

CHEMICAL AND MINERALOGICAL ANALYSIS OF TENAX USED AS A METALWORKING AUXILIARY MATERIAL IN ARTEFACTS FROM THE EARLY AVAR-PERIOD CEMETERY OF MAKÓ–MIKÓCSA-HALOM

ÖTVÖSTECHNIKAI SEGÉDANYAGOK (TENAX) KÉMIAI ÉS ÁSVÁNYTANI ÖSSZETÉTEL VIZSGÁLATA A MAKÓ–MIKÓCSA HALOMI KORA AVAR KORI TEMETŐBŐL*

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Abstract

In this study, we present the composition of tenax used as an adhesive and as a reinforcing or filling material, identified on two unique artefacts recovered from the Early Avar-period cemetery of Makó–Mikócsa-halom: a circular pendant forming part of a necklace and a belt mount. Based on our findings the main organic component was wax, similarly that recorded in the literature. The inorganic phase was somewhat different coming from sand (quartz, muscovite) and Mg-bearing calcite.

Kivonat

Ebben a tanulmányban a Makó–Mikócsa-halom kora avar kori temetőből előkerült leletek közül származó két egyedi tárgy, egy nyakék részét képező kerek csüngőből és egy övveretből származó, ragasztó és szilárdító vagy töltőanyagként használt tenax összetételéről számolunk be. Eredményeink alapján a fő szerves komponens a szakirodalomban rögzítetthez hasonlóan a viasz volt. A szervetlen fázis némileg eltérő volt, amely homokból (kvarc, muszkovit) és Mg-tartalmú kalcitból származott.

KEYWORDS: TENAX, CHEMICAL AND MINERALOGICAL COMPOSITION, EARLY AVAR AGE

KULCSSZAVAK: TENAX, KÉMIAI ÉS ÁSVÁNYOS ÖSSZETÉTEL, KORA AVAR KOR

Introduction

The design of the form and ornamentation of each individual piece of jewellery and dress accessories are primarily guided by artistic intent (Horváth, 2008, 2013; Riegl, 1989), regardless of the raw materials and the production technology. However, to gain a more detailed understanding of the methods, techniques, tools and circumstances of the objects' production, it is necessary to know the composition, geological deposits and value of the raw materials used, in addition to the typological analysis of the objects.

The pressing technique of Byzantine origin, employed in metalworking, became widespread in the Avar-period material of the Carpathian Basin (second half of the 6th century–early 9th century CE) from around the turn of the 6th and 7th centuries onward, and remained a dominant manufacturing technology until the end of the 7th century. The overwhelming majority of belt and horse-harness fittings, as well as jewellery, were produced using this technique.

* How to cite this paper: GULYÁS, S.; BALOGH, Cs. & FINTOR, K., (2026): Chemical and mineralogical analysis of tenax used as a metalworking auxiliary material in artefacts from the Early Avar-period cemetery of Makó–Mikócsa-halom, *Archeometriai Műhely* XXIII/1 25–36.
doi: [10.55023/issn.1786-271X.2026-002](https://doi.org/10.55023/issn.1786-271X.2026-002)

