture dating from the Ha A1 period (13th/12th century BC) investigated at Nagykanizsa–Bilkei-dűlő (County Zala) in southern Transdanubia included a small mug decorated with a longish knob and three rounds, regularly spaced stamped motifs retaining the residue of a whitish substance. A larger amount of this substance survived in one of the stamped circles, while the other two only retained a minimal amount. In the preliminary report of the excavation, L. Horváth suggested that this substance perhaps represented the remains of a glass inlay (Horváth, L., 1997).
The composition of the remains were analysed by PIXE (Particle Induced X-ray Emission**)** spectroscopy in the Wigner Research Centre for Physics, which revealed that the substance was not glass because it contained high amounts of copper (Cu) and tin (Sn). Alloys of this type are generally referred to as white bronze in archaeological studies.
Various articles made of white bronze, mostly small jewellery items and pendants, are principally attested in the early Urnfield period (Ha A1, 13th/12th century BC) in the Late Bronze Age of the Carpathian Basin. Funnel-shaped pendants are frequent in the southerly regions of Transdanubia, but they are also known from Croatia and Northern Italy. The archaeometric examination of the funnel-shaped pendants from northern Italy (e.g. from Bor and Lazise) indicated that their tin content was over 90%, resulting in a whitish sheen resembling silver.
M. Primas suggested that artefacts made of bronze with a high tin content, particularly jewellery, had a dual function: they served both as adornments and as ingots, since tin was indispensable for casting bronzes (Primas, M. 1984).

HORVÁTH, L., 1997. Késő bronzkori település feltárása Nagykanizsán. Excavation of a Late Bronze Age settlement at Nagykanizsa, *Archaeological Reports in Hungary MNM Budapest, 2001, 37-43*
PRIMAS, M., 1984. Bronzezeitlicher Schmuck aus Zinn, *Helvetia Archaeoligica* 15, 33-42

S5-P103.392

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Decorating techniques of pottery in the Villanovan Verucchio site (RN, Italy): evidence of metal-band use, or not?

Patrizi, G.[1], Masieri, M.[2], Rizzo, D.[1], Rodriguez, E.[3], De Benedetto, G.E.[1,2]

[1]Department of Cultural Heritage, University of Salento, campus Ecotekne, 73100 Lecce
[2]IBAM-CNR, campus Ecotekne, 73100 Lecce
[3]Director of the Archaeological Civic Museum of Verucchio

Keywords: Archaeometry; Raman; XRF; IR; Verucchio

The Villanovan site of Verucchio (RN, Italy), situated on the top of a hill that dominate the Marecchia Valley and about hundred kilometers of the Adriatic coast, is one of the most important centers of the Final Bronze-First Iron age. Thanks to its strategic geographical position, it was at the center of the crossroad of exchanging both with the Internal Etruria and the Baltic area.
Four nucleus of cremation burials, called Lavatoio, Moroni-Semprini, Le Pegge and Lippi, have been found over the hillslopes around the village and they have been investigated since the end of the nineteenth century until nowadays. About 600 entombments have been brought to light and they represent the main informative nucleus on the Villanovan Verucchio.
Analyzing the pottery found in the burials, a particular decoration has been noticed made by fixing metal strips after plastering the vase surface with an organic dark grey or dark brown substance.
In order to reveal the nature of the metals and the coated/adhesive material used to decorate the potteries, a series of non-destructive spectroscopical analysis have been made. XRF analysis revealed the presence of tin as a component in the strips, IR analysis shown that different coated/adhesive materials have been used to fix the strips, while Raman analysis helped in understanding the degradation processes of the metal strips and to identify and characterized a probably imitation of this technique in the same archaeological context. In the present communication will be reported the relevant results.

S5-P104.462

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric analysis of Late Medieval and Early Modern age glasses of a familial castle in Hungary

Sz. Osváth, Zs.[1], Sztankovánszki, T.[2], Bajnóczi, B.[3], Fórizs, I.[3]

[1]Department of Archeology, University of Szeged, Szeged, Hungary
[2]Móra Ferenc Museum, Szeged, Hungary
[3]Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Budapest, Hungary

Keywords: glass; late medieval; early modern age; plant ash glass; kutrolf

The excavations of a late medieval and early modern age familial castle located in Dombóvár (Hungary) during 2014–2019 resulted in the recovery of different kinds of finds including glass fragments. The main type of the glass assemblage investigated up to now was windowpane pieces, but fragments of prunted beakers, bottles, goblets, one piece of a neck of the so-called kutrolf (or Italian “angastare”), colourful pieces and some other unique and/or unidentified fragments also occurred. Due to the explosion of the castle in 1702, as well as other activities that caused the disturbance of the uppermost layers and the fact that glasses could be found in almost all trenches and any layer, the artefacts under discussion can be dated from the 14th century – fitting well the building date of the palace according to written sources – till the beginning of the 18th century. Compositional analysis of the glass artefacts was performed by using handheld X-ray fluorescence spectrometer and an electron microprobe (attached with EDS) in order to determine the base glass types and colourants, which can help chronological and provenance approaches.

Although many base glass types have been identified regardless of typology, majority of the glass finds were made of plant ash glass similarly to the “Venetian glasses”. However, it cannot be surely decided whether these plant ash glass pieces were imported or produced locally by Italian craftsmen. In addition, mixed alkali glasses, some unusual types and wood ash glass were identified as well. The specific composition of the kutrolf made of mixed alkali glass with high alumina content raises questions about its production technology and origin. Various base glass types indicate diverse relations including maybe local production – in certain periods – during centuries, and e.g. their Na_2O or CaO content allows us further groupings. Cobalt or copper, or both elements together colour the blue glasses, whereas manganese-bearing stripes cause brown shade in some of the windowpanes. Green glasses are coloured with copper probably simultaneously with iron.

S5-P105.324

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Applying pXRF Analysis on Unprovenanced Museum Glass and Metal Objects for Detecting Modern Reproductions: The Karam Collection of Lebanese Antiquities at the University of South Florida

Tanasi, D.[1], Tykot, R.H.[2], Hassam, S.[1], Kingsland, K.[1]

[1]Department of History, University of South Florida, Tampa, FL 33620 USA
[2]Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: pXRF; non-destructive analysis; museums; reproductions; archaeometry

Museums are often unable to vet every object that is donated to them, allowing for modern reproductions purchased by unsuspecting collectors to slip into museum collections. The ability to non-destructively verify an artifact's antiquity at low cost is beneficial for curatorial and collections management staff. The Karam Collection of Lebanese antiquities was donated to the University of South Florida Library in Tampa, FL in the 1990s to be held in perpetuity by the university. During a typological analysis of the 149 artifacts in the collection, several objects were flagged to be of uncertain antiquity. Five of the 61 glass artifacts in the collection were identified as having atypical patinas and did not conform to any Hellenistic or Roman glass types known in the literature. Two metal objects also had atypical patinas and did not conform to known iconographies of their subjects. Operating under the hypothesis that these objects may be modern reproductions produced for the collector's market, all of the 61 glass objects and 49 metal objects were analyzed using a non-destructive portable X-ray fluorescence spectrometer in order to quantify the elemental composition of the surface patinas. The five glass objects and both metal objects suspected of being reproductions were found to have surface compositions alien to those in the rest of the collection, supporting the hypothesis that the objects are not antiquities.

S5-P107.411

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chemical and geochemical composition of glass used in the renaissance stained glass windows of San Lanfranco (Pavia - Northern Italy)

Riccardi M.P.[1,2], Buganza S.[3], Morandotti L.[4], Visioli M.[5]

[1]DiSTA University of Pavia, via Ferrata 9, I27100 Pavia, Italy
[2]Arvedi Laboratory, University of Pavia, via Ferrata 9, I27100 Pavia, Italy
[3]Department of History, Archaeology and Art History, Università Cattolica Sacro Cuore, Campus Brescia, via Trieste 17, I25121 Brescia, Italy
[4]Studio Laura Morandotti, via della Commenda 33, I20122 Milano, Italy
[5]Department of Humanities, University of Pavia, Corso Strada Nuova 65, I27100 Pavia, Italy

Keywords: stained glass windows; renaissance; glass; chemical composition; geochemical composition; northern Italy

The three renaissance stained glass windows of San Lanfranco (Pavia) were created for the context that still hosts them: the presbytery area of the abbey church. This area was renovated at the beginning of the sixteenth century at the request of Pietro Pallavicini di Scipione, abbot of the important spiritual center of Vallombrosa.
The main window, located above the apse of the church, depicts the Madonna with Child, San Lanfranco and the customer Pallavicino kneeling. The minor rounds, inserted in the oculi of the apse, so as to highlight the important ark of San Lanfranco, depict two prominent personalities of the religious context of Vallombrosa and Pavia such as Cardinal San Bernardo degli Uberti and Cardinal San Tesauro Beccaria. Despite the abrasion of the grisaille, the elements of style allow the attribution of the three windows to the drawings by Bernardino Lanzani, an artist documented between Castel San Giovanni (PC), Pavia and Bobbio (PC) between 1490 and the early twenties of the sixteenth century, are evident.
The restoration, carried out in the years 2018 – 2019, has highlighted the integrity of the artifact, revealing that over time no glass substitutions had been made. Knowing the composition of the window glass made it possible to shed light on the use of flat glass in Pavia in the sixteenth century.
The archaeometric study was to determine the chemical and geochemical composition of the three Renaissance stained glass windows. The micro-fragments analyzed come from to the central drawings and from the outer frame (bigherini). The samples were studied by scanning electron microscopy (SEM) and analyzed by EDS microanalysis (major elements), and in LA ICP MS (minor and traces elements).
The analytical data were aimed to identifying the type of glass used, the raw materials and the coloring techniques.
Most of the glass belongs to the HLLA group; only a few samples show a Natron-glass composition. HLLA glass always contains P_2O_5 in values around 2 wt%, and MnO (values between 0.4 and 0.6 wt%). Natron-glass can be interpreted as a reuse of glass from previous artifacts, already during the first assembly of the glass. Colored glass shows a sequence of layers of colored glass (thicknesses of about 100 - 120 microns) enclosed within layers of colorless glass. The dyes used are Cu for green, Mn for purple and pink and Co for blue.

S5-P108.504

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

XVIII-XIX Century Glass bottles finds from Almada: typology, colour and composition for provenance and production techniques studies

Rocha, S.[1,2], Alves, L.C.[3], Coutinho, I.[1,2]

[1]Department of Conservation and Restoration, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[2]Research Unit VICARTE, Vidro e Cerâmica para as Artes, FCT NOVA, Campus da Caparica, 2829-516 Caparica, Portugal
[3]C2TN, Instituto Superior Técnico, Universidade de Lisboa, 2695-066 Bodadela LRS, Portugal

Keywords: glass bottles; raw materials; provenance; composition; color

During recent archaeological excavations at a Rua Latino Coelho building in Almada, several artefacts were recovered comprising glass, ceramics and metal objects. The aim of the present work was to study the dated 18th century cylindrical shaped wine glass bottles found following a typological, chemical composition and chromophores type identification (responsible for the brown, pale green and emerald green colours observed) approach, in order to be able or to try to establish their provenance and production methodologies.
The samples were initially grouped accordingly to their different typologies and 28 fragments were selected for elemental analysis using μPIXE ion beam technique. The samples were finally gathered in two groups through their similarities in chemical composition allowing comparison with glass bottles manufactured in several European production centres those including the Coina Royal Glass Factory in Portugal. Similarities with Bristol, England and with Coina, Portugal production centres, could be extracted from bottles belonging to the first group (totally composed of brown glasses), whereas the second group presented similarities with the bottles produced in Arles, France. It is interesting to notice that no correlation could be found with the bottles previously found in Lisbon whose results are reported elsewhere [1].
UV-Vis spectrometry was used for establishing the elemental species and/or oxidation states that could contribute to the different observed colours. The results obtained are inconclusive once both Fe^{2+} and Fe^{3+} were determined for all the samples with no apparent concentration differences.
Extended analytical glass bottles composition is needed both for glass found in Portugal and in European production centres (including Portuguese ones) as well as the origin of the observed colour responsible agents should be further investigated.

[1] - I. Coutinho, T. Medici, L.C. Alves, B. Gratuze, M. Vilarigues, Provenance studies of 18th century potassium-rich archaeological glass from Portugal, Journal of Archaeological Science: Reports 13 (2017) 185–198. http://dx.doi.org/10.1016/j.jasrep.2017.03.050

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

An interdisciplinary approach to the study of the Phoenician-Punic glassy beads of Southwestern Portugal – the case study of Vinha das Caliças 4

Mirão, J.[1,2], Barrulas, P.[1], Dias, L.[1], Arruda, A.M.[3], Costa, M.[1]

[1]HERCULES Laboratory, University of Évora, Portugal;
[2]Geosciences Department, School of Sciences and Technology, University of Évora, Portugal;
[3]UNIARQ – Archaeological Center of the University of Lisbon, Portugal

Keywords: Pre-Roman glass; Glass provenance; LA-ICP-MS

Vinha das Caliças 4 is an Iron Age necropolis located in the outskirts of the town of Trigaches (southwestern Portugal). This necropolis, dated to the 6th century B.C. and with over 45 inhumations, has an exquisite assortment of grave goods, including weapons, precious metal artefacts, fragments of furniture and glassy adornment objects.

Pre-Roman glassy artefacts have been found in significant amounts in southwestern Iberia, especially in burial contexts. However, typology has been the main focus of the research conducted on these artefacts, with little or no archaeometric analysis. In this study, a multi-analytical minimally invasive methodology was used to determine the chemical and mineralogical composition of the large bead assemblage found in Vinha das Caliças 4. Handheld X-ray fluorescence (hXRF) was used to establish the bulk chemical composition of these glassy artefacts, while a variable pressure scanning electron microscope coupled with energy dispersive X-ray spectrometry (VP-SEM-EDS) was used to determine microstructural aspects and to perform point analysis and elemental mappings. Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) was used to determine the trace element composition, which is essential for glass sourcing. Micro-X-ray diffraction and micro-Raman spectroscopy were used to identify possible micro-inclusions present in the glassy artefacts, including opacifying agents and remains of the raw materials used in the production of poor melting quality glass.

This study contributes to a deeper knowledge regarding pre-Roman glass production technology in the Iberian Peninsula. Moreover, it will provide insights into the trade routes present in the Mediterranean in the 6th century B.C. and contribute to the understanding of the influence of the Eastern and Central Mediterranean world in the Southwestern Iberia during the first millennium B.C..

Acknowledgements

This work has been financially supported by the PP-nGLASS – An interdisciplinary study of the impact of the Phoenician-Punic natron glass trade in Iberian communities – project (EXPL/HAR-ARQ/0381/2021) and by the UIDB/04449/2020 and UIDP/04449/2020 projects, which were funded by Fundação para a Ciência e Tecnologia (FCT) and by the European Regional Development Fund. M. Costa also acknowledges FCT for a Ph.D. Fellowship (SFRH/BD/128889/2017) co-funded by the European Social Fund and by Portuguese national funds. The authors would also like to thank ArqueoHoje for providing access to the archaeological artifacts.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Nondestructive analysis of Egyptian faience beads from Late Bronze Age and Early Iron Age necropolises in Alentejo (Southern Portugal)

Monge Soares, A.M., Gonçalves, A.P., Alves, L.C., Valério, P.

C2TN, Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa

Keywords: Orientalising trade; Ornaments; Scarabs; XRD, μ-PIXE

Faience beads from necklaces, associated with human inhumations in the Late Bronze Age (9-8th century BC) hypogea of Monte da Ramada 1 (Aljustrel) and also in the Early Iron Age (7-6th century BC) necropolises of Palhais (Beja), Montinhos 6 (Serpa) and Corte Margarida (Aljustrel), were studied using non-destructive techniques (XRD and μ-PIXE). Two scarabs, one also found in Palhais and another in Corte Margarida, were analysed as well. Results show that all these small items of high-valued ornaments have a ground-quartz body, while specific colorants of the glazing mixture were identified, namely metal ions like Cu for the green ones and Fe for the red/brown glazes.

It is the first time that this kind of glazed artefacts from proto-historic contexts in the Portuguese territory is identified and characterised. Moreover, its appearance in a Late Bronze Age necropolis located quite inland in Southern Portugal points to an Orientalising trade of luxurious products already in place before or just when the foundation of the first Phoenician settlements in Iberian seashores occurred.

Acknowledgements

The support of Fundação para a Ciência e a Tecnologia (FCT) to C2TN (UIDB/04349/2020) is gratefully acknowledged.

S5-P112.252

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Analytical methods for characterization of yellow glazes on the historical Renaissance ceramics

Šefců, R.[1], Kloužková, A.[2], Blažková, G.[3], Turková, I.[4], Bajeux Kmoníčková, M.[1], Dvořáková, P.[2], Kolářová M.[2]

[1]National Gallery Prague, Staroměstské nám. 12, 110 15 Prague, Czech Republic
[2] University of Chemistry and Technology, Prague, Technická 5, 166 28 Prague, Czech Republic
[3]Institute of Archaeology of the Czech Academy of Sciences, Prague, v.v.i., Letenská 4, 118 01, Prague, Czech Republic
[4] Institute of Criminalistics, Bartolomějská 12, 110 00 Prague, Czech Republic

Keywords: yellow glaze; ceramics; instrumental analysis; Raman spectroscopy; SEM-EDS

Presented results were obtained by an interdisciplinary survey of the yellow glazes on the historical renaissance ceramics of the 15th–16th century from the collection of the National Gallery Prague and Institute of Archaeology of the Czech Academy of Sciences in Prague. These lead glazes, which can be tin-opacified or transparent, present different yellow colors. The main objective was to identify yellow dyes in colored coatings using multiple instrumental techniques. The initial investigation of materials was realized in-situ by means of portable X-ray fluorescence analyzer and mobile Raman spectroscopy. The samples have been taken subsequently and analyzed using optical microscopy, scanning electron microscopy coupled with energy dispersive x-ray spectroscopy and micro-Raman and infrared spectroscopy. The use of the combination of the Raman spectroscopy and SEM-EDS were crucial for the analysis of some pigments in a non-destructive way. The measurements were performed on the individual grains or on the cross-sections using the mapping mode for better observation of individual components present in color glazed layers. The results showed that these are lead glazes with dyeing components consisted of ionic dyes Sb, Sn ions. EDS qualitative and quantitative mapping was performed on the yellow-glaze layers and showed the distribution of lead, silicon, tin and antimony in the individual layers. The yellow glazes contain the binary system Pb–Sb or system Pb–Sb–Sn composition. Using micro-Raman spectroscopy, the yellow pigments were identified in each glaze as Naples yellow, lead-tin yellow structure type II or Pb-Sb-Sn or their combinations.
The results obtained by the application of complementary analytical techniques allowed a qualitative evaluation of the ceramic and glaze materials used and significantly expanded the knowledge of yellow pigments used in each typology of different Renaissance ceramic works.
This contribution has been financially supported by the project of the Ministry of Culture of the Czech Republic: Technology of Treatment and Identification of Degradation Processes of Ceramic Finds from Hradčany Palaces - Methods of Restoration and Conservation of Porous and Dense Ceramics and Porcelain (DG18P02OVV028).

S5-P113.416

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric analysis of Hutterite-Haban blue-glazed stove tiles

Bajnóczi, B.[1], Ridovics, A.[2], May, Z.[3], Szabó, M.[1], Tóth, M.[1]

[1]Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Budapest, Hungary
[2]Hungarian National Museum, Budapest, Hungary
[3]Institute of Materials and Environmental Chemistry, Research Centre for Natural Sciences, Budapest, Hungary

Keywords: stove tile; blue glaze; tin glaze; Hutterite; Haban

Potters working in the Anabaptist-Hutterite (Haban) settlements in East-Central-Europe produced unglazed, unicoloured lead-glazed and varicoloured tin-glazed tiled stoves with rich and variegated motifs from the end of the 16th century. The double prismatic formed blue-glazed stoves, set with large rectangular tiles and crowned with battlements, form a distinct group. A representative stove from Liptónádasd (Trstené, today Slovakia) and another one from Besztercebánya (Banská Bystrica, today Slovakia) belong to this group. Main motifs of the tiles are floral trails; but the compositions are different on the two stoves. The decorations emerging from the blue-glazed background were emphasized by white tin glaze. Based on stylistic considerations the two stoves are closely related to each other, both can be dated to the second half of the 17th century.

The present archaeometric research aims at determining the composition of the ceramic body, the blue glaze and the white decorations. Tiles were analysed non-destructively by a handheld XRF, in addition, fragments detached from a Liptónádasd stove tile were examined by using XRD, ICP-AES and SEM-EDS.

The handheld XRF analysis performed on the two stoves succeeded similar results, namely blue glazes contain cobalt and a trace amount of tin, whereas white decorations contain much higher amount of tin. Circa 0.2 wt% uranium was detected in the blue glaze of the Liptónádasd stove, the blue glaze of the other stove also contains a trace amount of uranium. SEM analysis performed on the cross sections of a Liptónádasd tile confirmed the XRF results. Tiny amount of tin oxide and uranium-bearing particles are present in the alkali-lead (K-rich) type blue glaze coloured by cobalt. The white glaze is of lead-alkali type with high (circa 20 wt%) tin oxide content. The blue glaze covers an illitic ceramic body with low calcium and high iron content.

The tiles of two stoves show similarities in the material characteristics with the blue-glazed vessels produced by the so-called “mining town workshop”, which probably operated based on Hutterite-Haban tradition in the vicinity of Besztercebánya. The vessels of this workshop were also covered by alkali-lead (K-rich) type cobalt blue glaze with tiny amount of tin and detectable amount of uranium. It may be hypothesized that the “mining town workshop” and the workshop producing blue-glazed stoves operated in the same region, were related to each other or perhaps were the same.

S5-P114.622

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Middle Byzantine Ceramics from the Glavkis Shipwreck – Elemental and Microstructural Analysis

Klesner, C. E.[1], Hein, A.[2], Kilikoglou, V.[2], Tsanana, A.[3], Amprazogoula, K.[3]

[1]Department of Materials Science and Engineering, University of Arizona, Tucson, AZ, USA
[2]Institute of Nanoscience and Nanotechnology, NCSR "Demokritos", AghiaParaskevi, Attica, Greece
[3]Ephorate of Antiquities of Chalcidice and Mount Athos, Greece

Keywords:

Fifteen Byzantine ceramics were recovered from a shipwreck in the vicinity of the Glavki reef in the North Aegean Sea. The ceramics which consist of fine sgraffito glazed ware and a transport amphora date to the third quarter of the 12th c. CE and likely represent goods being transported for trade. The vessels were recovered southeast of Chalkidiki between Sporades and the island of Lemnos. To determine their production location, fourteen of the ceramics were subjected to compositional analysis by wavelength dispersive x-ray fluorescence spectroscopy (WD-XRF). This common compositional technique has been extensively used for Byzantine ceramics and enabled direct comparison to previous archaeometric studies, including of similar period ceramics from other shipwreck environments. Within the glazed ceramics from the Glavkis shipwreck, two compositional groups were identified. Although chemically altered by centuries of exposure to seawater, both groups bear strong chemical similarities to the Chalcis group identified by Waksman et al.[1]. The amphora sample was also identified as most likely belonging to the Chalcis reference group. Cross sections of all the ceramics were also analyzed by scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) to determine their production technology and to identify the mechanisms and extent of post-depositional chemical alterations in the ceramics. This case study adds to the evidence of trade of Middle Byzantine glazed ceramics in the Aegean, as well as revealing more information about the post-depositional alteration processes of low fired ceramics in marine environments.

[1] Waksman, S. Y., Koutsouflakis, G., Burlot, J., and Courbe, L., 2018. Archaeometric investigations of the tableware cargo of the Kavalliani shipwreck (Greece) and into the role of the harbor of Chalcis in the Byzantine and Frankish periods. Journal of Archaeological Science: Reports 21, 1122-1129.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The archaeological and scientific analysis of cobalt-blue-decorated ceramics from 7th to 13th century CE

Zhang, Y., Pollard, A. M.

School of Archaeology, University of Oxford, Oxford, UK

Keywords: blue-and-white porcelain; tang *sancai*; cobalt pigment; maritime silk road

This paper reviews studies of Tang (618-907 CE) and Song (960-1279 CE) blue-and-white porcelains, both archaeologically and scientifically, based on published data, and compares blue-and-white with sancai which represents the earliest use of cobalt pigment in Chinese ceramics. Thirty-nine Tang blue-and-white wares and seven Song wares have been excavated from city sites, kilns, a tomb, a shipwreck, and temples. These findings identify Tang blue-and-white as export ware related to the Maritime Silk Road or suggest it was produced for foreigners in China for daily use or personal collection. Also, the results reflected the connection with Buddhist culture. All Tang and Song blue-and-white are vessels and the motifs on them are plants, insects, child, geometric and combination patterns. Palmette and lozenge motifs show the influence of Middle Eastern taste. Additionally, we re-analyse and discuss the previous scientific evidence of Tang and Song blue-and-white, including body, glaze, and cobalt pigment to reveal the origin of materials and technology of Tang blue-and-white porcelain. The raw pigment of Tang blue-and-white was possibly inherited from sancai, probably from the Middle East, and the biscuit body and high-fired clear lime glaze were provided by white porcelain. And Song blue-and-white may utilise native cobalt ore, which possibly was from Zhejiang.

S5-P117.675

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Identification of glaze pigments by hyperspectral imaging: in situ characterization of 19th-century color palettes and museum pieces

Beauvoit, E., Javel, J.-B., Chapoulie, R., Ben Amara, A.

IRAMAT-CRP2A, UMR 5060 CNRS, Université Bordeaux Montaigne, Maison de l'Archéologie, Domaine Universitaire, Esplanade des Antilles, 33607 Pessac Cedex, France

Keywords: Vieillard manufactory; polychrome glaze; non-destructive analysis; hyperspectral imaging; 19th century

During the second half of the 19th century, fabrication of ceramic pieces decorated with polychrome relief glazes occupied a prominent place in French factories of white earthenware. It is through the example of the production of the Vieillard and Co. Manufactory (Bordeaux, France, 19th century) that we will approach the technical study of this decoration process. A preliminary step towards the non-invasive study of objects in the museum's collections was taken through the study of archaeological sherds of this manufactory. Multianalytical methods were carried out in order to characterize both the clay support and the colored glazes. One of the aim of this paper is to implement a non-invasive approach of colored glazes study based on the development of optical techniques (reflectance spectroscopy and hyperspectral imaging). This methodology is intended to characterize colored glaze and more specifically to identify the types of pigments used not known in the text.

A corpus of some pieces of polychrome glazed earthenware and decorated porcelain from Musée d'Aquitaine and Musée des Arts Décoratifs et du Design (Bordeaux, France) has been considered here. The selected pieces, representative of Vieillard and Co. decorative production, have a wide variety of colors. Twenty pieces, among them four color charts, were non-destructively analyzed *in situ* using a non-destructive hyperspectral imagery system. The data obtained made it possible to better understand the nature of the decoration chromogens and thus, to establish the color palette of the artists who produced this type of decorative objects. The goal is therefore to improve the database already established on hyperspectral imaging based on studies carried out on archaeological sherds and enriched by data acquired on color palettes.

[1] Beauvoit E., Cantin N., Sireix C., Marache V., Chapoulie R., Ben Amara A., 2019. *A multianalytical approach for the study of colored glaze decoration: the case study of Vieillard and Co. Manufactory (France, 19th century)*. *TechnArt*, Bruges, Belgium, 7-10 May 2019.

S5-P120.713

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Assessing provenance of Spanish 16th-17th centuries tin-lead glazed ceramics by Non-destructive Portable X-Ray Fluorescence

Iñañez, J.G.

GPAC, Built Heritage Research Group, Faculty of Arts, University of the Basque Country UPV/EHU, Vitoria-Gasteiz, Spain

Keywords: pXRF, tin-lead glazed ceramics, Spain, America, provenance

Non-destructive analytical techniques have emerged during the past years as an invaluable tool for archaeologists and museum professionals. Nowadays, these non-destructive and usually portable techniques are widely applied to numerous and heterogeneous kind of artifacts in museum collections, from natural (e.g. stony materials) to synthetic materials (e.g. ceramics or metals). In this regard, portable x-ray fluorescence (pXRF) instruments have emerged as a virtuous tool for non-destructive elemental analyses. Although the socialization and spread of such technologies may be considered as an important step forward for the Archaeological Community, several problems may be pointed out. Thus, and from the experienced gained in the past years, it is important to clearly highlight that such techniques should not be considered by no means as some kind of a “black box”. On the contrary, the limitations and strengths of any instrument and technique depending on the nature of the material to be study should be known in advance in order to generate reliable data. Experiences from the field of pottery analyses will be discussed, including provenance and technological studies. Thus, 16th and 17th centuries ceramics among tin-lead glazed well-established chemical reference groups of Spanish and Spanish American Colonial contexts, which were previously characterized by bulk analyses techniques (e.g. NAA and ICP-MS), will be assessed by pXRF. The strengths and limitations of such pXRF instruments for provenance studies on tin-lead glazed pottery, the occurrence of complex clay matrix arrays, as well as the presence of heavy metal coatings (e.g. lead glazes), are assessed. In light of the results, it can be concluded that nowadays pXRF instruments can become invaluable tools for screening among large set of ceramics for provenance studies, saving important efforts and resources when designing a project that includes destructive analyses and/or the use of complex facilities.

S5-P121.721

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Identification of glaze defects sources of archaeological ceramics from Renaissance Prague

Kloužková, A.[1], Kolářová M.[1], Šefců, R.[2], Kohoutková M.[1], Dvořáková, P.[1], Blažková, G.[3]

[1]University of Chemistry and Technology, Prague, Technická 5, 166 28 Prague, Czech Republic
[2]National Gallery Prague, Staroměstské nám. 12, 110 15 Prague, Czech Republic
[3]Institute of Archaeology of the Czech Academy of Sciences, Prague, v. v. i., Letenská 4, 118 01, Prague, Czech Republic

Keywords: glaze; ceramics, instrumental analysis, thermal analyses; renaissance

Ceramic findings show various types of damage such as crazing, peeling, pinholing, specking, efflorescence and biodegradation. Some types of glaze defects occur immediately after the ceramic product is fired, while other types appear during utilization of the product. Moreover, in the case of archaeological finds, defects may occur due to unsuitable environmental conditions (especially relative humidity and temperature).
Various types of tiles, stove tiles and unique large-format tiles with relief colourful decors are represented among the archaeological finds from Renaissance Prague. Glazes of these ceramic products is degraded by different types of defects. The basic characterization of ceramic bodies and glazes of selected fragments was done in combination of microscopic (OM, SEM) and X-ray (XRF, XRD) analyses. Thermal analyses (especially DIL a TMA) were used to identify possible sources of defects of degraded surfaces. The relative expansion data obtained from dilatometric measurements were used to study stress relations in two-component systems. The stress relations between the glazes and their ceramic substrates may have been influenced by higher amounts of unstable metaclays in the ceramic bodies.
Severe crazing and cracking was found on lead glazes of some brown stove tiles. It was proved that the main source of crazing was tensile stress within glazes developed already during production. The green glazes in both reliefs are crazed, but the research has shown that crazing was caused by irreversible moisture expansion in ceramic bodies. This type of glaze defects is referred to as delayed crazing. The stress in the system changed as the ceramic body expanded due to moisture in the waste pit environment, reversing the compressive stress in the green glaze to tensile stress. However, the second measurement, which indicated the state of the system shortly after it had been fired, confirmed that there was in fact a compressive stress in the surface layer. The corrosion of yellow glaze was caused by selective leaching and this caused the formation of defects called pits or pinholes.
By finding out what caused the defects on the surface glaze layer, we gain invaluable knowledge about how archaeological glazed ceramic products were made and used.
This research has been supported by the grant NAKI II DG18P02OVV028.

S5-P122.724

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Lead Isotope Analysis of Medieval Glazed Ceramics from Southern Kazakhstan

Klesner, C. E.[1], Renson, V.[2], Vandiver, P.B.[1]

[1]Department of Materials Science and Engineering, University of Arizona, Tucson, AZ, USA
[2] Archaeometry Laboratory, Research Reactor Center, University of Missouri, 1513 Research Park Drive, Columbia, MO 65211, USA

Keywords: Lead-glazed Ceramics, Lead Isotope Analysis, Silk Road

This research aims to characterize the fine decorated pottery from Central Asia to determine the scale of local production and long-distance trade of lead glazed ceramics from the 9-15th c. CE, and to determine the technological variation and similarities within and between the local productions in Central Asian and imported Islamic lead-glazed wares. To this end, compositional analysis is used in combination with lead isotope analyses. The compositional analysis of ceramics from seven Medieval Period cities in Southern Kazakhstan identifies three different paste compositional groups among lead-glazed ceramics. One group represents a local production and the two others corresponds to non-local material. However, while the chemical compositions of the ceramic pastes in the three groups are distinct, the elemental analysis in the glazes themselves reveals no difference in the major, minor, or trace element concentrations. The similarity of the glaze composition applied to ceramics with different pastes and origins could indicate that the lead-glaze technology that was introduced in Central Asia shared common glaze production methods and materials with those that were used over the wider Islamic world. Previous lead isotope research on Islamic lead-glazed ceramics also demonstrated the potential of long-distance trade in lead-ore for glaze production throughout the Medieval Islamic world.

To further understand the ceramic production and exchange in Central Asia, and more specifically to identify if potters from Kazakhstan and Southwest Asia were using the same raw material for the lead-glaze production, the lead isotopic composition of glaze samples was determined from the local and non-local ceramic groups. A representative selection of thirty-five lead-glazed ceramics were sampled for lead-isotope analysis including thirty-one glazed ceramics from the local and non-local paste groups and four glazed-ceramics not clearly assigned to a paste group by elemental chemistry. This research helps to define the spread of goods, raw materials, and technology along the Silk Road in the Medieval Period.

