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ANALYTICAL INVESTIGATION ON A COPTIC WOODEN ICON FROM THE 18TH CENTURY USING SEM-EDX MICROSCOPY AND FTIR SPECTROSCOPY

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ABSTRACT

In the present study, a comprehensive investigation has been undertaken into a wooden Coptic icon dated to the 18th century and painted by Ibrahim El-Nasekh. It is located in Saint Abanoub church in Samanoud, in the Nile Delta, Egypt. This study included the determination of the pigment palette, the gold layer and the white ground layer and the wooden panel. The analytical instruments used were Optical microscopy, Environmental scanning electron microscopy coupled with energy dispersive X-ray emission and Fourier transform infrared coupled with attenuated total reflectance "FTIR-ATR". The analysis revealed that the pigments used in this icon comprises indigo ($C_{16}H_{10}N_2O_2$), red lead (Pb_3O_4), white lead ($2PbCO_3 \cdot Pb(OH)_2$) and yellow lead (PbO). The gold areas were made of real gold leaves applied over an orange bole layer. The white ground layer comprises calcium sulphate dihydrate admixed with animal glue. The wooden panel was found to be made of Accacia Pennata (L.) Wild, while Cupressus sempervirens L. var. dupreziana (A. Camus) Silba was used for pegs and traverses.

KEYWORDS: Wooden icon, Coptic, pigments, Analysis, SEM-EDX, FTIR

1. INTRODUCTION

The 18th century is considered as the golden era of icon painting in Egypt. In this century, several icon painters were active whom which Ibrahim Al-Nasekh, Yuhanna Al-Armani and Mattary are the most famous.

The well established technique for Coptic icons' painting depends on painting on wooden panels made from one or several blanks held together with wooden pegs and supported by wooden traverses, nailed on the reverse with iron nails. The panels were covered with linen canvas in order to reinforce any joints, to even out flaws on the surface of the panel and to reduce the cracking of the ground. The linen covered panels were then primed with white ground layers made from gesso powder mixed with an aqueous protein component like animal glue. Over the paint layers, a protective varnish layer was finally applied (Abdel-Ghani et al., 2009).

Technically, two exceptions have been detected in icon painting in this period. The first was the replacing of the gesso layer with the paper ground layer in the entire icons, painted by Mattary (Skalova and Gabra 2003, p. 132). The second was the execution of the paint layer over a thin gesso layer which directly applied over the wooden panel, with the absence of the intermediate canvas layer.

In this study, for the first time, a wooden icon, painted by one of the most famous painters, was fully investigated. The purpose from this research is to set, beside the technical information about this type of icons, the knowledge about both the organic and the inorganic materials used. The study comprised the examination of the pigments, the media, the gilding, the ground layer and the wooden panel. A technical comparison was also carried out with the previously studied Coptic icons.

2. THE EXAMINED ICON

The icon of the Archangel Michael (registration number 1/ 49) is located at Abanoub church, in Samanoud, in Egyptian Delta (Fig. 1). It is dimensions are 88 cm H x 64 cm L. It Dates from the 18th century and was painted by Ibrahim Ibn Sam'an Al Nasekh who was a scribe, illuminator and icon painter. He worked between 1740 and 1780 and usually signed as "Al Haqir Ibrahim Al Nasekh ("the Humble Ibrahim the scribe") or Abraam as signed once in Harat Al Rum in Cairo (Abdel-Ghani et al., 2012).

The full-length figure of the archangel Michael (Fig. 1) is depicted frontally against a background, which is golden at the upper part and blue at the lower part, with Arabic inscriptions written on. His large wings are painted with a yellow pigment contoured with black thick lines forming feathers, which are decorated with delicate orange lines. His head, with brown hair, is surrounded by a golden halo with double red outlines. In his right hand, he is holding a long cross-staff with three horizontal bars while holding a balance scale in his left hand. The Archangel is wearing a long red under-tunic, a shorter white tunic, a red chlamys and brown shoes.

The reverse of the icon is plain with cut marks shown. The wooden panel comprises two wooden blanks joined together by wooden pegs and strengthened with three traverses.

