

## Gilding Technique and Conservation of a Gilded Greco-Roman Cartonnage in Hurghada Museum

Dr. Gehan Adel Mahmoud

Restoration Department, Faculty of Archaeology, Luxor University.

[gehanadel@farch.luxor.edu.eg](mailto:gehanadel@farch.luxor.edu.eg)

### Abstract

Hurghada museum includes a gilded cartonnage from Greco-roman period from Hawara, Fayoum governorate. This gilded cartonnage suffers from many deterioration phenomena, for example presence of cracks, micro cracks, completing of missing parts using wax as a previous conservation, and missing parts in the gilding layer. Examinations and Analysis were carried out by optical microscopy (LOM), Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray detector (EDX), X-ray diffraction (XRD), and Fourier transform infrared spectrometers (FTIR). EDX and XRD analysis show that the gilding layer is a gold leaf which includes gold as a principal component. It showed that the thickness of gold leaf is about 0.05 to 0.10 mm. The XRD analysis of the gilding layer sample showed that, it consists of gold, calcite and gypsum minerals. XRD analysis of the red - pink color revealed that it is red-lead  $Pb_3O_4$  (lead tetroxide), in addition of calcite and gypsum minerals. The analysis result of the brown color showed that it consists of hematite besides calcite and gypsum minerals. All samples included a ground layer of the cartonnage so that, calcite and gypsum minerals related to the Preparatory layer. FTIR results show that animal glue has been used as a pigment medium and a cohesion material for pigments and the gilding layer. The treatment and conservation plan of the gilded cartonnage includes mechanical and chemical cleaning, injection of cracks and micro cracks; completion the missing parts in colored and gilded parts, consolidating the fragile parts using paraloid-B72 (3%). Plexiglas has been used in display process of the gilded cartonnage in Hurghada museum.

### Key Words:

Gilding, Cartonnage, Greco-Roman, Conservation, Hurghada Museum.

### المخلص

يشتمل متحف الغردقة على كارتوناج مذهب يعود إلى الفترة اليونانية الرومانية من منطقة هواره بمحافظة الفيوم، يعاني هذا الكرتوناج المذهب من العديد من مظاهر التلف، على سبيل المثال وجود تشققات، تشققات دقيقة، إستكمال الأجزاء المفقودة باستخدام الشمع كترميم سابق، والأجزاء المفقودة من طبقة التذهيب. تم إجراء الفحوص والتحليلات بواسطة الفحص المجهرى البصري (LOM)، الفحص المجهرى الإلكتروني (SEM) المجهز بوحدة الأشعة السينية المشتتة للطاقة (EDX)، حيود الأشعة السينية (XRD)، مطياف الأشعة تحت الحمراء (FTIR). أتضح من التحليل بواسطة وحدة (EDX) وحيود الأشعة السينية (XRD)، أن طبقة التذهيب عبارة عن أوراق تذهيب، تتضمن معدن الذهب كمكون رئيسي. أتضح أن سمك أوراق التذهيب المستخدمة من 0.05 إلى 0.1 مم. أظهر التحليل بحيود الأشعة السينية (XRD) لعينة طبقة التذهيب أنها تتكون من معدن الذهب إلى جانب وجود معدني الكالسيوم والجبس، كما تبين من التحليل للون الأحمر الوردي أنه يتكون من الرصاص الأحمر  $Pb_3O_4$  (رباعي أكسيد الرصاص) بالإضافة إلى الكالسيوم والجبس، أما بالنسبة للون البني أتضح أنه يتكون من الهيماتيت إلى جانب معادن الكالسيوم والجبس. اشتملت جميع العينات على معدني الكالسيوم والجبس وهما

المكونان لطبقة التحضير أو الأرضية أسفل طبقة التذهيب. تبين من خلال التحليل بواسطة الأشعة تحت الحمراء FTIR أن الغراء الحيواني قد تم استخدامه كوسيط لوني ومادة لاصقة للألوان وطبقة التذهيب. تضمنت خطة المعالجة والصيانة، التنظيف الميكانيكي والكيميائي، وحقن الشقوق والشقوق الدقيقة؛ واستكمال الأجزاء المفقودة في الأجزاء الملونة والمذهبة، وتقوية الأجزاء الهشة باستخدام البارالويد - ب 72 بتركيز 3%. وتم استخدام البلكسي جلاس في عملية العرض المتحفي للكرتوناج المذهب بمتحف الغردقة.

### الكلمات المفتاحية:

تذهيب، كرتوناج، يوناني - روماني، صيانة، متحف الغردقة.

