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Characterization of the residues in Han Dynasty bronze lamps by pyrolysis–gas chromatography–mass spectrometry

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Abstract

The fuel residues in two bronze lamps excavated from Haihunhou Tomb in the Han Dynasty, in Jiangxi Province, China, were studied in this paper. The techniques of Fourier transform infrared (FT-IR) spectroscopy and pyrolysis–gas chromatography–mass spectrometry with thermal-assisted hydrolysis and methylation (THM-Py–GC/MS) were used to characterize the materials. Long-chain fatty acids, fatty alcohols and hydrocarbons were detected as the main components. By comparing the reference materials of beeswax and Chinese wax, the residues in the lamps were identified as beeswax. Furthermore, according to the morphology of the remains of the lamp fuel, the fuel remains were probably melted and then solidified, which suggests the lamps were used. Wax was found to have been used as an illuminating fuel as early as the Han Dynasty, enriching scholarly knowledge about the lighting materials used in ancient China.

Keywords: Py–GC/MS, Beeswax, Chinese wax, Illuminating fuel, Haihunhou Tomb

Introduction

Haihunhou Tomb (Western Han Dynasty, 202BC–AD8) was discovered in 2015 and is located south of Poyang Lake, Nanchang city, Jiangxi Province, China (Fig. 1). Liuhe was the emperor of the Western Han for just 27 days before being dethroned (63 B.C.). After being dethroned, he received his new rank of Haihunhou (the rank of nobility in the Han Dynasty) and the lands around Poyang Lake, where he eventually was buried. His coffin was in M1. Surprisingly, massive amounts of burial goods were found in this tomb, including a large pile of bronze coins (approximately 10 tons), a great number of gold and silver wares, bronze, lacquered musical instruments, porcelain goods, and very valuable lacquer paintings [1, 2]. Among these findings were two bronze lamps, numbered M1:1641 (Gang lamp) and M1:1681 (Yanzu lamp) [3] (Fig. 2). Luckily, remains of the illuminating fuel

were found inside the lamps, which is particularly rare and provides an opportunity to study the illuminating fuels used in the Han Dynasty. The sampling locations are marked in Fig. 3.

In recent years, there have been some reports about illuminating fuels. The most common kinds of lamp fuel are plant oil [4, 5], animal fat [6, 7], and wax. However, among these, archaeological examples of wax fuels are not abundant [8]. Moreover, in different areas, different kinds of materials were used as lamp oils. For example, olive oil has been found mostly around the Mediterranean, including in Roman [9] and Egyptian sites [4], because different material resources were available. According to the ancient literatures, in addition to beeswax, Chinese wax was also used in early China, which was recorded in *Compendium of Materia Medica* (本草纲目) [10]. This material is a white to yellowish-white gelatinous substance secreted by certain insects, such as *Ceroplastes ceriferus* and *Ericerus pela*. The main components in Chinese wax are wax esters formed by monosaturated fatty acids and monosaturated fatty alcohols [11], which are similar to the components in beeswax. The differences of molecular signatures between

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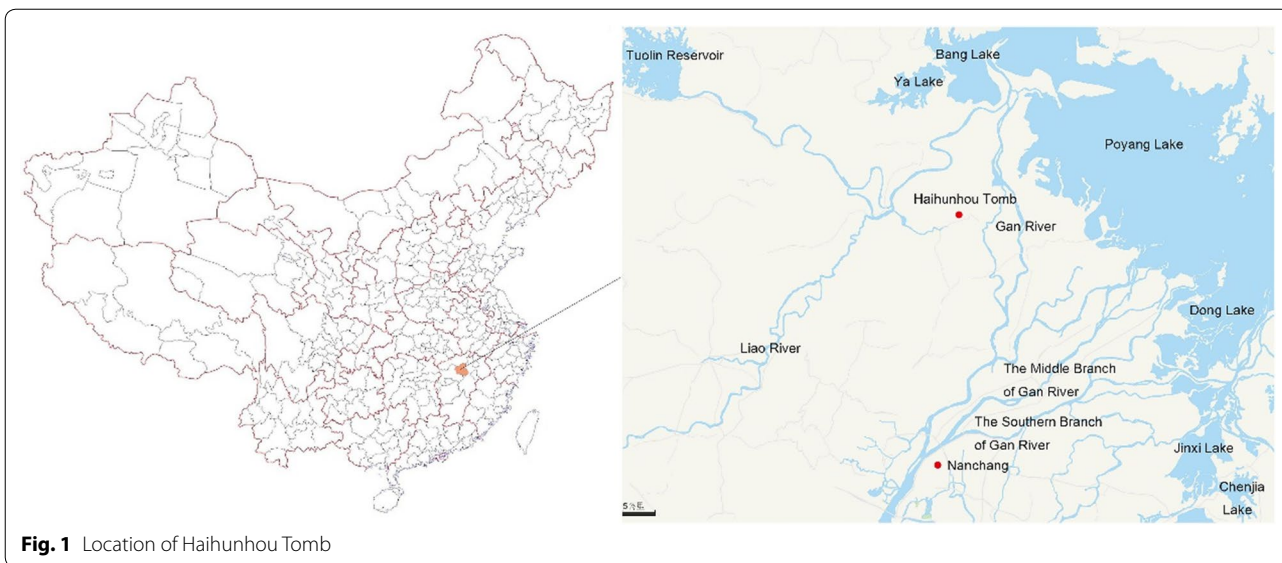Full list of author information is available at the end of the article