Our understanding of their manufacture is primarily based on the known goldsmiths' graves and assemblages containing metalworking tools (Fettich 1926; Csallány 1933; Balogh 2004, 266–267; Rác 2014; Ny. Kovacsóczy et al. 2022, etc.). Alongside the tools and pressing dies employed in production, however, the raw materials and the so-called auxiliary materials used in the manufacture of these objects may also provide important information regarding the technologies applied within the workshops, as well as the connections maintained between different workshops. One of the most used auxiliary materials in the Migration Period was putty or tenax, which was used to consolidate or fill in the settings. According to the literature, it was in use from antiquity (Hawthorne & Smith 1963; Brepohl 1987; Presbyter, 1986; Borbíró, 1996; Ogden 1982). It appears from the Hunnic period (5th century AD) and by the Merovingian period, dating from the first half of the 6th century, its use had become common practice (Horváth 2008, 2013; Horváth et al. 2024). It was also a popular filler material in the Avar period, used for stone and glass enclosures and as a filler and stiffener between metal sheets (Heinrich-Tamáška 2016). In the 12th century, Theophilus Presbyter, in his work on various works of art, mentioned a material called tenax. He suggested the use of tenax as a filling and stiffening agent for decorating thin-walled metal objects such as gold or silver cups and bowls. He described exactly the composition of the material and the technique for making it: „Grind a piece of brick or tile very small and melt some pitch in an earthenware dish and add a little wax. When these are both melted, mix in the powdered tile and stir it vigorously and pour it out into water. When it begins to grow cold, dip both your hands into the water and knead it for a long time until you can stretch the composition and draw it out like a skin.” (Hawthorne & Smith 1963, 129–130; Brepohl 1987). However, the composition of the tenax, consisting of crushed minerals and added organic binders, may have differed from age to age and workshop to workshop from what Presbyter describes. A reliable qualitative and semi-quantitative assessment of the compositional differences requires additional chemical analytical analysis of the putty mass beyond macroscopic and microscopic examination.

However, the composition of the Avar Age tenax was markedly different from that described by Presbyter, based on the results of a complex chemical analysis of the putty material of Avar potsherds available in the literature. X-ray diffraction analysis was generally used to measure the inorganic components, while infrared spectroscopic analysis was used to determine the organic components in the analysis of samples from Hungary from the 5th to the 8th centuries AD

(Arrhenius 1984, 1985; Daim 2002; Heinrich-Tamáška, 2006, 2016; Horváth et al. 2009, 2024).

The results of one of the most detailed chemical composition studies of the Avar filling masses can be found in Heinrich-Tamáška (2006). Based on individual sample analyses, the main organic component of the tenax was wax in most cases. The FTIR measurements were able to confirm the use of beeswax. Apart from wax the use of gypsum, quartz and lime was also common (Heinrich-Tamáška 2006, 2016; Arrhenius, 1985; Horváth et al. 2009). Beeswax was also used as a filler in a Keszthely-Dobogó disc fibula, according to Daim (2002). Birgit Arrhenius (1985) also recorded the use of wax in samples from Hungarian finds from the 5th and 6th centuries. Although at the present stage of research it is not possible to draw far-reaching conclusions about the use of tenaxes of different compositions, the continuity of the use of wax in the Carpathian Basin is already apparent (Heinrich-Tamáška 2006, 2016; Arrhenius, 1984, 1985; Horváth et al. 2009, 2024). For cloisonné goldsmith works the use of filling and firming substance called tenax composed of pulverized minerals and an organic binder was also common in the 5–6th centuries AD (Horváth et al. 2009). The tenax from the gold coated bronze pressed rosette of a horse-harness fitting from the cemetery of Komárom-Hajógyár dated to the last third of the 7th century AD was composed of wax mixed with millet seeds (Trugly 2008). In many cases, the finds include lead-containing paste e.g., from the Late Avar period at Zamárdi (Bárdos & Garam 2009), porous materials such as asphalt and clay (Kassa-Zsebes, Budinský-Krička & Točík 1984) and some kind of silicate-based tenax mixture (Pitvaros-Víztaózó, Bende 1998).

In this work we aim to show the results of chemical compositional analyses of Early Avar Age tenax recovered from two finds of the cemetery of Makó–Mikócsa-halom.

A brief overview of the archaeological material

In 2009–2010, 251 graves belonging to an Avar-period cemetery were excavated near Makó in south-eastern Hungary (Fig. 1). The cemetery may be regarded as fully excavated, and according to the preliminary analyses, its period of use can most likely be dated from the end of the 6th century to the middle of the 7th century AD (Balogh 2017; Balogh 2020).

Grave 33 was situated in the central part of the cemetery and contained the burial of a woman who died between approximately 23 and 35 years of age.

