

MULTI ANALYTICAL TECHNIQUES OF ANTHROPOID WOODEN COFFIN FROM EGYPT'S LATE PERIOD

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Abstract

The present study aims to identify the pigments, ground layer, and previous restoration materials used in a polychrome wooden coffin using multi-analytical techniques. It utilized optical microscopy, technical imaging, scanning electron microscope attached to X-ray dispersion unit, X-ray diffraction, Raman spectroscopy, and Fourier transform infrared spectroscopy. The results showed the use of yellow as goethite α -FeOOH, blue as Egyptian blue [Cuprorivaite ($\text{CaCuSi}_4\text{O}_{10}$)], red as haematite (α -Fe $_2\text{O}_3$), and white as calcite (CaCO_3). The black pigment as carbon (C) and the binding medium in both the calcite-based plaster layers and the polychrome layers was identified as animal glue. The previous consolidation material was primal AC33.

Keywords: Wooden coffin; Technical imaging; Pigments; SEM-EDX; XRD; FTIR

Introduction

The documentation, preservation, and treatment of artwork are enhanced by a deep understanding of its fabrication and history. Images taken of different parts of the electromagnetic spectrum provide the means to diagnose and determine the technical-material aspects of an artwork and to identify degradation and areas in need of repair [1]. Sufficient spectral accuracy of multispectral imaging is a major requirement for pigment mapping [2, 3]. Therefore, the non-destructive analysis of art materials has become the preferred method of analyzing valuable art objects [4].

The study examines a coffin dating back to the late period and stored in the Dahshour storeroom. The coffin measures 182cm long, 49cm wide, and 30cm high. It consists of the lid and base. Its lid was coated from the outside with two preparation layers. Because the coffin was made up of many wooden pieces, the first piece was a preparation layer composed of mud mixture applied on the entire surface of the lid to act as a leveling surface. The second one was a fine layer composed of calcium carbonate prepared for the coloring process and a painted layer decorated with red, blue, yellow, white and black [5].

This study aims to identify the pigments, ground layer, and previous restoration materials used on the studied coffin using different analytical techniques to provide necessary information for suitable future conservation works.

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Materials and Methods

Samples

Eight fallen samples 1, 2, 3, 4, 5, 6, 7 and 8 (Fig. 1) representing the different layers were carefully chosen for the analyses.

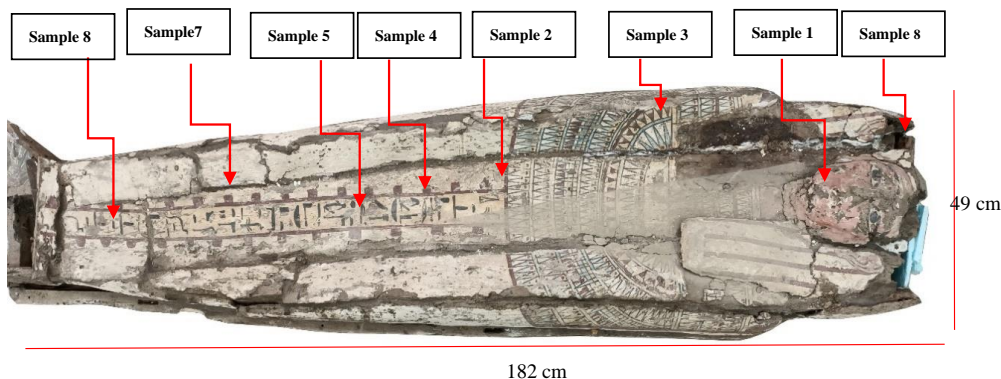


Fig. 1. A map of the sampling areas

Optical Microscopy (OM)

It was used to identify the stratigraphic structure of the painted layer and to view the details that cannot be seen with naked eye, this elements were studied by stereo microscopy (Zeiss Stereo Discovery V 20) equipped with Axio Cam MRC5 [6-8].

