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PHYSICAL ANALYSIS AND TREATMENT OF DISINTEGRATED ISLAMIC MURAL PAINTINGS FROM THE 15th CENTURY TAQI ALDIN ALBISTAMI HOSPICE

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ABSTRACT

An investigation of the disintegrated mural paintings at the Hospice (Takiya) of Taqi Al-Din Al -Bistami dated back to (AH 847 / AD 1443) by various physicochemical techniques is presented. Deterioration phenomena such as cracks, loss in the preparation layers, colour changing, calcifications, salt efflorescence and soot were analytically studied. Analytical work was made to determine the origin of the archaeological used materials, such as, light optical microscope, SEM-EDX, X- Ray diffraction (XRD), Fourier transform infrared (FTIR). It is found two different preparation layers of a mixture of calcite, gypsum, and quartz. Red hematite and cinnabar, green malachite, yellow goethite and blue ultramarine were used as pigments. Identifying animal glue binder indicated that mural painting followed the tempera technique. The results of the research contribute to the conservation project of mural paintings.

KEYWORDS: Disintegrated; Takiya; mural paintings; tempera; detachment; reinstalling; EDX, SEM; Arabic gum; ultramarine.

1. INTRODUCTION

Mural paintings are a cultural asset that require protection via conservation science and careful restoration. Takiya of Taqi Al-Din Al-Bistami is located at Darb Al-Laban in Cairo and it is a registered monument number 326. It dates back to the Mamluk sultan Al-Mansour Hossam Al-Din Lagen 607 AH (Nicholas, 2000) and has been refurbished by Al-Sultan Al-Zahr Abo-Saeed Jqmaq (1443 AD/ 847AH). Tikya is an archaeological house dedicated to Sufism and the Dervish who lean and depend on their livelihood from the returns they receive. It contains deteriorating frescoes known as the mihrab mural and the bright heart, which is a rectangle with a mihrab inside, and in the centre is a heart surrounded by a necklace of triple floral leaves on a floor of roses painted in pink, green and red (Fig. 1). There are also murals decorated with floral and geometric designs (Fig. 1b). Painting has always been a form of expression since early human dawn. From the first Palaeolithic cave drawings until today (Liritzis et al., 2019). Relevant works in the past include Coptic murals (Ali and Youssef, 2020), mural paintings in el Sakakeny palace (Salama et al., 2016), and on Byzantine encaustic mural painting (Basem Gehad et al., 2015).

Cairo was exposed to an earthquake in 1992 that led to the collapse of many Islamic monuments, including parts of the walls of Tekke, including the loads of wall paintings. This led to the increase of various cracks in the walls, the weak cohesion of the paintings, the fall of some stone blocks that bear some parts of the wall paintings, as well as, the weak foundations due to the effect groundwater, and, thus, weak in the mechanical properties of the foundations that bears the walls due to the ground water and its role in soil deterioration (Hemeda and El-Banna, 2014). The present research aims to study the materials and techniques used in wall paintings to protect Taqi al-Din. Emphasis is given to tempera, a technique in which colour is used with the medium (Ali and Youssef, 2020). We further aim to the conservation of mural paintings through a detachment of the mural painting by *stacco* technique[1] and reinstalling after the conservation. Most of the mural were disintegrated and became stable condition after treatment. Hence, for first time, the type of materials and techniques used in wall paintings to protect Taqi al-Din, are presented resulting to choosing proper procedures of restoring the murals, as well as, for the removing and reinstalling the panels.

2. MATERIALS AND METHODS

2.1. *Sampling*

Samples were taken from weak fallen coloured pieces of painted preparation layers, which are representative of the mural painting (Maryse, 2009). Samples were collected carefully from the destroyed edge, using scalpels and some sharp tools, to identify the constituents of the paintings. The samples were collected from places expressing all the existing colours in the mural paintings (Fig. 1a, b, c, d), and suffer from cracks, fading, salts, losses in the paint layer and part of the layers falling out.

Figure 1. a) The mural paintings calling the Mehrab (Niche) and the enlightened heart At Takiya Of Taqi Al-Din Al—Bistami, b) the mural paintings containing Geometric motifs, and Floral Ornaments, c) all the mural paintings and the condition assessment of the mural paintings, d) The deterioration factors facing the mural paintings.

[1] A thicker layer of plaster is retained along with the fresco and is smoothed flat on its back surface before the composite rigid layer is mounted to a prepared support.

2.2. Light optical microscope

A stratigraphic study was conducted to find out the stratigraphic structure of the mural, where samples were taken from the preparation layers and the staining layer. It was placed in an epoxy mould (Epon 812, Spur, Lx-112), then left to dry in the surrounding room atmosphere for 18-24 hours, and the surface polishing process was studied using Silicon Carbide Paper, while reducing the sanding process as much as possible as it could perform sanding process increasing abnormalities in the layers, and samples were then examined by optical microscopy. It is used to identify the sequence of construction and painting layers and their thicknesses.