The grave assemblage included a partial skeleton of a 3.5-year-old cow, a partial skeleton of a newborn calf, a partial skeleton of an approximately 12-year-

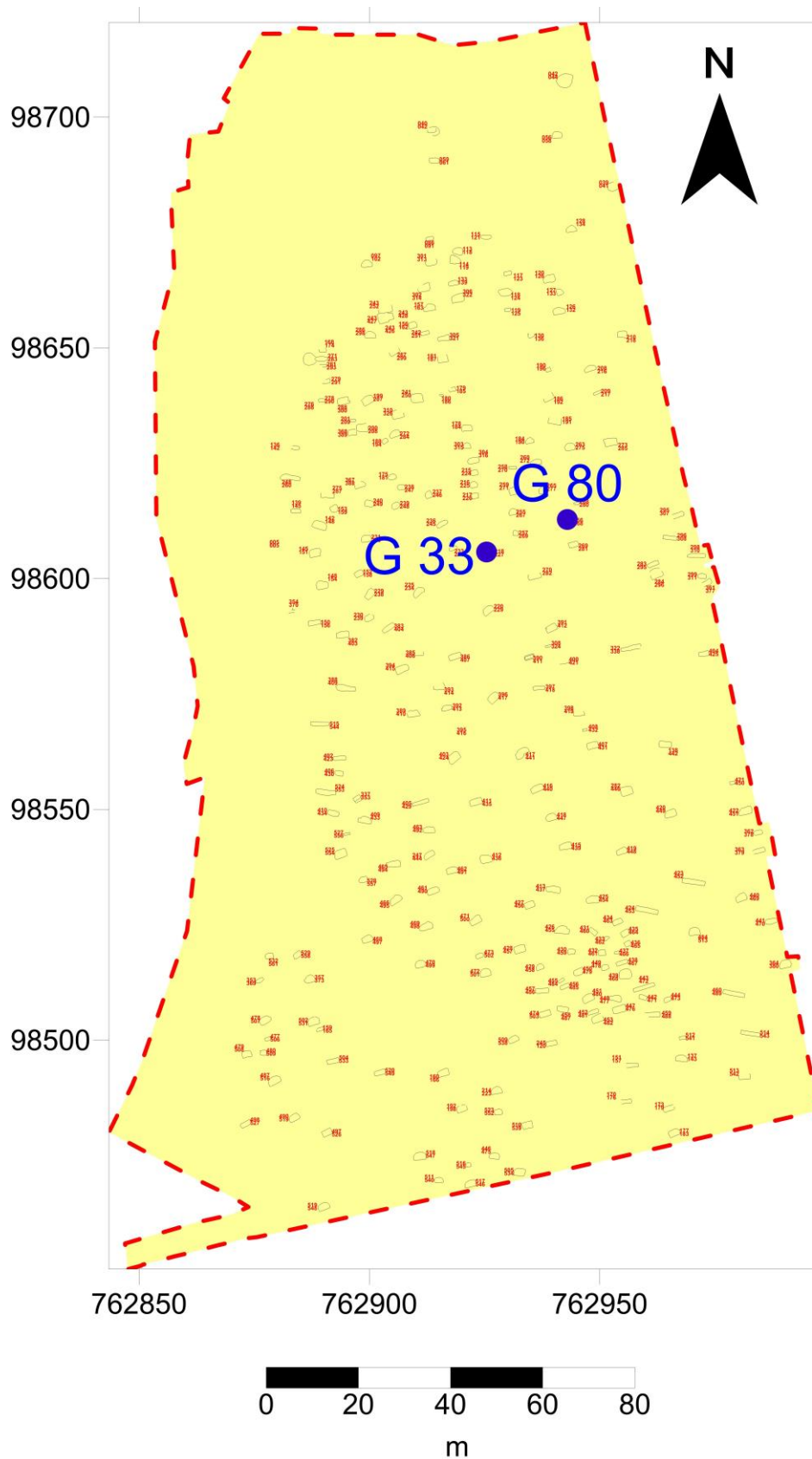


Fig. 1.: General plan of the Early Avar-period cemetery of Makó–Mikócsa-halom with the mentioned graves marked

