PIGMENTS CHARACTERIZATION AND CONSERVATION OF A LATE PERIOD WOODEN COFFIN FROM THE EXCAVATION SITE OF RASS ALGESR IN SAQQARA, EGYPT

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Abstract

This paper describes the results of a multi-technological analytical protocol and conservation of a polychrome wooden coffin from the Late Period discovered in Rass Algesr, Saqqara, to identify the pigments and ground layer. The investigation utilized visual observation, optical microscopy (OM), scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX), Raman spectroscopy, and Fourier transform infrared spectroscopy. The results showed the use of yellow ochre α-FeOOH and orpiment (As_2S_2) for the yellow-painted layer. Blue was Egyptian blue [Cuprorivaite ($CaCuSi_4O_{10}$)], red as hematite ($α-Fe_2O_3$), and white as calcite ($CaCO_3$). The black pigment was carbon (C), the ground layer was calcium carbonate, the fabric layer was linen, and the binding was identified as animal glue. The conservation of the wooden coffin included mechanical and chemical cleaning. Mechanical cleaning used soft brushes, while chemical cleaning used ethyl alcohol and distilled water for the painted layer, stabilization of the separated gesso layer used Primal AC33, and filling the cracks of the gesso layers used glass microballoons with Paraloid B72.

Keywords: Saqqara; Polychrome wooden coffin; Wood; Pigments; Raman SEM-EDX; XRD; FTIR

Introduction

Identifying the components of ancient Egyptian polychrome coffins is crucial for comprehending the production techniques that reflected the pharaohs' lifestyle and the state of the art. It is also critical to restore these wooden coffins to protect them for future generations [1], [2]. The identification of the components that make up an ancient Egyptian polychrome coffin is crucial to comprehending the production processes, which represent the pharaohs' way of life and the state of art. It is also necessary when proposing conservation projects for the future. The building methods, painting materials, and prior conservation materials of these coffins have all been extensively studied in recent years by numerous writers utilizing a variety of imaging and analytical approaches [3], [4], [5]. A variety of studies describe how Raman spectroscopy, attenuated total reflection, and the utilization of Fourier transform infrared spectroscopy (ATR-FTIR) and scanning electron microscopy in conjunction with energy dispersive X-ray spectroscopy (SEM-EDXS) are highly beneficial for the examination of diverse materials, particularly historical pigments [1], [6], [7].

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A polychrome wooden coffin was discovered in Saqqara, which is located to the west of the Nile and 28 km to the south of Cairo. It contains remains and monuments from different ages [8]. It was one of the principal necropolises of Memphis, an ancient capital of Egypt [9], [10].

A polychrome wooden coffin was discovered in March 2023 at the excavation site of the Secretary-General at Rass Algesr in Saqqara (Figs. 1 and 2). It was found in a rock-cut tomb from the late period, which consisted of two parts: the upper part was found empty, and the lower part was found untouched (Fig. 3). The coffin was found in a polychrome wooden box in good condition; however, the box wasn't as good as the coffin (Fig. 4).

Fig. 1. The map of Saqqara and the bubstion site by Google Earth

Fig. 2 shows the tomb where the coffin was found at the bubstion site at Saqqara by Google Earth

Fig. 3. The site of the Secretary-General's excavation to the north of Oserkaf Causeway and the cut rock tomb where the wooden coffin was found

Fig. 4. The wooden coffin and the box where they were found

The coffin was found inside a wooden box. It consisted of the lid and base. It was coated from the outside with linen and a gesso layer. Because the coffin was made up of many wooden

pieces, the outer coffin was rectangular, and it was used to preserve the inner coffin. The painted layer was decorated with blue, red, white, yellow, and black.

This study aims to identify the pigments and ground layer on the coffin using different analytical techniques and conservation processes to document the information for future generations.

Materials and Methods

Samples

For the analyses, seven carefully selected falling samples representing the various layers were used [11].

Optical Microscopy (OM)

Stereo microscopy (Leica Stereo) equipped with an Axio Cam MRC5 was used in the investigation to see the details invisible to the naked eye and to determine the stratigraphic structure of the painted layer [12], [13].

Scanning Electron Microscope attached with Energy Dispersive X-Ray (SEM-EDX)

Using a Quanta 250 FEG with SEM-EDX (Energy Dispersive X-ray Analyses), the painted layers were examined, and their elemental makeup was determined: the magnification was 500X, and the accelerating voltage was 30 kV [14], [15], [16].

