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SAVING THE MUMMY'S SHELL: AN INTERDISCIPLINARY APPROACH FOR ANALYSIS AND RESTORATION OF CARTONNAGE MUMMY CASE FROM EL-LAHUN EXCAVATIONS, MIDDLE EGYPT

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ABSTRACT

In this inquiry, a polychrome cartonnage mummy case, discovered at the archaeological site of El-Lahun at Middle Egypt, was investigated. Fragmented cartonnage samples were non-destructively examined by digital microscope and high resolution field-emission scanning electron microscope, which was outfitted with an energy-dispersive X-ray spectrometer (FE-SEM/EDX). For further understanding, spectroscopic study on some samples was fulfilled by Fourier-transform infrared spectroscopy (FT-IR). The results implied linen fibres as the base support, while thin calcareous layer mixed with gum Arabic worked as preparatory layer 'gesso'. The pigments on the cartonnage palette contained Egyptian blue (synthetic blue cuprorivaite), orpiment (brilliant arsenic sulfide mineral), yellow ochre (iron oxyhydroxide, silica, and clay mineral), and red ochre (iron (III) oxide, silica, and clay mineral). What's more, the green pigment was created through mixing together amounts of Egyptian blue and yellow ochre. While the yellow paints contained a blended layer of yellow ochre and orpiment. The results certified that gum Arabic was the adhesive used in both the 'gesso' and pictorial layers. Extreme physical damage, salt efflorescence, and biological attacks were documented on the cartonnage. Fungal species of *Aspergillus flavus*, *Aspergillus niger*, *Cladosporium cladosporioides*, and *Penicillium spp.* were verified. To minimize the damage and maintain the condition of the cartonnage, a unified restoration project was requested. The procedures covered disinfection using eco-friendly plant extracts-essential oil, cleaning, consolidation of the fragile surface, stabilizing the paint layers, reinforcing detached areas, and filling missing parts. Particularly, the results provided appreciable data on the painting materials and the proposed date of the cartonnage.

KEYWORDS: El-Lahun excavations; Cartonnage; Damage; FE-SEM/EDX; Pigments; Plant extracts; Restoration

1. INTRODUCTION

The ancient Egyptians had practiced fascinating funeral traditions to their mummies. An important feature was the encapsulation of the wrapped body into a decorated container or cartonnage case, which is then preserved inside a coffin, usually of wood (Sakr *et al.*, 2020). Each cartonnage case was designed to cover parts of the body, by multiple pieces (up to 6), or as complete segment for the whole mummy (Bartos, 2013; Cooney, 2015). A model cartonnage case combines: a) support base commonly made of linen or papyri sheets glued together, b) preparatory white layer 'gesso', and c) pictorial layer which represents funerary scenes and texts. On occasions, a varnish layer was employed to ensure additional protection. Since the 12th Dynasty (ca. 1991–1786 B.C.), cartonnage cases had become a prevalent custom, but their extensive employment was reported during the First Intermediate Period (ca. 2134–2040 B.C.) (Hussein *et al.*, 2020). Throughout the Ptolemaic and Roman periods, cartonnage was a favourite complementary technique to preserve the mummies (Scott *et al.*, 2009). Deserving of note that thin cartonnages were produced during the Ptolemaic era. But on contrary, thick plaster layers on coarse fibrous base were a distinguishable character for the Roman cartonnages. Often, linen fibres were constant materials used in making the cartonnage, but since the time of Ptolemy III (ruled from 246 to 222 B.C.), reused papyri scrolls were alternatively used (Gibson *et al.*, 2018).

Undoubtedly, the investigation of ancient Egyptian composite objects is a challenging concern. Respecting the characterization of the chemical structure of painted textiles and textile-based polychrome objects, several analytical attempts have been directed. In an excellent way, the micro-equipments allow a complete structural-chemical profile about each single pigment and the underlying layers. The implementation of Raman spectroscopy and reflectance spectroscopy with Laser-Induced Breakdown Spectroscopy (LIBS) showed remarkable benefits to study heritage composite materials, e.g. cartonnages (Edwards *et al.*, 2004; Siozos *et al.*, 2017), and for 712 BC until 332 BC cartonnages with multi analytical techniques by Ali *et al.*, (2020). Appreciably, the contribution of multiple analytical methods on Ptolemaic cartonnages assisted the reorganization of painting materials and chromatic alterations (Ali *et al.*, 2018; Gard *et al.*, 2020). By means of μ -Raman and Total Reflection X-ray Fluorescence (TR-XRF) spectrometers, precious arsenic-based pigments (Orpiment and Realgar As_4S_4) were tracked in Ptolemaic masks and mummy cartonnages (Vandenbeebe *et al.*, 2001). Since the Late Period (ca. 712–323 B.C.), the artists used pigment mixtures to prepare green colours (Marey Mahmoud,

2014). Laser-induced breakdown spectroscopy and scanning electron microscopy were selected to differentiate pigment mixtures on a Graeco-Roman cartonnage from Saqqara (Abd El Aal, 2014).

From conservation point of view, by the influence of the same deterioration agent, each single material in composite objects reacts in a specific way. In addition, the physical behaviour affects strongly the inner matrix, causing mechanical-chemical damage. The burial conditions, as well as the surrounding microclimate conditions, critically participate in the deterioration process. Accordingly, the requirements for an appropriate treatment are a necessity. Such studies contribute to the at-risk cultural heritage protection (Sideris *et al.*, 2017).

Since the time of its discovery 11 years ago, this study describes the first analytical and restoration approach applied on highly damaged cartonnage from a distinctive collection discovered at El-Lahun archaeological site and currently stored in the museum-storeroom of Kom Oushim at Fayoum. The results of this analytical study helped to correct archaeological data concerning the date and the materials used in the studied cartonnage. Thus, further analyses on the whole collection will establish an analytical database in terms of materials and manufacturing technology.

The aims of the present work were designed to cover three main points: (i) to determine the compositional structure of a cartonnage mummy case from El-Lahun excavations, Middle Egypt, (ii) taking the same importance, to diagnose the deterioration forms on the cartonnage, and (iii) to apply a combined restoration process to the cartonnage. Further, the study succeeds in presenting an eco-friendly disinfection process based on plant extracts and essential oils, and to emphasize its efficiency to be used as an alternative method for similar objects in the Egyptian museums.

1.1. The excavation site

El-Lahun site is located about 20 km far from the city Fayoum, and 100 km south of Cairo (Fig. 1). Early excavations in the site were complemented by the British archaeologist F. Petrie, in 1888–90 and in 1914. On 26 April 2009, the Egyptian Supreme Council of Antiquities (SCA) announced a significant discovery which included several rock tombs with amazing findings. The date of these findings goes back to the Eleventh and Twelfth Dynasties (ca. 2030–1840 B.C.), the New Kingdom, the Late Period, and the Roman age. Among the discovered artefacts, twelve wooden coffins were uncovered. The coffins were 'accumulated' on each other, suggesting a cache of multi-chronological objects. In *situ* observations led to conclude that the coffins contained mummies that were preserved in painted cartonnage cases.

1.2. The studied cartonnage

i. Description: A decorated cartonnage case with wrapped body, probably of an official man. The cartonnage expressed serious state of preservation (a detailed interpretation will be given later). The male-shape has been represented with two arms clasped beneath the chest area. The face area is painted white and surrounded by a blue wig, which completely hides the ears. The main part of the cartonnage is decorated with unpopular form of the god 'Khnum', wearing the 'Atef' crown, in a winged shape carrying the eternity symbol 'Shen', caught by his claws. Further, the observations performed on the cartonnage suggested a featured artistic style belongs to the northern Upper Egypt collection which covered a

huge district, from Beni Hassan to the Memphite necropolis. This design is distinguishable because the ancient artist used to create the motifs in a centred single stripe over the *gesso* layer (Taylor, 2009).

ii. Date and Geography: Third Intermediate Period (Dynasties 22–25, ca. 945–664 B.C.), at El-Lahun archaeological site (Tomb no. 27)

iii. Medium: Linen base, *gesso* layer, paint layer (pigments + medium)

iv. Gender/name/age of the mummified body: Male/demolished/unknown

v. Dimensions: Length: 180 cm, Height: 22 cm, Width: 37 cm

vi. Current location: the museum-storeroom of Kom Oushim, Fayoum (under registration number: 236).

