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ANALYTICAL STUDY AND TREATMENTS OF THE DECAYED MURAL PAINTINGS AT ATHRIBIS IN SHEIKH HAMAD TEMPLE, SOHAG GOVERNORATE, EGYPT

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ABSTRACT

This article focuses on the identification of the building materials used at Athribis temple – Sohag and their most common degradation factors, and then assessing the most appropriate consolidants for the decayed wall paintings at the temple. The building materials were studied through XRD, SEM-EDX, FTIR spectroscopy and light optical microscope to identify chemical decompositions and physical failures. Soluble salts were detected in the building materials. White pigment is calcium carbonate, yellow pigment goethite, red pigment hematite, blue pigment Egyptian blue, green pigment Egyptian green, the paint medium is animal glue. Conservation treatments were achieved using Ethanol + PLM.I for injecting detached plasters, Sturgeon glue 1.5% as a filler for cracks, Syton x30 for pre-consolidation, EDTA poultices for cleaning, EDTA for salt extraction, Cidal 50 L50 as a biocide and Klucel G as a final consolidant.

KEYWORDS

Attrips temple; Wall paintings; Pigments; Organic medium; Salts; Consolidation; Cleaning.

الملخص

يهدف هذا البحث الى التعرف على مواد البناء المستخدمة فى معبد اتريبس – محافظة سوهاج وعوامل التلف المؤثرة على مواد بناء المعبد، كذلك يتناول البحث مواد التقوية المناسبة لتقوية الاجزاء التالفة بالصور الجدارية بمعبد اتريبس. فى هذا البحث تم التعرف على مواد بناء المعبد باستخدام طريقة حيود الاشعة السينية والميكروسكوب الالكترونى الماسح المزود بوحدة التحليل والتحليل الطيفى بالأشعة تحت الحمراء بالاضافة الى الفحص بالميكروسكوب الضوئى. و قد تم الكشف عن الاملاح القابلة للذوبان فى الماء بمواد بناء المعبد. كذلك تم التعرف على اللون الابيض ويتكون من كربونات الكالسيوم بينما يتكون اللون الاصفر من الجوثيت، ويتكون اللون الاحمر من الهيماتيت ويتكون اللون الازرق من الازرق المصرى واللون الاخضر عبارة عن الأخضر المصرى وقد تم استخدام الغراء الحيوانى كوسيط عضوى ومادة رابطة للمواد الملونة. وتم اجراء عمليات ترميم باستخدام الايثانول و مونة الجبس والجير لحقن طبقات الملاط . و استخدم الغراء بتركيز 1.5% كمادة مالئة للشروخ و مادة سايتون30 للتقوية المبدئية و ككذلك استخدمت مادة اديتا فى عمل كمادات للتنظيف أسطح الصور الجدارية و استخلاص الاملاح. و استخدم سيدال 50 كمبيد للكائنات الحية الدقيقة و استخدم كلوسل **G** كمادة تقوية للاسطح الملونة.

الكلمات الدالة

معبدأتريبس، صور جدارية، مواد ملونة، وسيط عضوى، أملاح، التقوية، التنظيف.

1. INTRODUCTION

Athribis is an old Egyptian city located on the west bank of the river Nile, that belongeds to the ninth district of Upper Egypt "Akhmim"[1], located about 8 km from the southwest of Sohag city. The origins of the term Athribis goes back to ancient Egyptian language (Hwt rpyt – the headquarter repit) the goddess" Repit" the female

[1] - Kuhlman K., et.al, Recent Archaeological Exploration, p.143.

hippopotamus, who was mentioned in many texts discovered on the site especially in the temple of Ptolemy XII (Olitus the piper), then became the Greek "Atrips"[2], and in Coptic it was known through several names, including "Atrip - Adripp - Atripe[3]".