1. ábra: A Makó–Mikócsa-halomi kora avar kori temető összesítő térképe az említett sírokkal



Fig. 2.: Deszk-type necklace with sheet-metal pendants from Grave 33

2. ábra: A 33. sír Deszk-típusú lemezcsüngős nyakéke

old sheep, as well as five additional cattle limbs. Among the grave goods were a pair of bronze earrings with large globular sheet-metal pendants, a necklace composed of colored beads, a Byzantine gold cross (Balogh 2018; Gulyás et al. 2018), a cast bronze bracelet with funnel-shaped terminals, an iron buckle, a spindle whorl, and an iron awl. From the chest area of the deceased, a remarkable necklace was recovered (Balogh 2018, 25).

The necklace was composed of three circular pendants – one larger and two smaller examples – combined with thin bronze tubes (Figs. 2–3). All three discs were manufactured using the same technique. Their back plates were cut from thick bronze sheet, while the front plates consisted of gilded silver sheet decorated with pressed concentric motifs. An oval aperture was created at the center of each front plate, into which a convex pale-green glass inlay was inserted from behind.



Fig. 3.: Reconstruction of the manner of wearing the necklace (Balogh 2019)

3. ábra: A nyakék viseleti rekonstrukciója (Balogh 2019)

The suspension loops and the pendant ornaments attached to the lower edges of the discs were soldered onto the back plates. Tenax adhesive was employed both for fixing the glass inlays to the back plates and for joining the two plates together along their edges. Inv. No.: Móra Ferenc Museum, 2012.9.249.

Similar necklaces with sheet-metal pendants occur in the archaeological material of the Avar Khaganate exclusively in the Transtisza region, within cemeteries situated along the Maros River, more precisely in the vicinity of Makó and Deszk. Parallels are known from Grave 31 of the Deszk-G cemetery and Grave 5 of the Deszk-L cemetery (Garam 2001, Taf. 16.4; Taf. 17.1–2), while an additional unpublished example derives from Grave 214 of the Makó–Mikócsa-halom cemetery. A similar pressed disc with two crescent-shaped pendant ornaments was strung onto a necklace composed of coloured beads in Grave 16 of the Deszk H cemetery (Garam 2001, Taf. 16.4). It cannot be determined with certainty whether the fragmentary circular pendant recovered from Grave 60 at Mokrin–Vodoplav důl likewise belonged to a necklace composed of tubes and discs similar to

the examples discussed above, or whether it originally functioned as an isolated circular pendant (Ranisavlev 2007, T. XXI. 60:7)

These necklaces imitate Late Antique necklaces with circular medallion pendants and were produced after their prototypes. Their geographically concentrated distribution, together with their high degree of similarity, suggests that they may have been manufactured by a local goldsmith active in this region, in the vicinity of the Maros River (Balogh 2019).

On the basis of the findspots of the first known examples, these ornaments — designated in the scholarship as “Deszk-type” necklaces — can most likely be dated to the end of the 6th century and the opening decades of the 7th century (Balogh 2018).

Grave 80 was excavated at the eastern edge of the same cemetery. A set of silver mounts (**Fig. 4.**) exceptional within the archaeological material of the Avar Khaganate was recovered from the grave. The grave contained the burial of a man who died between 35 and 39 years of age; beside him were placed a 12–14-year-old horse with harness equipment and three partial sheep skeletons. The deceased was interred with a bow, sword, and quiver, while the silver mounts decorated his belt.