3. THE ICON CONDITION

The analysis of this icon carried out in the occasion of the restoration treatment. From the visual inspection and the cross section images, no intermediate canvas layer between the wooden panel and the pictorial layer presents. The condition of the icon (Fig. 2) is very poor. Failure in paint layers is the most apparent deterioration phenomenon (Fig. 2, a-b-c) as the canvas is normally used to hold the loose paint layers in place. In the case of absence of the canvas, the paint layers are fallen and mostly missed. This type of lose is due to the movement of the wooden blanks due to the environmental changes.

Figure 1 The icon of the Archangel Michael and the sample locations.

Another type of deterioration of the pictorial layer is the candle burns (Fig. 2, d-e). This phenomenon is due to putting candles in front of the icon with the absence of glass protection. Therefore, the pictorial layer suffered from severe loss and colour change around the lost areas. Soot accumulations are also found over the paint layer around those burnt areas. Holes of insects are spread through the wooden panel (Fig. 2, h) and tunnels can be seen under the optical microscope. This phenomenon made the wooden panel very fragile. As shown in the ESEM and LOM images, the wood is chewed into small fragments and discrete tunnels and surface channels were also recognized. Insect eggs can be also seen in the wood cavities made by insects (Fig. 2, f).

Wide splitting in the joint area between the wooden blanks is shown, in addition to narrow cracks found all over the panel (Fig. 3, i). There is also an evidence of old restoration treatment which is apparent by existence of over paint and paraffin wax (Fig. 3, f) on some areas of the pictorial layer. Moreover, ribbons of textile covered the splitting areas of the panel (Fig. 3, g).