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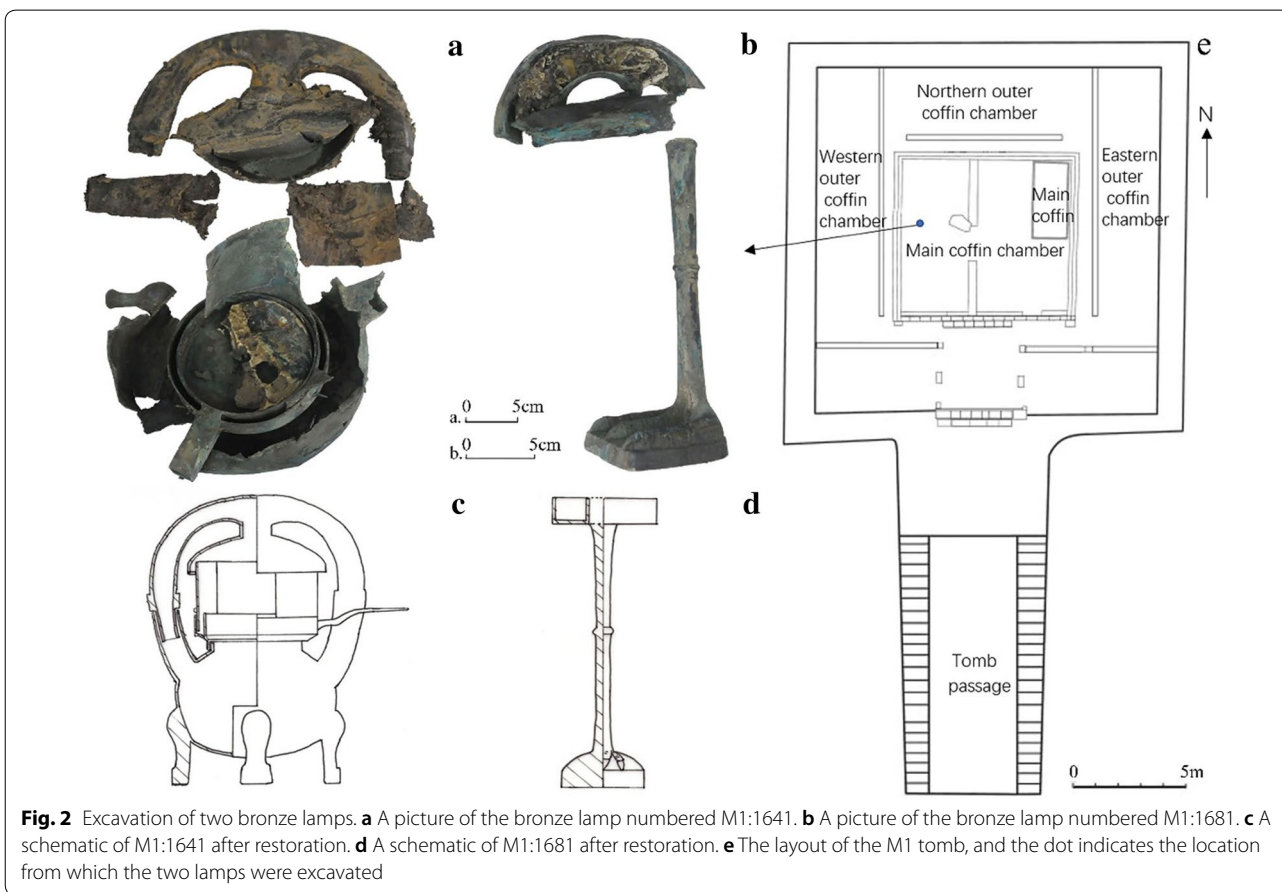

Fig. 1 Location of Haihunhou Tomb

Fig. 2 Excavation of two bronze lamps. **a** A picture of the bronze lamp numbered M1:1641. **b** A picture of the bronze lamp numbered M1:1681. **c** A schematic of M1:1641 after restoration. **d** A schematic of M1:1681 after restoration. **e** The layout of the M1 tomb, and the dot indicates the location from which the two lamps were excavated

these two materials mainly are relative content of the fatty acids and alcohols. Chinese wax has a higher melting point and harder than beeswax.

The main techniques used to characterize organic remains from cultural heritage sites are Fourier transform infrared (FT-IR) spectrometry [12], high-performance liquid chromatography–mass spectrometry (LC/MS) [9],
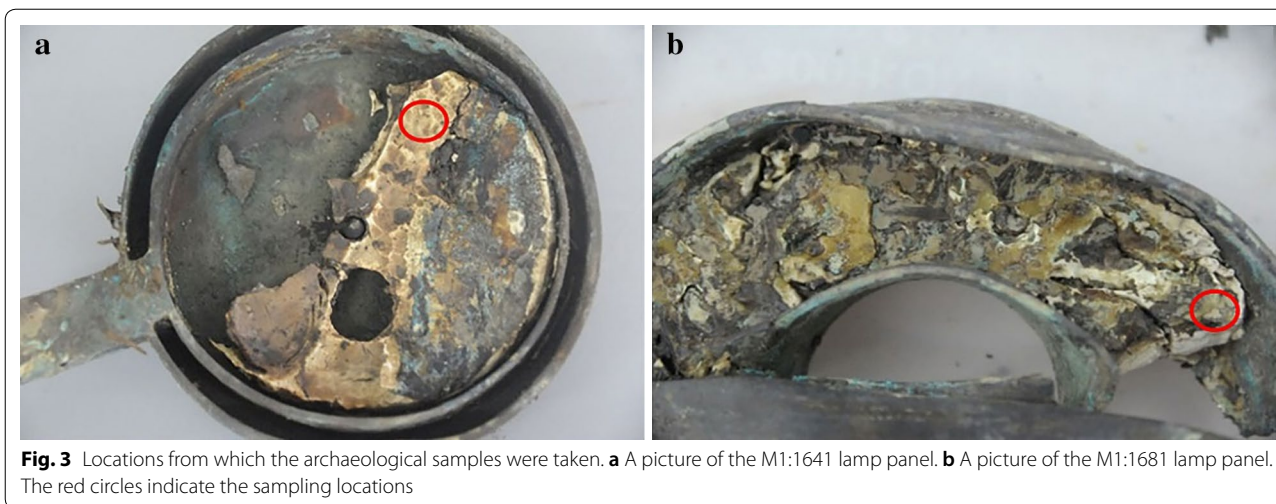
Fig. 3 Locations from which the archaeological samples were taken. **a** A picture of the M1:1641 lamp panel. **b** A picture of the M1:1681 lamp panel. The red circles indicate the sampling locations

