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Revealing the secrets of a lacquered leather artifact through molecular fingerprints

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This study reveals the composition and craftsmanship of a lacquered leather artifact from a Tang Dynasty tomb through molecular fingerprint analysis. The artifact was systematically analyzed and identified using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy–energy dispersive spectroscopy (SEM–EDS), and pyrolysis gas chromatography–mass spectrometry (Py–GC–MS). The results showed that FTIR and SEM analyses confirmed that the leather base of the artifact, while Py–GC–MS identified aliphatic aldehydes, unsaturated carbon chains, benzene rings, and long-chain hydrocarbons in the artifact, confirming the presence of lacquer. These findings demonstrate the effective integration of molecular fingerprinting techniques in the study of complex, deteriorated artifacts and provide scientific insights into the application of lacquer technology in Tang Dynasty artifacts.

Unlike artifacts of ceramics¹ or metals, organic artifacts are highly susceptible to deterioration over a long period of time due to the inherent properties of organic materials^{2–4}. This fragility results in a relatively small number of excavated organic artifacts, and research on their preservation and study is relatively limited. Collagen-based artifacts stand out among organic materials, and leather artifacts are the most important example^{5,6}. Leather, composed mainly of collagen, was widely used in ancient times for its durability and versatility⁷. It served as a key material for making various items such as clothing, footwear, containers, armor, and decorative objects. However, due to its proteinaceous nature, leather is highly sensitive to environmental factors, including humidity, temperature, and microbial activity. Over time, these factors can lead to the deterioration of its collagen fibers, resulting in significant structural and chemical changes. These challenges make the study and preservation of leather artifacts particularly complex and critical for understanding ancient craftsmanship and material use.

Lacquered leather artifacts are known for their complex craftsmanship and aesthetic appeal^{8,9}, and are an indispensable part of ancient cultures, especially in the Tang Dynasty (618–907 CE) of China^{10,11}. These artifacts play an important role in daily life and ritual practices, often serving as burial objects symbolizing the social and cultural status of their owners. However, environmental factors can lead to varying degrees of deterioration in them, affecting their appearance and

structural integrity, which may result in significant loss of material composition and process details¹². The irreversible deterioration makes the identification of these artifacts more complex, making it increasingly challenging to distinguish the raw materials and production techniques. In recent years, advances in analytical techniques, particularly molecular analysis, have provided new opportunities for studying these deteriorated organic materials. Pyrolysis gas chromatography–mass spectrometry (Py–GC–MS) and Fourier-transform infrared spectroscopy (FTIR) have been shown to play an important role in detecting and analyzing the molecular fingerprints of lacquers and leather^{13–17}. In addition, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM–EDS) has been widely used to study the microstructure and elemental composition of deteriorated artifacts, while X-ray diffraction (XRD) is crucial for examining the crystalline structure of inorganic components or residues, which may provide necessary evidence of environmental exposure or burial conditions.

The Asian lacquer used in Oriental artifacts usually comes from the sap of three distinct trees: *Toxicodendron vernicifluum* (in China, Japan, and Korea), *Toxicodendron succedaneum* (in Vietnam and Taiwan), and *Gluta usitata* (in Laos, Myanmar, Cambodia, and Thailand)¹⁸. Although the geographical distribution of these trees is more complex than commonly described, their sap is mainly related to these regions. The sap of the three tree species has a similar compositional profile,

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including water (30%), glycoproteins (2%), polysaccharides (7%), laccase enzyme (1%), and catechol derivatives (60–65%). However, the proportion and specific chemical structure of the catechol derivatives differ among the species¹⁹, which allows for the chemical differentiation of lacquers originating from different tree species. Laccase enzyme is crucial for triggering the polymerization process that leads to the formation of hardened lacquer²⁰. The variation in catechol and phenol mixtures between different species allows for the chemical differentiation of lacquers from different sources. The main component of *T. vernicifluum* sap is urushiol, while *T. succedaneum* produces laccol, and *G. usitata* produces thitsiol²¹. The alkenyl chains of these compounds are different: urushiol primarily contains C15 alkenyl chains, laccol features C17 alkenyl chains, and thitsiol is characterized by catechol derivatives with ω -phenylalkyl chains of C10 and C12^{18–22}. These differences are retained in the final polymer, allowing for chemical differentiation between the lacquers.

Py-GC-MS can recognize these molecular fingerprints even in heavily deteriorated samples, making it a powerful tool for analyzing complex organic materials. FTIR, on the other hand, can detect and characterize both collagen, the primary protein in leather, and lacquer. By identifying these compounds through Py-GC-MS and FTIR, the materials and methods used to manufacture these ancient artifacts can be reconstructed, providing valuable insights into their original composition and craftsmanship^{23,24}. While molecular analysis has been successful in the extensive study of organic artifacts, its application in lacquered leather artifacts still to be explored. Given the historical and cultural significance of these artifacts, it is evident that further research is needed using molecular fingerprinting techniques to uncover their secrets.

This study aims to contribute to the limited research on lacquered leather artifacts, particularly those that have severely deteriorated. By using molecular fingerprinting techniques, including Py-GC-MS and FTIR, a

severely deteriorated lacquered leather artifact was identified. By identifying molecular markers of lacquer and leather, the composition and original construction techniques of the artifact were revealed, deepening our understanding of ancient lacquered leather artifacts and their conservation.