The development of the temple of Athribis started during the reign of Atrips temple dates back to the period of Ptolemy XII (about 80 -50 BC)[4]. Some additions were made by the Roman Emperors Teripios and Claudios, the temple was finished during the ruling of Hadrian (117-138A.D.). According to the Excavations results, this temple is the only Ptolemaic temple built in limestone in Upper Egypt. The temple extends from the southeast to the northwest with a length of 78m and a width of nearly 45m, it is surrounded by a huge wall of mudbricks. The temple was dedicated to the Trinity of the goddess “Repit”, her divine husband “Min“and the child “Kolanthis”[5]. In 2000,[6] A preliminary Study for Restoration and Conservation of the remains of the Temple was conducted. The Egyptian-German expedition, which has been working (discovery, Documentation and Registration) in the temple since 2003, has discovered a large part of the Atrips temple (Figure 1), which retains many beautiful inscriptions [7]. Many parts of the temple were lost as it was use as a quarry for many ancient buildings

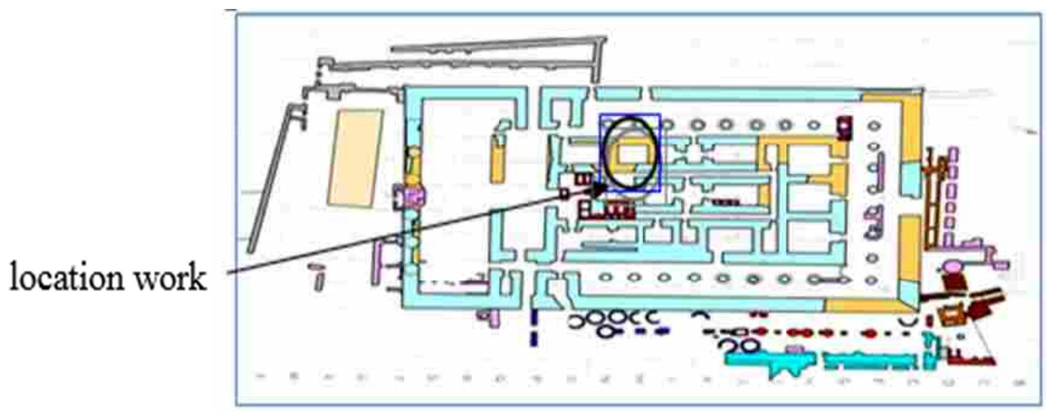

Fig. (1) a general plan of Athribis temple shows location work

Indeed, many of its stone blocks were used in constructing the nearby White monastery in the 5th century AD[8]. The villagers of the modern (Athribis – Sheikh Hamad) used some of the temple’s stones as bases for thier buildings and to erect dams reducing the flood risks[9]. A part of the temple was also used as a barn by the villagers for assembling animals during floods[10]. This has been demonstrated by the various missions’ excavations, through studying the sediments seen in the drilled layers. Despite this, the temple walls contain dozens of scenes and traditional texts, commonly seen in Greek and Roman eras’ temples [11], In addition to being recorded,

2 - Nour Eldin A., the ancient archeological sites, p. 139.
3 - Habib R., The Monastery Monasticism, p.177.
4 - Nashi I., The History of Egypt, p. 265.
5 - Mansour N., Akhmim the capital of the ninth region, p 264.
6 - Omran A., A scientific study for the restoration and Conservation, p. 30.
7 - German Mission: Atrips Temple Project.
8 - Meiemardus O., et al., Ancient Egypt, P.294.
9 - Zaki S., Archaeological finds from the excavations, p. 155.
10 - Ibid, p. 6.
11 - Nour Eldin A., The location and museums of the Egyptian monuments, p, 161.

these should be preserved. This All of these are the reasons prompted the researcher to select this temple for study.

2. MATERIALS AND METHODS

2.1 Investigated materials

The Limestone, mortars, plasters and pigments from Athribis temple were sampled and prepared for laboratory investigations and analyses which helped to determine their physical structure and chemical composition.

2.2 Methods

2.2.1 A stereo microscope type Stemi dr 1663 Zeiss was used.

2.2.2 X-ray diffraction (XRD) analysis was performed using a Philips Model PW 1710 control unit Anode material CU 40 K.V, 30 M.A, Optics: Automatic divergence slit Beta filtering using graphite monochromator, error = 0.16%.