Raman Spectroscopy

To determine the composition of colored materials, a Senterra Raman spectrometer (Bruker) with a 20× objective lens and 785 nm lasers with 5–20 second integration durations and 1–10 mW power was utilized. The method's small spectral footprint, non-destructive nature, and capacity to capture the molecular spectra of inorganic materials made it highly considered [17].

Fourier Transform Infrared Spectroscopy (FTIR)

It was used to identify the organic binding medium and the inorganic functional groups in the painted layerby Bruker VERTEX 70 FTIR equipped with the Universal ATR, 4 cm⁻¹ resolution spectra in the 500–4000 cm⁻¹ region, averaging 32 scans [18], [19].

Results and Discussion

The intricately designed and painted polychrome wooden coffin was a remarkable find. The vibrant colors and detailed motifs on the coffin depicted its significance in late-period burial customs. The interior of the coffin revealed valuable insights into the burial practices and beliefs of the Late Period. The craftsmanship and symbolic elements within the coffin highlighted the cultural and religious significance of the deceased individual. Moreover, the method of construction and the materials used in the coffin's creation provided clues about the technological capabilities and artistic skills of the ancient craftsmen during the Late Period.

Deterioration aspects

The coffin was covered with a thick layer of sand and dust resulting from the burial environment (Fig. 5).

Fig. 5. The wooden coffin before restoration

The condition of the coffin revealed that the lid had flaking, hydrated salts, macro- and microcracks, separation of the painted layer from the mud layer, and coloration issues (Fig. 6).

Fig. 6. The wooden coffin before restoration

Yellow pigments

OM image (Fig. 7a) showed that the yellow-painted layer had two colors: one was faded, and the other was bright yellow. Some grains of sand were on the surface of faded color due to the burial environment. (Fig. 7g-h) It shows the yellow painted layer and the blue painted layer above it.
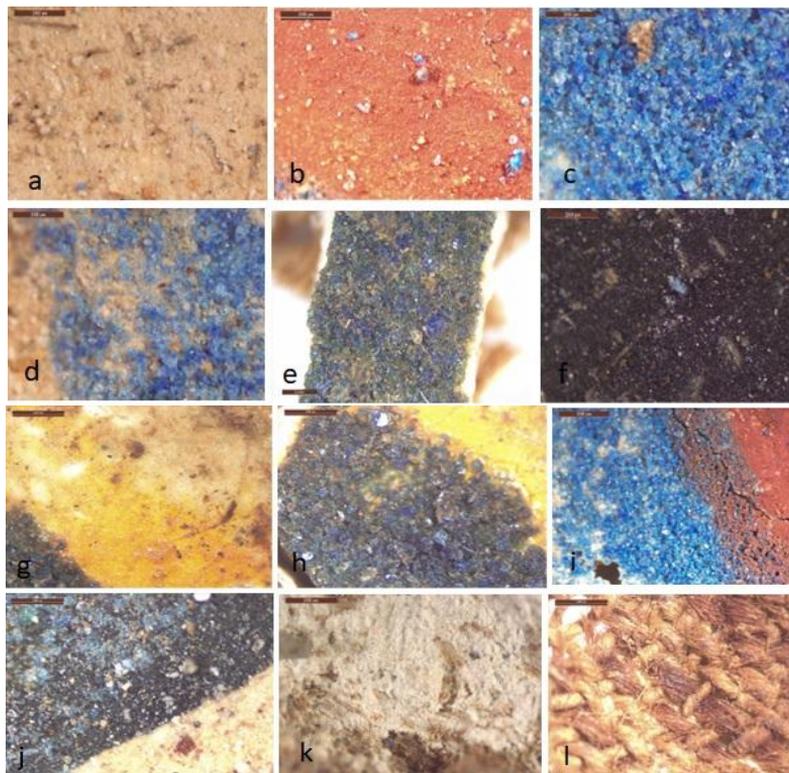

Fig. 7. Optical photomicrographs showing the coarse morphology of the painted layers surface used in the coffin: a) Yellow pigment; b) Red pigment; c-e) Blue pigment; f) black pigment; g-h) Yellow with blue pigment; i) Blue with red pigment; j) Yellow, black, and blue pigment; k) Gesso ground layer; l) Textile ground layer