Figure 1 shows the burial environment where the leather artifact was unearthed from Jiaozuo, Henan Province, in December 2020. Observing the structure of the tomb, a relatively long sloping passage can be found leading to the main chamber, which has an arched ceiling. Further examination of the placement of the tomb owner and accompanying objects revealed that the skeleton was in a supine and extended position, with a leather artifact near the top of the head (indicated by a dotted line). According to the structure of the tomb and the arrangement of the tomb owner and accompanying objects, the cemetery was preliminarily determined to be from the Tang Dynasty²⁵. The deceased was placed in a supine position with the limbs extended, which is consistent with the burial practices from the Tang Dynasty²⁶. In addition, the discovery of the artifact near the head is consistent with the common use of luxurious burial objects serving the deceased. This combination of a sloped tomb passage, an arched ceiling, and specific burial customs strongly indicates that the tomb originated from the Tang Dynasty.

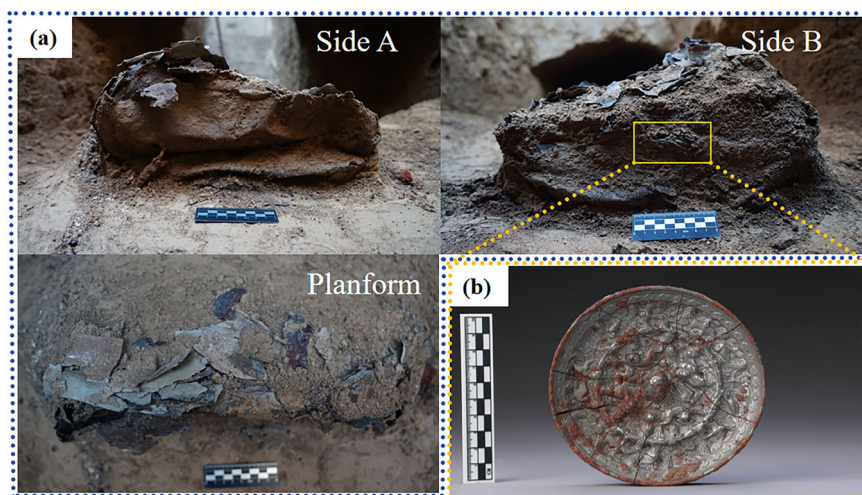
During the Tang Dynasty, Shanyang County (now Jiaozuo City, Henan Province) was located along a major transportation artery, with developed agriculture, abundant mineral resources, active cultural and religious life, and a high level of economic and cultural development²⁷. Shanyang County was located at the southern foot of the Taihang Mountains and was first mentioned in historical documents such as *Records of the Grand Historian (Shiji)* and *Book of Han (Hanshu-Geography)*. Although it was not one of the most developed regions in the Tang Dynasty, it played an important role in the regional economy, military defense, and cultural exchange.

The unearthed leather artifact is a leather bag, as shown in Fig. 2a. Severe fragmentation posed significant challenges to determining material

Fig. 1 | The digital pictures of a Tang Dynasty tomb and the excavation site of the leather artifact.



Fig. 2 | Overview of the leather artifact and the associated Sea-Creature Grape Mirror. **a** The all-around view of the leather artifact. **b** The digital picture of the Sea-Creature Grape Mirror.



composition and manufacturing techniques. It is worth noting that a marine life grape mirror was found inside this leather bag, as shown in Fig. 2b. The Tang Dynasty marked the pinnacle of Chinese bronze mirror craftsmanship and was renowned for its innovative spirit. These mirrors were mainly circular, occasionally square or lozenge-shaped, and usually decorated with patterns of grapevines and sea marine life. The design evolved from the complex and rigid patterns of mirrors in the Han Dynasty to a simpler and smoother style, with extremely exquisite craftsmanship²⁸. Among them, the marine life grape mirror is particularly eye-catching, and its mysterious pattern makes it known among scholars as “Mysterious Mirror”²⁹. Similar burial practices have been reported before. For example, a lacquer box containing a bronze mirror was unearthed from the Tomb M56 in Machang, Pingba County, Guizhou Province³⁰. However, as observed in this artifact, the combination of leather craftsmanship and lacquer production is very complex and rarely documented in Chinese archaeology. So far, to our knowledge, four Tang Dynasty lacquered leather artifacts are preserved in the Shōsōin Repository in Japan, providing rare insights into the complex craftsmanship. The discovery of the marine life grape mirror provided crucial support for determining the age of the tomb to the Tang Dynasty.

Methods

Sampling

The samples were collected from the fragments of the lacquered leather and the surrounding soil. The artifact fragments were carefully removed from the excavation site to minimize further damage, and the soil samples were taken from the immediate area around the artifact to better understand the burial environment. After collection, all samples were stored at a stable temperature of 22 °C and a relative humidity of 50%, to prevent further degradation before analysis.

The fresh lacquer sample was purchased from Xiaoli Natural Lacquer Co., Ltd. (Mianyang, China), and naturally cured at 25 °C and 80% relative humidity for 7 days, simulating traditional curing conditions. After curing, the sample was referred to as the newly prepared lacquer to reflect its final state for further analysis. This sample was used to validate the identification results obtained from the artifact analysis.

FTIR characterization

The residual fragment samples were analyzed using a Nicolet iS5 Fourier-transform infrared spectrometer (Thermo Fisher Scientific) with the KBr pellet method. The sample was finely ground and mixed with KBr powder, which does not absorb in the infrared range, and then pressed into a transparent pellet to ensure optimal transmission of infrared light and obtain clear spectral data. The spectra were collected in the wavenumber range of 400–4000 cm^{-1} , with 64 scans and a resolution set at 4 cm^{-1} .

SEM–EDS characterization

Due to the severe deterioration of the lacquered leather artifact, it was impossible to extract a large amount of leather fibers. Therefore, a small amount of soil around the artifact was collected and analyzed using a scanning electron microscope (SEM) to determine any residual fibers in the soil. The microscopic images of the samples were obtained using a FEI Quanta 250 SEM. The images were captured using an accelerating voltage of 20 kV. Before SEM observation, a thin gold coating was applied to the surface of the sample to increase conductivity and improve image quality. Energy dispersive spectroscopy (EDS) analysis was performed using the Bruker Quantax 200 XFlash 6/60 system.

