ANALYTICAL STUDY OF THE MATERIALS USED IN MURAL PAINTINGS IN THE LOVE CHAMBER OF EL SAKAKENY PALACE

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1. Introduction

The 19th century is considered to be the most important period in Egyptian art, and is distinguished by the great paintings produced by foreigners in the country at that time. During this century, several mural paintings were executed on the ceilings and walls of several rooms in a style that differed from the usual oriental one used in Egypt [1].

1.1. Stratigraphic studies

The first layer of rough coating, applied on the wall support of limestone, is a mortar composed of a mixture of hydrated lime (calcium hydroxide) and coarse sand. The second layer consists of zincate and gypsum. The third layer is the pictorial layer, made of pigments, the medium being applied in several coatings with a brush [2]. In this study, three mural paintings were fully investigated. Besides gaining technical information about this type of painting, the purpose of this research was to acquire knowledge about both the organic and inorganic materials used. The study comprised the examination of the pigments, media and ground layer [3].

1.2. The examined paintings

The mural paintings are located in the Love Chamber on the first floor of El Sakakeny Palace in El Sakakeny Square. The style is Rococo, as the colours are light and there is obvious movement in the flight of the birds and in the movement of the planets (Figure 1). Their dimensions are about 20 x 25 cm, 20 x 45 cm and 20 x 25 cm without the frame, whose width is 1.8 cm. El Sakakeny Palace dates back to the 19th century.

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1.3. Conservation state of the paintings

The analyses of these paintings were carried out during restoration treatment. From the visual inspection and cross section images, the condition of the paintings is poor, with weakness in the paint layers, which is the most apparent deterioration phenomenon (Figure 2 A-F).

There are several areas of the pictorial layer that have become detached, with chromatic alterations around the missing parts, grease and soot spots, and detachment of decorative elements.



Figure 1. The three mural paintings in El Sakakeny Palace showing sampling points.



Figure 2. Condition assessment of the mural paintings: (A) detachment of decorative elements; (B) loss in paint layers; (C-D) soot and grease spots; (E) blistering; (F) cracks.

2. Materials and methods

2.1. Sampling

Samples 1-8 were collected carefully from the damaged edges using a micro scalpel to identify the constituent materials and degree of deterioration of the paintings. All the analyzed and investigated samples were collected from areas without aesthetic value or from severely damaged parts.

2.1.1. Optical Microscope (OM)

The samples were examined with an optical microscope to identify their shape and chromatic alterations. In addition, the Optical Microscope helped us identify aspects of the damage within the samples, including the minutest cracks.

2.1.2. X-Ray Diffraction (XRD)

The X-ray diffraction patterns of the paintings were obtained using a diffractometer (Philips PW 1840) operated at 40 kV and 25 mA, using Cu K α radiation and a receiving slit of 0.2 mm. Measurements were made at room temperature. Preparation of each sample consisted of grinding it in the dry form by using a mortar and pestle to obtain a fine powder.

2.1.3. Scanning Electron Microscope coupled with EDX (SEM-EDX)

Samples were analyzed with an Environmental Scanning Electron Microscope (FEI, Netherlands) coupled with Energy Dispersive X-ray analysis. The microstructure and morphology of the mineral compounds in the paintings were analyzed with a FEI Quanta 200 Scanning Electron Microscope. The microscope operated at an accelerating voltage of 30 kV. Sample preparation consisted in the application of a superficial gold film by sputtering to prevent electrostatic charge.

2.1.4. Fourier Transform Infrared - Attenuated Total Reflectance (FTIR-ATR)

Samples were analyzed with an FTIR spectrometer (Model 6100 Jasco, Japan). Spectra were obtained in the transmission mode with a TGS detector and using an ATR crystal which represents (2mm/sec) Ni-Colet 760 added scans at spectral region ranging from 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

3. Results and discussion

3.1. Optical microscope (OM)

After examining the samples from the paintings, the following observations were made: the sample of black color was composed mainly of fine to medium-grained quartz, iron oxides applied as a layer on the ground layer; the reddish brown contained black spots; the blue color was of a powdery consistency, the deep green color was applied in multi layers, as was the golden green color (Figure 3A-F).

3.2. X-ray diffraction (XRD)

The XRD patterns of the samples from the mural paintings in El Sakakeny Palace indicate the results resumed in Table 1 and Figure 4.

XRD analysis results revealed the following:

The presence of 100% gypsum in sample 1 indicates that the frames of the mural paintings are made entirely of gypsum.