2.3. SEM-EDX

Quantum 3D 200 I (FEI Philips - Holland) scanning electron microscope with 60P column pressure specification, low vacuum, was used in the background dispersion mode and sample images were obtained in the background dispersion mode (BSE). It was helpful to analyse the quasi-quantitative elements of the samples (Hanlan, 1975) and to identify specific minerals that could not be easily identified using XRD (Perdikatsis et.al., 2000). SEM-EDX was used to study the morphology of the sample, the mineralogical composition, the micro-textural composition of the sample, and for the identification of chemical elements of the paint layer and the preparation layers. All are very helpful data in choosing the same colour composition in treatment, restoration and retouch work.

2.4. X- Ray Diffraction (XRD)

A diffraction device was used Philips X-ray Pert 32W X-ray Pert 32W (Ni-Filtered, Cu K) - PXRD data was collected using a Philips-related PW 3290 diffraction meter with NI filtered CU Kα and only directed at samples: The count statistics for the X-ray diffraction method were as follows; step size: 0.01°, The diffraction pattern was scanned over 3°–63° 2θ at a scanning speed: 0.02° 2θ/s. The 2006 high-grade X software was used for a semi-quantitative determination of detection threshold of ± 2% w / w for minerals components present. The measurements were made at room temperature. Preparation of each sample by grinding it in the dry form, by using a mortar and pestle to obtain a fine powder (Stuart, 2007).

2.5. Fourier Transform Infra Red (FTIR)

It is one of the most important spectral methods in quantitative and qualitative analysis to determine organic compounds, and sometimes inorganic compounds (Madejova, 2003). FTIR (Wavenumber Vs. Absorbance) was used to identify the medium which used in the paint and preparation layers, as identifying the medium helps to know the technique used (is the technique *fresco or tempera*). The most frequently used areas are the central region and in some devices the outermost region of infrared as the infrared spectrum was obtained in the medium term using the type of device JASCO-6100 FTIR. The sample was ground and placed in KBr potassium bromide tablets, where 0.1Mg of the sample was mixed with 5Mg of KBr.

Figure 2 (a,b,c,d,e,f). Details of deterioration of the mural paintings (cracks, change in colour, missing parts, flaking, and lost in the preparation layers).

3. RESULTS AND DISCUSSION

3.1. *Deterioration aspects*

The case study of the mural painting revealed status of deterioration and instability in most of the walls of the rooms, where the mural paintings are executed from outside, which naturally affects the painting. It appears in spreading cracks in the remaining parts of the painting, painting erosion and loss in the preparation layers, covering with a dusty layer, dirt on all the decorations, brittleness, fading, flakes, and loss in the paint layers, as shown in Fig. 2. The most severe deterioration factors affecting the mural paintings are the salts and the daily sun light (Salama et.al., 2016).

3.2. *Light optical microscope (LOM) investigation*

The stratigraphic structure was studied using a light optical microscope through cross sections (Madejova 2013; Katsibiri and Boon, 2004; Duran, et al., 2011) of the sample of the red and blue colour taken from the mural painting (Fig.3 a, c). That was composed of a support made of lime stone and preparation layer, the paint layer was executed on a very fine plaster layer which mostly consists of gypsum, and lime that was applied on a layer made of a mixture of gypsum, lime, a small percentage of sand, and some impurities. Its thickness reaches between 0.5 to 0.15 mm, while the colour thickness ranges from 0.2 to 0.4 mm with obvious flacks and loss in the red colour (Fig.3b). A cross section of the blue colour shows how thin and weak the blue colour is. Cracks and burrs are seen in the blue colour (Fig. 3d) and the colour thickness ranges from 0.3 to 0.5 mm. Present are some clay minerals and remains of the pigment mixed with gypsum and lime, some fine particles of the pigment which sprinkled on the stone surface, and in some parts loss of pigment (Fig. 3b,d). Examination of the colours shows the fading and dirt on the colour surface and the loss in the black colour layer (Fig. 4a). Remains of plant additives appear within the preparation layer (Fig. 4b). Finally, presence of flacks and salt efflorescence on the yellow colour surface is seen (Fig.4c). Fading, loss in the green colour, mixing of the red and green colour in some places and salt efflorescence (Fig. 4e, f) and the presence of flacks as yellow (Fig. 4d) are also present.