Technical Photography (TP)

Technical images in different spectral ranges (from 360 to about 1100 nm) [9-11]. The study used this portable, customizable, non-invasive, and relatively inexpensive system in situ [12]. It utilized the Visible-induced Infrared Luminescence (VIL) using a Sony A6000 digital camera modified to “full spectrum” with a 90C IR filter and a LED lamp to view the fluorescence (luminescence). The Infrared False Color images (IRFC) [13] were made by digitally editing the visible-induced infrared luminescence (VIL) and visible images using the Adobe Photoshop program. The Infrared False Color image was made by digitally editing the VIS and IRF images using the Adobe Photoshop program. The copy of the VIS image was edited to become the IRFC image. The VIS green channel substituted the blue channel, the red channel, and the green channel. Then, the IRF image constituted the red channel of the edited VIS [14].

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

Examining and identifying the elemental composition of the painted layers were performed using a Quanta 250 FEG with SEM-EDX (Energy Dispersive X-ray Analysis). The accelerating voltage was 30kV, and the magnification was 500× (Table 2) [15, 16].

X-ray Diffraction Analysis (XRD)

It was used to identify the components of the painted layers by a nondestructive mode without any sample preparation (X-ray diffraction model PW3040 /60– Analytical Equipment – PANalytical pro model with a Cu anode, working at 40mA/45kV) [17, 18].

Raman spectroscopy

It was used by a Senterra Raman spectrometer (Bruker) with a 20× objective lens and 785nm lasers with 5-20 second integration times and 1-10mW power to identify the composition of colored materials. The technique is well-appreciated for its non-destructive

character, its small spectral footprint, and its ability to record the molecular spectra of inorganic materials [19-22].

Fourier Transform Infrared Spectroscopy (FTIR)

It was used to identify the organic binding medium, the inorganic functional groups in the painted layer, as well as the previous consolidation materials by Bruker VEREX 70 FTIR equipped with the Universal ATR, 4cm^{-1} resolution spectra in the $500\text{--}4000\text{cm}^{-1}$ region, averaging 32 scans [23-27].

Results and discussion

Deterioration and degradation aspects

The condition of the coffin showed that the lid was affected by the separation of the mud and paint layer, macro- and microcracks, hydrated salts, flaking, coloration, and wrong previous restoration interventions, which used primal materials to cover the painted layer without proper cleaning of the surface that caused the consolidation of the painted layer and high viscosity of the consolidation materials, causing brightness of the painted layer without penetration to the ground layer. For the missing parts filled with paraloid B72 by cotton, and the crack filled with a new mud layer, polyurethane foam was used to make stability on the head area, using primal with white oxide to recolor some parts of the coffin. Consequently, severe damage appeared on the coffin.

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

Table 1: EDX, XRD, and Raman results of the painted and ground layers

Sample Number	Color	EDX Elements	XRD Components	Raman Components
1	Light Red	C, O, Fe, Al, Mg, Si and Ca.	Fe_2O_3	Fe_2O_3
2	Dark Red		Fe_2O_3	Fe_2O_3
3	Blue	Ca, C, O, AL, Si, Cu, Fe, Na and Cl	$\text{CaCuSi}_4\text{O}_{10}$ $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	----
4	Fading Blue	C, O, Si, AL, Ca, Cu, Mg, Fe, Na and Cl	----	----
5	Cross Blue	C, O, Si, Fe Cu, AL, Ca, Mg, Cl and S	----	----
6	Yellow	O, C, Mg, Al, Si, Fe and Ca	FeOOH	----
7	Black	C, O, Mg, Al, Si, Ca and Fe	----	----
8	Ground Layer	Ca, C, O, Al, Mg and Si	CaCO_3	----
9	Mud	O, C, Mg, Al, Si, Fe, Ca, Na, C and K	SiO_2 CaCO_3 , $\text{CaAL}_2\text{Si}_2\text{O}_8$	

Previous consolidation materials

Some parts of the painted layer was white due to the wrong consolidation process with high viscosity materials. UV-induced showed the restored sites in the coffin (Fig. 2a-d).

OM of the painted layers (Fig. 3a-i) showed that the painted layers were covered with shiny layers resulting from the previous consolidation materials, and the red-painted layer had some sand grains because the restorer made consolidation without good cleaning of the surface.