The ground layer consists of zincate mixed with gypsum in the ratio 2:1.

The colors used in the painting were common in that period. The red color is hematite, the yellow color is litharge, the green color is malachite, the blue color is wollastonite, the golden color is gold oxide and the black color is magnetite. The reddish brown consists of hematite mixed with azurite in the ratio of 3:1 and the light green color is a mixture of litharge with malachite and calcite in the ratio of 3:2:1 (Figure 4).



Figure 3. Optical microscopy shows structure of paintings: (A) crystals of the ground are large and seem to be homogenous; (B) red color mixed with a percentage of fine black color and there are cracks on the paint layer; (C) blue color has a powdery consistency; (D) flecks of white and black appear in the deep green color; (E) the layers of the deep green color; (F) the irregularly applied golden color.

Halite constitutes 21% of the ground layer, 19% of the painting layer and 16% of the deep green color sample. The presence of salts determines a major deterioration factor [4] as the growth of salts can cause interior pressure which leads to detachment of the paint layer in mural paintings [5].

Compounds %	1s	2s	3s	4s	5s	6s	7s	8s
Gypsum	100	61.3			16.5		20.1	37
Calcite					15	16		
Silica		9.5				4		6.5
Malachite					15	24		
Halite		21	19		16			
Albite		6						
Zincate			35.5	35	28.5		55	
Hydrocrossite			17	14				
Azurite			6.5					
Litharge				17.5		47.5		
Hematite				14			14.9	10
Magnetite							45.8	10.8
Wollastonite				11.8				
Anhydrite						8		9
Gold oxide								21

Table 1.	The	XRD	analysis	results	of	eight	samples	from	the	mural	paintings	at	ΕI	Sakakeny
Palace.														



Figure 4. (a) X-Ray diffraction patterns of the frame sample show results that indicate it consists of gypsum; (b) the original ground layer consists of calcite and quartz and a small percentage of zincate and halite; (c) X-Ray diffraction patterns of the reddish-brown colored sample show it is magnetite with a small percentage of hematite; (d) the green blue color is wollastonite; (e) X-Ray diffraction patterns of the light green sample show it is malachite; (f) X-Ray diffraction patterns of the light green sample show it consists of malachite with litharge; (g) X-Ray diffraction patterns of the red color show it consists of hematite and a mixture of magnetite with zinc oxide; (h) X-Ray diffraction patterns of the golden color sample show it consists of gold oxide.

3.3. Scanning electron microscope coupled with EDX (SEM-EDX)

The EDX patterns of the mural paintings from El Sakakeny Palace indicate the following results, resumed in Figure 5-10. Observations, made by SEM, of the deteriorated mural painting samples, show the presence of halite crystals (the salt was identified by XRD) on the surface and in depth, as well as calcite crystals homogenously connected to large-sized quartz crystals.

Sample	Elements %											
	Ti	Р	Si	S	Са	К	Fe	CI	Zn	Cu		
1s	0.01	0.53	17.38	1.31	39.76	0.27	1.01	0.42	0.25	0.18		
2s	0.20		5.21	15.82	2.84	0.96	1.56	2.84	2.62	3.05		
3s	0.19		0.51	18.34	34.68	0.67	0.46	1.16	1.12	0.21		
4S	0.80		2.21	14.64	28.29	3.20	0.81	5.65	5.22	0.50		
5s	0.62		14.50	12.16	20.26	2.07	0.75	4.46	0.56	0.42		
6s	1.47		1.47	20.96	25.18	0.49	0.47	2.44	2.50	0.01		

Table 2. Results of X-ray Fluorescence analysis of six samples from paintings at El Sakakeny Palace, confirming results of the X-Ray diffraction analysis.



Figure 5. On left Sample A taken from ground layer showing large quartz crystals which evidence the homogeneity of the mortar and on right EDX image showing the elements, the major elements being Ca, Si, S.



Figure 6. On left Sample B taken from reddish brown color showing the large quartz crystals, the homogeneity of the paint layer and on right EDX image showing the elements and the major elements being Ca, Si, S.



Figure 7. On the left Sample C taken from ground layer showing large quartz crystals connected with gypsum which evidence the homogeneity of the mortar and on the right EDX image showing elements in the samples, the major elements being Ca, S, Cl and Cu.



Figure 8. On left Sample D taken from green blue color showing large crystals of color consisting of wollastonite (after XRD analysis) and on right EDX image showing elements in the samples, the major elements being Ca, S, Cl and Cu.