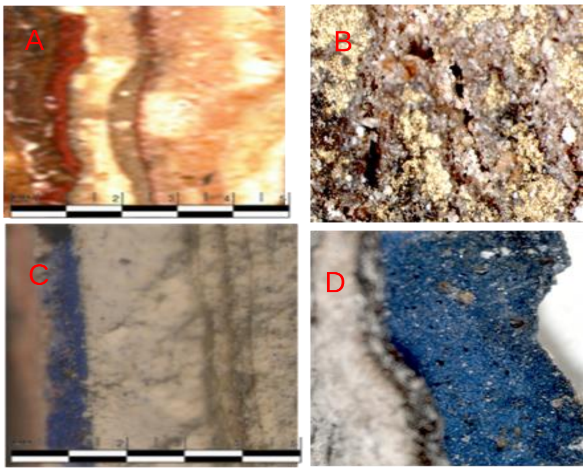

Figure 3. Stratigraphic structure of the painted ground layers (red &blue) by the stereomicroscope. (A) red pigment, under the reflected light and shows over painting (support- coarse ground layer, paint layer, dust- fine ground layer- red colour dust). (B) Shows of the Flacks, loss in the red colour. (C) A cross section of the blue colour showing six layers (support- coarse ground layer, dark preparation layer, fine ground layer, dark preparation layer, fine ground layer blue colour. (D) Cracks and burrs in the blue colour.

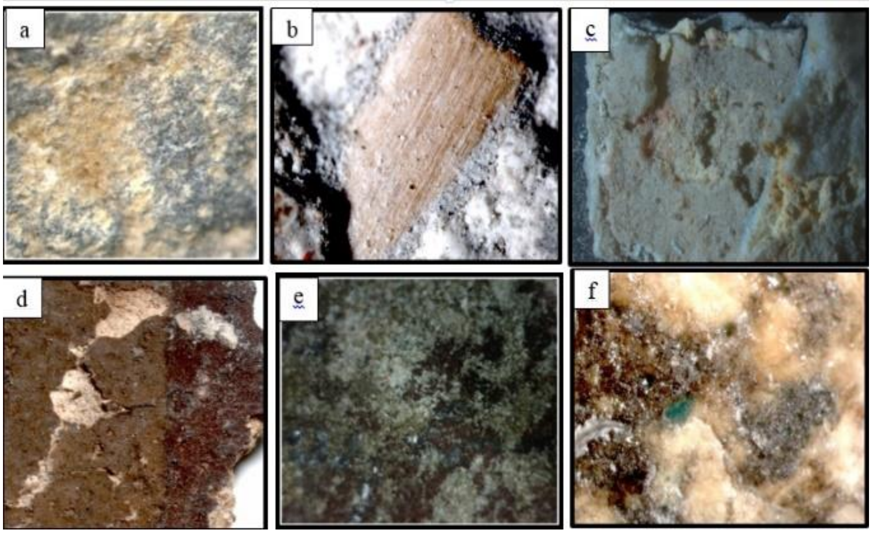

Figure 4. Imaging by the stereomicroscope (deterioration of Colours): (a) Black colour deterioration 150x, (b) The appearance of plant additions at the under of the colour layer 160x, (c) The crystallization of salts on the painted surfaces 150x , (d) Detachment in some layers of yellow colour 100x, (e, f) Degradation of red pigment and mixing with green colour 190x.

3.3. Results of Mineralogical Analysis

3.3.1. Ground layers

Through examination of the stratigraphic structure of the preparation layer using light microscope, it was clear that it composed of more than one layer where the first layer and it's a coarse and heterogeneous layer consists of different grains of quartz, calcite, and some plant additives to consolidate it and be coherent. Then a second layer with a color tends to red, maybe it resulted from red lead oxide and it is one of the second layer components. It is more homogenous among its components and finer than the first layer (Fig 5a.b). Through XRD analysis of the first layer it was clear that it is composed of 33% gypsum $CaSO_4.2H_2O$, 26% Calcite $CaCO_3$, 20% quartz and 19% Vaterite, which is one of calcium carbonate phases, though it may have been exposed to a higher temperature during preparation wih 2% Halite NaCl, indicating that the main component of the first preparation layer is gypsum and lime (Fig 5c). By XRD analysis of the second layer (Fig 5d) it was clear that it consists of 41% Zinc oxide, 30% Pb_3O_4, 18% PbO_2, and 4% $CaSO_4.2H_2O$. Also, the presence of zinc as one of the second layer components may be resulted from some previous restorations.