S5-P123.758

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Pigment identification using non-destructive techniques on glass artifacts dated in the Archaic and Classical periods from the ancient Cemetery of Sindos in Northern Greece

Gara, E.M.[1], Nazlis, I.[2], Kampasakali, E.[3], Christofilos, D.[3], Litsardakis, G.[1, 4]

[1]Postgraduate Course in Protection, Conservation and Restoration of Cultural Monuments, Faculty of Engineering, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece
[2]Department of Conservation, Chemical - Physical analysis and Archaeometry, Archaeological Museum of Thessaloniki (AMTh), 54621 Thessaloniki, Greece
[3]School of Chemical Engineering and Laboratory of Physics, Faculty of Engineering, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece
[4]School of Electrical & Computer Engineering, Laboratory of Materials, Faculty of Engineering, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece

The present study focuses on the analysis of ancient pigments found in 21 glass artifacts from the ancient cemetery of Sindos in Thessaloniki, Greece. The cemetery was accidentally discovered in the early 1980s and the systematic excavation of the site revealed a significant number of ornate tombs, dug into the clayish earth of a low hill. Based on the artifacts found, the cemetery was chronologically placed in the Archaic and Classical periods spanning from the second half of the 6th century B.C. until the end of the 5th century B.C. The graves of Sindos indicate a variety of materials and artifacts, testifying to the existence of a fairly active and economically wealthy settlement. The artifacts under consideration are glass vases and beads dating back to the aforementioned Archaic and Classical periods. The artefacts are kept and exhibited in the Archaeological Museum of Thessaloniki.

The aim of the study was the identification and characterization of the pigments detected on the surface of the artifacts, as well as the correlation of laboratory analysis with archaeological data, for more accurate documentation. At the same time, the ancient technology of the different materials was investigated with particular emphasis on the colour rendering and the pigments used to decorate the objects under consideration. The analysis of the coloured decoration was carried out using non-destructive analysis techniques. Initial testing involved the analysis by X-ray fluorescence spectroscopy, in order to collect information regarding the elemental composition of the colouring matter.

Furthermore, selected artifacts were examined using Raman spectroscopy for the characterization of pigments and confirmation of elemental analysis. The outcome of the analysis of the glass artifacts was largely identified with the bibliographic and archaeological data of the period under review. Quantitative analysis has shown that these are high- sodium silicon glasses, which are easier to treat at lower temperatures. Concurrently, calcium levels are essential in glass as a network stabilizer.

The opaque colour is due to high antimony rates, while cobalt, iron, lead and manganese oxides give colour to the glass. Thus, the Archaeological Museum of Thessaloniki's analytical data was enriched concerning the Archaic and Classical period in the Macedonia territory.

S5-P127.3006

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Micromineralogical collections in the service of ceramic provenance studies – the collection of supervisory authority of regulatory affairs (formerly mining and geological survey of Hungary)

Péterdi, B.[1], Szakmány, Gy.[2], Józsa, S.[2], Miklós, D.G.[2], Szilágyi, V.[3], Gyuricza, Gy.[1]

[1]Supervisory Authority of Regulatory Affairs
[2]*Department of Petrology and Geochemistry, Eötvös Loránd University*
[3]*Centre for Energy Research*

Keywords: micromineralogy; heavy minerals; ceramic provenance; Hungary

Determination of exact provenance of the non-plastic components is an important question in ceramic archaeometric studies. Heavy mineral content of ceramics can reflect on the clastic raw materials applied for the manufacturing even in the case of tempering or mixing of different clay types. Therefore the knowledge on the heavy mineral assemblages occurring in the potential raw material territories is very important for the successful provenance determination.

The micromineralogical collection of the Supervisory Authority of Regulatory Affairs (formerly Mining and Geological Survey of Hungary) covers the surficial/near surface alluvial clastic sediments of the whole territory of the country with 863 sampling points. The samples originate from 754 sites: 510 mines (sand- or gravel-mines, Miocene to Holocene age sediments), 145 recent river-bars, 99 samples from shallow drillings on older (Pleistocene) alluvial cones. From each sampling point 10-100 kg of sediment were collected (according to the average grain size of the sediment), except for the drillings. Due to a sieving – heavy-media separation – magnetic separation method, each sample was separated into 5 or 6 fractions: ferromagnetic fraction (fr. A: practically magnetite); paramagnetic sequences (fr. B: ilmenite, hypersthene etc.; fr. C: garnets, pyroxenes, magmatic amphiboles etc.; fr. D: metamorfic amphiboles, epidote-group, staurolite, etc.); diamagnetic fractions (fr. E: zircon, kyanite, gold etc. and residual light minerals; fr. F (in case of a part of the samples): fr. E without residual light minerals). The whole collection contains 4326 individually inventoried samples with data about the sampling site (and sampled layer or depth etc.), grain category of the original sample and the original mass of the fractions mentioned above. Though the collection originally was set for sedimentary researches (e.g. sedimentation history of the filling up of the Pannonian Basin), it is well applicable for archaeometric studies (e.g. ceramic provenance investigations) too. Due to the continuous evaluation, the partly accumulated qualitative and quantitative information on the overall mineralogy provide the possibility of direct comparison to mineral species detected in archaeological pottery by conventional petrography or SEM-EDS. This important knowledge can promote the success of ceramic provenance studies.

S5-P130.175

Session 6 Technology/provenance – metals

ORAL

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Analyses of prehistoric gold artefacts from the Carpathian Basin

Dani, J.1, Kertész, Zs.2, Tarbay, J.G.3, Angyal, A.2, Szikszai, Z.2, Kiss, V.4

[1]*Déri Museum, Debrecen, Hungary*
[2]*Institute for Nuclear Research (Atomki), Laboratory for Heritage Science, Debrecen, Hungary*
[3]*Hungarian National Museum, Budapest, Hungary*
[4]*Institute for Archeology, Research Centre for Humanities, Budapest, Hungary*

Keywords: prehistoric gold finds; PIXE; metallurgical traditions

This paper discusses the study of prehistoric gold finds that were used during three thousand years in the Carpathian Basin from the Early Copper Age (4500 BC) until the beginning of the Late Bronze Age (1500 BC). The composition of 150 artefacts from Eastern and Western Hungary, as well as from the Transylvanian region were determined by non-destructive particle induced X-ray emission (PIXE) spectrometry. Gold hair rings, bracelets and other ornaments from the collection of the Hungarian National Museum, the Déri Museum (Debrecen), Laczkó Dezső Museum (Veszprém), the Ferenczi Museum Centre (Szentendre), and the Katona József Museum (Kecskemét) were analysed in cooperation with the Momentum Mobility Research Group of the Research Centre for the Humanities (Budapest) and Laboratory of Heritage Science at Atomki (Debrecen). Our primary aim was to define raw material compositions (major, minor and trace element concentrations) drawing their possible correlation with time periods and cultural and/or regional groups, metallurgical traditions. Based on the results three main metallurgical traditions can be observed: a Copper Age horizon with silver content lower than 10w%, Bell Beaker tradition with very high (up to 50w%) silver concentration, and Bronze Age artefacts dating from 2000 BC onwards with silver content usually between 12-30w%. At the same time, the Eastern European origin Yamnaya jewellery around 3000 BC reflects a special tradition, with special raw materials. Analyses of gold jewellery from burials and hoards prove potential cross-cultural connections between Middle Bronze Age populations of Western, Central and Eastern Hungary, but also the existence of different workshops. We also collect data of production techniques and traces of use wear applying 3D microscopy. By these non-destructive methods a great amount of information can be gained for the better understanding of production and use of prehistoric gold jewellery in Central Europe.

.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Lead isotope analyses of Iron Age Luristan bronzes and their comparison with Iranian ore resources

Oudbashi, O.[1], Renson, V.[2], Hasanpour, A.[3]

[1]Department of Conservation of Cultural and Historical Properties, Faculty of Conservation, Art University of Isfahan, Isfahan, Iran.
[2]Archaeometry Laboratory, Research Reactor Center, University of Missouri, Columbia, MO 65211, USA.
[3]Department of Archaeology, Cultural Heritage, Handcrafts and Tourism Organization of Lorestan Province, Khorramabad, Iran

Keywords: tin bronze; lead isotopes; Luristan; Iron Age

Despite its key role in the development of early metallurgy and in the production of tin bronzes, the Iranian Plateau remains largely under studied when it comes to the identification of the ore sources used for metal production in the area. Pioneer work to investigate Chalcolithic sites and possible ore sources used for metallic production shown that some objects presented compatibilities with ores in Central Iran, the Urumieh-Dokhtar and the Sanandaj-Sirjan (e.g., Pernicka et al. 2011, Nezafati et al. 2008). Here, we have analyzed the lead isotopic composition of 15 tin bronze objects recovered at Baba Jilan, an Iron Age cemetery in Luristan, Western Iran. The results are compared with a compilation of over 300 lead isotopic data available in the literature for ore sources across the different geological zones in Iran, and objects and slags recovered at different sites dated from the Chalcolithic to the Bronze Age. The samples from Baba Jilan have a signature that does not match that of the data for the ores from the Zagros Fold Belt, the Tabas Block, the Lut Block and Yazd Block. Most of the objects analysed here (12/15) have a lead isotopic composition that is compatible with multiple ore sources located in the Urumieh-Dokhtar Zone, the Sanandaj –Sirjan and the Alborz Zones. Two other objects have a signature compatible with that of ore sources located in Central Iran, and the Alborz Zones. One sample does not show good compatibility with any of these sources.

Nezafati, N., Pernicka, E., and Malek Shahmirzadi, S., 2008. Evidence on the Ancient Mining and Metallurgy at Tappeh Sialk (Central Iran). In: Ü. Yalcin, H. Özbal, A. G. Paşamehmetoğlu (Eds.). Ancient Mining in Turkey and the Eastern Mediterranean, pp. 329-350, Atilim University, Ankara, Turkey.

Pernicka, E., Adam, K., Böhme, M., Hezarkhani, Z., Nezafati, N., Schreiner, M., Winterholler, B., Momenzadeh, M., and Vatandoust, R., 2011. Archaeometallurgical research on the Western Iranian Plateau. In: A. Vatandoust, H. Parzinger, B. Helwing (Eds). Early mining and metallurgy on the Western Central Iranian Plateau, pp. 631-688, Archäeologie in Iran und Turan 9.

S6-O02.500

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

On Stranger Tides: the ebb flow of copper in the Eastern Mediterranean

Howarth, P.J., Sainsbury, V.A

School of Archaeology, University of Oxford, UK

Keywords: Bronze Age (Middle and Late); Archaeometallurgy; Eastern Mediterranean; Maritime trade

Seafaring trade across the Mediterranean has been a focus of antiquarian and archaeological research for centuries, as has the trade of copper throughout the Bronze Age. This paper uses the novel approach of the FLAME Project to revisit peoples' relationship with copper, whether as transporters, traders, or consumers. By looking at the variation of copper chemistry in the corpus of copper in use at coastal sites, island sites and as cargo we hope to re-evaluate the use of copper by communities along the pathways via which it was moved in the 2nd millennium BCE, rather than focussing on the origins of the material itself.

FLAME, the FLow of Ancient Metals across Eurasia, is an ERC project aimed at investigating the movement, exchange, and transformation of metal in Eurasian societies during the Bronze and Early Iron Age through a synthesis of legacy data. During the course of this project a subset of more than 500 analyses of copper-base objects from the Mediterranean Coast has been collated. The typologies included range from rivets to diadems, allowing us to focus on more than elite or prestige items, which was historically a pitfall in the study of Late Bronze Age metallurgical assemblages.

Chronologically, we have also attempted to push such discussions both temporally earlier and metaphorically 'beyond' the Late Bronze Age, with the aim of better reconciliation of datasets collected and produced across Eurasia. In so doing, we necessarily touch on how named chronologies - whether semi-universal, such as 'Bronze Age', or highly localised, such as 'Late Cycladic I' - impede pan-regional syntheses. With the increasing exploitation of absolute dating, and the development of more holistic approaches to archaeology, a move away from regionally bound narratives, the 'fuzziness' and pitfalls of such terms must be addressed. Spatially, we use ArcGIS to create geographical polygons which combine many datapoints within a standardised lattice. This allows us to include single find material in context with larger scale analyses of contemporaneous finds from other closely localised sites. The polygons chosen for this research question are 20 km square, based on an estimated daily distance covered by a human on foot, as used by the ORBIS Project. By focussing on distance in human terms, we aim to address the behaviour of the people behind the copper. First and foremost, this paper aims to look at chemical changes in copper composition as markers of human intervention.

S6-O03.198

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Preliminary study of two antique Illyrian helmets (V-IV B.C.) excavated in northern and central Albania

Çakaj, O.[1], Duka, E.[2], Schmidt, G.[3], Stamati, F.[4]

[1]Department of Physics, Faculty of Natural Science, University of Tirana, Albania
[2]Department of Engineering Physics, Faculty of Engineering Mathematics and Engineering Physics, Polytechnic University of Tirana, Albania
[3]Institut für Keramik, Glas - und Baustofftechnik, TU Bergakademie Freiberg, Germany
[4]Centre of Albanian Studies, Tirana, Albania

Keywords: Illyrian helmet; SEM-EDS; OM; Vickers microhardness; copper-tin alloy

This study combines several analytical techniques in order to preliminary investigate two Illyrian helmets dating the V-IV century B.C. They have been exposed in the Center of Albanian Studies in Tirana, after being excavated in 1963 but have never been studied with physical methods until now. The first Illyrian helmet was excavated in Nënshat (near Shkodra, north Albania) while the second in Zgërdhesh (near Kruja, central Albania). They both have the same characteristics such as: half spherical shape, ridge along its length, quadrilateral or trapezoidal face space, triangular blinkers near the ears to protect the warrior cheeks. Although there is a similarity with the helmets excavated in North Macedonia, their distinct features define them as Illyrian helmets. The aims of this paper are the investigation of the helmets' alloy and the production technique used. In order to archive these goals SEM-EDS, OM and Vickers microhardness tester were used to study the alloy, microstructure inclusions and its characteristics due to the production process. The Illyrian helmets alloy resulted copper-tin with less than 1% of iron content. The microstructure was composed by bended twins, with mean Vickers microhardness values from 160.5 to 169.6 HV, which suggests that these objects' alloys were casted, annealed and then cold worked in order to obtain the final distinct shape. No relevant inclusions regarding the copper minerals were observed in the Illyrian helmets' samples hence no conclusions can be archived with the analytical methods used so far..

S6-O04.512

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Copper at ancient kerma: a diachronic investigation of alloys and raw materials

Rademakers, F.W.[1,2], Verly, G.[3], Degryse, P.[2,4], Vanhaecke, F.[5], Marchi, S.[6,7] and Bonnet, C.[6,8]

[1]*British Museum, Department of Scientific Research, London, United Kingdom*
[2]*KU Leuven, Department of Earth and Environmental Sciences, Leuven, Belgium*
[3]*Royal Museums of Art and History, Brussels, Belgium*
[4]*Leiden University, Faculty of Archaeology, Leiden, the Netherlands*
[5]*Ghent University, Department of Analytical Chemistry, Gent, Belgium*
[6]*Mission suisse-franco-soudanaise de Kerma-Doukki Gel*
[7]*CNRS, UMR 8167 (Orient & Méditerranée), équipe Mondes Pharaoniques, Paris, France*
[8]*Académie des Inscriptions et Belles-Lettres, Paris, France*

Keywords: copper alloys; metallurgy; provenance analysis; Sudan; Egypt

This paper presents the first comprehensive study of metal artefacts found at ancient Kerma, Sudan. Covering a period of several millennia, it investigates the development of copper alloy recipes as well as metal provenance through the trace element and lead isotope ratio analysis of forty-eight sampled objects. These include grave goods as well as production waste related to large-scale bronze casting performed at Kerma.

This study is part of a wider evaluation of copper alloy production at Kerma through targeted workshop excavation, materials analysis and experimental archaeology. The analytical results illustrate the gradual and flexible transition from arsenical copper to tin bronze alloys over time, in a pattern similarly observed in ancient Egypt. Trace element distributions and lead isotope ratios for copper used at Kerma are comparable to those of contemporary Egyptian artefacts too.

These findings indicate the exploitation of ores similar to those mined at the Sinai Peninsula, although copper ore deposits in Nubia remain poorly characterized and thus difficult to identify as source candidates. Nonetheless, it can be suggested that metal provisioning networks along the Nile Valley were likely interrelated to varying degrees over time. These results provide an important contribution towards mapping technological exchanges that took place between ancient Egypt and Nubia.

.

S6-O05.677

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Leaded bronze palstaves from NW Iberia in the Late Bronze Age – Iron Age transition: technology and metal provenance

Armada, X.-L.[1], Montero-Ruiz, I.[2], Arcidiacono, L. [3], García-Vuelta, O.[2], Hook, D. [4], Martinón-Torres, M. [5], Rovira, S. [6], Sureda, P. [1], Wilkin, N. [4]

[1]Institute of Heritage Sciences, Spanish National Research Council (Incipit - CSIC)
[2]Institute of History, Spanish National Research Council (Incipit - CSIC)
[3]UCL Institute of Archaeology
[4]The British Museum
[5]Department of Archaeology, University of Cambridge
[6]Museo Arqueológico Nacional, Madrid (retired)

Keywords: Atlantic Europe; Bronze Technology; Elemental composition; Hoards; Lead Isotope Analysis

Palstaves, usually occurring in isolated hoards, are the most abundant metal object in the northwestern Iberian Peninsula during the Late Bronze Age and the transition to the Earliest Iron Age. Many of these palstaves contain high quantities of lead and have morphological features (such as the presence of the casting jet) that are incompatible with their use as 'functional' tools or weapons. At the same time, the phenomenon of large hoards composed of high-leaded and unused (*as-cast*) axes has parallels in other areas of western Europe. Despite the long research tradition, many questions persist around the functionality, production technology and metal provenance of these artefacts.

These issues are currently being addressed by the ATLANTAXES project (*Mass production and deposition of leaded bronzes in Atlantic Europe during the Late Bronze Age - Iron Age transition*. Funding body: Spanish Ministry of Science, Innovation and Universities, HAR2017-84142-R, 2018-2020) through a number of analytical techniques (XRF, lead isotope analysis, metallography, neutron tomography, neutron diffraction…) and experimental archaeology.

The aim of this contribution is to present and discuss new data on the elemental and isotopic composition of c. 30 leaded palstaves preserved in museums of the Iberian northwest and in the British Museum. Lead isotope analyses suggest that many of these metal resources come from the mining areas of the south of Iberia (Linares, Gádor and Cartagena) and could have reached the northwest of Iberia within the framework of the exchange networks promoted by Phoenician communities. The production technology still poses several issues and, although the use of metallic lead is documented, it is also possible that some palstaves were produced from copper-lead ingots that were converted into bronze by cementation using tin ores (cassiterite). Furthermore, some palstaves have a thick lead core as well as a large lead ball inside the casting sprue, raising complex issues about their manufacturing technique that are also discussed in this contribution.
.

.

S6-O06.251

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Quantifying uncertainty and deriving structure from an archaeometallurgical legacy dataset: the case of pre-Hispanic goldwork in Colombia

Vieri, J.[1], Crema, E.[1], Uribe Villegas, M.A.[2], Sáenz Samper, J.[2], Martinón-Torres, M.[1]

[1]Department of Archaeology, University of Cambridge, Cambridge, United Kingdom
[2]Museo del Oro, Banco de la República, Bogotá, Colombia

Keywords: legacy data; multi-level modelling; gold metallurgy; pre-Columbian

With the increasing availability of large legacy datasets, archaeometallurgy is facing new opportunities and challenges in the building of large-scale narratives for technological innovation, transmission and adaptation in the past. Systematic, cross-comparative studies of archaeometric data (e.g. compositional, mineralogical, isotopic etc.) can offer unprecedented insight into movement of materials and technologies through time and space. Nevertheless, insufficient attention has been paid to the development of methodological approaches that account for the complexity of these legacy datasets, which are influenced not only by sampling biases, but also exhibit varying degrees of variable/sample interdependence. This paper presents a new approach that explicitly incorporates such sources of uncertainty into modelling spatio-temporal variation in a compositional legacy dataset, using a case study on the pre-Hispanic gold metallurgy of Colombia.

The research is presented as part of a larger project focused on collating and analysing the known legacy data on pre-Columbian gold and copper alloys, with the aim of building a continental model for variation in alloying practices and their relationship with environmental and cultural factors. Exploratory data analysis from Colombia (n=2,130) has allowed us to test previous hypotheses about alloying practices in the region and their spatio-temporal variability. In addition, the results highlight the importance of copper – likely traded further distances than gold – and hint to a 'duality' of metallurgical practice, whereby the use of gold and copper was intertwined. Multilevel regression modelling, in combination with GIS based approaches, is used to gain insight into factors influencing such alloying decisions, as additional object attributes, geospatial data and/or archaeological data are considered as independent variables influencing composition. Importantly, multilevel regression modelling enables pooling information across different clusters in the data, e.g. different analytical techniques, recovery locations, laboratories etc., while variability within each is explicitly modelled, highlighting major sources of uncertainty in the dataset. We argue that similar methodologies can have far-reaching implications for the ways in which we can incorporate data produced by seemingly disparate research projects, ultimately promoting sustainability within archaeology.

.

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Evidences for polymetallic copper metallurgy in the Chalcolithic site of Fazael (middle Jordan valley)

Rose, T.[1], Bar, S.[2], Rosenberg, D.[2], Buchman, E.[2], Shalev, S.[2], Goren, Y.[1]

[1] *Department of Bible, Archaeology and Ancient Near East, Ben-Gurion University of the Negev, Be'er Sheva, Israel*
[2]*The Zinman Institute of Archaeology, University of Haifa, 199 Aba-Khushi Avenue, Haifa 3498838, Israel*

Keywords: Chalcolithic; southern Levant; melting, lost wax casting; polymetallic copper alloys; metallurgy; Fazael

Recent finds from the Chalcolithic site of Fazael (middle Jordan valley, 4,000 to 3,900 BCE) revealed a rich assemblage of copper objects and slagged pottery fragments. Beside a macehead, a standard and a few chisels, the majority of the metal objects was found in a courtyard building and consists of fragments from different objects. Many of the fragments were made of high-As and high-Sb polymetallic copper alloys and were part of so-called crowns, mace heads and standards. The casting quality of these objects is very divers. The slagged ceramic fragments were found within the same building complex, and they show a different fabric than the other pottery fragments found. The combination of the slagged ceramic fragments and the numerous fragments of polymetallic copper alloys hints to the location of a workshop for re-melting metal objects, most of them produced with the lost wax casting technique. At the present state of research, their production on the site cannot be excluded, although there are also no arguments in favour, yet. Lost wax casted objects are well known from the Chalcolithic southern Levant, *e.g.* from the Nahal Mishmar Hoard, but no production site was identified yet and the metallurgical process is still widely unknown. The site of Fazael might bear the potential of a major breakthrough concerning this problem. A multi-method approach is applied to reconstruct the processes the objects were involved in and the origin of the raw material. Which were the processes the slagged ceramic fragments were used in? Can we identify failed castings, probably directly assigned for re-melting? Were the casted objects reworked and/or used before? Where did the ore of the alloys came from? Can archaeological experiments help to identify the appearance of lost wax casting moulds and their identification in the archaeological record? Comparison with the local tradition of pure copper metallurgy and evidences from archaeological experiments will help to put the results into a broader cultural perspective and to reconstruct the technological choices.

.

S6-O08.297

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A technological tradition: double-layered crucibles in the Roman period

Gardner, C.[1], Freestone, I.C.[2], Rehren, Th.[2,3]

[1]Fitch Laboratory, British School at Athens
[2]Institute of Archaeology, University College London
[3]STARC, The Cyprus Institute

Keywords: Roman crucibles; technological tradition; multi-layered ceramics

It is widely accepted that the Roman influence on economic, social, and cultural factors was significant across the Empire. Roman social systems and cultural influences impacted on many aspects of daily life, from the buildings people experienced, the clothes people wore, through to people's religious beliefs and practices. With this came an apparent increase in demand for Roman material culture, which saw many industries flourish, e.g. the introduction of mass-produced pottery and sophisticated metalworking practices. The expansion of industry saw Roman technologies spread across the Empire rapidly, and even into areas formally outside the Empire, too.

Through a systematic and widescale study and analysis of Roman period crucibles from Britain and Germany, the re-analysis of data within the literature of crucibles from France and Switzerland, and an extensive literature review, it has been possible to trace a Roman technology across the Empire and in some cases, beyond its borders. This paper will demonstrate that the use of wheel-thrown, refractory white-ware crucibles with applied extra outer layers of less refractory, red clay, potentially intentionally tempered with wood-ash, was a technological tradition which spread across the Roman Empire and which continued into the medieval period. Reasons for this spread and the possible mechanisms in which it was transferred will be explored in this paper.

.

.

S6-O09.636

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

When alloy meets chronology
Bronze Age gold alloys from Northern Portugal

Tissot, I.[1], Guerra, M.F.[1,2]

[1]LIBPhys-UNL, Costa da Caparica, Portugal
[2] MONARIS - UMR 8233 CNRS & Sorbonne Université, Paris, France

Keywords: Gold; Bronze Age; Northern Portugal; XRF; alloys

First analysis of a selected group of Portuguese Bronze Age (BA) gold objects has shown compositional variations that could be associated with chronological and regional practices [1,2]. To approach these variations and provide an overview of the gold alloys used during the BA, we concentrate this study on Northern Portugal, one of the richest gold-bearing areas of the Iberian Peninsula.

We selected for analysis a large number of gold objects dated from the Early BA (EBA) to the Iron Age (IA), preserved at five Portuguese museums, from sites situated north of the Douro River, in the regions of Trás-os-Montes, Minho and Douro Litoral. The objects were examined using a mobile XRF equipment and an optical microscope. The most significant objects from the typological, regional, or chronological point of view in the same collections that were analysed in the past using other techniques, were reassessed in this work by XRF for the composition of their alloys.

The results obtained show that the EBA objects, containing less than 0.2 wt% Cu, are made from native gold used as found, but the Ag contents suggest regional exploitation of local gold resources. The presence of higher amounts of Ag in some EBA objects found in Galicia [3], a Spanish neighbouring region north of Portugal, confirms regional exploitation of gold. The objects dated from the Middle BA onwards reveal that native gold was used as found until the end of the Late BA (LBA), possibly beginning of the IA, but in the LBA the amounts of Cu start to increase slightly, suggesting addition (in the beginning intentional or unintentional) of this element to native gold. From the Early IA onwards, the alloys start to contain increasing amounts of Ag and Cu, suggesting the use of metallurgical practices that include production of well-established intentional gold alloys and recycling.

The analytical data collected in this work provide the compositional evolution of gold alloys in northern Portugal and, together with the technological description of the objects, can serve in the future as a support to the attribution of objects without context.

References

[1] I. Tissot, M.F. Guerra. In: *Archaeometallurgy in Europe IV*, I.M. Ruiz & A. Perea (eds.), BPH 33, CSIC (2017), 159-167.

[2] M.F. Guerra, I. Tissot. Analytical Study of Overlooked Bronze Age and Iron Age Goldwork from Northwest Portugal, Metalla 26.1 (2022) in press.

[3] Guerra M.F., I. Tissot. Analytical study of Bronze Age goldwork from Northwest Iberia, Journal of Archaeological Science: Reports 39 (2021) 103117.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Arsenical copper production in Old Kingdom Egypt: a case study of Dynasty 4 Giza

Kmošek, J.[1], Odler, M.[2], Fikrle, M.[3], Kochergina, Y.V.[4], Racek, M.[5]

[1]Department of Chemical Technology, Faculty of Restoration, University of Pardubice: kmosekj@gmail.com
[2]Czech Institute of Egyptology, Faculty of Arts, Charles University: martin.odler@ff.cuni.cz, martin.odler@gmail.com
[3]Nuclear Physics Institute of the Czech Academy of Sciences: fikrle@ujf.cas.cz
[4] Czech Geological Survey: julia.kocergina@geology.cz
[5] Institute of Petrology and Structural Geology, Faculty of Science, Charles University

Keywords: arsenical copper; NAA; LIA; SEM/WDS; Old Kingdom Egypt

Austrian team, led by Karl Kromer, worked in the south-eastern part of the Giza necropolis in 1970s and discovered settlement remains from Dynasty 4, from the reigns of Khufu and Khafra (c. 2500 – 2450 BC). It can be inferred from the archaeological context that the debris was coming from craft installations, where craftsmen attached to the pyramid building were working. Among the remains was also metallurgical waste, including crucible fragment and many fragments of slag, prills and even finished artefacts. Our paper focuses mainly on the metallurgical remains, an evidence of arsenical copper production, which are curated in the Studiensammlung des Instituts für Urgeschichte und Historische Archäologie, Universität Wien. Recently, with this study were compared also metallurgical remains currently in Egypt, from the excavations of American, Ancient Egypt Research Associates mission at Giza.

The results presented in this paper include a team of scientists and an Egyptologist, needed for the understanding of archaeological context. A range of archaeometallurgical methods was applied to the remains, in order to identify technological processes: ED-XRF, optical microscopy, HV, SEM/EDS/WDS, NAA, MC-ICP-MS, and µXRD. Analysed copper alloy tools were made from alloy of arsenical copper with 0.7 to 5.3 % of arsenic and their hardness ranges between 65 and 162 HV units. Fragments of vitrified crucibles and samples of slags contain copper and arsenical copper prills, rich in arsenic, and also iron and antimony. These metallic prills were incorporated in the slag phases formed by fayalite, magnetite, wüstite and amorphous glassy phase, and exceptionally also by quartz and copper/iron sulphides. The study brought evidence of an active remelting and casting operations and, most likely, the alloying of copper with arsenic. Thus, Old Kingdom Egyptians most probably actively sought also for sources of arsenic for the production of their artefacts. Trace elements composition and lead isotope ratios of arsenical copper tools and arsenical copper prills are, with a few exceptions, very homogeneous and point out to the provenance in the Sinai Peninsula and/or Eastern Desert of Egypt, similarly to others recently published data of copper alloy artefacts from the Old Kingdom Egypt (Kmošek et al. 2018; Rademakers et al. 2018).

.

S6-O11.569

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Sourcing Mesopotamian Gold – Possibilities and limitations of trace element patterns and isotope ratios

Jansen, M.[1], Hauptmann, A.[2], Klein, S.[2]

[1]Center for the Analysis of Archaeological Materials, University of Pennsylvania Museum of Archaeology and Anthropology
[2] Forschungsbereich Archäometallurgie, Deutsches Bergbau-Museum Bochum

Keywords: gold; alloy components; trace elements; isotope composition; Bronze Age

Gold artifacts recovered from the Early Dynastic Royal Tombs of Ur in Mesopotamia represent a truly unique archaeological assemblage, due both to the size of the assemblage (hundreds of objects) as well as evidence for highly-skilled processing techniques. Other sites in Mesopotamia and beyond provide similar jewelry pieces for the second half of the 3rd millennium. The fact that there are no natural gold sources in Southern Mesopotamia prompts a question concerning the origin of the raw material used to create these artifacts. Gold was imported from other regions such as Anatolia, Iran, Afghanistan, Egypt or even India. The rich corpus of cuneiform tablets from Ur could help to identify trade relations through the naming of specific trading posts. However, the tablets date slightly later than the ED period, and little is known about the provenance of the earlier gold artifacts. Therefore, this project seeks to define the geochemical fingerprint of objects in order to identify the origin of gold from Ur, Ebla, Kish and Nippur through its chemical and isotopic composition. Ideally, this method could trace the gold back to the location of its natural deposit(s). For our analytical work, we used two different methods: first, the chemical composition with main, minor, and trace elements was determined by both non-destructive portable X-ray fluorescence analysis and – for samples – by electron probe micro-analysis and laser-ablation plasma mass spectrometry. Second, the isotopic composition of selected elements (Pb, Os, Cu) was measured by multi-collector plasma mass spectrometry. In special focus are the numerous inclusions of natural alloys of the Platinum Group Elements which were previously analyzed and indicate that the gold derived from alluvial sources panned at a river. The geochemical pattern indicates the use of a single alluvial gold source, or closely related sources of the same geologic origin, for artifacts deriving from different sites. The numerous deposits of Egypt can be excluded due to our analysis, while the geochemical fingerprint pinpoints the source of the gold to rich deposits located in Western Iran or Northern Afghanistan, the latter indicating additional evidence for cultural contact between Mesopotamia and Bactria-Margiana. These findings must be confirmed by a geochemical characterization of those gold occurrences in future, as part of a larger contribution toward our understanding of the extensive trade networks of EBA Mesopotamia.