FTIR spectrum of this material (Fig. 4) showed the characteristic peaks of C-H stretching 2980 and 2873cm^{-1} , C=O stretching 1726cm^{-1} , C-H bending 1425 and 1380cm^{-1} , C-O stretching 1143 and 1046cm^{-1} , C-H rock 871 and 848cm^{-1} [28, 29]. Primal AC33: an ethyl acrylate-co-methylmethacrylate, made and commercialized in water dispersion by Rohm and Haas, was used in the conservation of different materials [30-32].

Red pigments

OM image for sample 1 (Fig. 3a) showed that the red-painted layer was coated with previous consolidation material and salt crystallization on the surface of the color. Some grains of sand were on the surface after consolidation due to the lack of proper cleaning of the surface before the previous restoration.

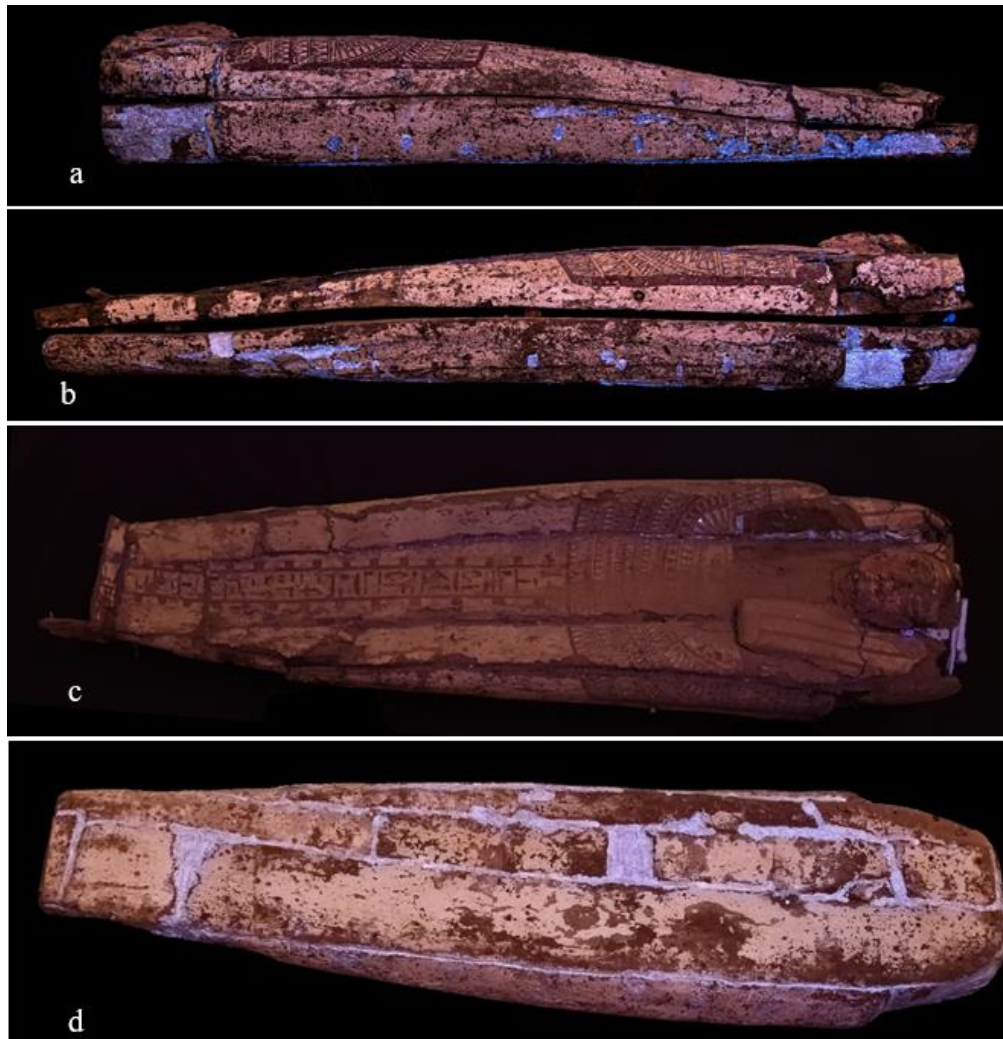


Fig. 2. Technical images of the coffin made by UV to show the different restoration parts of the coffin

EDX analysis suggested that the red pigment was (hematite, Fe_2O_3) due to the high amount of iron (Fe) [33]. Pigments made from red ochre are often discovered as long-lasting colorful remains in archaeological contexts [34]. XRD data confirmed that the red-painted layer made of red ochre was a natural product colored by anhydrous iron-oxide hematite (Fe_2O_3) [35, 36]. There was a light red tone color for sample 1 (Figs. 3a and 4a) and a dark red tone color for sample 2 (Figs. 3b and 4b) composed of hematite.