gas chromatography coupled to mass spectrometry (GC/MS) [13–17], high-temperature gas chromatography coupled to mass spectrometry (HT–GS) [18] and pyrolysis–gas chromatography–mass spectrometry (Py–GC/MS) [19–22]. Py–GC/MS with thermally-assisted hydrolysis and methylation (THM) has the advantages of offering high sensitivity and requiring a facile pretreatment of the samples [23], and this method has been successfully used to characterize the remains in Tang Dynasty lamps [20]. Thus, THM-Py–GC/MS in conjunction with FT-IR spectrometry was chosen for this study. The results provide evidence for the materials used by the aristocracy of the Han Dynasty to illuminate dark spaces.

Experimental
Samples
The two canary-yellowish samples were taken from the bronze Gang lamp numbered M1:1641 and the bronze Yanzu lamp numbered M1:1681, which were found in the western chamber of Haihunhou Tomb (Fig. 3). Reference samples were Chinese wax, purchased from Emeishan in Sichuan Province and beeswax, bought from apiarist in Shanxi Province. Another name of Chinese wax is Sichuan wax, because it was original produced in Sichuan Province. These reference samples can represent the kinds of wax typically used in ancient China.

Fourier transform infrared (FT-IR) spectrometry
The FT-IR analyses were carried out on a Nicolet 6700 advanced Fourier transform infrared spectrometer (American ThermoFisher Scientific Cooperation). The spectra were collected over the 4000–500 cm^{-1} region using attenuated total reflectance (ATR) for the measurements.
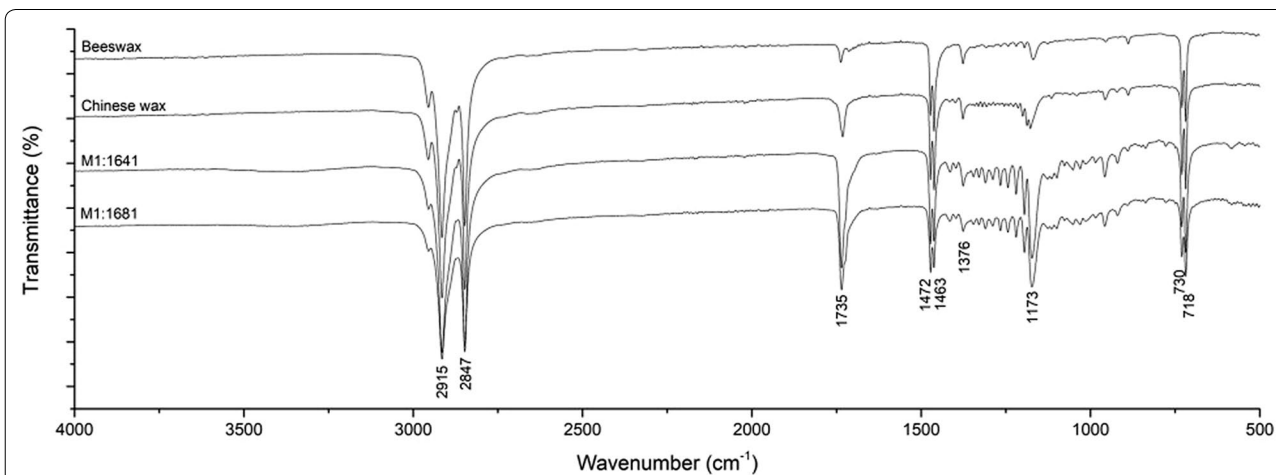