XRD characterization

The collected soil samples were characterized using an X-ray diffractometer (Rigaku Ultima IV, Rigaku Corporation, Tokyo, Japan), with Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$) as the X-ray source. The scanning range was set from 5° to 90° to ensure comprehensive coverage of all possible crystalline phases in the sample. A scanning rate of 10°/min was adopted to allow sufficient signal collection time to obtain high-resolution diffraction patterns.

Characterization of Py-GC–MS

Approximately 10–20 mg of the sample was placed into a micro-sample tube. Then it was pyrolyzed using an EGA/PY-3030D pyrolysis probe at 550 °C, producing volatile small-molecule products for subsequent GC–MS analysis. For chromatographic separation, a UA-5MS column (30 m \times 0.25 mm \times 0.25 μm) was used on a Shimadzu QP2010 ultra gas chromatograph-mass spectrometer. The initial column temperature was set to 40 °C and maintained for 4 min, then the temperature was raised to 280 °C at a rate of 6 °C/min, and finally maintained for 5 min to ensure complete separation of the components. High-purity helium was used as the carrier gas at a flow rate of 1 mL/min. Mass spectrometric detection was conducted in electron ionization (EI) mode with an ionization energy of 70 eV. A scan range of 14–500 amu was applied to capture molecular ion and fragment ion peaks from the pyrolyzed products to ensure comprehensive detection of all relevant components.

Results and discussion

SEM–EDS analysis

Residual fibers were found in the soil around the artifact, and their SEM images and elemental composition are shown in Fig. 3. Elemental analysis confirmed that these fibers are primarily composed of carbon (C) (Fig. 3a1), nitrogen (N) (Fig. 3a2), and oxygen (O) (Fig. 3a3). It is worth noting that the distribution of elements is closely related to fiber morphology, with oxygen having the highest proportion at 54.14%, followed by carbon at 28.70%, and

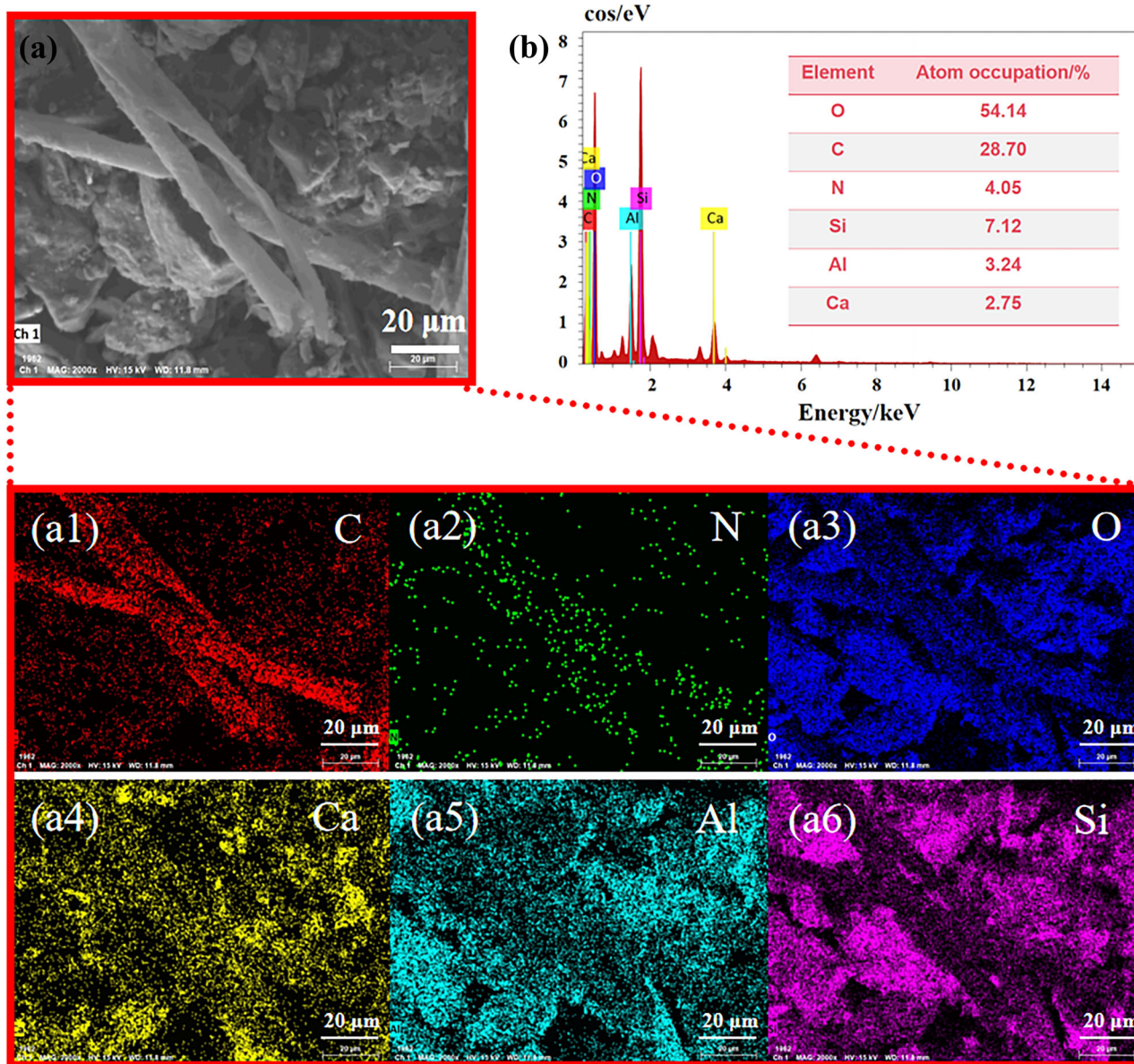


Fig. 3 | SEM analysis and elemental distribution of leather fibers found in the soil. **a** The SEM images of leather fibers and the elemental distribution of (a1) C, (a2) N, (a3) O, (a4) Ca, (a5) Al, and (a6) Si. **b** The corresponding atom occupation.

nitrogen at 4.05%, as shown in Fig. 3b. These significant findings provide essential evidence that the fibers might be originated from leather, which is correlated with the FTIR results, thus providing key clues about the original material of the artifact.

