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Systematic Investigation into Evolution of Materials and Techniques Used in Lacquer Lian from the Warring States Period to the Yuan Dynasty

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Abstract: In order to investigate the evolution of Chinese lacquering techniques, seven pieces of lacquer Lian from the Warring States Period to the Yuan Dynasty (475 BC–1368 AD) were analyzed by means of cross-section observation, Raman spectroscopy (RS), and thermally assisted hydrolysis and methylation pyrolysis coupled with gas chromatography/mass spectroscopy (Py-GC/MS). The results revealed that the lacquer Lian consisted of a three-layer structure, encompassing a pigment layer on the surface, an undercoat layer in the middle, and a ground layer. The red mineral pigment utilized was cinnabar, while a combination of Chinese lacquer and drying oil served as the primary organic material. Although lacquering techniques had undergone minimal changes from the Warring States Period to the Yuan Dynasty, the species of drying oil had changed, based on the fact that boiled tung oil was found in the ground layer of lacquerware from the Song Dynasty and the Yuan Dynasty. The present research provides direct evidence for the inheritance and development of Chinese lacquer technology.

Keywords: Lacquer Lian; Py-GC/MS; Chinese lacquer; boiled tung oil; cross sections; lacquering techniques

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1. Introduction

Lacquer, a natural coating material for wood, porcelain, and metal, is tapped from lacquer trees growing in different regions of East and Southeast Asia: Rhus vernicifera (China, Japan, and Korea), Rhus succedanea (Vietnam and Taiwan), and Melanorrhoea usitata (Laos, Burma, Thailand, and Cambodia) [1,2]. The main component of lacquer is a mixture of catechol and phenol derivatives (60%–65%), proteins (glycoproteins (2%) and a laccase enzyme (1%)), polysaccharides (7%), and water (30%) [3]. Lacquer, when catalyzed by laccase, can undergo polymerization in the natural surroundings, resulting in the formation of a film characterized by an intricate three-dimensional network [4,5]. Exhibiting exceptional stability and possessing remarkable abilities to resist water, corrosion, and microbial growth, lacquer emerges as an exemplary environmentally friendly substance.

Lacquered artifacts have been revered and esteemed due to their unparalleled fortitude, enduring resilience, imperviousness, and exquisite beauty across a prolonged span of time. The utilization of lacquer throughout history can be traced back to the Neolithic era, and in the context of China's heritage, it can be classified into six distinct phases. The initial phase is the period of incubation, predominantly during the Neolithic epoch. The subsequent phase is the period of germination, transpiring during the Bronze Age, encompassing the Xia, Shang, and Zhou dynasties. This is followed by the phase of growth, occurring in the Iron Age, specifically during the Chunqiu Zhanguo dynasty. The fourth phase emerges as the heyday, characterized as the era of lacquer, flourishing primarily during the Qin and Han dynasties. The fifth phase, known as the recession stage, arises during the Buddhist era, spanning from the Wei to Tang dynasties. Ultimately, the final stage unveils itself as the pinnacle of prosperity within the porcelain era, comprising the Song, Yuan, Ming, and Qing dynasties [6]. Initial evidence of Chinese lacquerware can be traced back over 8000 years ago to the Kuahuqiao culture, where an ancient wooden bow delicately adorned with raw lacquer was unearthed [7]. A red lacquered wooden bowl was unearthed at Hemudu, a Neolithic site in the Yangtze River Delta, that can be traced back to 6000–7000 years ago [8]. In the present day, lacquerware products continue to hold their esteemed position as the most esteemed and sought-after handicrafts globally.

The red lacquer film commonly includes a subsequent mineral constituent such as iron oxide red (Fe_2O_3), lead red (Pb_3O_4) [9], or cinnabar (HgS). The primary constituents of the yellow lacquer film are orpiment or realgar. The black lacquer film frequently incorporates carbon black [10] or ferroferric oxide. Throughout the annals of lacquerware artistry, the red, yellow, and black colors have assumed significant roles. In this investigation, the main colors observed in the lacquer film specimens were red and black, aligning harmoniously with traditional aesthetics.

As we know, a number of modern analytical methods have been used to characterize lacquerwares, such as optical microscopy (OM) [11], scanning electron microscopy augmented by energy-dispersive X-ray spectrometry (SEM-EDS) [12], Raman spectroscopy [13], Fourier transform infrared spectrometry (FTIR) [14], and pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS) [15]. A large number of scientific studies have been undertaken to characterize Asian lacquer, focusing on molecular markers to identify the three lacquer species [16], the interactions between plant oils and lacquers [17], and the pathways of lacquer degradation [18]. Most research has been aimed at a single item of ancient lacquerware, several in one burial, or several in different burials but from the same period [19–22]. Although China was one of the earliest countries to make and use lacquerware, few studies about its inheritance and evolution of lacquering techniques have been reported. The sole extant treatise on the art of lacquering is *Xiushilu*, authored by Huang Cheng during the Ming Dynasty. Despite providing the groundwork for the exploration of lacquer techniques, the accounts within *Xiushilu* are regrettably concise, limiting our contemporary comprehension of the materials and methodologies employed in the creation of such exquisite lacquerware. Lacquer Lian was an important daily necessity in ancient times for Chinese women to store their toiletries, and is very common in unearthed cultural relics from the Warring States Period to the Tang and Song Dynasties, making it very suitable for research on the inheritance and evolution of Chinese ancient lacquer techniques. In this study, seven lacquer Lian samples from the Warring States Period, Han Dynasty, Song Dynasty, and Yuan Dynasty were analyzed via numerous modern analytical methods to investigate the types of lacquer, pigments, and drying oil added to the lacquer. The inheritance and evolution of Chinese ancient lacquer techniques is discussed by comparing the analysis results of seven lacquerware samples. Moreover, this study can also provide scientific support for the preservation and conservation of unearthed lacquerware.