The belt set comprised an iron buckle, three shield-shaped and three double shield-shaped mounts, one shield-shaped mount of differing form, five small strap-ends, and one large strap-end. Loop attachments were soldered to the reverse sides of the shield-shaped and double shield-shaped mounts, perpendicular to the longitudinal axis of the fittings. The reverse sides were then filled with tenax adhesive, which reinforced the thin and fragile mounts. The tenax from one of the shield-shaped mounts was subjected to analysis. Inv. No.: Móra Ferenc Museum, 2012.9.638.

The only known parallel within the Carpathian Basin for the shield-shaped and double shield-shaped mounts of this belt set is the fragmentary fitting from Grave 211 at Csákberény–Orondpuszta (László 2015, Taf. 18. 211:1). Outside the Carpathian Basin, close analogies are known from Graves 90 and 119 of the Castel Trosino Longobard cemeteries in northern Italy (Paroli & Ricci 2007, T. 67. 12a–g, T. 100. 30b–l). As in the case of the Makó mounts, loop attachments were soldered onto their reverse sides; however, no tenax is visible within the Italian examples (cf. Paroli & Ricci 2007, T. 152.h–d).

In the case of pressed belt mounts from the Avar Khaganate, fastening was most commonly achieved



Fig. 4: Belt fitting from Grave 80

4. ábra: A 80. sír övveretei

either by rivets or by loop attachments fixed into the tenax filling. The soldered loop attachment employed on the Makó mounts, by contrast, represents a characteristic technical solution of Byzantine–Mediterranean metal fittings. On the basis of this feature, as well as the North Italian analogies of the mounts, we assume that these objects were not produced within the Carpathian Basin but rather constitute Italo-Byzantine products.

According to the radiocarbon date obtained from the anthropological material, Grave 80 at Makó was most likely constructed during the first half of the 7th century.

Taking into consideration both the age of the deceased and the nearly unused condition of the mounts, the fittings themselves were probably manufactured during the opening decades of the 7th century CE.

Material and methods

Sampling was made from the bottom of the sheets of the clasp and the belt fitting. Raman confocal spectrometry was used for the determination of the mineralogical structure of the inorganic components. A Thermo Fisher Scientific DXR confocal Raman spectrometer with an Olympus BX41 microscope using 50X objective lens (NA: 0.75) and Peltier cooling CCD detector was used to obtain the Raman spectra. The measurements were performed using a 532 nm wavelength Nd-YAG solid state laser and a 900 lines/mm optical grating, with a spectral resolution of 3–4 cm^{-1} . The confocal aperture was set to a 50 μm slit, and the laser power was varied between 1 and 5 mW to avoid photodegradation.

X-ray powder diffraction analysis (XRD) was used for determining the mineralogical composition of the samples besides crystalline or amorphous structure. The sample under study was ground in an agate mortar and pestle. The instrument used was a Shimadzu XRD-6000 X-ray diffractometer using a Cu X-ray tube (I: 1.5405 Å) with 40 kV, 30 mA Cu $K\alpha$ radiation. Scanning was carried out for 2θ in the range 0–75°.

Results

Figs. 5 and 6 display the Raman spectra of two tenax samples from the cemetery of Makó–Mikócsa-halom. The first four distinct bands in the wavenumber region of 100 and 1100 cm^{-1} are associated with the carbonate ion (Gunasekaran et al. 2006). These are the ν_1 symmetric stretching vibration (1087 cm^{-1}); the ν_4 symmetric deformation vibration (712 cm^{-1}) and two translational vibrations (277 and 150 cm^{-1}), which are specific to the carbonate lattice of CaCO_3 (Gunasekaran et al. 2006). In each of the curves of the spectral series these spectral bands appear with higher intensities, demonstrating that a part of the samples is most certainly composed of the trigonal crystalline mineral calcium carbonate, i.e. calcite (inorganic component).