2.2.3 SEM study was performed using SEM study was performed using JEOL JSM

5400 LV EDX Link ISIS-Oxford "high vacuum".

2.2.4 FTIR analysis was performed using Nicolet 6700 FT-IR Spectrometer from Thermo Scientific.

3. RESULTS

3.1 Light optical microscope results:

Most of the pigments were applied directly on the limestone surface after adding a thin layer of whitewash, the latter containing quartz particles in a small amount. All pigments were fine grounded and pulverised as they have a high covering power, homogeneity and interference with the whitewash layer is also observed (Figure 3 to Figure 7,x 50)

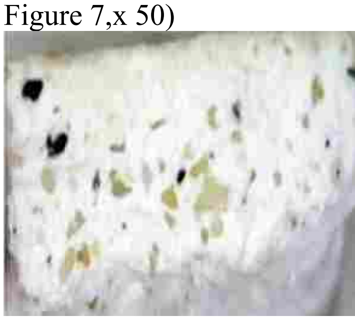

Fig. 2: Thin layer applied to the white pigment from Athribis temple.

Fig. 3: Thin layer applied of the yellow pigment from Athribis temple.

Fig. 4: Thin layer applied of the red pigment from Athribis temple.

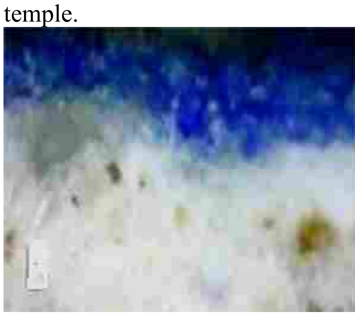

Fig. 5: Thin layer applied of the blue pigment from Athribis temple.

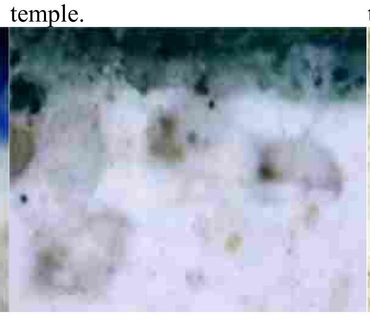

Fig. 6: Thin layer applied of the green pigment from Athribis temple.

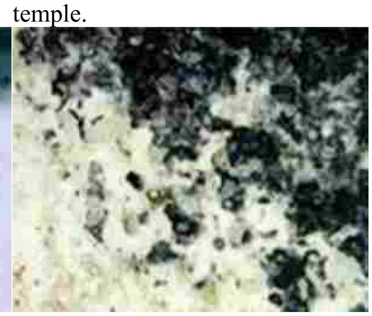

Fig. 7: Thin layer applied of the black pigment from Athribis temple.

3.2 *XRD results:*

According to XRD analyses, the first sample is a hard limestone as it is a pure calcite 100% ($CaCO_3$) (Figure 8). The second sample white pigment is a calcium carbonate pigment made of 84.8% calcite ($CaCO_3$) and 15.2% quartz (SiO_2) (Figure 9). The yellow pigmented plaster sample contains 73.2% $CaCO_3$, 13.9% goethite FeO(OH) and 12.9% hematite (Fe_2O_3) (Figure10). The red pigmented sample contains 79.5 % gypsum ($CaSO_4.2H_2O$), 14.5% hematite (Fe_2O_3) and 6% halite (NaCl) (Figure 11). The blue pigmented sample is a pure Egyptian blue as it is composed of 100% cuprorivaite $CaCu.Si_4O_{10}$ (Figure 12). The green pigmented plaster sample contains 75.7 % of Egyptian green (Wollastonite $CaCu.Si_3O_9$) complemented with 24.3% of calcium carbonate ($CaCO_3$) (Figure 13).

