

## Article

# Identification of Coatings on Persian Lacquer Papier Mache Penboxes by Fourier Transform Infrared Spectroscopy and Luminescence Imaging

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**Abstract:** In this study, Fourier transform infrared spectroscopy (FTIR) and luminescence imaging were used to identify the coatings of seven Persian lacquer papier mache penboxes, of which two were contemporary, one was from the Pahlavi era, and four belonged to the Qajar era. First, FTIR was used to identify the nature of the coating. Then, UV-induced visible luminescence imaging at the spectral ranges of 420–680 nm (UVL), 425–495 nm (UVIBL), and 615–645 nm (UVIRL) was performed for further examination. The FTIR results showed that the coatings were made of alkyd resin, oil-resin varnish (Kaman oil), and shellac. In UV-induced visible luminescence images, synthetic alkyd resin showed no fluorescence, which made it distinguishable from the natural organic coatings. While it is slightly challenging to differentiate Kaman oil from shellac based on FTIR results, these two coatings can be easily distinguished by their fluorescence in UVL and UVIBL images. The results suggest that the combined use of spectroscopy and spectral imaging methods can provide substantial information about the organic coatings of historical objects.

**Keywords:** lacquer papier mache; historical organic coatings; kaman oil; shellac; alkyd resin; FTIR spectroscopy; spectral imaging; luminescence imaging



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## 1. Introduction

Persian lacquer objects, especially penboxes (*Qalmadan* in Persian), are among valuable Persian historical and artistic objects that have survived in significant numbers from multiple historical periods. Despite their global renown as lacquer art, as some researchers have pointed out, these lacquerwares are essentially painted surfaces that are coated with a protective oil or resin [1,2]. In addition to its protective role, this coating often contributes to the aesthetic value of the coated object. However, research into lacquerwares has been generally focused on their historical significance, fabrication technology, and illustrations. This is why the identification of organic coatings is also an important part of the study of these historically and culturally significant objects [3–8].

In the case of penboxes, these coatings used to comprise a thin layer of oil-resin veneer. According to historical records, this veneer, generally referred to as “Kaman oil”, used to be prepared by mixing a terpenoid resin, typically sandarac, with linseed oil at boiling temperature. The use of this coating in Persian artworks dates back to at least the 11th century. From the 18th century onwards, Persian artists started to use shellac as well as Kaman oil [9], until the twentieth century, when synthetic resins, especially alkyd, became common. While the process of making papier mache penboxes has not changed much over these centuries, the coatings applied to these objects have changed depending on circumstances [10].

Given the limitations in taking samples from such objects, how often they are recoated or restored, and how often they may have lost a significant part of their original coating, the identification of these original coatings is one of the main challenges for researchers. There

are a variety of methods for identifying historical resins and coatings, including techniques based on chromatography and mass spectrometry, which are relatively expensive [11–16]. However, more affordable spectroscopic methods such as Raman spectroscopy and especially Fourier transform infrared spectroscopy also have a good track record in identifying the molecular structure of complex organic compounds like those of coatings used on historical objects [17–24]. With these methods, it is possible to study a wide range of synthetic and natural organic oils, resins and coatings very quickly and at reasonably low cost [25].

This identification can also be done with the help of ultraviolet fluorescence examination. The use of ultraviolet light sources to examine visible fluorescence emissions from the outermost layer of painted artworks dates back to the 1920s [26–30]. Despite recent advances in instrumental analysis methods, this method is still one of the fundamental techniques for evaluating the quality of coatings based on their fluorescence in the visible region [31]. However, the results of this method depend on a variety of factors including the experimental process, the post-processing method, and the data usage procedure [32,33]. While ultraviolet imaging allows us to collect data from an object without physical contact, it cannot provide definite results for complex coatings. Therefore, recent years have seen more interest in the compilation of ultraviolet fluorescence databases for artwork materials [34,35].

In this study, we used Fourier transform infrared spectroscopy (FTIR) and spectral imaging to identify the coatings of multiple historical and contemporary lacquer papier mache penboxes in order to determine whether these methods are suitable for the systematic study of such historical and artistic objects. The results of this study can pave the way for the wider use of non-destructive imaging methods in the technical study of historical lacquer objects.