## 1. Introduction

Hurghada is a city located in the Arab Republic of Egypt. It is one of the most famous tourist centers in Egypt. It is located about 400 km from Cairo city. The nature of the Hurghada Museum is different from the other museums in Egypt because it is a partnership between Egyptian government and a private sector. The area of this museum is about 10,000 square meters. (Ministry of Tourism and Antiquities, 2020). This museum includes a funerary cartonnage dating back to the Greco-Roman era, which is gilded and colored. Its dimensions are 41 x 51 cm, and dates back to the first century related to Julio Claudian period, (gettyimages, 25 years), fig. (1). The cartonnage in this period is characterized by the presence of a thick plaster layers, which are reinforced by a coarse fibrous material. The cartonnage in Hurghada museum is gilded, there is a red wreath in the right hand, and there is a purple belt in the reward, (Picton, J., 2007). The eyes of this cartonnage were made of calcite and glass, and the frames of these tow eyes and the eyelashes is made of bronze, (Pancaldo, S. 2010). Fig. (2). The museums and stores of Egypt contains many cartonnages (mummy masks), and these masks are subject to deterioration. (Sabino, R., 2019). Cartonnage is made of linen. The linen layers are fixed with an adhesive, then the plaster and colored layers were added. The layers leave to dry and the surface of the plaster layer is smoothed, and then the surface is colored and gilded. (Ali, M. et al, 2016). Cartonnage represents the face of the deceased and sometimes it represents parts of the deceased such as the foot or parts of the whole body, (Alawneh, F. 2019). Cartonnage was first applied in the Middle Kingdom (2025-1700 BC), (Wright, 1983). Cartonnage gilding continued into the era of the New Kingdom (1570-1070 BC), (Nicholson, E.D., 1979). The method of gilding depends on fixing gold foil or gold leaves with thicknesses ranging from (1 and 10  $\mu\text{m}$ ), (Adams, 1996). There are different ways to fix gilding leaves, such as mechanical methods, or the use of an adhesive from organic material, (Oddy, W.A., 1993). The method of applying gilding leaves to the surface of the gilded cartonnage is done by cleaning the surface first, then a layer of adhesive is applied to fill the voids. (Oddy, W.A., 1981). Leave the layer to dry. Layers of calcium carbonate are applied with the adhesive, and then the gilding layer was applied, (Felder, E., C, 2010). The layers were left to dry, (Darque-Ceretti, E., 2011). There are many deterioration phenomena in the gilded cartonnage such as soil deposits, Cracks of deep sizes and different directions, Gaps, missing parts from the gilding layers, blur the gilding layer, in addition some parts are weak and need strengthening, fig. (3). The aim of the present paper is to identify the layer structure and techniques, characterize painting and gilding materials used to decorate the cartonnage by using light optical microscopy (LOM), Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray

detector (EDX), X-ray diffraction analysis (XRD), and Fourier Transform Infrared Spectroscopes (FTIR). On the other hand, to carry out the conservation processes of the gilded cartonnage and to display it in the Hurghada museum.



Fig. (1) Show the gilded cartonnage in Hurghada museum dates back to the first century related to Julio Claudian emperor. (After gettyimages, 25 years)



Fig. (2) show the gilded Greco-roman cartonnage in Hurghada museum before conservation.



Fig. (3) show the Greco-roman gilded cartonnage and completing parts with wax as a previous restoration in addition to presence of cracks and micro cracks.

## 2. Materials and Methods

Examinations and analysis have been performed to diagnose the deterioration phenomena and to characterize gilded layer, pigments, ground layer, and binding media of the gilded cartonnage and pigments. Light Optical microscopy (LOM), Scanning electron microscope equipped with energy dispersive x-ray analysis (EDX), Analysis by X-Ray Fluorescence (XRF) of the gilding layer, X-ray diffraction analysis and Fourier transform infrared spectroscopy were used for examination and analysis of the gilded cartonnage.

### 2.1. Sampling

Samples were taken from parts that were separated from the cartonnage to identify the constituents and the deterioration phenomena. These samples were representative of red-pink, brown pigments and gilding layer as well as the preparation layers.

### 2.2. Light Optical Microscopy (LOM)

Samples were observed by a Wild M8 stereomicroscope, an Olympus BX51 optical microscope. The surface of the gilded cartonnage and three cross sections of gilded layer, red-pink and brown pigments were examined.

### 2.3. Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray analysis (EDX)

Samples were investigated by Philips (XL30) microscopy, equipped with EDX micro analytical system to obtain the total element content qualitatively and quantitatively by EDX unit in the samples. The beam voltage for the quantitative determination of elements was set to 25 kV, in order to obtain better excitation of the low-energy and low-concentration compounds. Samples from the gilding layer were examined.

### 2.4. Analysis by X-Ray Fluorescence (XRF) of the gilding layer

Samples of the cartonnage gilding layer were analyzed using NITON XLt 800 Series Alloy Analyzer, Version 4.2, Thermo Electron Corporation Portable Elemental Analysis, and Produced in the United States of America (USA).

### 2.5. X-ray diffraction analysis

X-ray diffraction analysis carried out with Phillips X-ray diffraction equipment model pw/1840 with Ni filter, Cu radiation  $1.54056 \text{ \AA}$  at 40 KV, 25mA, 0.05 /sec. Measurements were carried out on powders of the samples, in the range  $0^\circ < 2\theta < 60^\circ$  with a step of  $0.02^\circ$ . This method was used to analyze samples of the gilding layer and the pigments layer.