2. Experimental

2.1. Archaeological Samples

Seven fragment samples of lacquer Lian used in this study were supplied by Jingzhou Museum, Yangzhou Museum, Changzhou Museum, and Jinsha site Museum, respectively. Two of the samples (Samples 1 and 2) dating back to the Warring States Period were unearthed from Jigongshan and Yangjiashan tomb, Hubei Province, respectively. Two samples (Samples 3 and 4) were traced back to Han Dynasty from Tianhui town tomb and Fenghuang mountain tomb, Sichuan Province. Two samples (Samples 5 and 6) dating back to the Song Dynasty were supplied by Yangzhou Museum and Changzhou Museum, respectively. The last one (Sample 7), tracing traced back to the Yuan Dynasty, was provided by Yangzhou Museum. Images of the lacquer fragments are shown Figure 1.

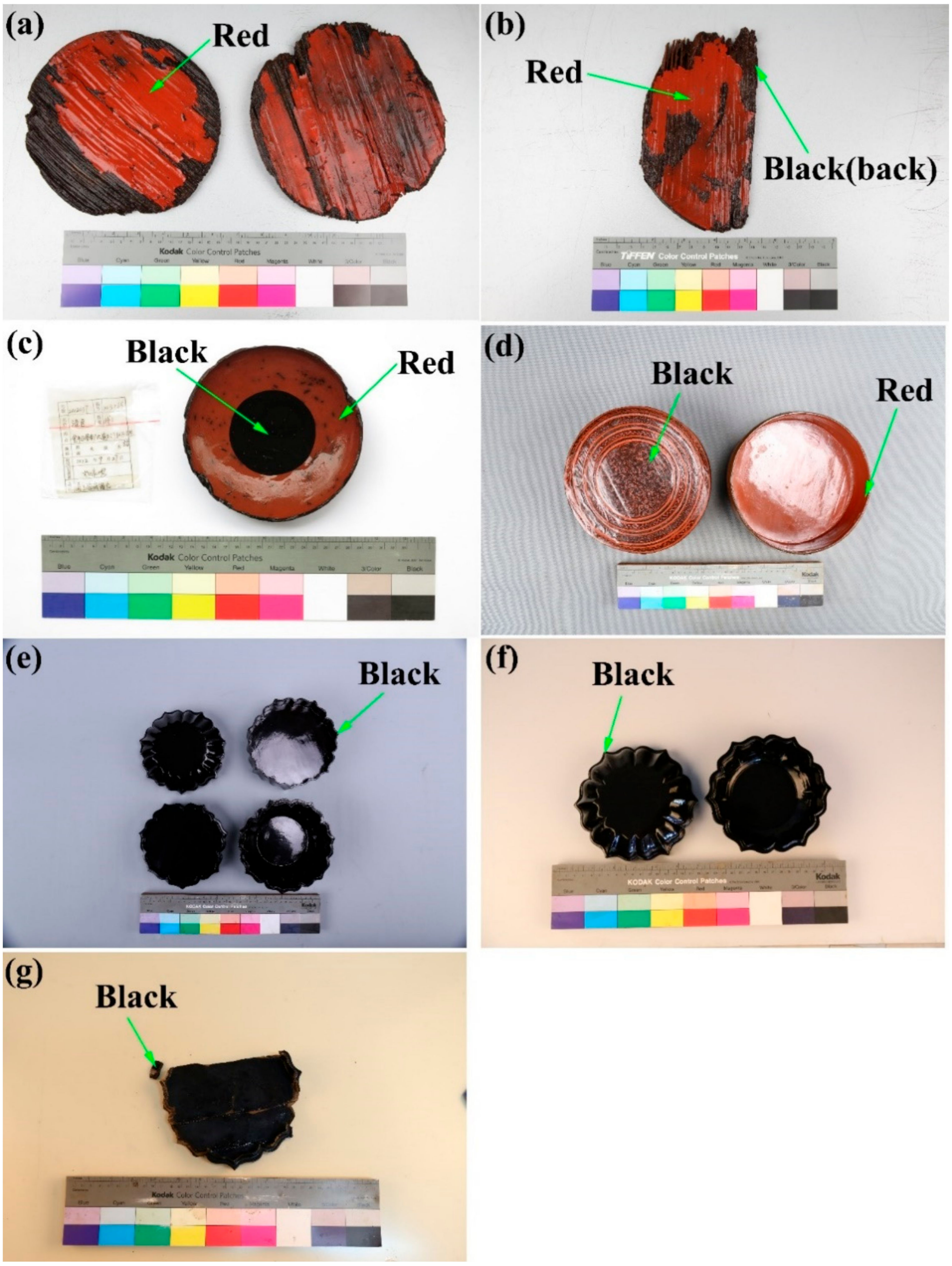


Figure 1. Images of lacquer fragments of (**a**,**b**) Warring States Period, (**c**,**d**) Han Dynasty, (**e**,**f**) Song Dynasty, and (**g**) Yuan Dynasty. The sampling locations are indicated by green arrows.

2.2. Analysis Methods

2.2.1. Cross-Section Observations

Lacquer samples (length × height, 10 mm × 5 mm) were embedded in epoxy resin and the bottom surface was polished with dry sandpaper (up to 12000#) after the epoxy resin was completely cured. The cross-section observations were carried out with an optical microscope (Axio Scope A1, Zeiss, Goettingen, Germany) under blue light (BL). The thickness of each layer was acquired by measuring ten data points using AxioVision software (Version 4.9) and calculating their average value.

2.2.2. Raman Spectroscopy

A micro confocal Raman spectrometer (LabRAM HR Evolution, HORIBA, Loos, France) was used to determine the mineral pigment in the lacquer film layers. The analysis was conducted using an excitation wavelength of 532 nm. The Raman spectra were acquired across the spectral range of 100–800 cm^{-1} with a resolution of 2 cm^{-1}, and subsequently cross-referenced with the RRUFF Raman spectroscopy databases.

