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ANALYTICAL INVESTIGATION OF MATERIALS USED IN THE CONSTRUCTION OF ISLAMIC MOSAICS IN AL SULTAN EL MANSOUR KALAOUN SCHOOL, CAIRO, EGYPT

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ABSTRACT

Egyptian Islamic Buildings are suffering from a lot of deterioration types, mainly groundwater and salt weathering which have caused the complete loss of the decorations of some of these mihrabs. Many mihrabs in Egyptian Islamic buildings need restoration and conservation, as Islamic buildings are one of the most famous historical places in Egypt and over the world finding solution for this problem become very urgent. A physicochemical study using analytical techniques such as Fourier transform infrared, X-ray diffraction and scanning electron microscopy was done. This characterization study, on one hand helping us to reach for the optimum conservation and completion methods; on the other hand, the deterioration factors of mosaic were determined. All analytical methods indicated that the Al- Mansour kalaoun mosaic mihrab contains lime, Gypsum, Dolomite, Quartz, Calcite and Aragonite. It also proves that sodium chloride (halite) is the principal salt causing deterioration.

KEYWORDS: Mosaic, Mihrab, Dolomite, Calcite, Deterioration, Construction, Analytical, Conservation.

1. INTRODUCTION

EL Mansour Qalawun group was constructed in (684 AH -1285 AD), Prince "Aladdin Aq Sonkor" bought Qalawun a Mamluk Turks with a thousand dinars so he called him (El- Alfy) and after prince death his ownership became for the king El-Saleh Najm al-Din and he called him (El- Saly El-Najme), then he continue promotion from position to anther till he became a great prince. And he became a king in (678 AH - 1279 AD) with a lot of power and he continue for eleven years till his death in (689 AH -1290AD).

EL Mansour Qalawun constructed a mosque carrying his name attached to Bimaristan (hospital) and school teaching Qur'an science, and the dome of the king shrine was the most wonderful thing built in the Mamluk era, Construction of the mosque began in Rabee El-Akhar 683 AH and construction finished after only fourteen months. (Fig. 1.a, b, c)

The mihrab located within this group is made of marble and shell, the Mihrab and its dome where decorated with golden mosaic which made this Mihrab to be considered as the most beautiful Mihrab in Egypt. (Fig. 2)

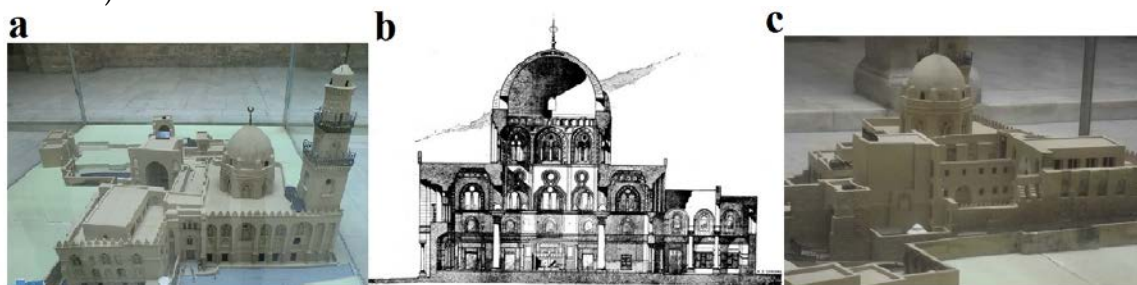


Figure 1. a) Front view for Qalawun group; b) Longitudinal sector of the school of Qalawun. c) Backside view for Qalawun group.

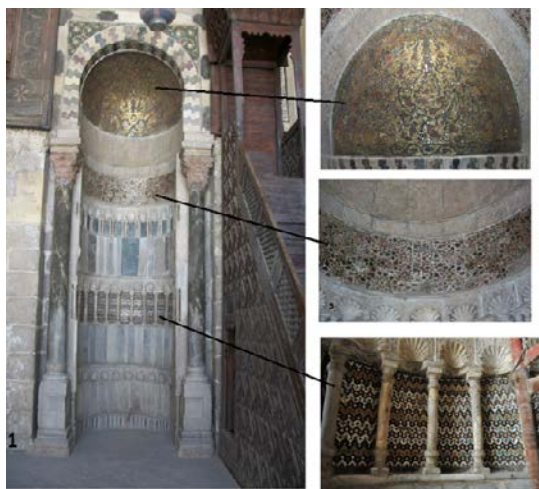


Figure 2. Mihrab of Qalawun decorated with marble and shell mosaics, the tomb decorated with golden mosaic

The mihrab was deteriorating due to some factors, mainly groundwater and salt weathering, which has caused the loss of some parts of the mosaic motives, as well as depositing thick layers of salt, soluble salts are considered to be among the most important causes of decay. Salts cause damage by the growth of salt crystals within the pores, which can generate

stresses that are sufficient to overcome the tensile strength of the material and turn it to powder (Doehne and Price, 2010).