The schematic structure of the pigment (Fig. 8) showed that the painted layer of the coffin contained wood as a support of the textile layer, the ground layer, and the painted layer, while the box of the coffin contained the wooden support, ground layer, and painted layer.
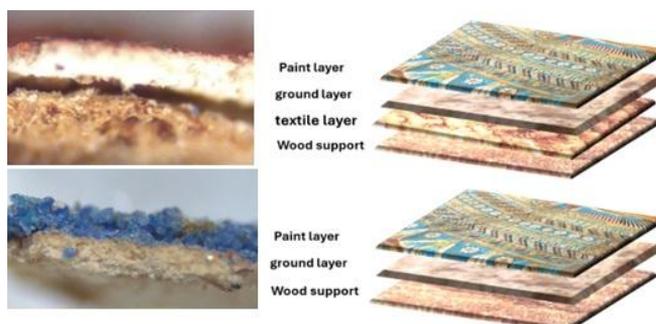

Fig. 8. The cross-section and the schematic structure of the the inner coffin and the outer box

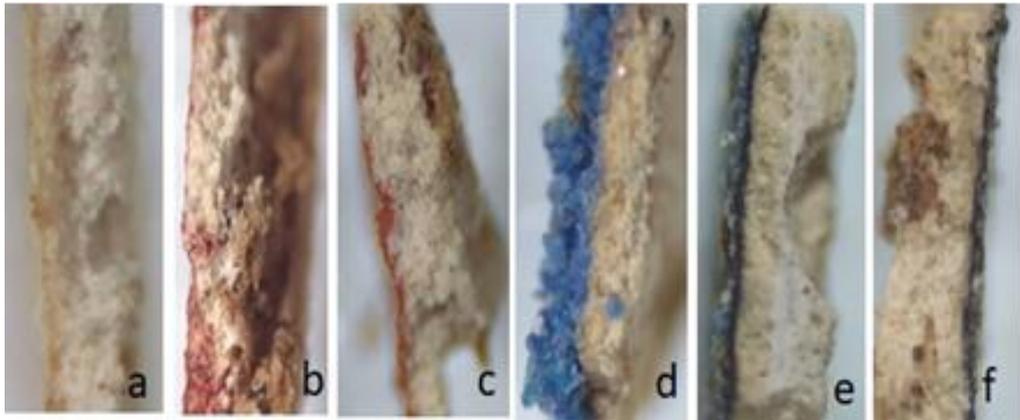The thickness of the yellow-painted layer (Fig. 9a) was 100μm.

Fig. 9. Stratigraphic structure of the ground and painted layers by OM:
a)   Yellow; b-c) Red; d) Blue; e) Black and blue; f) Black

SEM showed the surface of the yellow-painted layer. It also showed fine cracks on the surface of the material (Fig. 10c-d). EDX analysis (Fig. 11a) showed that the elements were calcium (Ca), iron (Fe), and silicon (Si), which proved the presence of the yellow ochre pigment. The bright yellow (Fig. 11b) contained arsenic (As) and sulfur (S), suggesting that it was an orpiment (As_2S_2). The presence of sodium elements (Na) and chlorine (Cl) was due to the presence of sodium chloride salt on the surface. Raman data confirmed that the yellow-painted layer (Fig. 8d) was made of yellow ochre in the form of goethite (Fig. 12a) [20], [21].
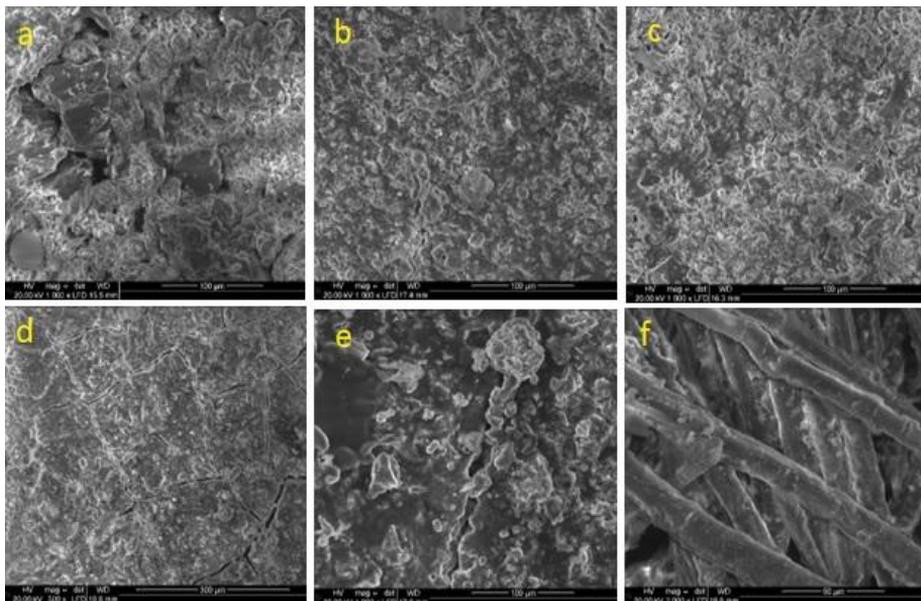