2.2.3. Thermally Assisted Hydrolysis and Methylation Pyrolysis Coupled with Gas Chromatography/Mass Spectroscopy (THM-Py-GC/MS)

The pyrolysis–gas chromatography/mass spectroscopy measurements were carried out using a PY-3030D pyrolyzer (Frontier Lab, Fukushima, Japan) attached to a GCMS-QP2020 gas chromatograph mass spectrometer (Shimadzu, Kyoto, Japan). A stainless-steel capillary column (0.25 mm i.d. × 30 m) coated with 0.25 µm of 100% dimethylpolysiloxane was used for separation. Online methylation with additions of less than 1 mg sample and 5 µL 25% aqueous solution of tetramethyl ammonium hydroxide was used to obtain methylated phenolic hydroxyl groups. The sample was pyrolyzed at 500 °C for 0.2 min. The temperature of the pyrolyzer and GC interface was 300 °C. The initial temperature of the gas chromatograph oven was set to 50 °C, held at this temperature for 5 min, and then increased from 50 to 300 °C at 4 °C/min. The temperature of the gas chromatograph oven was then maintained at a constant temperature of 300 °C for 15.5 min. The temperatures of the injector and ion source were set to 300 and 230 °C, respectively. Helium was used as the carrier gas at a flow rate of 1.0 mL/min with a split ratio of 1:20. The electron ionization energy for mass spectroscopy was 70 eV, and the scanning range was from m/z 10 to 600 with full scan mode. Compounds were identified using comparisons between mass spectroscopy and the NIST library.

3. Results and Discussion

3.1. Cross-Sectional Analysis

The morphology of the cross sections of seven samples were examined under optical microscope. As shown in Figure 2, their cross sections showed similar three-layer structures, including a colored paint layer with red pigment or a surface finish layer (Layer 1), an undercoat layer (Layer 2), and a ground layer (Layer 3). Layer 1 boasted a more uniform thickness in comparison to Layer 2. The detailed thickness of the lacquer films of the analyzed archaeological samples is shown in Table 1. The difference in thickness between the red layer and the black layer is distinct, with the black layer being significantly thicker than the red layer (21.6 vs. 19.2, 40.8 vs. 29.9, 39 vs. 27.6). The red colored layer became thicker from the Warring States Period to the Han Dynasty (21.6, 19.2 vs. 29.9, 27.6).

Table 1. The detailed thickness of lacquer films of the analyzed archaeological samples.

Sample	Color	Layer 1 (µm)		Layer 2 (µm)	
		Average	Standard Deviation	Average	Standard Deviation
1	red	21.6	2.89	56.7	8.84
2	black	21.6	1.34	28.1	11.84
	red	19.2	5.28	22.7	10.44
3	black	40.8	6.15	-	-
	red	29.9	0.84	23.0	3.20
4	black	39.0	3.92	11.7	1.74
	red	27.6	2.69	5.1	0.97
5	black	43.8	2.45	78.9	20.67
6	black	31.6	1.94	9.9	4.59
7	black	45.3	5.75	35.1	10.86

The colored paint layer, known to be a mixture of pigment, drying oil, and lacquer liquid, was not only used to decorate the lacquerware items, but also could increase the covering power of the lacquer film, prevent ultraviolet rays from penetrating the lacquer film, and delay the aging of the lacquer film. It is located on the top layer of the lacquerware, as shown in Figure 2a,c,e,g. The undercoat layer under the surface layer, which was usually a mixture of lacquer and drying oil, was used to cover defects on the next layer, such as the second layer in Figure 2, which resulted in uneven thickness. It can also be used as the surface finish layer for the original color of raw lacquer, as shown in Figure 2b,d,f,h–j. A mixture of clay, lacquer, and oil, known as the ground layer, was used to fill the pores of lacquer bodies, as shown in Figure 2. As an important daily necessity in ancient times, the manufacturing process of lacquer Lian involved a three-layer structure, which was a commonly used technique in ancient Chinese lacquerware such as ear cups [19].