These soluble salts are very harmful to building materials as they have the ability to exist in different states of hydration. The crystallization of these salts within the pores takes place if the salinesolution is between the state of saturation and super saturation. Salts such as sodium chloride and calcium sulphates (CaSO_4 and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) cause serious damage to building material because the change from one hydrated phase to another is commonly associated with a volume change which in turns gives rise to forces on the walls of the individual pores. (Mona & Abdullah, 2014).

The most important reasons of the damage which mihrab is suffering was Buckling due to the presence of groundwater and dissolved salts in addition to the temperature difference and this reflected in the separation of mosaic cubes and cause some parts to fall down. Figure (3.a, b, c).

On the other hand, the mihrab was suffering from growth of crystals salts, which present on the surface of the mosaic due to the high rela-

tive humidity which leading to dissolve the salts which found mainly in the composition of the mosaic cubes of marble and shell and at higher temperatures this salts are dehydration and crystallized, and we found that salts in the mosaic mihrab crystallized firstly between the cup of mosaics and also between the mosaic layer and gypsum mortar and also between mortar and lime stone.

One of the another damage the mihrab suffered was the glue that connects between the mosaic cubes, we did X.R.F analysis and we found Qlfona, and this Qlfona was found in fragile to the touch condition, which led to the fall down of many units of the mosaic during the process of restoration, also during the cleaning of mosaic unites Qlfona was found in a brittle condition.

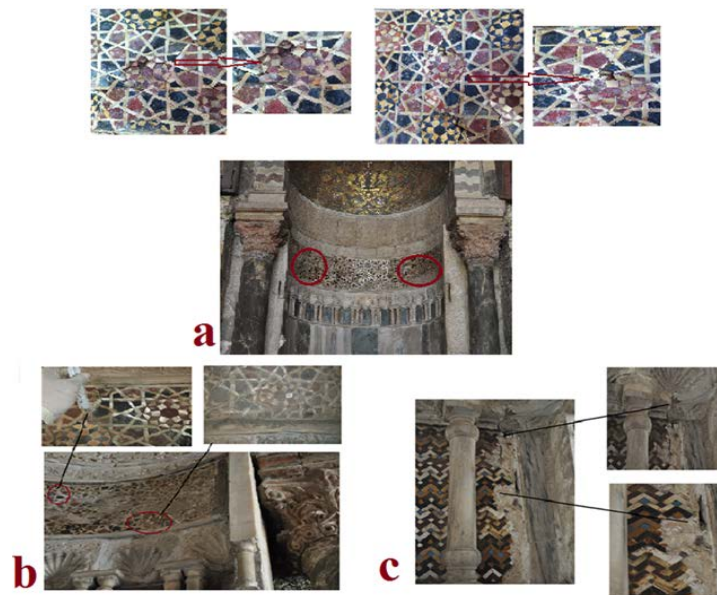
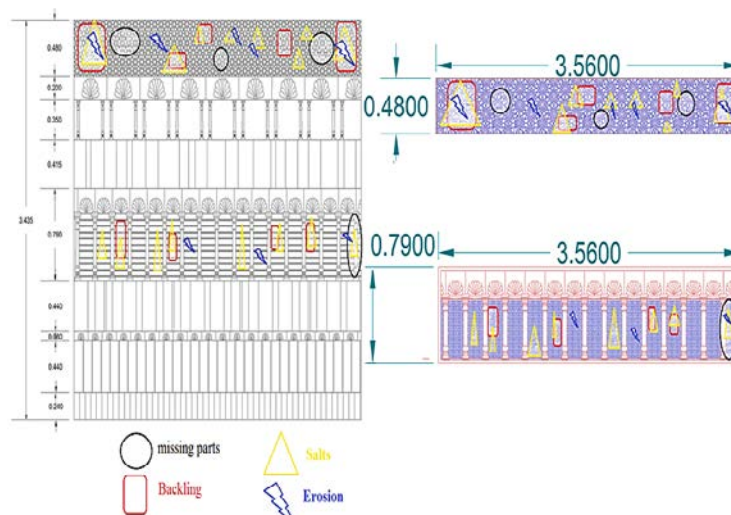


Figure 3, a) showing mosaic curvature which due to erosion of the mortar caused by ground water effects, b) Showing mosaic buckling due to growth of salt behind the mosaic cups, c) Showing fall down of mosaic cups due to different level of high relative humidity and soluble salt which came from ground water.