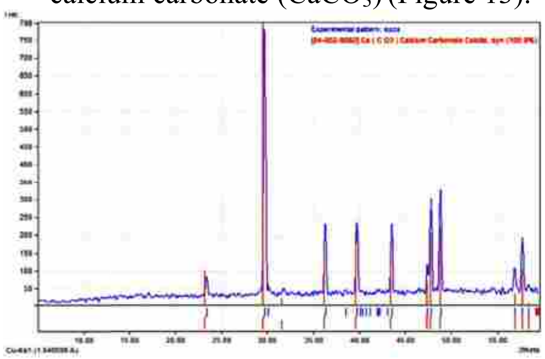

Fig. 8 XRD pattern of the lime stone sample from Athribis temple.

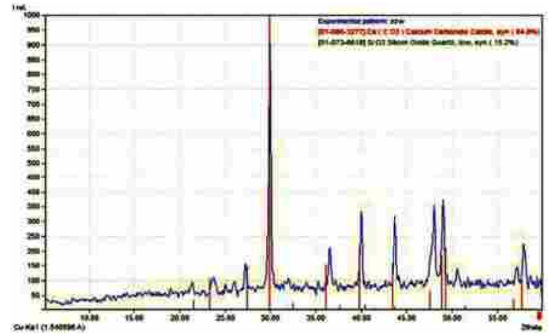

Fig. 9 XRD pattern of the white pigment sample from Athribis temple.

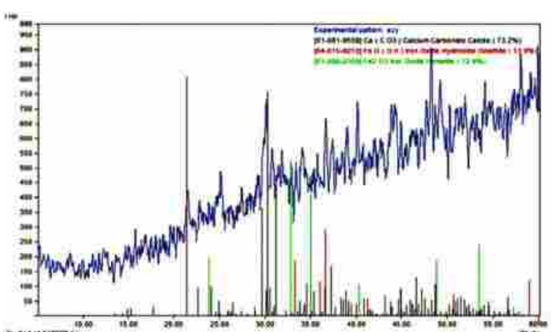

Fig.10 .XRD pattern of the yellow pigment sample from Athribis temple.

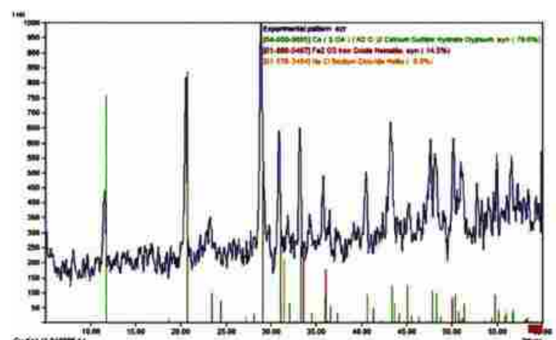

Fig. 11 XRD pattern of the red pigment sample from Athribis temple.

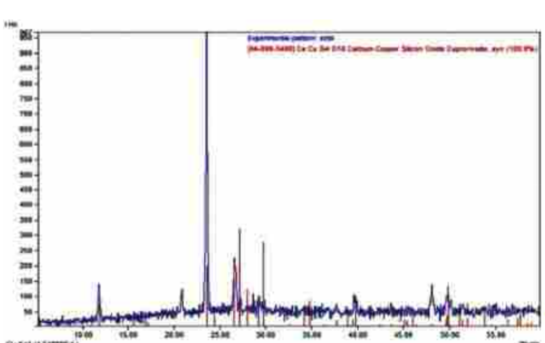

Fig. 12 XRD pattern of the blue pigment sample from Athribis temple.

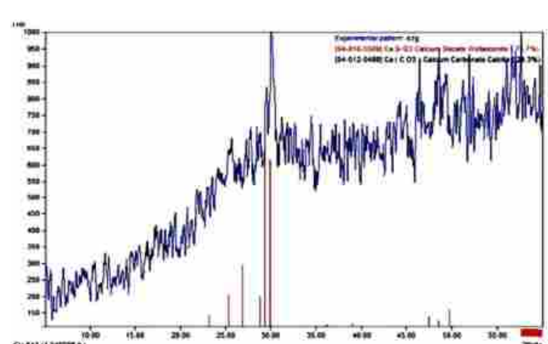

Fig. 13 XRD pattern of the green pigment sample from Athribis temple.