However, a much larger number of peaks appeared in the spectra in a visually obvious way. These can be divided into two distinct groups. On the one hand, there is a series of peaks belonging to an

amorphous, carbonized component (Corg). These are very broad, poorly defined peaks (~1587 and 1372 cm^{-1}) and must probably represent the charcoal pieces seen on the microscopic views of **Fig. 5b** (Ferrari 2007). At the same time, another series of peaks belonging to the organic phase also appeared in the spectra. In both samples tested, shown in the **Figs. 5 and 6**, this organic component was “enriched”, providing us with the possibility of identification. Based on literature analogies, we may assume the component to be wax, most likely beeswax (Edwards et al. 1996). FTIR measurements would be needed to confirm the presence of beeswax. The different vibrations we identified are summarized in **Table 1**. The vibration at 1127 cm^{-1} was a superposition of two vibrations.

Figs. 7–9 represent the x-ray diffractograms of the studied samples. **Fig. 7** has quartz and beta-cristobalite and Mg-bearing calcite as the main components. **Fig. 8** hints at the presence of quartz and muscovite. **Fig. 9** shows the diffractogram of Mg-bearing calcite. Based on our results the main organic component was beeswax in addition to charcoal in both samples. The major mineral components are sand (quartz, muscovite) and carbonate (Mg-bearing calcite). The presence of beta-cristobalite is also notable.

Summary of the results

Based on our findings, the organic composition of the filling material tenax is similar to that described in the literature (Horváth et al. 2024; Heinrich-Tamácska 2016). The major organic components are wax, most likely beeswax and flue ash. The confirmation of beeswax requires FTIR measurements. Charcoal-bearing backing paste is known from the literature as well (Horváth et al. 2024). The main inorganic components are sand (quartz, muscovite), Mg-bearing calcite. The latter appears in the soda lakes of the Danube-Tisza Interfluve (Molnár & Molnár-Murvai 1975; Molnár et al. 2014). The presence of beta-cristobalite in the samples point to the presence of potential volcanic glass production residues which might have been transported to the site after weathering and long-distance fluvial transportation of the sediment (Kristály & Török 2020).