### 2.6. Fourier Transform Infrared Spectroscopy (FTIR)

Samples of pigments and gilding layer were analyzed by FTIR method using a Bruker, Equinox 55/S spectrometer. Transmittance spectra were collected in the range  $400\text{-}4000 \text{ cm}^{-1}$  with a spectral resolution of  $4 \text{ cm}^{-1}$  and acquiring 400 scans.

## 3. Results

### 3.1. Light Optical microscopy (LOM)

LOM examination showed that there was adherent dust and missing parts in pigments, Deep cracks in the gilding layer. Scaling and loss in the gilding layer. From the polished cross sections, it showed that homogeneity in the pigment layer in some parts of the cartonnage and its gilding layer. It showed that the thickness of gold leaf is about 0.05 to 0.10 mm; the thickness

of the red color is between 0.2 to 0.3 mm and the thickness of the brown color is between 0.2 to 0.4 mm. Fig (4).

### 3.2. Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray analysis (EDX)

SEM photomicrographs of the gilding layer showed the presence of cracks in the gilding layer, losing some parts of the gilding layer, the ground layer is appeared under the gilding layer through cracks and missing parts, Fine dirt, grains of sand and dust attached to the surface. There are ripples in the gilding leaves due to the method of fixing the gilding leaves on the surface of the cartonnage, fig. (5).

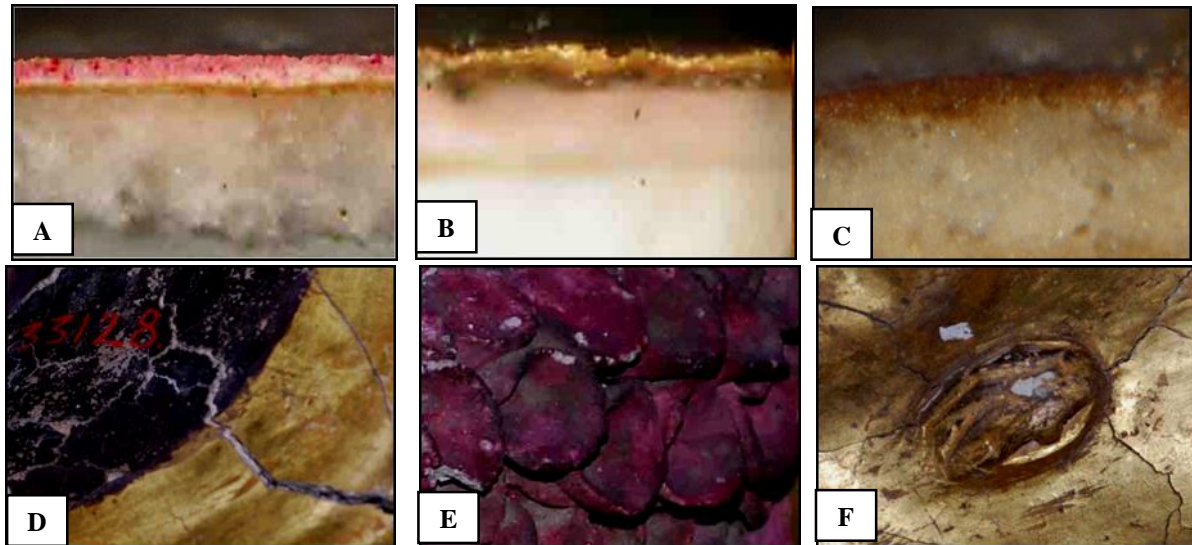


Fig. (4), A, B, C show cross sections of gilding and pigments layers and D, E, F show deterioration phenomena of gilding and pigments for example missing parts in addition to presence of cracks.