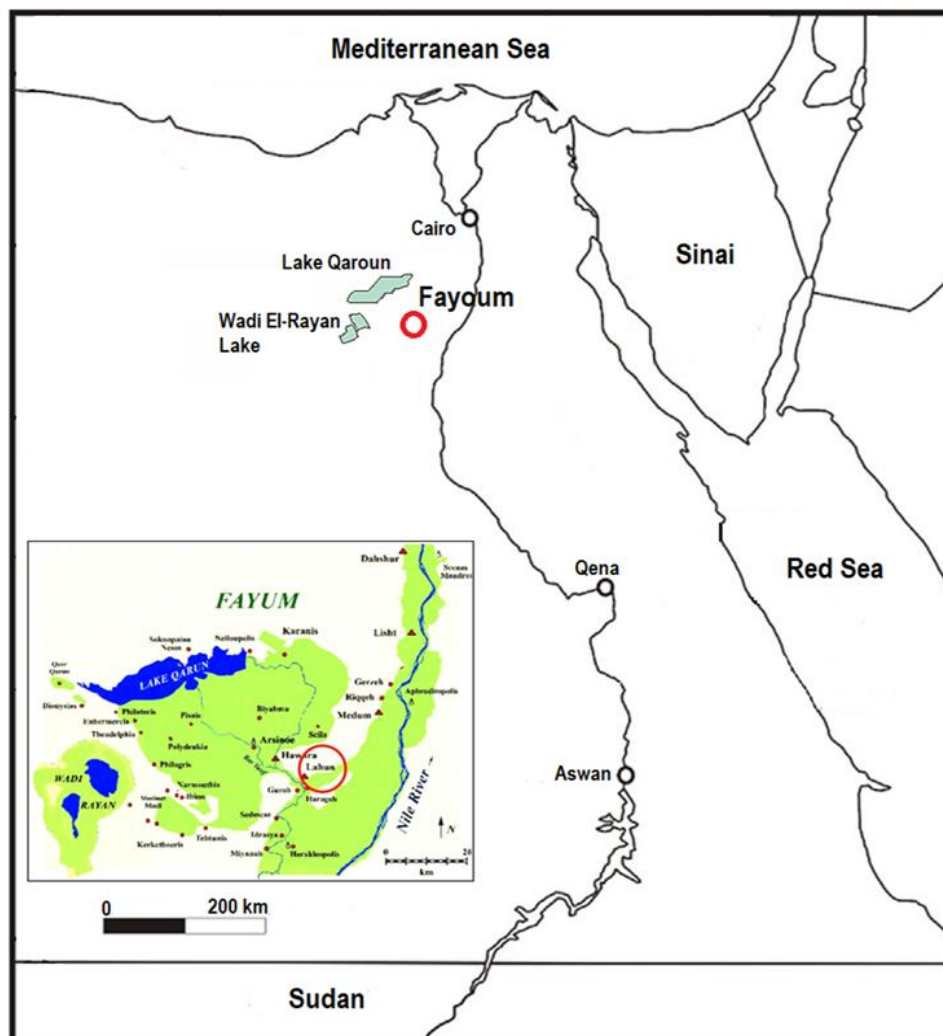


Figure 1. Schematic map of Egypt and close-up map of Fayoum region, the location of El-Lahun site is highlighted by a circle.

1.3. State of preservation

In Fig. 2, the main deterioration aspects on the studied cartonnage are recorded. The simple examination exhibited physical weakness with a notable

wrapping. Microbiological and insect attacks were clear in form of spots, holes and broken edges. Beside the burial environment, the insufficient storage conditions in the museum-storeroom of Kom Oushim, led to additional influential damage. The condition

monitored that the cartonnage has an irregular surface with different push-up areas. Several deterioration forms were registered, including micro cracks, decomposition/carbonization of the linen fibres, exfoliation and paint flakes, detachments, areas of loss,

crumbling of chest area, distortion, and dust accumulations. The feet area and the right side displayed a severe damage, and the textile fabrics were partially exposed.



Figure 2. Serious damage of the cartonnage: detachments, areas of loss, and holes produced by insects are observed.

2. MATERIALS AND METHODS

2.1. Samples

The studied cartonnage is painted with a palette contains white background covered with blue, red, black, and yellow pigment particles. Whereas the cartonnage was damaged heavily, numerous tiny detached pieces were available for micro and non-destructive investigation.

2.2. Materials

Conservation materials used included:

1- Klucel® G: hydroxypropylcellulose (HPC) is a form of cellulose that non-ionic water-soluble, produced by CTS Srl, Italy.

2- Paraloid™ B44: a solid grade acrylic co-polymer, can be dissolved easily in a variety of organic solvents, produced by Rohm and Haas, USA.

3- Microballons: Hollow Glass Microspheres (HGM), obtained by thin walled glass bubbles and usually used as a filler, with an approximate size of 50 µm.

4- Ethanol: (Ethyl alcohol, CH₃CH₂OH; ACS reagent, ≥99.5%), produced by Sigma-Aldrich.

5- Acetone: ACS reagent ≥99.5%, produced by Sigma-Aldrich.

6- Tissue paper: an acid-free tissue paper (pH= 7–7.5).

7- Plant extracts: extracts of Frankincense and common Juniper were prepared by solvent extraction,

while clove essential oil was obtained from the aerial parts of plants by hydro distillation.

8- Tulle: lightweight nylon fabrics, used to support the cartonnage.

2.3. Analytical instruments

A detailed microscopic documentation of the cartonnage was necessary to access the main chromatic features and the state of preservation. The examination was made by a “DNT” digital easy-to-use microscope (dnt, Germany) which operates at magnifications up to 500x, with 1µm accuracy. The morphological-anatomical features of the textile fibres and paint layers, in addition to their chemical content, were evaluated using a high resolution field-emission scanning electron microscope FE-SEM (Model: Quanta FEG 250, The Netherlands), which is combined with an energy-dispersive X-ray analyzer (EDX). The microanalysis was resolved through an Oxford Aztec software. Instrument accelerating voltage of 20 kV was used for all samples. To achieve a molecular interpretation, some samples were analyzed by a Jasco model 4100 Fourier-transform infrared spectrometer (FT-IR) (Jasco, United Kingdom). Few milligrams of each sample were mixed and encapsulated with a KBr powder. Generating the spectra was operated in the mid-Infrared region (400–4000 cm⁻¹), with co-added accumulation of 32 scans and a resolution of 4 cm⁻¹.

3. RESULTS AND DISCUSSION

Table 1 unifies the micro-chemical analysis, by EDX unit, (Element, At. %) of the studied samples. Below the fabric structure, the gesso layer, the pigment

layers (blue, green, yellow, red), the deterioration forms and restoration and treatment results, are presented.

Table 1. Chemical composition of the studied samples, measured by EDX (At.%)

Elm.	Linen fibres	Gesso layer	Blue paint	Green paint	Yellow paint	Yellow paint	Salt deposits	
C	39.21	10.98	16.87	15.25	16.78	15.08	20.43	22.72
N	0.75	-	-	-	-	-	-	-
O	54.16	67.9	61.82	61.09	59.59	49.93	56.32	38.3
Na	2.23	-	-	1.02	-	1.57	3.76	18.54
Mg	-	-	-	1.51	-	2.33	-	-
Al	-	-	-	1.27	1.52	1.69	-	-
Si	-	-	7.55	7.11	3.32	3.96	0.55	-
S	-	-	-	-	3.82	1.65	7.27	-
Cl	1.02	-	-	-	-	-	1.79	9.63
Ca	2.62	21.13	12.29	9.32	7.88	10.6	9.87	-
Fe	-	-	-	0.96	0.98	1.45	-	-
Cu	-	-	1.47	1.41	-	-	-	-
As	-	-	-	-	6.1	-	-	-

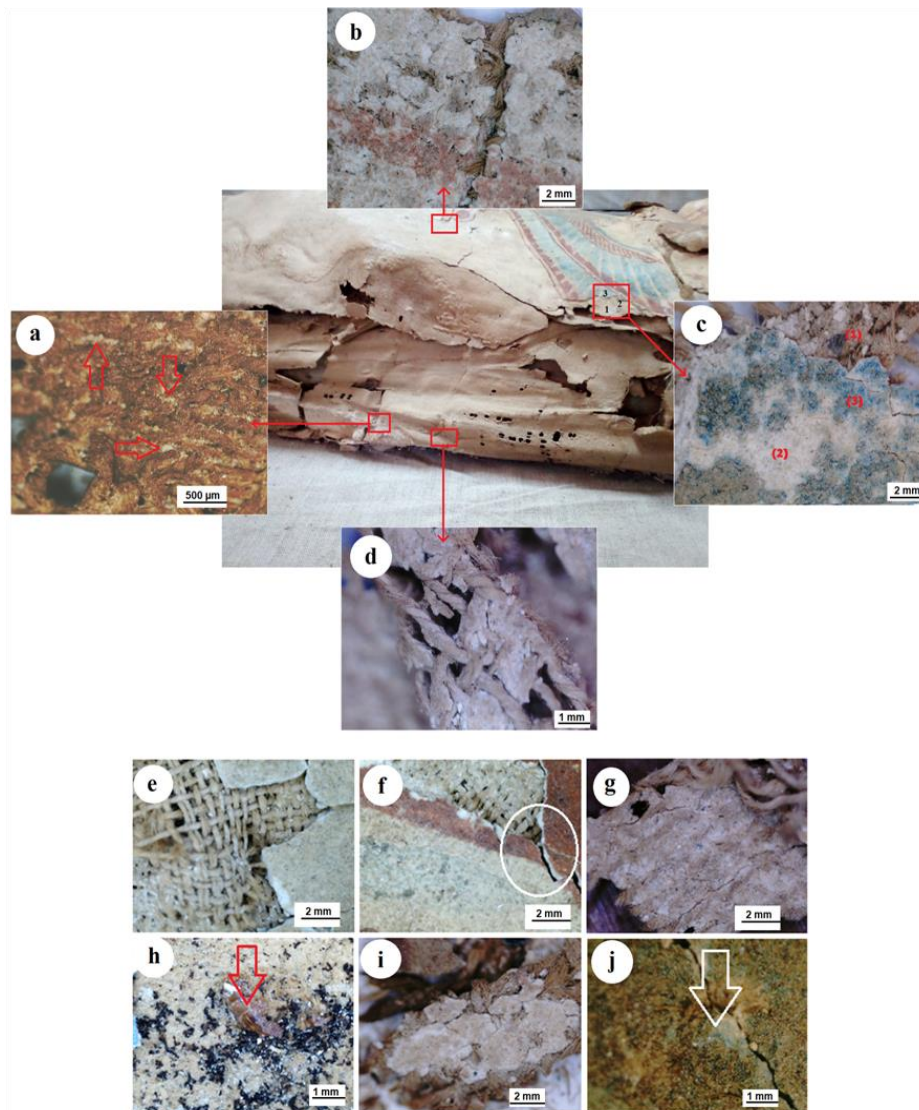


Figure 3. Microscopic images captured on the surface of the cartonnage: a) salt deposits over the linen fibres, b) wide crack in the gesso layer, c) stratigraphic profile of the paint layers, d) some damaged fibres, e) exposed areas of the fabric matrix, f) cracks and detachments in the paint layers, g) broken fibres, h) insect attack, i) fragile gesso layer, and j) wide crack and chromatic alteration in the paint layers.