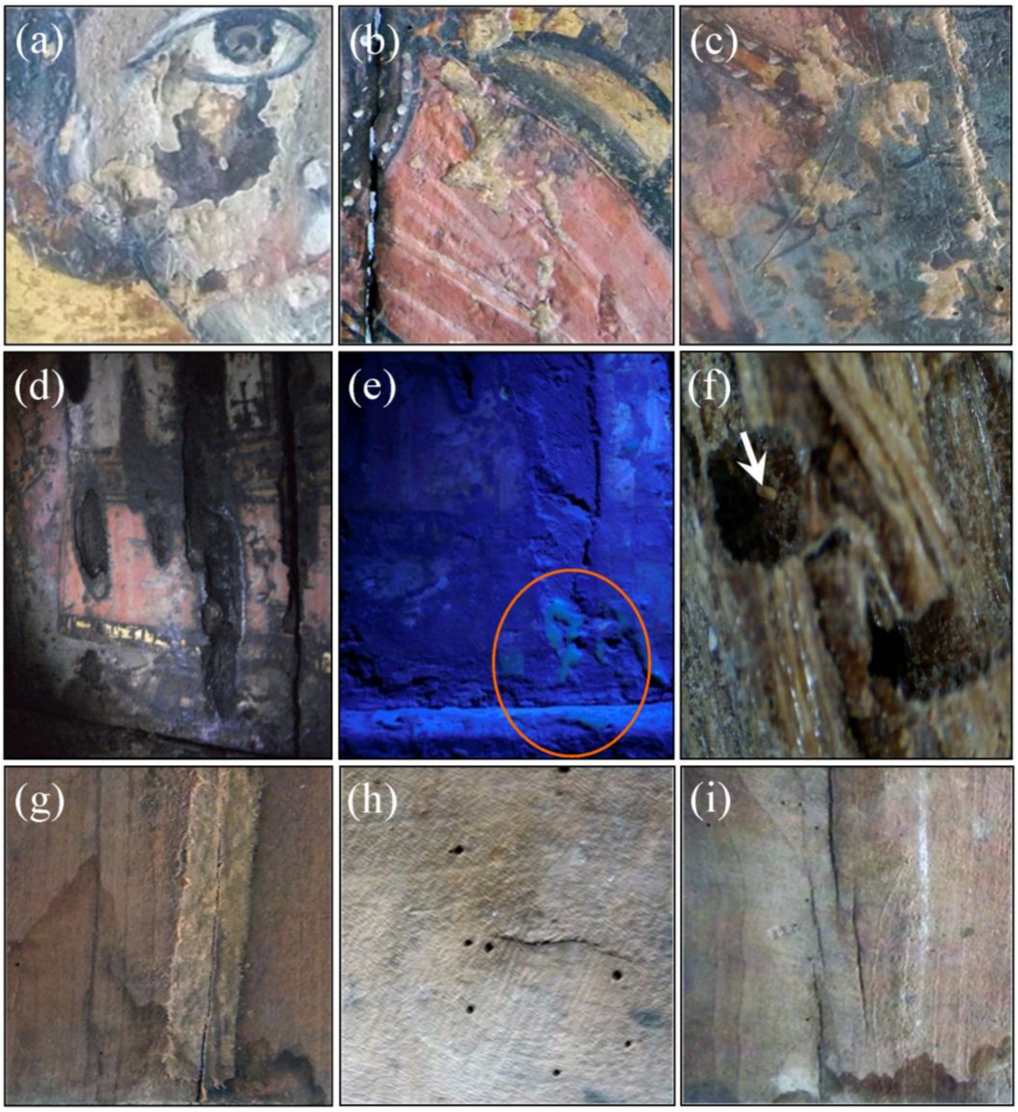

Figure 2: The condition assessment of the Archangel Michael icon (a-c) failure in paint layers (d) candle burns (e) paraffin wax from old conservation treatment (f) the insect larva in the insect tunnels (g) ribbons of textile covered the splitting areas (h) holes of insects are spread through the wooden panel (i) narrow cracks found all over the panel

4. METHODOLOGY

4.1 Samples and sample Preparation

Samples from the very edges of the icon from damaged areas were taken using a scalpel. The samples under study comprise five paint samples and four samples taken from the wooden panel and traverses. The colours under study are blue, red, yellow, white and gold. Samples were studied non-destructively using Optical microscope, Fourier transform infrared coupled with attenuated total reflectance "FTIR-ATR" and Environmental scanning electron microscope coupled with energy dispersive X-ray "ESEM -EDX".

4.2 Fourier Transform Infrared –Attenuated Total Reflectance "FTIR-ATR"

Samples were analyzed with a FTIR spectrometer (Model 6100 Jasco, Japan). Spectra were obtained in the transmission mode with TGS detector and using ATR crystal which represents (2mm/sec) co added scans at spectral region ranging from 4000 to 400 cm^{-1} with 4 cm^{-1} resolution.

4.3 Scanning Electron Microscope coupled with EDX

Samples were analyzed with an Environmental Scanning Electron Microscope

(FBI, Netherlands) coupled with Energy Dispersive X-ray analysis. The samples were examined without coating at low vacuum. Backscattered Electron images of the samples' fragments were examined at 20 keV. Acceleration voltage with backscattered detector (BSE) was at 10.6 mm working distance and a spot size 7.0 with scale ranging from 50μm to 200μm and magnification start from 500x to 2500x.

5. RESULTS AND DISCUSSION

5.1 The Paint area

5.1.1 The blue paint area

The blue sample from the background of the inscription was investigated (Fig. 1, sample 1). The characteristic bands revealed by the FTIR analysis appeared at 1623, 1400 and 688 cm^{-1} suggesting the presence of indigo pigment ($C_{16}H_{10}N_2O_2$).

This result was confirmed by using EDX analysis (Fig. 3, a) which gave rise to silicon, aluminium, potassium, chlorine, iron, sulpher and sodium which are ascribed to indigo pigment originated from indigofera tinctoria plant (Abdel-Ghani et al. 2012). These elements occurred due to the presence of residual salts and oxides in the indigofera plant, while woad leaves contain a high amount of calcium and phosphorous (Abdel-Ghani et al. 2012).

Indigo was found to be admixed with lead white [$2PbCO_3$. $Pb(OH)_2$] to obtain a lighter shade as proved by the presence of lead and calcium (Ganitis et al., 2004).

This is the second detection of indigo pigment in Egyptian panel paintings. By the same painter, Ibrahim Al-Nasekh, indigo was used alone as a dark pigment or admixed with lead white to give light blue shade. It was also used as a complex mixture with red lead (Pb3O4), lead white and dolomite [$MgCa(CO_3)_2$] (Abdel-Ghani et al. 2012).

Chemically, indigo (Indigofera tinctoria) contains indicant (indoxyl-β-D-glucoside) and indican precursors which serve as starting materials for indigo production. Indigo (indigotin, blue) is formed in damaged old leaves by the aerial oxidation of products of hydrolysis of these precursors. The chemical process of the formation of indigotin is carried out by the enzymes present in the leaves. Indican forms indoxyl and glucose then the colourless, soluble indoxyl is oxidised into blue, insoluble indigotin by aeration in the fermentation vat or by pouring the liquid from one container to another. It forms lumps to be ground for use as a pigment or reduction in alkaline solution for use as a dye.