Fig. 10. SEM spectrum of a) blue; b) red; c & d) yellow; e) black; f) textile
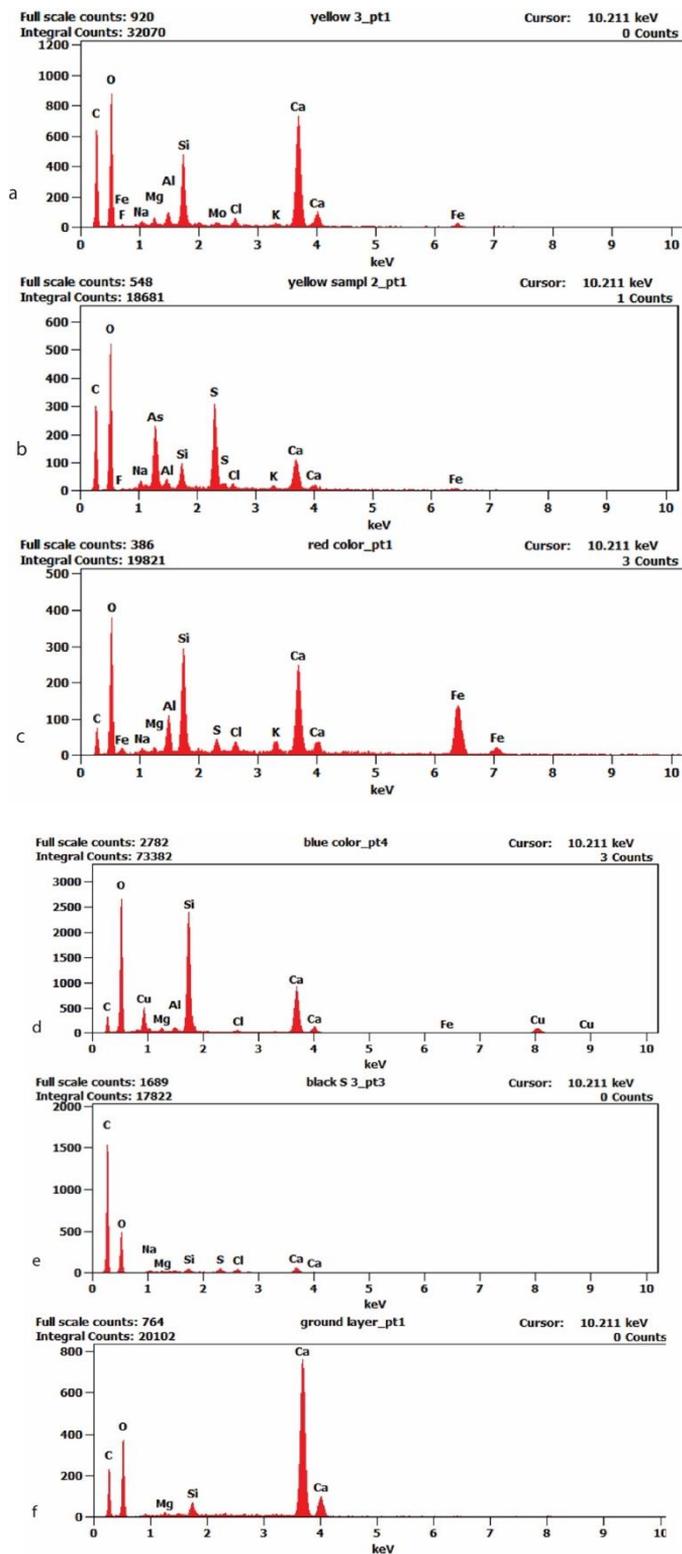
Fig. 11. EDX spectrum of a & b) yellow; c) red; d) blue; e) black; f) ground layer
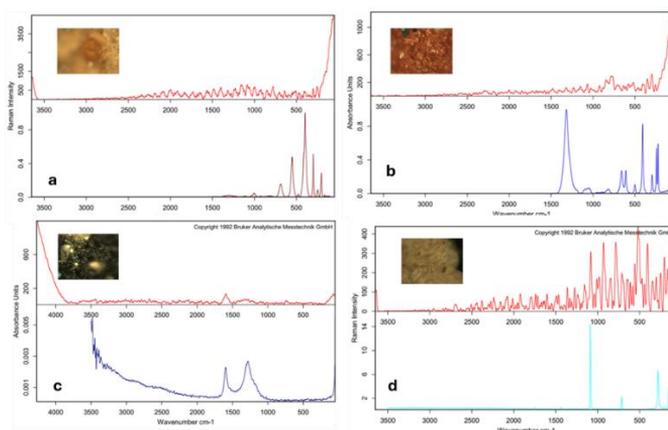
Fig. 12. Raman spectrum of the pigments compared with the standard sample:
a) red; b) yellow; c) black; d) ground layer