Blue pigment

The OM image samples 3 (Fig. 3d) showed that the blue paint layer had deep and light blue crystals spread within a white matrix. The blue-painted layer appeared white bright in the VIL image (Figs. 5b and 6b), indicating the use of Egyptian blue [37, 38] but did not appear in visible light (Figs. 5a and 6a).

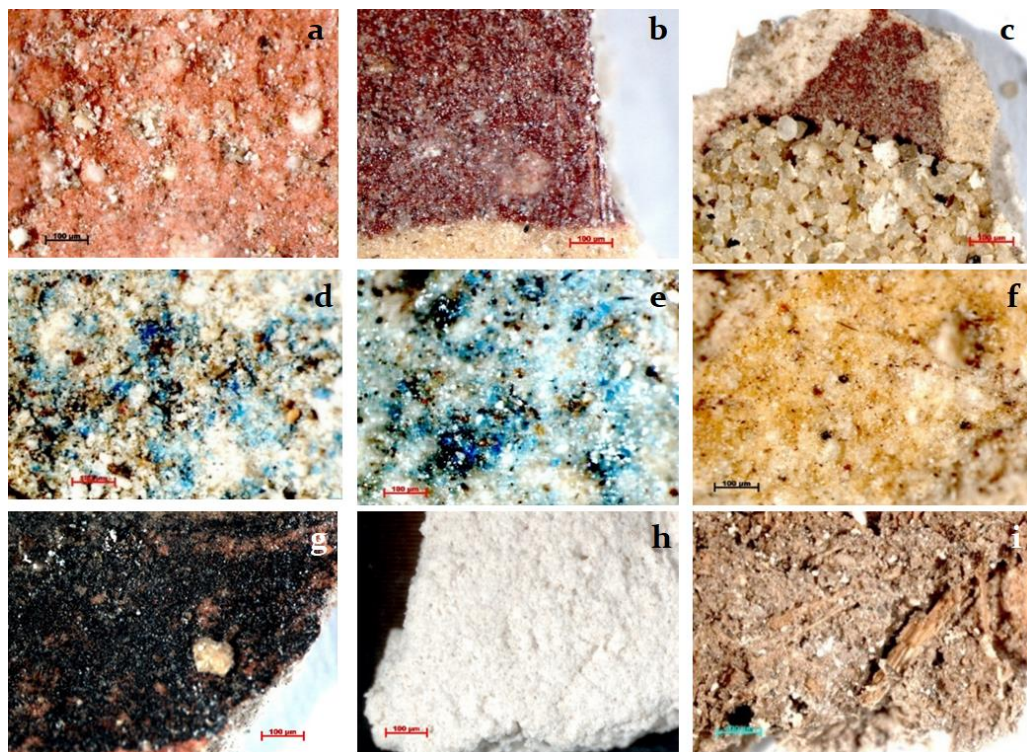


Fig. 3. Optical photomicrographs showing the course morphology of the painted layers surface used in the coffin: a, b. and c. Red pigment; d, and e. Blue pigment; f. Yellow pigment; g. black pigment; h. gesso ground layer; i. Mud ground layer