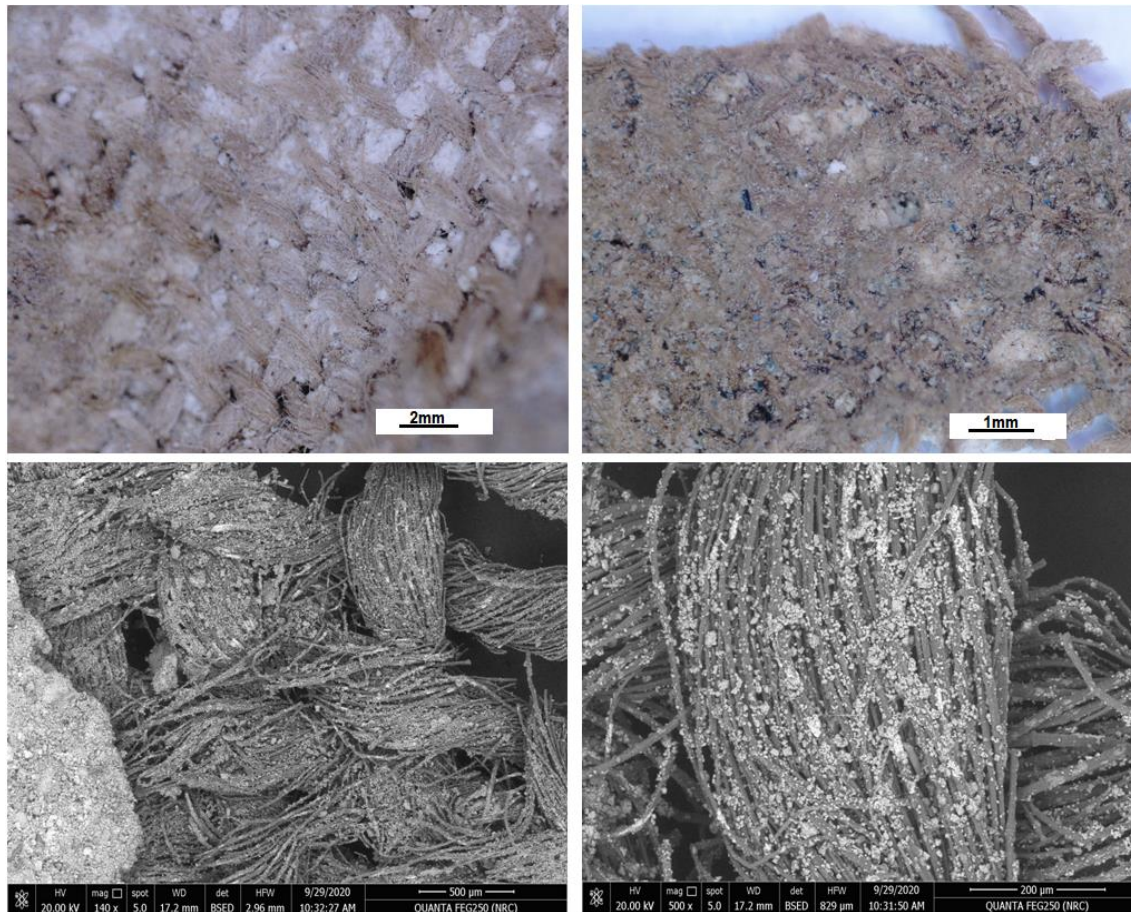


Figure 4. Microscopic images (up) and FE-SEM micrographs (bottom) recorded on the fabric structure.

3.1. The fabric structure

Fig. 3 displays a collection of microscopic images, which highlights the structure of paint layers and some deterioration forms, mostly the physical damage occurred to the flax fibres. A sample of the linen fabric was microscopically investigated (Fig. 4, up), in which the linen fragment appeared as fine-woven textile and the *gesso* particles deposited in the pores between fibres. Low magnification (140x and 500x) FE-SEM investigation on the sample (Fig. 4, bottom), revealed a slightly high thread count which reflects the quality of the textile weaving process used to manufacturing the cartonnage. Observing the microscopic appearance, the fabric appeared with damage and broken-detached fibres. White *gesso* deposits and some salt crystals, were observed onto the fibres. The EDX analyzer showed high concentrations of oxygen (O, 54.16%) and carbon (C, 39.21%), together with calcium (Ca, 2.62%), sodium (Na, 2.23%), chlorine (Cl, 1.02%), and nitrogen (N, 0.75%) (Fig. 5a).

The prevalence of sodium chloride salts is very likely due to the saline soil of the burial site. A minute portion of the fabric structure was studied by the FT-

IR spectrometer (Fig. 5b). The spectrum unveiled featured peaks at 3350 cm^{-1} (for the free O-H bond), 2912 cm^{-1} (representing the $\nu(\text{C-H})$ vibration), 1625 cm^{-1} (which expresses H_2O in the cellulose structure) (Margariti, 2019). A supported group of peaks are present at 1056 cm^{-1} ($\nu\text{C-O-C}$, distinctly imputed to glycosidic ether), 1374 cm^{-1} (which is related to $\delta(\text{C-H})$), 1323 cm^{-1} (consistent with $\delta(\text{CH}_2)$). The bands at 1161 and 1030 cm^{-1} are correlating with ($\nu(\text{C-C})$) and ($\nu(\text{C-OH})$), respectively (Cao *et al.*, 2010). Calcite peaks were noted at 1798 , 1426 , 874 , and 710 cm^{-1} . Other, Si-O stretching vibrations (of quartz) at 477 and 600 cm^{-1} together with S-O bending band (of gypsum) at 663 cm^{-1} , were issued. Immensely, the ancient Egyptians harvested flax plants for weaving textile-based objects (Clark, 1944; Vogelsang-Eastwood, 1992). Talking about technique, painting on fibres is a simple technique, usually executed after finishing the weaving. Then, fibres are treated with animal glue, followed by brushing the fabric with fine render (Maehler, 1980), or alternatively, saturating the fibres into *gesso* suspension. After the complete drying, the paints are applied according to the pre-designed drawings. Here, it is worthy to account that painting on textile was probably applied for limited purposes, e.g. funerary objects.