Auto cad drawing for the Mihrab with scale 1:100 showing deterioration positions.

MATERIALS AND METHODS

Sampling

Fifteen representative samples were collected carefully, using a micro-scalpel, to identify the

constituents and degree of deterioration of the mosaic. All the analysed and investigated samples were carefully collected from areas without aesthetic value or from severely damaged parts.

- **Polarizing microscopy (P.M)**

Mosaic has been studied samples for microscopic examination of the polarizer, as this depends on the light microscope into force on the sample and reflected from the sample surface and given optical properties of the metal distinctive him. So that samples can be tested by any polarized light vibrates in one direction and it gives more accurate than ordinary light details.

The samples were examined polarized microscopy to identify the samples refraction coefficient, shape and colour of the crystals, as well as internal disconnect and colour change. In addition, it helps to identify the aspects of damage within the samples from the minute cracks and corrosion.

- **X-ray powder diffraction (XRPD)**

The X-ray powder diffraction patterns of the mosaic cup powders were obtained using a diffract meter (Philips PW 1840), operated at 40 kV and 25 mA, using Cu Ka radiation and a receiving slit of 0.2 mm. The 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.

- **Scanning electron microscopy (SEM)**

The microstructure and morphology of mineral constituting of the mosaic material were analysed with a scanning electron microscope FEI Quanta 200. The microscope operated at 30 kV accelerating voltage. Sample preparation consisted of application of a superficial gold film by sputtering to prevent electrostatic charge.

- **X-ray Fluorescence (XRF)**

Five mosaic samples were analysed by XRF to determine the chemical composition of the stucco samples. The apparatus used is a Philips PW1400. The investigated samples were prepared in the form of pellets, using a manual press under a 20 tons load, and were analysed using an Rh-Ka (rhodium) radiation tube at 50 KV and 50 mA. The chlorine ion was analysed by potentiometer titration. The loss on ignition (LOI) of the samples was determined by igniting 1g of the sample in a weighted platinum crucible heated to 950°C in an electric furnace for 30 minutes (first time) and then for 15 minutes (second time). The crucible and samples were

cooled for 10 minutes before each weighting, performed after heating and weighting were repeated until constancy of weight was observed.

- **Fourier transform infrared Spectroscopy (FTIR)**

IR spectra were obtained using a FT-IR Thermos Nicolet 760. The resolution is of: 4 cm⁻¹ (Region 4000: 400 / Absolute threshold 0.002 /Sensitivity: 50). The sample preparation process consisted of grinding the sample to obtain fine Marble, shell and mortar powder which was then mixed with KBr powder, with a sample/KBr ratio of 1:15.

RESULTS AND DISCUSSION

- **Polarizing microscopy (P.M)**

After examining mosaic samples of EL-Sultan EL-Mansur Qalawun School Mihrab with polarizing microscopy the petrographic investigation of the samples revealed that the samples of reddish colour composed mainly of fine to medium-grained of Quartz, Feldspars and Iron oxides embedded in very fine-grained groundmass of clay materials admixed with considerable amounts of Iron oxides giving the reddish colour of the sample (Fig. 4.a, b, c, d).

Quartz occurs as fine-grains, well-sorted and of angular to sub angular outlines usually monocrystalline except few grains are polycrystalline with sutured edges and display wavy extinction. Feldspars are represented mainly by plagioclase and Orthoclase which occur as medium grains of elongated, euhedral to subeuhedral crystals. Most of which are strongly altered to clay materials and carbonates. Iron oxides occur as fine sub rounded grains disseminated in the ground mass.

The samples of light grey to dark grey colour composed mainly of carbonates and clay material. They occur as very fine grained interlocked and compact with each other's and mostly masked by clay material.

Tresses of opaque are also observed disseminated in the carbonates.