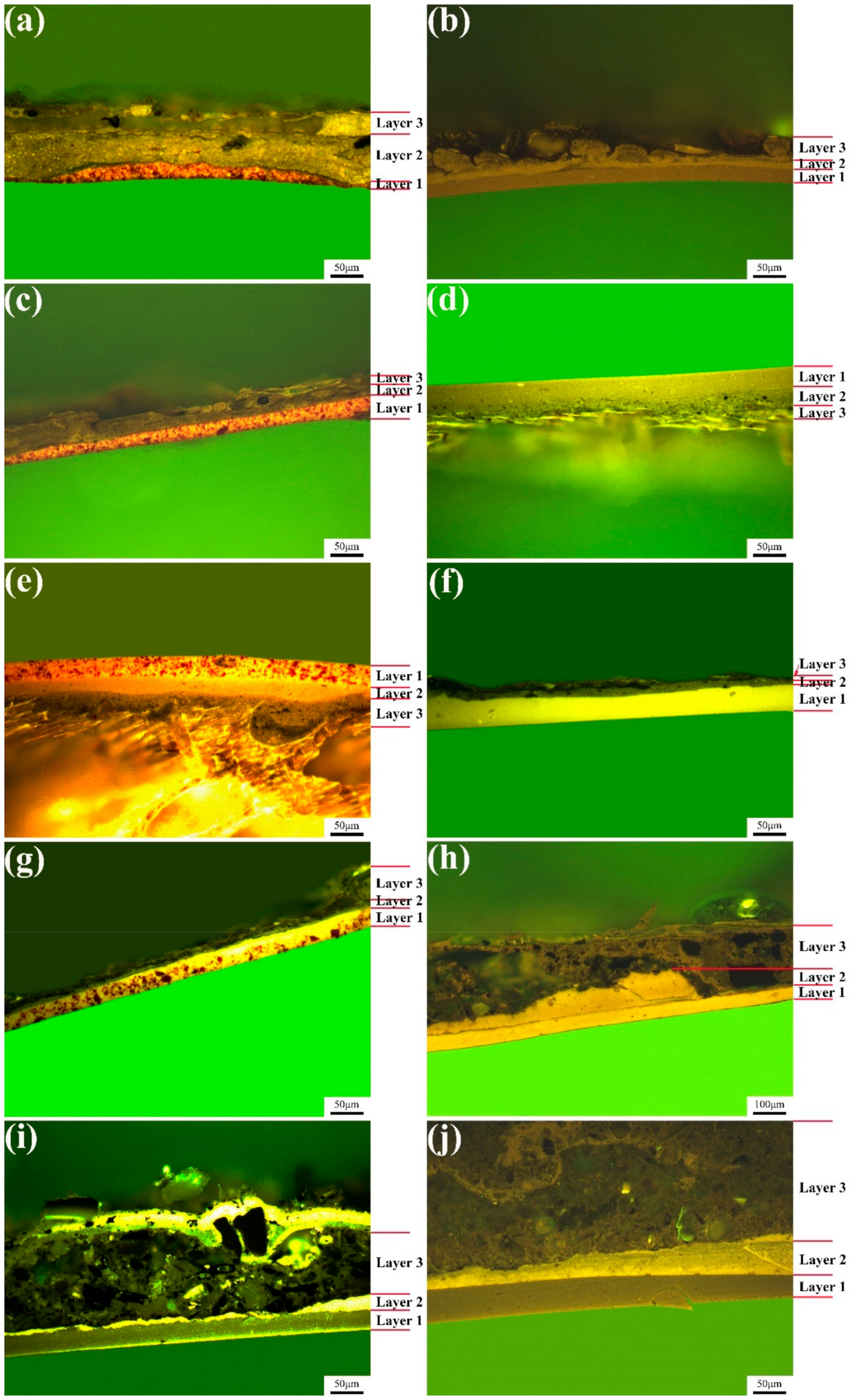


Figure 2. Cross-sectional photos of (**a**) Sample 1, (**b**) Sample 2 (black), (**c**) Sample 2 (red), (**d**) Sample 3 (black), (**e**) Sample 3 (red), (**f**) Sample 4 (black), (**g**) Sample 4 (red), (**h**) Sample 5, (**i**) Sample 6, and (**j**) Sample 7 under blue light.

As shown in Figure 2, the lacquering techniques of lacquer Lian underwent minimal changes during nearly 1800 years from the Warring States Period to the Yuan Dynasty. Firstly, the ground layer was painted on the lacquer bodies to fill the pores. Secondly, the undercoat layer was used to cover the defects of the ground layer. Lastly, a colored layer or a lacquer layer was used as the surface finish layer to decorate the lacquerware. However, the thickness of the same layer in different eras varied greatly; for instance, the red colored layer became thicker from the Warring States Period to the Han Dynasty, as discussed above.

3.2. Pigment Analysis

According to Xiushilu, cinnabar, red ochre, and crimson melanterite could be used to make red lacquer. Raman analysis was used to clarify the minerals in the red pigments. Figure 3 shows the Raman spectra of the red pigments. All the four red pigments show similar Raman spectra, with peaks at about 254 and 343 cm^{-1}, which are characteristic bands of cinnabar. Due to its excellent color, gloss, and anti-corrosion properties, cinnabar is the best choice for making red lacquer. The results were consistent with previous studies [12,23].

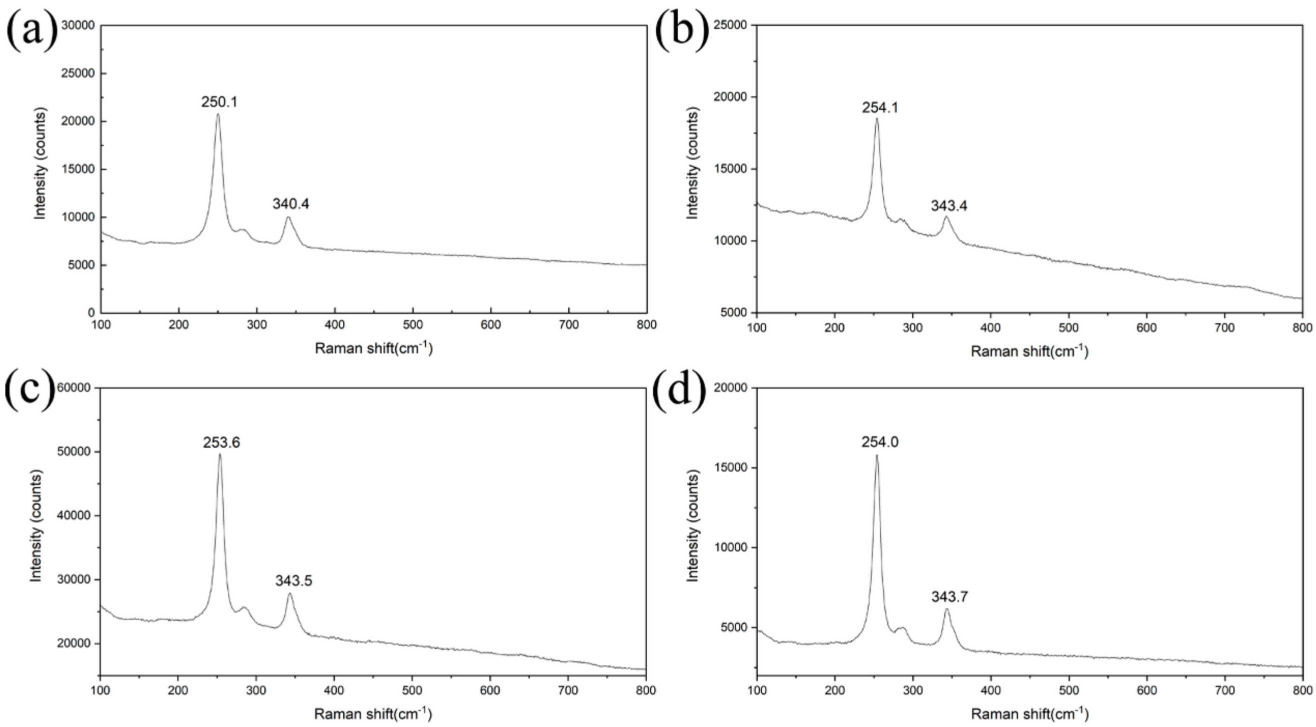


Figure 3. Raman spectra of red pigments of (**a**) Sample 1, (**b**) Sample 2, (**c**) Sample 3, and (**d**) Sample 4.