5.1.2 The white paint area

The white paint was sampled from the neck of the Archangel (Fig. 1, sample 2). The characteristic bands revealed by the FTIR analysis appeared at 3405, 1410, 847 and 670 cm^{-1} suggesting the presence of lead white [$2PbCO_3$. $Pb(OH)_2$] (Ganitis et al., 2004; Bevilacqua et al., 2003).

The strong absorbance at 1410 cm^{-1} is attributed to the antisymmetric stretching vibrations of CO_3^- group and the peak at 681 cm^{-1} is associated with the rocking deformation of the same group (Ganitis et al., 2004).

The distinction between calcite and lead white can be achieved by infrared spectroscopy since the principal bands corresponding to lead carbonate appears approximately at 1400-1410 cm^{-1} while the one for calcium carbonate appears at higher values of the wave number at ~1440-1460 cm^{-1}.

The EDX spectrum of the same sample confirmed the application of lead white by yielding lead and calcium as main elements (Ganitis et al., 2004). Some more elements were also detected in the spectrum, namely; iron which is found in an appreciable amount, silicon, aluminium, sodium and potassium. These elements suggest the presence of clay and iron oxide compounds (Hradil et al., 2003) which may come from a bole layer applied underneath an adjacent gold area.

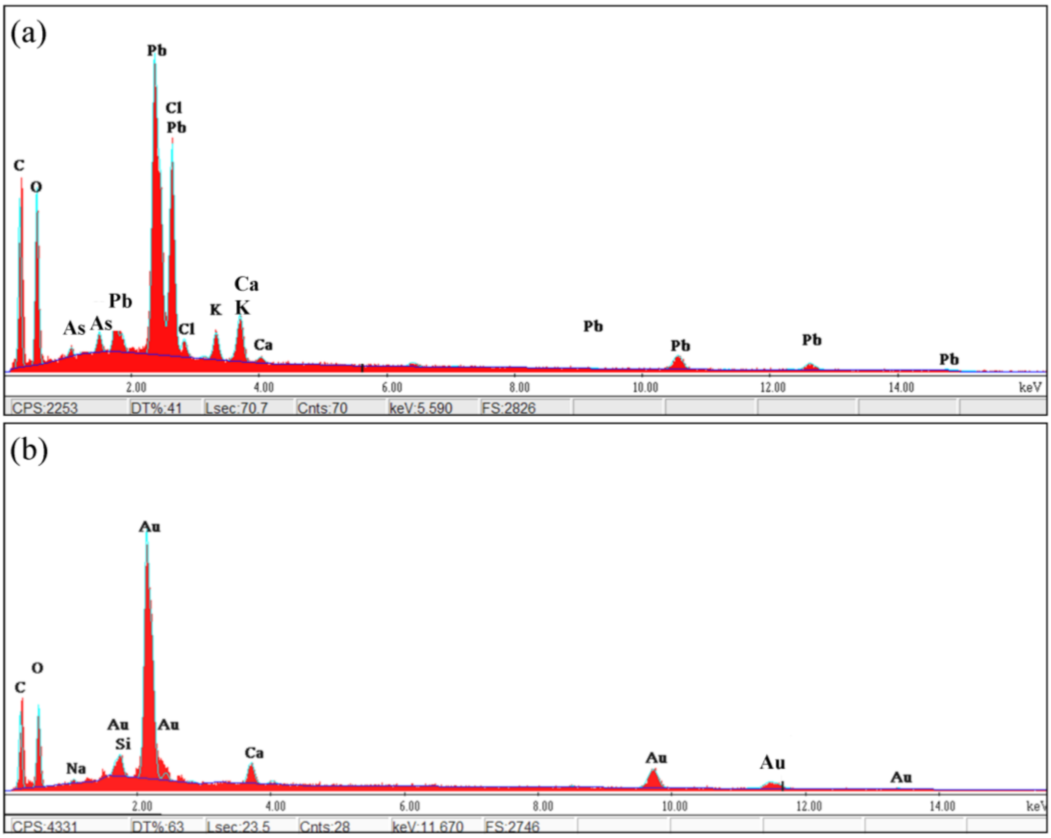

Figure 3 The SEM-EDX of (a) the blue paint sample (b) the yellow paint sample

Lead white was previously detected in two 18th century and a 19th century Coptic icons [3, 7] while hydromagnesite [$Mg_5(CO_3)_4(OH)_2.4H_2O$] was used in a 13th century Coptic Byzantine icon Abdel-Ghani et al., 2009). Gypsum, calcite, Barite and dolomite were sometimes used as filling materials in Coptic icons (Abdel-Ghani et al., 2012, 2009, 2008].

5.1.3 The yellow paint area

The yellow sample was taken from the left wing of the Archangel (Fig. 1, sample 3). This pigment was found to be very fragile and we found difficulties in sampling without causing harm to the pictorial layer.

The FTIR analysis failed to give any characteristic features that allows the identification of the pigment while the EDX spectrum revealed lead (Pb) as a predominant element suggesting the application of the lead containing pigment; massicot (orthorhombic lead (II) oxide, PbO) or litharge (tetragonal lead (II) oxide, PbO) (Eastaugh et al. 2004, p. 241). In addition, a significant content of sulpher and calcium (S, Ca) was also present, which most probably came from the white ground layer. The elements; potassium, silicon, aluminum, magnesium and phosphorous were also exist as trace elements in the yellow paint (Fig. 3, b).