Figure 9. On left Sample E taken from deep green color showing large color crystals which evidence the homogeneity of the paint layer and consisting of malachite and hematite and on right EDX image showing elements in the samples, the major elements being Ca, S, Cl and Cu.



Figure 10. On left Sample F taken from light green color showing large crystals of colors which evidence the homogeneity of the paint layer and on right EDX image showing elements in the samples, the major elements being Ca, S, Cl and Cu.

3.4. Fourier Transform Infrared – Attenuated Total Reflectance (FTIR-ATR)

The samples were placed inside a spectrophotometer to obtain the analyses as spectra on graph paper, [6] OH stretching 3545.49 cm⁻¹, asymmetric C-H stretching of aliphatic group 2924.52 cm⁻¹, 2853.17 cm⁻¹, overtones 2362.37 cm⁻¹ and C-O stretching 1140.69cm⁻¹. Readings show that the medium used in the painting is linseed oil, though the gold color is applied using the medium of animal glue [7].



Figure 11. (a) The medium in the red color shows asymmetric OH stretching absorption at 3545.49 cm⁻¹, OH bending at 1317 cm⁻¹ and C-O stretching at 1140.69 cm⁻¹: readings proved that the binding medium used in the painting is linseed oil; (b) the medium in the wollastonite color shows asymmetric OH stretching absorption at 3545.49 cm⁻¹, OH bending at 1317 cm⁻¹ and C-O stretching at 1140.69 cm⁻¹: readings proved that the binding medium used in the painting is linseed oil; (c) the medium ised in the readish brown color shows asymmetric OH stretching absorption at 3545.49 cm⁻¹, OH bending at 1317 cm⁻¹ and C-O stretching at 1140.69 cm⁻¹: readings proved that the binding medium used in the readish brown color shows asymmetric OH stretching absorption at 3545.49 cm⁻¹, OH bending at 1317 cm⁻¹ and C-O stretching at 1140.69 cm⁻¹: readings proved that the binding medium used in the painting is linseed oil; (d) the medium in the deep green color shows asymmetric OH stretching absorption at 3545.49 cm⁻¹, OH bending at 1317 cm⁻¹ and C-O stretching at 1140.69 cm⁻¹: readings proved that the binding medium used in the painting is linseed oil; (e) the medium in the gold color shows absorption of NH stretching at 3545.49 cm⁻¹ and C-O stretching at 1622 cm⁻¹: readings proved that the binding medium used in the gold color is animal glue; (f) the medium in the ground layer shows absorption of NH stretching at 3545.49 cm⁻¹, and C=O stretching at 1622 cm⁻¹: readings proved that the binding medium used in the gold color is animal glue; (f) the medium in the ground layer shows absorption of NH stretching at 3545.49 cm⁻¹, and C=O stretching at 1622 cm⁻¹: readings proved that the binding medium used in the gold color is animal glue; (f) the medium in the ground layer shows absorption of NH stretching at 3545.49 cm⁻¹, and C=O stretching at 1622 cm⁻¹: readings proved that the binding medium used in the gold color is animal glue; (f) the medium in the ground layer shows absorption of NH stretch

4. Conclusion

This paper presents the initial stages for restoration of three mural paintings in El Sakakeny Palace. The study focused mainly on the physical and chemical properties of the painting compounds and the main changes that occurred in the painting materials, which caused deterioration and detachment of parts of the paintings. This allowed us to find the best solution for their restoration.

Based on the analysis and testing results, below are the findings and conclusion of the research project:

- 1) The ground layer of the paintings consists of zincate with gypsum mixed together in the ratio of 3:2.
- 2) The medium used in the ground layer is animal glue.
- 3) The main chemical component of the frame is only pure gypsum.
- 4) The main reason for deterioration and detachment of the painting layers is the halite which is considered to be one of the major factors of deterioration.
- 5) The colors used in the paintings are:
 - red, which is hematite;
 - yellow, which is litharge;
 - green, which is malachite;
 - black, which is magnetite;
 - blue, which is wollastonite;
 - the mixture of reddish brown consists of hematite and azurite mixed together in the ratio of 3:1;
 - and the light green color consists of litharge, malachite and calcite mixed together in the ratio of 3:2:1.
- 6) All Italian artists in that period mixed their colors with linseed oil to make them bright and shiny, but in the golden color the medium is animal glue.

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