.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Metals and metallurgical remains from the Outeiro de Baltar hillfort (Ourense, Spain): production of tin and bronze

Figueiredo, E.[1], Fonte, J.[2,3], Meunier, E.[1], Rodrigues, A.[1], Dias, F.[4], Lima, A.[4], Silva, R.J.C.[1], Veiga, J.P.[1], Mirão, J.[5]

[1]Cenimat/i3N, Faculdade de Ciências e Tecnologia, Universidade NOVA de Lisboa, Portugal
[2]Department of Archaeology, University of Exeter, UK
[3]Institute of Heritage Sciences (Incipit), Spanish National Research Council (CSIC), Spain
[4]ICT, Faculdade de Ciências, Universidade do Porto, Portugal
[5]Hercules Lab, Universidade de Évora, Portugal

Keywords: Tin smelting; bronze; Iron Age; Roman transition

Austrian team, led by Karl Kromer, worked in the south-eastern part of the Giza necropolis in 1970s and The site of Outeiro de Baltar hillfort is located in the Ourense Province, in NW Spain, next to a tin rich mineralised area. During the first half of the 20th century tin mining works by the company Somar were undertaken near a stream next to the hillfort. Because the mineralized veins were relatively close to the surface, the works spread through the area and did not reach great depth. During these mining works, in the slope of the hillfort, miners discovered stone walls of round houses and between them they found several pieces of gangue. During the works and by sieving the sediments they also found numerous archaeological artefacts including small metallurgical debris. The archaeological artefacts include fibulae, hair pins and others, which can be attributed to a late phase of Iron Age/Roman transition. Most of these materials were allocated to the Museo Arqueolóxico Provincial de Ourense.

In the present work, we will present elemental and microstructural analysis by XRF, SEM-EDS and XRD of a selection of archaeological materials which are related to metallurgical processes, such as ores, slags and metallic debris found at the site during those mining works. We will also provide a geological context for the site and include in the discussion other nearby mining areas that have also been worked in ancient times.

The analytical study allowed the identification of one tin slag, different ores and bronze metallurgical debris, indicating that by the end of Iron Age tin smelting was being performed at the site as well as metallurgical activities involving bronze (Cu-Sn) with some lead.

The present results point out to the exploitation of the local tin resources by the local communities during Late Iron Age-Roman transition, similarly to what has previously been demonstrated for the Carvalhelhos hillfort (N Portugal) [1]. Given the limited evidence for tin smelting all over Europe during pre-Medieval times, this site is of high relevance within the debate of ancient tin sources in Western Europe.

The present work is part of the project *IberianTin* (PTDC/HAR-ARQ/32290/2017) funded by FCT and COMPETE2020.

[1] Figueiredo E., Fonte, J., Lima, A., Veiga, J.P., Silva, R. & Mirão, J. (2018). Ancient tin production: slags from the Iron Age Carvalhelhos hillfort (NW Iberian Peninsula). Journal of Archaeological Science, 93: 1-16.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeology, geology, rock chemistry, lead isotopes, and 3D modelling: keys to unlocking the 3000 year history of the Lavrion silver mines in Greece

Ross, J.[1,2], Voudouris, P.[3], Melfos, V.[4], Vaxevanopoulos, M.[5], Soukis, K.[3], and Kayafa, M.[6]

[1]*School of Earth Science, The University of Western Australia, Perth, Western Australia.*
[2]*John de Laeter Centre, Curtin University, Perth, Western Australia.*
[3]*Faculty of Geology and Environment, National and Kapodistrian University of Athens, Greece.*
[4]*Faculty of Geology, Aristotle University of Thessaloniki, Thessaloniki, Greece.*
[5]*Ecole Normale Superieure de Lyon, CNRS, Universite de Lyon, lyon, France.*
[6]*Environmental Education Centre, Lavrion, Greece.*

Keywords: Lavrion; Mining; Silver; Copper; Lead isotopes

Unlocking the mining history of the extensive Lavrion silver mining district is central to understanding the availability of silver, lead and copper through the Bronze age in Greece and the Aegean. It is also central to understanding the phenomenal rise of Athens in the first millennium.

This paper integrates the archaeological record, new geological and geochemical data, abundant lead isotope data, and 3D modelling, to review metal production at Lavrion over a period of more than 3000 years. The current archaeological record indicates that silver was mined first and recovered by cupellation, most likely from mid-4th millennium. Nevertheless, surface showings of oxide copper may have led to earlier or simultaneous copper production. Production of lead was also likely.

Archaeology and lead isotopic data tell us that during the Bronze Age, silver, copper and lead from Lavrion was widely distributed through mainland Greece, the Aegean, and some distant locations. At times Lavrion appears to have been the dominant source, especially of lead. However, 19th century observations, in combination with geological and geochemical data, strongly suggest that production of silver, copper and lead from Lavrion would have been constrained for much of its history by the character and low grade of mineralisation being mined at the first contact. This mineralisation occurred in numerous, widely dispersed, small, oxidised, thin and irregular near-surface deposits, likely to support small-scale mining and processing operations with fluctuating output.

Discovery of the deeper third-contact mineralisation, most likely early in the fifth century BC, brought a stunning change. It was much richer, thicker, more continuous, more centralised, and capable of supporting much higher levels of production and processing. A sharp surge in silver output was inevitable. This discovery transformed Lavrion and Athens, and helped usher in the Classical Period. It was also likely to have led to increased shaft sinking to explore the third contact. Shaft sinking and lateral exploration in marble required specialised iron tools that were most likely forged from the high-grade hematite/goethite outcrops at Lavrion. These occurrences may have become a key enabler of future silver production.

.

S6-O14.1502

Session 6 Technology/provenance – metals

POSTER

Vector of technological evolution of blacksmithing production on rural sites of Ancient Rus

Terekhova N.N., Zavyalov V.I.

Institute of Archaeology RAS, Moscow

Keywords: archaeometallography; iron artifacts; rural settlements

Handicraft production on rural sites, in particular ironworking, was an integral part of the economic structure of Ancient Rus. As known, the basis of the feudal state was precisely the villagers. It is impossible to obtain a complete description of the industrial culture of Ancient Rus without studying the role of rural craft.
Significant materials have been obtained for the Myakinino archaeological complex as a result of excavations at the turn of the 20th and 21st centuries. These materials permit to solve many questions of the development of the iron industry on Ancient Rus rural sites. Our paper presents the results of archaeometallographic studies of iron artifacts collection from two villages located in the immediate vicinity of Moscow: Myakinino 1 (second half of the 12th - 15th centuries) and Myakinino 2 (mid-12th - early 17th centuries). It is significant that numerous wastes of metallurgical production were found on Myakinino 1, while they were not recorded on Myakinino 2. The long-term existence of the Myakino archaeological complex allows us to trace the dynamics of the technological development of forge production.
It was studied a large amount of various blacksmithing artifacts: tools, agricultural tools, household items, weapons. The study was carried out in the laboratory of natural sciences of the Institute of Archeology of the Russian Academy of Sciences (Moscow) according to the methodology for studying archaeological objects developed by the authors.
As a result, it was established that the main raw material was the bloomery steel obtained during the metallurgical process. To improve the mechanical properties of artifacts, such techniques as cementation and heat treatment (sharp hardening) were used.
The use of welding-on technologies represents of particular interest. As known, according to the studied Ancient Rus blacksmith artifacts, this technology had become the basis of the urban ironworking from the middle of the XII century. It was also represented at Myakinino villages. At the same time, there is an increase in production using the complex technology in the nearest city - the center of Moscow. Items manufactured in welding-on technology should be considered urban imports, in consideration of that it was no signs of ironworking were recorded on Myakinino 2.

The work was supported by the Russian Science Foundation (grant number 19-18-00144)

S6-P02.061

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Scientific studies of Balkan white metal jewellery of the 19th-20th centuries

Wang, Q.

Institute of Cultural Heritage, Shandong University, 72 Jimo Binhailu, Qingdao 266237, China

Keywords:

Blue copper corrosion was identified on Balkan ‘silver’ jewellery in preparation for the exhibition ‘Traditional jewellery and dress from the Balkans” at the British Museum. How can copper corrosion products be present on white metal thought to be ‘silver’? Does the ‘silver’ contain copper and what its purity is? These questions prompted scientific examination of the metals used for the jewellery, including 90 Bulgarian and 30 Yugoslav pieces. It was hoped that the technical investigation would help better understand the materials used and contribute to the wider study of 19th to 20th century Balkan jewellery.

The objects were analysed by X-ray fluorescence spectroscopy, from which some items were selected for metallography and compositional analysis using SEM-EDX. They were found to have been made using a variety of alloys, including copper-silver-arsenic (Cu-Ag-As), copper-silver, copper-nickel-zinc (Paktong), copper-nickel (cupronickel), copper-zinc (brass), copper, silver, as well as arsenic brass. No association between jewellery types and the alloys used for their production has been found.

Most of the items studied have multi-components which are soldered together. The Cu-Ag-As alloys were found to be dominant among the jewellery and were used for the main components in the majority of the items. Silver plating and gilding have been applied to some pieces.

The alloys of Cu-Ag-As are similar to aghiari, an alloy of copper-silver widely used in Greek traditional jewellery with silver contents of less than 50% and arsenic being added to produce a silvery colour. It seems that aghiari-type alloy may have been widely used in Balkan traditional or folk jewellery to reduce material cost.

S6-P03.074

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

From OXALID to GlobaLID: A substantial upgrade of a well-known data pool of lead isotopes for metal provenancing using R and Shiny App

Rose, T.[1,2,3], Klein, S.[3,4,5], Westner, K. J.[6], Hsu, Y.-H.[3]

[1]Department of Archaeology, Ben-Gurion University of the Negev, P.O.B. 653, Beer-Sheva 8410501, Israel
[2]Dipartimento di Scienze dell'Antichità, Sapienza – Università di Roma, ex vetrerie Sciarra, Via dei Volsci 122, 00185 Rome, Italy
[3]Forschungsbereich Archäometallurgie, Leibniz-Forschungsmuseum für Georessourcen/Deutsches Bergbau-Museum Bochum, Am Bergbaumuseum 31, 44791 Bochum, Germany
[4]Institut für Archäologische Wissenschaften, Ruhr-Universität Bochum, Am Bergbaumuseum 31, 44791 Bochum, Germany
[5]FIERCE, Frankfurt Isotope & Element Research Centre, Goethe Universität, Altenhöferallee 1, 60438 Frankfurt am Main, Germany
[6]Ecole Normale Supérieure de Lyon, CNRS, Université de Lyon, Lyon

Keywords: archaeometallurgy, lead isotopes, provenancing, database, web application, FAIR

Lead (Pb) isotope geochemistry is an approved key method in archaeological sciences to reconstruct the resource provenance of metals and trade networks of the past civilisations. Successful application and interpretation of Pb isotope signatures of metal artefacts rely crucially on the published ore data, which are partly only available from pre- or re-digitalised publications. Most Pb isotope reference data collections were compiled by individual working groups, usually focussing on their projects and regions of interest. A great step towards a large-scale collection of Pb isotope data came with the release of the OXALID database in the early 2000s, which has benefited the scholars in the natural science discipline as well as the more untrained users from the archaeological community. Still up today, OXALID is the most used and cited source for reference data, despite the accumulation of many additional data sets since then. All of them are set up as static data collections, limiting the possibilities to expand, correct, and modify them with the publication of newer results or analyses. Additionally not all of them are easily available for people from across the world and only recently compilations for regions outside of Europe and the Mediterranean became widely available.

Riding the wave of open science and new data infrastructures, the authors are endeavouring to digitalise and construct a global Pb isotope data base using the statistical environment R and Shiny App. The presentation will demonstrate this highly promising application for the modernisation of archaeometry as an applied geoscience discipline.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometallurgical research into the ironworking activities of the Medieval Harbour at Hoeke (Belgium)

Biernacka, P.[1,2], De Clercq, Wim.[1], Dewaele, S.[3], Vanhaecke, F.[2], De Grave, J.[3]

[1]Department of Archaeology, University of Ghent, St.-Pietersnieuwstraat 35, 9000 Gent, Belgium
[2]Atomic & Mass Spectrometry – A&MS Research Unit, Department of Analytical Chemistry, Ghent University, Krijgslaan 281 - S12, 9000 Gent, Belgium
[3]Laboratory for Mineralogy and Petrology, Department of Geology, Ghent University, Krijgslaan 281 S8, 9000, Ghent, Belgium

Keywords: Medieval Harbour of Bruges; archaeometallurgy; iron slags; smithing; metallography.

Despite a number of archaeological studies on Medieval harbour in Bruges, little attention has been paid to metallurgical activities in this region. Recent excavations, however, revealed great quantities of iron slag, fuel and other wastes demonstrating the presence of large-scale iron working activities at the site.
This study presents new archaeometrical data on iron production at one of the Late Medieval outer harbours of Bruges (Belgium), located at Hoeke and focuses on the geochemical and mineralogical analysis of iron slags.
Samples were primarily analysed by portable X-ray Fluorescence (pXRF) and X-ray Diffraction (XRD) determined the bulk elemental and mineralogical composition of the examined material, then subjected to the further study by using reflected light optical microscopy to characterise the microstructural features of iron slags. This approach helped identify which step of *chaîne opératoire* the slags were involved in and provided a first insight into iron production process carried out onsite.
Distinctive microscopical characteristics such as presence of Plano-Convex Bottom (PCB) slags and the occurrence of hammerscales give evidence of smithing activity. A geochemical and metallographic examination of iron slags show a complex mixture of iron oxides (wüstite FeO and mainly magnetite Fe_3O_4), fayalite (Fe_2SiO_4) and quartz SiO_2. Microstructural analysis highlights the presence of spinifex texture in some examples (indicating a rapid cooling rate), occurrence of metallic iron inclusions and incorporation of partially dissolved hammerscales in the slag. Moreover, the investigated slag has a significant level of quartz which could have been used as a flux.
The results of these analyses support the hypothesis that smithing iron production process took place at Hoeke. Small macroscopical and mineralogical discrepancies in iron slag assemblage suggest the use of different techniques during smithy activity such as fashioning an object and forging. This present study makes noteworthy contributions to understand the technological aspects of iron production in late Medieval Bruges. Considering there is no iron ore extraction possible at Hoeke or in the wide region, iron was imported from other, distanced areas. Therefore, the isotope analysis will be performed on iron slags from Hoeke to evaluate Pb isotopes composition using Inductively Coupled Plasma Mass Spectroscopy (ICP-MS).

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

An experimental approach on the Kythnian metallurgical furnace from Paliopyrgos/Aspra Spitia

Giannakopoulou, M., Olliffe, T., Doonan, R., Marks, Y.

University of Sheffield

Keywords: metallurgy; experimental archaeology; Early Bronze Age Aegean; copper smelting; perforated furnace

The investigation into Early Bronze Age Aegean copper smelting has been a very interesting topic during the last few decades, with yet unanswered questions. Remains of a furnace have been found at Aspra Spitia/Paliopyrgos on Kythnos, at West Cyclades. In contrast to the Chrysokamino furnace from East Crete, the wall fragments from the Kythnos furnace are thicker and the perforations have an inclination averaging around 40º. At the same time the lack of bellows indicates that the smelting was conducted by wind force. The reconstruction of the Kythnian furnace aimed to a observe and record the processes of the furnace, from its construction to its firing and smelted product. The effects of the different thickness of the wall, the inclination of the perforations, the operation without bellows and the efficiency of the furnace were investigated.

The furnace was constructed from 60 kg of clay with the use of a cone mould and 37 perforations with an inclination of 45º were opened on its walls. During the firing the fan was placed 2.5m away from the furnace, resulting in an average speed of 4,4 m/s, which equals to 3 on Baeufort scale. This is slower than the normal speed of meltemia, the strong Aegean winds (4-7bft). The fuel used was charcoal, in total 13 kg and the 1,4 kg of malachite ore contained 36,5% copper. Throughout the 4,5 hours of the firing, temperatures were recorded every 10 minutes from 3 points; top, middle and bottom. The addition of the fuel was also recorded.

The temperature remained generally stable throughout the procedure at all three points, whilst the middle temperature exceeded 1200ºC within 30 minutes of the fan being on. Thus, not only was the use of bellows unnecessary, but also the procedure could take place during lower windspeeds than those of the meltemia. The average burn rate of the furnace was 16kg/h. After the smelting the furnace walls, although cracked, remain intact and could have been possibly used for another smelting. The XRF analysis of the furnace walls showed that the highest Cu level was close to the fusion zone and even other parts of the wall contained Cu in relatively high levels. The efficiency of the furnace was impressive, reaching 60.3%. The 308 g of copper recovered was of 98,9% purity, with a normal small percentage of iron which would be reduced if remelted.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A multidisciplinary approach to the study of Polish silver denars minted by the early Piasts concerning their provenance and archaeological aspects

Matosz, M.[1], Hrnjić, M.[2,3], Röhrs S.[3], Denker, A.[4], Weisser, B.[5], Stoess Ch.[5], Bolewski, A.[6], del Hoyo-Meléndez, J.[1]

[1]Laboratory of Analysis and Non-Destructive Investigation of Heritage Objects, National Museum in Kraków, ul. Piłsudskiego 14, 31-109 Kraków
[2]Centre for Urban Network Evolutions, School of Culture and Society, Aarhus University, 8000 Aarhus, Denmark
[3]Rathgen-Forschungslabor, Staatliche Museen zu Berlin, Stiftung Preußischer Kulturbesitz, Schlossstr. 1a, 14059 Berlin, Germany
[4]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin, Germany
[5]Münzkabinett Staatliche Museen zu Berlin, Staatliche Museen zu Berlin, Stiftung Preußischer Kulturbesitz, Geschwister-Scholl-Str. 6, 10117 Berlin, Germany
[6]Faculty of Physics and Applied Computer Science, AGH University of Science and Technology, Reymonta 19, 30-059 Kraków, Poland

Keywords: silver denar; medieval Poland; x-ray fluorescence; high energy particle induced x-ray emission; neutron activation analysis

Near-surface analytical techniques, such as X-ray fluorescence (XRF), high energy particle induced X-ray emission (PIXE) as well as particle induced gamma-ray emission (HE-PIGE) and neutron activation analysis (NAA), which provides an analytical data from greater depths, have been used to study more than one hundred medieval denars of the Piast dynasty. Two collections housed in the National Museum in Krakow (69 denars) and Numismatic Cabinet of the National Museums Berlin (39 denars), were evaluated in the present study. All of them were minted between 995 and 1020 by two of the first Polish kings from the early Piast dynasty, namely Boleslaus the Brave and his son, Mieszko II Lambert.

An archaeometric approach for the study of these denars is fundamental to establish any possible correlation between the chemical composition of these objects and their historical context, since it concerns the beginning of a Polish state. The analysis had the objective of gaining a better understanding of the chemical composition of the alloys utilized in the early Polish coinage system.

The major elements present were Ag and Cu, while minor elements such as Pb, Au, Bi, Hg and Zn were also detected. An evaluation of the results obtained with the different techniques showed that the content of Ag and Cu near the surface and in the bulk varies in some of the coins. Quantitative analysis revealed a distinction between Boleslaus the Brave and Mieszko II Lambert mintage procedures. In addition, a correlation between Ag, Au and Bi was observed after evaluating the analytical results providing a first step in the determination of silver provenance.

S6-P08.141

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Copper production and use during the Third Millennium BC in the Western end of Iberian Peninsula: the testimony of Castro de Chibanes (Portugal)

Valério, P.[1], Soares, J.[2], Alves, L.C.[1], Silva, R.J.C.[3], Araújo, M.F.[1], Tavares da Silva, C.[2]

[1]C2TN, Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa.
[2]Museu de Arqueologia e Etnografia do Distrito de Setúbal (MAEDS).
[3]CENIMAT/i3N, Department of Material Science, NOVA School of Science and Technology, NOVA University of Lisbon.

Keywords: Arsenical copper; Crucibles; Artefacts; Chalcolithic; Iberian Peninsula

Early archaeological works during the beginning of the last century suggested that the archaeological site of Castro de Chibanes (Setúbal Peninsula, Portugal) had been occupied from Neolithic to the Roman period. Recent archaeological excavations conducted by MAEDS clarified the different occupation periods through stratigraphy, ceramic typologies and radiocarbon analyses. In this settlement, the copper metallurgy arises during the middle and second half of the third millennium BC, with local production activities evidenced by the occurrence of ceramic crucibles with copper-bearing prills. A complete melting crucible with pouring lip and four feet deserves a special highlight. Archaeological works also recovered small metal tools such as awls and a chisel, although with uncertain chronology. The present work concerns a chemical (p-XRF and PIXE) and microstructural (optical microscopy and SEM-EDS) characterisation of metallurgical remains and metal artefacts from Castro de Chibanes. Additional items (metal prills and artefacts) from two nearby coeval and related sites, namely Rotura and Pedrão, were also studied to enhance the knowledge about the early metallurgy in this western region of Iberian Peninsula. The main results point to the production and use of copper with variable contents of arsenic. Moreover, the post-casting manufacture of artefacts comprised hammering and annealing operations, although with incipient working conditions that prevented the compositional homogenisation of those low-arsenic copper alloys. A few artefacts proved to be composed of bronze or leaded bronze alloys, thus belonging to the later occupation phases of Castro de Chibanes and Pedrão. Overall, the third millennium BC metallurgy in Setúbal Peninsula seems to present analogous features to what has been identified in the neighbouring and best-studied regions of the Portuguese Estremadura and Southern Portugal.

Acknowledgements
The support of Fundação para a Ciência e a Tecnologia (FCT) to C2TN (UIDB/04349/2020) and CENIMAT/i3N (UIDB/50025/2020-2023) is gratefully acknowledged. Authors also recognise the financial support of Associação de Municípios da Região de Setúbal (AMRS).

S6-P09.3011

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Where do you come from? Copper ores in archaeological contexts

Montero Ruiz, I.[1], Lackinger, A.[2], Murillo-Barroso, M.[2]

[1]*Instituto de Historia, CSIC. C/Albasanz, 26-28, 28037, Madrid (Spain)*
[2]*Departamento de Prehistoria y Arqueología, Universidad de Granada, Campus de Cartuja s/n 18074 Granada (Spain)*

Keywords: Copper ores; Provenance Analyses; Lead Isotope Analyses; Later Prehistory; Iberian Peninsula

One of the main issues in current archaeometallurgical research is defining the catchment areas of copper resources in order to infer the organisation of metallurgical production at the local, regional or supra-regional scale. Consequently, provenance studies based on Lead Isotopes Analysis (LIA) has become recurrent (Montero Ruiz, 2018), however, lead isotope analysis requires a comparative geologic dataset. Currently, the main and bigger mineralisations in the Iberian Peninsula are well characterised with around 1300 analysis. Some of these mines have been radiocarbon dated between c. 3200-1500 BC and thus mining works during the Recent Prehistory are well documented. But the diversity and wide distribution of copper resources in different geological domains all over the peninsula makes difficult to cover in detail all the small copper occurrences. Moreover, extensive modern mining works may have erased any evidence of these small outcrops.

Some copper ores recovered in archaeological context from different regions (Catalonia, Central Spain or the Southeast), mainly from Chalcolithic and Early Bronze Age, have isotopic signatures that are not possible to match with the known geological data, and especially with those mines with radiocarbon dates. This fact suggests the use of diverse small scale resources and reinforces the idea of local productions based on a wide variability of resources with not dependence or restriction to a main or unique mining district (eg. Murillo-Barroso et al. in press).

In this paper we present a different approach to lead isotope data of these copper ores recovered in archaeological sites. We define isotopic fields for each archaeological site or region based on the copper ores recovered in archaeological contexts to define the field of resources used by each site or region regardless their mine of provenance. This information can shed some light on different regional patterns of copper ore catchment and can be campared with metal objects from the same or close archaeological sites. The absence of concordance between copper ores and objects of one single site would indicate exchange of metal objects between different sites.

References

Montero Ruiz, I. (2018): La procedencia del metal: consolidación de los estudios con isótopos de plomo en la Península Ibérica. *Revista d'arqueologia de Ponent*, 28: 311-328.

Murillo-Barroso, M.; Montero Ruiz, I.; Camalich Massieu, M.D.; Martín Socas, D.; Labaune, M.; Cattin, F.; Nieto, J.M.; Martinón-Torres, M. (In press): Raw material procurement and selection in Iberia's earliest metallurgy.

S6-P10.155

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The effect of socio-economic dynamics in the selection of tin-bronze alloying techniques: A case study from Iron Age Iberia

Montes-Landa, J.[1], Pons Brun, E.[2], Rovira Hortalà, C[3]., Martinón-Torres, M.[1]

[1]University of Cambridge
[2]Museu d'Arqueologia de Catalunya-Girona
[2]Museu d'Arqueologia de Catalunya-Barcelona

Keywords: tin-bronze; alloying techniques; technological choices; slag

From co-smelting through cementation to scrap recycling, there are five ways to make tin-bronze. We can only tell them apart through the analysis of production residues. Interestingly, the evidence indicates that the oldest techniques are not always replaced by the most modern and 'advanced' ones, and that several techniques often co-exist at the same production contexts. This trend demands to be explained.

Selection of a technique(s) is conditioned by its performance characteristics, and by the socio-economic dynamics related to bronze production. While the former are universal and easier to establish, the latter demand linking contextual parameters to the choices observed archaeologically. This will allow explaining the appearance of different techniques, their acceptance, development, rejection and/or co-existence over time: i.e. to understand the production strategies embedded in the bronze making system under study.

We present a case study from two adjacent, indigenous sites in Iron Age Northeast Iberia: Mas Castellar (Pontós), and Ullastret. Slags and slagged technical ceramics were analysed using pXRF, OM and SEM-EDS to relate their microstructures and compositions to specific alloying techniques and characterise the production strategies.

Mas Castellar followed a diversified raw material acquisition strategy and a versatile selection of techniques. Conversely, the two assemblages from different but contemporaneous Ullastret nuclei (Puig de Sant Andreu and Illa d'en Reixac) show distinct patterns that favoured different alloying techniques over the rest. Moreover, our results point to an absence of technological transmission of the co-melting technique to indigenous sites, in contrast with the results of a previous study at Emporion, the nearby Greek colony.

The diverse indigenous alloying strategies observed are mainly dictated by the socio-economic position of each site. If they were, on the contrary, mainly conditioned by technical limitations of each technique and/or environmental aspects (i.e. ore availability), this would produce a more uniform pattern of choice in these nearby sites.

Our results demonstrate the potential of our approach to reconstruct bronze making systems. Our on-going project expands the chronology and geographic scope of this case study to understand the development and evolution of the bronze making system in Northeast Iberia from the Chalcolithic to the Iron Age. This is the first comprehensive study focused on technological choices concerning bronze alloying techniques.

S6-P11.179

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Where to smelt: at the mine or in the city? The case of Pistyros, N. Greece (5th-3rd c. Bce)

Nerantzis, N.[1], Photos-Jones, E.[2], Papadopoulos, S.[3]

[1]Marie Skłodowska-Curie Fellow, Centre de Recherches en Archéologie et Patrimoine, Université Libre de Bruxelles - CP 133/01, Avenue F.D. Roosevelt, 50 B-1050 Bruxelles
[2]Analytical Services for Art and Archaeology (Ltd), Glasgow G12 8JD, UK / Archaeology, School of Humanities, University of Glasgow, Glasgow, UK
[3]Ephorate of Antiquities of Drama, Archaeological Museum of Drama, Greek Ministry of Culture, Patriarchou Dionysiou 2, 66133 Drama, Greece

Keywords: metallurgy; litharge; speiss; silver; coinage

Ongoing excavation (2014-2019) of a Thasian *emporion*, identified with Pistyros, near Kavala in N. Greece, provided, for the first time, a unique opportunity to study large scale smelting activities within an urban context. The settlement of Pistyros (second half of the 6th century BC -1st century BC) revealed nearly 0.6 of a ton of slag dated to the Classical period. About 2-2.5 km NE of the site, near the modern villages of Perni and Petropigi, there are mining galleries dated tentatively to Classical/Hellenistic period. Analyses of small scatters of slag, found there, suggest compositional similarities (at least based on pXRF analysis) with slag from Pistyros. So, given such close proximity between mine and 'city', why smelt in the city?

By focusing on an area bounded by the three sites (Pistyros-Perni-Petropigi), this paper attempts to focus on the clarification of the type of metallurgy practiced at both contexts, urban and rural, for the period concerned. How does Pistyros metallurgical waste compare with that from Perni-Petropigi? Is it the same metallurgy practiced at both sites? Given the polymetallic nature of the local ores (Au bearing Fe-Mn, Pb-Ag and Zn bearing Fe-Mn, Au bearing pyrite-arsenopyrite) is it about precious metals extraction at both sites? Or base metals in one and precious metals in the other?

By delving into the Pistyros/Petropigi metallurgical waste, this paper seeks to resolve issues pertaining to the complex, and still little understood, relationship between the indigenous Thracian tribes and the Thasians, colonists from the nearby island of Thasos. Numismatic evidence suggests that both Thasians and Thracians mint separate coinage with strikingly similar iconography (Silenus with nymph/centaur with nymph). Is it a case of shared responsibilities ('collaborative mining'?) as highlighted by (Picard 2006) whereupon Thracians work the mines and Thasians smelt 'their' ore, payment being made in kind i.e. in the shape of large staters with shared iconography? This work expects to shed some light in a region where most information derives from the Greek sources and relatively little from the local material culture.

References:

Picard O. 2006 Mines, monnaies et impérialisme: conflits autour du Pangée (478-413 av. J.-C). In A-M. Guimier-Sorbets, M.B. Hatzopoulos and Y. Morizot (eds.) *Rois, cités, nécropoles: institutions, rites et monuments en Macédoine: actes des colloques de Nanterre, Decembre 2002 et d' Athènes, Janvier 2004,* Centre de Recherches de l'antiquité Grecque et Romaine, Fondation Nationale de la Recherche Scientifique. Meletemata 45. Paris: de Boccard, 269-283.

S6-P12.182

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Mobility of metals in the Iron Age. Archaeometry might be the answer to historical questions

Danielisová, A.[1], Bursák, D.[1], Strnad, L.[2], Trubač, J.[2], Pajdla, P.[3]

[1]*Institute of Archaeology CAS, Prague, v.v.i.*
[2]*Faculty of Science, Charles University in Prague*
[3]*Faculty of Arts, Masaryk University in Brno*

Keywords: Iron Age; provenance analyses; lead isotopes; trace elements; multivariate statistics

Exact data on trends in using metals in prehistory are solid part of the archaeological narration and their importance is indisputable. There are quite numerous studies on the provenance and production of the copper and its alloys in the areas like Mediterranean or the Alps, however, the case for central European Iron Age remains largely obscure. For decades there was a paradigm that primarily local deposits were exploited and the proximity to sources of polymetallic ores significantly influenced the settlement structure, distribution patterns and socio-political networks. This assumption, however, was not backed by much (if any) scientific data.

Within our current project we have analysed several hundreds of copper alloy objects spanning the time period of the so called Celtic expansion (4th century BC), through the industrial development (3rd century BC) and founding the oppida agglomerations (2nd- 1st cent BC) to the massive migrations of Germanic tribes (1st century AD). Archaeological record and namely the exact data give a rather confused picture. While, during the period in question, we have recorded several cultural and socio-economic changes (massive migrations, new materials, introduction of coinage, change in burial customs), our data are extremely difficult to interpret in relation to addressing these issues. Therefore we have developed a combined archaeological and archaeometric approach based on the principles of provenience analyses of materials which is compared with the evidence on mobility of people. Our methodology involves evaluation of bulk and trace composition (including less usual tracers), accompanied by isotopic (namely Sr, Pb) tracers.

Possible structures in data are evaluated statistically by multi-variate methods. Analysed finds come from different stages of the life cycle of metals, from the raw material to scrap. We follow the line of introduction and standardisation of alloys, the question possibly related to the primary acquisition and/or recycling or various sources. Even in periods when traditional provenance questions are much more difficult to answer, we believe that mixing of sources tends to follow certain trends that can be traceable by available analytical methods. By establishing these “trends” historical events related to “mobility” can be explained.

S6-P13.185

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

From bronze to brass: Investigating continuity and change in early Islamic metallurgy

Davis, M.[1], Mongiatti, A.[2], Simpson, S.J.[2], Martinón-Torres, M[3]

[1]The University of Cambridge and The British Museum
[2]The British Museum
[3]The University of Cambridge

Keywords: Archaeometallurgy; Islamic; brass; alloys; technology.

Late Antiquity saw a prevalence of bronze alloys across the Middle East and Western Asia, while middle and later Islamic societies were characterised by the popularity and sophistication of their brass wares. This project seeks to understand this transition in alloy choice through the study of archaeological objects excavated from sites across the region.

For this study, we are undertaking XRF and SEM-EDS analysis of excavated materials from the early Islamic sites of Siraf, Merv and Kush (UAE), focusing in particular on small, everyday objects that were manufactured for personal use and decoration. Early results from Siraf, a key port on the Persian Gulf in this period, reveal the range of alloys employed by early Islamic metalworkers, as well as evidence for controlled alloy selection in both bronze and brass and their application to specific object forms. Indications that silver and high-tin alloys were used to enhance such everyday objects are also a reminder of the wealth and prosperity of the citizens of this major trading hub in the early Islamic period.

In addition to these excavated materials, we are also analysing a range of copper alloy objects in the wider British Museum collection. These include objects typologically dated to Late Antiquity or the early Islamic period, as well as objects associated with historic excavations in Nineveh and Babylonia. Together, these analyses will allow us to identify more detailed spatial and temporal trends of alloy usage in the region, and, through this, an understanding of cultural interaction and personal choice in the early Islamic period.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Diverse Strategies for Copper Production in Chalcolithic Iberia

La Duc, E.[1], Montero-Ruíz, I.[2], Freestone, I.[3], Martinón-Torres, M.[1]

[1]University of Cambridge, Cambridge, U.K.
[2]CSIC, Madrid, Spain
[3]UCL, London, U.K.