Yellow lead has not been identified in the previously studied Coptic icons. Three yellow pigment have been employed in Coptic icons; yellow ochre which was identified in a 13th century Coptic-Byzantine icon (Abdel-Ghani et al., 2009), orpiment which was used in two 18th century icons (Abdel-Ghani et al., 2012) and Chrome yellow that was found in a 19th century icon (Abdel-Ghani et al., 2008).

5.1.4 The red paint area

Two red paint samples were examined of which one is lighter than the other. The dark one was sampled from the upper part of the Archangel's chlamys (Fig. 1, sample 4) while the second was originated from the under-tunic (Fig. 1, sample 5).

The Characteristic bands revealed by the FTIR analysis at 586, 558, 533, 509 and 485 cm^{-1} suggesting red lead as the red pigment applied (Bevilacqua et al., 2003). The EDX spectrum (Fig. 4, a) yields lead (Pb) as the main element confirmed that the pigment used is red lead. The presence of calcium and the very high percentage of lead (30%) may suggest the presence of lead white admixed with red lead.

The lead (II, IV) oxide, Pb_3O_4, is thought to have been one of the earliest artificially produced pigments and one which is still in use today. More commonly known under the name 'red lead', it is the synthetic analogue of the mineral minimum (Eastaugh et al., 2004, p. 256). The pigment was known as early as lead itself.

However, as reported by Lucas and Harris (1962, p.348) its earliest use commenced in Egypt during the Graeco- Roman times (Gettens and Stout 1966).

Red lead is either used in Coptic icons as a single red pigment or sometimes admixed with cinnabar, α-HgS, to reduce the cost of using cinnabar, the most expensive red pigment [Abdel-Ghani et al. 2012; Howard 2003).

5.1.5 The gold area

The cross-section of the gold sample taken from the halo (Fig. 1, sample 6) was investigated under the optical Microscope. It was found that the gold leaf was applied over an orange underlayer of bole. The Characteristic bands revealed by the FTIR analysis of the gold layer appeared strongly at 526 cm^{-1} and less intense at 501, 600, 647 and 669 cm^{-1}.

The gold layer is made with a very thin gold leaf, around 2–5 μm, applied on a gesso layer covered with an orange ochre layer of the bole. The function of this coloured layer of fine bole is double; a chromatic one which is giving a warm red or ochre tonality to the hue of the gold surface, and allowing further treatments of the metallic leaf by burnishing (Crina Anca Sandu, et al., 2011).

The same result was confirmed by using EDX analysis which gave rise to gold (Au) as a main element, beside calcium silicon and sodium as trace elements (Fig. 4, b) (Fig. 4, b).

The absence of silver from this sample is of interest, as the previously studied gold leaf contained different amount of silver as a trace element. The purity of Egyptian gold ranges from high purity to those containing 40% silver (Colinart, 2001, p.1) and about 1.4% copper (Hatchfield and Newmann, 1991, p. 27).