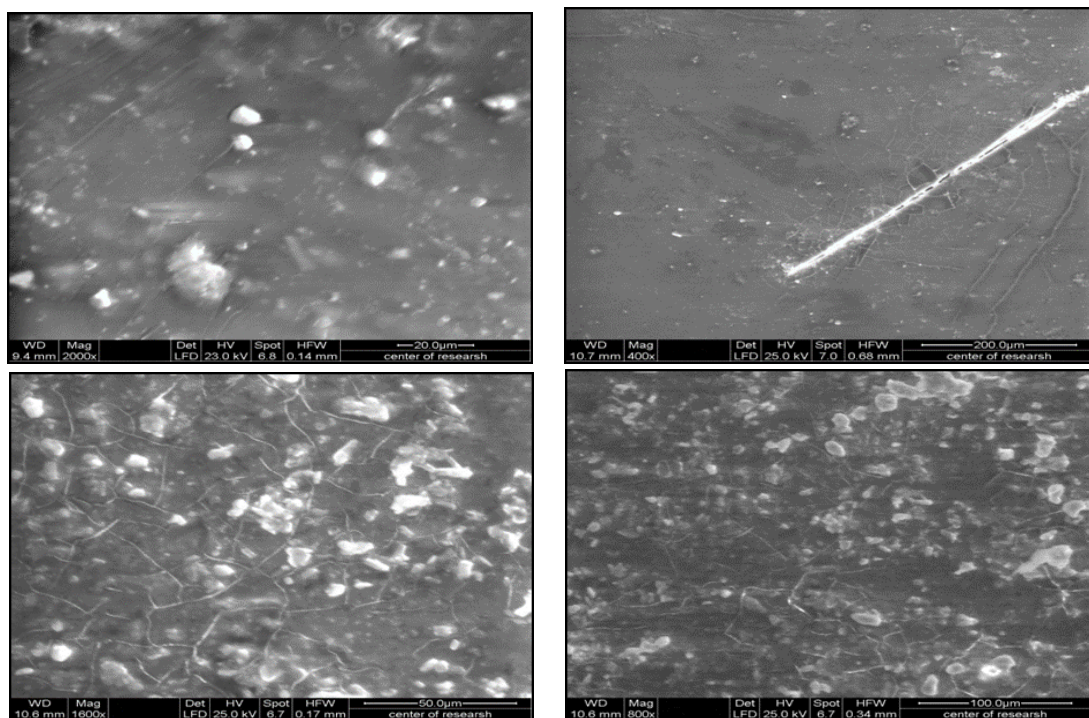


Fig. (5) SEM micrograph for the gilding layer of cartonnage show deep and micro cracks in addition to the ground layer which appear through the gilding layer and presence of missing parts from the gilding layer.

### 3.3. Analysis by (EDX) unit of the gilding layer

The gilded layer was analyzed by the (EDX) unit attached to the scanning electron microscope (SEM). It showed that, the presence of gold metal by 2.84 % in a sample and by 7.78 % in the other sample. Elements were appeared related to the ground layer and bole material ( A clay mineral) under the gilding layer including C, O, Al, Si, Fe and Ca elements. Ratios of other elements have been found and the results are complete shown in figs. (6), (7).

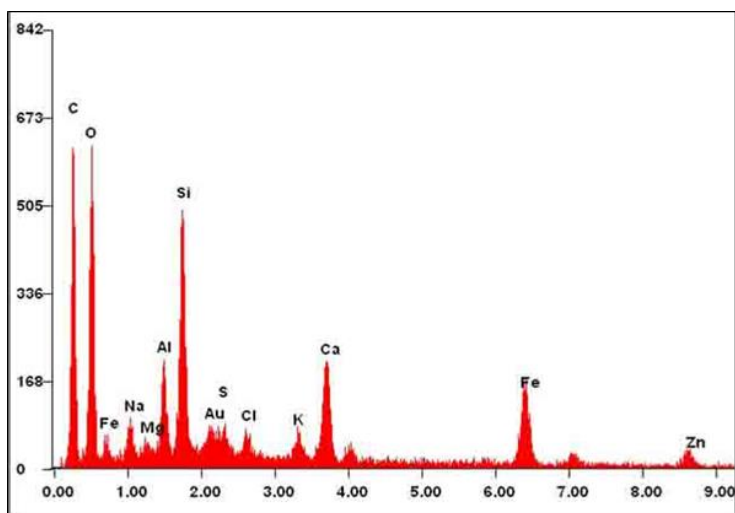


Fig. (6) EDX pattern of a cartonnage gilding layer sample.

| Element | Wt %  | At %  |
|---------|-------|-------|
| C K     | 47.02 | 62.71 |
| O K     | 28.07 | 27.52 |
| NaK     | 01.55 | 01.06 |
| MgK     | 00.52 | 00.33 |
| AlK     | 02.05 | 01.19 |
| SiK     | 05.22 | 02.91 |
| AuM     | 02.84 | 00.15 |
| S K     | 00.60 | 00.29 |
| ClK     | 00.55 | 00.25 |
| K K     | 00.74 | 00.30 |
| CaK     | 03.04 | 01.19 |
| FeK     | 05.73 | 01.61 |
| ZnK     | 02.08 | 00.50 |

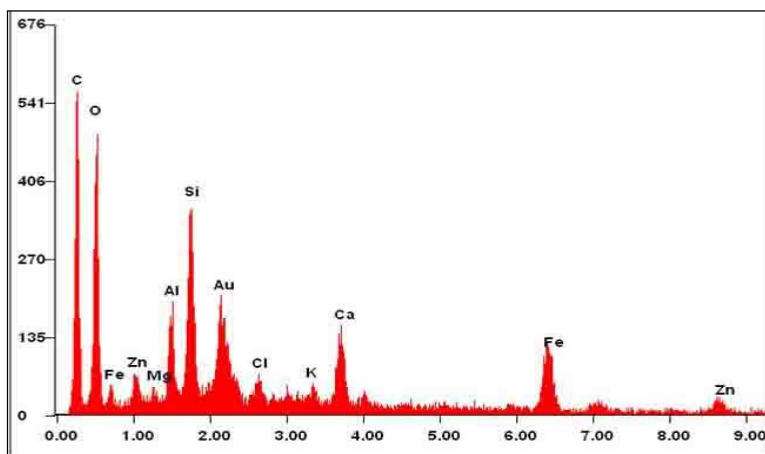


Fig. (7) EDX pattern of another cartonnage gilding layer sample.