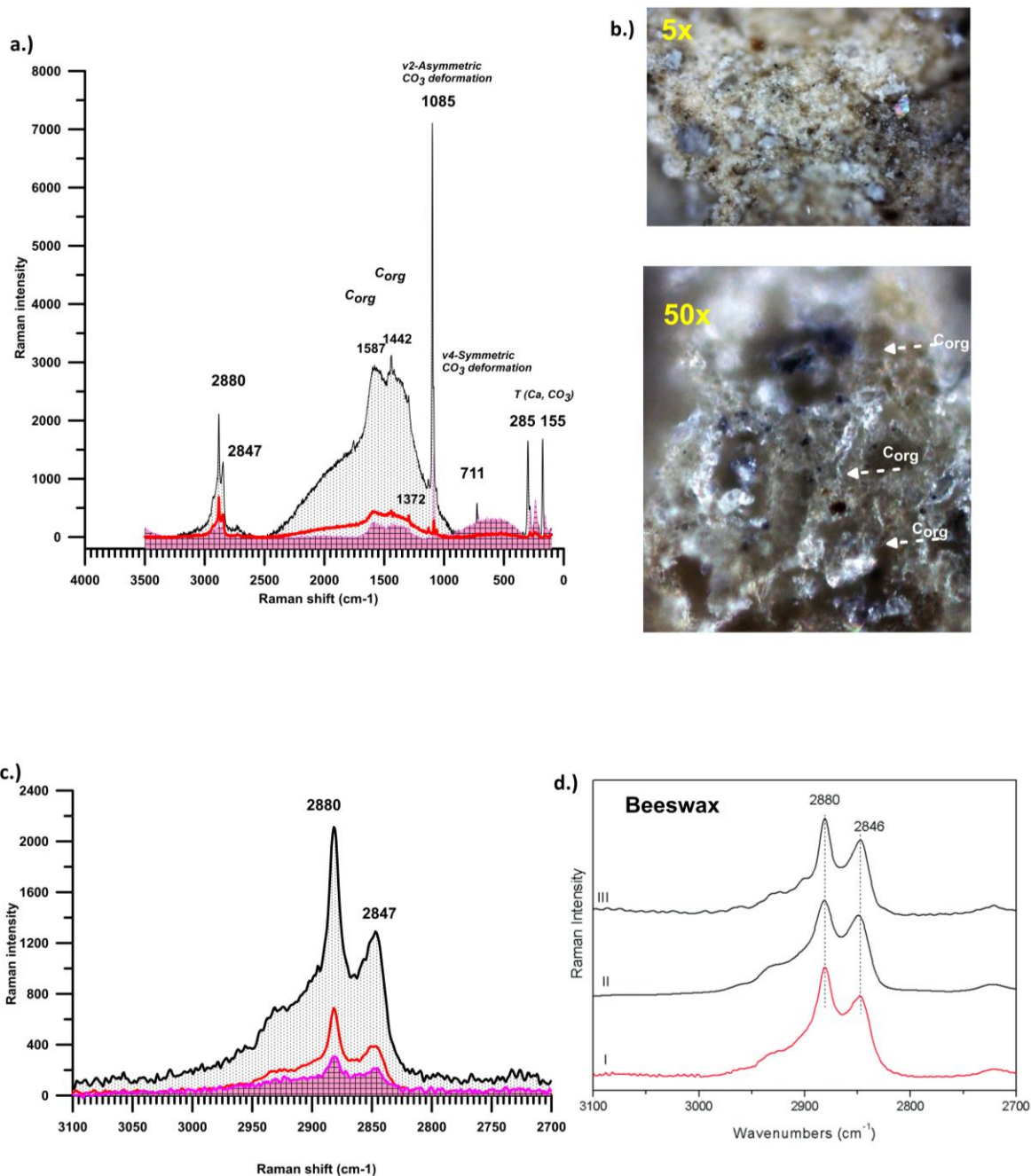


Fig. 5.: Raman spectra of the tenax recovered from the shield-shaped mount of Grave 80: **a.)** full spectrum, **b.)** microscopic view of the tenax, **c.)** area between 2700 and 3100 cm^{-1} wavenumbers enlarged giving signal of organic component wax **d.)** the spectrum of beeswax for reference.

5. ábra: A 80. sír pajsz alakú veretéből származó tenax Raman spektrumai: **a.)** teljes spektrum, **b.)** a tenax mikroszkópi képe, **c.)** a 2700 és 3100 cm^{-1} közötti rész kinagyítva a viaszra jellemző csúcsokkal **d.)** a méhviasz referencia spektruma.

Table 1.: Characteristic wavenumbers of the wax

1. táblázat: A viasz karakterisztikus csúcsai

Wavenumbers (cm^{-1})	$\nu(\text{CH}_2)$ asymmetric	$\nu(\text{CH}_2)$ symmetric	$\nu(\text{CH}_3\text{CH}_2)$	$\delta(\text{CH}_2)$	$\delta(\text{CH}_2)_2$	$\nu(\text{COC})$	$\nu(\text{CC})$ asymmetric	$\nu(\text{CC})$ symmetric
	2881	2847	2727	1435	1291	1127	1127	1060