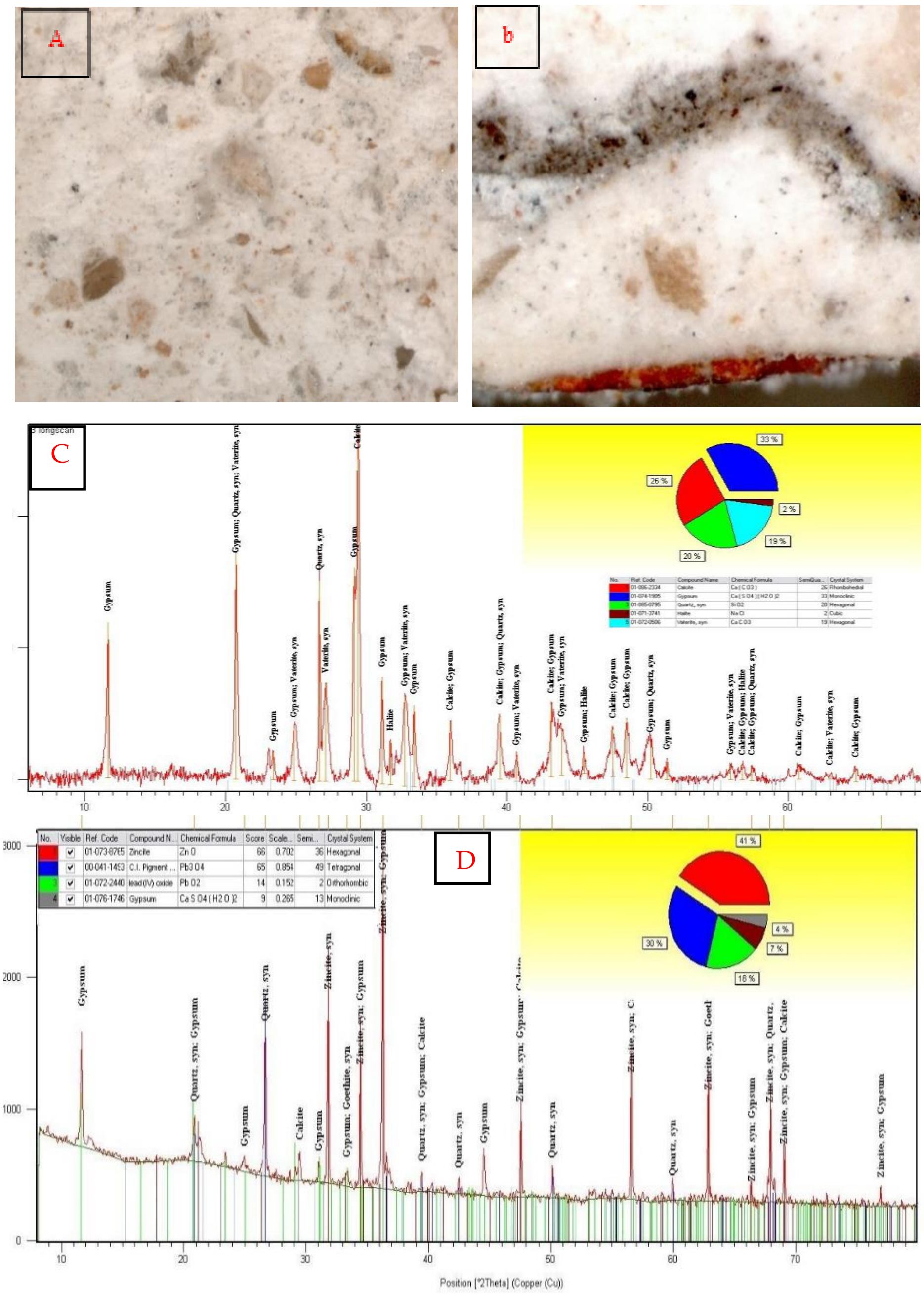

Figure 5. XRD patterns of the preparation layers and Imaging by the stereomicroscope. (a, b) Examination of preparation layers 300x, (c, d) patterns of the preparation layers (the first preparation layer and the second preparation layer.

3.3.2. *Red pigment*

The XRD results (Fig.8a) showed the compounds of the red colour which composed of 4% hematite Fe_2O_3 and its source is ferric oxide, where this oxide is widely found in Egypt and used since pre-dynastic period and over the Egyptian history (Moussa et al., 2009), and also, 89% calcite $Ca(CO_3)$, in addition to, 7% quartz SiO_2. Those distinct features are matched with the red ochre under a light microscope (Fig. 3b), where its colour ranges from red brown to black, transparency in some particles and opaque in other particles due to the association with some impurities. These results are concordant with those by EDX (Fig.8b), reinforcing that the sample containing the elements: Zn, Ca, Na, Si, Mg, S, Al, Cl, Zn, resulted from the preparation layer. The presence of iron at its both alpha and beta phases indicates red hematite also sodium chloride and some impurities. Examination by SEM shows appearance of non-distinct red colour where different crystals of red and brown are present (Fig.7a). Red colour grains are heterogeneous in size and composition in the case of decomposition and weakness, containing some spaces in the colour layer and incoherence of the colour grains and separating areas in the colour layer.

3.3.3. *Green pigment*

The X-ray patterns of the green colored powder shown in XRD (Fig. 8C) indicates the presence of gypsum recognized, in spite of the shifted peaks at 7.7, 4.3 and 2.88Å. The peaks observed in 3.39, 3.11Å are probably due to the red lead (lead tetroxide, Pb_3O_4), which main peaks are at 3.38 Å, with weaker patterns in 2.90 and 2.79 Å. A strong peak at 2.88 Å might be attributed to gypsum and overlap with malachite green, which main line appears at 2.86 beside weaker lines at 3.69 and 5.06 Å. The background of the diffractogram is diffused and reflects the presence of organic matters. These results agree with the EDX results (Fig.8d), where it shows that the sample contains the following elements: 21% Cu, 2% Ca, 2% Si, 1.42% Cl and 2.74% S. This indicates that the green pigment refers to green malachite (basic copper carbonate Cu_2CO_3 $(OH)_2$) and it has dark green color. Through examination using SEM the green color layer appeared clear and distinct where crystals of different sizes and interstitial spaces between crystals are appearing, also some color luster (Fig.7b). The green color, appearing of the granules that are heterogeneous in size and composition and in case of decomposition and weakness with some spaces and some salt efflorescence on the grain surface.