Keywords: archaeometallurgy; copper; technical ceramics; Chaclolithic

Our knowledge of early copper metallurgy in the Iberian Peninsula is mostly based on analysis from well-studied regions in the Southeast and Southwest, which may have led to an overly homogeneous picture. This paper focuses on two recently recovered Chalcolithic assemblages outside these traditional research foci: Cueva del Cañaveralejo (Córdoba) and Lugar Viejo III (Zaragoza). Two slag cakes from Cueva del Cañaveralejo and two slagged crucibles from Lugar Viejo were analyzed using optical microscopy and scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS). Analysis of the composition and microstructure of the artifacts suggests they are related to primary copper production, namely smelting in crucible-furnaces under relatively oxidizing conditions, as is standard for this period. However, the slags from Cueva del Cañaveralejo are atypical in their large size (c. 125 g each), fayalitic composition, unusual efficiency as demonstrated by a low copper content, and lack of arsenic; furthermore, the high sulfur content raises the possibility of the use of sulfidic ores. On the other hand, the slagged layers on crucibles from Lugar Viejo indicate the production of arsenical copper, the customary alloy found in this period. Of particular interest is the use of organic temper in the crucibles from Lugar Viejo, a practice found at the nearby site of Moncín but rare at other sites in Iberia. Results from both sites are compared against published data from well-known sites such as Los Millares, Las Pilas, Almizaraque, and Bauma del Serrat del Pont. The new data from Cueva del Cañaveralejo and Lugar Viejo reinforce the interpretation of metallurgy in the Iberian Peninsula as a low-skilled, conservative technology but also indicate the need for more research into regional variations.

S6-P16.208

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The metal sheathing and fastenings of wooden ships. Archaeometallurgical study of Carron, Ancla Macuca, HMS Forth and El Pesquero sites located at the Gulf of Mexico (late 18th to mid 19th Centuries)

Arano, D.[1], Ciarlo, N.C.[2], Bethencourt, M.[3], Carrasco, G.[4], Barba, H.[5]

[1]Sección de Restauración, Instituto Nacional de Antropología e Historia, Campeche, México.
[2]Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET) – Instituto de Arqueología, Facultad de Filosofía y Letras, Universidad de Buenos Aires – Programa de Arqueología Histórica y Estudios Pluridisciplinarios, Departamento de Ciencias Sociales, Universidad Nacional de Luján, Argentina.
[3]Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica y Química Inorgánica, Facultad de Ciencias del Mar y Ambientales, Instituto de Investigaciones Marinas (INMAR), Universidad de Cádiz, España.
[4]Departamento de Historia Moderna, Universidad de Cádiz, España
[5]Subdirección de Arqueología Subacuática, Componente Península de Yucatán, Instituto Nacional de Antropología e Historia, México

Keywords: archeometallurgy; nautical and underwater technology; 18th and 19th century shipwrecks

After four decades of research, the Vice-Directorate of Underwater Archaeology of the National Institute of Anthropology and History in Mexico (SAS-INAH) recorded around 400 underwater archaeological contexts in the Gulf of Mexico and Mexican Caribbean. One of the great challenges has been the correlation of historical, archaeological and material information for dating and assessing the provenance of the vessels. The archeometallurgical analysis of copper-base sheathing and hull fastenings (bolts and nails) stand as a relevant mean for the interpretation of modern shipwrecks. During the 18th century and the dawn of the 19th century, the naval construction in the European nations was focused on increasing the quality of wooden vessels for the Atlantic sailing, given the conditions of tropical waters. For instance, the introduction of metal sheathing to protect the ship's bottoms from the action of wood boring organisms, was an essential measure. The studies developed in metallurgy and mining, combined with archeological data from shipwrecks, historical research and characterization of sheathing and fastenings samples from the shipwrecks Carron, Ancla Macuca, HMS Forth (1849) and El Pesquero, located at Campeche Bank and coast, allow assessing its main technological features. Furthermore, the results obtained helped to date the remains and discuss the sites formation processes, in particular the existence of palimpsests on two of the sites studied.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

First approach to the Prehistoric Metallurgy of Alto Guadiato valley (NW Córdoba, Spain)

Perez-L'Huillier, D[1]., Gonzalez Zambrano, P[1]., Lacinger, J[1]., Cristo Ropero, A[1]., Murillo Barroso, M[1]., Contreras Cortes, F[1]., Vera-Rodriguez, J.C.[2]

[1]*Universidad de Granada. Dpto. de Prehistoria y Arqueología*
[2]*Universidad de Huelva. Dpto. de Historia, Geografía y Antropología*

Keywords: Metal, Late Prehistory, color, XRF, South of Iberian Peninsula

In this work we present a study on copper-based metal objects from Recent Prehistory, from the Alto Guadiato Valley. This valley is located in the northwestern part of the province of Córdoba (Andalusia, Spain) and acts as a natural passageway between the Guadalquivir and the Guadiana Valleys.The set is composed by 33 copper-based objects, 4 chisels, 4 palmela points, 15 awls, 1 axe fragment, 1 punch, 1 rod, 1 saw, 1 plate and 4 indeterminate.We present a elemental composition analysis by X-Ray Fluorescence (XRF) and a colorimetry study with the objective of assesing color variation according to the amount of the different elements (Cu, As, Sn). A Cu-As-Sn ternary color diagram, as well as photos and colorimetric measurements were used. We also present the evolution on the appearance of metals as a consequence of the surface patina that appears, taking meassurements every 7 days over 3 months. This will allow us to document the evolution of patina and color according to the percentage of tin and arsenic in copper objects and evaluate the role that metals appearance may have played on their social valuation.

S6-P19.1554

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Nails and anchors - quality and smithing technology of maritime ironwork in the Viking Age

Jouttijärvi, A.

Heimdal-archaeometry

Keywords: Iron; anchors; nails; smithing; Viking

It is likely that anchors of iron for use in Viking ships were the largest type of object that was made by forging in the Viking Age. Dated anchors from Denmark and Norway show that they most often weighed about 10 kg, but in some cases weighs up to 30 kg. It has therefore been very large pieces of iron to handle when heated in the forge, and by forging on the anvil. The production must therefore have required a specialized knowledge.

In order to describe the technology and quality of the iron used, a number of anchors have been metallographically investigated. Despite the big difference in size, there seems to be a relatively uniform technology, which however, at least in some places, seems to have been in use almost up to now. However, it is possible on the basis of the structure of the iron, and especially slag inclusions in it, to see that this is Bloomery iron, which differs significantly from the later iron. The different processes: bloomery smelting, fining of pig iron and puddling leaves slag inclusions with different compositions and can be recognized by analysis. A total view of technology, material and form may therefore form the basis for an assessment of anchors found without context. A development of technology and production of iron from the Viking Age and until the beginning of the 19th century is outlined

It has been suggested that for maritime purposes, iron with a high content of phosphorus has been deliberately used as it would provide better resistance to corrosion. However, from the studies of the anchors and a number of analyzes of iron nails from ships, this cannot be demonstrated unequivocally. Phosphorous iron and steel might however have been deliberately used for nails in different parts of boats and ships.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

ULFBERHT - a fragment of a sword and a very special type of steel

Jouttijärvi, A.

Heimdal-archaeometry

Keywords: High carbon steel; swords; Viking Age; carburization; metallography

A small, wedge-shaped fragment of iron was found at an excavation site at Tørslev Hage near Roskilde fjord, off the Skuldelev barrier. Upon examination, it was found to consist of steel with an unusually high carbon content. Therefore, it is likely that this is a fragment of a ULFBERHT sword, and it is possible that the steel came from India or Pakistan.

In recent years, fragments of the so-called Damascus steel have been found several times in connection with archaeological traces of smiths from the Viking Age in Scandinavia. This has, to our knowledge, not previously been identified, and it has been believed that blacksmiths in Scandinavia did not master the special forging technique to be used to forge this type of steel. Usually the blades of ULFBERHT swords are believed to be forged in the Frankish area.

Previous studies on ULFBERHT swords have shown that only those swords whose name is spelled + VLFBERH + T are made of high-carbon steel (Williams 2009).

Simultaneous imitations of ULFBERTH swords with similar inscriptions may be copies of poor quality, but a sword from Telemark in Norway shows that they can also be of high quality, although not the same special type of steel.

Recently, however, traces of the production high carbon steel by carburization of common iron or steel, have been found on a Norwegian smithing site. The pieces of steel produced have been small but sufficient for the cutting egg on a knife.

It is therefore possible that in Scandinavia there may have existed a local tradition of making and using a material similar to the Indian and Pakistani crucible steel. A review of previous metallographic studies shows that high carbon steel does in fact occur from time to time in knives and other tools, especially from the Viking Age. So far, however, in most cases they have been regarded as random deviations from the ordinary.

Williams A., 2009, A metallurgical study of some Viking swords, Gladius, 29: 121-184.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometallurgical study of an ornamental copper-based figurine with gilded surface decoration obtained by means of a gold-mercury (Au-Hg) amalgam from s'Olivera (Ibiza, Balearic Islands)

Sureda, P.[1,3], García-Vuelta, O.[4], Gener-Moret, M.[2], Montero-Ruíz, I.[4], Marí-Casanova, J.[5], Graziani, G.[6]

[1]Instituto de Ciencias del Patrimonio. Incipit-CSIC
[2]McDonald Institute for Archaeological Research. University of Cambridge
[3]Homerton College, University of Cambridge
[4]Instituto de Historia. Consejo Superior de Investigaciones Científicas.
[5]Universitat de les Illes Balears.
[6]Universitat Autònoma de Barcelona

Keywords: Archaeometallurgy; Amalgam; XRF; SEM-EDX; Metallography

The archaeological site of s'Olivera (Ibiza, Mediterranenan Sea), gathers a large area of crop ditches dated on the Late Punic period, roman building remains, Byzantine tombs, buildings and also reoccupations of the area during the Andalusian period (III centuries BC - XIII AD). The archeometallurgical study on the metallic objects from the site is conducted on a set of 116 copper-based items typologically diverse (i.e. coins, ornaments, small artifacts and ingots stand out).

The current case study focuses on one specific object from the site: an ornamental copper-based figurine with gilded surface decoration obtained by means of a gold-mercury (Au-Hg) amalgam. The object is related to the Roman occupation of the site, specifically between the I-III centuries AD and has been analysed combining different methods and techniques: X-ray fluorescence spectrometry (XRF), scanning electron microscopy with microanalysis by Energy Dispersive X-ray Spectroscopy (SEM-EDX) and metallography by optical microscopy observations.

Our results reveal interesting information on the object production process and the complexity of the decoration involved, including specific details concerning the amalgam technique. Obtained data could be also relevant to approach its particular functionality, that may be related with magic/religious practices during the Roman period in Ibiza. Besides, crucial data on the corrosion interacting with Au-Hg slides has been collected and represents a first approach for future restoration actions.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Sr-Nd isotope fingherprinting as a tool for Bronze coins provenance: the case study of Nabataean Kingdom

Ercoles, I.

Department of Earth Sciences, Sapienza University of Rome, Piazzale A. Moro, 5, 00185, Rome, Italy

Keywords: Provenance study; Strontium Isotopes; Neodymium Isotopes; Coin; Timna Deposit (Arabah Valley)

This innovative study was developed by means of combined use of Strontium (Sr) and Neodymium (Nd) isotope ratios and chemical elemental analysis carried out on 40 copper-based coins, belongingto the Nabataean Kingdom and hailing from the Israel Antiquities Authority at the Israel Museum (Jerusalem).
The Nabataean Kingdom was a political entity, composed of a nomadic tribe, expert in trades, which flourished in modern-day Jordan, Israel and Syria between the 4th century B.C.-106 A.D.
Sr-Nd isotope ratios are measured to detect the metal provenance source of the coin, prill and ore samples from Timna area, the most important local copper source in Levant zone.
The elements concentration in the coin-solutions was analyzed using ICP-AES at the Geological Survey of Israel while the isotopic compositions of Sr and Nd were measured with both TIMS and MC-ICP-MS- “Nu Plasma” at IGAG-CNR laboratories at La Sapienza and at the Geological Survey ofIsrael.
Lead isotopes, commonly used to trace metal provenance source, are valid geochemical tracers but sometimes could not be sufficient to identify the original ore deposits due to mixing/re-melting processes.
The use of Sr and Nd isotope ratios as geochemical tools could overcome this obstacle, but they havenot yet been explored probably for the low elements' concentrations in metals.
The results show that Sr and Nd isotope ratios can trace the metal provenance and combined with those of Pb allow obtaining more complete information.
The ubiquitous geological database of the Sr isotope ratio and the resistance to the weathering of theNd allowed to investigate the Nabataean copper archaeometallurgy, identifying the Arabah Valley asthe local source of Nabataean copper coins.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Bronze votive objects from the sanctuaries of Tegea, Arcadia, Greece, 9th-7th c. BCE: A technological study using optical microscopy (OM), portable micro X-ray fluorescence spectrometry (micro-XRF) and micro particle induced X-ray emission analysis (micro-PIXE)

Kladouri, N.K.[1,2], Karydas, A.G.[2], Orfanou, V.[3], Tsampa, K.[2], Kantarelou, V. [2], Karapanagiotou, A.V.[4], Zacharias, N.[1]

[1]Department of History, Archaeology and Cultural Resources Management, University of the Peloponnese, 24133, Kalamata, Greece
[2]Institute of Nuclear and Particle Physics, N.C.S.R. "Demokritos", 15341, Agia Paraskevi, Athens, Greece
[3]School of Archaeology, University College Dublin, Ireland
[4]Ephorate of Antiquities of Arcadia, Hellenic Ministry of Culture and Sports, Kalavryton 211, 22100, Tripolis, Greece

Keywords: Tegea; Bronze; Micro-XRF; Micro-PIXE; Optical microscopy

Bronze objects during antiquity have widely fulfilled peoples' daily but also votive needs. Bronzes deposition in early Greek sanctuaries, the most renowned being the cult at Olympia, has been a long-standing tradition originating to the Bronze Age. From the 9th century onwards, metals constitute the majority of inorganic votive offerings preserved at the sanctuaries of Tegea, whereas bronze is the dominant alloy of the Geometric and Orientalizing period (9th-7th c. BCE). An assemblage of 182 copper-based votive objects from the sanctuaries of Athena Alea and of Demeter and Kore at Tegea, Arcadia, dating to the 9th-7th centuries BCE, was examined by means of optical microscopy and elemental characterization techniques. The study aimed to investigate the technology used in the metalworking practices of the sanctuaries. The integrated micro-invasive and invasive analytical protocols allowed for the technological characterization of the copper-based votives at Tegea, in order to address questions about the characteristics of the workshop(s) involved in their production. The compositional elemental analyses of the samples were carried out using a portable micro X-ray Fluorescence (micro-XRF) spectrometer at the X-ray Fluorescence laboratory of the Institute of Nuclear and Particle Physics at the NCSR "Demokritos", in Athens. A selection of samples ensuring statistical independence was examined with the external scanning micro Particle Induced X-ray Emission (micro-PIXE) technique at the facilities of the Accélérateur Grand Louvre d' analyse élémentaire (AGLAE), C2RMF, Paris. The combined information obtained through the comparative evaluation of the two data sets, allowed to deduce compositional data of improved reliability and accuracy.

The present archaeometallurgical study proves useful, given the relative lack of comparative data, concerning the continuity of the use of tin bronze at the sanctuaries of Tegea during the Geometric and Orientalizing period. Results confirmed that the assemblage consists of Cu-Sn binary alloys, with an average tin content of approximately 8 wt%. The relative concentration of the two main elements of the alloy was not correlated to object typology. The results also suggest that fresh rather than scrap metal was used, along with indications for a local metallurgical technology, linked with the use of the sites as sanctuaries. The present study offers a glimpse on the role of the sanctuaries in the local community and the agents involved within a complex social, political and economic landscape.

Acknowledgements: The project was partially funded by the project CALIBRA/EYIE (MIS 5002799), which is implemented under the Action "Reinforcement of the Research and Innovation Infrastructures," funded by the Operational Programme "Competitiveness, Entrepreneurship and Innovation" (NSRF 2014–2020) and co-financed by Greece and the European Union (European Regional Development Fund).

S6-P24.246

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometallurgical study of copper-base production in the Iron Age settlement of Sierra Boyera (Belmez, Córdoba, Spain)

González-Zambrano, P., Pérez-L'Huilliere, D., Cristo-Ropero, A., Adroher Auroux, A. M., Murillo-Barroso, M.

University of Granada

Keywords: Metallurgical production, Protohistory, Mining, Archaeometry, Iberian Culture.

The Iron Age settlement of Sierra Boyera is located in the north of the Andalusian province of Córdoba, in a transition region between the Guadalquivir and the Guadiana river basins, which are connected by the corridor formed by their tributaries, the Guadiato and the Zujar respectively. In the 2021 excavations, a productive area of copper-based and iron metallurgy with the whole chaine operatoire was documented, including ore fragments, nozzles, a smelting pit, smelting furnaces, an anvil, slag fragments and crucibles, as well as manufactured objects, some of which might have been destined for remelting.

This site, dated between the 4th and 2nd centuries B.C., allows us to tackle an archaeological problem such as the evolution of the metallurgical production and mining in the Sierra Morena region, and sheds more light on the production processes of Iberian societies. This region has a long mining tradition, thanks to the richness of its polymetallic seams. While it has been possible to trace it back to the Chalcolithic, there has been hardly any research regarding its Iron Age societies. This study has made it possible to reconstruct the entire production process of copper-based metallurgy from the mine to the final product.

A set of several slags samples, 3 crucible fragments and 8 bronze objects have been analysed. Slag samples and slagged ceramic sherds, were analysed by SEM-EDX while elemental composition of objects was determined by pFRX.

With all this, we have shed new light on the metallurgical production of a singular site from the Iron Age in Sierra Morena Central, aided by archaeometrical studies of the elements preserved in this metallurgical workshop.

S6-P25.1558

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Untangling golden threads: Interpreting the technology of an ancient woven gold textile using scanning macro XRF spectroscopy

Ganio, M.[1], MacLennan, D.[1], Lansing Maish, S.[2], Hart, M. L.[2], Trentelman, K.[1]

[1]Getty Conservation Institute, Los Angeles, CA
[2]The J. Paul Getty Museum, Los Angeles, CA

Keywords: Gold; MA-XRF; textile weaving; ancient Roman

Gold has been used to embellish luxury textiles since the Bronze Age. Gold threads were embroidered, interwoven with other fibres in tapestry, and even woven alone to create stand-alone ornamentations. Gold is a highly ductile and malleable metal, and fine threads could be produced by twisting gold wire or strips of gold foil around a vegetable- or animal-derived fibrous core. For example, in the 1st century AD, Pliny the Elder (NH 33.19.62-63) describes a gold textile that appears to be spun and woven like wool *sine alia materia*.
A group of fifty-six small woven gold fragments (#79.AO.75.125) from the J. Paul Getty Museum collection comprises one of the rare extant ancient woven gold textiles. Acquired in a single tangled ball, the original shape, dimensions, and even the use of this textile are unknown.
A historical and scientific study was carried out in order to better understand this unique artifact. Scanning macro X-ray fluorescence (MA-XRF) spectroscopy was performed on a subset of 21 fragments to characterize and map the chemical elements of the textile. Unlike point analyses, XRF mapping can significantly increase the understanding of the materials used, since it allows the distribution of different chemical elements across an entire object to be visualized. Gold is the main element detected in all of the scanned fragments, but perhaps most surprisingly, the results also reveal an enrichment of copper and silver in distinct ‘bands’ that run parallel to the warp direction of the objects. The copper and silver-rich ‘bands’ observed in some of the fragments may provide important clues as to how the fragments relate to each other and how many different objects may be represented by the collection. Additional complementary analyses, including SEM-EDS mapping and digital microscopy, were carried out to investigate the manufacturing technology and to describe the weaving technique.
The results of the scientific investigation have provided important evidence supporting our reassembly of the disparate fragments into an approximation of their original shape, allowing a reappraisal of the golden ribbon’s original context and function.

S6-P26.260

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The study of chrysocolla and its thermal decomposition to understand extraction of copper in pre-Hispanic metallurgy

Manya, S.[1,2], Mille, B.[2,3], Figueroa Larre, V.[4], Bataille, T.[1], Lacaillerie, J-B.[5], Le Polles, L.[1]

[1]Institut des Sciences Chimiques de Rennes (UMR CNRS 6226), École nationale supérieure de chimie de Rennes, France
[2]Centre de Recherche et de Restauration des Musées de France, Paris, France
[3]Technologie et Ethnologie des Mondes Préhistoriques (UMR 8068), Nanterre, France
[4]Instituto de Arqueología y Antropología, Universidad Católica del Norte, San Pedro de Atacama, Chile
[5]Sciences et Ingénierie de la Matière Molle (UMR CNRS 7615), École supérieure de physique et de chimie industrielles de la ville de Paris, France

Keywords: chrysocolla; characterization; thermal analysis; pre-Hispanic copper

A French-Chilean team from the IRP CNRS Atacama-SHS (Fondecyt 1201603) involving archaeologists, anthropologists, archaeometallurgists and geologists, discovered in Ujina-Collahuasi (Tarapacá, Chile) a camp of miners and more than fifty smelting furnaces, constituting what appears to be the largest known pre-Hispanic copper production site for northern Chile.
These discoveries have made it possible to collect many witnesses of the functioning of the furnaces: fragments of ore, tools, slags, metallic copper. Among the copper ores used, chrysocolla, a copper hydroxysilicate, is always present in substantial proportion.
A preliminary study of the slags showed that chrysocolla introduction created a significant excess of silica, but also that the pre-Hispanic metallurgists achieved a surprisingly good copper recovery despite this strongly unbalanced chemical composition.
The principal goals of the INKACOPPER project is firstly to characterize chrysocolla and its thermal decomposition, and secondly to get a better understanding of the pre-Hispanic copper smelting process used in the Collahuasi furnaces.
In this communication, we will focus on the first objective by carrying out a detailed study of what is chrysocolla and the examination of its thermal decomposition. The chrysocolla mineral is poorly crystallized or even amorphous, and its organization at the atomic scale remains largely controversial.
This structural complexity of chrysocolla led us to use a combination of structural study techniques (MEB-EDX, TG-DTA, optical and resonance spectroscopies, XRD) to try to have a better understanding of its organization at the atomic scale.
At the same time, the study of the thermal transformation of chrysocolla into copper was initiated; in particular with the study of the chrysocolla's behavior during heating. The decomposition proceeds in a few stages reaching to intermediate copper oxides, along with additional unidentified crystallized phases are formed over 600 °C.

S6-P27.1557

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

First analyses of the chemical composition of buckles from Bulgaria dated to the great migration period

Zlateva, B.[1], Lesigyarski, D.[2], Traikova, L.[3], Vagalinski, L.[3]

[1]Faculty of Chemistry and Pharmacy, Sofia University "St. Kl. Ohridski", 1, James Bauchier Blvd., 1164 Sofia, Bulgaria
[2]National Institute of Geophysics, Geodesy and Geography – BAS, 3, Acad. G. Bonchev Str., 1113 Sofia, Bulgaria
[3]National Archaeological Institute with Museum – BAS, 2, Saborna Str., 1000 Sofia, Bulgaria

Keywords: bronze; brass; XRF; ICP-AES; Great Migration Period

The belt accessories style has accommodated to fashion of their era and thus is very important chronological indicator. It carries potential information about social status, gender and ethnicity of the owner as well as about commercial contacts and production centers. Studies of the development of belt accessories from the Great Migration Period in Bulgaria lag significantly with respect to the large amount of finds of this kind.

In the present study more than 250 copper alloy buckles collected from archaeological museums in several regions of Bulgaria are analyzed using XRF, ICP-AES and ICP-MS. Additionally, to a small part of the investigated samples metallographic analysis is performed in order to obtain more information about the technology of production used. The finds are dated to the 3rd-7th centuries CE. Concentrations of As, Bi, Co, Cd, Cu, Fe, Mn, Ni, P, Pb, Sb, Se, Sn, Zn are determined by an external calibration strategy using well characterized matrix-matched standards and standard reference materials with similar chemical and physical properties. Determination of the selected elements can provide information about the technological issues of production of the ancient bronze and brass buckles. Statistical treatment of the analytical data using the Statistica 10 software is also performed.
The investigated belt accessories found in West Bulgaria are made predominantly of brass with various concentrations of Zn, however, additions of tin and lead in moderate concentrations are common, which suggests that part of the items are produced by adoption of older bronze or brass artifacts. Within the specimens from Central and East Bulgaria artifacts made of Sn-bronze and brass are almost equal in number, however, small group of buckles form Central South Bulgaria (the Stara Zagora Region) are made of nearly pure copper. Statistical treatment of the data obtained show separation of the analyzed buckles in several groups according to the elemental composition that match to a great extent the typological and geographical characterization of the artifacts.

Acknowledgements:
This investigation is financially supported by the Bulgarian National Science Fund under the Contract No. H 10-15/2016

S6-P29.318

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Geochemical study of raw materials of metallurgical crucibles of Mari I: key information for the study of pottery in the Syrian Euphrates Environment

Sanjurjo-Sánchez, J.[1], Montero Fenollós, J.L.[2]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]Departamento de Humanidades, University of A Coruña, Campus de Esteiro, 15403 Ferrol, Spain

Keywords: geochemical study; crucibles; Mari, pottery; Syrian Euphrates

The city I of Mari (ca. 2900-2650 B.C.), in the Middle Syrian Euphrates, was an important center of copper and bronze metallurgical activity. Numerous fragments of clay crucibles used in the smelting process have been found in its workshops. In this work, the geochemical composition of a large sample of these crucibles has been studied. The study consisted in the analysis of the mineral composition by means of X-Ray Diffraction (XRD) and the analysis of the elemental composition in majority and minority elements, and some trace elements, by means of X-Ray Fluorescence (XRF).

The objectives of this study are as follows:

1. To estimate the firing temperature reached in the crucibles during their use in metallurgical production from their mineral composition. This could show if the temperature control technique of the smelting was very precise or not.
2. Check if there are geochemical similarities and/or compositional differences between the different samples studied. The existence of a similar pattern should indicate a local production of crucibles, using nearby raw materials. This could indicate if raw materials or manufactured metals were imported being the crucibles used for the local metallurgical manufacture or recycling materials.
3. Contrast the results with other ancient pottery from different periods in the Middle Syrian Euphrates. Specifically, it is of great interest to compare the result obtained with the analysis of the bevelled rim bowls (BRB) of the Middle and Late Uruk periods (ca. 3500-3100 B.C.). BRB samples are available from various sites in the area; the aim is to establish the local or non-local character of this pottery.

S6-P30.337

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The Iron Age bronze workshop of Cabeço Redondo (Portugal): preliminary evaluation of copper sources using lead isotopes

Valério, P.[1], Soares, R.M.[2], Soares, A.M.M.[1], Gomes, S.S.[1], Araújo, M.F.[1]

[1]C2TN, Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa.
[2]UNIARQ, Universidade de Lisboa; Câmara Municipal de Silves.

Keywords: Copper; Bronze; Ingots; Provenance; Southwestern Iberian Peninsula

Cabeço Redondo (Moura, southern Portugal) is a singular and monumental building of the 5th century BC, located on the left bank of the Guadiana River, whose stone and adobe structures were severely damaged in 1990 during agricultural works. However, among the preserved non-structural remains recorded by archaeological excavations, we highlight those referring to a metallurgical workshop of copper/bronze production, whose facilities in the ground have not yet been found. The evidence of its existence is quite strong since several pieces of copper and bronze ingots, including a large 6.4 kg fragment of a plano-convex copper ingot, numerous lumps and prills, tuyeres, slags, as well as several artefacts, showing one of them signs of a faulty casting, have been recorded during archaeological surveys. Previous chemical and microstructural study provided some answers about local foundry activities and identified the composition and manufacture of copper, bronze and leaded bronze artefacts. The present work concerns the determination of Pb isotopic ratios of copper and bronze ingots and lumps at Cabeço Redondo, and their comparison with Pb isotopic fields of Peninsular and Mediterranean copper sources. The exercise suggests that this workshop dealt with raw materials from multiple sources located in the Iberian Pyrite Belt and/or in distant Sardinia (Central Mediterranean). However, considering the absence in the Iberian Peninsula of coeval metallurgical structures compatible with the production of such a large plano-convex ingot (slag-tapping furnaces), the copper trade with Sardinia seems a highly feasible hypothesis. Although the precise location of the workshop in the site is still unknown, all this new evidence makes Cabeço Redondo one of the most important and most deeply studied metallurgical complexes of this kind in the Iberian Peninsula.

Acknowledgements
The support of Fundação para a Ciência e a Tecnologia (FCT) to C2TN (UIDB/04349/2020) is gratefully acknowledged. Rui M. Soares acknowledges the FCT grant SFRH/BD/111511/2015. The authors thank for technical and human support provided on LIA by Servicio General de Geocronologia y Geoquímica Isotópica de la Universidad del País Vasco (SGIker of UPV/EHU/ERDF, EU).

S6-P32.344

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chemical composition of metal artifacts found in ancient Emporion Pistiros and Sboryanovo (ancient Helis ?), Bulgaria

Bonev, V.[1], Zlateva, B.[2]

[1]Faculty of History, University of Sofia, 15 "Tzar Osvoboditel" str, 1504 Sofia, Bulgaria;
[2]Faculty of Chemistry and Pharmacy, University of Sofia, Bul. James Bouchier 1, 1164, Sofia, Bulgaria.

Keywords: Archaeometry; metal artifacts; Thrace

This study presents the results of the chemical analysis of large number of metal artifacts (203 in number), chronologically dated between second half of the 5th century BC – beginning of the 2nd century BC. 127 of them were found in Emporion Pistiros archaeological site. It is supposed that Pistiros was an ancient Greek emporion with particular importance as trade center. 76 artifacts originating from ancient Helis (?), the supposed capital of the Getic dynasty from the Early Hellenistic time. The city was well fortified and was an economic centre of import, redistribution of goods and local production. Evidence for metal processing and jewelry production workshops is found in both settlements.

Significant part of the samples are taken from fibulae, but data collected from other jewelry, elements of weaponry, precious metal ingots etc. is also included in the research. The fibula as an element of the costume combines two functions – utilitarian one (serving to fasten the garments) as well as aesthetic and decorative one. This circumstance has predetermined its widespread use in time and space. Among the pieces known to us, the ones made of bronze prevail although there are examples of some other metals employed as well – gold, silver and iron. The attempt to compare the obtained data from these settlements is likely to answer questions that have been of concern to the researchers for long time.

The study on the chemical composition of the metal artifacts will provide important information about the technology of ore processing and production of the findings. In the initial stage of this study the concentrations of fifteen elements was determinate (Al, As, Bi, Co, Cr, Cu, Fe, Mn, Ni, P, Pb, Sb, Se, Sn, and Zn). The analytical techniques used for analysis were ICP-AES (inductively-coupled plasma atomic-emission spectroscopy, Perkin-Elmer Optima 7000 DV) and p-XRF (Bruker S1 Titan). An external calibration strategy with well characterized matrix-matched standards and different standard reference materials with similar chemical and physical properties (i.e. BAM 211) were chosen for the analysis. Multivariate statistical interpretation of the analytical data (statistical program SPSS 16.0 for Windows) reveals the types of alloy and their connection to topological characterization of the metal objects.

S6-P33.371

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The work of gold and silver under the Wari in the Cusco region

Guerra, M.F.[1], Sierra Palomino, J.[2]

[1]MONARIS, UMR 8233 CNRS & Sorbonne Université, Paris, France
[2]Laboratorio Fisicoquímico, Dirección Desconcentrada de Cultura de Cusco, Peru

Keywords: Wari; Cusco; precious metals; alloys; technologies

The Wari state controlled during the Middle Horizon (600-1000 AD) a vast region around the Ayacucho administrative centre, which comprised monumental settlements such as Pikillacta, situated southeast of Cusco. Despite numerous archaeological excavations leaded in the Cusco region, few silver and gold items were found in Wari sites. Pikillacta only delivered, in 2004 and 2019, two ceremonial offerings containing small silver figurines representing nobles, felines and warriors [1]. A few other objects were found in sites such as Marcaconga and Batan Urqu [2]. However, the discovery in 2011 at Espíritu Pampa of an important ritual site [3] changed the vision of the Wari state presence in Cusco, as one of the tombs delivered remarkable gold and silver goods (gold bracelets, a silver mask, a silver pectoral, a sceptre and a staff covered with silver sheet, etc.).

The aim of this work is to provide more insight into the work of gold and silver in the region of Cusco under the Wari. Using optical microscopy and a portable XRF equipment, we carried out at the *Dirección Desconcentrada de Cultura* (Ministry of Culture) in Cusco the *in-situ* study of the objects from the Espiritu Pampa main tomb, from Marcaconga, and from the ceremonial offer found in 2004 at Pikillacta. Data obtained showed the skilled metallurgical practices of the Wari. Some objects are hollow and others made in plain gold and plain silver. A few of them are made from silver plated copper.

Data recently published for gold and silver objects made in the Andean area by the Incas revealed the use of standardised metallurgical practices and particular intentional alloys [4]. The gold and silver alloys made by the Wari are of quite good quality, but their compositions are different from those employed by the Incas. In addition, the Wari productions do not seem to follow standardised criteria.

[1] Tuni C., Tesar L. 2011. The Pikillacta 2004 Eastern Gate offering pit, Ñawpa Pacha 31(1), 1-44.
[2] Zapata J. 2019. Unidades territoriales, residencias y tumbas de la elite wari en la región del Cusco, Research Papers of the Anthropological Institute 8, 177-229.
[3] Fonseca J. 2011. El rostro oculto de Espíritu Pampa, Arqueología Iberoamericana 10, 3-5.
[4] Guerra M.F., Fischer M., Radtke M., Reinholz U. 2017. Inca figurines from the Ethnologisches Museum in Berlin, Bull. de l'Institut Français d'Etudes Andines 46(1), 221-252.