Fig. 4 Infrared spectra of the reference samples and archaeological samples

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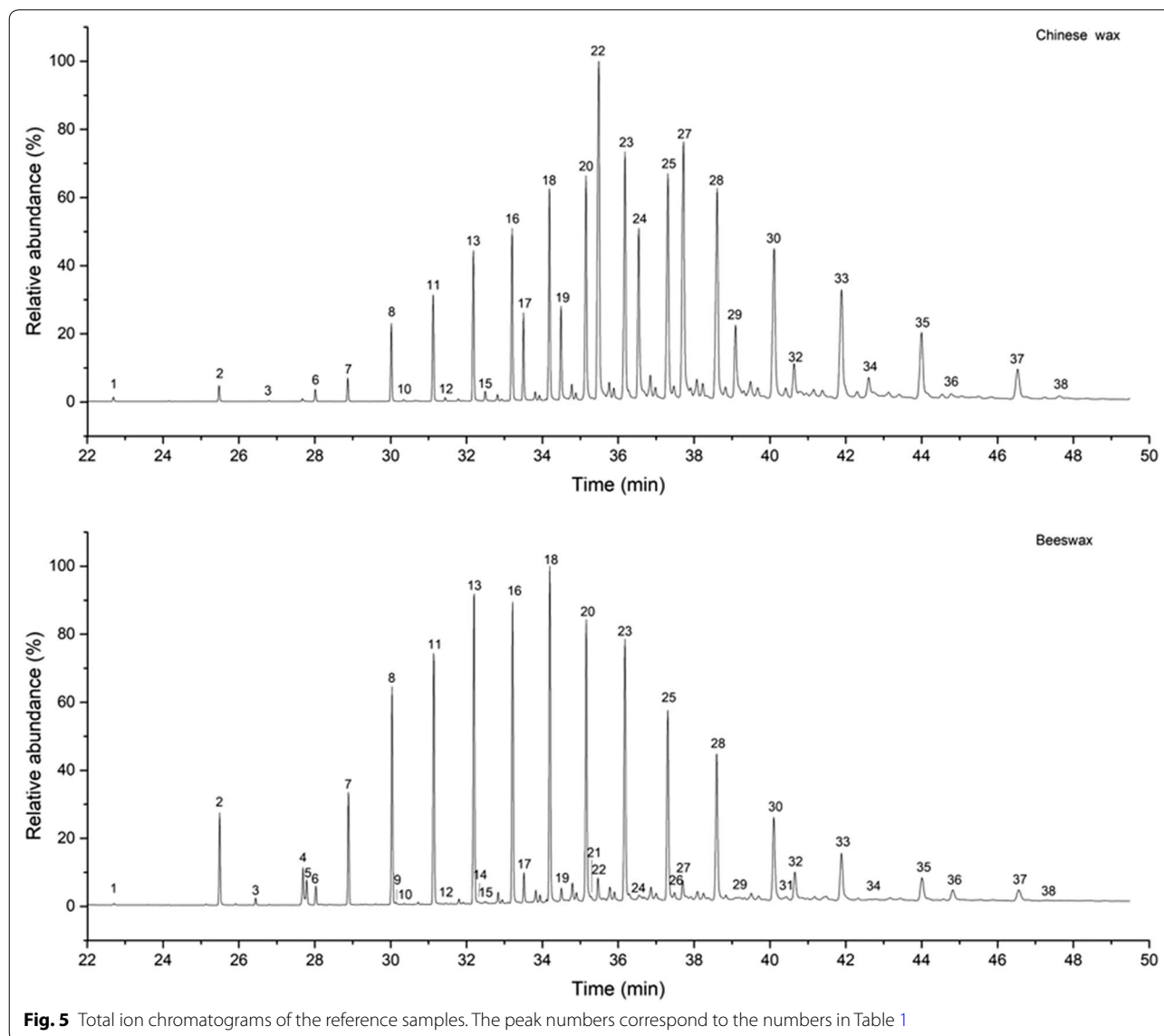## Pyrolysis–gas chromatography–mass spectrometry (Py–GC/MS)

The Py–GC/MS analyses were carried out on a pyrolyzer EGA/PY-3030D (Frontier Lab, Japan) attached to a GC–MS QP2010Ultra instrument (Shimadzu, Japan). The pyrolyzer furnace temperature was set at 550 °C, and the injector was set at 300 °C. The samples were held in the furnace for 6 s to achieve pyrolyzation completely. The initial chromatographic temperature was 50 °C, and the temperature was increased at 10 °C/min to 320 °C and then held for 20 min. The carrier gas was helium, and the split ratio was 1:100. The electron ionization voltage of the mass spectrometer was 70 eV. The temperatures of the interface and the ion source were 280 °C and 200 °C, respectively. The mass data was collected from m/z 30 to 750 with a scan time of 0.5 s.

Approximately 0.2 mg of the sample was placed in a sample cup, and 3 μl of 25% aqueous TMAH (tetramethyl ammonium hydroxide, analytically pure, Sinopharm Chemical Reagent Co., Ltd.) was added. The TMAH converted the fatty acids and alcohols into homologous methyl esters and methyl ethers, respectively. The NIST14 Library and NIST14 s Library were used to identify the compounds. The normalized areas were used to evaluate the peaks.

Results and discussion

FT-IR analysis results

The results of FT-IR analysis are shown in Fig. 4, and all the samples, including both the archaeological samples and the reference samples, exhibited similar spectra. The major infrared transmittance peaks appear at

Fig. 5 Total ion chromatograms of the reference samples. The peak numbers correspond to the numbers in Table 1

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wavenumbers (cm^{-1}) 2915, 2847, 1735, 1463, 1376, 1173, 730, and 718. Among these, the bands at 2847 cm^{-1} and 2915 cm^{-1} are associated with $-CH_2-$ stretching vibrations, indicating the presence of long-chain alkyl groups; the band at 1462 cm^{-1} represents bending vibrations of $-CH_2-$; the bands at 718 cm^{-1} and 730 cm^{-1} are from planar rocking vibrations of $-CH_2-$, and the appearance of two peaks indicates that there are crystallized compounds with long-chain alkyl groups; the band at 1735 cm^{-1} is from the stretching vibrations of the carboxyl groups of esters; the bands at 1173 cm^{-1} and 1130 cm^{-1} are from the stretching vibrations of C–O–C moieties, confirming the presence of esters. All the samples showed similar absorption peaks ranging from 1376 to 1195 cm^{-1}, and these bands can be attributed to out-of-plane vibrations of $-CH_2-$ in long-chain aliphatic groups. In addition,