XRD analysis

In addition, EDS elemental analysis revealed the presence of Ca, Al, and Si (as shown in Fig. 3a4, a5, and a6, and further confirmed their sources from quartz, calcite, and albite through XRD analysis (Fig. 4). The identification of these minerals indicated that the fibers come into contact with soil components containing these minerals. The presence of quartz (SiO_2), calcite (CaCO_3), and albite ($\text{NaAlSi}_3\text{O}_8$) suggested the possible existence of sedimentary inclusions, providing clues for understanding the burial environment and preservation conditions of the artifact.

FTIR analysis

Figure 5 shows the FTIR spectrum of the artifact fragment sample, revealing the key functional groups characteristic of both lacquer and leather. The broad peaks at 3627 cm^{-1} and 3419 cm^{-1} correspond to the

stretching vibrations of hydroxyl ($\nu(\text{O-H})$) and amino ($\nu(\text{N-H})$) groups, respectively. The weak signal at 1641 cm^{-1} indicates the stretching vibration of the carbonyl group in the amide I band ($\nu(\text{C=O})$). The disappearance of other characteristic peaks of amide bands suggests that the collagen structure in the sample has undergone extensive deterioration^{31–34}. The peak at 1436 cm^{-1} is attributed to the bending vibration of methylene groups ($\delta(\text{CH}_2)$). A prominent signal at 1031 cm^{-1} arises from overlapping stretching vibrations of carbon–oxygen ($\nu(\text{C-O})$) and silicon–oxygen ($\nu(\text{Si-O})$) bonds, and the Si–O contribution was enhanced by silica and silicates from the burial environment. This observation is consistent with the results of the XRD analysis, which also confirmed that silica and silicate minerals are the main components of the inorganic residues, further supporting their origin from the burial environment. The signal near the 803 cm^{-1} corresponds to the out-of-plane bending vibrations of C–H bonds in substituted benzene rings ($\gamma(\text{C-H})$). The lower wavenumber signals may be due to stretching or bending vibrations of inorganic compounds. These spectral features collectively confirm the presence of both lacquer and leather components in the sample.

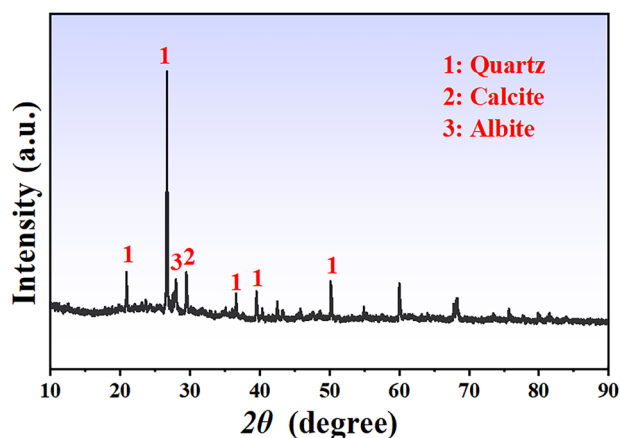


Fig. 4 | The XRD pattern of the collected soil samples.

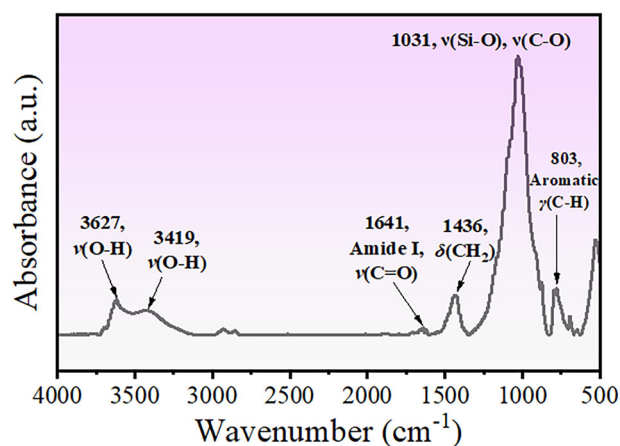


Fig. 5 | The FTIR spectrum of the fragment sample.

Py-GC-MS analysis

To further confirm the composition and manufacturing of the leather artifact, Py-GC-MS was used to analyze the fragment samples. Figure 6 shows the total ion chromatogram (TIC) of the residual fragments, which can be divided into two main zones: Zone I (0–8 min), characterized by a broad peak, and Zone II (8–22 min), composed of a series of sharp peaks. In-depth analysis was conducted on the mass spectrometry data from these two zones to elucidate the chemical characteristics of the artifact residues.