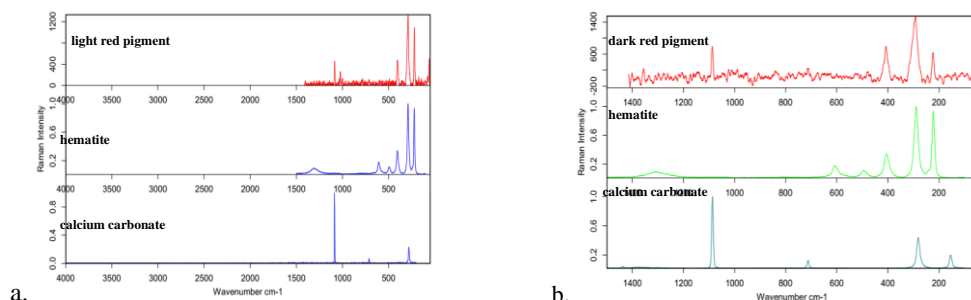


Fig. 4. Shows the Raman pattern of red pigments compared with the standard sample of hematite and the standard sample of calcium carbonate:

a) light red tone pigment; b) dark red tone pigment

The IRFC image showed that the areas painted with Egyptian blue appeared red [39] (Fig. 6c). The presence of the characteristic silicon (Si), calcium (Ca), oxygen (O), and copper (Cu) peaks in the spectra obtained by EDX analysis was identified as Egyptian blue

(cuprorivaite, $\text{CaCuSi}_4\text{O}_{10}$) [40-42] (Fig. 7c). No Raman signal was detected from Egyptian blue due to the intense luminescence from the pigment when excited at 785nm [43]. The investigation of pigments by XRD illustrated the presence of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), calcite (CaCO_3), quartz (SiO_2), and cuprorivaite ($\text{CaCuSi}_4\text{O}_{10}$) (Fig. 8c) the main component of Egyptian blue-in accordance with the attribution made by VIL [44].

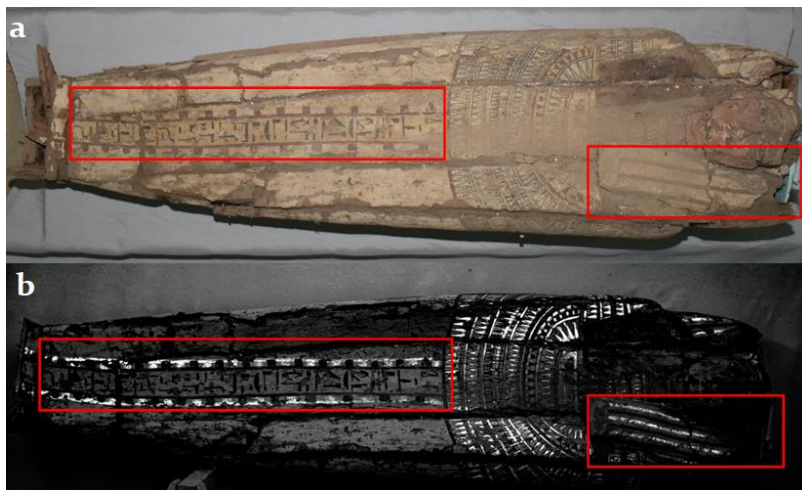


Fig. 5. Technical images of the coffin made by different methods:
a. visible (VIS); b. Infrared Fluorescence (IRF)

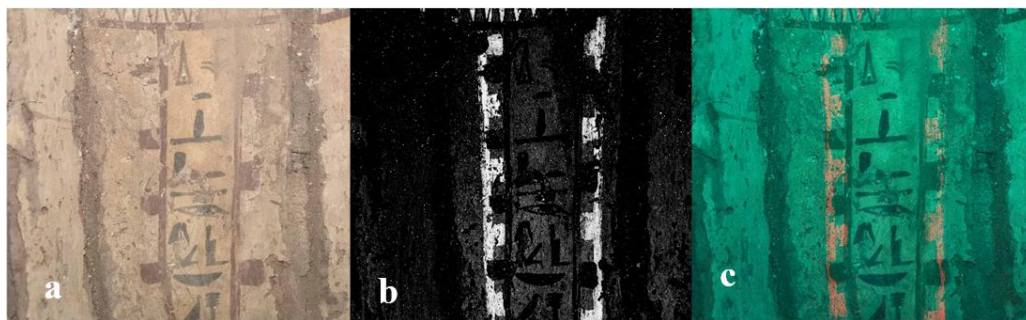


Fig. 6. Technical images of the coffin made by different methods:
a. visible (VIS); b. Infrared Fluorescence (IRF); c. Infrared False Color (IRFC)

The EDX of the fading Egyptian blue on both sides of the central decorative panels (samples 4) showed the same elements of the blue-painted layer beside iron (Fe), and oxygen (O) that indicated that ancient Egyptians used the yellow-painted layer from goethite then used Egyptian blue in the same area (Fig. 7d).