Table 1 Compounds identified in the chromatograms of the reference samples

Peak number	Ret. time (min)	Identified compound	Original molecular formula	Peak area (%) Mean ± SD	
				Chinese wax	Beeswax
1	22.70	Tetradecanoic acid methyl ester	$C_{14}H_{28}O_2$	0.30 ± 0.16	0.05 ± 0.02
2	25.49	Hexadecanoic acid methyl ester	$C_{16}H_{32}O_2$	0.78 ± 0.39	2.67 ± 0.72
3	26.44	Heneicosane	$C_{21}H_{44}$	0.03 ± 0.03	0.13 ± 0.04
4	27.69	9-Octadecenoic acid methyl ester	$C_{18}H_{34}O_2$		0.57 ± 0.29
5	27.78	15-Methoxyhexadecanoic acid methyl ester	$C_{16}H_{32}O_3$		0.29 ± 0.12
6	28.02	Octadecanoic acid methyl ester	$C_{18}H_{36}O_2$	0.27 ± 0.13	0.67 ± 0.19
7	28.88	Docosane	$C_{22}H_{46}$	0.65 ± 0.07	2.56 ± 0.71
8	30.03	Tricosane	$C_{23}H_{48}$	0.57 ± 0.15	5.15 ± 1.42
9	30.13	17-Methoxyoctadecanoic acid methyl ester	$C_{18}H_{36}O_3$		0.02 ± 0.01
10	30.35	Eicosanoic acid methyl ester	$C_{20}H_{40}O_2$	0.06 ± 0.02	0.02 ± 0.01
11	31.13	Tetracosane	$C_{24}H_{50}$	2.09 ± 0.46	6.95 ± 1.38
12	31.45	Docosanol methyl ether	$C_{22}H_{46}O$	0.05 ± 0.03	0.01 ± 0.00
13	32.19	Pentacosane	$C_{25}H_{52}$	3.07 ± 0.43	9.03 ± 1.31
14	32.30	19-Methoxyeicosanoic acid methyl ester	$C_{20}H_{40}O_3$		0.02 ± 0.01
15	32.50	Docosanoic acid methyl ester	$C_{22}H_{44}O_2$	0.43 ± 0.18	0.04 ± 0.01
16	33.21	Hexacosane	$C_{26}H_{54}$	4.07 ± 0.48	9.42 ± 0.70
17	33.51	Tetracosanol methyl ether	$C_{24}H_{50}O$	1.25 ± 0.60	0.60 ± 0.23
18	34.19	Heptacosane	$C_{27}H_{56}$	4.07 ± 0.54	10.05 ± 0.48
19	34.50	Tetracosanoic acid methyl ester	$C_{24}H_{48}O_2$	3.66 ± 0.99	0.33 ± 0.09
20	35.15	Octacosane	$C_{28}H_{58}$	4.79 ± 0.58	9.23 ± 0.42
21	35.28	Methoxytetracosanediol methyl ethers	$C_{24}H_{50}O_2$		0.02 ± 0.01
22	35.46	Hexacosanol methyl ether	$C_{26}H_{54}O$	10.10 ± 2.20	0.60 ± 0.14
23	36.17	Nonacosane	$C_{29}H_{60}$	6.41 ± 0.77	9.26 ± 0.77
24	36.55	Hexacosanoic acid methyl ester	$C_{26}H_{52}O_2$	8.74 ± 3.93	0.16 ± 0.10
25	37.30	Triacontane	$C_{30}H_{62}$	6.83 ± 0.64	7.41 ± 0.88
26	37.48	Methoxyhexacosanediol methyl ethers	$C_{26}H_{54}O_2$		0.19 ± 0.01
27	37.69	Octacosanol methyl ether	$C_{28}H_{58}O$	9.56 ± 0.67	0.50 ± 0.10
28	38.59	Hentriacontane	$C_{31}H_{64}$	7.89 ± 0.15	6.78 ± 0.88
29	39.09	Octacosanoic acid methyl ester	$C_{28}H_{56}O_2$	4.36 ± 1.61	0.05 ± 0.02
30	40.10	Dotriacontane	$C_{32}H_{66}$	7.71 ± 1.42	4.98 ± 0.72
31	40.43	Methoxyoctacosanediol methyl ethers	$C_{28}H_{58}O_2$		0.14 ± 0.01
32	40.65	Triacontanol methyl ether	$C_{30}H_{62}O$	0.81 ± 0.40	1.09 ± 0.29
33	41.88	Tritriacontane	$C_{33}H_{68}$	5.46 ± 0.64	4.28 ± 1.02
34	42.62	Triacontanoic acid methyl ester	$C_{30}H_{60}O_2$	1.38 ± 0.51	0.04 ± 0.01
35	44.00	Tetratriacontane	$C_{34}H_{70}$	3.13 ± 0.51	3.15 ± 1.33
36	44.81	Dotriacontanol methyl ether	$C_{32}H_{66}O$	0.15 ± 0.06	0.48 ± 0.21
37	46.56	Pentatriacontane	$C_{35}H_{72}$	1.51 ± 0.23	3.04 ± 1.52
38	47.67	Dotriacontanoic acid methyl ester	$C_{32}H_{64}O_2$	0.33 ± 0.23	0.01 ± 0.00

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the band at 3300–3400 cm^{-1} indicates that alcoholic hydroxyl groups are present in the samples [24]. In conclusion, the main components of the samples are long-chain alkyl groups, including long-chain hydrocarbons, esters, fatty acids and alcohols, which indicates that the archaeological samples are wax [25, 26].