| Element | Wt %  | At %  |
|---------|-------|-------|
| C K     | 48.21 | 65.04 |
| O K     | 26.47 | 26.81 |
| MgK     | 00.40 | 00.27 |
| AlK     | 02.08 | 01.25 |
| SiK     | 04.35 | 02.51 |
| AuM     | 07.78 | 00.64 |
| ClK     | 00.56 | 00.26 |
| K K     | 00.52 | 00.22 |
| CaK     | 02.55 | 01.03 |
| FeK     | 05.30 | 01.54 |
| ZnK     | 01.76 | 00.44 |

### 3.4. Analysis by X-Ray Fluorescence (XRF) of the gilding layer

XRF data shows that, the gilding layer of the gilded cartonnage consists mainly of gold metal. The analysis was carried out for the gilding samples as a percentage and ppm. concentration. Gold metal was appeared in three samples by 2.61 %, 4.67 %, 4.41 % as a percentage in addition

to a small percentage of Al, Fe, Pb, Zn. On the other hand, gold metal was appeared in tow samples by 159. k and 153. 4k as ppm. concentration. Al and Fe elements related to a layer of bole (A greasy clay with iron oxide impurities that produce a red, yellow and/or brown color and with about 24% water), (Deer. H. 1997) was applied. The structure of this clay, plates, formed a smooth cushion on the gypsum layer, figs. (8), (9), (10), (11), (12).

### 3.5. X-Ray Diffraction analysis (XRD)

The XRD analysis of the gilding layer sample showed that, it consists of gold, calcite and gypsum minerals, fig. (13). XRD analysis of the red - pink color revealed that it is red-lead  $Pb_3O_4$ .(lead tetroxide), in addition of calcite and gypsum minerals, fig. (14). The analysis result of the brown color showed that it consists of hematite besides calcite and gypsum minerals, fig. (15). All samples included a ground layer of the cartonnage so that, calcite and gypsum minerals related to the ground layer.

### 3.6. Fourier Transform Infrared Spectroscopy (FTIR)

The results of the Fourier transform infrared spectroscopy (FTIR) of the gilding layer evidenced the C=O bond stretching and NH bond bending bands found between 700 and 1500  $cm^{-1}$ , both of them characteristic of the amide groups of proteins. This finding is confirmed by the presence, between 1500 and 1700  $cm^{-1}$  of the  $CH_2$  bond of the methylene group as well as by the  $-CH_3$  methyl group band at about 2900  $cm^{-1}$ , all of them suggesting the presence of animal glue. It was used as an adhesive material to fix the gold leaves on the surface of cartonnage, fig. (16). A sample of the red-pink pigment showed that, the presence of a band at 1540  $cm^{-1}$  associated with the deformation vibration of the N-H link in the protein. The bands that appear in the IR spectrum can be attributed to animal glue, fig. (17).

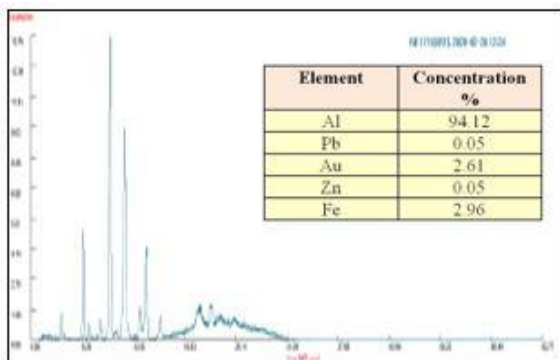


Fig. (8) XRF pattern of cartonnage gilding layer as a percentage concentration.

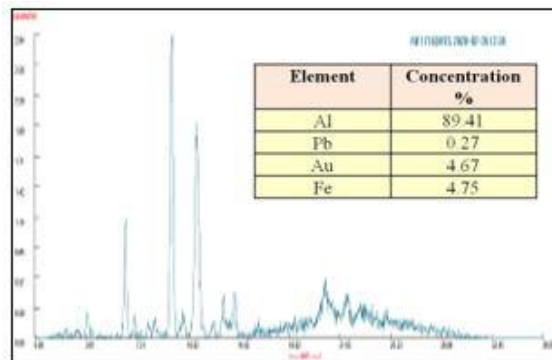


Fig. (9) XRF pattern of cartonnage gilding layer as a percentage concentration.