5.2 The white ground layer

The cross-section examination showed the application of the white ground layer as a single layer on which the paint layers were directly executed. Gypsum was use in the white ground layer mixed with animal glue as an organic medium. The FTIR spectrum of the ground layer revealed absorbance bands at 3546, 3405 and 1621 cm^{-1} which are attributed to water molecules (Ganitis et al., 2004; Daniilia et al., 2002). In addition to bands at 2927 and 2855 cm^{-1} which are ascribed to the methylene groups of animal glue [4, 15, 16], while the bands at 1117 and 1139 cm^{-1} are attributed to the sulfate group (SO_4^{2-}) (Daniilia et al., 2002).

The EDX analysis of the white ground layer (Fig. 4, c) confirmed the application of gypsum, calcium sulphate dihydrate ($CaSO_4$. $2H_2O$), as a white ground layer. It revealed calcium and sulpher as main elements, in addition to chlorine and silicon as trace elements

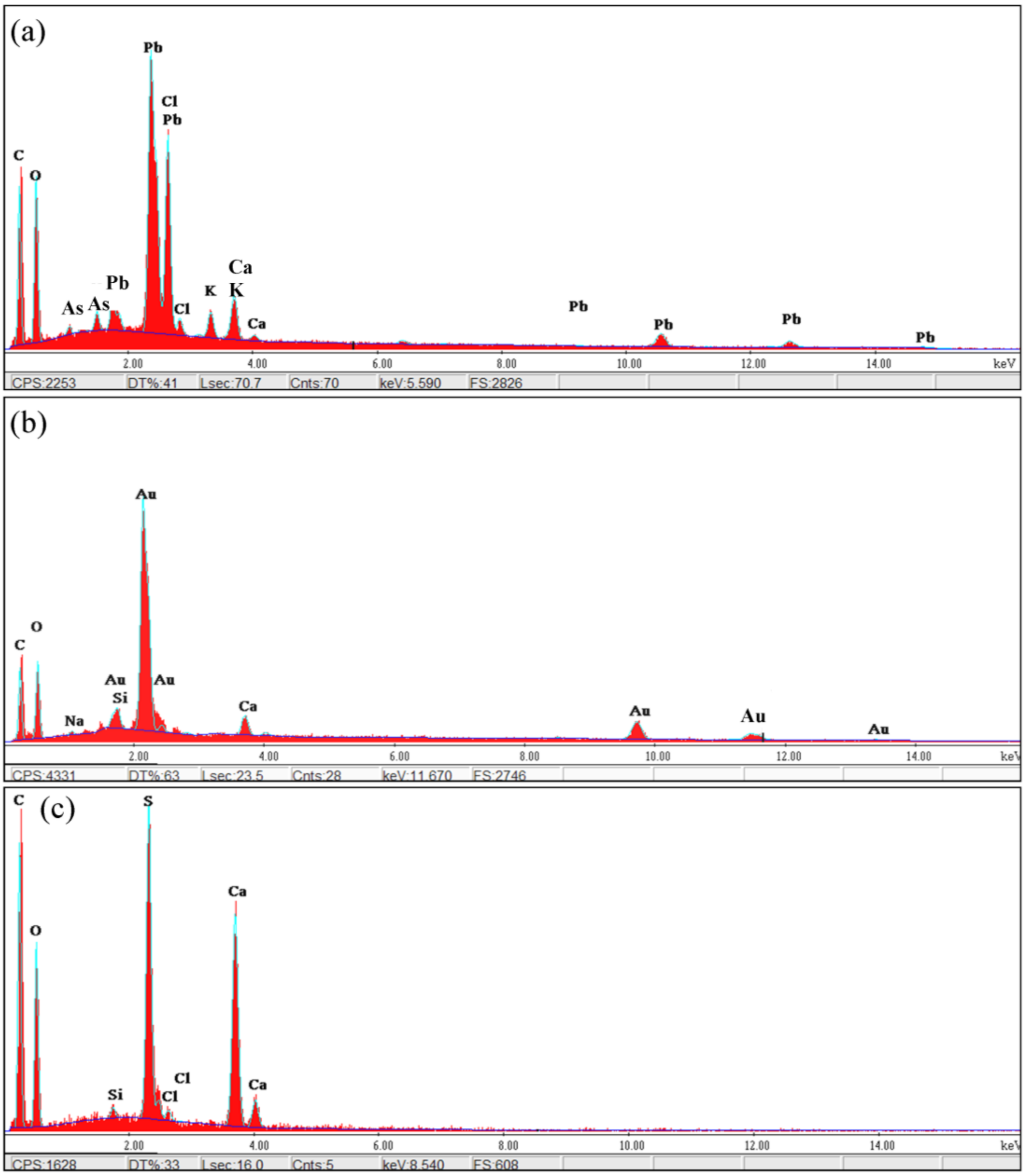

Figure 4 The SEM-EDX of (a) the red paint sample (b) the gold area (c) the white ground layer

The application of the gesso in a single layer is the structure that normally found in Coptic icons (Abdel-Ghani et al., 2012, 2009). Two exceptions have been reported which comprised two different ground layers. The first was applied by Ibrahim Al-Nasekh as two distinct layers of which the upper was made of lead white while the under layer was made of gesso (Abdel-Ghani et al., 2012). The other exception was found on a 19th century icon by Anastasi el-Romi in which both layers were made of gesso with different amount of animal glue (Abdel-Ghani et al., 2009).

5.3 The wooden panel

5.3.1 The Anatomical Pattern of wood