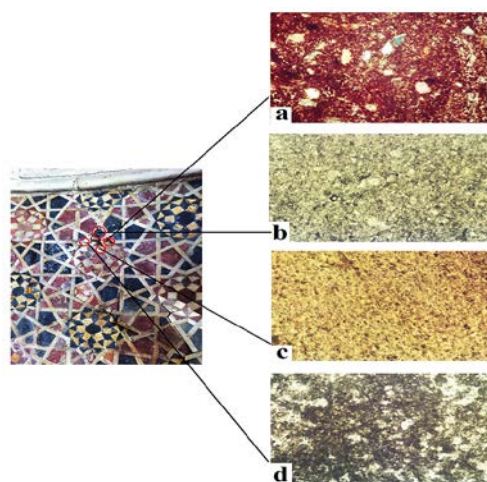


Figure 4. a) a sample of red marble from the mosaic mihrab, quartz grains with sharp angles with a longitudinal feldspar crystals (power magnification X-63PPL) b) Sample of black marble taken from the mosaic mihrab show very finely divided carbonate (power magnification X-63C.N), C) sample of shell explain compact and overlapping micro- carbon granules with each other (power magnification X-63C.N). d) a sample of mortar show

amalgamating carbonates with each other (power magnification X-63PPL).

• *X-ray powder diffraction (XRPD)*

The XRPD patterns of the mosaic samples from El Mansour Kalawun mosaic mihrab indicate the following results which are resumed in Table 1.

From the XRPD analysis the results discovered were as following:

1-Existence of a high percentage of sodium salt (Halite) in all mosaic samples (marble, shell, mortar and limestone), the existence was directly behind the mosaic layer between the mosaic and mortar also between the mortar and limestone as well as the surface of the mosaic tresses, and this due to

a. NaCl was found as part of the mosaic materials.

b. Soil contain a lot of ground water as well as sewage and this sewage contain NaCl and nitrate.

Table 1. The approximate XRD analysis results of five mosaic samples from KALAWUN mosaic mihrab.

| Samples | a (red marble) | b (black marble) | c (shell) | d (mortar: 3mm depth) | e (mortar between 1-2cm depth) |
|----------------------------|----------------|------------------|-----------|-----------------------|--------------------------------|
| Calcite | 22.50% | 48.50% | 35% | 19.50% | 0 |
| Dolomite | 51% | 32.50% | 14.50% | 0 | 0 |
| Aragonite | 0 | 0 | 40% | 0 | 0 |
| Quartz | 0 | 11.50% | 0 | 30% | 28.50% |
| Halite | 15% | 7.50% | 7% | 0 | 12% |
| Hematite | 11.50% | 0 | 0 | 0 | 0 |
| Gypsum | 0 | 0 | 0 | 50.50% | 46% |
| Kaolinite | 0 | 0 | 0 | 0 | 3.50% |
| Sodium Carbonate Phosphate | 0 | 0 | 3.50% | 0 | 0 |
| Gypsum dehydrate | 0 | 0 | 0 | 0 | 10% |

2. Dolomite $\text{CaMg}(\text{CO}_3)_2$ was found as one of the main component of marble samples with a percentage of (51%), also calcite CaCO_3 was found by 22.5% and 48.5% in red marble and black marble respectively, also the main reason of the red colour in red marble mainly due to Hematite (Figs 5,6).

3. Aragonite was found as the main component of shell sample not Calcite as usual, which shows transformation for the crystalline form of calcite the main usual component of shell and this is may be due to

The high temperature for a long period of time, beside the aragonite also Dolomite, Sodium salt and Phosphate was found as well and this due to the existence of shell in sea water for a long period Figure (7).

4. CaCO_3 was found in the wall of Mihrab which built from limestone, Also CaCO_3 was found in mortar sample as part of limestone powder as a filler which used along with Gypsum in the mortar (this was the famous mortar in the Islamic period) (Figs 8, 9).