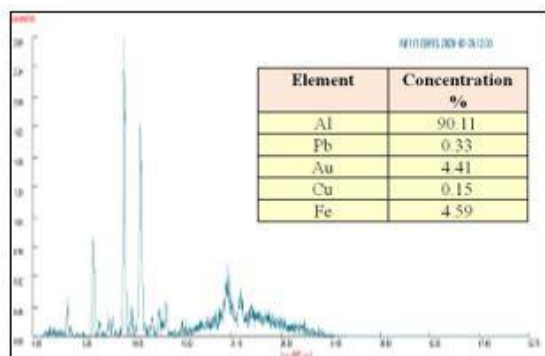


Fig. (10) XRF pattern of cartonnage gilding layer as a percentage concentration.

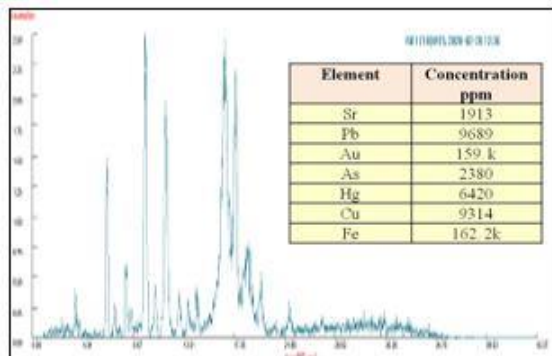


Fig. (11) XRF pattern of a cartonnage gilding layer as a ppm. concentration.

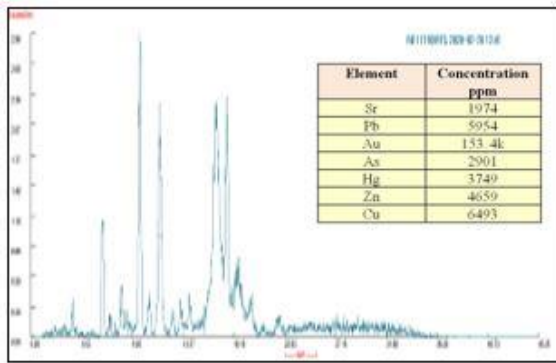


Fig. (12) XRF pattern of a cartonnage gilding layer as a ppm. concentration.

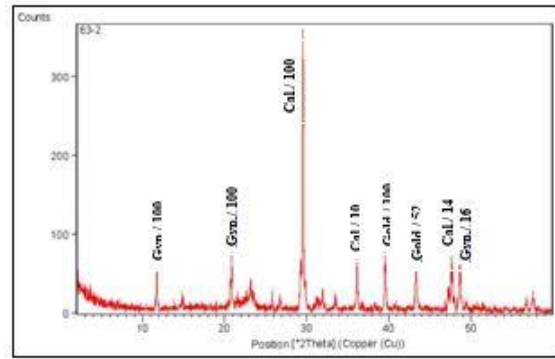


Fig. (13) XRD pattern of a cartonnage gilded layer.