S6-P34.376

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Egyptian jewellery in gold: puzzling practices in early periods

Guerra, M.F.

MONARIS, UMR 8233 CNRS & Sorbonne Université, Paris, France

Keywords: Egypt; gold alloys; colour; casting; soldering

The analytical study of gold jewellery produced in Egypt [1], dated to the earliest periods until the New Kingdom, allowed approaching questions related to their production [2]. The aim of this work is to provide an overview of the most significant results, in particular those related to workshop practices in early periods. The results obtained seem to indicate controlled “large-scale” productions with a workshop organisation that includes artisans with different range of skills. The low quantity of gold necessary to make part of the items contrasts, in fact, with the quantity of work that has to be achieved by the artisans.

The observation using optical and scanning electron microscopes of the size, shape and use-wear marks of the simplest and most common gold beads made since the Predynastic period, revealed their wide variety, allowed separation by type, and suggested reuse without melting. The XRF, EDS and PIXE analysis of these beads revealed the heterogeneity of the alloys employed within a same string. This contrasts with the homogeneity by type of the elaborated gold beads, necessarily made by skilled goldsmiths for particular objects.

In addition to exploitation of gold mechanical properties, the regular use of casting to shape the objects and of hard-soldering to assemble their different parts could be demonstrated. The other properties of the gold alloys that were exploited are the optical ones. Since the earliest periods, jewellery items of different colour and reflectivity were produced. The analytical data obtained suggested the use of colouring techniques based on addition of copper, silver and silver alloys to gold. The recurrent presence of PGE inclusions at the surface of the objects suggested exploitation of placer gold deposits and extensive use of recycling practices.

[1] In the collection of Petrie Museum, British Museum, National Museums Scotland, Manchester Museum, Garstang Museum, Louvre Museum.

[2] The totality of the data will be published in 2020: *Ancient Egyptian gold: archaeology and science*, M.F. Guerra, M. Martinón-Torres, S. Quirke (eds.)

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chemical and Pb isotopic study of Roman lead artefacts recovered in the Arade River, Portimão (Portugal)

Gomes, S.S.[1], Araújo, M.F.[1], Valério, P.[1], Monge Soares, A.M.[1], Fabião C.[2], Freitas, V.[3]

[1]Centro de Ciências e Tecnologias Nucleares (C²TN), Departamento de Engenharia e Ciências Nucleares, Instituto Superior Técnico, Universidade de Lisboa
[2]Departamento de História, Faculdade de Letras, Universidade de Lisboa
[3]Museu de Portimão /CMP

Keywords: Elemental composition; Pb isotope ratios; Late Roman Empire; Mediterranean trade routes; lead amphora labels

Arade River, the main water line located in southern Portugal (Algarve), played an important role in ancient cultural and commercial exchanges of Mediterranean and Atlantic World with local communities. During the Late Roman Empire, the city of Portimão (Lusitania province), located at the mouth of the Arade River, was an important harbour where several fish-processing factories were installed, displaying an important commercial activity, evidenced by numerous archaeological underwater finds uncovered during modern dredging programs. These include amphora fragments from Africa Proconsularis, widely used in fish products transport, and miscellaneous roman artefacts with special emphasis on lead plaques, rectangular in shape and variable sizes (33 – 129 mm length, 32 – 66 mm width, and a few millimetres of thickness). Some of them exhibit decorative motifs in relief similar to those of amphora labels reported in the literature [1]. In the present research 37 lead samples was analysed, composed by rectangular plaques with decoration in relief, such as tridents, fishes or palms, usually displayed in amphora labels, small plaques with perforation and inscriptions, strips and shapeless fragments without any decoration. Elemental analysis was performed by ICP-MS to determine nickel, copper, arsenic, silver, tin, antimony and bismuth contents. Results were later interpreted using multivariate statistical analysis, which assembled elements with similar behaviour/composition suggesting different raw materials, namely lead obtained by the reduction of litharge or nonargentiferous galena. Cluster analysis displayed a distinctive group of plaques showing decoration. Therefore, these decorated plaques were further analysed by MC-ICP-MS to determine Pb isotope ratios in order to assess ore sources trying to infer trade routes. The distribution of Pb isotope ratios points to the use of different lead sources, ones from Iberia Peninsula and amphorae lead *etiquettes* from Africa, despite not bearing any epigraphic inscription as usual in other lead *etiquettes* known before.

[1] Quevedo, A.; Fernández-Diaz, A.F. 2020. Lead labelling on Roman Amphoras. A Short-lived Fashion? The International Journal of Nautical Archaeology, 1-11.

C²TN/IST authors gratefully acknowledge the FCT support through the UIDB/04349/2020 project and RNEM – Portuguese Mass Spectrometry Network. Authors are also grateful to the technical and human support provided by SGIker (UPV/EHU/ERDF, EU).

S6-P36.1542

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

About the colour of copper alloys in the systems Cu-Sn, Cu-Zn, Cu-Sn-Pb, Cu-Zn-Pb, Cu-Sn-Zn, Cu-Sn-Zn-Pb

Mecking, O.

Thuringian State Office for Heritage Management and Archaeology, Germany

Keywords: color of copper alloys; CIELAB

The colour of decorative objects is decisive for the appearance of the object. Archaeological copper alloys have become greenish as a result of the floor storage and thus give a different colour impression than the objects originally had. In order to reconstruct the original colour of the object, 424 copper alloys were produced. These samples cover the following alloys: Cu-Sn, Cu-Zn, Cu-Sn-Pb, Cu-Zn-Pb, Cu-Sn-Zn and Cu-Sn-Zn-Pb. The CIELAB system was used to determine the colour of these samples. The chemical composition of the archaeological samples can be used to deduce the colour of the object from the alloys produced. The colours show a very large variability from reddish copper tones to gold and silver tones. These systems will be presented. Copper-tin alloys develop into silver tones with increasing tin contents. Brasses show golden hues with increasing zinc content. If lead is added to brass or copper-tin alloys, the colours change only slightly. Copper-tin-zinc alloys show the greatest colour differences. Lead also has only a minor influence on the colour of these alloys.
The results will be applied to two archaeological examples. Hallstatt and Late Period fibulae partly consist of several parts in Central Europe. Lead as well as the tin content in the individual parts can be different. The examples show that, despite the different composition, an attempt was made to have a similar colour for the individual parts. Copper-tin-zinc alloys of the Roman Empire in Central Europe show very different compositions. A large group lies exactly in the range of gold tones. Therefore, it can be assumed that this colour should be achieved with the objects.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The determination of Platinum group elements in archaeological bronzes - New ways to determine the provenance of copper alloys

Mecking, O.

Thuringian State Office for Heritage Management and Archaeology, Germany

Keywords: Platinum group elements; provenance; copper alloy; Kupferschiefer

Until now, the origin of archaeological copper objects has been determined by trace element patterns and isotope ratios. For the application of trace element patterns these elements have to follow the copper during smelting. Simplified the elements follow the copper which are more noble or have a similar behavior to the copper. Until now, mainly Ni, Ag, Sb, As, Sn, Au, Zn, Pb, Co and Bi have been used for copper provenancing, most of which occur in µg/g content in copper objects. Platinum group elements (PGE) are more noble than copper and therefore follow copper during smelting. This is exploited today by accumulating PGEs in copper and recovering them. Since PGEs have significantly lower contents than the other trace elements, a very powerful method must be used. ICP-MS devices are ideal for this. Interferences can form in the plasma of these devices. For example, the copper can react with argon to form CuAr, which is then measured on mass 105 and 103. However, rhodium is also measured on mass 103 and palladium on mass 105. In order to eliminate these interferences safely, a reaction gas must be used, which reacts either only with the analyte or with the interference. In order to prevent other masses from reacting to the target mass, these masses must be separated before. This was achieved using an ICP-MS/MS with a mass spectrometer in front of the reaction cell. By using different gases, it can be shown that all possible interferences can be safely eliminated. With this method, detection limits of less than 1 ng/g for the solid at 20 mg sample weight can be achieved. This method with the first applications to archaeological objects shall be presented.
In a first test series, approximately 70 samples from different times were examined for the PGEs and the classical trace elements. In the early Bronze Age in Central Europe, the contents of PGEs are low and do not provide a meaningful extension for the determination of origin. In Central Germany the Pt and Pd values increase during the Hallstatt and Latene periods and thus give further indications of the deposits. In the Middle Ages there are very large differences between the PGEs. Kupferschiefer was mined near Eisenach. These contain PGE. In some finds from the Wartburg in Eisennach, unusual PGE contents were found. These values may indicate the use of the Kupferschiefer. The porphry copper deposits (Bor, Majdanpek and others) also contain PGE and PGEs can be an important marker for these deposits.

S6-P38.390

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Lead Bricks for the Qin First Emperor's Acrobats: entertaining function and metallurgical features

Li, X.[1,2,3], Rehren, T.[2,4], YinXia[1], Zhang, W.[1], Liu, S.[5], Fu, J.[1], Shao, W.[1]

[1]*Emperor Qin Shihuang's Mausoleum Site Museum, Xi'an, China*
[2]*UCL Institute of Archaeology, London, United Kingdom*
[3]*School of Archaeology, University of Oxford, United Kingdom*
[4]*Science and Technology in Archaeology and Culture Research Center, The Cyprus Institute, Nicosia, Cyprus*
[5]*Institute of Historical Metallurgy and Materials, University of Science and Technology Beijing, China*

Keywords: Qin Empire, bronze, lead, SEM, ICP-MS

Lead, as one of the important metals of bronze alloys, was widely used for producing ritual vessels and weapons in Bronze Age China. The Qin Terracotta Army was equipped with a large quantity of lethal bronze weapons to be buried with the Qin First Emperor (259-210 BC) to protect him in his afterlife. However, among the most spectacular finds at his mausoleum complex, the terracotta acrobats were very unique to be buried for the emperor's entertainment. Comparing to the large quantity of bronze weapons with which the terracotta warriors were equipped, metals found with these acrobats are very different. Particularly outstanding is a pile of lead bricks, some of which had molten and the liquid lead run into the originally paved wooden floor due to the fire in the pit. In addition to study the function of the heavy lead bricks for performance of the Qin acrobats, this study also aims to investigate the lead bricks regarding: a) metallurgical features; b) compositions of metal and patina; c) trace elements and lead isotope ratios, in order to understand the raw materials, burial environments, and corrosion in their archaeological and social context. For the broader view, it also aims to address the logistics for the construction of the Qin Emperor's mausoleum complex, and metal production in ancient China.

S6-P40.400

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Technological interpretation of the manufacturing process of the Vittoria Alata di Brescia through application of Neutron Diffraction

Cantini, F.[1], Patera, A.[1], Morandini, F.[2], Brini, A.[1], Galeotti, M.[1], Cagnini, A.[1], Porcinai S.[1], Scherillo, A.[3], Grazzi, F.[4,5]

[1]Opificio delle Pietre Dure, Firenze (Italy)
[2]Fondazione Brescia Musei, Brescia (Italy)
[3]Science and technology Facility Council, ISIS Neutron and Muon Source, OX110QX (UK)
[4]Consiglio Nazionale delle Ricerche, Istituto di Fisica Applicata Nello Carrara, Sesto Fiorentino (Italy)
[5]Istituto Nazionale di Fisica Nucleare, Laboratorio di Tecniche Nucleari per i Beni Culturali, Sesto Fiorentino (Italy)

Keywords: Roman statuary; Bronze analysis; Technological analysis; Neutron diffraction

The Vittoria Alata di Brescia is a large size Roman bronze statue that, according to recent studies, was made during the 1st century A.D., and discovered underground in the Capitolium of Brescia in 1826. It represents a winged female figure with the arms protruded forward writing the name of the winner on a shield (missing).
An extensive restoration has been performed by Opificio delle Pietre Dure (OPD), settore Bronzi, between 2018 and 2019 to remove the filling material added in 19th century.
The statue was found disassembled in five pieces: the body, the two arms and the two wings. There are possibilities that the wings (and maybe the arms) were added (or modified) after the first modelling. During restoration activity, several technological questions were raised and discussed: is the alloy the same in all the components; how much lead was used; is the lead observed on the surface originating from the statue or from the environment; was the body made in a single or multiple cast; if multiple cast in which way was it assembled?
The Scientific Laboratory of OPD previous studies were aimed to analyse the core materials, the patina and, where possible, the bronze composition with different tecniques: X-ray radiography, microscope observations, SEM, XRF, XRD, Eddy current and FTIR. At the same time, a technological study about casting procedure (direct or indirect casting), was performed by detailed investigations, using portable microscope and endoscope, of the inner and outer surface.
In order to obtain high accuracy quantitative phase analysis, details about dendritic compositional gradients, and casting related microstructural features (columnar growth, crystalline defects density and crystallographic domain size), we performed time of flight neutron diffraction analysis at the INES beamline of the ISIS spallation neutron source, measuring one broken end of the left wing, a fragment from the inner surface of the torso, and several sub-mm scale samples from the different pieces and areas of the body, arms and wing.
The combination of neutron diffraction analysis together with the laboratory methods allowed us to identify clearly that the body was cast in at least three pieces, and the alloy and the dendritic microstructure of the examined areas were similar in some parts and significantly different in others as well as the lead content.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Collective action:
The making and breaking of gold objects in bronze age crete
.....

Martinón-Torres, M.[1], Legarra Herrero, B.[2], Benzonelli, A.[2]

[1]Department of Archaeology, University of Cambridge, Downing Street, Cambridge CB2 3ER, United Kingdom
[2]UCL Institute of Archaeology, 31-34 Gordon Square, London WC1H 0PY, United Kingdom

Keywords: archaeometallurgy; gold; Mediterranean; Bronze Age; heterarchy

Studies of prehistoric goldwork in Crete have predominantly discussed typology and style, often focusing on unusual objects and seeking parallels in Egypt or the Near East to discuss diffusion of iconography or style. Conversely, studies that concentrate on a contextual understanding of local traditions have been limited, particularly those taking advantage of analytical techniques.

Here we present a high-resolution analytical study of some 100 gold objects recovered from Early and Middle Bronze Age contexts at the sites of Mochlos, Hagios Charalambos and Sissi, focusing on artefact biographies and seeking to understand island-specific dynamics in the provision, manufacture, use and depositon of goldwork. Our results challenge traditional narratives and support the claim that gold was not a medium used to reinforce hierarchies between individuals. Instead, we show gold as a collective resource, managed by the community, manufactured by many, and employed to reinforce horizontal rather than vertical social structures, or as a medium for competition between rather than within communities.

Gold was predominantly used for relatively simple shapes obtained through hammering and mechanical decoration. However, it is possible to document the hands of multiple artisans, with variable technical repertoires and skillsets. This argues strongly against the existence of specialists attached to an elite. Extensive marks of wear and modification are indicative of extended periods of utilisation, most likely in rituals and by multiple users, and again questioning the assumption of a single and powerful owner. Finally, we reveal that virtually all objects were deliberately fragmented before their parts were scattered in different collective tombs: cut, broken, ripped or otherwise taken apart, fragmented gold objects were used for enchainments between the living and the dead, but also to connect communities of people. The technology and value of gold, however, begin to change around 2000 BC.

Overall, our study demonstrates the potential of science-informed studies of goldwork to inform debates about craft organisation, power and social hierachies, while also introducing a new material to broader discussion of fragmentation practices in the prehistoric Aegean.

S6-P49.520

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Chemical, mineralogical, and morphological properties of ancient iron slag founded in Katund Plak, in eastern Albania

Duka, E.[1] Çakaj, O[2], Civici, N.[3], Dilo, T.[4], G. Schmidt, G.[5]

[1]Faculty of Mathematical Engineering and Physical Engineering, Polytechnic University of Tirana, Albania
[2]Faculty of Natural Science, University of Tirana, Albania
[3]Centre of Applied Nuclear Physics, University of Tirana, p.o. Box 85, Tirana, Albania4
[4]Institut für Keramik, Glas- und Baustofftechnik, TU Bergakademie Freiberg, Germany

Keywords: iron slag; XRF; XRD; OM; SEM/EDS

Slags, which are the best material for studying ancient metallurgy, can help in the search for the origin of the artifacts of iron as well as production techniques. The size, shape, microstructure of slag, resistance to corrosion and chemical composition enable connection of slag and minerals, to detect specific treatments, minerals, and technologies. The aim of this paper is to study slags that belong to End of Bronze Age - Early Iron Age founded in an expedition in Katun Plak village, of Elbasan district in Eastern Albania. Archaeometallurgy methods used to complete this study are analytical techniques of X-ray fluorescence (XRF), X-ray diffraction (XRD), optical microscopy (MO) with reflecting and polarized light, stereomicroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray (EDAX), and other chemical and analytical methods. The densities of measured slag samples was between 3.5- 4 gr/cm^3. With a magnet we founded that samples are magnetic. Chemical elements content shows that those slags are iron slags with 35 % Fe, and have metallic iron grain inside of slags. The XRD analyses pointed out the presence of magnetite, fayalite, wustit and quarc. SEM micrographs showed that examined slags are fayalite-wustit type slags, and are considered products of iron metallurgy. Wustite such as nodular and shaped dentrite, and fayalite crystals are placed on a glassed matrix. These dentrites are fine and scattered.

S6-P52.559

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometrical investigation of „Thracian“ and „Chalkcidian“ helmets from Bulgaria

Zlateva, B.[1], Lesigyarski, D.[2], Totomanova, M.[3], Bonev, V.[4], Stoyanov, T.[4]

[1]Sofia University “St. Kl. Ohridski” Faculty of Chemistry and Pharmacy, 1, J. Bauchier blvd, 1164 Sofia, Bulgaria
[2]National Institute of Geophysics, Geodesy and Geography – BAS, 3 Acad. G. Bonchev Str., 1113 Sofia, Bulgaria
[3]Regional Museum of History Veliko Tarnovo, 6, N. Pikolo Str., 5000 Veliko Tarnovo, Bulgaria
[4]Sofia University “St. Kl. Ohridski”, Faculty of History, 2 Tzar Osvoboditel blvd, 1000 Sofia, Bulgaria

Keywords: Thracian; Chalkcidian helmets; XRF; ICP-AES; ICP-MS

The main objective of the project is to study and identify Thracian armaments (Chalkidian and Thracian helmets) through analytical methods and their classification on the basis of their chemical composition and archaeological contex.
The chemical characterization of the various types of materials used in the production and artistic decoration of artefacts from defensive armory is of significant importance in specifying the most appropriate methodology for their conservation and restoration.
The analysis of their chemical composition via different analytical methods brings various information regarding their origin, actual age, changes resulting from their aging, and the technologies used in the process of their elaboration.
In this study are presented the results obtained from the XRF, ICP-AES and ICP-MS analysis of 46 helmets collected from different historical museums in territory of modern Bulgaria and the data are compared with similar results. The results are important in the field of the Bulgarian archaeology, which would concern directly the archaeology of the Balkans, especially the development of metal work and particularly the helmet production as well as the cultural and historical relations between the Balkans, the Apennine and Iberian peninsulas.

Acknowledgments

This study is done as part of a bigger project N ORP 05/13/2018 funded by National Scientific Fund-Bulgaria

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Early medieval fine metalwork from the Carpathian Basin – changes in metal composition, manufacture and technology during the 5th century AD

Mozgai, V.[1], Horváth, E.[2], Bajnóczi, B.[1]

[1]Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Eötvös Loránd Research Network (ELKH), H-1112 Budapest, Budaörsi út 45, Hungary
[2]Department of Archaeometry and Archaeological Methodology, Institute of Archaeological Sciences, Eötvös Loránd University, H-1088 Budapest, Múzeum krt. 4/B, Hungary

Keywords: gilding, niello, garnet, silver alloy, gold alloy, non-destructive

In the Carpathian Basin the second half of the 5th century AD represents a transition period in the history of fine metalwork, separating the Hunnic Period rich in unique luxury objects and the Langobardic / Gepidic Period with rather mass products. More than two hundred gold and silver objects from the 5th century AD Carpathian Basin and their decorations (niello, gilding, garnet inlays) were analysed in detail by using non-destructive analytical methods (optical microscopy, hXRF, EPMA/SEM-EDX, μ-XRD, and μ-Raman). The comprehensive and detailed analyses have shown that no sudden changes occurred neither in metallic materials, nor in decorative techniques during the period. The quality of gold was unchanged and although silver has taken over the dominance, gold objects (> 90 wt% Au) still played a significant role. Different Ag/Au and Cu/Au ratios of the gold alloy indicate the use of gold from different sources. Silver-related technologies such as lost-wax casting, mercury-gilding, niello inlaying all have indirect local roots. Silver objects were manufactured from silver alloys (> 80 wt% Ag) intentionally alloyed with copper. Various amounts of gold and bismuth in the silver alloys possibly reflect the use of silver from different sources. The composition of silver objects changes towards the end of 5th century AD containing elevated amounts of tin, zinc and lead up to several wt%, indicating remelting and alloying with bronze and brass. In case of niello the continuation of the Late Roman practice using pure silver sulphide was supposed, however the quality deterioration is obvious, both in material and in technical sense. The objects were inlaid with various silver-copper sulphides ranging in composition from acanthite to chalcocite. The decrease in quality becomes even more emphasised by the end of the century, affecting even the fineness of the silver and the design of chip-carving. There is no sign of any standardisation of alloys, but recycling was detected with increasing frequency and complexity. Almandine of Northern-Indian origin appeared among the used garnet types, however only scarcely, barely indicating the major change in the 6th century. Long-used garnet sources of Sri Lanka and Southern India and their trade routes were preferred until the end of the 5th century.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Non-destructive analysis of Hunnic Period horse tacks from the Carpathian Basin

Piros, R. Á.[1], Mozgai, V.[2], Bajnóczi, B.[2]

[1]Department of Archaeology, University of Szeged, H-6722 Szeged, Egyetem utca 2, Hungary
[2]Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, H-1112 Budapest, Budaörsi út 45, Hungary

Keywords: horse tacks; Hunnic; handheld XRF; gilding

Different types of horse tacks from the Carpathian Basin were analysed non-destructively by using handheld X-ray fluorescence spectrometry (hXRF) in order to determine their chemical composition and the type of gilding. In addition, optical microscopy was applied to examine the used manufacturing and decorating techniques. The detailed analysis can help to prove the belonging of the objects to the same assemblages and to detect any later replacement/repair of a damaged or lost object. The analysed horse-tacks are dated to the late Hunnic Period (first half of the 5th century AD) representing the horizon of objects manufactured from pressed, gold plates (*'Pressblechtechnik'*). Based on the results, the objects are grouped into three groups: (i) gold objects, (ii) gilded silver objects and (iii) gilded copper-based alloy objects. The silver objects can be further classified based on the type of gilding. Presence of mercury indicates use of fire-gilding on some objects. However, on other objects no mercury was detected pointing to the use of leaf gilding proved by optical microscopy as well. All the copper-based alloy objects were decorated with fire-gilding. Replacements/repairs were also observed denoted by their distinct chemical composition and different quality of manufacture and decoration.

S6-P57.632

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Silver and copper isotopes reveal ancient gold refining: implications from experiments and gold artefacts

Berger, D., Brauns, M., Brügmann, G., Lockhoff, N.

Curt-Engelhorn-Zentrum Archäometrie Mannheim, Germany

Keywords: Stable isotopes; Gold refining; Parting; Cementation; Gold objects

The process of gold parting is closely linked with Sardis and the Lydian Kingdom of Croesus. From there, the first archaeological evidence for the metallurgical separation of silver from gold is known (6th c. BC) which allowed the production of almost pure gold. This is considered an important prerequisite for establishing monetary economies. The pyrometallurgical process of parting (also called cementation) involves the reaction of common salt and brick/fireclay powder with impure gold alloys at medium high temperatures. The simplicity of the procedure makes it likely that gold parting represents an ancient process already practiced in earlier times such as the Bronze Age. A considerable number of objects with low silver contents (0.5–3 %) are known since the 16th century BC. However, it is still an open question whether these artefacts are the product of a deliberate refining or the result of melting natural, silver-poor gold. We present a new approach into this archaeological problem by measuring the compositions of Ag and Cu isotopes in experimental material and ancient gold artefacts with MC-ICP-MS. Cementation experiments were carried out with gold alloys containing up to 53 % Ag and 3.7 % Cu using the common NaCl method either without or in combination with alum. The original metal was cemented in three consecutive steps. This progressively reduced Ag to 5.6 % (1. step), 2.0 % (2. step) and 0.6 % (3. step) using NaCl and to 2.9 %, 1.4 and 0.6 %, respectively, using NaCl and alum. Cu concentrations decreased from around 2 % to very low contents already after the second cementation step. The reduction is accompanied by a large fractionation of isotopes as $\varepsilon^{109}Ag/^{107}Ag$ increases from –0.28/0.95 to 100 ‰ and $\delta^{65}Cu/^{63}Cu$ from 0.17/0.28 to 11 ‰. This fractionation is much larger than that observed in primary and secondary gold deposits. The process of parting can thus be identified by measuring the bulk isotopic composition of Ag and Cu in gold. The analysis of Roman coins minted from cemented gold (0.1–0.6 % Ag) confirms this conclusion as they also have high isotopic values (e.g. $\varepsilon^{109}Ag/^{107}Ag$ = 8–25 ‰). In contrast, the studied Early Iron Age gold objects with moderate silver contents (11 % Ag) have $\varepsilon^{109}Ag/^{107}Ag$ of about 1.5 ‰, a value which is well within the natural isotopic range. Although we did not yet find indication of an early use of parting, it is demonstrated that parted gold can be isotopically distinguished from non-parted gold.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Late Roman Iron blooms of extraordinary size from Pannonia: a metallographic and experimental investigation

Török, B.[1], Barkóczy, P.[2]

[1]Institute of Metallurgy, University of Miskolc, Hungary
President of the UISPP Commission for Archaeometry
[2]Institute of Physical Metallurgy, Metalforming and Nanotechnology, University of Miskolc, Hungary

Keywords: iron bloom, microscopy, metallography, experiment, Roman

Five split iron blooms were unearthed at a former Pannonian Late Roman fortress in Keszthely-Fenékpuszta (Hungary). Because of their extraordinary parameters, ranged in size around 39-43cm X 25-38cm X 12-16cm and weighed 59-69kg each, these finds are one of the most interesting features of the archaeometallurgy of ancient ironmaking in Europe. The manufacturing process of them has been a technological mystery for decades.

A complex analysis was carried out on two of the blooms to find answers for some fundamental questions: Which are the characteristics of the microstructure and chemical composition? What could be revealed about the technological circumstances of the smelting process? What about the further usability of these blooms?

A slice, 10mm thickness was cut from one of the blooms by waterjet. Ten 4X4cm pieces, cut from the slice, were examined by optical microscopy and SEM-EDS. The C-content of the samples was detected by OES.

Based on the results it can be concluded that the bloom is a product of a single smelting process. There was no plastic deformation or jumping after the smelting. The bloom has a large number of gas pores and an extremely high C-content (in many parts higher than 2%). Surprisingly low number of slag inclusions can be observed, which originated from the smelting.

Based on the structure, the bloom was manufactured at about 1200 °C temperature for a long smelting time. It is conceivable that smaller slaggy pieces of blooms, broken off during the purification hammering after previous smelting processes, were charged back to the furnace and smelted again.

Three experimental forgings were prepared from the slice of the bloom, these pieces were heated up and hammered, in order to investigate the behaviour of the material during the metal-forming process. Some pieces were forgeable more or less, but other one broke into pieces.

A similar metallographic analysis of a small piece, cut from the corner of another bloom revealed that the materials of these blooms have similar properties. Some slag samples and two iron artefacts, found in the site, were also examined by SEM-EDS and OM.

S6-P65.690

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A Roman urban workshop for non-ferrous metal processing – preliminary data on gold and silver refining vessels

Cholakova, A., Penkova, P., Katsarova, V.

National Institute of Archaeology with Museum, Bulgarian Academy of Sciences

Keywords: Roman period; gold; silver; parting vessels; ED-XRF

Recent excavations in Serdica (present-day Sofia, Bulgaria) produced unexpected results, pointing to the existence of a workshop for non-ferrous metal working in the centre of the Roman town. In one of the rooms of a big public building, in a layer generally dated to the first half of the 3rd c. AD, numerous fragments of ceramic crucibles (with traces of gold, silver, copper alloy), copper alloy scrap pieces, charcoal were found. The most intriguing part of this debris assemblage consists of fragments of ceramic vessels belonging to well-known household coarse ware bowl types, with partly preserved additional external insulation layers of different type of ceramic fabrics, traces of burning, metal prills and slag-like residues, pale coloured internal surface, often with remains of a clay-like substance, with which some of these re-used domestic vessels were apparently filled.

The initial identification of the pieces as remains of vessels for gold and silver refining, based on macroscopic observations, was confirmed by preliminary non-destructive ED-XRF analyses which proved high-silver content of the metal prills and residue, and the presence of tiny gold particles trapped in the clay-based filling of the bowls.

Despite the fragmented state of the finds, in some cases, due to the numerous sherds recovered from the layer, it is possible to achieve an almost complete reconstruction of the reused domestic bowls and their insulation layers. Among the finds from Serdica, fragments indicative of gold parting are of special significance for the study of ancient technology, given the fact that such finds are very rare in the archaeological context. This poster presentation will provide an overview of the main macroscopic features of the vessels, as well as a summary of the preliminary ED-XRF data obtained so far.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Reassessment of the Evidence for Copper Metallurgy in Teuchitlán, Jalisco, Western Mexico

Maldonado, B.[1], Tropper, P.[2], Heredia, V.[1], Angerer, T.[2]

[1]Center for Archaeological Studies, El Colegio de Michoacán, A.C., La Piedad, Michoacán, México.
[2]Institute of Mineralogy and Petrography, University of Innsbruck, Austria

Keywords: Archaeometallurgy; ancient copper; Western Mexico

Metallurgy and metalworking in the form of sumptuary objects, represented one of the most characteristic socio-technological developments in Pre-Columbian Western Mexico [1]. Such precious goods were used as symbols of social and political elite status, as well as in religious ceremonies and other rituals, though some utilitarian implements were also crafted [2]. Based primarily on copper and its alloys, this technology represents a valuable reference for understanding the cultural context in which it developed.

The present paper discusses research pertaining to the West Mexican site of Teuchitlán, Jalisco, framed in the general context of mining and metallurgy in the region. This work represents a systematic attempt to characterize the metallurgy associated with the Teuchitlán Tradition, a pre-Hispanic complex society whose rise and development have been generally dated from the end of the Formative period (200 CE) to the end of the Classic era in Mesoamerica (ca. 900 CE) [3][4].

In 2016, sixteen metal samples of artefacts recovered from Teuchitlán, were analysed by wavelength-dispersive electron probe microanalyser (WD-EPMA) and laboratory-based micro-X-ray fluorescence (micro-XRF). The studied fragments represent adornments such as lost–wax-cast bells and cold-worked rings, as well as implements, including needles and awls. The WD-EPMA results indicate that 14 of the samples are almost pure copper (98 - 100 wt.%) with trace amounts of silver (<0.3 wt.%). Two samples (103 and 68CA2) turned out to be bronzes (3.8 - 4.7 and 8.5 wt.% Sn). Trace element analyses by micro-XRF show the additional elements Fe (<0.5 wt.%), Zn (<0.4 wt.%), Cr, Ag, Cd, Sb, Te, Pb (<0.3 wt.%), Mo (<0.2 wt.%). The two bronze samples show highest Sb and Te. In order to obtain refined analyses with new analytical procedures, all sixteen samples are currently being re-analysed using a different analytical method, which will allow comparison with our previous results.

[1] Beekman, C. S., Recent Research in Western Mexican Archaeology, Journal of Archaeological Research 18, pp. 41-109, 2010.
[2] Hosler, D., The Sounds and Colors of Power, MIT Press, Cambridge, 1994.
[3] Weigand, P. C., J. Herrejón y S. M. Smith, Proyecto Arqueológico “Los Guachimontones”, 2001-2002, Informe Técnico de las Unidades de Excavación Talleres 1 y Talleres 2, 2002.
[4] Smith, M. E., Form and Meaning in the Earliest Cities: A New Approach to Ancient Urban Planning. Journal of Planning History 6 (1), pp. 3-47, 2007.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Casting bronze vessels in ancient Anyang: the technological and organizational problem solving of the bronze casters

Cheng, Wen Y.,[1] Chen, S.[1,2]

[1]Univeristy of Toronto, Department of East Asian Studies
[2]Royal Ontario Museum

Keywords: bronze foundry technology; Anyang bronze mould; foundry organization

The various stages of bronze vessel production during the Shang (1500-1046 BC) and Zhou (1046-221 BC) dynasties transcends their social hierarchy (Franklin, 1999). This research will continue Franklin's (1999) discussion, based on the late Shang capital of Anyang, with the focus on bronze casters within the larger social construct. Although the completed bronze vessels were used by the elites, the bronze casters played a central role in manufacturing these artefacts.

In order to produce the bronze vessels for the elites, the bronze casters had to solve various technological and organizational challenges. The bronze casters' ingenuity in answering these problems can be found in the production sequence left behind in the bronze casting moulds. However, there is a lack of systematic analysis of bronze casting mould's sequence of production. Thus, in this research the various steps involved in producing these moulds based on the mould types and vessel forms will be discussed through the petrographic and SEM analysis of mould samples housed at the Royal Ontario Museum.