## 2. Materials and Methods

### 2.1. Samples

The study was performed on seven Persian lacquer papier mache penboxes (Figure 1). The length of these penboxes varied from about 20 to 25 cm. In terms of age, the penboxes labeled LP1 and LP2 were contemporary, LP3, LP4, LP5, and LP6 belonged to the Qajar period (late 18th to early 20th century), and LP7 belonged to the first Pahlavi period (2nd to the 4th decade of the 20th century). Despite some conservation actions done on these penboxes, they still had their original coatings, making it possible to determine whether such coatings can be identified by FTIR spectroscopy and spectral imaging methods.



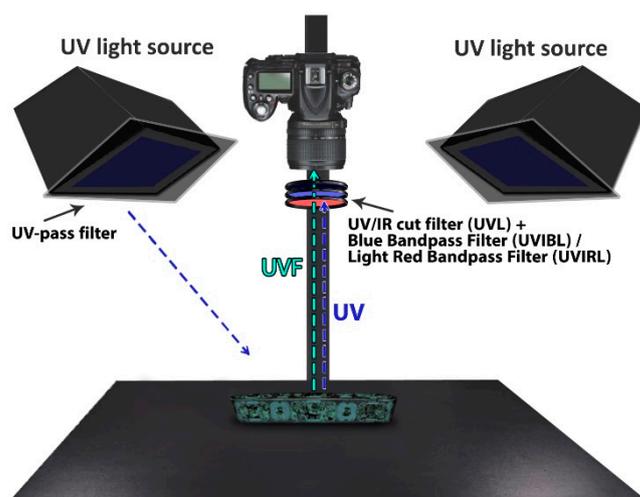
Figure 1. The studied lacquer papier mache penboxes and their codes.

## 2.2. Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) was used for the structural study of the coating applied to the papier mache base. Before sampling, the surfaces of the penboxes were cleaned with ethanol. Sampling was performed with a scalpel. The samples were analyzed using the KBr pellet method with an FT/IR-680Plus spectrometer (Jasco, Tokyo, Japan). The spectra were recorded in 32 scans with a resolution of  $4\text{ cm}^{-1}$  in the range of  $400\text{--}4000\text{ cm}^{-1}$ . The spectra of Indian shellac, alkyd resin, and a historical sample of Safavid Kaman oil were also recorded to be used as the reference.

## 2.3. Luminescence Imaging

All images were taken with a modified D750 camera (Nikon, Tokyo, Japan) equipped with an AF Nikkor 50 mm f/1.8D lens. To take full advantage of the CMOS sensor (350–1100 nm), the internal UV-IR block filter was removed. Following the instructions of Verri [36], two electronic xenon flashes equipped with a U360 UV beam filter (Hoya, Tokyo, Japan) fixed at a 45-degree angle to the subject were used as the light source (Figure 2). Visible reflected images (VIS) and visible luminescence images were recorded in RAW format at the highest possible resolution (24 megapixels:  $6016 \times 4016$ ) using the filters described in Table 1 and then converted to 16-bit TIF format in Adobe Photoshop (Adobe, San Jose, CA, USA). Post-processing and calibration procedures were performed using the Kushel method [37] and as instructed by Cosentino [38]. In the UVL1 method, PTFE was used as the white balance reference.



**Figure 2.** Arrangement of the camera, light sources, penboxes, and filters used in UV fluorescence imaging.

**Table 1.** Radiation sources and filters used in each imaging method.

Imaging Technique	Filter(s) in Front of Radiation Sources	Filter(s) in Front of Camera	Range Investigated
Visible-reflected Imaging (VIS)	2 × Youngenu NY660 Xenon flashlight (without filter)	Baader UV/IR Cut	420–680 nm
UV-induced visible luminescence imaging (UVL)	+2 × Hoya U-360	Baader UV/IR Cut	420–680 nm
UV-induced Blue luminescence imaging (UVIBL)	+2 × Hoya U-360	Baader UV/IR Cut + MidOpt BP470	425–495 nm
UV-induced Red luminescence imaging (UVIRL)	+2 × Hoya U-360	Baader UV/IR Cut + MidOpt BP635	615–645 nm

### 3. Results and Discussion

#### 3.1. FTIR Spectroscopy

Until the 18th century, the coating used in the majority of Persian lacquer works was Kaman oil [39,40]. However, starting from the Safavid era, the use of shellac as an alternative coating also became common. In the 20th century, an increasing number of Persian artists began to use alkyd resins in their work. While acrylic resins have been occasionally used in the restoration of other coatings, in such cases, the structural differences are quite easily distinguishable in FTIR, but it is fairly difficult to distinguish Kaman oil from shellac, alkyd, and triterpenoid varnishes that some artists have used as its substitute. However, given the differences in the FTIR spectra characteristics of these materials, several researchers have suggested using these spectra to distinguish them from another.