3.3 SEM-EDX results:

Building materials of the temple were studied through SEM-EDX to emphasize the results of the XRD analyses, samples were first investigated and then analysed. The first sample shows that the limestone is suffering from different weathering factors as halite crystals are of different sizes. While the binder is lost in some parts, the EDX analysis shows that the sample is made of 80% calcium with quartz which may indicate that sand stuck to the stone gaps due to weathering and some traces (Figure 14). The white pigmented plaster sample shows severe deterioration including gaps, cracks, plant fossils and salts. This sample contains some wooden or plant fibers which may be due to the artist's brush (Figure15). EDX analyses show that, the sample contains 72% calcium, 26% Sulphur, a small ratio of quartz and some iron traces (Figure 16). The yellow pigmented plaster sample shows gaps and salt crystals between and above pigment particles, EDX analyses show that the sample contains 62% calcium, 20% iron and 18% chlorine (Figure17). According to SEM, the red pigmented plaster sample shows the effect of dissolved salts in the groundwater, mainly gypsum and halite. The EDX shows that the major components are calcium and sulphur, followed by chlorine, a minor amount of iron was also detected. These results are exactly the same as those of the XRD analyses (Figure17). The blue pigmented plaster sample shows under SEM many blisters on the pigment surface, while the EDX proved that the sample is composed of Ca, Cu, and Si as major components (Egyptian blue – cuprorivaite) with minor amounts of Na and Cl (Figure 18). According to the SEM results, the green pigmented plaster sample suffered from a loss of cohesion between pigment particles, the sample shows also gaps and salt crystals upon its surface, EDX analyses proved that the sample contains Ca, Cu and Si as major components also (Egyptian green – wollastonite), Sulphur was also detected in a significant amount (Figure 19).

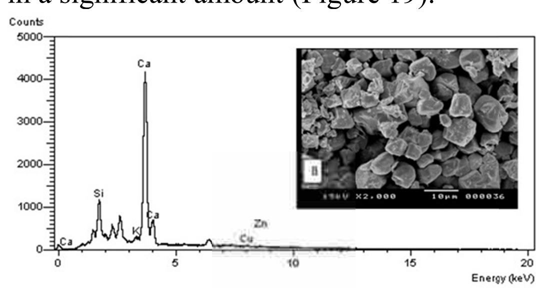

Fig. 14: SEM spectrum and SEM micrograph (2000x) of the limestone sample from Athribis temple.

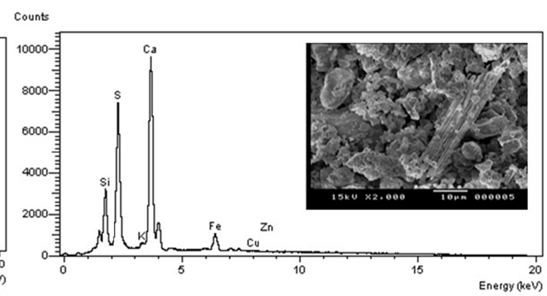

Fig.15: SEM spectrum and SEM micrograph (2000x) of the white pigment sample from Athribis temple.

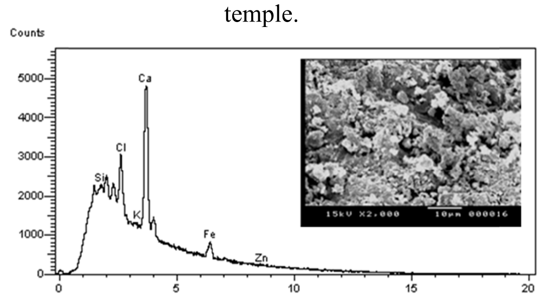

Fig. 16: SEM spectrum and SEM micrograph (2000x) of the yellow pigment sample from Athribis temple.

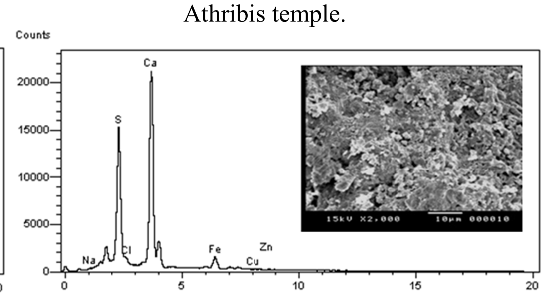

Fig. 17: SEM spectrum and SEM micrograph (2000x) of the red pigment sample from Athribis temple.