Red pigment

The OM image (Fig. 7b) showed that the red-painted layer had fine cracks and salt crystallization on the surface. Some grains of sand were on the surface due to the burial environment. The uneven thickness of the pigment surface was most likely the result of the paint layers not being properly prepared (Fig. 9b-c) [22]. SEM showed that the red-painted layer had microcracks (Fig. 10b)

The red pigment was identified by EDX analysis as hematite (Fe_2O_3) because of the high iron (Fe) content. In archeological environments, pigments derived from red ochre are frequently found as persistently colored remnants. Sodium (Na) and chloride (Cl) indicated the presence of sodium chloride (NaCl). The presence of aluminum (Al) and silicon (Si) was due to the aluminum silicate in the dust or to the impurities in the colored material. Additionally, silicon (Si) might be due to quartz present on the surface of the sample due to storage in a desert area (Table 1, Fig. 11c). Anhydrous iron-oxide hematite (Fe_2O_3) was the natural substance that gave the red-painted layer its color, as proven by Raman data (Fig. 12b) [23], [24], [25].

The results of the correlational analysis are presented in Table 1.

Table 1. The correlation of the chemical data

Sample number	Color	EDX Elements	Raman
1	Red	C, **O**, Si, **Fe**, Ca, Al, Mg	Fe_2O_3
2	Blue	C, **Cu**, Si, **Ca**, O, Al, Cl	-----
3	Bright Yellow	C, **O**, **As**, **S**, Ca, Si, Na, Cl	As_2S_2
4	Fade Yellow	**O**, C, **Fe**, Ca, Al, Mg, Si	FeOOH
5	Black	**C**, O, Al, Mg, Ca, Si	C
6	Ground Layer	**C, O, Ca**, Mg, Si, Na, Cl	$CaCO_3$

Blue pigment

The OM images and SEM (Fig. 7c-e and Fig. 10a) revealed that the grain of the blue-painted layer could be seen easily. A glaze matrix between the blue crystals, a light blue pigment layer with coarse surface morphology, and varying chromatic degrees ranging from blue to pale green crystals pointed to a heating temperature greater than 900°C. The mixture's main component was quartz, and the presence of glass denoted a high flux content (either wood ashes or alkali flux) [26]. (Fig. 7i) it showed cracks and poor coverage of the blue painted layer; the thickness of the yellow painted layer (Fig. 9d) was 100 μm.

EDX of Egyptian blue (Fig. 11d) was recognized by the presence of the distinctive silicon (Si), calcium (Ca), oxygen (O), and copper (Cu) peaks in the spectra produced by EDX analysis [27], [28].

Black pigment

OM images (Fig. 7f) revealed that the black color was in good condition. They (Fig. 7j) showed that some grains of the blue-painted layer on the surface of the black-painted layer might result from the burial environment or the chemical cleaning process of the coffin during the excavation. The schematic structure (Fig. 9e) showed the ground layer, the black painted layer, and the blue painted layer above it. The thickness of the yellow-painted layer (Figs. 9e and f) was 100 µm.

SEM (Fig. 10e) showed the crystallization of salt grains on the surface and fine cracks. EDX analysis (Table 1, Fig. 11e) revealed that carbon was the primary ingredient. Additionally, Raman spectra demonstrated that carbon (C) was used for the black-painted layer (Fig. 12c) [29], [30].

Ground Layer

OM of the ground layer (Fig. 7k) showed white with some parts of the wooden support attached to it. The ground layer's EDX examination (Fig. 11f) revealed the presence of calcium (Ca), carbon (C), and oxygen (O). These components recommended using calcium carbonate as a ground layer [31]. The FTIR spectrum of the ground layer (Fig. 13) showed the characteristic peaks at 2988, 2925, 2873, 2517, 1422, 710, and 874 cm^{-1} as calcium carbonate. and Raman spectra (Fig. 12d) demonstrated that calcium carbonate was used for the ground layer.