Although the infrared spectra obtained from the beeswax and Chinese wax were quite similar to those from the archaeological samples, there were small differences in the peaks at 1195–1376 cm^{-1}, 1730 cm^{-1}, and 3300–3400 cm^{-1}. The 1730 cm^{-1} peak of the archaeological residues is more intense than that of the reference samples, indicating that there are more free fatty acids in the archaeological samples. In addition, the peaks at 1195–1376 cm^{-1} and 3300–3400 cm^{-1} in the spectra of them are more intense than the corresponding peaks in the spectra of the reference materials, meaning that there are more long-chain aliphatic constituents in the archaeological samples. These differences indicate that some of the wax esters in the residues of bronze lamps degraded during burial [12]. However, classifying the kind of wax was still difficult, so further investigations using other complementary analytical techniques were needed.

Py–GC/MS analysis results

Following TMAH derivatization, the reference samples of beeswax and Chinese wax were analysed by Py–GC/MS. Figure 5 shows the total ion chromatograms, and the identified compounds with their retention times, as well as the peak areas (three parallel analyses were carried out for each sample) are reported in Table 1. Using the THM method, fatty acids and alcohols, including both free and ester-bound compounds,

can be converted into the corresponding methyl esters and ethers respectively, and this conversion is highly efficient [21]. In the case of beeswax, long-chain fatty acids, fatty alcohols and hydrocarbons were detected. The fatty acids present including linear monocarboxylic acids (saturated fatty acids ranging from C14 to C32, with C16 being the most abundant, and the unsaturated fatty acid C18:1) and linear ω-1-hydroxy fatty acids (ranging from C16 to C20, with the most abundant being 15-hydroxyhexadecanoic acid), and all with carbon atoms of even number. The fatty alcohols present including linear monohydric alcohols (ranging from C22 to C32, with the most abundant being C30) and linear α-(ω-1) diols (ranging from C24 to C28), also with carbon atoms of even number. The hydrocarbons present including linear alkanes (ranging from C21 to C35, with the most abundant being C27) with carbon atoms of both odd and even numbers, and the alkanes with carbons of odd numbers were more abundant than with even-numbered, and minor amounts of alkenes are present. The main chromatographic features of raw beeswax are in accordance with the literature [16, 19, 21]. Moreover, according to the research about fingerprints of beeswax from 10 provinces in China, the components vary slightly on relative amount as their fingerprint characteristics [27], meaning that although the beeswax samples have different origins, the main features are same.
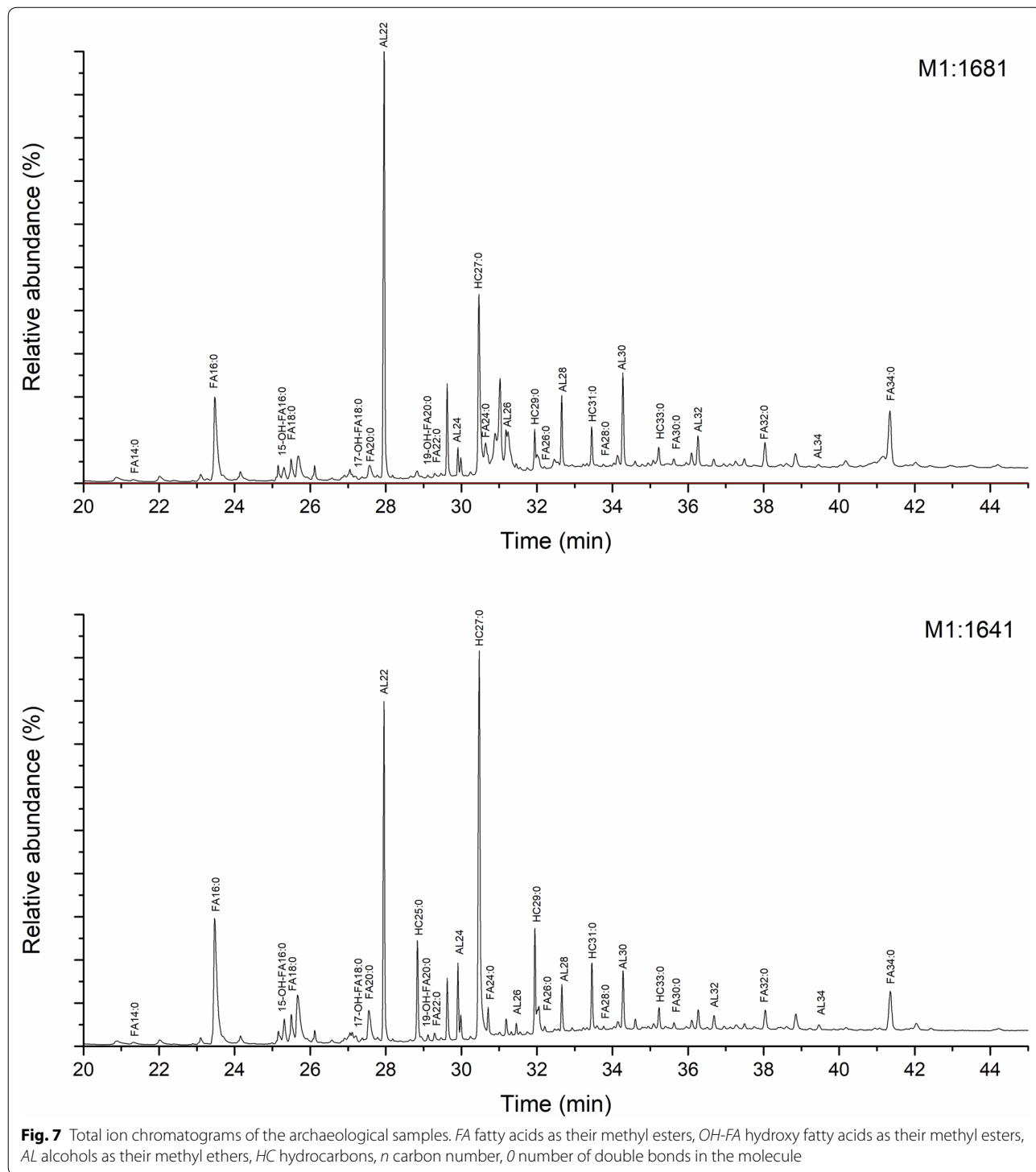
The main fractions of Chinese wax are similar to those of beeswax. Linear monocarboxylic acids with carbons of even numbers ranging from C14 to C32 were detected, and the most abundant species was C26:0. Linear monohydric alcohols with carbons of