3.3. Lacquer Film Analysis

In order to identify the types of lacquer and oil in different layers of the seven samples, THM-Py-GC/MS analysis was carried out. All the seven samples showed similar chromatographic profiles; only the chromatographic profile of the red layer of sample 1 is presented in Figure 4, and the rest are shown in Supplementary Materials. The presence of lacquer was assessed by using EICs, as shown in Figure 4. Characteristic pyrolysis products were confirmed. Aliphatic hydrocarbons (C, m/z 55 and 57, Figure 4b,c) are present from 1-Decene (C9:1) to 1-Pentadecene (C15:1), peaking at 1-Tetradecene (C14:1), and present from decane (C10) to pentadecane (C15), peaking at pentadecane (C15); the alkylbenzenes (B, m/z 91, Figure 4d) show a decreasing profile from benzene propyl- (B3) to benzene octyl- (B8). These results are identical to those of urushi [24]. In addition, 1,2-Dimethoxy-3-pentadec-8-enylbenzene (P1, 3-pentadecenyl-catechol) and 1,2-Dimethoxy-3-pentadecylbenzene (P2, 3-pentadecyl-catechol) were detected in the red layer; both are characteristic components of urushi [25,26]. Based on the above results, it could be concluded that the lacquer of the seven samples was Chinese lacquer (urushi).

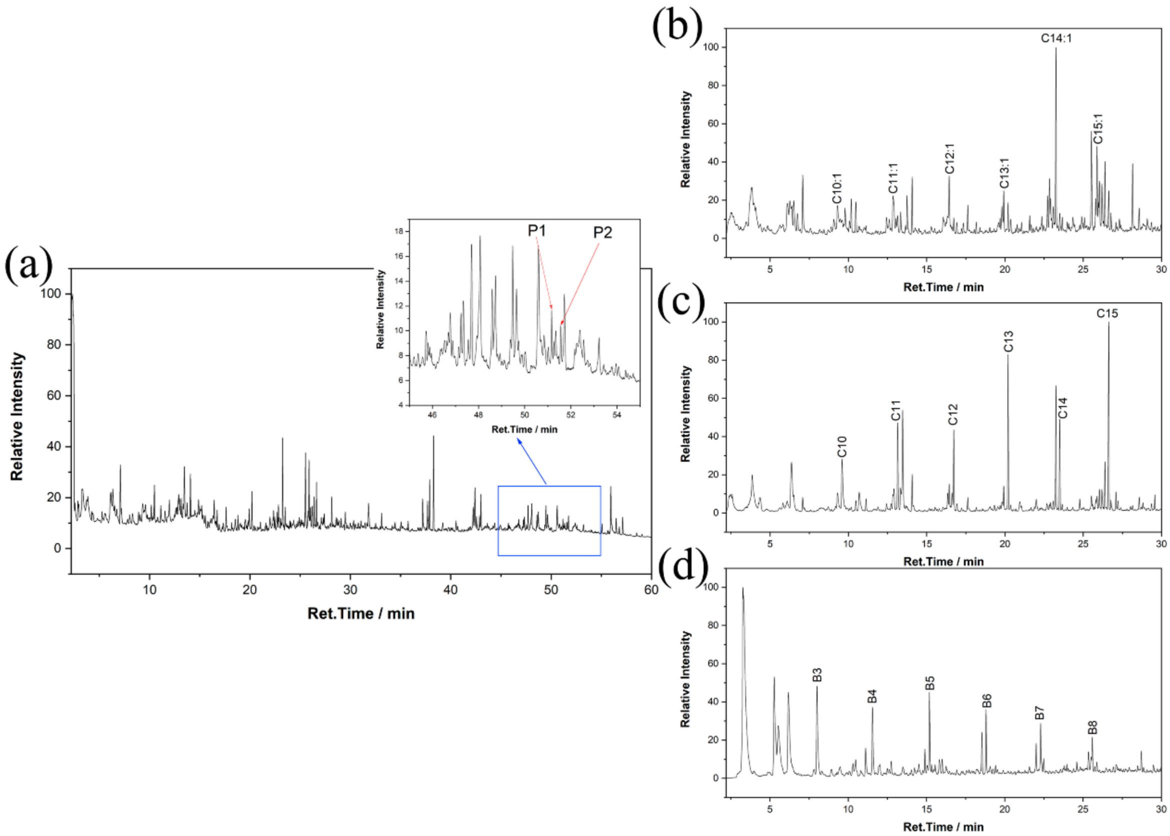


Figure 4. Chromatographic profiles obtained using THM-Py–GC/MS of red layer of sample 1: (**a**) total ion pyrogram; (**b**) m/z 55 extracted ion pyrogram; (**c**) m/z 57 extracted ion pyrogram; (**d**) m/z 91 extracted ion pyrogram.

Figure 5 shows the relative concentration of fatty acids detected from the red layers. It can be seen that a large number of mono-carboxylic acids and di-carboxylic acids were detected, especially nonanedioic acid (C8) and octanedioic acid (C9), which are typical pyrolytical and aging products of dying oil. These results indicate that drying oil was used in the red layer at least during the Warring States Period, which is identical to previous reports [19]. Lacquer could be easily mixed with pigments by drying oil to obtain colorful lacquerware.