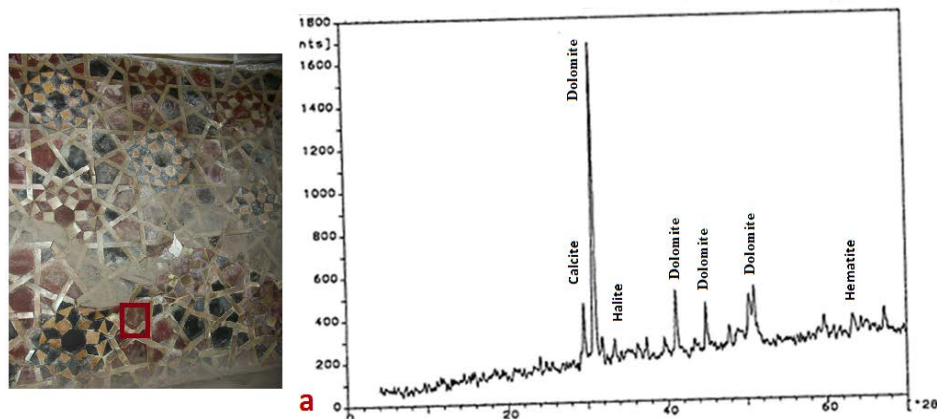


Figure 5. X-Ray diffraction patterns of archaeological red marble sample from Kalawon mosaic mihrab used to Reconstruct the archaeological mosaic material, the results proved that the red marble consists of dolomite, calcite, halite, and hematite.

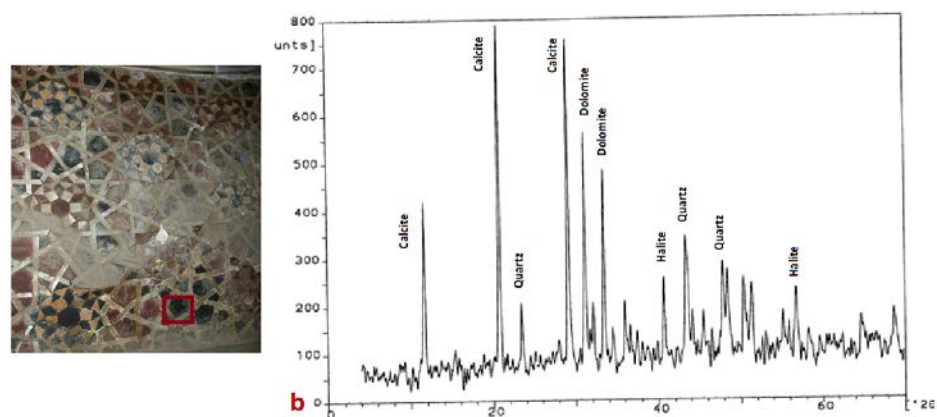


Figure 6. X-Ray diffraction patterns of archaeological black marble sample from Kalawon mosaic mihrab used to reconstruct the archaeological mosaic material, the results proved that the black marble consists of calcite, dolomite, Quartz and halite.

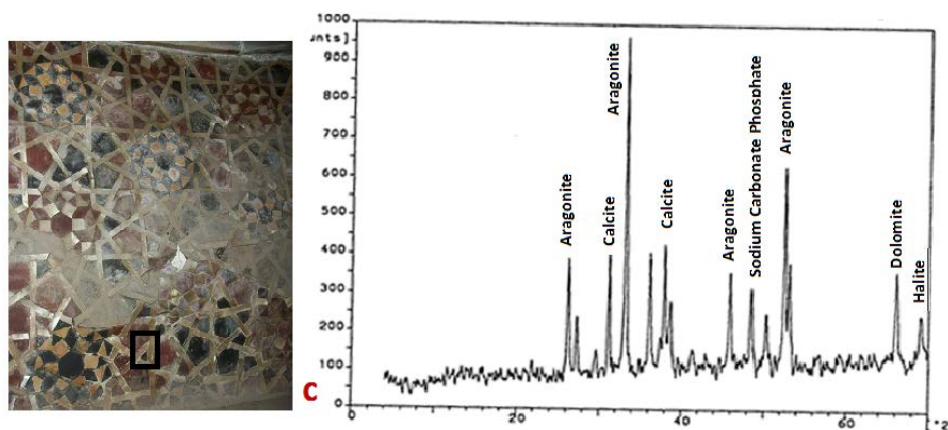


Figure 7. X-Ray diffraction patterns of archaeological shell sample from Kalawon mosaic mihrab used to reconstruct the archaeological mosaic material, the results proved that the shell consists of aragonite, calcite, dolomite, sodium carbonate phosphate and halite.