In Zone I, a characteristic signal of aliphatic aldehyde was observed at 6.9 min with an m/z of 44, as shown in Fig. 7a. This signal is generated by McLafferty rearrangement of unsubstituted α -C under mass spectrometry ionization conditions, producing oxonium ion ($C_2H_4O^+$)^{35–37}. This specific rearrangement is important because it is typically observed in aliphatic aldehydes during pyrolysis, providing a unique molecular fingerprint that helps confirm the presence of aldehydes in the sample. In addition, unsaturated hydrocarbon chains were detected, including $m/z = 55$ ($C_4H_7^+$) and $m/z = 67$ ($C_5H_7^+$). Their corresponding chemical structures are shown in Fig. 7b. These signals indicate the presence of aldehydes and short unsaturated carbon chains in the pyrolysis products, suggesting that the sample might contain unsaturated aliphatic compounds. The detection of these aldehydes and other unsaturated hydrocarbon fragments suggested that they might be degradation products of the original lacquer compounds, thereby strengthening the identification of this artifact as lacquered leather.

As shown in Fig. 8a, a notable shift in the mass spectrometric spectrum is observed at 7.9 min. In Fig. 8b, the strongest signal peak appears at $m/z = 91$, corresponding to tropylium ion ($C_7H_7^+$) that was formed by a rearrangement reaction of the α -C attached to the benzene ring under ionization conditions³⁷. In addition, the signal at $m/z = 79$ is attributed to the

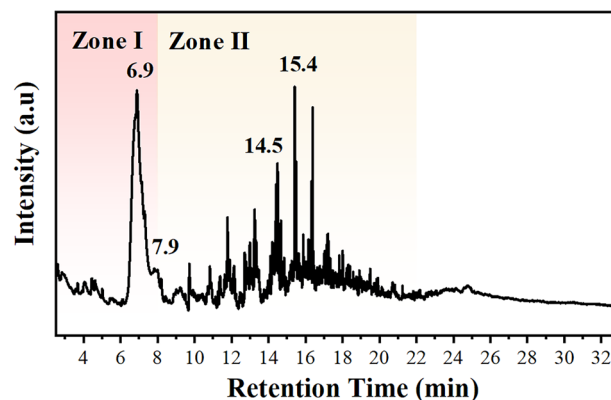


Fig. 6 | The TIC of the residual fragments of the leather artifact.

further cleavage of the tropylium ion, producing cyclohexadienyl ion ($C_6H_7^+$), while the signals at $m/z = 77$ and $m/z = 78$ correspond to the phenyl ion ($C_6H_5^+$) from the benzene ring. These signals indicate the presence of compounds with a benzene ring core structure in the sample, consistent with the result of infrared analysis (Fig. 3).

In Zone II, mass spectrometric analysis revealed a series of more complex organic compounds. As shown in Fig. 9a, the 14.5 min spectrum displays signals with $m/z = 43, 57, 71,$ and 85 , corresponding to the $C_3H_7^+$, $C_4H_9^+$, $C_5H_{11}^+$, and $C_6H_{13}^+$ (Fig. 9b), which are characteristic fragment ions of long-chain alkanes. This indicated the presence of long-chain alkanes in the sample components. Subsequently, in the 15.4 min spectrum (Fig. 9c), signals with $m/z = 41, 55, 69,$ and 83 (Fig. 9d) represent alkene fragments with double bonds ($C_3H_5^+$, $C_4H_7^+$, $C_5H_9^+$, and $C_6H_{11}^+$), corresponding to the alkanes observed in Fig. 9b. Furthermore, other alkenes such as $C_5H_9^+$ ($m/z = 97$) and $C_8H_{15}^+$ ($m/z = 111$) were also detected. These findings further support that the signals in Zone II mainly comes from the pyrolysis products of long-chain alkanes and alkenes.

It is worth noting that, as shown in Fig. 10a–c, the signals at $m/z = 91$, $m/z = 107$, and $m/z = 123$, representing $C_7H_7^+$, $C_7H_7O^+$, and $C_7H_7O_2^+$, respectively, span almost the entire analytical period. This observation is consistent with previous findings reported by Okamoto et al.³⁸, who also identified these ions as characteristic of aromatic structures. Although the signal at $m/z = 91$ appears relatively weak in Zone II, its trend aligns with the major peaks in the TIC, as illustrated in Fig. 10a. The extracted ion chromatogram for $m/z = 91$ further shows stable tropylium ions present in Zone II (8–22 min), suggesting that the benzene ring structures may coexist with long-chain hydrocarbons in the sample. In Fig. 10b, c, derivatives of phenol and catechol were detected, as shown in Fig. 10d, which confirms it as the main characteristic chemical component of Asian lacquer³⁹, supporting the sample's attribution to Asian lacquer.

As shown in Fig. 11, signals from lacquer were identified in the artifact sample, mainly composed of alkyl-substituted catechols and benzenes with different chain lengths. For the catechol fragments (Fig. 11a), the maximum substitution observed was C12, with the main signals corresponding to the C6–C12 substituted catechols. For the alkylbenzene fragments (Fig. 11b), longer chain substitutions were observed, with the main signals corresponding to C11–C15 alkylbenzenes. No high-chain substituted catechols or benzenes were detected. These results suggested that the lacquer in the artifact sample may have originated from *T. vernicifluum*, a lacquer tree widely distributed in China, which produces urushiol. Urushiol is a natural resin mainly composed of 3-pentadecylcatechol, which is consistent with the structural information obtained from Py-GC-MS analysis.