No previous analytical studies were performed on the Coptic icon's wooden panels. However, it was suggested by Skalova in 1995 that the icons' panels were made from local wood which was mostly sycamore. She also stated that acacia, cypress, olive and tamarisk may sometimes be used in addition to some imported wood (Skalova, 1995).

In our case study, the ESEM for the wooden support showed the anatomical pattern attributed to the Acacia species (family: Lenguminosea) which is based on various species. All Acacia types are very similar in many anatomical features such as the growth ring boundaries, the size of fibers, the different types of parenchyma including; marginal, axial, paratracheal vasicentric tracheid, aliform, confluent, banded, apotracheal. The distinction between the species is normally based on quantative and /or variable qualitative features such as ray height, ray width, crystal diameter and parenchyma type.

In the ESEM micrographs of the icon's support (Fig. 5, a-c), vasicentric parenchyma in the longitudinal and radial section was obvious. It appeared wider than the one found in Acacia niloltica (L) Willd suggesting being Accacia Pennata (L.) Wild (Fig. 5, a-c).

tion work, furniture, coffins, bows, arrows and dowels (Gale et al., 2000, p. 334).

By studying the ESEM micrograph of the traverse samples (Fig. 5, d-f), it was suggested that the wood analyzed was Cupressus sempervirens L. var. dupreziana (A. Camus) Silba (Cupressaceae family) (Amri et al., 2013; Normand, 1950, 1956, 1960). This was established by the presence of its anatomical features such as the tracheid short (size class < 3000µm long), the bordered pits, the uniseriate ray in stem wood (Fig. 5, d), in addition to, the absence of the resin duct (canals) and the presence of axial tracheids of polygonal section. The slightly-defined growth rings, the bordered pits present on the tangential walls of the axial tracheids (Fig. 5, e, f) and the ray height ranging between 1 to 12 cells (Fig. f, d) are also supported our results.