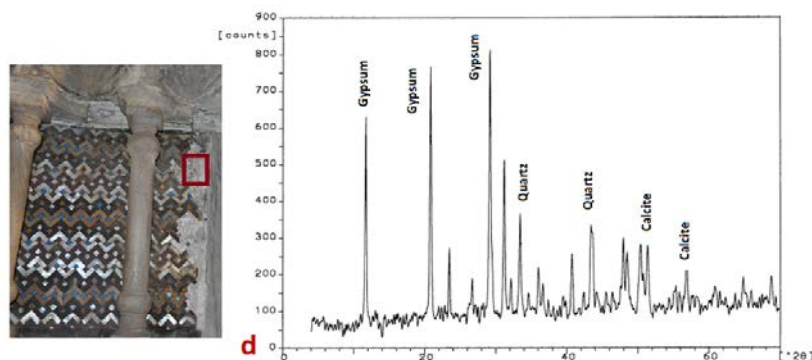


Figure 8. X-Ray diffraction patterns of archaeological gypsum mortar sample from Kalawon mosaic mihrab used to reconstruct the archaeological mosaic material, the results proved that the mortar 3mm consists of gypsum, calcite and Quartz.

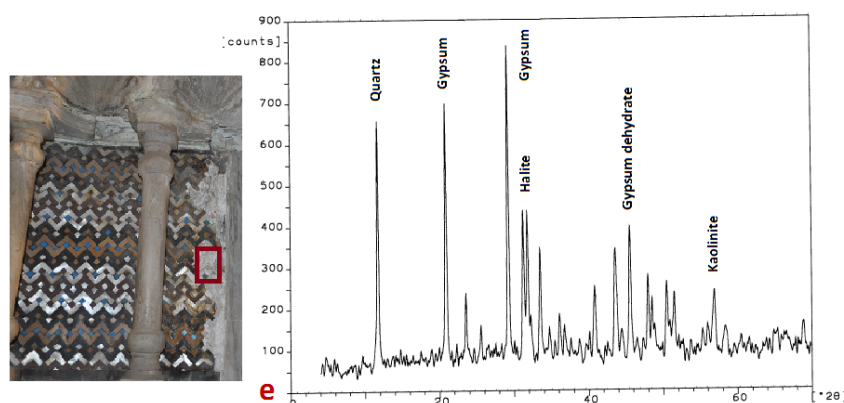


Figure 9. X-Ray diffraction patterns of archaeological gypsum mortar sample from Kalawon mosaic mihrab used to reconstruct the archaeological mosaic material, the results proved that the mortar 1-2cm consists of gypsum, calcite, halite, kaolinite, gypsum dehydrate and Quartz.

- *Scanning electron microscopy (SEM)*

The EDX patterns of the mosaic samples from El Mansour Kalawun mosaic mihrab indicate the following results which are resumed in Table 2.

The observations made by SEM of deteriorated mosaic samples from the mihrab show the appearance of halite crystals (the salt was identified by XRD) on both surface and depth, as

well as the severe disintegration of the surface which may be related to the transportation of some mineralogical components by the effect of sodium chloride solutions. Calcite crystals appear as an irregularly result of the impact of groundwater which bearing high proportion of salts and acids.

Table2. The results of X-ray Fluorescence analysis of five mosaic samples from Kalawon mosaic mihrab. The results are confirming the results of X-Ray diffraction analysis.

| Samples | a (red marble) | b (black marble) | c (shell) | d (mortar: 3mm depth) | e (lime stone) |
|---------|----------------|------------------|-----------|-----------------------|----------------|
| Ca | 64% | 83% | 76% | 65% | 40% |
| S | 14% | 12.50% | 0 | 17% | 45% |
| Si | 7% | 3.20% | 0 | 0 | 4.50% |
| AL | 0% | 0 | 0 | 0 | 5% |
| CL | 4.50% | 0 | 0 | 5.50% | 3.50% |
| Fe | 11% | 1,3% | 4.50% | 1.50% | 1.50% |
| Mg | 0 | 0 | 2.50% | 8.50% | 0 |
| K | 0 | 0 | 3% | 2.50% | 0.50% |
| Zn | 0 | 0 | 4% | 0 | 0 |
| Na | 0 | 0 | 10% | 0 | 0 |

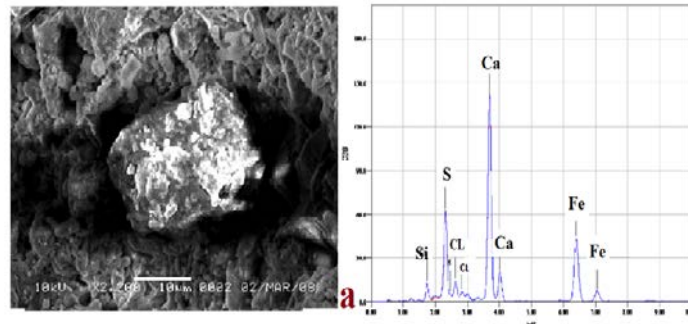


Figure (10, sample/a) sample taken from the mihrab and shows the dolomite crystals Rhomboid shape added to the growth of crystals of iron and sulphur.