3.3.4. *Blue pigment*

The XRD results (Fig.9a) revealed that the blue colour consists of sodium silicates with aluminium and sulphur, where the ultramarine appeared 16%, calcite 56%, gypsum $CaSO_4.2H_2O$ 38%. Maybe the blue colour is the ultramarine $[(Na,Ca)_8(AlSiO_4)_6(SO_4.S,Cl)_2]$ (Plesters, 1993). This confirms that using scanning electron microscope with EDX (Fig.9b), the sample contains the following elements: Ca, 9.5% Na, 3% Si, 2.38 % Al, Cl, S resulted from gypsum. This indicates that the blue pigment maybe Ultramarine blue, where it was artificially prepared with a similar composition of azurite, therefore a natural and artificial term is used to distinguish between the two types (Eastaugh et al., 2004). Ultramarine blue was also identified in the Islamic wood decorations (Abdel-Ghani et al., 2013) also in the mural paintings which date back to Mohamed Ali dynasty (Darwish, 2013). Examination by using SEM, the blue colour layer appeared heterogeneous and the colour grains was not well prepared had a lot of impurities, calcifications and some grains that are not well grind and with apparent crystals of different sizes (Fig. 7c). The blue colour, appears loose with the decomposition of its grains, spreading of the cracks in the colour layer, as well as, the presence of some ungrounded grains during mixing and preparation of the colour, presence of spreading cracks on the colour and different calcifications on the colour surface and incoherence of the colour grains.

3.3.5. *Yellow pigment*

The X-ray diffraction analysis indicates the presence of gypsum beside possible peaks of red lead. Examination by using SEM, the yellow color layer appeared heterogeneous, full of voids and has some dirt and calcifications (Fig. 9a). Through scanning electron microscope EDX results of the sample (Fig.9c) it turned out the presence of some elements such as: (Fe, Ca, Al, Si) which refer to the presence of hydrated iron oxide, FeO(OH) (goethite) with the presence of a percentage of calcite and other elements, and this is due to the preparatory layer. Thus, it can be said he used the yellow ochre in obtaining the pigment and it turned out the presence of some elements such as: Si, Al, Ca K, Na, Fe, Mg and the presence of the sodium element refers to glauconitic of yellowish green color and that's what makes the yellow color tends to dark color.

Through SEM examination of the yellow color, it appears the weakness and decomposition of its grains, spreading of the cracks in the color layer, presence of some ungrounded grains during mixing and preparation of the color, presence of spreading cracks on the color and different calcifications on the color surface and incoherence of the color grains figure and separating areas in the color layer (Fig. 7d).

3.4. Fourier Transform Infra-Red (FTIR)

By comparing the functional groups of the sample taken from the colour layer with the standard functional groups of gypsum and animal glue, it became clear that gypsum and animal glue had been in the colour layer. This indicates that the medium used is animal glue (Table 1) (Fig 6).

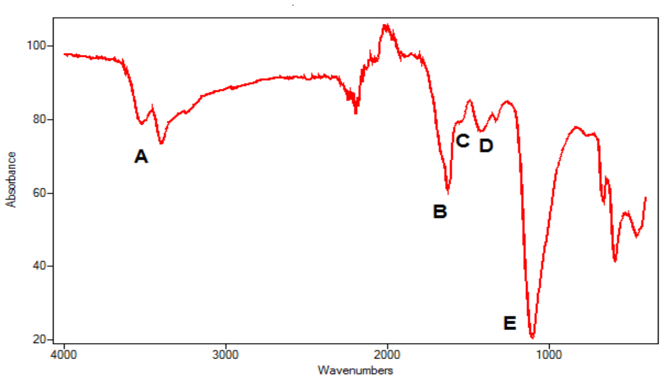

Figure 6. FTIR pattern of samples from the color layer.

Table 1. The main functional groups obtained from the color layer.