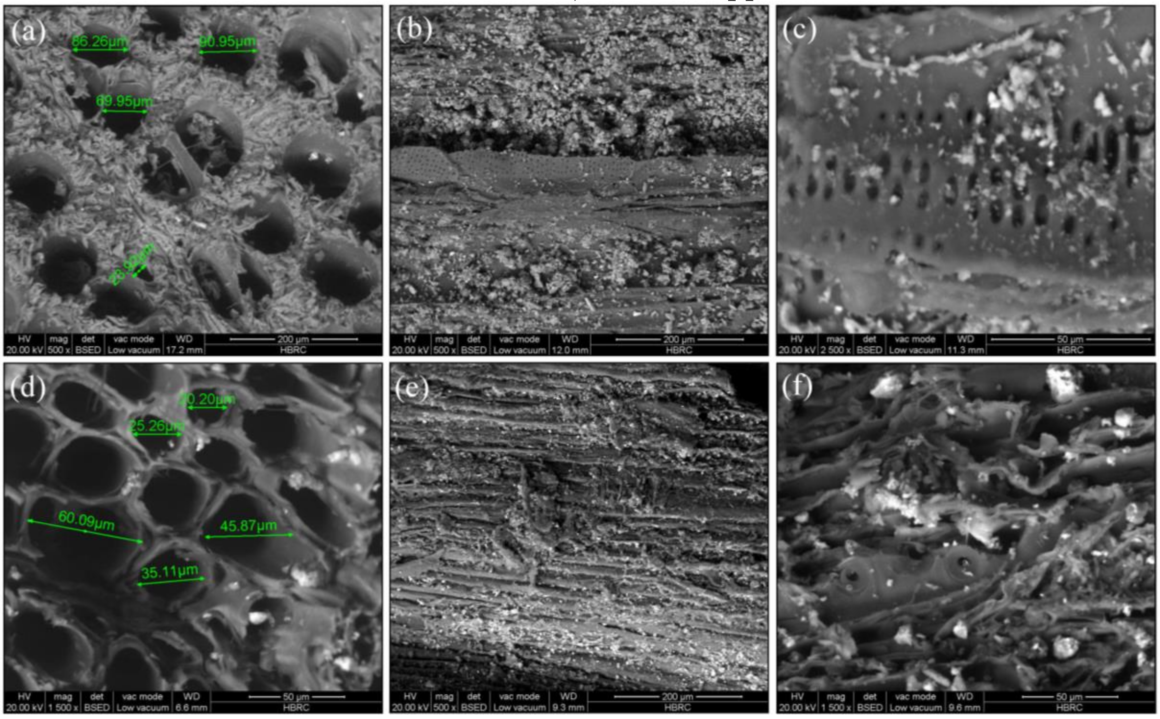

Figure 5 SEM micrograph of (a-c) the wooden panel, Acacia Pennata (L.) Willd (a) transverse view showing Vasicentric parenchyma (b) longitudinal view showing foreign deposits presumably resinous material mixed with dust. Erosion and defibration of cell wallsare evident (c) Radial view showing the parenchyma with deterioration of simple pits and detachment within cells due to fungal attack (d-f) the traverse wood, *Cupressus* sempervirens L. var. dupreziana (d) transverse view showing Vasicentric parenchyma (e) Radial view showing deterioration of bordered pits resulting from weathering and foreign deposits on the wood surface (f) radial view showing deterioration of bordered pits.

The Acacia tree is found in hot deserts in Egypt, Jordan and Iraq. It is also found in tropical Africa. In ancient Egypt, Acacia wood was used for boat-building, construc-

Cypresses are normally found in hilly locations (from 300 to 1500 meters above sea level), in Jordan, Syria, Lebanon, Turkey, Iran, Crete and Rhodes (Amri et al., 2013). Cupressus sempervirens L. var. dupreziana

(A. Camus) Silba, Saharan cypress, is a very rare coniferous tree native to the Tassili n'Ajjer mountains in the central Algerian Sahara Desert (Normand, 1956). In ancient Egypt, Cypresses used in construction work, doors, furniture, boat building and carving (Gale et al 2000, p. 350). This species is distinct from the Cupressus sempervirens, Mediterranean cypress, which can be difficultly identified through its distinct morphological features (Gale et al., 2000, p. 350, Neumann et al., 2001).

6. CONCLUSIONS

The examination of the Coptic wooden icon of Archangel Michael by optical microscopy, Scanning electron microscopy coupled with Energy Dispersive X-ray analysis and Fourier Transform Infrared – Attenuated Total Reflectance spectroscopy revealed that the icon consists of a wooden panel made of Acacia species, precisely; Accacia Pennata (L.) Wild and strengthened with three traverse of Cupressus sempervirens L. var. dupreziana (A. Camus) Silba. It was also revealed that Ibrahim Al-Nasekh had used limited pigments to produce a simple palette. The pigments identified are indigo, red lead, white lead and yellow lead. Real gold leaves over an orange bole layer were used in the golden background and in some ornamental details.

Comparing the wooden icon investigated in this study with the well-established technique of two icons painted by the same painter (Abdel-Ghani et al., 2012), it was found out that both types are similar in all aspects except; the absence of the intermediate canvas layer and the application of lead oxide as a yellow pigment in the wooden icon.

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