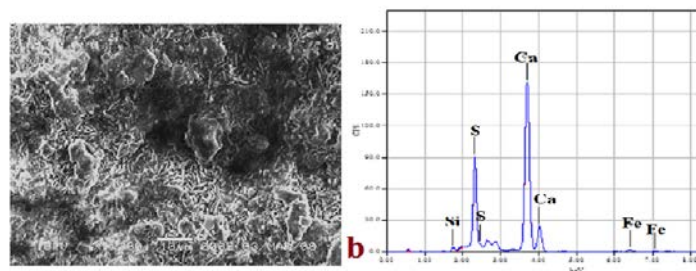


Figure (11, sample/b) Clear erosion of the calcite crystals and Salt crystals growth between calcite crystals (2.200X).

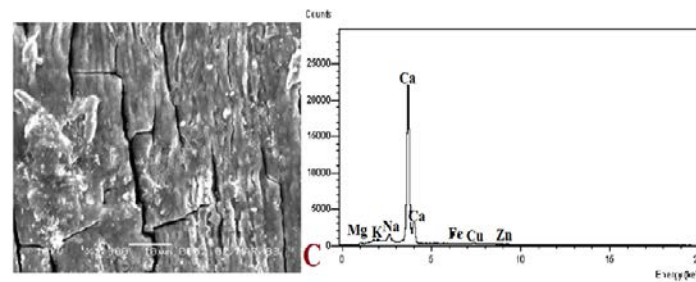


Figure (12, sample/c) Shows the structure of seashell from the inside in the form of a monolithic layers, as the gaps between the layers, which may be erosion due to the presence of salts in the presence of moisture led to dissolve the salts causing these gaps (2.200X).

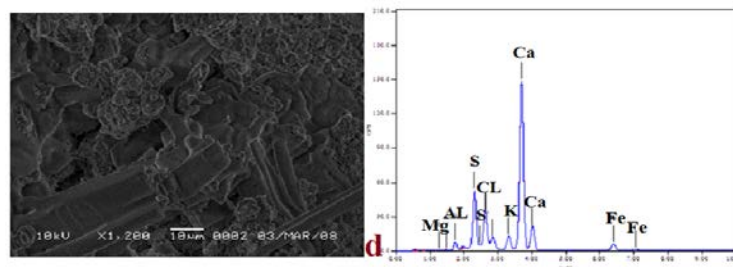


Figure (13, sample/d) shows spread of crystallized salts between quartz crystals and gypsum where hardly apparent by the proliferation of salts (1.200X).

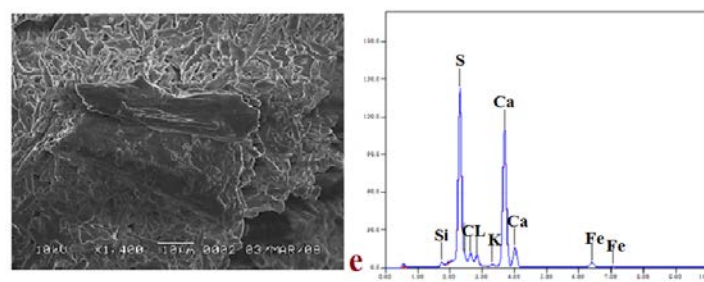


Figure (14, sample/e) shows Calcite crystals irregularly as a result of the impact of water ground a bearing high proportion of salts and acids (1.400X).

•Fourier transform infrared Spectroscopy (FTIR)

Where one sample was taken from the adhesive behind the mosaic cubes from niche Kalawon to see composition, as well as some other known adhesives samples were tested and Mihrab sample result showed the same standard composition for one of them.

The sample preparation was done by mixing powder of sample with potassium bromide and then the sample was compressed inside the piston to get round disk shape with diameter about 1cm free from air and water completely.

Then the sample was placed inside a spectrophotometric analysis, the device is running to give the analysis as a chart graph paper, the same was done for the other standard samples which are:

1. Standard sample (taken from the mihrab)
2. Qlfonah.
3. Wax.
4. Arabic gum.

After the scan and comparing all the results we conclude that the archaeological standard sample is Qlfonah, and this is clearly seen in the results. Figure 15.