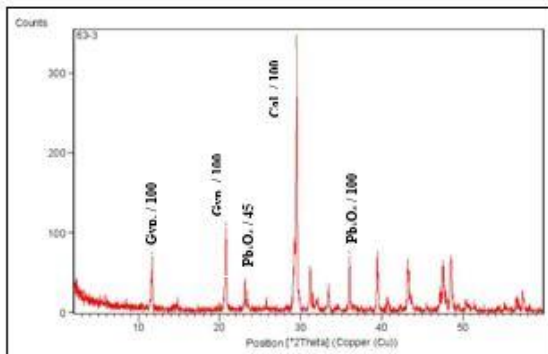


Fig. (14) XRD pattern of a cartonnage pink color

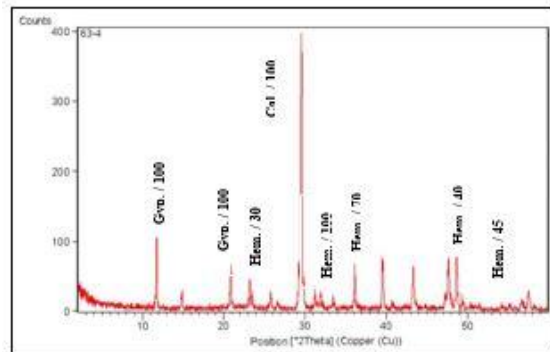


Fig. (15) XRD pattern of a cartonnage red - brown color

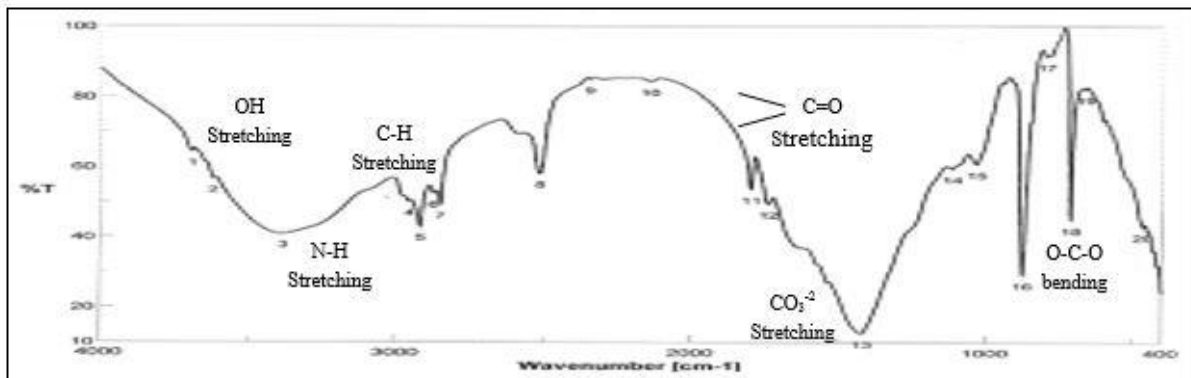


Fig. (16) FTIR pattern of a cartonnage gilding layer.

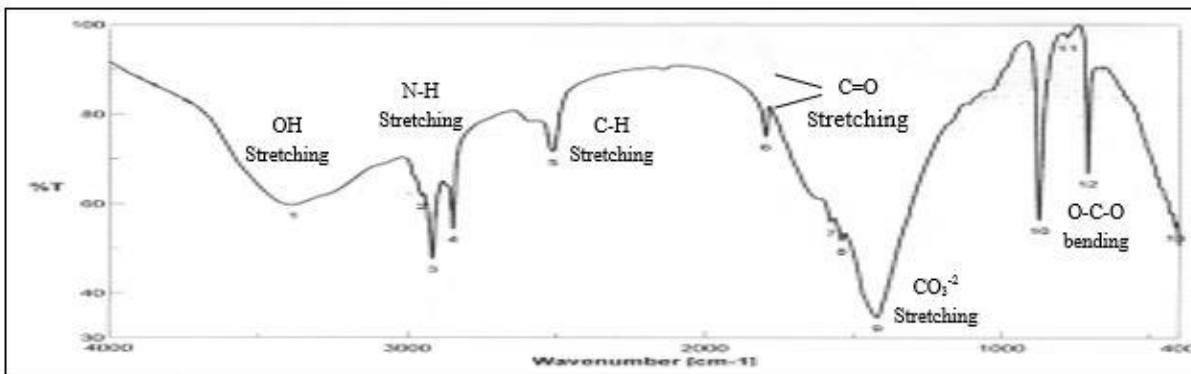


Fig. (17) FTIR pattern of a cartonnage red-pink color.



#### 4. Discussion

Examinations and analyses were carried out for the gilded cartonnage to understand the current situation of its deterioration phenomena and the contents of the cartonnage, especially the gilding layer and the gilding technique, (Rowe, S., 2010). Examination of the optical microscope revealed the presence of deep cracks. Among the cracks appeared the preparation layers located below the gilding layer and the various deterioration phenomena. Ripples appeared in the gilding layer indicating the technique used to fix the gilding leaves on the surface of cartonnage. By examining cross-sections the thickness of the gilding layer and the coloring layers have been identified. (Ali, M. et al, 2016). It turned out that there is homogeneity in the mineral grains of pigments. Joints in the gilding layer were appeared indicating the presence of holes and confluent parts on the surface, (Scott, D. A., 2003). It is clear that there is a loss of the gilding and pigment layers. The results of the scanning electron microscope and the optical microscope were matched, in the presence of deep cracks and fine cracks in the gilding layer. The ground layer appeared below the gilding layer and this indicates the extent of the thickness of the gilding leaves used, (Cyril A., 1998). It appeared clearly through the elemental analysis by (EDX) and (XRF) of the elements present in the gilding layer and the preparation layer below that the gilding layer is made of gold metal, whose presence has appeared in different values in the results of the analyses. As a result of penetration of the beam of radiation used in the analyses of the gilding layer and its penetration into the original layer below it, the calcite mineral (calcium carbonate) appeared where the elements of calcium, carbon and oxygen were found, (Afifi, H.A.M, 2011). Sulfur element also appeared, which indicates the presence of gypsum in the preparation ground. In addition, other elements appeared in the results of the analyses in a small percentage, the most important of which are iron, aluminum related to bole material (A clay mineral), sodium and magnesium minerals. The results of the X-ray diffraction analysis showed that, the red-pink pigment is red-lead  $Pb_3O_4$  (lead tetroxide), in addition of calcite and gypsum minerals, as it represents one of the important sources of red color in the Greco-Roman era, (Vaticani, M. 2007), and the brown pigment is hematite besides calcite and gypsum minerals. It is an iron oxide mineral that was widely used as a source of red color in the past as well as in the Greco-Roman era. (Ali, M. et al, 2016). On the other hand, the gilding layer consists mainly of gold metal. A percentage of gypsum and calcite minerals appeared due to the presence of the preparation layer below the gilding layer, similar to the results of (EDX) and (XRF). The FTIR results indicated that the adhesive used to fix gilding leaves on the surface of cartonnage is animal glue and has also been used as a color medium with the pigment materials which used in the cartonnage. (Derrick M.R., 1999).