Figure 12a shows the Py-GC-MS chromatogram comparing the newly prepared lacquer (as a reference material of a known source) with the artifact sample, with a focus on verifying that the artifact indeed contains lacquer components, while highlighting the similarities and differences between the two. Significant differences were observed in thermal degradation behaviors. The artifact sample showed clear peaks before 7 min, while the newly

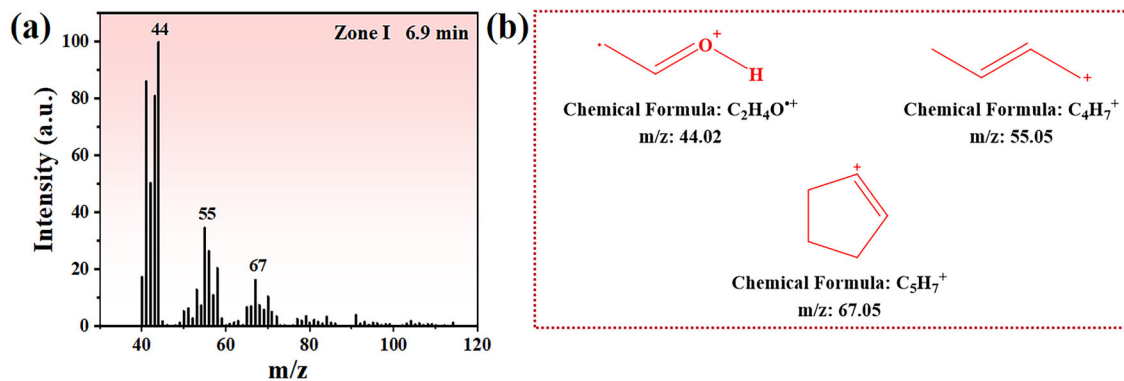


Fig. 7 | Mass spectrometry analysis of volatile compounds detected in Zone I. a The mass spectrum obtained at 6.9 min, and b the corresponding chemical structures of the identified compounds.

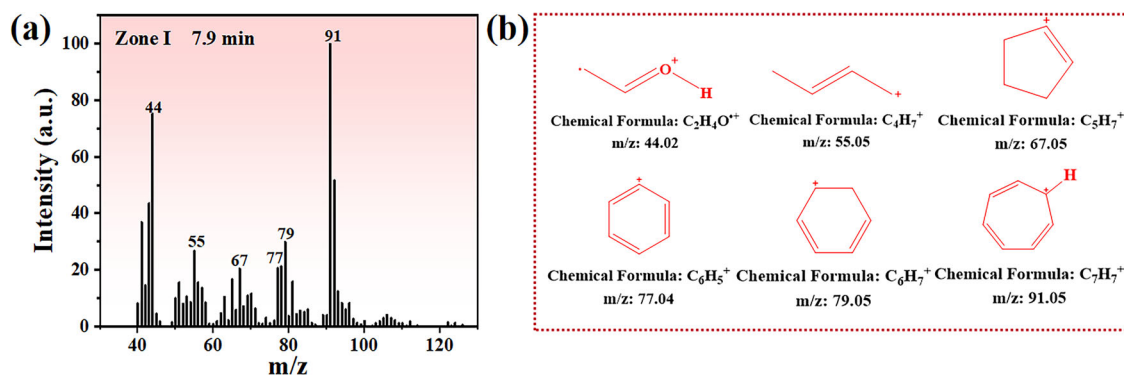


Fig. 8 | Mass spectrometry analysis of volatile compounds detected in Zone I. a The mass spectrum obtained at 7.9 min, and b the corresponding chemical structures of the identified compounds.

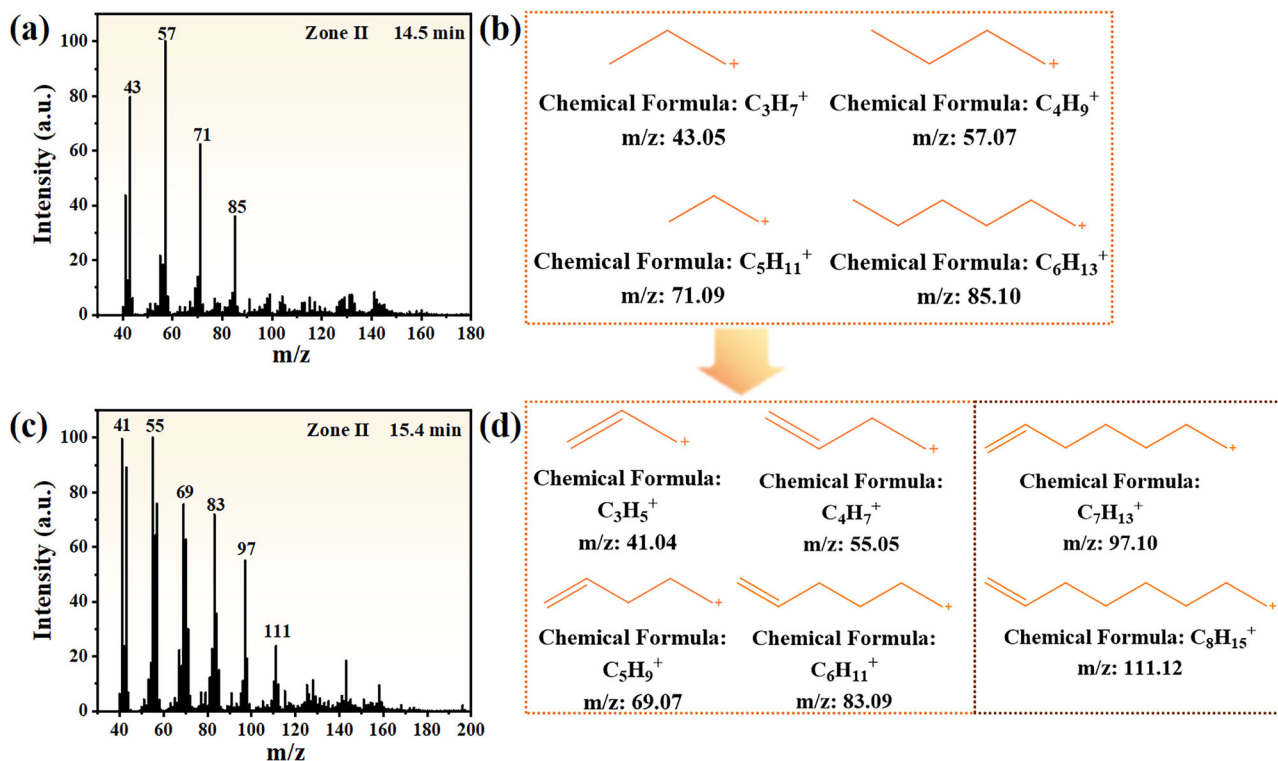


Fig. 9 | Mass spectrometry analysis of volatile compounds detected in Zone II. a The mass spectrum obtained at 14.5 min and c at 15.4 min, and b, d their corresponding chemical structures of the identified compounds.