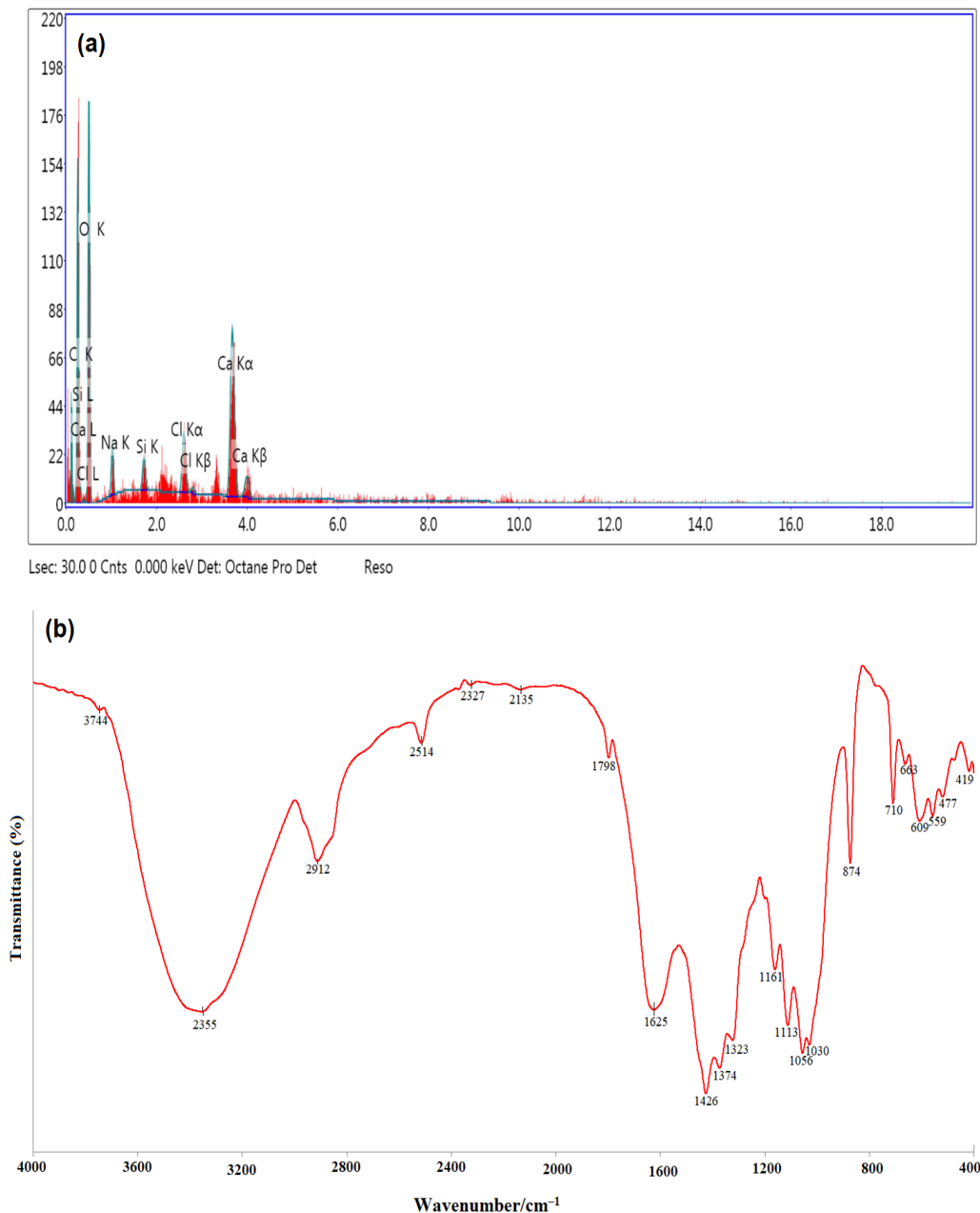


Figure 5. (a) EDX of energy KeV vs counts, spectrum and (b) FT-IR spectrum with respective peaks and troughs recorded on the fibre sample.

3.2. Gesso layer

FE-SEM morphology of the *gesso* layer demonstrates smooth grains (Fig. 6a, up). The EDX microanalysis measured atomic concentrations of carbon (10.98%), oxygen (67.9%), and calcium (21.13%) (Fig. 6a, bottom), which reference the presence of calcium carbonate (Calcite, CaCO_3) (Abdelmoniem et al., 2020). A microscopic image of the studied sample suggests that the *gesso* layer was brushed directly onto the linen fibres. Micro-morphological inspections of several points on the sample allowed the detection of featured crystals (Fig. 6b, up). The EDX spectrum of these crystals (Fig. 6b, bottom), gave major atomic concentrations of calcium (Ca, 9.87%) and

sulphur (S, 7.27%), which imply phases of calcium sulphate.

Substantial values of sodium (Na, 3.76%) and chlorine (Cl, 1.79%) indicate accumulations of sodium chloride (Halite, NaCl). The analysis using FT-IR technique of this layer (Fig. 6c), revealed calcite featured absorption band at 1420 cm^{-1} , which is assigned to the asymmetric stretching mode of carbonates (Marey Mahmoud and Abo El-Yamin, 2020). Complementary absorptions appeared at 874 cm^{-1} ($\omega(\text{C}=\text{O})$ wagging and 710 cm^{-1} (associated to CO_3 ion). Combination modes at 1799 and 2513 cm^{-1} , as well Si–O–Si vibrations at 665 and 416 cm^{-1} , were also detected. An indication on the existence of organic material was

suggested for bands at 3437 cm^{-1} (linked to hydroxyl stretch vibration), 2919 and 2872 cm^{-1} (for C–H bond). The symmetric O–H stretching band recorded at 1619 cm^{-1} is for the water molecules related to carboxyl group. Besides, peaks in the region ($1200\text{--}900\text{ cm}^{-1}$) are linked to carbohydrates (Kharbade and Joshi, 1995).

Building on these analyses, it was concluded that the *gesso* layer in the studied cartonnage is made of calcite combined with gum Arabic. Similar composition was previously reported by Afifi *et al.* (2020) in a

cartonnage from El-Lisht archaeological site. A microscopic image accompanied to the FT-IR spectrum on a cross-section of the *gesso* layer shows a white thick irregular layer (with a thickness $\sim 2\text{ mm}$) over the linen fabrics. It was pointed out by several scholars that the *gesso* formula used by the ancient Egyptians was made through a mixture of calcium carbonate, or calcium sulphate, and an organic adhesive (Hatchfield and Newman, 1991). In this direction, different plant gums were identified in *gesso* layers in multi-chronological cartonnages (Wright and Wheals, 1987).

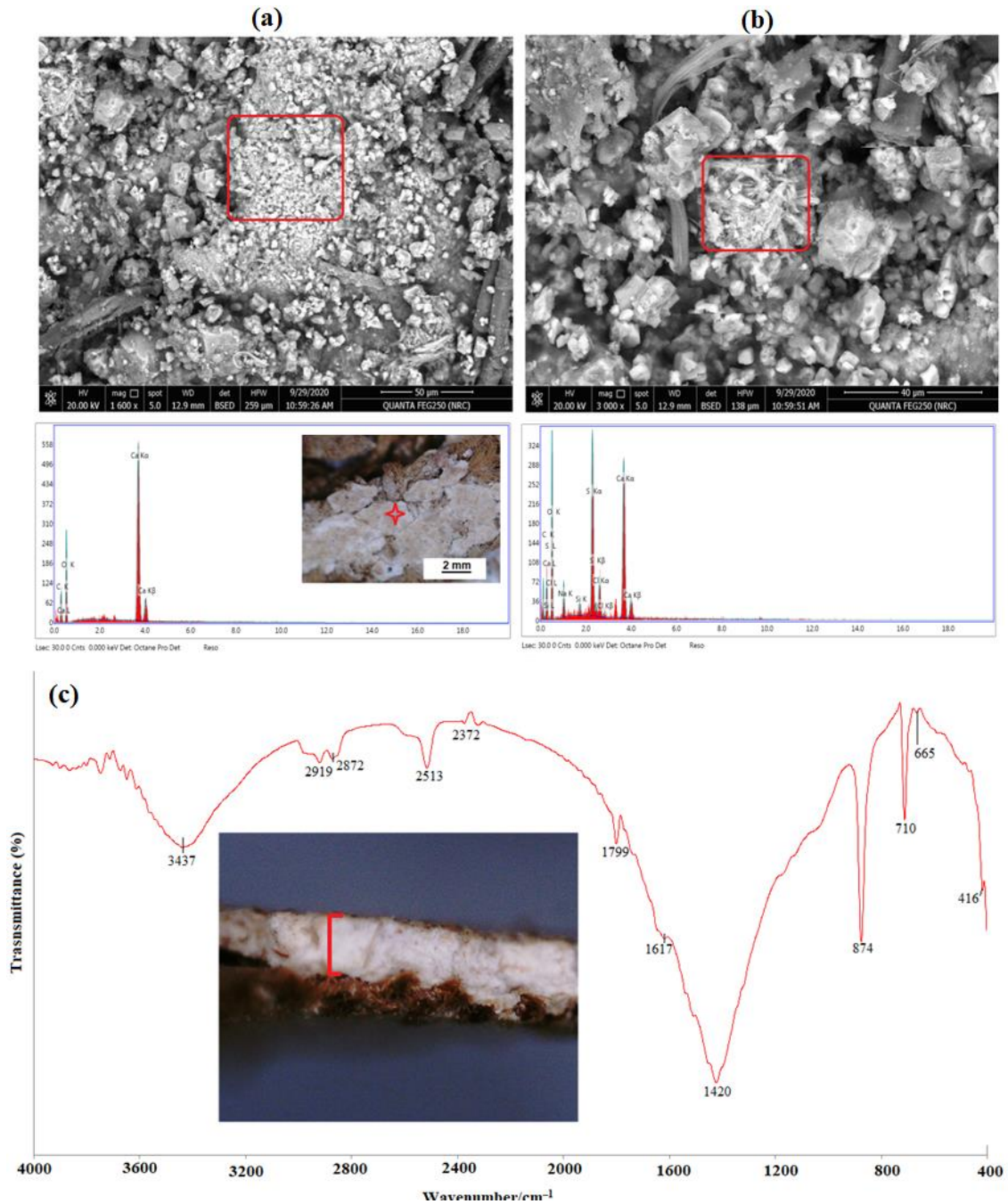


Figure 6. a) FE-SEM micrograph and EDX spectrum of the *gesso* sample, b) FE-SEM micrograph and EDX spectrum of some crystals (of gypsum) in the matrix, c) FT-IR spectrum obtained on the sample (a microscopic image on a cross-section of the *gesso* layer is also included).