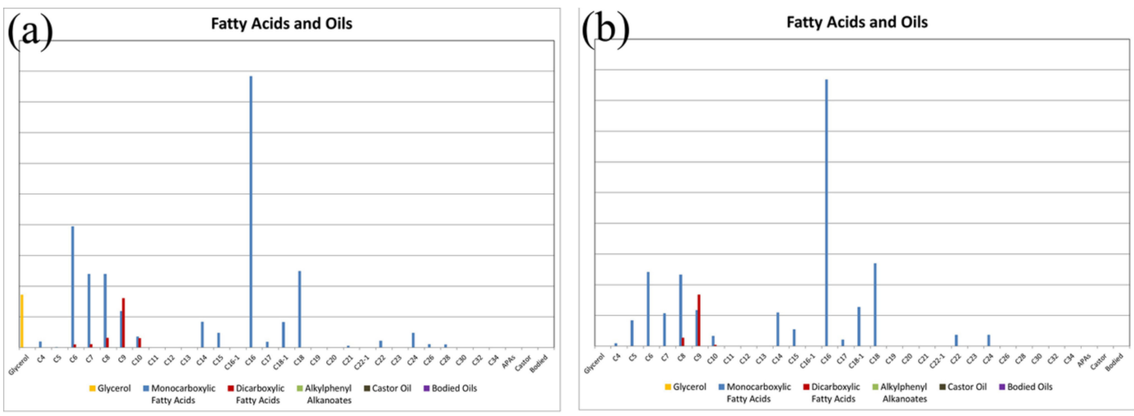


Figure 5. *Cont.*

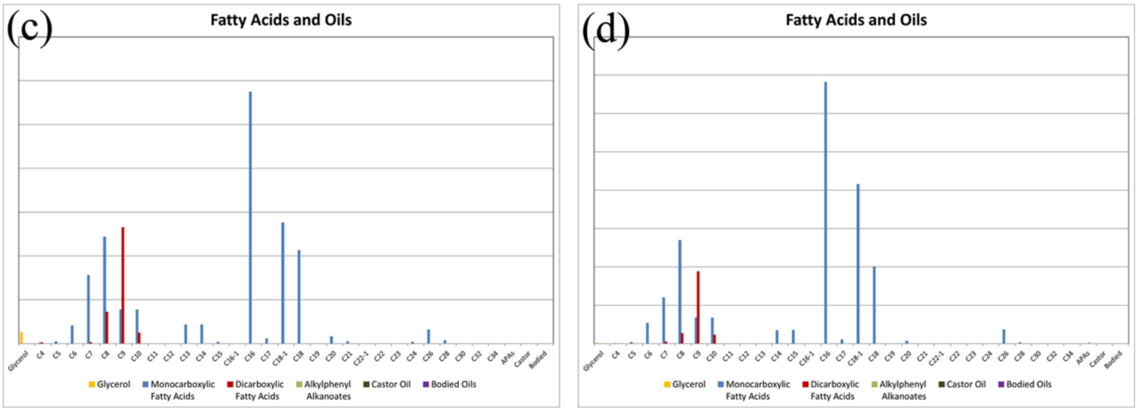
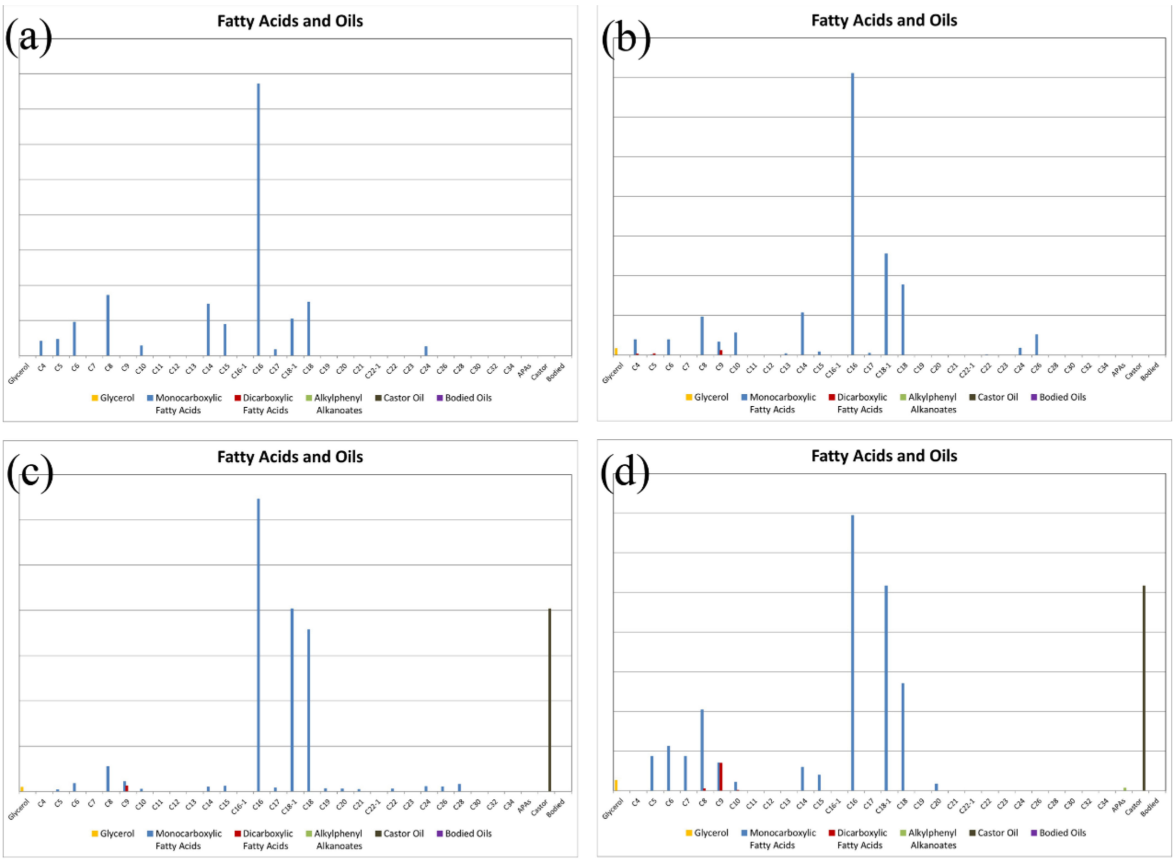


Figure 5. The relative concentration of fatty acids in red layers of (**a**) Sample 1, (**b**) Sample 2, (**c**) Sample 3, and (**d**) Sample 4.