N	Functional Groups bands	cm^{-1}	Substance
A	OH stretching	3200-3420	Gypsum
B	stretching S-O C=O (Amide I)	1643	Gypsum, Animal Glue
C	CN stretching + NH stretching (bending)	1542	Animal Glue
D	CH_2 bending(Amide II)	1428	Animal Glue
E	C-O stretching	1102	Animal Glue

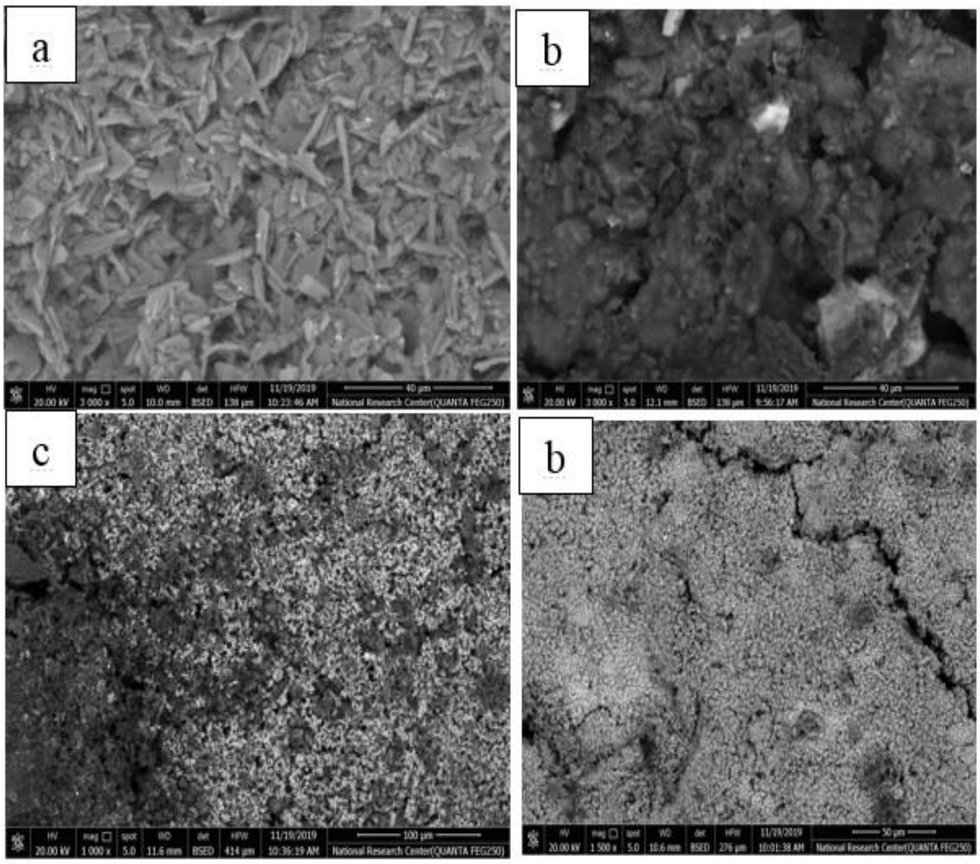

Figure No 7. Examination by SEM: (a) The investigations of red pigment 30007x , (b) The investigations of green pigment 3000x, (c) The investigations of blue pigment 1000x, (d) The investigations of yellow pigment, 1500x.