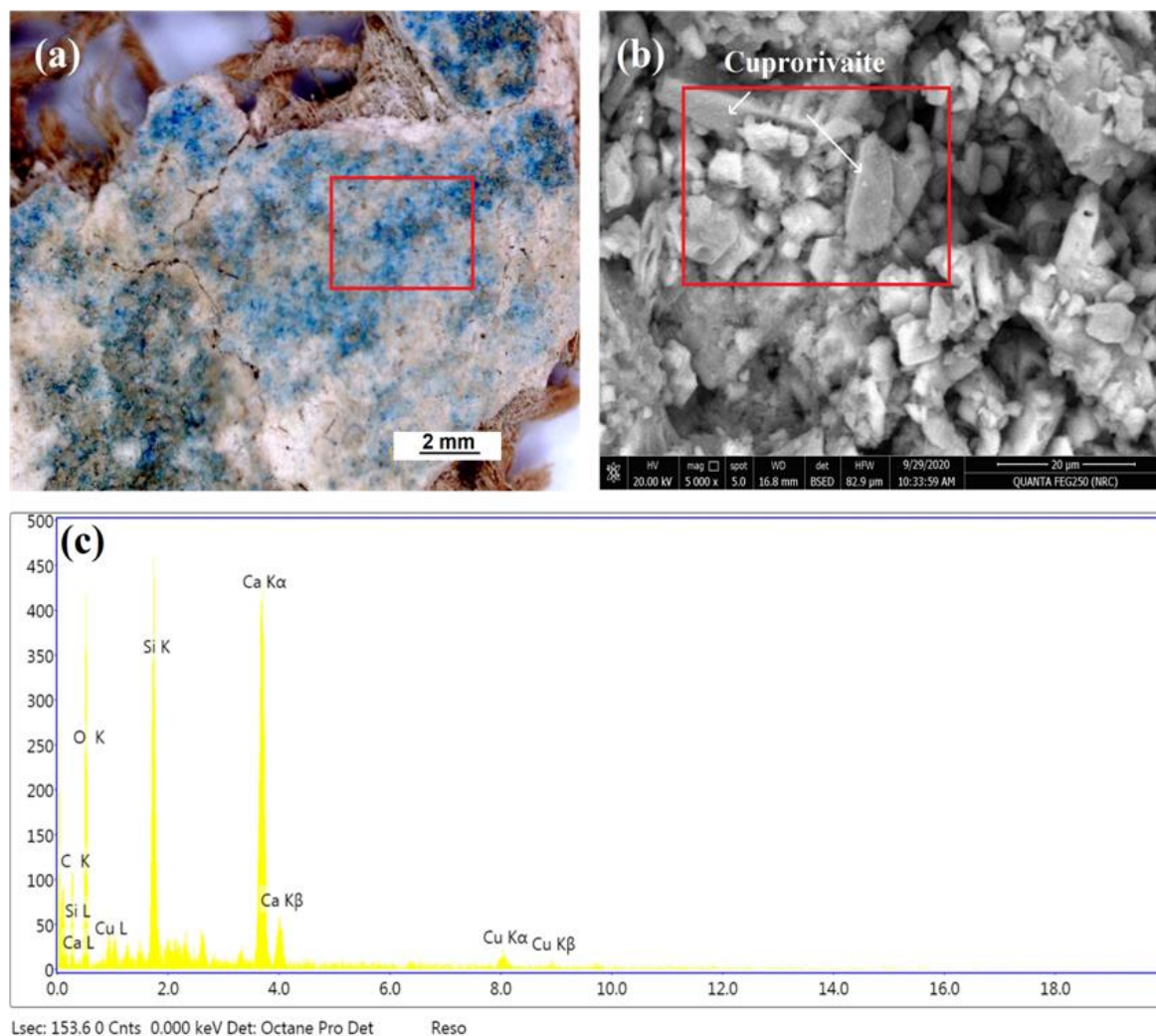


Figure 7. a) Microscopic examination of the blue paint layer, b) microstructure of crystals, by FE-SEM, c) EDX microanalysis of the sample.

3.3. Blue paint layer

The blue paint layer comprises a significant element in the cartonnage case, principally in the Upper and Middle areas. The microscopic image obtained on the blue paint layer (Fig. 7a) demonstrated bright blue particles scattered on the underlying *gesso* layer. In the image, the exposed fabric support is clearly observed. FE-SEM micrograph registered on the sample (Fig. 7b), evidenced varied matrix with a notable occurrence of siliceous aggregates. Also, large-size pigment particles are detectable in the image (with a length reaches 20 μm). Figure 7(c) shows the atomic concentrations of the occurring elements: calcium (Ca, 12.29%), silicon (Si, 7.55%), and copper (Cu, 1.47%), connected to the cuprorivaite mineral. Egyptian blue was a leading permanent artificial pigment in ancient Egypt (Jaksch et al., 1983; Mirti et al., 1995; Pradell et al., 2006; Marey Mahmoud, 2011). It is obviously stated that its desirable application is linked to the lack of alternative suitable blues and its high stability, resulting from the intense bonding of copper

ions in the silica phase in the pigment structure (Bianchetti et al., 2000; Pagés-Camagna and Colinart, 2003).

3.4. Green paint layer

The examination of green paint layer in the polychrome surface showed multi-morphological aspects. The FE-SEM micrograph (Fig. 8a, up), reported coarse and fine particles, with dark and light grey tonalities. Moreover, penetrating flax fibres from the support base were observed in the micrograph. Concentrations of a huge number of elements were as follows: sodium (1.02%), magnesium (1.51%), aluminium (1.27%), silicon (7.11%), sulphur (1.07%), calcium (9.32%), iron (0.96%), and copper (1.41%) (Fig. 8a, bottom). Actually, these elements refer to multi-components, mainly Egyptian blue (Si, Ca, Cu), ochre pigments (Si, Al, Mg, Fe), and calcite/calcium sulphate (Ca and S). Green pigments in ancient Egypt were derived mainly from two minerals: malachite ($\text{Cu}_2(\text{CO}_3)(\text{OH})_2$), used mainly for cosmetic purposes, and the artificial Egyptian green (appeared

since the 6th Dynasty, ca. 2323–2150 B.C.) (Lucas, 1962).

But, mixing pigments together to fabricate multiple hues was also involved. In our case, it seemed that the green colour was produced through mixing proportions of Egyptian blue and yellow ochre (goethite, $\alpha\text{FeO}\cdot\text{OH}$). More, it seems that amounts of red ochre (haematite, $\alpha\text{Fe}_2\text{O}_3$) and gypsum were added to create extra special hues. Indeed, the microscopic inception of the sample showed blue, yellow and red particles mixed together in the same surface.

3.5. Yellow paint layer

The microscopic examination of the yellow paint area showed pale and shiny particles. In the morphological analysis of the sample, bright crystals were clearly observed (Fig. 8b, up), which suggest the existence of heavy metals. EDX analysis of these crystals (Fig. 8b, bottom), presented pronounced atomic concentrations of arsenic (As, 6.1%) and sulphur (3.82%), which propose an arsenic-based pigment (e.g. Orpiment, As_2S_3). However, bulk analysis showed aluminium (1.52%), silicon (3.32%), sulphur (3.82%), calcium (7.88%), and iron (0.98%), which are assigned to minerals of iron oxide, clay minerals, calcite, and gypsum. The two later minerals are coming from the underlying *gesso* layer.

It seemed that the ancient artist used a blended layer of yellow ochre and orpiment, purposely, to

minimize the used amounts of the precious orpiment. As analyzed in many objects, orpiment was usually added to ensure a shiny appearance to the produced yellow paints. It was widely applied for decorating sarcophaguses during the 18th Dynasty (ca. 1549–1292 B.C.) (El Goresy, 1997). In addition, it was found together with goethite in the tomb of Amenhotep III (ruled from ca. 1386 to 1349 B.C.) and in his palace (currently known as Malkata site) at Luxor (Uda *et al.*, 1999; Uda *et al.*, 2004). Other examples were highlighted in the ruins of Hatshepsut's shrine in the Karnak temples and in El-Deir El Bahary temple.

3.6. Red paint layer

FE-SEM image of the red paint sample unveiled fine aggregates scattered within the matrix (Fig. 8c, up). A diverse penetration of linen fibres into the paint layer is clearly observed. The EDX measured a variety of elements (Fig. 8c, bottom), namely iron (Fe, 1.45%), aluminium (Al, 1.69%), and silicon (Si, 3.96%), which asserted the occurrence of iron oxide chromophore, together with clay minerals. Calcium (Ca, 10.6%) and sulphur (S, 1.65%) are associated with components of the *gesso* layer (calcite and gypsum). Uninterruptedly, red ochre pigments were used since the Predynastic Period, and later in polychrome objects and cartonnages (Ali *et al.*, 2016). Frequently, iron ores spread over large areas of the Egyptian land (Nakhla, 2011).

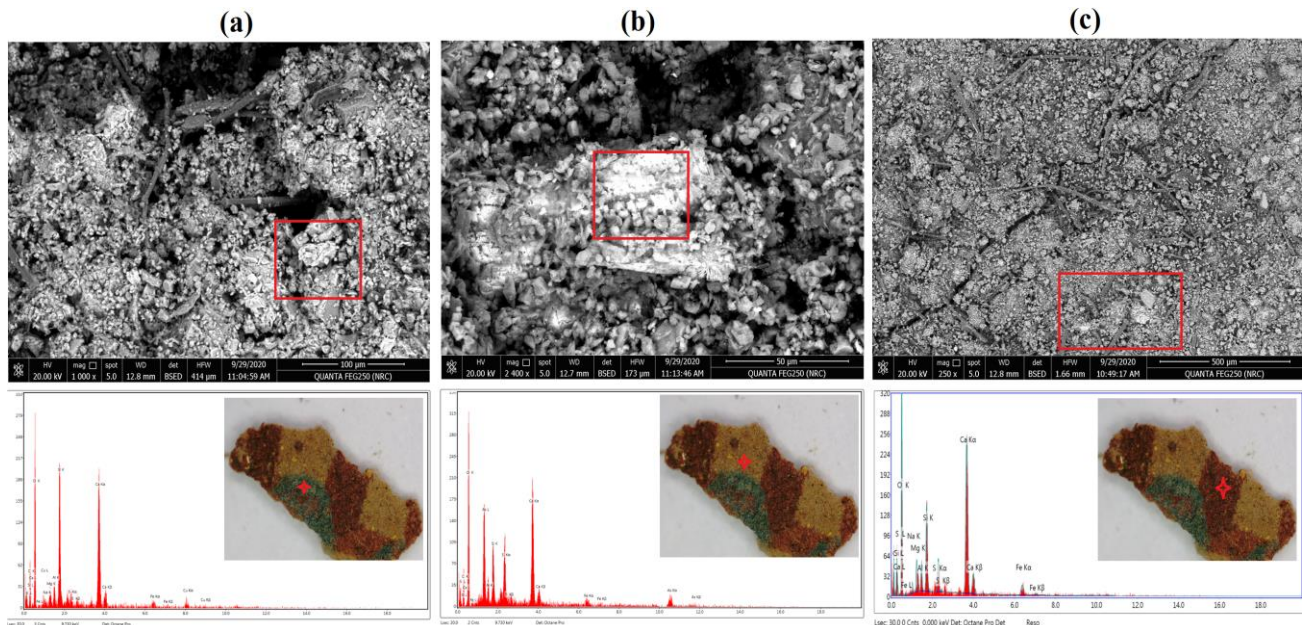


Figure 8. FE-SEM micrographs and EDX spectra collected on a polychrome surface of the cartonnage: a) green paint layer, b) yellow paint layer, and c) red paint layer.