The FTIR spectra of the studied samples are illustrated in Figure 3. Initial examinations showed a clear difference between LP7 and other samples. In the spectrum of LP7, the multiple peaks belonging to OH groups at  $3470\text{ cm}^{-1}$ , symmetric and asymmetric stretching vibrations of  $\text{CH}_2$  at  $2925$  and  $2855\text{ cm}^{-1}$ , bending vibrations of  $\text{CH}_2$  at  $1457\text{ cm}^{-1}$ , and the strong peak related to the stretching vibration of  $\text{C}=\text{O}$  at about  $1733\text{ cm}^{-1}$  are among the characteristics of alkyds. This spectrum also shows the bending vibrations of  $\text{C}-\text{H}$  and the stretching vibrations of  $\text{C}-\text{O}$  and  $\text{C}-\text{C}$  at  $1124\text{ cm}^{-1}$  and especially a strong peak at  $1268\text{ cm}^{-1}$  (probably related to the vibrations of  $\text{C}-\text{O}-\text{C}$  esters). The absorption bands  $1603$ ,  $1071$ ,  $704$  and  $742\text{ cm}^{-1}$  have also been attributed to the stretching and bending vibrations of the aromatic ring and aromatic  $\text{CH}$  in alkyds [41–44]. Comparison of this spectrum with the reference alkyd spectrum also showed the remarkable similarity of the two, further indicating that the coating applied on LP7 is alkyd. Furthermore, a weak absorption band at about  $3020\text{ cm}^{-1}$  was only observed in this spectrum and the reference alkyd spectrum.