Figure 6 shows the relative concentration of fatty acids detected from black layers. There is no drying oil in the black layers of lacquer Lian from the Warring States Period, as shown in Figure 6a,b. Castor oil, which is one kind of drying oil, was detected in the black layers of lacquerwares from the Han Dynasty and Song Dynasty, as shown in Figure 6c,d. Figure 6e,f indicate that there is drying oil in the black layers of lacquerwares from the Song Dynasty and Yuan Dynasty. Drying oil can decelerate the hardening process of lacquer while enhancing the luster and flexibility of the lacquer, thus elevating the quality of lacquerware.



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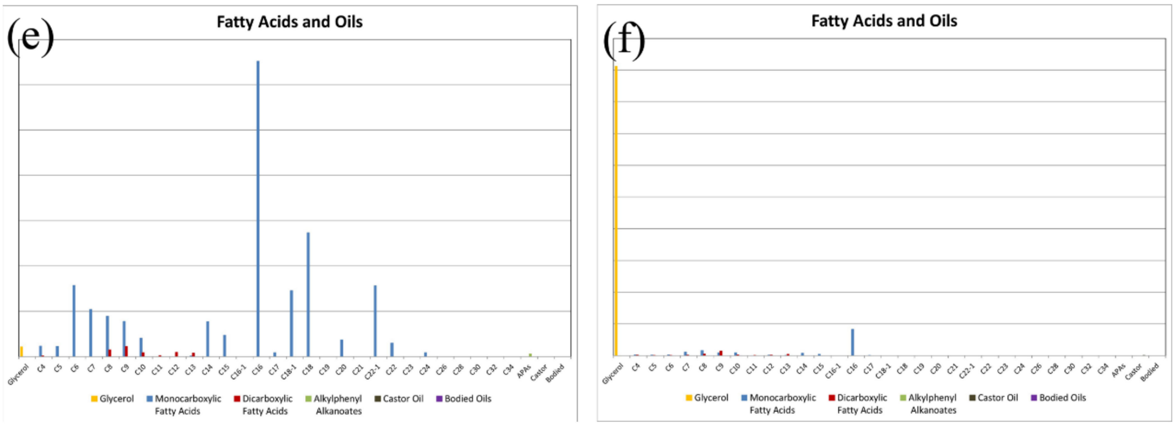

Figure 6. The relative concentration of fatty acids in black layers of (**a**) Sample 2, (**b**) Sample 3, (**c**) Sample 4, (**d**) Sample 5, (**e**) Sample 6, and (**f**) Sample 7.

The relative concentration of fatty acids detected from the ground layers is shown in Figure 7. Drying oil was used in the ground layer for Sample 1, but not for Sample 2, as shown in Figure 7a,b. Figure 7c,d shows that drying oil was widely used in ground layers in the Han Dynasty. Castor oil was detected in the ground layer of Sample 5 (Figure 7e). Since methyl alkylphenyl alkanoate (APA) is the marker component of boiled tung oil, it could be concluded that boiled tung oil was used in the ground layers of Samples 6 and 7, based on the results of Figure 7f,g. Boiled tung oil, serving as an organic binder for ground layers, emerged in the Song Dynasty at the latest, in accordance with other findings [27].