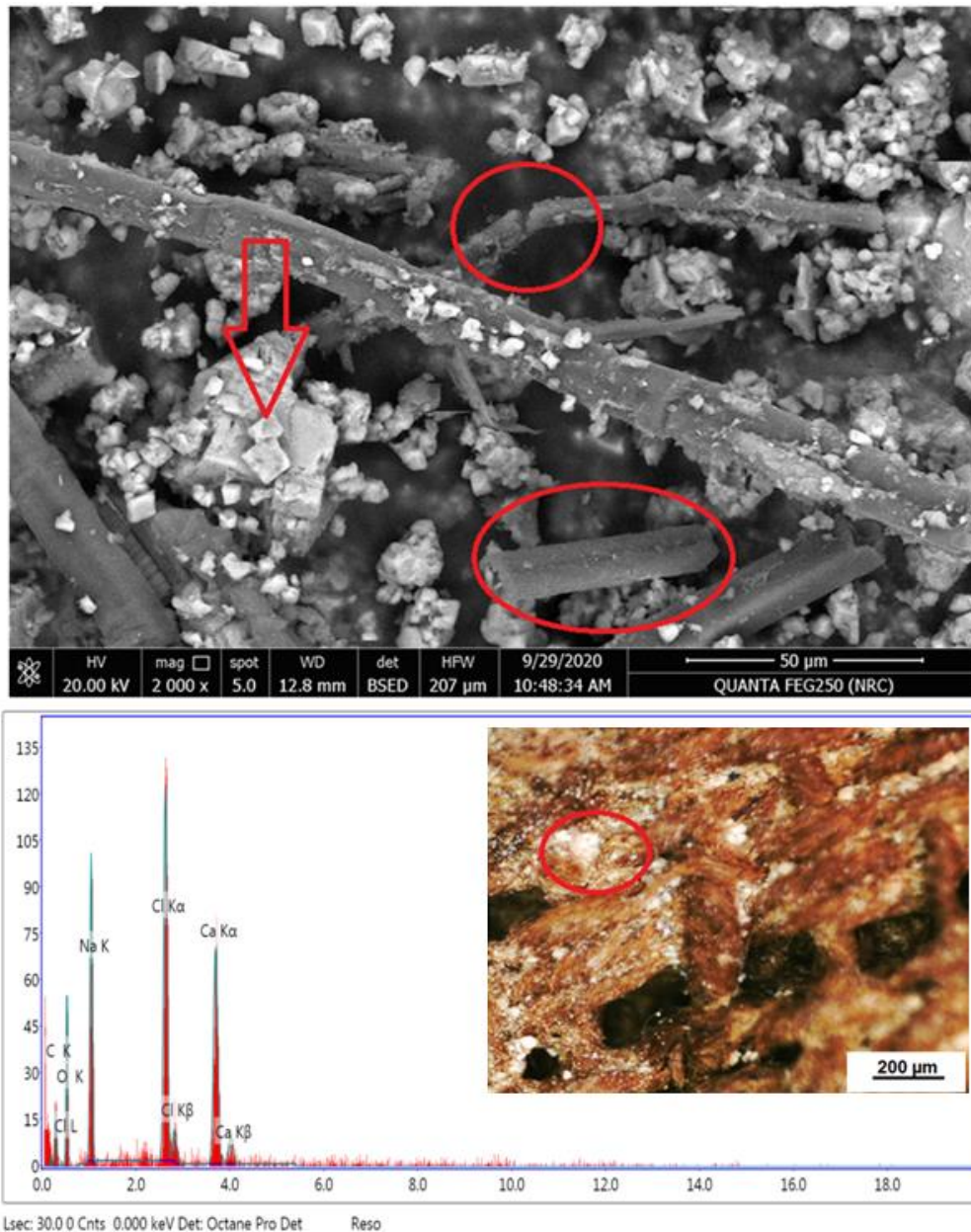


Figure 9. FE-SEM micrograph and EDX spectrum of salt deposits on the fabric structure.

3.7. Deterioration forms

As previously described, the cartonnage suffered from a severe damage. The microscopic examination on the linen structure showed the deposition of white particles. The morphological scan of the sample (Fig. 9, up), shows broken fibres. Also, cubic-shape crystals were reported. EDX analysis displayed extreme amounts of sodium (18.54%) and chlorine (10.81%), suggesting the crystallization of halite salt (Fig. 9, bottom). The efflorescence of this salt is highly accredited to the migration of saline solutions within the burial soil in the excavation site (Neate et al., 2011).

3.7.1. Microbiological analysis

Microorganisms are able to degrade easily organic and inorganic materials. The growth of microorganisms, e.g. fungi, causes a serious fracturing to the fibres through the penetration of hyphae into the macrostructure. In addition, destructive chemical degradation and microbial films are developed due to their enzymatic activity. To identify the affecting fungal species, multi-swabs were collected over several areas in the cartonnage. The examined areas covered: inner part from the head (SWAB (A)), the chest (SWAB (B)), the pelvis (right side) (SWAB (C)), the right-side end of the leg (SWAB (D)), and the feet area (SWAB (E)).

Table 2. *The isolated fungi over several areas in the studied cartonnage.*

Swab no.	Area	Species
A	Head	<i>Penicillium polonicum</i> ; <i>Aspergillus flavus</i> ; <i>Aspergillus niger</i> ; <i>Cladosporium cladosporioides</i>
B	Chest	<i>Penicillium polonicum</i> ; <i>Aspergillus flavus</i> ; <i>Cladosporium cladosporioides</i>
C	Pelvis	<i>Penicillium polonicum</i> ; <i>Aspergillus flavus</i> ; <i>Aspergillus niger</i>
D	Leg	<i>Cladosporium cladosporioides</i> ; <i>Aspergillus flavus</i> ; <i>Aspergillus niger</i>
E	Feet	<i>Cladosporium cladosporioides</i> ; <i>Aspergillus flavus</i> ; <i>Aspergillus niger</i>

Potato Dextrose Aga (PDA) rich medium was used for the cultivation of fungi. The isolated species are highlighted in Table 2. Fig. 10 shows swab areas on the cartonnage and the microscopic appearance of the isolated fungi. As shown in Fig. 10, the microscopic examination allowed the identification of the inducing fungi which manifested *Aspergillus flavus* with characteristic shaped cells spreading in random directions. *Aspergillus niger* appeared with long mitospores and structure full of spores. In case of *Cladosporium cladosporioides*, a tree-like branching can be observed. The microscopic image of *Penicillium polonicum* showed conidiophores representing simple and divided habit.

3.8. Restoration and treatment

As described above, the initial visual examination of the cartonnage showed dirt, spots, cracks, peeling, loss in the paint layers, detachments, and missing parts. Obvious white spots associated with holes and cavities distributed along the object were also documented. Ideally, conservation aims to slow down the rate of deterioration as possible. Based on assessing the present condition of the cartonnage, a restoration intervention was necessary. The restoration project started with a secure lifting of the cartonnage using a hydraulic lift to transport the cartonnage from the storage room to the laboratory with the aid of sponge sheets and Tyvek straps.

3.8.1. Disinfection

Disinfecting microorganisms is an essential step in the conservation process of organic and composite artefacts. Synthetic chemical biocides are widely used to prevent the microbiological growth, but they are environmentally dangerous for indoor application (Li *et al.*, 2013). For this, alternative natural eco-friendly anti-microbiological substances are highly recommended (Dos Santos *et al.*, 2018; Bahmani and Schmidt, 2018).

The use of plant extracts and essential oils has become an alternative global trend to disinfecting archaeological objects (Salem *et al.*, 2016; Veneranda *et al.*, 2018). Gomez De Saravia *et al.* (2008), have reported the efficiency of plant extracts to control the fungal growth on several archaeological objects. For

sterilizing purpose of the studied cartonnage, plant extracts that contain volatile oils were used. Natural extracts are low cost products that have the ability to minimize and prevent a wide range of microbiological agents (Fierascu *et al.*, 2014). In our case, extracts of Frankincense and Common Juniper in addition to clove essential oil were used in the disinfection process of the cartonnage. The extracts were diluted in Ethyl alcohol and applied by spraying technique.