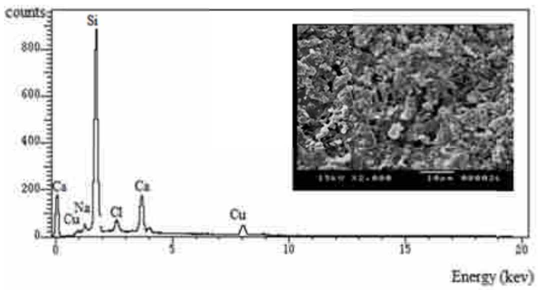

Fig. 18: SEM spectrum and SEM micrograph (2000x) of the blue pigment sample from Athribis temple.

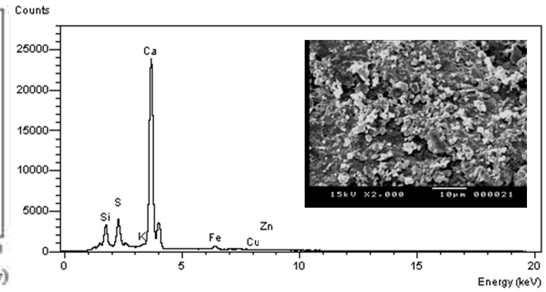

Fig. 19: SEM spectrum and SEM micrograph (2000x) of the blue pigment sample from Athribis temple.

b. FTIR results:

One paint media sample from mural paintings of the Athribis temple were analysed by mean of FTIR. The results show that the paint medium is animal glue (Figure 20, 21).

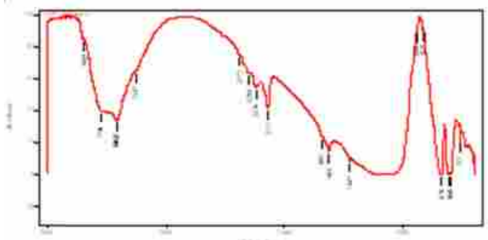

Fig. 20: FTIR spectrum paint medium sample clearly is animal glue.

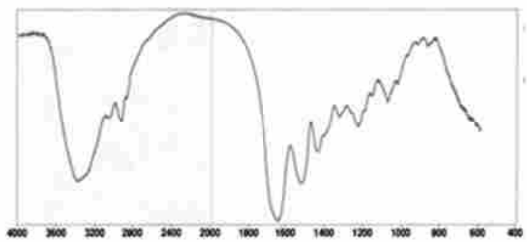

Fig. (21) Standard sample (after Derrick, Stulik and Landry[12])

Table (1) shows the wavelength and function groups of Archaeological paint medium sample and animal glue Standard sample.

Archaeological paint medium sample		animal glue Standard sample	
wavelength	**function groups**	**Wavelength**	**function groups**
3411 cm^{-1} - 3246 cm^{-1}	N-H stretching band	3400-3200 cm^{-1}	N-H stretching band
3020 cm^{-1} - 2919 cm^{-1}	C-H stretching bands	3100-2800 cm^{-1}	C-H stretching bands
1683 cm^{-1}	C=O stretching band	1660-1600 cm^{-1}	C=O stretching band
1623 cm^{-1} - 1520 cm^{-1}	C-N-H bending band	1565-1500 cm^{-1}	C-N-H bending band
1446 cm^{-1}	C-H bending band	1480-1300 cm^{-1}	C-H bending band
876 cm^{-1} - 680 cm^{-1}	N-H group		

12 - Derrick M. R, Stulik D.and Landry J. M., Infrared Spectroscopy, p.181.