Fig. 6 The relative abundances of fatty acids (left) and alcohols (right) in the reference samples
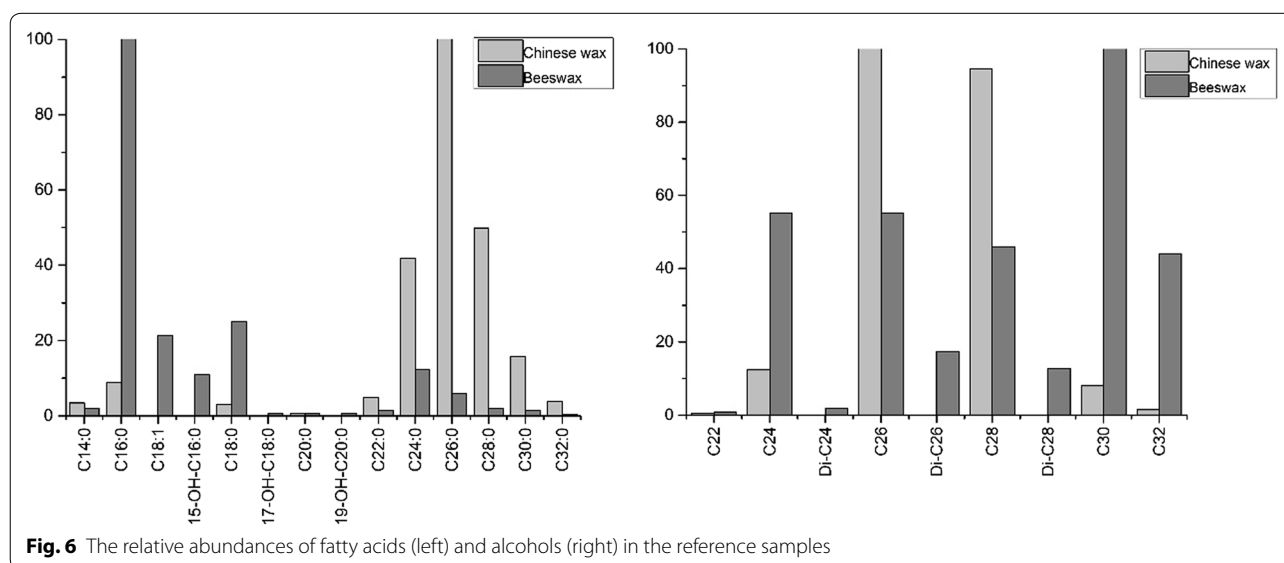
even numbers ranging from C22 to C32 were also identified, and the most abundant was C26. The observed long-chain hydrocarbons were primarily linear-chain alkanes, and both odd and even numbers of carbons were found. Triacontane and hentriacontane are the dominant species in Chinese wax, and the relative abundance of alkanes increased gradually from C21 to C29 and decreased from C30 to C35.

The main difference between Chinese wax and beeswax is the relative abundance of long-chain fatty acids, fatty alcohols and alkanes. In beeswax, the major compounds are hexadecanoic acid, triacontanol, and heptacosane,
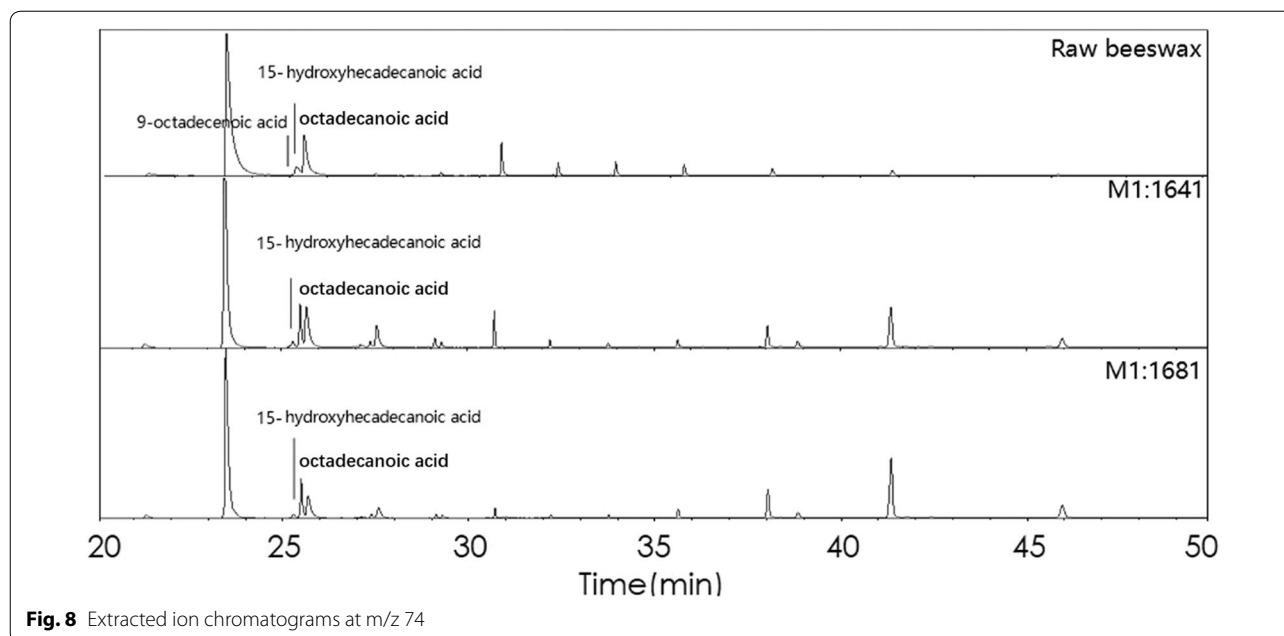