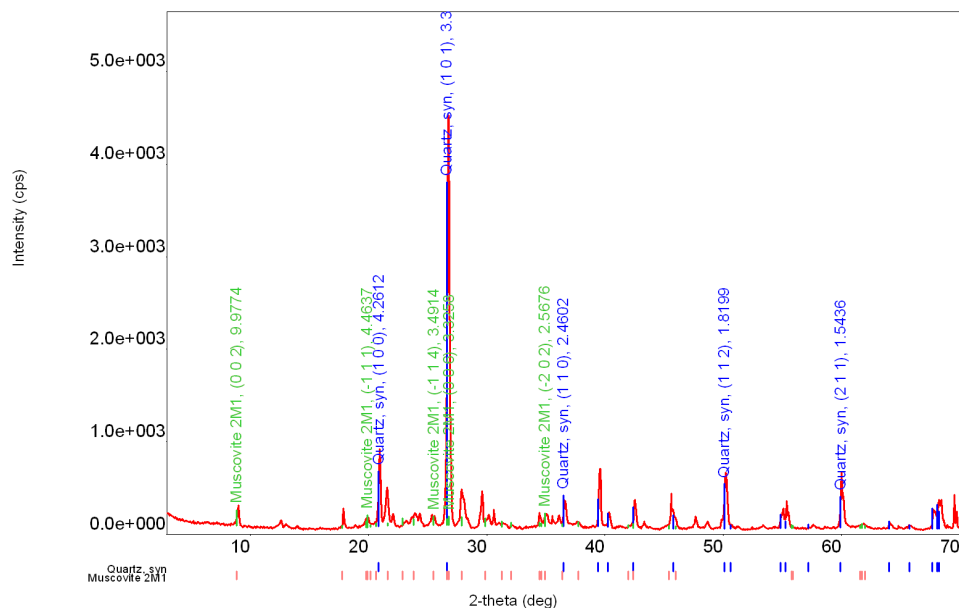


Fig. 8.: XRD diffractogram of tenax of sample G80

8. ábra: A G80-as minta tenaxának XRD diffraktogramja

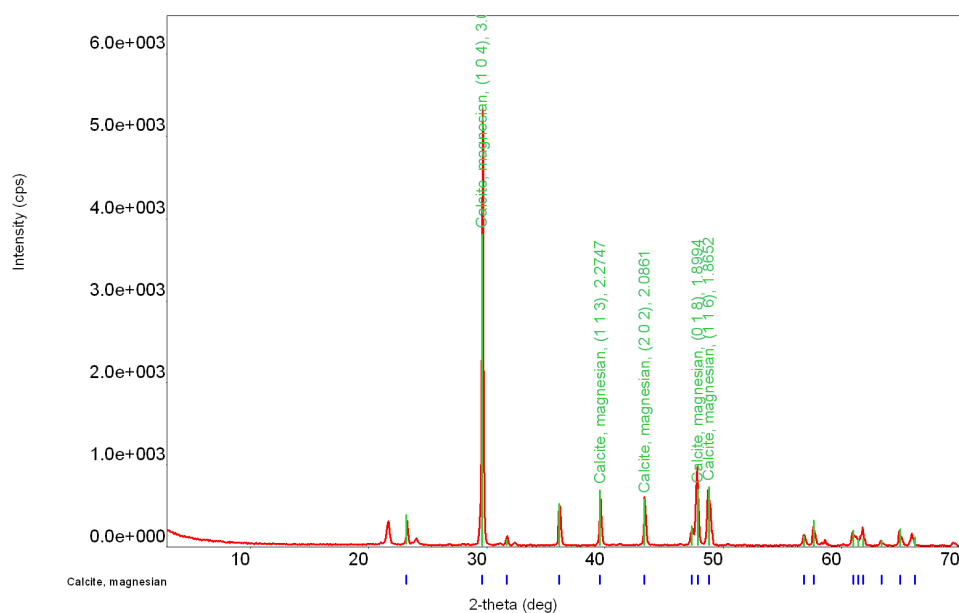


Fig. 9.: XRD diffractogram of tenax of sample G80

9. ábra: A G80-as minta tenaxának XRD diffraktogramja

Contribution of authors

Gulyás Sándor Conceptualization, Interpretation, XRD analysis, Writing – Original draft, Review & Editing. **Balogh Csilla** Material provision, Archaeological description, Circumstances of discovery, Archaeological and historical data, Dating, Editing. **Fintor Krisztián** Raman spectroscopy.

Acknowledgements

This work has been carried out as part of the Terra Tempus Research Group, University of Szeged. Support from OTKA Grant K109510 is acknowledged.

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