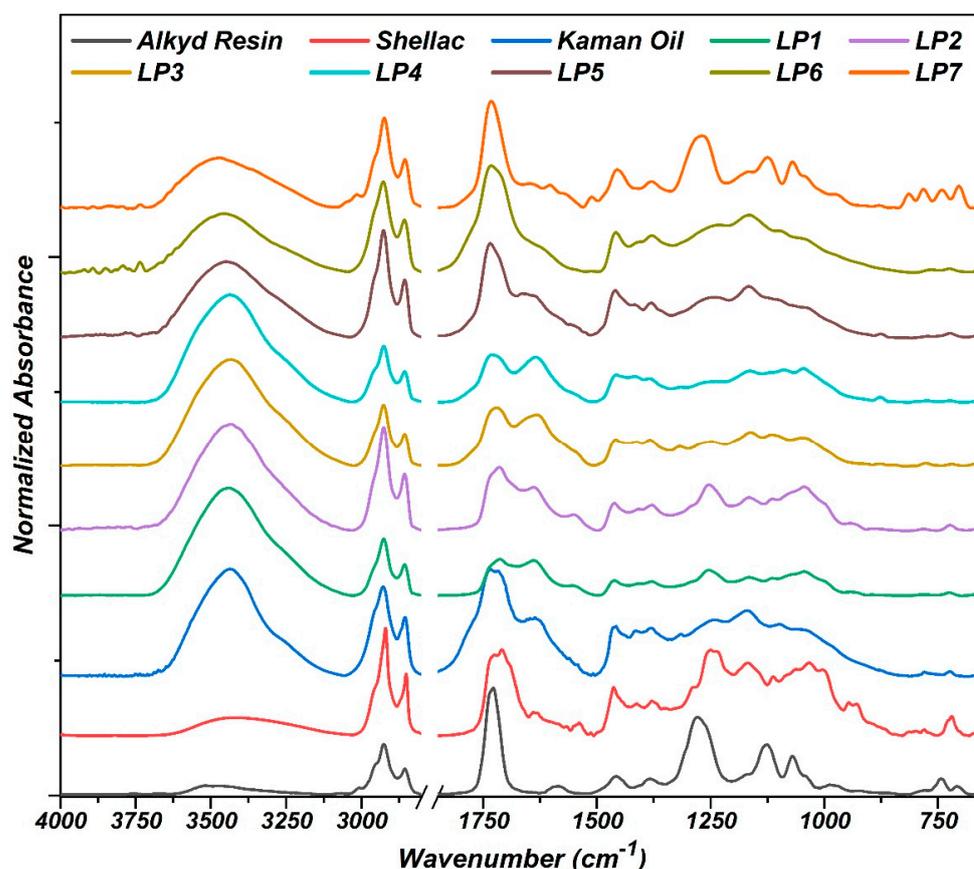
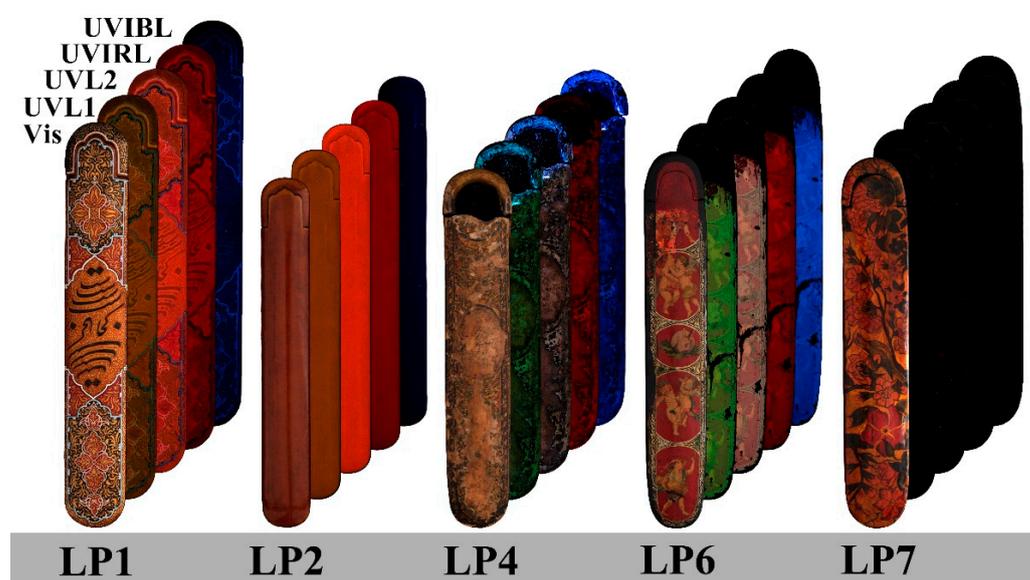


Figure 3. FTIR spectra of the studied samples and references in the range of  $650\text{--}4000\text{ cm}^{-1}$  after baseline correction.

For LP1 and LP2, the spectra showed a weak twin absorption band related to C-H vibrations at  $929$  and  $946\text{ cm}^{-1}$ , which is similar to the one in the reference shellac spectrum. These spectra also showed C-O vibrations at  $1045$  and  $1165\text{ cm}^{-1}$  and a pronounced peak around  $1250\text{ cm}^{-1}$ , which is a feature distinguishing shellac from Kaman oil. In the reference Kaman oil spectrum, the peak of this region is positioned around  $1165\text{ cm}^{-1}$  and there is a clearly visible shoulder peak around  $1245\text{ cm}^{-1}$ , which can also be seen in the spectra of LP3, LP4, LP5, and LP6. Like shellac, LP1 and LP2 showed the stretching vibrations of carbonyl at around  $1714\text{ cm}^{-1}$ . It should be noted that in fresh shellac, C=O vibration appears as a twin peak around  $1715$  and  $1735\text{ cm}^{-1}$  [44–47]. In shellac, vinyl (C=C) vibrations were also observed around  $1640\text{ cm}^{-1}$  [47]. Unlike Kaman oil, shellac had a clearly visible absorption band at  $1540\text{ cm}^{-1}$ , which was also observed in LP1 and LP2. These results suggested that LP3, LP4, LP5, and LP6 have an oil-resin coating and LP1 and LP2 have a shellac coating. However, the high similarity of the FTIR spectra of these two coatings makes them difficult to distinguish.