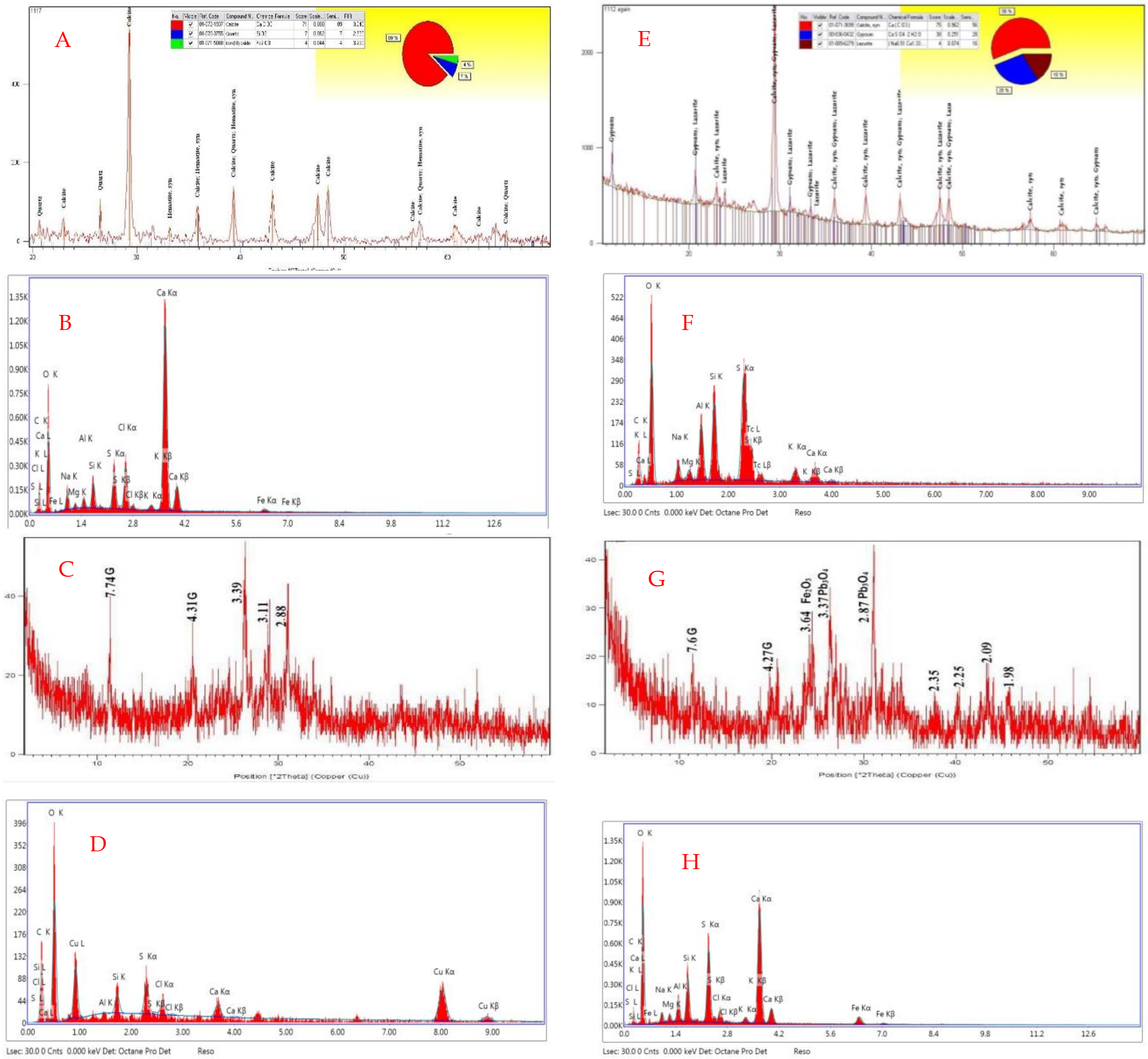

Figure 8. XRD patterns and EDX analysis of the pigments layers; (a, b) XRD patterns and EDX analysis of the red pigment; (c, d) XRD patterns and EDX analysis of the green pigment; (e, f) XRD patterns and EDX analysis of the blue pigment; (g, h) XRD patterns and EDX analysis of the yellow pigment.

4. TREATMENT AND CONSERVATION

4.1. Detachment of the mural painting

The mural paintings were divided into sections (Fig.9, 10) which ensures the safety of the detachment through sawing lines. In the sawing areas which were selected as far as possible away from rich areas with decorations some difficulties were faced, such as irregularity of mural paintings layers thickness, the presence of old iron screws of different lengths which the mural paintings were fixed with, the presence of cracks in most of the paintings, the collapses found in the support, and the irregularity of walls where it were constructed using irregular stones, stone blocks, and mud bricks.

Important steps had been done before detachment process such as cleaning of the painting surface from the suspended dust which impedes the protection layer. Removing the old iron bars used to fix the panels, consolidation of the decorations found using Paraloid B-72, and making of the facing layer using acid free gauze and apply it on the mural painting using Paraloid B-72 in concentration of 3% for the first layer and 5% for the second layer then 10% for the third layer, the first layer was executed so that it is horizontal. The decorations and the second layer is vertical and this ensures the coherence and adhesion of the protection layer on the mural paintings (Fig.11 a, b). Some of the electrical tools used for sawing and several saws were made of different lengths ranges from 50cm to 150cm which suits with all the mural paintings length (Fig.11c). During detachment, the focusing was on the weak areas in the support, where there were missing parts in the mural paintings in most of its layers. Hence, the focusing was on these weak areas in the support. In the beginning of the sawing and cutting through these parts through special holes at the edges were opened in the areas of the cutting borders. There, where, the sawing had been done in the mural painting's back, and only in the plaster layers and separated it from the support cautiously one part from another. Extreme caution was required when approaching the end of the detachment (Fig.11d), which was reinforced from the front using reinforced foam boards' 3 cm thickness and where the detachment is one of the methods of treatment. Similar treatment has been made to some monuments such as the tomb of Queen Nefertari in Luxor and the Church of Santi Quattro in Rome (Corzo, 1987; Svahn Garreau 2010; Herman, 2001; Lotfi, 2016).

4.2. *Re-attachment and conservation of the mural paintings*

The removed mural paintings were assembled with the fallen remains using 25% Paraloid B-72 dissolved in acetone. The assembling was done depending on the nature of the existing decorations, its shape, its boarders, and the shape of the edges, its thickness, and the fingerprint of the preparation layer's background and the existing plant fibers. After doing the detachment process and assembling the painting parts, parts of the weak preparation layers were removed and replaced by different mortars, where back support is done using mortar based on traditional materials such as lime, and salt free sand and addition of consolidation materials primal EC-33 to the mortar. Using of fiberglass net to reinforce the mortar layers which were added in the background (Fig.11 e, f), primal AC33 10% was used to detachment of the mural paintings (Basem, 2015). This helped in making every painting in a case of complete coherence and make it one easy to handle the block during re-attachment of it again on the old support, then cleaning of the stone support from the calcifications and dust and removing of some burrs that might hind the mural paintings during installing it on the support. This was done using chisels and electric saw after making sure the panels are in the right place, the external borders are fixed in its place on its own support in every painting and this made by scaffoldings equipped with movable jacks which help in pressing on the painting and fix it. After that the electric saw was used in making holes with the same diameter of fiber skewers. Then putting the skewers and fix them in the holes to make them alike between the mural paintings and the support (Fig. 11 g, h, m), and thus the paintings are in a more safe mode, if vibrations occur. Subsequently, the injection process, removing of the protective layer, cleaning and consolidation of the weak parts (Fig.11 n, o, p) and external walls were sprayed with water-repellant materials, such as the OH100 supported with titanium nano-oxide granules (Manoudis et al., 2009). Hence, the room and its mural paintings appeared before, during and after the restoration process (Fig.11 q, r, s, t, u).