The blue color of the original changed to *green blue* because of the alteration to a mixture consisting predominantly of the basic copper chlorides [45] or the use of inappropriate solvents in the chemical cleaning process in the previous restoration process.

Yellow-painted layer

The (OM) image (sample 5) (Fig. 2f) showed that the yellow-painted layer was coated with a previous consolidation material. SEM-EDX analysis (Fig. 7e) showed that the elements were calcium (Ca), iron (Fe), and silicon (Si) that proved the presence of the yellow ochre

pigment. XRD data confirmed that the yellow-painted layer (Fig. 8d) was made of yellow ochre in the form of Goethite. The most common yellow pigments are goethite, yellow ochre, α -FeOOH [46-49].

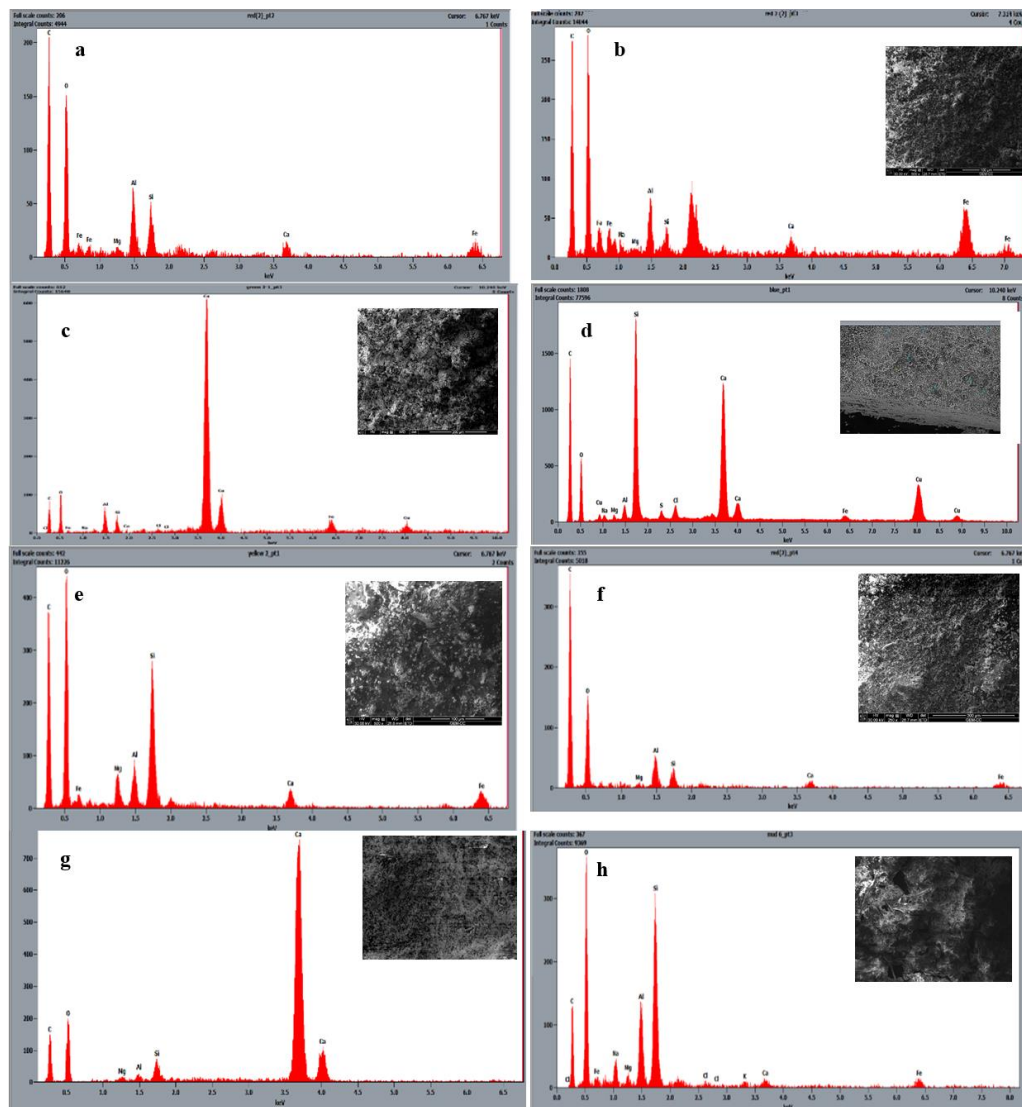