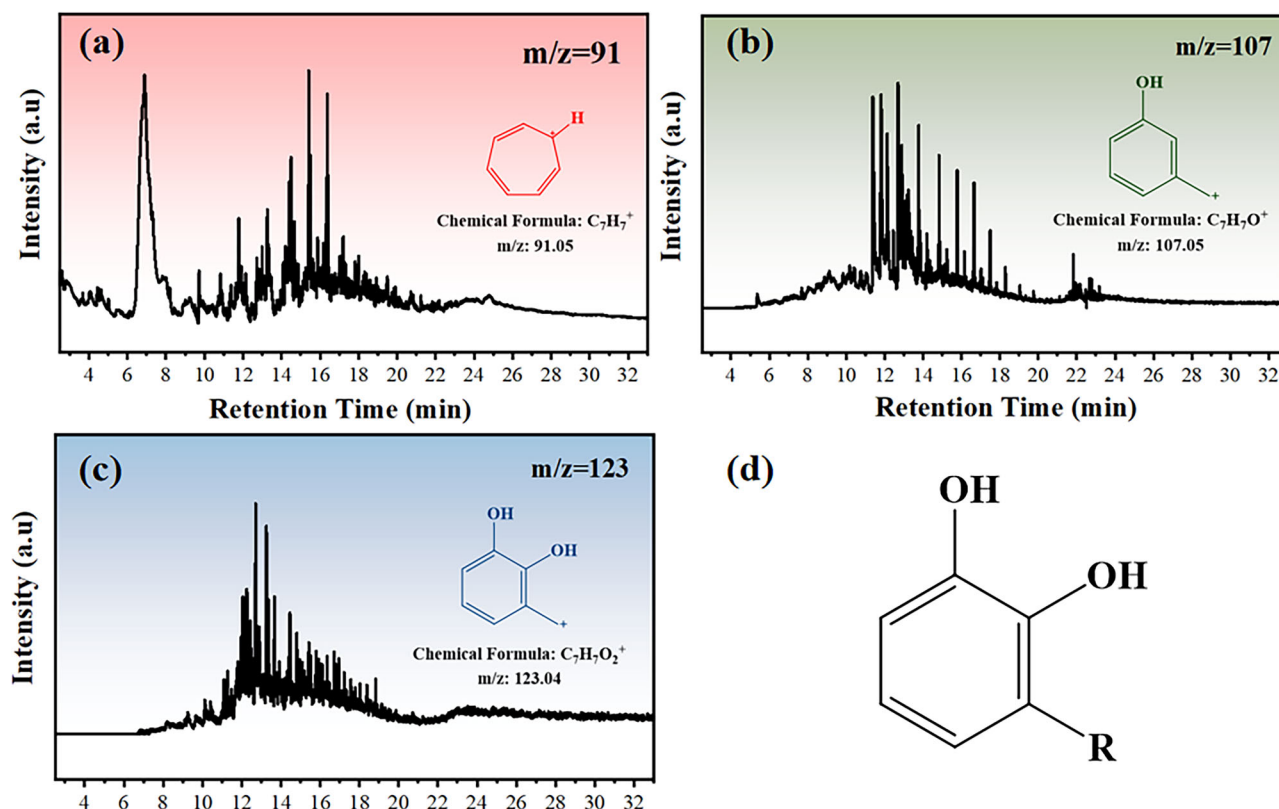
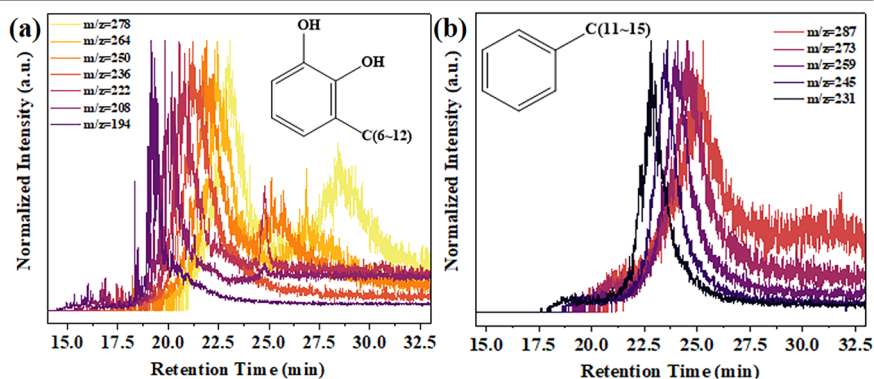


Fig. 10 | TIC and chemical structure of alkyl-substituted catechols from residual fragments of the leather artifact. **a** TIC at $m/z = 91$, **b** $m/z = 107$, **c** $m/z = 123$, and **d** the general structure of alkyl-substituted catechols (R represents an alkyl group of varying chain length).