3.8.1.1. Clove

Clove (*Syzygium aromaticum*) is a well-known evergreen tree that grows in tropical climates. Literature studies referred that the essential oil of clove is antibacterial agent. Actually, studies have shown that clove oil has an effective activity against some fungi species, e.g. *Aspergillus niger*, *Aspergillus clavatus*, *Penicillium sp.*, and *Fusarium sp.* (Borrego *et al.*, 2012; Hasheminejad *et al.*, 2019). The study of Veneranda *et al.* (2018) showed that clove oil, in low concentrations, succeeded to produce a remarkable inhibition to the growth of *Aspergillus niger* isolated from mural painting from Pompeii, Italy. Indeed, hydrophobicity of the components of clove oil plays an important role in their inhibiting measures (Burt, 2004).

3.8.1.2. Frankincense

Frankincense, also known as Olibanum, is obtained from a kind of trees called *Boswellia*, profusely cultivated in tropical countries. The extracted amount of the essential oil of this tree (*Boswellia carterii*) reaches 5–9% (Al Amri *et al.*, 2019), and it is based on several ingredients, e.g. *a-pinene*, *linalool*, and *1-octanol*.

3.8.1.3. Common Juniper

Common Juniper is a small evergreen tree spreads in the dry regions (Orav *et al.*, 2010). This plant has been recognized with its antimicrobial behaviour. The juniper berries extract (*Juniperus communis* L.) consists a wide variety of compounds, mainly *a-pinene*, β -*pinene*, *sabinene*, and *D-limonene*.

3.8.1.4. Preparation of extracts (Frankincense and juniper)

Each kind of these plants was first washed by distilled water, air dried and then pulverized into fine

powdered substances by a grinding machine, which creates a good surface contact between the particles and the used solvents. A total of 10 g of each plant powder was transferred into two 250 ml conical flasks separately, after which 100 ml of ethanol were poured into flask. The conical flasks were closed with foil paper and placed in a shaker in dark condition for one day at 25°C. The ethanol extracts were then filtered by

passing the extracts over four layers of cheese-cloth and then through Whatman No. 1 filter paper. After that the extracts were evaporated at 40°C using a rotary evaporator. These residual extracts were dissolved in ethanolic crude extracts and stored at 4°C in the refrigerator for further phytochemical studies on the isolated fungi (Othman, Saada, and Matsuda 2019).

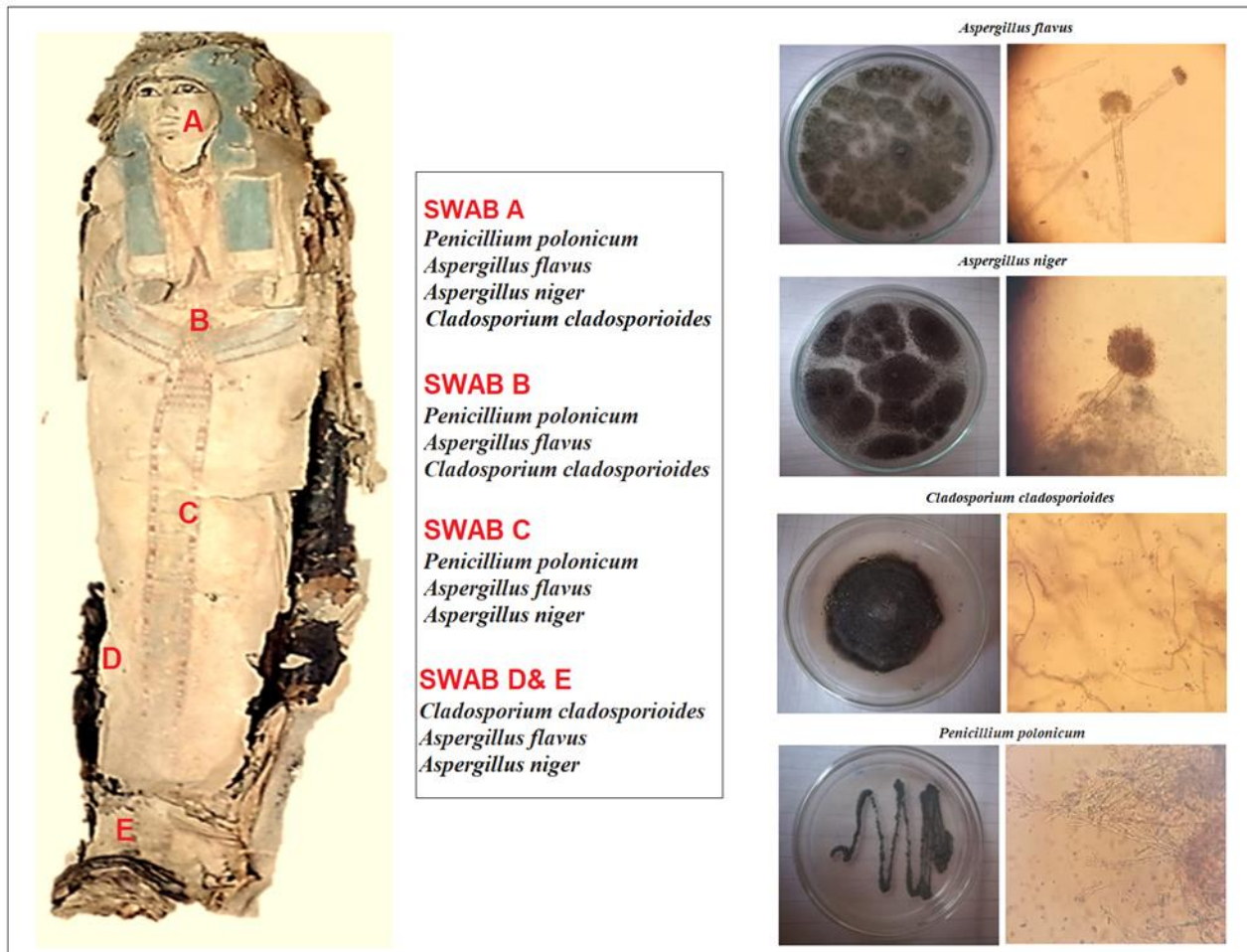


Figure 10. Identification of the fungal species isolated from infected areas on the cartonnage.

3.8.1.5. Clove essential oil

Clove oil was obtained from Natural Oils Department, National Research Center, Dokki, Egypt. The tested clove essential oil was obtained from the aerial parts of plants by hydro distillation using a Clevenger-type apparatus for 3 hours. The extracted essential oil was kept in dark at 4°C until further investigation.

3.8.1.6. Antifungal activity

The antifungal activity of clove, frankincense, and Juniper extracts were evaluated using the Mycelial growth inhibition (MGI). A melted-dilution of the tested materials in the Potato dextrose agar, at a concentration (100 μml^{-1}), was applied. After shaking,

the mixture was poured and cooled in Petri dishes until reaching the complete solidification. The plates were then inoculated at the center with fungal discs (each with 4 mm in diameter) from 7 day-old cultures. The tested triplicate dishes were incubated at 27°C for 7 days. The inhibitory percentage for each extract on the fungal growth was evaluated using the following equation (Stupar et al., 2014): % of inhibition = $(dC - dT) \times 100 / dC$.

In the formula, 'dC' represents the fungal colony's average diameter measured in a negative control, while 'dT' refers to the fungal colony's average diameter of the treatment group.

In Fig. 11 and Table 3, the antifungal efficiency of some extracts (of Frankincense, Juniper extracts in addition to clove essential oil) are given. Results showed

that the lowest inhibiting act was induced by the extract of frankincense on all the tested species. While its best effect was reported against *Aspergillus flavus*, while *Penicillium polonicum* was the most resistant. In the second level is the extract of common Juniper, and

its best effect was reported against *Aspergillus niger*. Positively, the four fungal species were sensitive to the essential oil of clove, and the highest percentage of the growth inhibition (%) was achieved, ensuring a super growth inhibition.

Table 3. Antifungal activity of extracts against the isolated fungi.

Fungal species	Growth inhibition (%) of the tested extracts (100 μml^{-1})		
	Clove	Frankincense	Common Juniper
<i>Aspergillus flavus</i>	100	50	62
<i>Aspergillus niger</i>	100	48	70
<i>Cladosporium cladosporioides</i>	100	40	58
<i>Penicillium polonicum</i>	100	8	48