Fig. 7. SEM-EDX spectrum for samples: a. light red; b. dark red; c. blue; d. fading blue; e. yellow; f. black; g. gesso ground layer; h. ground mud layer

Black pigment

The OM images for sample 6 (Fig. 3g) revealed that the samples were coated with a previous consolidation material. Therefore, the grain of the black-painted layer could not be seen, and red pigments appeared on the missing parts of the black pigments, suggesting that ancient Egyptians used red pigment under the black pigment on this coffin.

The black painted layer appeared darker in the infrared image that suggests the presence of carbon-based black. EDX analysis (Fig. 7f) showed that the main element was carbon (C), which confirms the use of carbon for the black painted layer [50, 51].

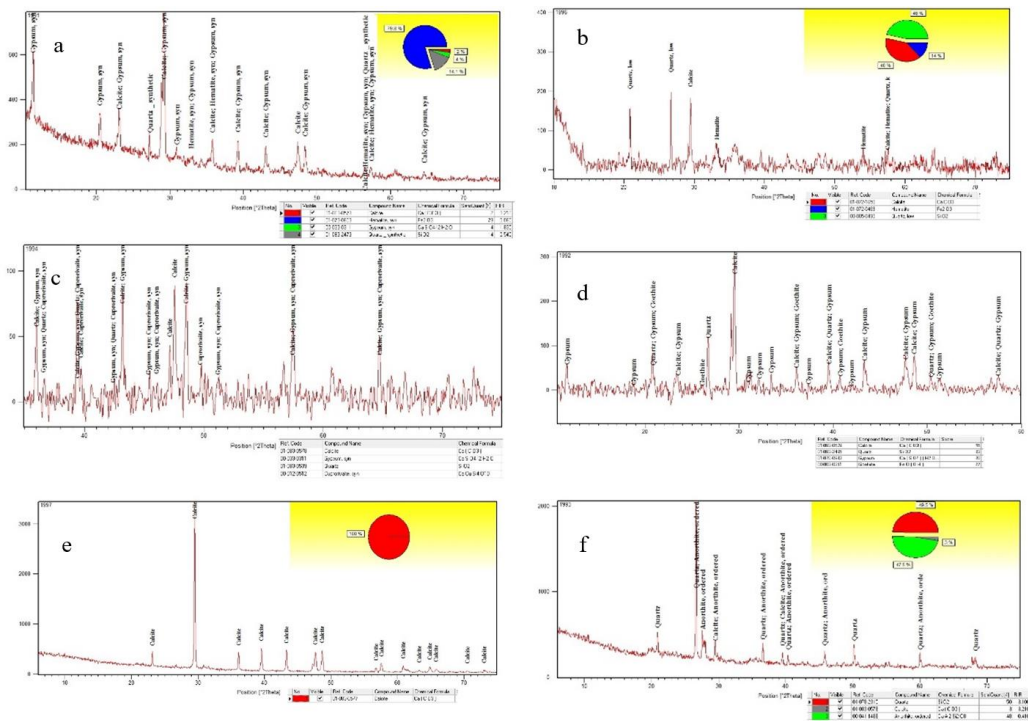


Fig. 8. XRD spectrum of a) light red tone; b) dark red tone; c) blue; d) yellow; e) Gesso ground layer; f) mud ground layers