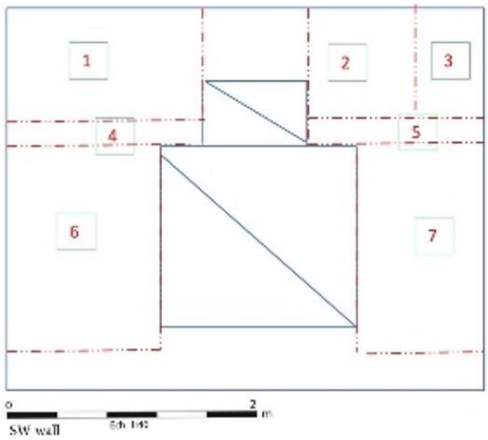

Figure 9. A drawing showing the cutting points in the eastern wall before detachment.

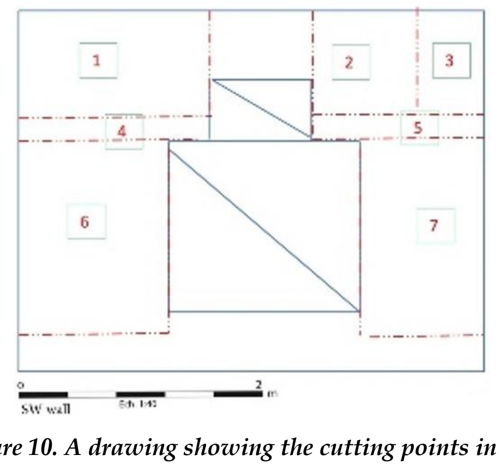

Figure 10. A drawing showing the cutting points in the northern wall before detachment.

Figure 11. Treatment, restoration detachment and re-attachment steps (a, b); Apply a surface protection layer (facing) Before detachment (c); the tools used for sawing. mural painting after detachment (d); During detachment (e, f); Add a fiberglass layer to strengthen the mortar (g,h,m); During re attachment the murals painting with the fiberglass bars (n); Remove the facing layer (o, p); cleaning the colours by wash up and chemical cleaning (s); mural painting before detachment (r); Wall paintings after detachment (s, t, u); Wall paintings after re-attachments) mural painting before detachment (r); Wall paintings after detachment (s, t, u); Wall paintings after re-attachment.

5. CONCLUSION

In this study a detailed investigation via analytical methods (light microscope, electron microscope, FTIR, X-ray analysis, and scanning electron microscope equipped with EDX) were used to identify mural paintings layers executed in Takiya of Taqi Al-Din Al-Bistami in Cairo. The study have shown the deterioration and instability of the painting's support and the mural painting, this can be shown in the cracks, erosion, loss in the preparation layers, dust, dirt on most of the painting's decorations, extreme weakness, general colour fading, gaps, cracks in the paint layer and separation of the paint layer from the preparation layer. There most of the damage was due to architectural and structural defects of the mural paintings increasing also the humidity factor. Most of the samples exhibited presence of chlorine and sodium that confirm the presence of halite salt but also the 1992 destructive earthquake. From the stratigraphic structure of the mural paintings it turned out that the used support is made of lime stone followed by two preparation layers; the first layer is a coarse thick layer composed of a mixture of gypsum, lime and a small percentage of sand and some impurities and the creator added some plant remains to the preparation layer and seems heterogeneous, and, the second layer, is a fine layer composed of gypsum and lime and in some cases zinc, appears homogenous.

Above these two layers there appears a paint layer that has a little thickness. Some colour was executed on the mural paintings and among the colours that have been recognized, red colour which consists of red hematite, green colour consists of malachite, blue colour consists of ultramarine and yellow colour consists of goethite. The FTIR study proved for some samples that the applied execution method of the mural painting is the tempera technique and the binder used in binding the colours is the Arabic gum. The mural paintings had been treated and removed using acid free gauze and apply it on the mural painting using Paraloid B-72in concentration of 3% and for the second layer 5% and 15% for the third. One of the best protection layers is the application of vertical and horizontal layers which increase the bearing strength of the protective layer of the mural painting. The use of net fiberglass layers as an addition for the permeated layers of the mural paintings increase the strength and coherence of the mural painting's layers. Use of wash up in the mechanical cleaning helps in removing and absorbing dust easily without abrasion to the paint layer.

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