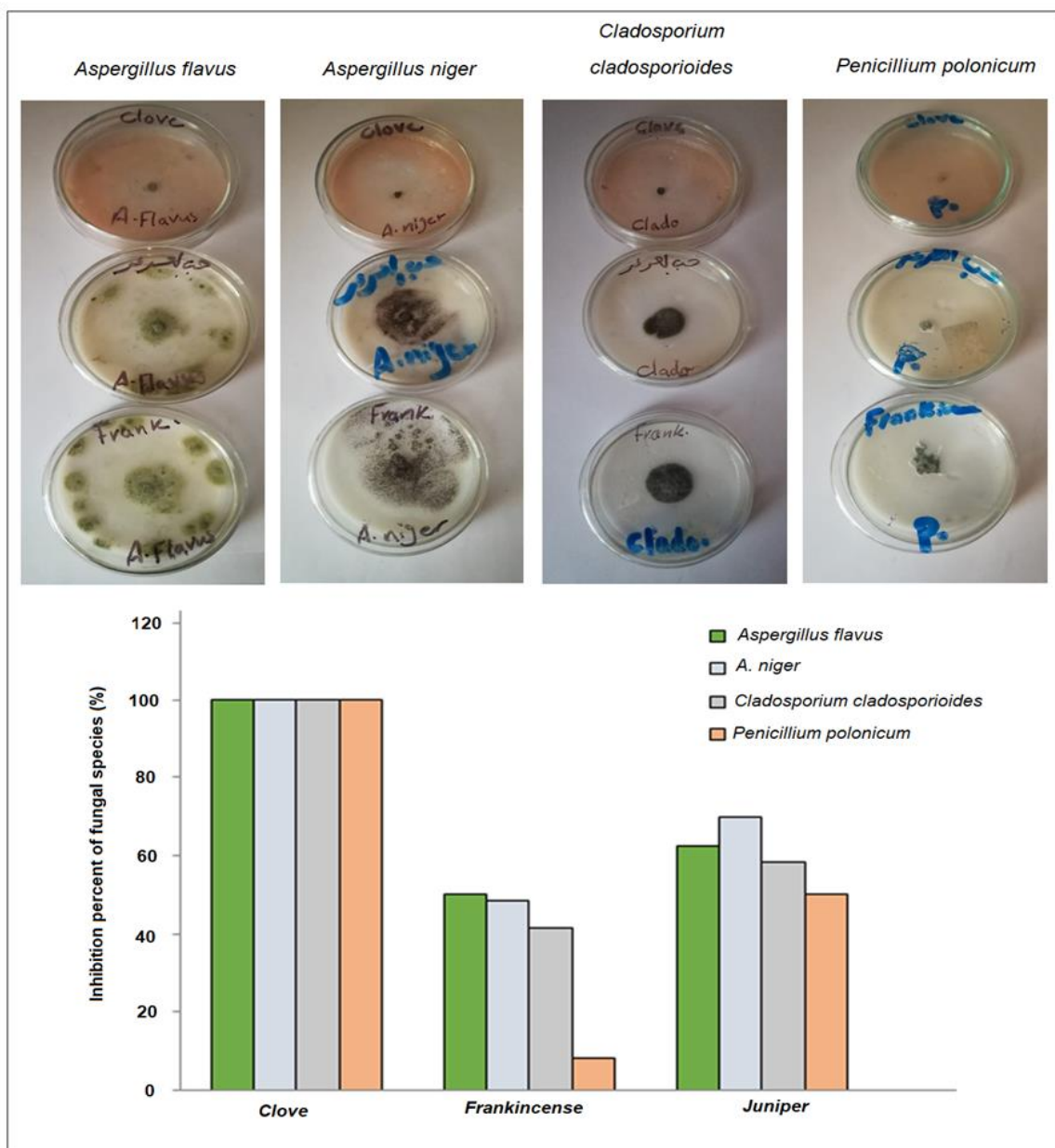


Figure 11. Antifungal activity represented by inhibition percent (%) of clove oil, frankincense, juniper extracts on the isolated fungal species (*Aspergillus flavus*, *A. niger*, *Cladosporium cladosporioides*, and *Penicillium polonicum*).

3.8.2. Surface cleaning

The cleaning process involves the removal of accumulations and deterioration products from the surface of artifacts, using different tools and techniques. Several dust and soiling layers resulted from the burial soil, in the excavation site, were reported. Cleaning of these materials was done using soft and rough sable brushes keeping one direction, a 45 degree angle, to prevent any damage to the fragile surface (Fig. 12a, left side). In some areas, a hand air pump was used to push the dirt away. To facilitate the removal of the soiling materials, cotton swabs dampened with ethanol were rolled gently over the surface.

3.8.3. Stabilizing paint flakes and detached layers

Several detachments in the *gesso* layer and separated paint flakes were documented. Reasonably, the degradation of the organic binder used for the pigment grains contributes seriously in the flaking process. But, due to the effect of microclimatic factors and the difference in the response behaviour of each single material used in the composite objects, as in case of cartonnage, results in form of detachments of the *gesso* layer. For this, securing and re-adhere these layers was a necessary procedure to prevent any further damage that may lead to the complete loss. The detached pieces and flakes were first dampened and supported by strips of Japanese paper. Then, the paint flakes were injected using Klucel® G (Hydroxypropyl cellulose) (10% in ethanol, weight/volume) and re-adhered to the underlying base (Fig. 12a, right side). While high concentration of Klucel® G (15%) was used for the detached *gesso* layers. A gentle pressing over the detached layers, using different sizes of metallic spatulas, was applied to ensure a sufficient adhesion to the substrate.

3.8.4. Consolidation

Consolidation is usually applied to create a solidification for the damaged material. The consolidating action depends mainly on the penetration depth of the consolidant into the pores and its distribution within the substrate. The painted surface of the studied cartonnage showed a severe fragility. The fragile areas were consolidated, repeatedly, by soft brushes, using Klucel® G in ethyl alcohol (3%, w/v). While, some cracks in the surface were consolidated by injecting the Klucel® G solution through a syringe, to maintain their stability. Practically, Klucel G was used to treat several composite objects in several museums, e.g. Penn Museum and the British museum.

3.8.5. Reinforcing

As stated before, serious physical damage with broken pieces were reported mainly in both sides of the cartonnage, right and left. First, cleaning the area from the carbonized linen fibres was performed. Afterwards and for supporting the sides, a wall of sterilized linen pillows filled with medical cotton was tapped to allow the resin to expand into the voids and gaps of the edges (Fig. 12b). An alternative treated linen support was fixed to compensate the missing parts of the cartonnage. The fallen pieces were reattached to this new fabric using an adhesive while sewing was used for other pieces. To strengthen the head area, a high concentration of an acrylic resin (Paraloid B44, 10% in acetone, w/v) was used. While Japanese strips, saturated in Paraloid B44, were applied to stabilize the area. Then, pieces of well-dyed, treated and sterilized linen textile was necessary to support the fabric base in both sides of the cartonnage. For completing the missing parts, a paste made of microballoons and Klucel G was employed.

3.8.6. Filling the missing areas

To fill the missing parts in the body of the cartonnage, a collection of tissue papers with microballoons filler (Hollow Glass Microsphere) and Klucel G (7%, in ethyl alcohol) was appropriate to fill the cracks and to strengthen this part. A supporting board made from sterilized treated linen was bedded under the cartonnage, then a transparent tulle was fixed by stitching it on some parts of the cartonnage to ensure a long term stability.

3.8.7. Supporting the cartonnage

A new support of treated linen that respects the cartonnage shape, with extra size of 1 cm from all sides, was applied. Then, the cartonnage was transferred to this new support to build up an additional reinforcement to the lower part. A clothing for the head and feet areas with nylon net was applied and fixed to the linen support using simple sewing. Further, almost equal sizes of nylon strips were added to backing the cartonnage. To facilitate lifting the cartonnage, a separate wooden support of treated wood was covered with medical cotton. After that, the wood was cushioned by wrapping gauze and dammur textile and cloth strips to the support to safely accommodate the cartonnage.

Figure 12 summarizes the main restoration steps and the final condition of the cartonnage after completing the intervention.



Figure 12. A group of restoration procedures performed on the cartonnage: a) cleaning and fixing paint flakes, b) reinforcing edges of the cartonnage, c) details of the cartonnage show the condition before and after restoration (left side) and the head/chest areas after final restoration (right side), and d) general and side view of the cartonnage after restoration.

4. CONCLUSIONS

The present interpretation allowed the fulfilment of beneficial data related to characterizing materials used in a decorated cartonnage case, from EL-Lahun excavations, Middle Egypt. Therewithal, to diagnose its state of preservation and to apply the required restoration procedures. Considerations on the artistic style of the cartonnage helped to determine its date, which ascertains that it belongs to the Third Intermediate Period (Dynasties 22–25, ca. 945–664 B.C). A number of analytical methods (mainly OM, FE-SEM/EDX and FT-IR) were used to characterize the materials of cartonnage and the main deterioration forms. The analyses proved the fabric base as linen fibres, while the *gesso* layer was prepared by mixing calcium carbonate with gum Arabic. Amounts of gypsum were also found, probably occurred as degradation of the lime matrix. The pigment palette was analyzed as Egyptian blue, red ochre, yellow ochre and orpiment. A blended layer consists of orpiment and yellow ochre was identified. A combination

of Egyptian blue and yellow ochre was applied for the green hue. The cartonnage showed several deterioration forms, including cracks, detachments, loss, etc. Fungal species of *Aspergillus flavus*, *Aspergillus niger*, *Cladosporium cladosporioides*, and *Penicillium spp.* were isolated from several areas on the cartonnage. The results showed that the plant extracts can provide eco-friendly and safe disinfection to composite artefacts. In the same way, the clove essential oil showed a super action against the fungal activity, compared to other tested extracts. A complete restoration succeeded to eliminate the damage affected the cartonnage. Mechanical and chemical cleaning succeeded to remove dust and soiling accumulations adhered to the painted surface. Stabilizing the paint flakes, *gesso* layers and consolidation of the friable areas were achieved by different concentrations of Klucel G, diluted in ethyl alcohol. Restoration steps included securing the cartonnage sides using a high concentration of Paraloid B44, and filling the missing parts using tissue papers with microballoons filler (HGM),

and Klucel G. The cartonnage case was placed onto a new linen base to provide an additional support to the lower parts. Obviously, the results helped to preserve the cartonnage and to correct previous records concerning the cartonnage in terms of its chronological date and the material used in the fabric substrate. In

addition, the results revealed the importance of applying eco-friendly products for restoration purposes. Accordingly, integrated scientific approaches for similar objects in the museum-storerooms of Egypt are a high priority.

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