4. DISCUSSION

Based on the visual investigation, much deterioration is visible at Athribis temple, such as fragility and detachment of pictorial layers, due to salt crystals, wind erosion, and biological infections. Through an optical microscope, one sees that the pigment layer is homogeneous although salt effloresces. The yellow pigment was applied upon a red pigment layer, this superposition may have been done to increase the covering of the pigment or to give the effect of gilding. SEM investigation showed that the pigment particles lost their correlation as salt crystals grew among them. Moreover, some particles are loosening and falling due to the fragility and detachment of the pictorial layer. According to XRD analyses, the support of the paint is a fine pure hard limestone, a rare instance in Upper Egypt, due to the geological profile of Egypt. Limestone tended to be the support of choice in the north, while sandstone was in the south. Grounded limestone was also the source of the white pigment at the Athribis temple. The presence of sand refers to the painting ground mortars which consisted primarily of gypsum, lime and sand. This portion had been an integral part of masonry structures for thousands of years[13]. Although the temple belongs to the Greek era in Egypt, the same pigment pallet has been in use of the ancient Egyptian style of achieving tempera wall paintings, yellow pigment is still goethite, and the red is hematite, both used in Egyptian murals for millennia. Blue is Egyptian blue, which had been made and use since the fourth dynasty[14] till the Greco-Roman period[15]. The Egyptian green has the same value than the Egyptian blue, but it started to be use later. EDX has assured the previous results. The FTIR analysis of the organic media proved that the artist used animal glue. This strong binding material was used as a color mediator connecting the color beads together and sticking to the dry painting ground layer in mural paintings since ages[16].

5. Treatment and conservation:

a. Pre-Consolidation:

The temple's mural paintings are in a bad condition. Its pre-consolidation process is fundamental. The stability of the fragile separated parts must be enhanced before initiating restoration processes. The physicochemical similarity between the treated material and it′s consolidated is an important aspect in the consolidation process[17]. Syton x30[18] (Ethyl Silicate protect against corrosion especially underground and industrial atmosphere[19] , Table 2), it used dissolved in water 4% to re attach the detached pigment layers (Figure 22,23,24,25), table 2. Cracks were injected and

13 - Moussa A., 2018, Nano Treatment of Decayed Cement-Lime Motars p.75.
14 - Lucas A., Materials and industries pp:560-561.
15 - Orabi E., Study and conservation of pigments deterioration, p. 67.
16 - Orabi E., Mural Painting Technology, p. 55.
17 - Victoria E., García V., Antonio J., Tenza A., Afonso M. and Marcos L., Calcium hydroxide nanoparticles, p.1.
18 - Langston L., Conservators of Wall Paintings, p.8.
19 - Parashar G., Srivastava D. and Krumar P., Progress in Organic Coatings,Ethyl silicate binders p. 1.

strengthened with Sturgeon glue 1.5%. (treated with heating process under 75°C)[20] (figure 26). This transparent glue does not cause any change in the surface and has excellent adhesive properties, it is also flexible and workable. Japanese paper immerged in Syton30 and dissolved water (10%) has been used as a fixative for the fragile pigment particles. Klucel G (hydroxypropyl cellulose) dissolved in Ethanol 5% has also been also used for the same purpose[21,22].

b. Biological Control: -

The places of insect's infections have been sterilized by using Ethoxy Carbon di Methyl Phosphor Rothioate known commercially as (Cidal 50 L 50). It has a long-lasting phosphoric component that is easy to be applied and excellent safety profile[23]. Methyl bromide was also used in cleaning the biological spots[24].

c. Cleaning: -

Mechanical cleaning has been done by brushes of different size, by metallic spatulas sometimes, by scalpels, and by air blower to clear the surface accumulation and to remove dust and sand from the mural painting (Figure 27). EDITA poultice (Tetra Sodium Salt Ethylene Diamine Tetra Acetic Acid) was used conjointly for this purpose (Figure 28, 29, 30), in order to overcome different spots and calcifications[25,26,27,28,29]. Micro emulsions are also used as they are transparent and stable materials[30.]

d. ***Salt's extraction:*** *-*

Poultices of distilled water were used to remove the halite salts, in addition to cotton poultices filled with EDTA solution. This enabled the extraction of insoluble salts such as carbonate and sulphate[31].