Mapping out the sequence of production of specific samples will reveal how the bronze casters tackled the technological and organizational challenges in producing different types of bronze vessels for the elites of Anyang. This work reports a very standardized sequence of production based on the two mould types found at the Anyang site. The result is cross examined based on Li's (2003) and Liu's (2018) spatial analysis of mould types uncovered at the different foundry sites, linking the technology to the spatial distribution of the foundries and how the production was organized between the foundries.

FRANKLIN, U. M., 1999. *The Real World of Technology*. Anansi, Toronto.

LI, Y.-T., 2003. *The Anyang Bronze Foundries: Archaeological Remains, Casting Technology and Production Organization.* Ph.D. Thesis. Harvard University.

LIU, Y., 2018. *Yinxu chutu qingtong liqi zhuzao gongyi yanjiu.* Guangdong renmin chubanshe, Guangzhou.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometry of the “Tomares Hoard”. A first approach based on a statistical sample

García Vargas, E.[1], Chaves Tristán, F.[1], Pliego Vázquez, R.[2], Scrivano, S.[2,3], Gómez-Tubío, B.[3,4], Almeida, Rui de [5,6], Conejo Vázquez, N.[1], Ortega-Feliu, I.[3,4], Ager, F.J.[3,7], Respaldiza, M.A.[3,8]

[1]*Dpto. Prehistoria y Arqueología. Universidad de Sevilla. C/ Dña. María de Padilla, s/n.*
[2]*Centro de Investigación Tecnológica e Innovación (CITIUS). Laboratorio de RX. Universidad de Sevilla, Avda. Reina Mercedes*
[3]*Centro Nacional de Aceleradores (US-CSIC-JJAA), c/ Thomas A. Edison 7.*
[4]*Dpto. Física Aplicada III. Universidad de Sevilla, Camino de los Descubrimientos s/n.*
[5]*Museu Minicipal de Loulé. Câmara Municipal de Loulé.* Praça da República, 8104-001 Loulé.
[6] Uniarq. Universidade de Lisboa. Faculdade de Letras. Alameda da Universidade, 1600-214 Lisboa
[7]*Dpto. Física Aplicada I. Universidad de Sevilla, c/ Virgen de África 7.*
[8]*Dpto. Física Atómica, Molecular y Nuclear. Universidad de Sevilla, Avda. Reina Mercedes s/n.*

Keywords: coin; nummus; XRF; non-destructive technique; bronze

The so-called "Tomares Hoard" is a treasure trove of about 55.000 Roman plated coins issued between 294 and 310-312 A.D. contained in 19 olive oil amphorae of Tejarillo 1 type. The hoard was found accidentally in April 2016 in the Olivar de Zaudín site (Tomares, Sevilla). 8 of the 19 amphorae that constituted the deposit were completely broken during the works that caused the discovery, 9 remained intact and have not been still opened and 2 were damaged, but maintained their contents accessible through the longitudinal rupture of their bodies. In the archaeological excavation carried out *a posteriori* at the site of the find, 102 new coins were recovered. There is no doubt that they remained at the site of the find once the hoard had been collected and deposited in the city's Archaeological Museum. We, therefore, have now several coin collections directly related to the circumstances of the find:

1. The coins found in the amphorae which remain closed (amphorae Nos. 1-9). We estimate a total figure of 25-27,000 coins for this set; 2. The pieces preserved inside the fractured amphorae which have been stratigraphically micro-excavated (amphorae no. 10 and 11). They are 2776 and 2797 coin, respectively; 3. The group of coins not attributable to specific amphorae, which amounts to 25,000 (we call this set of coins the "regular collection"); 4. A small group of 102 coins that were recovered in situ in the salvage excavation carried out at the find site immediately after the find.3.

So far, the 2797 coins from amphora 11 (set 1), 3000 coins from the regular collection (set 3) and all of the coins collected at the find site during the excavation (set 4) have been catalogued and documented. A significant sample of coins from the catalogued coins from groups 2, 3 and 4 were also analysed from an archaeometric point of view using in most cases the XRF spectrometer (Fischerscope X-RAY XUV® 773 from X Ray Laboratory of CITIUS), with a Mo anode, 1 mm diameter beam and 500 µm thick. We present in this paper the characterization of the composition of the alloys, a quaternary bronze alloy (Cu, Sn, Pb and Ag) of coins from groups 2 and 3 in order to perform a statistically significant study of the different mints and periods present in the hoard.

Acknowledgments
Work partially supported by the project PGC2018-093511-B-I00 From the Spanish Ministry of Science, Innovation and University, the PGC2028-093511-B-100 Andalucia FEDER project and VI Plan Propio de Investigación of the University of Seville. The authors wish to thank the Archaeological Museum of Seville and the CITIUS of the University of Seville for their invaluable help during this study.

S6-P72.791

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

SEM-EDS and optical microscopy study of two bells from the carillon of the South Tower of the Royal Building of Mafra

Saraiva, A.S.[1,2], Silva, R.J.C.[1], Figueiredo, E.[1], Bottiani, C.[3]

[1]CENIMAT/I3N, Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologia, Universidade NOVA de Lisboa, Quinta da Torre, 2829-516 Caparica, Portugal
[2]Departamento de Conservação e Restauro, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Quinta da Torre, 2829-516 Caparica, Portugal
[3]HERCULES Laboratory, Department of History, University of Évora, Portugal

Keywords: Royal Building of Mafra; Bell metal; High-tin bronze; Composition; Microstructure

The most typical composition of bronze used to manufacture bells lies within 20-25 wt.% Sn. This has been the alloy of choice for bell foundry from as early as the 5th century BC in Ancient China due to its good mechanical resistance and sound resonance. This alloy has thus been given the name of bell metal.

Samples from two bells of different sizes from the carillon of the South Tower of the Royal Building of Mafra (UNESCO's World Heritage list) were studied by optical microscopy (OM) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS) with the aim of characterize their metal composition and microstructural corrosion patterns. The results were compared to other 4 archaeological bell fragments from different locations of the Portuguese territory and from a time span from the 13th to the 19th centuries, previously studied by OM, SEM-EDS and Raman micro-spectroscopy.

The comparison between the archaeological samples and Mafra's bells cross-sections allowed the visualization of similar corrosion phenomena occurring in the most internal corrosion layers but with some differences in the external corrosion layers, namelly the presence of high levels of copper(I) chloride (CuCl) that can be justified by the proximity of Mafra Palace to the sea.

Regarding the alloy composition the two bells from Mafra showed to have different tin contents although both are near to the ideal bell metal composition range. Contrary to previously expected, the largest bell has more tin than the smallest bell, conferring greater fragility to this large size bell.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Reflectance Transformation Imaging for studying coins surface

Corregidor, V.[1], Dias, R.[2], Catarino, N. [3], Cruz[3], C. Alves, L.C.[1], Cruz, J. [4]

[1]C2TN, IST/CTN, Universidade de Lisboa, E.N. 10, 2686-953 Sacavém, Portugal
[2]IPFN, Instituto Superior Técnico, Universidade de Lisboa, Portugal
[3]MEEC, Instituto Superior Técnico, Portugal
[4]LIBPhys-UNL, Universidade Nova de Lisboa, Portugal

Keywords: RTI; images; surface; corrosion

This work describes the development of a portable set-up controlled by an Arduino board to perform Reflectance Transformation Imaging (RTI) technique. The set-up consists of 45 high intensity light emitting diodes (LEDs) distributed over an hemispherical mesh and a Canon digital camera.
RTI is an imaging technique which documents information about surface reflectance per pixel [1]. This technique needs multiple photographs taken from the same position while the object is illuminated from different light positions in each shot. Using a specially developed computer program (RTI builder) the images are compiled into one single file. Other dedicated viewing software (RTI viewer) enables the visualization of the file and the user interaction by choosing different filters and light position. Both software have been developed by the US non-profit corporation "Cultural Heritage Imaging" [2].
RTI technique allows the visualization of bas-relief of objects and thus, a better representation of the surface than a single standard image, since surface self-shadowing or inter-resections are recorded, enhancing the realism of the image.
Different objects can be studied by this technique. In this work, different coins mint in Portugal were studied. Different features of the surface can be highlighted when using different filters as specular enhancement, where colour information is extracted from the photograph, or using unsharp masking filters. Scratches and corrosion features can be highlighted by means of RTI technique. Furthermore, with this technique, the coins can be analysed by remote researchers, interested stakeholders or the general public, avoiding the transportation, minimizing the manipulation and ensuring their conservation state.

[1] T. Malzbender, D. Gelb, H. Wolters, doi:10.1145/383259.383320
[2] CHI, http://culturalheritageimaging.org/

S6-P83.882

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Technology and functionality on Iron Age weapons from the Iberian Peninsula

Gener-Moret, M.

McDonald Institute for Archaeological Research, University of Cambridge, Cambridge CB2 3ER

Keywords: iron; technology; weaponry; metallurgy; functional analysis

In the field of early iron technology research, besides the works focused in metal production, there is a comparatively less explored area of study that deals with the actual objects made of ferrous alloys. The use of these alloys in manufacture according to the function of the objects constitute a technological corpus of knowledge in itself, with its own patterns of acquisition, development, transmission and exchange, from which conclusions about social change, contacts, and cultural practises can be extracted. For this to be feasible, the analyses must be approached from an archaeological, technological and functional/anthropological point of view, always interpreting "function" beyond the strictly utilitarian sense. Indeed, as technology and purpose are so strongly linked, from the study of the objects we seek to understand, in different cultural contexts, the relationship of manufacturers and users with their technological tradition and with interpersonal violence. This is the basis of the project I present here (IBERIRON), which focuses in the technology of manufacture of weapons and other ferrous objects in the context of the Iberian Peninsula in the Iron Age.

We will present some results of the analytical study and functional analysis of weapons from the sites of Mianes (Tarragona, Spain. Iberian culture, 6th- 5th c. b.c.e.) and La Osera (Ávila, Spain. Vettonian Culture, 4th – 3rd c. b.c.e). Samples have been obtained from various specimens (spearheads, *soliferrea*, atrophied antennae swords and falcatas) and studied by optical metallography and SEM-EDS, showing complex procedures, including uneven use of selective carburization and composite welding. The use of different materials and techniques open the possibility of identifying technological differences between different cultural areas, whereas the purpose and use of the weapons seems to follow similar patterns.

The results also highlight some of the systemic methodological problems encountered in this kind of research, such is the distorting effect of the prevalent cremation rituals on the study of original heat-treatment and carbon distribution, or the difficulties encountered because of corrosion. These problems are addressed with the techniques used, and some additional innovative techniques are proposed, like the use of LA-ICP-MS for the characterization of the slag inclusions or the use of Neutron Diffraction techniques for non-destructive analyses.

S6-P85.496

Session 7 Human-Environment Interactions

ORAL

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multi-analytical study of soluble salt formation on Renaissance wall paintings of Valencia Cathedral (Spain)

Etxebarria, I.[1], Costantini, I.[1], Huidobro, J.[1], Irizar, P.[1], Villate, A.[1], Catala, J.[2], Prieto-Taboada, N.[1], Madariaga, J.M.[1]

[1]Department of Analytical Chemistry, Faculty of Science and Technology, University of the Basque Country UPV/EHU, P.O. Box 644, 48080 Bilbao, Basque Country, Spain.
[2]Catalá Restauradors S.L., Mossen Sorribes avenue, 19 ground floor, 46111 Rocafort, Valencia, Spain.

Keywords: Raman spectroscopy; EDXRF; Ion Chromatography; Wall paintings; Salts.

This work concerns the study of mural paintings preserved in the Valencia Cathedral, known as the Angel Musicians. They were discovered in 2004 and are considered the earliest example of Renaissance painting. Although the paintings, executed in *buon fresco,* were restored after their discovery; some issues related to the emergence of salts and the lifting of the polychrome began to appear again in 2014. Before carrying out new restoring works, it was decided to perform a diagnostic study of the state of conservation in order to identify the cause of the salts and to be able to prevent their appearance in the future. In this research, *in-situ* portable Raman analysis were focused on the most damaged areas, where an extensive formation of salt efflorescence was visible as a means to determine their composition and create damage maps. Samples were also taken to corroborate the in-situ analysis in the laboratory by means of Raman and micro-energy-dispersive X-ray fluorescence (μ-EDXRF) spectroscopies combined with Ion Chromatography (IC). Even though sampling was restricted in the polychrome area, 28 different superficial efflorescences were sampled to corroborate their characterization in the lab, and a depth sampling of up to 27.3 cm was conducted in an area with total loss of polychromy, taking a total of 15 samples. It was established that the main salts damaging the paints were potassium and sodium nitrate, found along with calcium sulphate and calcium carbonate salts. It was concluded that these nitrates did not come from the materials employed for the construction of the vault and they had probably been washed away by water infiltration from the roof. The origin of the nitrate salts could have been the remains of the pigeons that inhabited the site during centuries up to their rediscovery. The results also demonstrated the loss of consolidation suffered by the mortar due to the salts formation. It is intended that the results obtained will help in the restoration and conservation of the paintings in the near future.

S7-O01.1546

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Terrace Archaeology and Culture in Europe (TerrACE): Using Multple Approaches to agricultural terraces and lynchet history

Brown, A.G.[1,2], Fallu, D.J.[1], Cucchiaro, S.[3], Zao, P.[4], Pear, B.[2], Snape, L.[5], Walsh, K.[6], Tarolli, P.[3], Lang, A.[5], Rosa-Albert, M.[7], Van Oust, K.[4], Waddington, C.[8]

[1]The Arctic University Museum of Norway, Tromsø
[2]Geography & Environment, University of Southampton, UK
[3]Department of Land, Environment, Agriculture and Forestry, University of Padova, Italy
[4] Earth & Life Institute UCLouvain, Belgium
[5]Geography & Geology, University of Salzburg, Austria
[6]Archaeology, University of York, UK
[7]Humanities, University of Barcelona, Spain
[8]ARS Ltd., UK

Keywords: lynchets, TLS, OSL, Portable OSL, Stratigraphy, Geoarchaeology, phytoliths, SOM, *seda*DNA

Agricultural terraces and lynchets (unwalled terraces created through ploughing and erosion), are a conspicuous, and nearly ubiquitous but poorly understood, feature of human-altered hilly landscapes. This paper reports on the first results from a new project; *Terrace Archaeology and Culture in Europe* (TerrACE). This is a five-year European Research Council grant funded archaeological research project hosted at the Tromsø University Museum, Norway. The project is applying modern geoarchaeological and palaeobotanical techniques to the investigation of agricultural terraces across Europe from Greece to Norway. TerrACE aims to uncover how and when these landscapes were created, as well as what crops were cultivated in these engineered landscapes, many of which are today under threat.

One aim of the project is to develop an integrated framework for multiple methods – starting with automated surveying using ground based laser scanning (TLS), UAV-generate DEMs' using SfM and automated feature extraction. Terrace and lynchet staircases are then investigated using test-pits, exposures and sedimentological profiling using portable optically stimulated luminescence (pOSL) and portable X-ray flourescence (pXRF). Samples are then taken for phytoliths, soil organic matter (SOM) and sedDNA. So far fieldwork has taken place at 5 sites in the UK, Belgium and Italy with approximately 6 sites yet to be investigated.

In all the field sites the work is being conducted in parallel with archaeological survey and in most cases excavation. This allows the terraces to be put into their local archaeological contexts allowing site comparisons at a higher levels. This is desirable in order to understand complex construction and land use histories which frequently involve partial or complete abandonment and re-use driven by regional socio-political trajectories.

The study of SOM is the part of the project allows us to assess the role of terraces in carbon sequestration and storage and answer the question – how important are terraces as carbon sinks? This relates to the future management and conservation of terraced landscapes. Many terraced landscapes are valued as of great heritage value (e.g. Cinqua-Terra in Liguria) and some have been designated by the FAO as Globally Important Agricultural Heritage Systems (GIAHS). This project is feeding into this program with the aim of conserving terraced landscapes across Europe for archaeological, socio-cultural and environmental benefits.

Session 7 Human-Environment Interactions

POSTER

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Population Regimes and Stable Carbon ($\delta^{13}C$) Isotopes in Hunter-Gatherers from Central Texas, USA.

Mauldin, R.[1], Freeman, J.[2], Hard, R. J.[3], Munoz, C.[1], Anderies, J. M.[4], Carpenter, M.[3]

[1]Center for Archaeological Research, University of Texas at San Antonio, San Antonio, Texas, USA.
[2]Anthropology Program and Ecology Center, Utah State University, Logan, Utah, USA.
[3]Department of Anthropology, University of Texas at San Antonio, San Antonio, Texas, USA.
[4]School of Sustainability, Arizona State University, Tempe, Arizona, USA

Keywords: demography; intensification; dietary change; stable isotopes

Using approximately 1,000 radiocarbon dates and stable carbon isotopes from 125 human interments, we document the adaptive patterns of hunter-gatherers over roughly 8,000 years within a 50,000 square kilometer area in Central Texas, USA. A summed probability distribution (SPD) of the radiocarbon dates serves as a proxy for population levels and allows us to define three demographic regimes, periods over which population patterns were broadly similar. After attempting to control for sampling and taphonomic bias, the SPD pattern suggests that each of these regimes sequentially support higher population levels. Much of the earliest period, which dates from 8500 to 5800 BP, is characterized by little or no growth. Populations do spike near the end of this period, establishing a higher level which appears stable from 5800 through 2800 BP. The final period, from 2800 BP to roughly 350 BP, is characterized by accelerated population growth. Using these three time frames, we then consider patterns in stable carbon ($\delta^{13}C$) derived from bone collagen and apatite to gauge the impacts of these shifting population levels on hunter-gatherer adaptations. The stable carbon isotopic patterns document two temporal trends. First, there are directional changes in the average diet, with a shift of over 3‰ present in $\delta^{13}C$ values in collagen and carbonate that suggests movement away from C_4 resources and intensification on C_3 plants and animals. Second, when measured either by the inter-quartile range (IQR) or by the coefficient of variation (CV), there is a substantial increase in carbon isotopic variation for each of these population regimes through time. This second trend suggests that individual diets were more localized with increasing populations. Increased variation is especially apparent in the most recent time period (2800-350 BP) when the SPD curve suggests that prehistoric populations were at their apex. These patterns of intensification and dietary variation are consistent with reduced mobility anticipated at higher population levels.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Study of conservation state of open-air prehistoric rock art site affected by natural and antropogenic agents

Costantini, I.[1], Etxebarria, I.[1], Prieto-Taboada, N.[1], Ruiz, J.F.[2], Castro, K[1], Arana, G.[1] Madariaga, J.M.[1]

[1]Department of Analytical Chemistry, Faculty of Science and Technology, University of the Basque Country UPV/EHU, P.O. Box 644, 48080 Bilbao, Spain
[2]Departamento de Historia, Área de Prehistoria, Facultad de Ciencias de la Educación y Humanidades, University of Castilla-La Mancha (UCLM), Avda. de los Alfares 42, 16002 Cuenca, Spain

Keywords: Raman spectroscopy; µ-EDXRF; XRD; rock shelter Levantine art; conservation

In this work, six microsamples belonging to the open-air rock art site of *Cueva de la Vieja* (Alpera, Albacete, Spain) were analysed. This study is contextualised in an intervention aimed at recovering the visibility of the pictographs, which includes a diagnostic phase, subsequent cleaning and consolidation by specialized restorers, and a final phase that will produce a new digital tracing. The rock shelter includes 131 figures, most of which are in the Levantine style, while the rest are of Schematic style. Cueva de la Vieja were included in the UNESCO World Heritage list in 1998, being one of the most significant sites of the Rock Art of the Mediterranean Basin of the Iberian Peninsula property. A whitish layer of concretion, produced by the repeated humidification in order to improve the visualization of the pictographs, affects the painted panel. On the other hand, other alteration phenomena like cracks, alveolization, dissolution and biological patinas are nowadays visible. Thus, a diagnostic study by means of micro energy dispersive X-ray fluorescence (µ-EDXRF) spectrometry, Raman spectroscopy and X-ray diffraction (XRD) were carried out for the characterization of the degradation layer as well as of mineral substrate and pigments. The microsamples analysis demonstrated that the painted layer was applied on a dolomitic limestone with silicon aggregates and aluminosilicates as well as iron oxides, mainly hematite. The whitish crust was composed by sulfate compounds like gypsum ($CaSO_4 \cdot 2H_2O$) with minor amount of epsomite ($MgSO_4.7H_2O$) generated by the dissolution of carbonates, from the dolomitic limestone, and the following reprecipitation as sulfates. An extensive phenomenon of biological activity was demonstrated since, in almost all the samples analysed, the presence of calcium oxalates monohydrate ($CaC_2O_4H_2O$) and dehydrate ($CaC_2O_{42}H_2O$) were found. Probably the presence of both calcium oxalates had favoured the conservation of pictographs. In addition, some carotenoids pigments such as scytonemin ($C_{36}H_{20}N_2O_4$) and astaxanthin ($C_{40}H_{52}O_4$) were characterized both by Raman spectroscopy and by X-ray diffraction. The detection of this kind of pigments underline that the rock shelter was affected by a hard environmental stress since they are produced by microorganisms as a defence mechanism in extreme environments. Traces of hematite were found as a pigment voluntarily used for the painting of the panels.

S7-P02.1545

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Microclimates and landscape archaeology: the case of Enlène cave

Bourges, F.[1], Pastoors, A.[2] Bégouën, R.[3], Lartiges, B.[4], Genty, D.[5], Perrier, F.[6], Girault, F.[6]

[1]Géologie Environnement Conseil, 30 rue de la République, F-09200 St Girons, France
[2]Friedrich-Alexander-Universität Erlangen-Nürnberg, Institut für Ur- und Frühgeschichte, Kochstraße 4/18, Germany, D-91054 Erlangen
[3]Association Louis Bégouën, 113 Pujol, 09200 Montesquieu-Avantès, France
[4]Université de Bordeaux, UMR CNRS 5805 EPOC – OASU, Allée Geoffroy Saint-Hilaire, F-33615 Pessac, France
[5]Université de Toulouse III Paul Sabatier, Géosciences Environnement-Toulouse, 14 av. Edouard Belin, F-31400 Toulouse, France
[6] Institut de Physique du Globe de Paris, Université de Paris, CNRS, F-75005 Paris, France

Keywords: cave; microclimate; habitat; palaeolithic

Connecting the material culture with its natural context is a successful practice in archaeology. This landscape-archaeological approach was applied to the cave environment to investigate to what extent the microclimate influenced the use of the underground space by prehistoric humans.
Investigations took place in the Enlène cave, one of the so-called Volp Caves located in a multiphase karstic massif of the French Pyrenean piedmont. The river Volp flows in a low gallery while the two upper galleries contain most of the prehistoric remains. The Enlène cave is interpreted as a basecamp while the connected Trois-Frères cave contains mainly symbolic representations such as rock art. Despite archaeological excavations in the 19th and 20th centuries, the cave and its entrances geometry were not modified justifying the assumption that the original microclimate pattern has been preserved.
The Enlène cave is connected to the outside by two entrances at different levels, to the drainage gallery by a vertical pit, and to the Trois-Frères cave by a long and narrow tunnel. These relationships with outside and inside air at different levels induce a complex cave aerology resulting in successive seasonal regimes and space partitioning microclimates. In the upper entrance (a porch largely open to outside), winter temperature is 10°C higher than exterior for 1036 h/year, that is at least 1h/day during 82 days from November to March. Inside, the entrance galleries are preserved from the direct influence of winter temperature by steady outflow of subterranean air (systematically > 10°C). This attractive climatic entrance sector hosts different locations of rich archaeological layers. Located 200 meters away from the entrance, the Salle du Fond is a remote large chamber protected from draughts and temperature changes by confined microclimate. More than 42,000 units of sandstone slabs were brought there from the outside forming a large-scale paved area with fireplaces. The huge quantity of artifacts and elements of “chaînes opératoires” for pearls, needles and tools support the interpretation of basecamp activities. Attractive microclimatic locations coincide with an intensive use by prehistoric humans. In the large intermediate galleries with permanent air circulations, archaeological strata are also present, but show a lower density of artifacts.
The climatic zoning identified in Enlène is therefore in direct relationship with the occupation of the underground landscape.

S7-P05.165

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Influence of deterioration on the preservation of mud brick architecture based on the monuments from the Tell el-Retaba archaeological site

Zaremba, M.[1], Bobrowska, A.[2], Trzciński, J.[2], Szczepański, T.[2], Welc, F.[1]

[1]Institute of Archaeology, Cardinal Stefan Wyszynski University, Wóycickiego 1/3, 01-938 Warsaw, Poland
[2]Faculty of Geology, University of Warsaw, Żwirki i Wigury 93, 02-089 Warsaw, Poland

Keywords: Residential and defence structures; Physical and mechanical properties; Reconstruction of mud brick; Weathering processes; Nile Delta; Climate change

Properties of mud bricks and reconstructed brick material from the Tell el-Retaba archaeological site in Egypt have been compared. The site is located in the transitional zone between humid sub-tropical and dry tropical climate. The influence of deterioration on changes of the physical and mechanical properties of mud bricks has been assessed as a factor of time. Effects of deterioration of the mud brick material have been presented: exfoliation as an effect of heating enhanced by colour, mud brick deformation and salt crystallisation as a result of cyclic watering and evaporation, and loosening of the structure caused by numerous factors. With brick age, decrease of bulk density and increase of porosity has been observed. The values of these parameters in the reconstructed brick material do not show a similar trend. Differences in grain size and mineral composition of the material used for brick production influenced not only the speed of deterioration processes but also the prevailing type of the process. Comparison of the properties of mud bricks and the reconstructed material indicates that mud bricks were characterised by good initial values. However, with time, the physical and mechanical properties of the mud bricks became significantly worsened following the long-term influence of deterioration processes.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Evaluation of the use of natural materials for the cleaning of bronze leaches on marble using spectroscopic tecniques.

Barbier, I., Vazquez-de la Fuente, I., Etxebarria, I. [1], Prieto-Taboada, N., Arana, G., Madariaga, J. M.

Department of Analytical Chemistry, Faculty of Science and Technology, University of the Basque Country UPV/EHU, P.O. Box 644, 48080 Bilbao, Basque Country, Spain.

Keywords: Agar, Kudzu, Konjac, bronze sculptures, spectroscopic techniques.

The cleaning of the leachates appeared in the marbles close to bronze sculptures is an important goal to be solved. The use of aggressive reactants could damage the materials and, in the field of cultural heritage, this point must be addressed carefully. For this purpose, the use of agar gels has been the best option to put the reactant in contact with the materials, controlling its application. However, the use of agar gels could promote the appearance of fungi because they are a good substrate for them. Taking all of this into account, new natural alternatives have appeared in the last years. The most studied one in our research group is Kudzu starch which seems to be also antimicrobiological activity improving the classical applications. Analogously, Konjac gel seems to show also good qualities as alternative to Agar gels. Thus, the usefulness of both materials was evaluated prior to the cleaning of the marble support of a bronze sculpture of the first middle of the 19th century located in the city cemetery in Getxo (Bizkaia). This marble is affected by the leaching of the bronze sculpture promoted by the rain and acid atmosphere but it is also affected by microbiological activity. Therefore, a careful study of the efficiency of these materials was carried out comparing its effectiveness with agar gels in marble mock-ups. The gels were checked without any reagents, as well as with different chelates as EDTA, citrate, acetylic acid, oxalate and gluconic acid. These pieces were characterized using X-ray fluorescence, Raman spectroscopy and colorimetric measurements before and after their ageing. Moreover, the gels were analyzed by means of inductively coupled plasma mass spectrometry (ICP-MS). The results show that these new natural materials are a good alternative to the cleaning of this type of marbles affected by bronze leaching, and the results will be useful in the restoration on this sculpture.

This work has been supported by the DEMORA (Grant No. PID2020-113391GB-I00) projects funded by the Spanish Agency for Research AEI (MINEICO/FEDER-UE).

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Stony materials as source of information in Braga (NW Portugal)

Alves, C.[1], Sanjurjo-Sánchez, J.[2]

[1]LandS/Lab2PT - Landscape, Heritage and Territory Laboratory (FCT UID/AUR/04509/2013; FEDER COMPETE POCI-01-0145-FEDER-007528) and Earth Sciences Department/School of Sciences, University of Minho, Braga, Portugal;
[2]University Institute of Geology, University of A Coruña, 15071 A Coruna, Spain

Keywords: water springs; stone typology; stone alteration features

Braga (NW Portugal) has a long documented history and its tangible heritage can provide further information on human interactions with the geological environment, as we will attempt to show with a synthesis of around three decades of research.

The idol's fountain is a unique case in Braga where a granite outcrop is the preservation medium for the information (sculptures and engravings showing the importance of groundwater for early civilizations). Its unique character might reflect the attribution of specific properties to the place, absence of other water springs in suitable outcrops or the destruction or concealment of similar situations (the concealment hypothesis suggests that they could be found in the future).

In other cases, the displaced rocks (stones) are "per se" a source of information. In Braga, two situations can be highlighted: variations of the local granite (darkish biotite-rich granite), including more weathered varieties (easier to work) and big stones (indicating the extraction of blocks with great dimensions), and imported rocks, especially light coloured granites that seem to indicate an aesthetic option (some fine-grained varieties could be confounded with marbles). Some structures present these two broad granite types with distributions that could be related to temporal or architectural patterns.

Crystal neoformations on stones (formed after their emplacement in human structures) can also highlight specific activities when they can be associated with specific sources. In Braga, the most significant cases concern efflorescences of sodium chloride, which in this inland location could mark situations of use or storage of common salt, and nitrate potassium, representing contributions from feeding products for herbivore domesticated animals and from these animals wastes. One can also refer carbonate crusts that signal the use of modern cements (and that might also be a source for dating of structures, a hypothesis that suggests the interest of their preservation).

One can also include here the study of mortars as they occur in intimate association with stones: mortar joints (a potential element for dating and where the presence of cement mortars indicates modern interventions) and mortars used for stone replacements (indicating extreme stone degradation).

The synthesis presented here shows how the study of rocks, stones and their alteration products can contribute to the study of human activities along time in the town of Braga.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Understanding the ancient Marmarica landscapes: cognitivism and networks in harsh environments

Laguna-Palma, D.[1]

[1]*Department of Prehistory and Archaeology. University of Granada (Spain).*

Keywords: Landscape Archaeology; Social Networks Analysis (SNA); Human-environment; Uncertainty principle; Marmarica.

In the last two decades, there has been an exponential growth in the number of projects studying the connectivity around the Mediterranean in the past, from ecological to social and economic dynamics(Horden & Purcell 2000; Broodbank 2013; Leidwanger & Knappett 2018). This means that we can interrelate different entities, from communities to ideas or actors to environments, in a simpler but complex system.The present work is part of the results obtained by the PERAIA project, which aims to study the human-environment interactions along the Marmarica region from the Late Bronze to the Early Iron Age (c. 1500 – 700 BC). We present a reflection on how to understand and study network modellingby using different proxies (e.g. pottery and mobility patterns) and the importance of assessing criticallyhow to deploy SNA. These digital challenges, we argue, should be confronted with other landscape data, such as understanding the mental-phenomenological approach of cultural movement in harsh environments – and, conversely, the environmental effects on the development of these societies.

In turn, this research seeks to enrich the study of land/seascapes -where interactions occur. In consequence, a landscape must be understood as a space that encompasses different agents and scales (Moreland, 2010; Criado-Boado, 2017). Precisely, these different scales mean to focus on a multi-scalar analysis (Tartaron, 2013). Such an approach has enhanced our understanding of what the ancient Mediterranean was: a warped community (Broodbank, 2013).

On the other hand, all data and consequently geospatial analyses involve uncertainty. Although its importance has been widely recognised, uncertainty issues are not yet addressed in deep. In this sense, and aware of the aforementioned theoretical debate regarding data, the project has developedsome scales of 'trustiness'. These scales are applied to the identification and mapping of archaeological sites, as well as to the digitisation of ancient routes.

Summing up, we will seek to reflect on some of the theoretical caveats brought by this approach. More specifically, this work aims to provide an overview of concepts, sources, and tools, and how theproject manages uncertainty to map material remains that serve as proxies for tracing ancient networks, especially in harsh environments.

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

The Paleoecological Context of the Lower Chincha Valley, Peru Using Pollen Analysis and pXRF of Soils

Bergmann, C.L., Tykot, R.H.

Department of Anthropology, University of South Florida, Tampa, FL 33620 USA

Keywords: pollen, pXRF, soil, Peru

Andean archaeologists have long assumed that the coastal valleys in Peru possessed a barren desert environment which was irrigated for agricultural use at the time of initial human settlement. Recent research in the Chincha Valley is now challenging that assumption. The Chincha Valley, located 200 km south of Lima, is one of the largest valleys on the south coast of Peru. Previous archaeological research demonstrates a long history of occupation in the valley beginning in the 2nd millennium BCE with the archaeological site of Pozuelo. Pozuelo is the earliest known site in the Chincha Valley as well as possesses the oldest U-shaped mound structure on the south coast of Peru. Radiocarbon dates on human bones date to about 1200-1000 cal BCE, while $\delta^{13}C$ values averaging -13.3‰ suggest that seafood was a major part of the diet. Recent excavations at Pozuelo, as well as at surrounding sites, suggest the region was inhabited in an environment vastly different from what is seen today.

Our research tests the hypothesis that during initial settlement of the region, individuals had the ability to occupy the land in the absence of irrigation agriculture using landscape modification strategies, such as the draining of marshlands, to permit the cultivation of agricultural resources. Evidence for a marshland environment in the Chincha Valley comes from pollen analysis of ten soil samples from Huaca Soto, a site adjacent to Pozuelo, and twenty soil samples from Pozuelo. The results demonstrated the presence of *Typha*, a plant that only thrives in a wetland environment. In addition to pollen analysis, a case study was performed using portable X-ray fluorescence spectrometry (pXRF) on twenty soil samples from three units excavated in the court of the U-shaped mound at Pozuelo. The samples were taken from varying levels within the units to get a comprehensive picture of the different environments prior to initial settlement and after occupation. The samples were analyzed for barium, calcium, copper, iron, potassium, manganese, strontium, and other elements as well as plotted with baseline data acquired from different environments in Peru. The results suggest pXRF is a valid technique to use to differentiate soils from different environments.