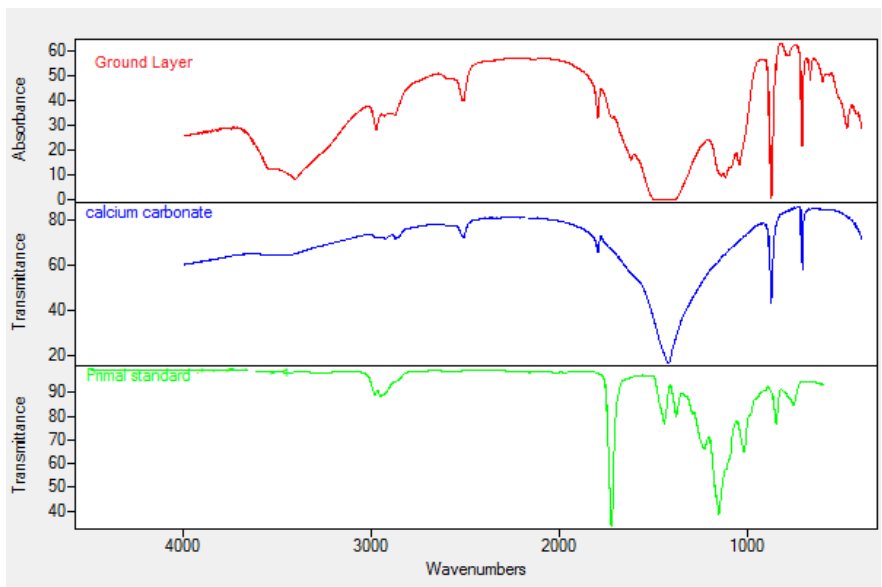


Fig. 10. Comparing FTIR spectra between reference and sample of the ground layer

Gesso ground layer and white pigment

The OM of the ground layer (Fig. 3h) showed white, but not shiny and weak because the consolidation materials used in the previous restoration did not penetrate enough to reach the ground layer. SEM-EDX analysis of the ground layer (Fig. 6g) showed the presence of carbon (C), oxygen (O), and calcium (Ca). These elements suggest the use of calcium carbonate as a ground layer. Magnesium (Mg), aluminum (Al), and silicon (Si) were also found due to the dust on the surface of the coffin [52]. The FTIR spectrum of the ground layer (Fig. 9) showed the characteristic peaks at 2976, 2875, 2516, 1800, 1425, 875 and 851 cm^{-1} as calcium carbonate [53]. The XRD spectrum of the ground layer (Fig. 7e) showed that calcium carbonate was used for preparation layers. The number of white colors used in ancient Egypt was limited for a long time to only two types of pigments, Calcium carbonate (CaCO_3) obtained from the mineral calcite [54, 55] which ascribed to calcium carbonate, confirming the attribution by SEM-EDX and XRD [56].

Mud ground layer

The OM of the mud ground layer for sample 8 (Fig. 3i) revealed a mixture of mud, clay, and plant fibers in the ground mud layers.

Calcium (Ca), aluminum (Al), silica (Si), and oxygen (O) indicated the presence of anorthite found in clay minerals (Fig. 7h). Calcium (Ca) and oxygen (O) were due to calcium carbonate used with clay minerals to make the ground layer, whereas the presence of chloride and sodium indicated the existence of halite that may be related to the burial soil. The mud layer was used to cover the wooden coffin from the outside and to fill the gaps between boards from inside. The chemical composition of the mud layer was composed mainly of clay minerals (quartz, anorthite, and calcium carbonate) [55, 56] (Fig. 7f).

Conclusion

The study investigated an anthropoid wooden coffin from the late period, Egypt. Optical Microscopy (OM) was used to investigate the stratigraphic structure of the painted layer, whereas technical photography and SEM-EDX helped identify the ground and painted layers. Moreover, complementary techniques, such as XRD and Raman were used to confirm the painted layer. FTIR analysis allowed us to characterize the ground layers. The pigments were hematite for the red-painted layer, the Egyptian blue (cuprorivaite) for the blue-painted layer, and the yellow ochre was goethite for the yellow-painted layer. Furthermore, carbon was used for the black-painted layer and calcium carbonate was used for the ground layer. The previous consolidation material was Primal AC33. The analyses provided more information concerning the original and previous treatment materials, which will help choose the most suitable materials in future conservation works.

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