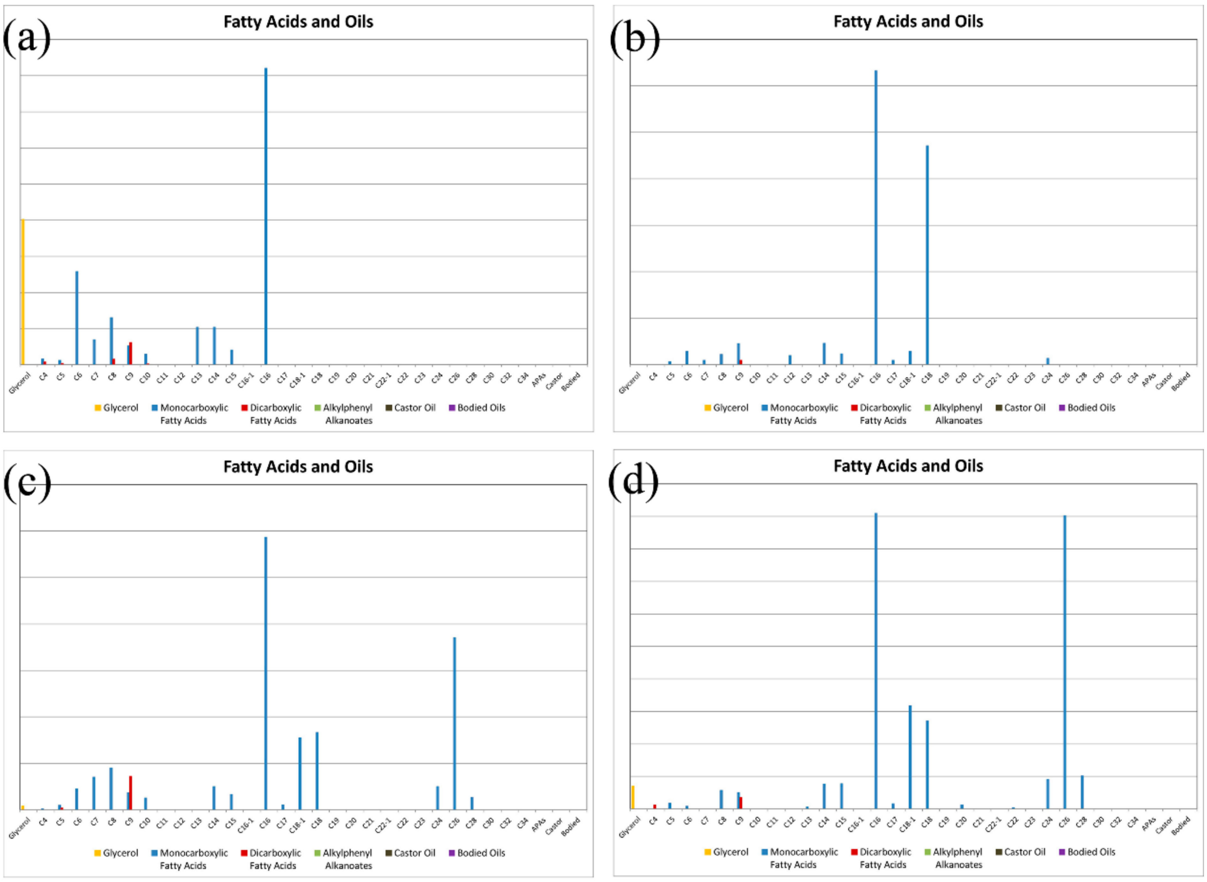

Figure 7. *Cont.*

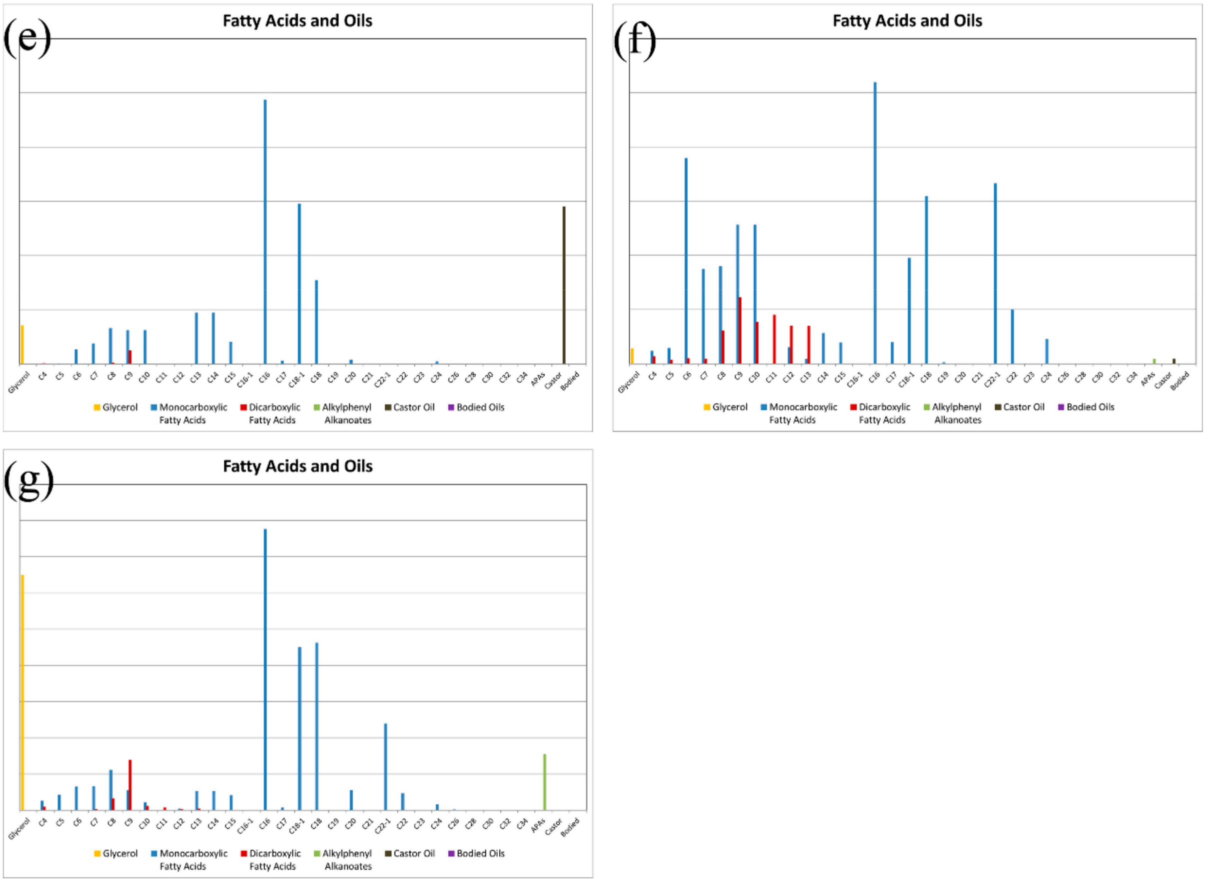

Figure 7. The relative concentration of fatty acids in ground layers of (**a**) Sample 1, (**b**) Sample 2, (**c**) Sample3, (**d**) Sample 4, (**e**) Sample 5, (**f**) Sample 6, and (**g**) Sample 7.

4. Conclusions

Seven lacquer Lian samples from the Warring States Period, Han Dynasty, Song Dynasty, and Yuan Dynasty were comprehensively investigated using a scientific analytical approach. The cross-section photos indicated that the lacquer Lian included a pigment layer, undercoat layer, and ground layer, and lacquering techniques underwent minimal changes from the Warring States Period to the Yuan Dynasty, which indicates the inheritance of lacquer techniques. Raman analysis clarified that the red pigment was cinnabar. The THM-Py-GC/MS results indicated that all the lacquerware items were coated with lacquer sap collected from a Rhus vernicifera lacquer tree. Drying oil was mixed with lacquer sap to increase the luster and elastic of lacquer film. Boiled tung oil was found in the ground layer of lacquerware from the Song Dynasty and Yuan Dynasty, reflecting the development of lacquer materials. This study not only provides a better understanding of the inheritance and evolution of Chinese lacquering techniques and lacquering materials, but also provides scientific support for the preservation and conservation of unearthed lacquerware.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/coatings13101750/s1.

Author Contributions: H.W., the lead author, carried out the Raman experiments on the samples, analyzed the data, and designed the article. The second author, Y.Z., carried out THM-Py-GC/MS of the samples. The third author, B.F., provided archaeological samples and their archaeological information. The corresponding author and the fourth author, J.D., designed the article and funded the research. All authors have read and agreed to the published version of the manuscript.

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