S7-P15.587

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Fungi and molds in museum environment: Fungal biodeterioration of exhibited materials in the Criminology Museum of National & Kapodistrian University of Athens

Tziamourani, E.[1], Mitronatsios, D.P.[1], Nikolaidou, N.V.[1], Stefanidou, M.[2], Panagiaris, G.

[1]*University of West Attica, Faculty of Applied Arts and Culture, Department of Antiquities and Works of Art Conservation*
[2] *National and Kapodistrian University of Athens, Faculty of Medicine, Museum of Criminology*

Keywords: museum environment fungi molds biodeterioration

In museum and archives collection environments fungi are a critical artefact biodeteriotation factor, whereas most infections are airborne. Poor ventilation and temperature variations can produce water condensation points and adverse local micro-climates. These conditions favour fungal growth activity in specific museum exhibit areas; along these lines, colonizing paper made documents is typically caused by species of slow-growing *Ascomycetes*, as well as mitosporic xerophilic fungi of the genera *Aspergillus, Paecilomyces, Chrysosporium, Penicillium* and *Cladosporium*. Plus, some of the fungi present in paper documents, surfaces and air from archives, libraries and museums are also a threat to human health.

In this paper we present the results from indoor/outdoor measurements of particulate matter mass/number concentrations and viable, cultivable microbial load were performed in museum for a period of 2 years at selected time intervals. Measurements of inhalable particulate mass (PM) and viable, cultivable airborne microorganism concentrations in air were performed. Also the isolation of microorganisms and fungi with a classical microbiological techniques are presented. The fungal populations and the effect of environmental conditions on the paper surface were identified and characterized using optical microscopy (OM) and scanning electron microscopy (SEM).

The indoor PM and microbial concentrations were higher than the outdoor levels showing the influence of the indoor sources, such as the presence of people and indoor activities, as well as, anthropogenic outdoor sources and natural emissions. Two fungal species belonging to different genera were isolated from paper artefacts in case environments. Samples exposed to the environment showed positive results in contrast to the samples of the back cover. *Aspergillus niger* appeared to be the dominant fungus with a maximum number of colonies growing on Sabouraud Dextrose Agar (SDA) medium, and opportunistic pathogenic heterotrophic bacteria in the museum showed to be enriched inside the closed showcases.

This work aims to show that the identification of a fungal genus or species on a document does not necessarily mean this fungus is the actual cause. Showcases offer not always a protection for PM and specific airborne bacteria. In contrast, an enrichment of viable, cultivable airborne bacteria inside the showcases was observed that may be related to a possible utilization of the exhibits materials as sources for growth. An appropriate management of the museums microclimate according the ISO 11799, in accordance to proper cleaning and disinfestating of the rooms, is the best practice to protect the exhibits and the health of people who work in them.

S7-P17.699

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Relationships between human activities and the environmental of Cyprus and South Levant area

Dzwoniarek-Konieczna, M., Węcławska, M.

Institute of Geology, Adam Mickiewicz University in Poznań

Keywords: Cyprus; Levant; geoarchaeology; settlements studies

The degree of human assimilation with the natural environment, as well as the ability to overcome subsequent settlement barriers and use of available natural resources affects settlement development to this day. In settlement studies, the geological factor is analyzed in the context of research on paleoecology of the site, paleoclimate or provenance of rock raw materials used in material objects from site. Geological and geomorphological data (e.g., information about rocks basements, tectonics, topography, landforms and types of sediments or soils) is often used in settlement studies aimed on place settlements in the environmental context.

The aim of this paper is pointing out main geological factors that influenced settlement development and its functioning from Early Bronze Age to Roman period based on examples sites from coasts of Cyprus (Paphos and Limassol region) and South Levant area (Akko and Tyre region), based on an analysed of satellite imagery from contemporary data available through Bing Maps and Google Earth; geological and geomorphological maps; and comparised field survey.

This work analyses and presents two groups of factors. Positive or neutral factors like: location at a strategic point between different geomorphological zones, land elevations, watercourse, on a sheltered coast, availability of raw materials conditioning the development of industrial and trade type of settlements. Numerous settlement barriers (negative factors), such as climate impacts, storms, bedrock and active geological processes (erosion, landslides, earthquakes and other natural hazards) leads to the limitation or fall of these settlement functioning.

A preliminary study on iron gall inks in historical documents of the Portuguese Inquisition

Nunes, M.[1], Wanzeller Martins, G.[1], Correia, D.[1], Mitchell, S.[2], Claro, A.[3], Ferreira, T.[1,4]

[1]HERCULES Laboratory, University of Évora, Largo Marquês de Marialva, 8, 7000-809 Évora, Portugal
[2]Instituto de Nanociencia y Materiales de Aragón (INMA-CSIC), CSIC-Universidad de Zaragoza, c/Pedro Cerbuna 12, 50009 Zaragoza, Spain
[3]Centre for the Humanities (CHAM), NOVA School of Social Sciences and Humanities, Avenida de Berna, 26-C, 1069-061 Lisboa, Portugal
[4]Chemistry Department at the Science and Technology School, University of Évora, Rua Romão Ramalho, 59, 7000-671 Évora, Portugal

Keywords: historical documents; written sources; analytical characterization; degradation processes

The conservation of written heritage with iron gall ink (IGI) is a subject of deep interest which encourages further investigation on the subject. A plethora of recipes to prepare this dark ink is known and includes three main ingredients: plant tannins extracted from oak tree galls, metallic salts, usually iron sulphate, and a binder [1]. Initially, it presents a violet-black hue that fades over time to a rusty brown tone. IGI historical formulations were said to be usually unbalanced due to an excess of transitions metals in relation to tannins or the other way round. Thus, these formulations play a key role in triggering degradation mechanisms in the paper supports as acid-catalysed hydrolysis and oxidative depolymerization [2]. Both mechanisms are also activated by exogenous factors such as temperature and humidity fluctuations and exposure to radiation [1-3]. An immense written heritage is threatened by the ink itself, being the official documents of the Portuguese Inquisition Courts, hold by the Arquivo Nacional da Torre do Tombo (ANTT), an example of that. Unfortunately, a considerable part of those archives can no longer be accessed by the general public or the researchers due to their delicate condition. Hence, is fundamental to characterize the materials used, deepen the understanding of the degradation processes and develop innovative methodologies for the conservation of the supports.
The work here presented is a preliminary analytical study carried out on collected fragments from historical records of the Inquisition Courts of Coimbra, Lisboa and Évora, from the 16th to the 19th centuries. An integrated methodology that included morphological and chemical characterization was complemented with the historical context of the samples. Optical microscopy, as a first step, allowed to characterize the surface morphology and pathologies and scanning electron microscopy coupled with energy dispersive spectroscopy (VP-SEM/EDS) was used to provide deeper morphological and chemical information. Micro-Fourier transform infrared spectroscopy (FT-IR-ATR) and Micro-Raman analyses complemented the study.

Acknowledgements: The authors acknowledge the ANTT team for providing access to the samples and FCT for funding (IRONIC project PTDC/ART-HIS/32327/2017, UIDB/04449/2020 and UIDP/04449/2020). G. Wanzeller thank the support by the FCT grant PTDC/ART-HIS/32327/2017 in the scope of IRONIC project and M. Nunes also thanks FCT for a PhD scholarship (SFRH/BD/147528/2019).

S7-P19.865

Session 8 Nuclear and Radioactive-based Techniques in Cultural Heritage

ORAL

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

INVITED LECTURE

Heritage Science at the Budapest Neutron Centre

Kasztovszky, Zs.

[1]*Centre for Energy Research – H-1121 Konkoly Thege str. 29-33., Budapest, HUNGARY*

Keywords: Research reactor; neutrons; activation analysis; imaging; diffraction

Neutrons, as particles having zero electric charge, are ideal tools to study the inner parts of objects, independently of their chemical or physical forms. By detecting the prompt- or delayed gamma photons emitted in (n,γ) radiative capture reaction, “bulk” elemental composition of the irradiated volume can be determined (PGAA and NAA techniques).
Furthermore, by detection of scattered neutrons (TOF-ND and SANS techniques), structural information on 1 to 1000 Å scale can be obtained.
Finally, by detecting the attenuation of a neutron beam caused by an extended object with invisible inner structure, hidden details of the object can be revealed without any destruction of it. 2D and 3D grey-scale images can be recorded (NR and NT techniques). Moreover, by combination of compositional and imaging methods, elemental distribution of a bulky object can be reconstructed (PGAI).
Most of the above mentioned methods do not require any sampling, and due to the relative low neutron intensities, it can be considered absolutely non-destructive.
At the Budapest Neutron Centre several neutron-based methods are extensively used in various projects related to cultural heritage already since the beginning of 2000s. For more years, we are access providers in European user access programs in the heritage science discipline (e.g. CHARISMA, IPERION-CH and IPERION-HS)
Compositional data measured by PGAA or NAA are used for provenance studies of various stone objects, ceramics, glass or metal objects. Neutron diffraction (ND) is used to identify ancient production techniques or corrosion states of metal objects. With recent developments in neutron imaging methods, important technological information about unique, precious objects (sculptures, vessels) have been obtained.
Description of the experimental neutron techniques used in heritage science, as well as examples of their successful applications at the Budapest Neutron Centre are presented.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Neutron tomography - a tool to virtually unwrap folded lead amulets

Mannes, D[1], Wilster Hansen, B.[2], Kutzke, H.[2], Ødeby, K.[3], Langsholt Holmqvist, K.[3]

[1]Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institut (PSI), Switzerland
[2]Museum of Cultural History, University of Oslo (KHM), Norway
[3]Norwegian Institute for Cultural Heritage Research (NIKU), Norway

Keywords: Lead amulet; runic inscription; neutron tomography; non-destructive testing

Wearing amulets as lucky charm and to ward off evil was very common in medieval Scandinavia as well as in the rest of Europe. Some of these amulets consisted of lead sheets covered with inscription, which was grooved into the surface. The sheets were then rolled or folded, partially to conceal and protect the inscription to preserve or enhance the protective power of the inscription. The text itself could be written with runes or Roman letters and could consist of biblical verses, prayers, the names of saints, Jesus or god or even the names of demons.

In recent years, several of such textual amulets were found in Scandinavia. The content of the inscriptions on these amulets represents a valuable source of knowledge to study the beliefs and practices among common people. Many of these folded lead amulets show unfortunately signs of corrosion. Mechanical unwrapping of such fragile and brittle objects would not only be time-consuming but also change the original appearance of the object and most of all would bear the risk of damaging or destroying the object. In order to avoid mechanical unwrapping, non-destructive methods have to be taken into account. While X-ray tomography is a widespread method, which has already been successfully employed to virtually unwrap parchment scrolls or even scrolls of thin metal, it appears it appears less suitable for the study of lead objects. Lead features a very high attenuation coefficient for X-rays and is hence commonly used as shielding material. An alternative method working along similar principles (i.e. transmission images) is neutron tomography. This method shows a partially complementary behaviour in comparison to X-ray. While lead is largely opaque to X-rays it shows high transparency for neutrons. On the other hand some light elements such as hydrogen (e.g. organic material) show high contrast for neutrons but scarce for X-rays.

In the presented study an amulet found in an urban excavation in the historic centre of Oslo (Norway) was investigated by means of neutron tomography at the neutron imaging facilities of the Paul Scherrer Institut (PSI) in Switzerland. The resulting tomography data was subsequently virtually unfolded using the visualisation and analysis software VG studio max. This virtual unwrapping allowed to unveil the runic inscription hidden inside the folded amulet. Based on the neutron tomography data it was possible to read and interpret the inscription to large extents.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Non-destructive and position selective elemental analysis method using negative muon

Ninomiya, K.[1], Kubo, M.K.[2], Miyake, Y.[3], Shinohara, A.[1], Saito, T.[4]

[1]*Graduate School of Science, Osaka University*
[2]*College of Liberal Arts, International Christian University*
[3]*Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK)*
[4]*National Museum of Japanese History*

Keywords: non-destructive analysis; elemental depth-profiling; quantitative analysis; muonic X-ray; muon

Elemental analysis is one of the most fundamental and essential techniques for various research fields. Many elemental analysis methods have been developed, for example, chemical separation, X-ray fluorescence, mass spectrometry, and applications of some nuclear processes such as nuclear activation. Among them, needless to say, non-destructive analysis methods are of great benefit to the practicing scientist, and some useful methods have been developed, such as X-ray fluorescence analysis and neutron activation analysis, and these methods have already been applied for analysis of heritage. However, an analysis method that is non-destructive, three-dimensionally position selective, and can detect multiple elements including light ones for a bulk sample has remained elusive.

Here, we propose a new probe for non-destructive elemental analysis: negative muons. Muons have the same charge as an electron, but are 207 times heavier. Muons form muonic atoms in material that contain one muon in an atomic orbital instead of an electron. Due to the large mass of the muon, the energy of characteristic X-ray from muonic atom (muonic X-ray) is very high, and the "X-ray fluorescence analysis" method using a muon can solve the problem of self-absorption in an ordinary method using an electron. As a result, position selective analysis for a bulk sample is possible using muons.

We have been developing a new elemental analysis method with muonic X-rays at the muon science facility (MUSE) in the Japan Proton Accelerator Research Complex (J-PARC), and applied this method for some archeological artifacts; bronze coin [1, 2] and gold coin [3]. The elemental components of bronze (mixing ratio of Cu, Sn and Pb) are important to investigate production method and producing area of the material. Usually, such analysis is mainly performed by fluorescent X-ray analysis, but the surface of bronze is oxidized, and the elemental components may different between surface and deep parts. In fact, the different analysis results from muonic X-ray measurements were obtained by analysis depth; the elemental component of lead becomes low near the surface of the bronze. In the analysis of gold coin, the concentration of gold near the surface was increase about 1.5 times than the deep side.

[1] K. Ninomiya et.al., Bull. Chem. Soc. Jpn., **85** (2012) 228
[2] K. Ninomiya et.al., JPS Conf. Proc., **8** (2015) 033005
[3] K. Ninomiya et.al., Anal. Chem., **87** (2015) 4597

S8-O03.222

Lipari Obsidian and Neolithic communities: new chronological elements through fission track dating

Bonizzoni, L.[1], Balestrieri, M. L.[2], Coltelli, M.[3], Guglielmetti, A.[1], Manni, M.[3], Martinelli M. C.[4], Oddone, M.[5]

[1]University of Milan, department of Physics "Aldo Pontremoli",via Celoria 16, 20133 Milano, Italy
[2]IGG-CNR, Via G. Moruzzi 1 56124 Pisa, Italy
[3]Istituto Nazionale di Geofisica e Vulcanologia, Osservatorio Etneo, Piazza Roma 2, 95125 Catania, Italy
[4] Parco archeologico delle Isole Eolie Museo Luigi Bernabò Brea, Lipari, Italy
[5] University of Pavia, Department of Chemistry, Via Taramelli 12, 27100 Pavia, Italy

Keywords: fission track dating; Lipari obsidian; Neolithic populations

The present work is part of a wider project aimed at studying the connection between obsidian flows on the island of Lipari and Neolithic populations on the Aeolian archipelago, in Italy. As it is well known, obsidian is of particular interest to trace prehistorical trading patterns; indeed, Lipari obsidian has the widest distribution and has been found in southern France, Dalmazia, Sicily and mainland Italy.
The project outputs will give a general vision of both archaeological and volcanological aspects through the stratigraphic and radiometric dating of eruptions which produced obsidian, in relationship with the first phases of the human settlements and row material exploitation. To reach this goal, we considered both raw materials from different flows and artefacts from Neolithic settlements on the Aeolian islands, and performed fission track dating. To this end, obsidian samples were divided in two fractions, one of which was irradiated at the TRIGA Mark II Research Nuclear Reactor of the LENA Laboratory (University of Pavia, Italy). Standard glasses, together with mica foils acting as external detectors, were also irradiated for thermal neutron fluence determination. The irradiated and non-irradiated fraction of obsidian samples were then prepared for etching and microscope observation, applying the standard technique consisting of mounting samples in epoxy resin. Etching was performed with HF in monitored conditions and samples were observed with total 500x magnification for spontaneous and induced fission track identification. This procedure allowed to get the density of fossil and neutron induced tracks and therefore the cooling age of obsidian source and artefacts.
Obtained results are expected to shed some new light on the raw material procurement and on the ability of the Neolithic populations to move from their locations, with particular attention to the consequences of environmental features on the first human settlements on the Aeolian islands.

S8-O04.237

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Complex analytic researches of a Medieval enamelled Bronze pendant from Rus'

Zaytseva, I.[1], Stolyarova, E. [2], Kovalenko, E. [3], Podurets, K.[3]

[1]*Institute of Archaeology of Russian Academy of Sciences*
[2]*M. V. Lomonosov Moscow State University*
[3]*National Research Center "Kurchatov Institute"*

Keywords: Rus'; bronze pendant; enamel; X-ray neutron and synchrotron methods

2018 archaeological researches in the Suzdal region (250 km far from Moscow) discovered a quatrefoil bronze pendant decorated with enamelled images on either side. On the one side, there was Christ Emanuel, and on the other, as the inscription states, Holy Martyr Nestor of Thessaloniki. The pendant measures 33×27×2.5 mm. This artefact dates from the second half of the twelfth to the early thirteenth century. The pendant is the unique artefact first found in an archaeological context.

The studies of the manufacturing technology and chemical composition of the metal backing and enamels applied a complex of analytic methods. Neutron tomography supplied a complete picture of the inner structure of the artefact. Synchrotron analysis only uncovered the structure of enamel layers as deep as ca. 400–500 μm from the surface. The metal backing is 0.9–1.2 mm thick, and enamel layers are 0.6–0.9 mm thick. Phase constitution of the enamel was analysed with thermal neutron diffraction. Crystalline quartz was discovered: tin dioxide was opacifier in deep blue and white enamels, and cobalt magnetite was colouring pigment in deep blue enamels. Elemental composition of the surface enamel layer and the metal has been studied with the SEM-EDS methods. It has been revealed that the metal backing was moulded of tin-lead bronze containing 15% of tin and ca. 2% of lead. Visible metal areas were covered with gold amalgam. Our analyses of enamels of various colours have uncovered the use of sodium glass of Mediterranean or Near Eastern origin.

Initially, the images were supposed to apply cloisonné technique. However, our researches proved that although there were no visible fragments of partitions in between of colours (deep blue, reddish brown, black, white, and gray), there were a few small fragments of partitions measuring about 300 μm in the depth of enamel. The traces of partitions on the surface of enamels are documented as hollows separating colours, with clearly visible red contours around, which indicate that these partitions were made of copper. From the nature of the making of these hollows there are reasons to suppose that the images were created first and later on partitions were applied to contour the images and to enhance their artistic effect. Plausibly, the partitions were fixed onto the enamelling under non-sufficient temperature, and therefore they were lost later on.

The study was funded by the Russian Foundation for Basic Research (project 17–29–04129).

S8-O05.193

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Non-destructive depth profile mapping tool at the New AGLAE facility

Pacheco, C.[1,2,3], Pichon, L.[1,2], Holé, C.[1,2,3], Lemasson, Q.[1,2], Moignard, B.[1,2], Chapoulie, R. [3]

[1]C2RMF, Palais du Louvre-Porte de Lions, 14 quai François Mitterrand, 75001 Paris, France
[2]NewAGLAE, FR3506 CNRS/Ministère de la Culture et la Communication/IRCP, C2RMF Palais du Louvre, 75001 Paris, France
[3]IRAMAT-CRP2A, UMR 5060 CNRS/Université Bordeaux Montaigne, Esplanade des Antilles, 33607 Pessac, France

Keywords: ion beam analysis (IBA); New AGLAE; non-destructive; depth profile mapping tool; Rutherford back-scattering spectroscopy (RBS)

Since 1988, the AGLAE facility is devoted to Cultural Heritage. Thirty years later, after a big refurbishment, the New AGLAE facility is upgraded and automated, welcoming French and European users with archaeometric issues.
Most of the time, the study of Cultural Heritage objects does not permit sampling and implies the use of non-destructive techniques such as Ion Beam Analysis (IBA) that provides precious information on their provenance, making process or conservation state, which are essential issues for AGLAE users. RBS/EBS is of utmost interest concerning layers at the surface such as degraded or corroded surfaces, patinas, metallic leaf decorations, concentration gradients induced by a specific making process, etc.
In the frame of the New AGLAE project (grant ANR-10-EQPX-22), a multi-parameter acquisition system has been coupled to a vertical magnetic deflection of the beam and an XY stage movement allowing to map the object over area of interest of several square centimetres with a pixel resolution side size of typically 20 to 40 µm. X-rays, gamma-rays and backscattered particles are simultaneously recorded and used by a homemade software for rebuilding the matrix of any detector and/or re-bin the data with different pixel size off-line [1].
PIXE maps enable to represent the spatial repartition of the quantitative elements without information of their depth distribution. The new tool conceived and developed by the AGLAE team consists in extracting this depth quantitative information from RBS spectra contained in each pixel and in representing it with the *ad hoc* scale.
The tool and methodology will be presented on two types of decorations present on ancient ceramics: gold leaf and lustre decoration. If the former is a "simple" case - a discrete layer of metal above a glass/ceramic substrate - the second one consists in layer(s) of metallic nano-particles in a glassy matrix. This work will present the information on the making processes reached with this new tool as well as its limits and perspectives it opens to Heritage Science objects presenting layers that cannot be sampled.

Acknowledgement
The New AGLAE project is funded by the Investissement d'Avenir programme of the French ANR (ANR-10-EQPX-22), French Ministry of Culture, Paris City Council and the DIM-MAP programme of Ile-de-France region.
Reference
[1] L. Pichon*, et al.,* 2014: Nucl. Instr. and Meth. in Phys. Res. B. 318, p.27-31.

S8-O06.794

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Ceramic body of tiles characterization by means of Ion beam analytical techniques

Corregidor, V., Dias, M.I., Prudêncio, M.I., Alves, L.C.

C2TN, IST/CTN, Universidade de Lisboa, E.N. 10, 2686-953 Sacavém, Portugal

Keywords: ceramics; raw materials; firing; ionoluminescence

Tiles are composed of a ceramic body covered by a vitreous glaze layer, usually coloured. The ceramic body acts as support of the glaze and its quality is essential for the good conservation and preservation of tiles along the centuries. Some of the factors which will affect the ceramic body quality are the kiln temperature during the manufacturing process and the raw materials used. For example, underfired bodies will tend to be soft or when they are overfired ceramics will be brittle. The initial materials, as clays, will also affect the final composition of the ceramic body, with influence for instance in its hardiness or final colour.

The first uses of tiles appeared in the region of Mesopotamia, Egypt and Persia, being the beginning of an enduring tradition. From there, the tile manufacturing technology and utilization spread worldwide, usually through commercial circuits, and consequently the tile evolved and adapted to each culture and local styles. In Portugal, tiles are everywhere, they can be found for example for wall decoration in official buildings, churches or monasteries, or in fountains. They often represent scenes from the history of the country, or geometrical design.

In this work we propose the combination of non-destructive Ion Beam Analytical (IBA) techniques to assess the manufacturing conditions of ancient tiles, especially the ceramic body. The conditions to be determined are:

-the identification of the raw materials through the study of the elemental composition by means of PIXE and PIGE techniques;

- the firing temperature through the study of the compounds or particular mineral phases present in the ceramic body by means of Ionoluminescence measurements.

In this work a set of tiles with quite different chronological production (from the XVII to the XX centuries) were analysed and the obtained results will be presented and discussed. Composition results will be compared with other more stablished techniques, as for example XRD.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Applying neutron techniques to the earliest Large Hollow-cast bronze statues from Ancient Egypt

Perucchetti, L.[1], Fedrigo, A.[2], O'Flynn, D.[1], Craddock, P.T[1], Taylor, J.[1], Shearman, F.[1], Hook, D.R.[1]

[1]The British Museum
[2]ASTFC, ISIS Neutron and Muon Source

Keywords: Egyptian Statues; Large Hollow Casts; IMAT; ENGIN-X; X-Ray CT scan

In this study, we used a combination of techniques including neutron imaging and neutron diffraction, to study the details of the production of three Egyptian bronze statues, dated to the 9th-8th century BC. These statues are amongst the first examples of large statuary ever made by hollow casting techniques and understanding how they were made may help to understand the development of lost-wax technology in the ancient world. Investigating whether they were produced using the same or different procedures may suggest whether they were produced in the same or a variety of workshops, with implications for the organisation of metal object production in Ancient Egypt. Preliminary studies were undertaken using X-ray computed tomography (CT) at the British Museum, but, although an acceleration voltage of 450 kV was applied, the thickness of the metal and high lead component of the alloys restricted the penetration of the X-rays. This, in addition to the significant X-ray scatter from the statues, affected the image quality and prevented the elucidation of the internal structures of the statues. Neutron CT conducted on the IMAT instrument at the ISIS neutron and muon source (Didcot, UK) confirmed that the statues consist of several different parts, with separations observed between the main body, the arms, the head and the peruke (wig). The components were all found to be hollow and were produced using the direct lost-wax casting technique.

In addition, neutron diffraction performed using ENGIN-X at ISIS shed some light on the composition of the armatures of the statues. We found that a variety of material was used for armatures, including iron rods, leaded bronze rods and tubes, and possibly some organic material. It is also possible that some objects have been (re)used as armatures.

The details of the production techniques and the differences in the composition of the metal revealed a variety that seems more consistent with the statues being produced at different workshops. This new investigation also provided an opportunity to re-assess the extent and method of historical restorations on the statues.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

‘To be or not to be della Robbia, that is the question’: methods of analysis in art history and archaeometry in the study of 16th century Italian sculptures

Flor, P.[1], Dias, M.I.[2,3], Rodrigues, A. L.[2], Prudêncio, M.I.[2,3]

[1]Universidade Aberta / Instituto de História da Arte – NOVA/FCSH
[2]Centro de Ciências e Tecnologias Nucleares (C2TN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal
[3]Departamento de Engenharia e Ciências Nucleares (DECN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal

Keywords: Della Robbia Sculpture, Renaissance, Luminescence dating, Compositional characterization

Nowadays, in the art market, enamelled and glazed terracottas appear numerous times, in the perception of the Italian Renaissance. They are invariably ascribed to Della Robbia, a dynasty of Florentine sculptors active in Italy and France between the 15th and 16th centuries. The question is: are all these works of art due to this important workshop? We know from ancient sources that in Florence at that time several sculptors were working in terracotta, in which clay modeling, enamelled colouring and glazing gloss were the common denominator.

However, art history through its own working methodology, based on careful observation of the pieces, comparative analysis of iconographies and models, without forgetting the documentary and bibliographic research on them, has not by itself managed to answer the doubts that legitimately arise when we are dealing with such works at a scientific point of view, or even at an auction. For this reason, an archaeometric approach was applied to a large set of sculptures, particularly chemical and mineralogical characterization of both ceramic body and glaze, as well as luminescence dating.

In recent years, we have been developing as an interdisciplinary team a methodology of analysis of several pieces attributed to Della Robbia in Portugal, present in national collections, namely, National Museum of Ancient Art, National Tile Museum, Calouste Gulbenkian Museum and Berardo Museum - Quinta da Bacalhôa. Focused on around twenty pieces, the preliminary results of this important and vast research have already been published in articles and reference books. Several origins of clay used in the pieces have already been identified and they have been placed in distinct chronologies. Fortunately, some mistakes have been clarified, which the historiography of Portuguese art could not solve.

Based on two case studies in private collections of Della Robbia pieces acquired in the antiques market, we will present some historical-artistic evidences and laboratory novelties about them, as a living example of an interdisciplinary methodology that may, in the future, bear fruit in terms of education and raising society's awareness of the problems of authenticity and value of works of art.

Session 8 Technology/provenance - stone/pigments/plaster

POSTER

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Neutron tomography and diffraction, SEM, XRF and ESA: determination of the internal structure and chemical composition of a silver multiple dirham.

Khramchenkova, R.[1], Kichanov, S.[2], Sitdikov, A.[1,3]

[1]*Institute of Archaeology of Tatarstan Academy of Science*
[2]*Joint Institute for Nuclear Research*
[3]*Kazan Federal University*

Keywords: silver coin of 10th c.; internal structure; chemical composition

Medieval multiple dirhams are interesting numismatic objects: it is believed the original silver raw materials without prior enrichment were used to make them. In this regard, the chemical composition and structure of these finds is of great interest from the perspective of comparing analytical data and identifying a possible source of raw materials for the production of silver coins. The multiple dirham of Samanids dating from the first half of the 10th century is studied in the work by neutron diffraction and tomography (ND and NT), scanning electron microscopy (SEM), X-ray fluorescence (XRF) and emission spectrum (ESA) analyzes. ND and NT made it possible to "examine" the internal structure of the entire object; XRF determined the surface composition. These methods are non-destructive, and detailed studies of the physicochemical properties of ancient coins are one of the important directions in non-destructive testing of objects of cultural heritage. Neutron studies gave the phase composition and spatial distribution of chemical components in the volume of the coin. The average volumetric content of copper and silver was about 50%. XRF showed 96% silver on the surface. Micro-particle about 1-2 mm in size were broken off from the coin for SEM and ESA. SEM gave the element distribution's cartograms on the transverse kink of the coin. The composition of the metal alloy varies greatly between sites. ESA analysis was carried out for two pieces of particle and gave very different results too. Lead, antimony, bismuth, tin, arsenic and gold were identified in addition to silver and copper. The distribution of elements in the coin structure is uneven, that is evidence of poor mixing of the alloy and can indirectly confirm the hypothesis about the use of unprepared raw materials.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Development of the PATRIC14 project for the dating of low-carbon content cultural heritage materials.

Moreau, C., Thellier, B., Hain, S., Messager, C., Beck, L., Caffy, I., Delqué-Količ, E., Dumoulin, J.P., Goulas, C., Perron, M., Setti, V., Sieudat, M.

Laboratoire de mesure du Carbone 14 (LMC14), LSCE/IPSL, CEA-CNRS-UVSQ, Université Paris-Saclay, Gif sur Yvette, France

Keywords: Radiocarbon dating; Accelerator Mass Spectrometry; microsample; gas ion source; culture heritage

Carbon-14 dating is a method that allows materials containing organic carbon to be dated up to 50,000 years ago. The development of Accelerator Mass Spectrometry (AMS) dating technique has made it possible to considerably reduce the quantity of material necessary to carry out a dating. However, for archaeological works or objects low in carbon, the quantities of material to be sampled often remain too voluminous for dating to be considered. This is particularly the case for certain old ferrous objects for which previous studies have revealed very low carbon contents. This is also the case with other materials such as plaster and stucco, materials for sculptural and architectural creation, widely used since the ancient periods of Archeology and Art History. However, currently their dating is based solely on stylistic criteria, which are debated for certain corpora. The objective of the project is therefore to develop a new instrumental device which will be installed on the AMS ARTEMIS of the Laboratoire de Mesure du Carbone 14 (LMC14) located on the CEA site in Saclay (France). The aim is to develop an ion source allowing the injection of samples directly in gaseous form, avoiding the transformation into solid graphite as at present. This solution will thus make it possible to considerably reduce the quantity of carbon necessary to carry out dating by AMS. The first applications using the equipment will be carried out in collaboration with other partners in differents fields: archaeometry (LAPA), research and restoration (C2RMF) and museum (Louvre). The PATRIC14 project will make it possible to remove the technological barriers that prevent the dating of samples with very low carbon content. It allows us to glimpse new perspectives for the history of art, the history of techniques and ancient societies.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Xiongnu-period pit kilns for roof tiles production in Khustyn Bulag (Mongolia): comparison of radiocarbon and luminescence ages

Saran, T.[1], Lauer, T.[2], Ishtseren, L.[3], Solongo, S.[4], Isao, U.[5], Amartuvshin, C.[3], Lo Giudice, A.[1], Re, A.[1], Guidorzi, L.[1], Amapane, N.[1]

[1]Department of Physics, University of Torino, Torino, Italy Sezione di Torino, National Institute for Nuclear Physics (INFN), Torino, Italy
[2]Max Planck Institute for Evolutionary Anthropology, Leipzig, Germany
[3]Institute of Archaeology, Mongolian Academy of Sciences, Ulaanbaatar, Mongolia
[4]Institute of Physics and Technology, Mongolian Academy of Sciences, Ulaanbaatar, Mongolia
[5]Sapporo Gakuin University, Sapporo, Japan

Keywords: Optically Stimulated Luminescence; Thermoluminescence; Xiongnu Empire; Radiocarbon dating; Luminescence Dating

The archaeological record of ancient nomadic civilizations allows insights into the technologies, the state formation, and the emergence of nomadic pastoral tribes. The expansionistic attributes of the Xiongnu Empire (209 BC–155 AD) are evidenced by the discovery of an iron-smelting site with charcoal pit kilns at Khustyn Bulag, Tuv province, Mongolia (1). Pit kilns for pottery and roof tiles production indicate the development of nomadic settlements and towns signified by the presence of the Tereljiin fortress. The chronology of the kilns is provided by radiocarbon dating on charcoal, yielding 174-50 BC (95.4%) and 208-92 BC (86.5%). Recent research on charcoal kilns revealed that ages obtained by radiocarbon dating of randomly selected and botanically unidentified charcoal fragments are potentially affected by old-wood effect (2).
In this work, we investigate the quartz Optically Stimulated Luminescence (OSL) (3) from heated sediments associated with the rammed earth walls (4) of kilns and the fine grain Thermoluminescence (TL) (5) of the architectural roof tiles and tapestry brick found inside of the kilns. The luminescence results will be discussed and compared with the independent C14 ages obtained on charred material from the kilns.