a. Completion

A grout consisting of (sand + matt lime + limestone powder size 250 microns concentrations 2: 1: 1) was applied to fill the gaps, while a mortar of (Ethanol) and

[20]- Eriksen A. M., Kristensen H. V. and Boellingtoft p., Bo Botfeldt K. and Rasmussen, Identification of Animal Adhesives, p.369.
[21] - Dan L.,et al, The effect of adding PDMS-OH and Silica nano particles on Sol-gel ,p.368.
[22] - Brus, J., and Kotlik, P., Consolidation of Stone, PP: 109-110.
[23] - Kovacs R., Gesztelyi R., Berenyi R., Doman M., Kardos G., Juha´sz B. and Majoros L., Killing rates exerted by caspofungin in 50 % serum, p. 186.
[24] - Valentin, N., Libstrom, M., and presser, F., Microbal Control by low oxgen, p. 222.
[25] - De Guichen , G., : Object Interred , p . 22 .
[26] - Moncreiff, A., and Weaver, G., Cleaning in Science for Conservators, pp. 13-21.
[27] - Mora et al, Conservation of Wall painting,p.289.
[28] - Torraca,G., Solubility and Solvents for Conservation,p.4.
[29] - E. De Witte, M. Dupas, Cleaning poultices based on EDTApp:1023–1031.
[30] - R. Giorgi and E. Carretti, Cleaning: Applications and Case Studies, pp.236-237.
[31] - Ashurst , J. and Ashurst , N., Practical Building Conseruation , p. 77 .

(PLM.I) powder was prepared. This mortar was injected in fine cracks and used to re-attach of the plaster layer.

Fig. 22 Detachment part in the mural painting

Fig. 23 The Detachment part on Japanese Paper

Fig. 24 Replace the separated part in its place

Fig.25 Re attaching the detached pigment layers.

Fig.26 Cracks injected and strengthened with Sturgeon glue

Fig.27 Air blower to remove dust and sand from the mural painting

Fig.28 Scene before applying the poultice.

Fig. 29 Scene during the application.

Fig. 30 Scene after cleaning.

6. CONCLUSIONS

The mural paintings in Athribis temple were done on a of limestone base that was coated with a whitewash, the ground oxides colors were then applied using the Tempra technique. The analyses showed that the white pigment is calcium carbonate, the yellow pigment goethite, the red pigment hematite, the blue pigment Egyptian blue while the green pigment is Egyptian green. The paint medium is animal glue. The research has pointed out that Athribis temple is suffering many failures such as salts found on the surface of inscriptions and detected in most building materials. There were also cracks in addition to weakness and separating of some colored inscriptions in the form of peeling some insect damaged the appearance also. The research team achieved thorough restoration to enhance the durability of the temple, and to improve the service life of decayed building materials at the temple.

Appendix 1 Materials treatment:

Table (2) Additional appendix of the materials treatment**.**

No	commercial name	scientific name	Company	Properties
1	**Kalx kP1060**	Plaster lime Mortar injection (PLMI)	Steadfast	high quality slaked lime injected in cracks and voids, lime grains size between 1-2.5 Microns
2	**Klucel G**	hydroxypropyl cellulose	GMW (Geräte Material Werkzeug Germany)	Soluble in water and polar solvents. Used in low concentrations and in higher concentrations as an adhesive
3	**Sturgeon glue**	manufacturing of animal glue	TALAS - Brooklyn, New York	Treated with heating process under 75°C
4	**Sytonx30**	ethyl silicate	Kremer Pigmente GmbH & Co. KG (Germany)	An aqueous silica acid dispersion, 30 % concentration of silica and a specific weight of 1200 g/l
5	**Cidal 50 L 50**	Ethoxy Carbon di Methyl Phosphor Rothioate	States of America. Patent and Trademark Office	strong in effect, easy to apply, effective for long periods and excellent safety profile
6	**EDITA**	Tetra Sodium Salt Ethylene Diamine Tetra Acetic Acid		Its reaction is shallow and does not penetrate the pores of the murals, highly capable of dissolving various materials and planktons and Easily removed

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