#### 5. Treatment and Conservation of the gilded cartonnage

The gilded cartonnage contains completion of missing parts in the ground and gilding layers using wax as a previous restoration. The first step is to remove the previous restoration materials from the internal parts of the cartonnages. Wax which was used as an old adhesive in a previous restoration was removed very gently and slowly by applying small amounts of acetone. After removing wax a new ground layer and gilding layer were applied.

### 5.1. Mechanical and Chemical Cleaning

Cleaning process was carried out using soft brushes and manual tools to remove dust and dirt. The adherent deposits on the gilded layer were removed using organic solvents including, acetone, n-butyl alcohol, and ethylene glycol. Diluted organic solvents were used to prevent dissolving the color or the white preparatory layer, (Jaeschke, L., 1997). In the case of solid dirt, a solution of ethyl alcohol and turpentine (1: 2) was first used to dilute it and it was then removed. This method gave good results. Some colored parts were cleaned by eraser which was safe. (Alawneh, F. 2019).

### 5.2. Completing the preparatory layer and the gilding layer.

The preparatory layer was completed using clean brushes and spatulas. This was completed in three stages so that each layer was lighter in texture than the previous one, using a low glue concentration. The first layer consists of coarse calcium sulfate, as well as a 10% polyvinyl alcohol adhesive. This layer was left to dry to ensure easy and complete bonding with the subsequent layer. (Alawneh, F. 2019). The second layer contained fine calcium sulfate with 7% adhesive ensuring that the thickness of each layer did not exceed 1 mm. Thick layers increase the probability of separation and cracking of the preparatory layer. The second layer was left to dry at room temperature. Then a third layer with a concentration of 5% glue and a thin texture was applied. After this layer was completely dried, the surface was smoothed with fine sand paper till the surface was ready for gilding process with gold leaves. A coat of animal glue was applied on the surface of the ground layer then the gold leaves were applied. Fig. (18).

### 5.3. Injection of cracks and Completing the missing colors

The deep cracks were filled with linen fibers, 10% polyvinyl alcohol, and alkoxide was used as fungicide. Fine and small cracks were injected using paraloid-B72, 3% and Acrylic paints were used to complete the missing parts of the red and brown colors. Acrylic colors are characterized by reversibility and transparency which make them consistent with the background color. Fig. (19).

### 5.4. Protection process of the gilded cartonnage

It is essential to protect the gilded cartonnages from the surrounding atmospheric conditions. The cartonnage was completely isolated using 3% Paraloid- B72 dissolved in xylene by brushing both sides (outside and inside), (Podany, J., 2001). During application of the coating layer of Paraloid- B72, the direction of the brush strokes was taken into account, so one direction was used to eliminate the appearance of brush marks on the surface. All processes were conducted in a closed and clean environment. Fig (20). After this process, the conservation steps of the gilded cartonnage were completed and now, it is ready to display in Hurghada museum.

### 5.5. Display of the gilded cartonnage in Hurghada museum

The Plexiglas was used to create a base and stand for the gilded cartonnage in order to put it in the museum display. Plexiglas sheet is a material that has many commercial, industrial, and professional uses. Plexiglas is a thermoplastic material that can be formed according to the required shape; therefore, Plexiglas is one of the best materials used in museum display works. After completing the preparation of the Plexiglas according to the required shape, the gilded

cartonnage was placed in the museum display and a collection of jewelry was placed from the same period with the gilded cartonnage. Fig. (21)

## 6. Conclusion

The study showed that gilding leaves were used in gilding of this cartonnage. X-ray diffraction analysis showed that, the red-pink pigment is red-lead  $Pb_3O_4$ . (lead tetroxide), and the brown pigment is hematite and the gilding layer consists mainly of gold metal. The FTIR results indicated that animal glue was used as an adhesive to fix gilding leaves on the surface of cartonnage and as a color medium with the pigment materials which used in the cartonnage. A previous restoration in which wax was used Instead of the gilding layer. A new ground layer was applied and gilding leaves were used to re-gild these parts again. Deep cracks were injected and restored to strengthen them. The cracks were filled with linen fibers, 10% polyvinyl alcohol, and alkoxide as adhesives fungicide material. The mechanical and chemical cleaning processes of the cartonnage are fully performed. Plexiglas was used to create a base and a holder for displaying the gilded cartonnage in Hurghada Museum.



Fig. (18) Show completion of the ground layer and the re-gild process by gold leaves.



Fig. (19) Show injection of cracks, the gilded parts after the gilding process and the protection of the gilded cartonnage surface using paraloid – B72, 3 %.



Fig. (20) Show the gilded cartonnage after conservation and its details.



Fig. (21) Show the gilded cartonnage after displaying in Hurghada museum.

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