References:

1. Sasada, T., Ishtseren, L. “Two Types of Iron Smelting Furnaces in Ancient Mongolia” Materials Science Forum Vol. 983, pp 7-13 Revised: 2019-08-15© 2020 Trans Tech Publications Ltd, Switzerland.
2. Deforce, K., Groenewoudt, B., Haneca, K., *Quaternary International* 593-594 2021. 295-305.
3. Martini, M., Sibilia, E. *Radiation Physics and Chemistry* 61 2001. 241-246
4. Solongo, S., Tengis, S. *Quaternary Geochronology* 30 2015. 18-23.
5. Guidorzi, L., Fantino, F., Durisi, E., Ferrero, M., Re, A., Vigorelli, L., Visca, L., Gulmini, M., Dughera, G., Giraudo, G., Angelici, D., Panero, E., Lo Giudice, A., *ACTA IMEKO* 10 2021. 32

S8-P05.1532

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

A non-destructive study of a silver enriched Portuguese coin using nuclear techniques

Marcucci, G.[1,2], Di Martino, D. [1], Scherillo, A. [2], Agoro, T.[2], Hillier, A.D. [2], Clemenza, M.[1]

[1]Dipartimento di Fisica "G. Occhialini", Università degli Studi di Milano Bicocca and INFN, Sezione di Milano Bicocca
[2]ISIS Neutron and Muon Source, STFC Rutherford Appleton Laboratory, Didcot, UK

Keywords: muonic X-ray Emission Spectroscopy (μXES); Neutron Resonance Capture Analysis (NRCA); Time-of-flight Neutron Diffraction (ToF-ND); X-Ray Fluorescence (XRF); Portuguese coin.

An ancient silver-copper Portuguese coin, dating to the late 18th century and with a Cu depletion on the surface, has been analysed through several non-destructive techniques.
In particular, in the framework of IAEA CRP (International Atomic Energy Agency Coordinated Research Project) F11021: feasibility assessment of nuclear analytical techniques for accurate measurements of silver artefacts composition [1], muonic X-ray Emission Spectroscopy (μXES) [2] was applied as a reference technique for non-destructive elemental measurements for Cultural Heritage or forensic reasons.
μXES works as standard X-Ray Fluorescence (XRF); however, the characteristic fluorescence of an atom is induced by a muon replacing an electron in the outer shell, resulting in the emission of high energy muonic X-rays from the bulk of the samples without photon self-absorption losses. Another unique feature of μXES is the possibility to change the depth of implantation of the negative muon by tuning the incident muon beam momentum. Hence, μXES is a valuable tool for performing non-destructive compositional analysis of a wide range of materials at different depths, up to several centimetres [3].
In this way, a depth profile analysis of the Portuguese coin has been performed, allowing the distinction between bulk and superficial compositions. In particular, the Ag/Cu ratio has been evaluated at different penetration depths up to 400 μm, investigating the thickness of the silver enriched surface.
An intercomparison between μXES and other nuclear techniques has been made. In particular, with XRF and neutron-based analysis (Neutron Resonance Capture Analysis, Instrumental Neutron Activation Analysis, and Time-of-flight Neutron Diffraction –ToF-ND), highlighting that the quantitative composition obtained through XRF is limited to the surface of the coin and therefore overestimate the Ag content. Neutron-based techniques returned information mediated over the bulk in terms of elements and phases. Muons and neutrons techniques allow us to overcome the common issue of obtaining non-destructively the composition of historical silver coins with enriched silver surface [4]
In addition, archaeometric considerations have been derived by microstructural characterization obtained through ToF-ND analysis.

[1] A. Fajgelj et al. IAEASM-344/3 (1997) [2] A.D. Hillier at al. Microchem. J. 125, 203–207 (2016)
[3] B.V. Hampshire et al. Heritage 2, 400–407 (2019) [4] J. Corsi et al. Microchem. J. 126, 501-508 (2016)

S8-P06.1573

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Non-destructive assessment of weathering granite stones in historical buildings by portable gamma-ray spectrometry

Sanjurjo-Sánchez, J.[1], Arce-Chamorro, C.[1], Alves, C.[2]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]LandS/Lab2PT - Landscape, Heritage and Territory Laboratory (FCT UID/AUR/04509/2013; FEDER COMPETE POCI-01-0145-FEDER-007528) and Earth Sciences Department/School of Sciences, University of Minho, Braga, Portugal

Keywords: GRS; gamma-ray spectrometry; weathering; stone decay; building environment

Stone decay in the built environment results of several weathering and erosive processes. It is one of the most important problems concerning the protection of Heritage buildings. Several methods have been extensively used for assessing the decay of stone in historical buildings, their causes and the result of the use of some conservative treatments. In the last years, the use of non-destructive methods has strongly increased due to the restricted sampling, but it is not always easy to assess the mechanisms and causes of decay of such stones. One of the ubiquitous problems, is the rock heterogeneity, above all in some rock types such as granite (due to previous weathering) which can explain variation in decay as well as in terms of conservation.

We propose here the use of a non-destructive technique to assess the weathering degree of granite stones: portable gamma-ray spectrometry (GRS). This technique allows a quick assessment of the radioisotope content of stone measuring a spot on the stone surface. ^{40}K, and both ^{238}U and ^{232}Th and radioisotope decay chains are present in variable concentrations in rocks. With this technique, their concentrations can be assessed in the rock, being ^{40}K directly estimated and ^{238}U and ^{232}Th indirectly from daughter radioisotopes. The method requires flat surfaces and some minimum rock thickness (25-30 cm). These requirements are usually met by stone surfaces of building walls. As chemical weathering usually causes the depletion or enrichment of some of the K, U and Th radioisotopes in rocks, and namely in granite, the measurement of such radioisotopes could be helpful to assess the weathering degree of stonewalls.

We have applied in situ GRS to assess the weathering degree of building stones of A Coruña (NW Spain). We have compared our measurements with others carried out on rock surfaces of the original quarry, located in the outskirts of the city, where several weathering degrees (from fresh to completely weathered) of the rock are observed. Comparative analyses of the radioisotope content of the quarry stone were carried out with other geochemical methods to precisely assess their content in radioisotopes, while rock surfaces were measured by in situ GRS and results compared. Afterwards, in situ GRS was used to assess and compare the weathering degree in buildings constructed with the same rock types.

S8-P08.339

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Portable Gamma-Ray spectrometry use for archaeological survey: an example of possible use

Sanjurjo-Sánchez, J.[1], Arce-Chamorro, C.[1], Alves, C.[2], Sánchez-Prado, J.C.[3], Blanco-Rotea, R.[3]

[1]University Institute of Geology, University of A Coruña, ESCI, Campus de Elviña, 15071 A Coruña, Spain
[2]LandS/Lab2PT - Landscape, Heritage and Territory Laboratory (FCT UID/AUR/04509/2013; FEDER COMPETE POCI-01-0145-FEDER-007528) and Earth Sciences Department/School of Sciences, University of Minho, Braga, Portugal;
[3]Facultade de Historia, Universidade de Santiago de Compostela, Praza da Universidade, 1, 15703, Santiago de Compostela, Spain

Keywords: GRS, gamma-ray spectrometry, geophysical survey, radioisotopes, prospection

Geophysical exploration methods are increasingly used in archaeology because the valuable data they provide to choose the best excavation locations. Among geophysical methods, the use of Portable gamma-ray spectrometry (GRS) was early suggested in the 1970s. The technique was widely used for geological purposes including mineral exploration and mapping. Few study cases have been reported but they have provided successful results. Still, the technique was not used and developed in archaeology.
The method consists on performing GRS measurements on the ground of the archaeological site of interest. The measurements are carried out in a grid of spots in a given square or rectangle of several meters in side. The measurements allows direct assessment of potassium-40 (^{40}K), and indirectly assessment of uranium-238 (^{238}U) and thorium-232 (^{232}Th) by measuring daughter radionuclides of their decay chains. These radioisotopes are ubiquitous in most rocks, sediments and soils. The method provides the concentration of such radioisotopes in the topsoil (from surface to 25-30 cm depth). As several rocks, archaeological materials, sediments and soils have variable concentrations of such radioisotopes, it is a potential method for non-destructive surveying archaeological objects, buried in the 0-25 cm of the topsoil. Such objects must contain a different concentration of radionuclides than the surrounding sediment or soil. Thus, it is potentially applicable for exploration of shallow structures or objects, providing a map of the sediment volume corresponding to such depth.
We have tested GRS in a partially excavated archaeological site of NW Spain (Cidadela, Lugo) to test the method. We have performed a GRS survey on a 10 x 7 m grid, measuring spots every meter. Remains of wall foundations made of stone have been excavated close to the explored grid, being the top of the structures at 10-30 cm depth. The stones used as building materials in the wall foundations were mostly metamorphic rocks with very low radionuclide content while the sediments contained significant amounts of K, U and Th radioisotopes. Results showed reliable results for surface exploration where shallow structures exist, despite the low radioactive content of the archaeological materials in the site.

S8-P09.340

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Studying alpha irradiation effects on quartz crystals as possible tool to unmask ceramic forgeries

Guidorzi, L.[1,2], Re, A.[1,2], Picollo, F. [1,2], Aprà, P. [1,2], Fantino. F.[3], Martire, L.[4], Artioli, G.[5], Peruzzo, L.[6], Boesso, S.[5], Rigato, V.[7], La Torre, L.[7], Lo Giudice, A.[1,2]

[1]Dipartimento di Fisica, Università degli Studi di Torino, Via Pietro Giuria 1, 10125 Torino (Italy)
[2]INFN – Sezione di Torino, Via Pietro Giuria 1, 10125 Torino (Italy)
[3]TecnArt S.r.l. – Via Modena, 58, 10153 Torino (Italy)
[4]Dipartimento di Scienze della Terra, Università degli Studi di Torino, Via Valperga Caluso 35, 10125 Torino (Italy)
[5]Dipartimento di Geoscienze, Università degli Studi di Padova, Via Giovanni Gradenigo 6, 35131 Padova (Italy)
[6]CNR, Istituto di Geoscienze e Georisorse, via Giovanni Gradenigo 6, 35131 Padova (Italy)
[7]INFN - Laboratori Nazionali di Legnaro, Viale dell'Università, 2, 35020 Legnaro, Padova (Italy)

Keywords: authentication; alpha irradiation; luminescence

The authentication of ceramic objects of artistic or archaeological interest is nowadays commonly achieved by means of the thermoluminescence (TL) technique [1]. However, one of the biggest problems about the great diffusion of TL as dating tool is that forgers have learned in time various tricks to mislead the authentication. One of the most complex is the "aging" of the object induced by artificial irradiation with X or γ rays using commercial sources, as it cannot be detected with certainty so far [2].
On the contrary α irradiation, that is present in naturally irradiated samples, is almost impossible to reproduce by counterfeiters. Hence, one possible method to determine the authenticity of a dubious relic could be based on the observation of the effect of α particles natural irradiation on the quartz grains contained in the clay matrix. An α particle is a Helium nucleus, that has a greater mass than β or γ particles and is thus capable of inducing lattice damage and differences in space charge. In geology this effect is well known as the results of constant irradiation, for millions of years, of the portion of crystal surrounding radioactive inclusions: the visible result is the formation of a luminescent halo, easily detectable with cathodoluminescence (CL) imaging [3].
Preliminary results of this study will be presented, starting from the procedures adopted for isolating quartz from the matrix without damaging the surface of the crystals (where α particles interacts with the material). Selected quartz grains from archaeological objects have been observed under CL before and after artificial He^+ irradiations; these irradiations were performed with different energies and fluences using the microbeam line of the AN2000 accelerator at INFN – LNL, in order to obtain a visual and immediate reference of the α irradiation effect on the same natural quartz. Irradiated areas have also been characterized with real-time acquisition of ionoluminescence (IL) signal and subsequent SEM-EDX and Raman spectroscopy analyses. However, the dependence of CL or SEM visible traces on the α particles fluence seems to indicate that the natural effect might be quite faint, requiring accurate experimental protocols for their identification, possibly using other analytical techniques.

[1] Aitken, M.J., *Thermoluminescence dating*, Academic Press, London, 1985.
[2] Neunteufel, R., *Terracotta artefacts: is thermoluminescence analysis sufficient for authentication?*,Mediterranean Archaeology and Archaeometry, 10(2010), 129-132.
[3] Owen, M. R., *Radiation-damage halos in quartz*, Geology, 16(1988), 529-532.

S8-P11.541

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Looking for new insights In the composition of tin based historical organ pipes by nuclear and raman techniques

Di Martino, D.[1], Merlo, C.[2], Bonizzi, C.[3], Scherillo, A.[4], Kasztovszky, Zs.[5], Harsányi, H.[5], Kovács, I.[6], Szőkefalvi-Nagy, Z.[6], Lorenzi, R.[7], Perelli Cippo, E.[8], Gorini, G.[1]

[1]Dipartimento di Fisica "G. Occhialini", University of Milano-Bicocca, Milan, Italy
[2]Laboratorio di diagnostica applicata ai Beni Culturali, Restoration School – Cr.Forma, Cremona, Italy
[3]Inzoli Cav. Pacifico & Brothers, Bonizzi F.lli Manufacturers and restorers, via Lodi 23A, Crema (CR), Italy
[4]ISIS neutron and muon source, STFC, UK
[5]Centre for Energy Research, H-1121 Konkoly Thege str. 29-33, Budapest, Hungary
[6]Wigner Research Centre for Physics, Konkoly -Thege M. út 29-33, 1121 Budapest, Hungary
[7]Dipartimento di Scienza dei Materiali, University of Milano-Bicocca, Milan, Italy
[8]Institute for Plasma Science and Technology (ISTP), CNR, Milan, Italy

Keywords: tin plague; nuclear and neutron-based techniques, Raman spectroscopy

It was a common knowledge in Middle Age that tin-based pipe organs of many important churches were subject to a progressive decay due to what was called *tin pest* or *plague* [1]. This alteration started with the presence of whitish, opaque spots on the once shining polished metal, and often resulted in deformation and even rupture. In modern times it was found that the cause of tin plague is an allotropic transformation of tin, spontaneously occurring at temperatures of about 13 °C. The transformation is strongly accelerated by lower temperatures, highly humid atmosphere or high concentration of halide, and delayed by the addition of Pb, Sb or other metals to the cast.

Due to the paramount relevance of pipe organs in European musical tradition, the importance of restoration and early non-invasive diagnosis of the degradation process of metal pipes is great.

During the last 4 years, a project of combined nuclear- and Raman spectroscopy studies is running to clarify the degradation process and help restorers in conservation practices.

We previously performed neutron studies on a single pipe fragment [2], and other studies have been carried out on organ pipes alterations in the literature, but many questions are still debated [3]. In particular, the characterization of the metal alloy, which was used both for the pipe and for the soldering is essential for the conservation of historical pipes and for chronological criteria too.

We could then analyse the composition of a larger set of historical organ pipes, in a non-destructive way, by neutron techniques (like neutron diffraction and prompt gamma activation analysis -PGAA), particle induced x-ray emission (PIXE), as well as by Raman spectroscopy, having access to a set of specimens and full pipes coming from the restoration of several organs in Italy, in a wide period ranging from the 17th to the 20th century. We will report the present status of conservation, how the restoration was performed in the past and hints for future preservation of tin based organ pipes.

[1] C.E. Homer and H.C. Watkins, Transformation of tin at low temperatures, The Metal Industry, (1942) LX 22 London.
[2] D. Di Martino, et al., A multidisciplinary non-destructive study of historical pipe organ fragments, Materials Characterization 148 (2019) 317-322.
[3] C. Chiavari, et al., Deterioration of tin-rich organ pipes, J. Mater Sci. 41 (2006) 1819–1826.

S8-P13.562

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Studying opacifiers with non-destructive techniques for manufacturing and provenance analysis of glass mosaic tesserae

Marcucci, G.[1], Lorenzi, R.[2], Scherillo, A.[3], Cazzaniga, C.[3], Fedrigo, A.[3], Raspino D.[3], Clemenza, M. [1], Di Martino, D.[1]

[1]Milano Bicocca University, Physics Department, Italy
[2] Milano Bicocca University, Material Science Department, Italy
[3]ISIS neutron and muon source, STFC, UK

Keywords: glass; neutron; activation; transmission; Raman

A set of glass mosaic tesserae, dating from the 2nd to the 11th century AD and coming from different geographical areas (Greece, Italy and Syria), has been investigated in order to trace back the composition in terms of crystalline phases and elements and the manufacturing technology.
Glass tesserae are composed of an amorphous matrix with the addition of various raw materials, generally minerals, with different functions like colorants and/or opacifiers.
The process of colouring glass tesserae is a demanding task, peculiar of the geographical area and historical period.
One way forward in exploring the manufacturing technique employed to produce the tesserae is through the analysis of the opicifiers composition. The goal of this study was the application of non-destructive analytical techniques to obtain a qualitative and quantitative description of the mosaic tesserae composition. At first, micro-Raman spectroscopy has been applied, which allowed for qualitative identification of the mineralogical phases acting as opacifiers. The type of opacifier should match the specimen production period and therefore gives us insight on its dating. A quantitative investigation of the bulk elemental and isotopicalcomposition was then provided by Neutron Activation Analysis, performed at the ISIS Neutron and Muon Source (UK).
Since glass mosaic tesserae are heterogeneous artefacts, a further technique has been applied: Neutron Resonance Transmission Imaging (NRTI) after implementation of the experimental set-up on the INES beamline at ISIS by introducing a time and space-resolved detector based on nGEM technology. NRTI relies on neutron time-of-flight method to reconstruct a 2D map that allows for the visualization of the isotopes distribution within the samples. The combined use of different non-destructive techniques returns an in-depth characterization of the mosaic tesserae.

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Investigation of Cultural Heritage objects by scanning XRF

Asvestas, A.[1], Mastrotheodoros, G.P.[2,3], Tzima, A.[1], Filippaki, E.[3], Beltsios, K.G.[4], Anagnostopoulos, D.F.[1]

[1]Department of Materials Science and Engineering, University of Ioannina, Ioannina, Greece.
[2]Conservation of Antiquities & Works of Art Department, West Attika University, Aegaleo, Greece.
[3]Institute of Nanoscience and Nanotechnology, NCSR "Demokritos", Agia Paraskevi, Greece.
[4]School of Chemical Engineering, National Technical University of Athens, Zografou, Greece.

Keywords: elemental distribution, depth profile

In the present work, a micro-XRF spectrometer is used for the study of the elemental distribution in cultural heritage objects, like paintings, manuscripts, and metallic items. The measurements are performed in-laboratory using a micro-XRF spectrometer equipped with a programmable motorized x-y-z sample stage, allowing 3D scanning. Target movement in the x-y direction (target's surface plane) enables point, line and area measurements of XRF spectra, while proper analysis of spectra offers handy elemental distribution visualization. Measurement of the XRF spectra as a function of the target movement in the z-direction (perpendicular to the target's surface) allows the differentiation between bulk and layered in-depth structures. Variable size beam slits, mounted along the X-ray path, limit the beam spot size at the sample side and contribute to the definition of the spatial resolution determination

The contribution of set-up parameters (e.g. beam spot size, step size, target holder, target orientation) to the quality of the measured spectra and on the derived results are discussed as a function of the object characteristics. The complementarity between the micro-XRF measured spectra with spectra acquired using a portable handheld XRF is discussed. The spectral analysis and the elemental distribution visualization is performed using the free open source PyMca code [1], while the results are interpreted applying the XMI-MSIM Monte-Carlo simulation code [2].

[1] V.A. Solé, E. Papillon, M. Cotte, P. Walter, J. Susini, Spectrochim. Acta Part B At. Spectrosc. 62 (2007) 63–68.
[2] T. Schoonjans, V.A. Solé, L. Vincze, M. Sanchez del Rio, K. Appel, C. Ferrero, Spectrochim. Acta Part B At. Spectrosc. 82 (2013) 36–41

S8-P20.860

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Archaeometric approach to the study of Phoenician pottery production in Santa Olaia (Figueira da Foz, Portugal)

Almeida, S.[1], Prudêncio, M.I.[2], Marques, R.[2], Dias, M.I.[2], Russo, D.[2], Vilaça, R.[3]

[1]FCT Scholarship - Foundation for Science and Technology (SFRH / BD / 129227/2017).CEAUCP - Center for Studies in Archeology, Arts and Heritage Sciences, Portugal
[2]Centro de Ciências e Tecnologias Nucleares (C2TN), Departamento de Engenharia e Ciências Nucleares (DECN), Instituto Superior Técnico, Universidade de Lisboa, EN 10 (km 139.7), 2695-066 Bobadela, Portugal
[3] Institute of Archeology. Department of History, European Studies, Archeology and Arts, Faculty of Arts, University of Coimbra. CEAACP - Center for Studies in Archeology, Arts and Heritage Sciences; Sub-Ripas Street, 3000-395 Coimbra, Portugal.

Keywords: Neutron activation analysis (NAA); Phoenician pottery; Ceramic Technology; Santa Olaia

Santa Olaia is the northernmost Phoenician establishment in the Atlantic coast of the Portuguese territory. The archeological site, presently object of research within a broader PhD project, is an unavoidable reference in the studies concerning the Mediterranean influences (Phoenician and Punic) in the Iberian Peninsula.
Usually associated with the metallurgical activity, the site would have welcomed other productive sectors, of which the pottery production stands out. The identification of a potter's neighborhood suggests the importance of this activity in the local economy. In this work, a compositional approach of ceramics (29 samples) was used to better understand this phenomenon. The strategy assumed consisted on the analysis of a set of ceramics of known wares (except black slip ware) recovered on the site, in layers covering a time span from the 6th to 4th cent. BC. Chemical contents were obtained by neutron activation analysis (NAA), and used as variables in a multivariate statistical analysis. A comparative study with data previously obtained by Prudêncio (1987) including local and regional clay deposits, was done.
The results obtained point to three main groups: (i) two fragments produced with marine clays and similar to ceramics previously identified in the near site of Tavarede (Ce negative anomaly); (ii) one group with a continental clay chemical pattern and similar to ceramics previously studied by Prudêncio, 1987; and (iii) a "calcitic" production with high contents of Ca, Sr and V in agreement with high amounts of calcite.
The outcome highlights an intensive and long-lasting influence of Phoenician pottery technology, thus incorporated into the local productions, mixed with traces of other cultural inspirations. The identification of this tendency lines up with the present-day results obtained in other sites linked to the orientalising world and represents a paradigm revolution regarding the study of ceramics in this emblematic establishment.

Prudêncio, M.I. (1987) "Contribuição para o estudo da proveniência das argilas usadas no fabrico da cerâmica cinzenta fina local e regional de Conímbriga". Dissertação, provas de acesso a Inv. Auxiliar, INETI, 133pp. (policopiado).

S8-P21.3000

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Nuclear analytical techniques applied to the study of ivory artefacts

Rodrigues, A.L.[1], Dias, M.I.[1,2], Prudêncio, M.I.[1,2], Marques, R. [1,2], Valera, A.C.[3,4], Kasztovszky, Z.[5], Harsányi, I.[5], Szilágyi, V. [5], Kovácsc, I.[6], Szőkefalvi-Nagyc, Z.[6]

[1]Centro de Ciências e Tecnologias Nucleares (C2TN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal
[2]Departamento de Engenharia e Ciências Nucleares (DECN), Instituto Superior Técnico, Universidade de Lisboa, E.N. 10 (km 139,7), 2695-066 Bobadela, Portugal
[3]ERA Arqueologia S.A., Calçada de Santa Catarina, 9C, 1495-705 Cruz Quebrada – Dafundo, Portugal
[4]Interdisciplinary Center for Archaeology and Evolution of Human Behavior (ICArHEB), Universidade do Algarve, Campo de Gambelas, Faro, Portugal
[5]Nuclear Analysis and Radiography Department, Centre for Energy Research, 29-33 Konkoly-Thege street, 1121 Budapest, Hungary
[6]Institute for Particle and Nuclear Physics, Wigner Research Centre for Physics, Hungarian Academy of Sciences, Budapest, Hungary

Keywords: Ivory; Chalcolithic enclosure; PGAA; PIXE; Chemical composition

For millennia, several cultures have carved ivory into items both decorative, utilitarian and as sacred objects. This exotic raw material usually plays a relevant social role in pre-historical contexts, being crucial its correct identification and provenance. Non-invasive nuclear analytical techniques (NAT) have already been applied to the study of ivory objects, but usually to differentiate ivory from other osseous materials. Other compositional approaches have also been done, but mainly relating the extent and nature of degradation with depositional environment as a tool to an appropriate conservation treatment. Only a few refers to the use of trace elements as indicators of ivory provenance. Due to the historical and cultural value of ivory artefacts, only non-destructive techniques can be used for compositional studies. Thus, this work proposes an alternative approach to the chemical composition study of ivory archaeological objects, complementing prompt-gamma activation analysis (PGAA) with external milli-beam particle induced X-ray emission spectroscopy (PIXE). This approach enables to determine the bulk concentration of the analysed artefacts by using PGAA to quantify major, minor and trace elements. In addition, PIXE measurements analyze the chemical composition of the artefacts' surface. Concerning the chemical elements obtained by PGAA, as expected, P is the element with major concentrations (58 - 90 wt%), and Ca the second most abundant element (28 - 45 wt%). In addition, C, H, Mg, Si, Na and Cl are also present in all samples. Ti, Al, Fe, Mn and K were also detected in trace amounts in most of the samples. Besides confirming the chemical composition obtained by PGAA, dominated by the presence of P and Ca oxides, PIXE analyses enhance the enrichment in Si, Fe and other first row transition elements, pointing to a contamination of soils particles in the ivory artefacts surface. The CaO/MgO obtained ranges around 0.08 reinforcing the ivory nature of material, instead of bone or antler. The CaO/ P_2O_5 and MgO/P_2O_5 ratios have been used to evaluate and discuss the preservation state of these ivory items and its relationship with burial conditions. The application of the proposed combined nuclear analytical techniques to the study of archaeological ivory material clearly contributes to characterize, identify and evaluate alteration rates, as well as to develop criteria for the chemical examination of artifacts of doubtful nature and provenance.
IPERION CH Project nr. 654028 supported this studied (VISUAL proposal number BRR_563, 2018)

S8-P23.3005

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Applying neutron techniques to the earliest large hollow-cast bronze statues from Ancient Egypt

Perucchetti, L.[1], Fedrigo, A.[2], O'Flynn, D.[1], Craddock, P.T.[1], Taylor, J.[1], Shearman, F.[1], Hook, D.R.[1]

[1]The British Museum
[2]ASTFC, ISIS Neutron and Muon Source

Keywords: Egyptian Statues; Large Hollow Casts; IMAT; ENGIN-X; X-Ray CT scan

In this study, we used a combination of techniques including neutron imaging and neutron diffraction, to study the details of the production of three Egyptian bronze statues, dated to the 9th-8th century BC. These statues are amongst the first examples of large statuary ever made by hollow casting techniques and understanding how they were made may help to understand the development of lost-wax technology in the ancient world. Investigating whether they were produced using the same or different procedures may suggest whether they were produced in the same or a variety of workshops, with implications for the organisation of metal object production in Ancient Egypt. Preliminary studies were undertaken using X-ray computed tomography (CT) at the British Museum, but, although an acceleration voltage of 450 kV was applied, the thickness of the metal and high lead component of the alloys restricted the penetration of the X-rays. This, in addition to the significant X-ray scatter from the statues, affected the image quality and prevented the elucidation of the internal structures of the statues. Neutron CT conducted on the IMAT instrument at the ISIS neutron and muon source (Didcot, UK) confirmed that the statues consist of several different parts, with separations observed between the main body, the arms, the head and the peruke (wig). The components were all found to be hollow and were produced using the direct lost-wax casting technique.

In addition, neutron diffraction performed using ENGIN-X at ISIS shed some light on the composition of the armatures of the statues. We found that a variety of material was used for armatures, including iron rods, leaded bronze rods and tubes, and possibly some organic material. It is also possible that some objects have been (re)used as armatures.

The details of the production techniques and the differences in the composition of the metal revealed a variety that seems more consistent with the statues being produced at different workshops. This new investigation also provided an opportunity to re-assess the extent and method of historical restorations on the statues.

S8-P24.330

ISA 2020/2022

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

New set-up for Neutron Resonance Capture Analysis (NRCA) and Neutron Resonance Transmission Imaging (NRTI) for cultural heritage at the Italian Neutron Experimental Station INES at the ISIS neutron and muon source

Scherillo, A.[1], Fedrigo, A.[1], Grazzi, F.[2], Raspino, D.[1]

[1]ISIS neutron and muon source, STFC, United Kingdom
[2]Istituto di Fisica Applicata Nello Carrara, CNR, Italy

Keywords: neutron, activation, transmission, imaging

The Italian Neutron Experimental Station INES, located at the pulsed neutron source ISIS (U.K.), is a general purpose neutron diffractometer that was built to focus its use on cultural heritage related studies. Thanks to the high penetration power of neutrons, archaeometric measurements performed through neutron diffraction allow us to determine bulk properties of the sample in a non destructive way, in particular regarding phase analysis and microstructure. This opens up the possibility of scientific investigation on objects otherwise unsuitable, due to their cultural and/or historical importance.
Complementary to the determination of the type and amount of the crystalline phases present in the samples, neutrons can provide elemental and isotope analysis, if neutrons of energy in the eV range are available. In particular Neutron Resonance Capture Analysis (NRCA) and Neutron Resonance Transmission Imaging (NRTI) can be exploited to determine, non destructively, the type and spacial distribution of elements, and in some cases isotopes, in the artefact under investigation.
A few years ago the suitability of the INES beam line for NRCA measurements was assessed using a compact γ detector made of a Yttrium Aluminum Perovskite (YAP) scintillation crystal coupled with a silicon photomultiplier (SiPM) readout. The current NRCA set up has been improved implementing a larger angular coverage. Recently, a new detector based on GEM technology has been used for transmission measurements, to obtain a bidimensional map of elements for calibration standards and archaeological artefacts. As an example of the potential of the combined approach of diffraction and elemental analysis, we present in this work the preliminary results obtained on two Chinese bimetallic sword fragments from 2nd 1st century BCE. In particular, NRTI provided a 2D map of the elemental composition of the artefacts, indicating the nature of the bronze alloy of the grip and of the iron blade. The study presented was complemented by Neutron Diffraction, Neutron Resonance Capture Analysis (NRCA), and Neutron Imaging, providing a full characterisation of the objects in terms of composition and microstructure in a non destructive way.

S8-P25.626

International Symposium on Archaeometry

Lisbon, 16th – 20th May 2022

Multi-analytical approach for the characterisation of decoration and glazing materials of late 19th-early 20th century French ceramics

Mazzinghi, A.[1,2], Mangani S.M.E.[2], Chiari M. [1], Legnaioli S. [3], Mandò P.A. [1,2]

[1]INFN/CHNet, Florence Division, Via G. Sansone 1, 50019 Sesto Fiorentino, Italy
[2]Università degli Studi di Firenze, Dept. of Physics and Astronomy, Via G. Sansone 1, 50019 Sesto Fiorentino, Italy
[3] ICCOM-CNR, 56124 Pisa, Italy

Keywords: IBA; MA-XRF; ceramic; porcelain; France

This study is aimed at obtaining a general view on glazing and decoration materials used in French ceramics around the end of the 19th century and the beginning of the 20th century. The great success of ceramic, and porcelain in particular, during this period led to a general modernization of the production processes, from a hand-made, craft-based system to an industrial one, more suitable to the increasing trades demand. This upgrade is evident not only in the new, industrialized, production processes but also in the materials, making a wider range of available colours (either pigments or colorants) available with different and new formulations. To study in depth these materials, twenty-one samples from six different French factories underwent a comprehensive technical characterisation by means of fully non-invasive approach. The non-invasive and not-deliberatively destructive protocol involved using nuclear techniques as Ion Beam Analysis (IBA), i.e PIXE, PIGE and EBS. These measurements were carried out at the LABEC laboratory in Florence, exploiting a Tandem Accelerator and an external beam set-up, and were aimed at quantitative analysis of the glazes, the detection of low-Z elements (such as Li and B) and for the stratigraphy of decoration layers and metals. Complementarily, the CHNet Macro X-Ray Fluorescence (MA-XRF) scanner was employed for the analysis distribution of the materials on the surfaces. Finally, Raman spectroscopy was employed to better characterise some of the compounds.
Briefly summarizing the results, two different types of glazing have been distinguished, likely related to the different kind of core material (faiance and porcelaine). Regarding the decorations, Co, Fe, Sn/Au and Cr are related to blue, red, pink and green colours (respectively), while browns, purples, greys and blacks were made of complex mixtures or spinels containing various chromophores. A wider variability has been detected on yellows instead and the use of the following materials has been hypothesized: Basic Zinc Yellow or Zinc and Iron Oxide mix; Titanium Yellow or mixture of Iron Oxides / Hydroxides with TiO_2; Naples Yellow and Lead, Antimony and Tin Yellow.
This study is an interesting example on how IBA can be decisive for a comprehensive analysis of the materials and shows how complementary methods, as MA-XRF, can make great advantage of this holistic approach.

S8-P26.773