Fig. 7 Total ion chromatograms of the archaeological samples. *FA* fatty acids as their methyl esters, *OH-FA* hydroxy fatty acids as their methyl esters, *AL* alcohols as their methyl ethers, *HC* hydrocarbons, *n* carbon number, *0* number of double bonds in the molecule

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Fig. 8 Extracted ion chromatograms at m/z 74

while hexacosanoic acid, hexacosanol, and nonacosane are the dominant species in Chinese wax. In addition, linear ω-1-hydroxy fatty acids, 9-octadecenoic acid (C18:1), and linear α-(ω-1) diols are only found in beeswax but were not detected in Chinese wax (Fig. 6).

Furthermore, artificial ageing of the reference materials by heating has been performed. Through analysis, no differences between the unaged and aged samples were found, so only the results of unaged samples were reported in this paper.

The two archaeological samples were analysed using the same method as were used to evaluate the reference materials, and the chromatograms obtained by THM-Py–GC/MS are presented in Fig. 7. The main components detected in the samples of M1:1641 and M1:1681 are similar to those in the reference wax samples, which contained long-chain linear fatty acids, alcohols and hydrocarbons. However, compared with the reference materials, the amounts of hydrocarbons in the archaeological samples were significantly lower, while the amounts of fatty acids and alcohols were relatively higher. Nevertheless, the characteristics of the alkanes and alkenes were not sufficiently clear to identify the kind of wax, but the characteristics of the fatty acids were obvious. Saturated fatty acids with even numbers of carbons ranging from 14 to 34 were found in both archaeological samples, and among these species, the major compound was hexadecanoic acid (C16:0). Linear ω-1 hydroxy fatty acids were also detected with 15-hydroxyhexadecanoic acid being the major species, which is consistent with the known characteristics of beeswax. By comparison

with the reference samples, the fatty acid profiles of the archaeological samples (M1:1641 and M1:1681) are in accordance with beeswax (Fig. 8). 9-Octadecenoic acid, an unsaturated fatty acid characteristic of beeswax, was not found in the archaeological samples because the unsaturated carbon chain is easily broken [5].

Conclusion

The residues in bronze lamps excavated from Haihunhou Tomb were characterized using Fourier transform infrared (FT-IR) spectrometry and thermal-assisted methylation gas chromatography with mass spectrometry (THM-Py–GC/MS). The main components of the archaeological samples were identified. Through THM-Py–GC/MS analysis, the relative abundance of long-chain saturated fatty acids and the presence of ω-1-hydroxy fatty acids, can be used to differentiate beeswax and Chinese wax. The remains in the two lamps could be classified as beeswax by comparison with the reference samples. So Py–GC/MS with thermal-assisted hydrolysis and methylation is an effective method for assessing the type of wax, and minimum sample preparation is required. The depletion of hydrocarbons in the remains might be attributed to preservation in a warm environment [12]. According to the morphology of the remains in the lamps, which tightly fill the interior of the lamp plates, the wax was most likely melted during burning and solidified upon cooling. This may provide additional support for the hypothesized the usage of the lamps in the past. It can be concluded that the remains were candles made of

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beeswax. The Chinese historical literature (*西京杂记*) reports that the vassal king of Minyue (*闽越王*) paid 200 candles (approximately B.C. 200), which were made of beeswax, as a tribute to the emperor of the Western Han Dynasty. Liuhe was the emperor of the Western Han for 27 days (B.C. 92–B.C. 59) before being dethroned. Although we do not know whether the beeswax residues found in the tomb of Haihunhou were the tribute from the vassal king of Minyue, it is obvious that beeswax candles were very precious at that time. In addition, the typical forms of the bronze lamps, in which beeswax residues were collected, were only used by aristocracy in Han Dynasty, indicating the precious of the bronze lamps and the wax.

Authors' contributions
Professor SW and HH conducted the experiments and analysed the results. JY, LG, and WL excavated Haihunhou Tomb, collected the archaeological samples and provided the pictures of the lamps shown in Figs. 2 and 3. All authors reviewed the submitted article. All authors read and approved the final manuscript.

Availability of data and materials
The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Competing interests
The authors declare that they have no competing interests.

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