Fig. 11 | The extracted ion chromatograms (EIC) of alkyl-substituted compounds. **a** Alkyl-substituted catechols and **b** alkyl-substituted benzenes.



prepared lacquer did not. These early signals indicate that, due to aging, the crosslinking structure of the lacquer has been disrupted, resulting in the formation of many low-molecular-weight fragments. In contrast, the newly prepared lacquer showed strong signals after 20 min, reflecting its intact crosslinked structure and higher thermal stability. Between 10 min and 20 min, both samples showed overlapping peaks of substituted benzenes with different alkyl chain lengths.

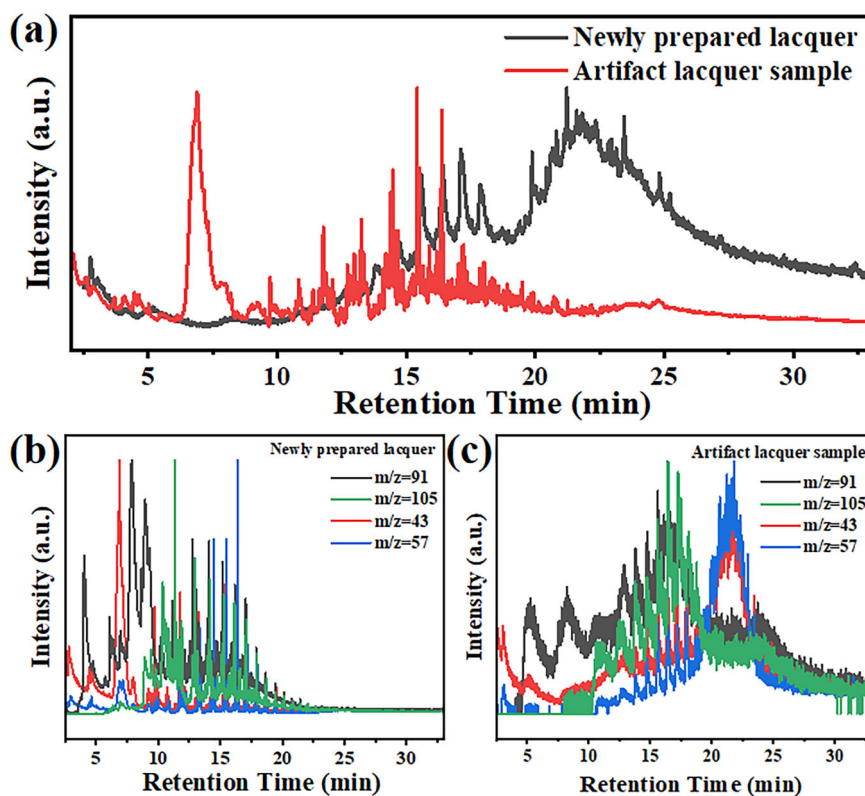
Figure 12b, c show the extracted ion chromatograms (EIC) of $m/z = 43$ and 57 (characteristic fragments of alkane chains) and $m/z = 91$ and 105 (related to alkylbenzenes) of the artifact sample and newly prepared lacquer. The artifact sample (Fig. 12b) displays weaker signals in these regions, reflecting the degradation of long alkyl chains due to aging. In contrast, the newly prepared lacquer (Fig. 12c) exhibited stronger alkane chain signals after 20 min, indicating that these signals originated from long-chain alkyl-substituted benzenes in the urushiol structure. This comparison not only verifies the presence of lacquer components in the artifact, but also reveals

differences in their chemical composition and degradation behavior, providing valuable insights into the preservation status and aging mechanisms of the artifact.

The discovery of lacquered leather artifacts as burial objects in Tang Dynasty tombs has immeasurable value, not only with profound historical significance, but also with rich cultural value, such as the cultural exchange and technological advancements of the Tang Dynasty. The application of lacquer techniques significantly improved the durability and aesthetic appeal of leather artifacts, symbolizing the social status and wealth of the tomb owner in daily life. In addition, the rarely recorded material evidence is provided for studying the funerary customs of the Tang Dynasty. This discovery provides valuable insights into the composition and craftsmanship of lacquered leather artifacts from the Tang Dynasty, enriching our understanding of their cultural significance and conservation.

In summary, this study investigated the composition of a lacquered leather artifact from a Tang Dynasty tomb by combining analytical

Fig. 12 | Py-GC-MS analysis of artifact lacquer sample and newly prepared lacquer. **a** The comparison of TICs. The extracted ion chromatograms (EIC) of **(b)** the artifact sample and **(c)** the newly prepared lacquer at $m/z = 43, 57, 91,$ and 105 .



techniques of FTIR, SEM-EDS, and Py-GC-MS. The results confirmed that the artifact was composed of lacquered leather, with the lacquer most likely being urushiol-based. These findings provide important insights into the application of lacquer materials in Tang Dynasty tomb artifacts. The importance of molecular fingerprinting in identifying the materials and composition of ancient artifacts is highlighted, particularly its applicability in analyzing severely deteriorated organic artifacts. This method provides a foundation for further exploration of historical craftsmanship and information for future conservation efforts.

Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Mingrui Zhang: conceptualization, investigation, writing—reviewing and editing, and methodology. Jie Liu: visualization, validation, and methodology. Bo Li: investigation and methodology. Yong Lei: visualization and validation. Mădălina Georgiana Albu Kaya: visualization and validation. Xiaohu Zhang: visualization and validation. Keyong Tang: supervision, validation, resources, and funding acquisition. All authors reviewed the manuscript.

Competing interest

The authors declare that they have no known competing interests that could have appeared to influence the work reported in this paper. Y.L. serves on the Editorial Board of *npj Heritage Science* but was not involved in the peer-review or editorial decision of this submission.

Consent to publish

All participants consented to the publication of this work.

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