### 3.2. Spectral Imaging

Spectral imaging, in general, and UV-induced visible luminescence imaging, in particular, are very suitable for studying organic coatings and coloring agents on historical objects. The scalability of this method and the fact that it does not require any sampling make it a good complement to spectroscopy-based methods in cultural heritage studies. The results of spectral imaging of the samples are presented in Figure 4 and a summary of the observations is provided in Table 2. While FTIR conducted in the previous step managed to identify the organic coatings, here, an obvious difference was detected between the synthetic coating and natural ones. More specifically, the alkyd resin in LP7 did not show any fluorescence in any of the imaging methods. Therefore, this resin can be easily distinguished from Kaman oil and shellac by its lack of fluorescence.



**Figure 4.** UV-induced visible luminescence imaging of the samples with two processing methods (UVL1 and UVL2) and with image capture in the bright red range (UVIRL) and the blue range (UVIBL).

While the similarity of the FTIR absorption bands of shellac (LP1 and LP2) and Kaman oil (LP3, LP4, LP5, and LP6) make them slightly difficult to distinguish by FTIR, imaging methods make it easy to differentiate them from another. As Figure 4 illustrates, the only image in which shellac and Kaman oil had a similar reddish appearance was UVIRL. The shellac coating had a brown appearance in UVL1 and an orange appearance in UVL2. But Kaman oil had a green color and a milky color in UVL1 and UVL2 respectively. Previous studies have also reported an orange fluorescence for shellac [48–50]. In UVIBL, shellac and

Kaman oil appeared in dark blue and light blue colors, respectively. These results suggested that shellac and Kaman oil coating can be properly distinguished through spectral imaging.

**Table 2.** Summary of the results of luminance imaging of the coatings identified by FTIR.

Samples	Coating Type *	Imaging Methods			
		UVL1	UVL2	UVIRL	UVIBL
LP1	Shellac	Brown	Orange	Red	Dark blue
LP2					
LP3					
LP4	Kaman oil	Green	Milky	Red	Light blue
LP5					
LP6					
LP7	Alkyd resin	Black	Black	Black	Black

\* Based on FTIR spectroscopy results.

#### 4. Conclusions

In this study, FTIR spectroscopy and spectral imaging were used to identify the surface coatings of seven contemporary and historical Persian papier mache penboxes. The results of FTIR spectroscopy showed the use of alkyd resins, Kaman oil, and shellac in the coatings. It was found that alkyd resin can be distinguished from shellac and Kaman oil by the absorption bands 1124, 1268, and 3020  $\text{cm}^{-1}$  as well as the absorption bands related to the vibrations of aromatic structures around 1603, 1071, 742, and 704  $\text{cm}^{-1}$ . However, it is slightly more difficult to differentiate Kaman oil from shellac coating based on FTIR results. The twin peaks appearing at 929 and 946  $\text{cm}^{-1}$  and the absorption bands 1250, 1540 and 1714  $\text{cm}^{-1}$  can be regarded as the features distinguishing shellac from Kaman oil. Shellac was only detected in the two contemporary samples. Oil-resin base coatings typically showed a peak at 1165  $\text{cm}^{-1}$  with a shoulder at 1245  $\text{cm}^{-1}$ . Comparison of the absorption bands of the samples with the reference Kaman oil spectrum indicated the use of this coating in all four Qajar-era penboxes.

Spectral imaging also showed good performance in differentiating the coatings of penboxes. This method was found to be especially effective in distinguishing synthetic coatings from those made of natural oils or resins. While the similarity of the FTIR absorption bands of shellac and Kaman oil in historical objects make them difficult to differentiate, UV-induced visible luminescence imaging made it possible to quickly and easily distinguish them without needing to take any sample. Overall, the results clearly demonstrated the utility of luminescence imaging in the fast and inexpensive identification of organic coatings on historical objects, making it particularly useful for screening large numbers of these artifacts. However, to determine the exact type of coating with this method, a comprehensive reference database will be needed covering all types of coating or must combine this technique with other methods.

**Author Contributions:** A.K. designed the work process and analyzed and interpreted the data. B.J.B. and A.N.B. participated in experiments. All authors discussed the results and contributed to the final manuscript. All authors have read and agreed to the published version of the manuscript.

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