Textile

The coffin was covered with a layer of fabric, topped with a ground layer. OM (Fig. 7l) of the textile under the gesso layer illustrated that it consisted of basic weave types: (a) plain weave. The fiber appeared as smooth, cylindrical bundles with some swelling areas along the fiber, as demonstrated by SEM (Fig. 10f). This indicates that the fiber was linen [32], [33].

The bending medium

The FTIR spectrum of the painted layer (Fig. 13) showed the characteristic peaks of N-H stretching band at 3421 cm^{-1}, the C=O stretching of amide I band at 1638 cm^{-1}, N–H bending of amide II band at 1506 ccm^{-1}, and C-H binding band at 1393 cm^{-1}. This was attributed to proteinaceous materials like animal glue, which were frequently employed as binding agents in the ground layer starting in the very early times of ancient Egypt [27].
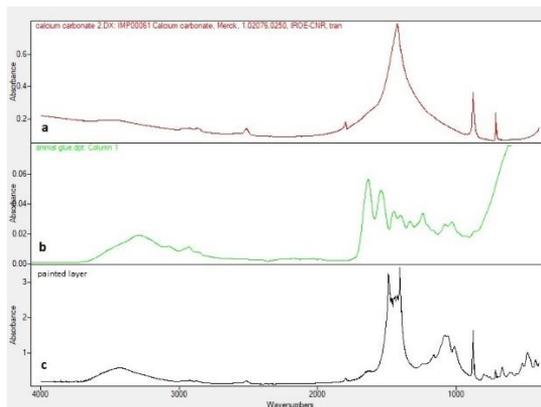

Fig. 13. Comparing FTIR spectra between references and samples:
a) calcium carbonate; b) animal glue; c) painted layer sample

Treatment and Conservation

Mechanical cleaning

One of the trickiest and most delicate parts of a restoration project is cleaning. In the present study, a rubber pump and gentle brushes were used to clear the dust and sand from the coffin and box [34] (Fig. 14).

Fig. 14. The wooden coffins during the restoration and consolidation of pigments and ground layer

Chemical cleaning

Ethyl alcohol and distilled water were employed with a 1:1 ratio for chemical cleaning (Fig. 6 D). After conducting many tests, it was determined to proceed with chemical cleaning of the painted layer [32].

Stabilization of the separated painted gesso layers

Before installing the separate ground layers, the facing was carried out using tissue paper with Kulcel G 0.5% in alcohol [35].

In addition to reversibility, compatibility and retreatability are typical characteristics of wood consolidation materials [36]. Thus, primal AC33 was used in these processes. A solution of 10% Primal AC33 was used for the reattachment of the separated parts to the wooden support by injection (Figs. 15-17).

Fig. 15. During the facing process of the painted layer

Fig. 16. The wooden coffins during restoration injection

Fig. 17. The wooden coffins during the restoration of the box and the laid

Consolidation of the painted layer

Soft brushes were used to consolidate the painted layer using solutions of Klucel G (hydroxypropyl cellulose) 1% W/V in ethyl alcohol [37], [38].

Filling the edges of the ground layer

Using a spatula, glass microballoons containing Paraloid B72 in 15% w/v acetone were applied. The microballoons were then coated with earth pigments. Figure 18 shows the coffin after restoration [39].

Fig. 18. The wooden coffins after restoration

Conclusion

An Egyptian wooden coffin from the Late Period was the subject of the investigation. SEM-EDX assisted in identifying the ground and painted layers, while optical microscopy (OM) examined the stratigraphic structure of the painted layer. Additionally, supplementary methods like Raman were employed to verify the painted layer. We could describe the pigments' bending medium by FTIR analysis. For the red layer, hematite was the pigment of choice; for the blue layer, cuprorivaite was the Egyptian blue; and for the yellow layer, orpiment was yellow and also ochre. Moreover, calcium carbonate was utilized for the ground layer, and carbon was used for the black-painted layer.

Consolidation, filling the ground layer borders, reattaching the ground layer to the support, and mechanical and chemical cleaning were the stages of restoration. The ground layer was reassembled using 10% Primal AC33, and the ground layer's edge was preserved using a combination of microballoon, 15% Paraloid B72, and earth pigments. Using a brush 1% Klucel G was used to consolidate the coffin. This analysis helped document the colored materials in wooden coffins for use in the future in comparing manufacturing techniques and materials used in wooden coffins across the ages.

Acknowledgments

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