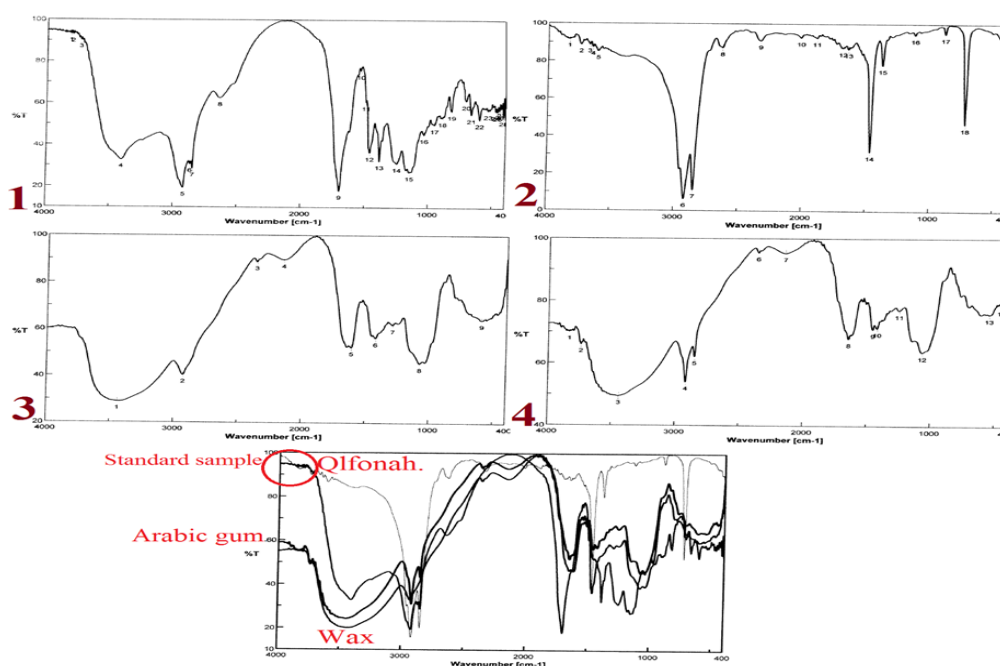


Figure 15. Infrared spectra of four adhesives samples from the mihrab confirmed the results of XRD and XRF analyses.

CONCLUSION

This paper is presenting the initial stages for restoration project of El Sultan El Mansour Kalawon school mosaic mihrab, this study focused mainly on the physical and chemical properties of the mosaic compound and the main changes happened for the mosaic material which caused deterioration and fall down of the mosaic parts, so we can find the best and optimum restoration ways for this project.

Biased on the analysis and testing results below are the findings and conclusion:

1-The mosaic Mihrab was mainly contain marble, shell, gypsum and lime stone.

2-The main chemical components of the mosaic are Calcite, Dolomite, Aragonite, Quartz, Halite and Hematite.

3-The main reason for deterioration and fall down of the mosaic cup was the groundwater which rich of sodium chloride salts which was the main reason of its crystal erosion.

4-This conclusion shows that elimination of the source of salts is very urgent on one hand, and water (in a poultice) is the acceptable method for extracting the soluble salt (halite), on the other hand.

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REFERENCES

- Boschetti C, Leonelli C, Macchiarola M, Veronesi P, Corradi A, Sada C (2008) Early evidences of vitreous materials in Roman mosaics from Italy: An archaeological and archaeometric integrated study. *Journal of Cultural Heritage* 9 21-26
- Abdullah M., Mona F. and Hala A. (2014) Chemical and physical characterization of the stucco mihrab of the mausoleum of Muhammad Al-Hasawati, Fatimid period, Cairo, Egypt. *Periodico di Mineralogy*, 83, 3, 313-327
- Shortland A.J., M.S. (2005) Technological study of Ptolemaic-early Roman faience from Memphis, Egypt, *Archaeometry* 47 (1) 31- 46.
- British Standards Institution. (2001) Building Lime – Definition, Specifications and Conformity Criteria. EN 459-1. London: BSI.
- Boschetti C, Corradi A, Leonelli C, Veronesi P, Santoro S, (2007) IL corpus dei mosaici alessandrini in Italia. UN progetto tra archeologia e archeometria, in: C. Angelelli, F. Rinaldi (Eds.), *Proceedings AISCOM 12th meeting*, Padova-Brescia, February 14-17 2006, Scripta Manent, Tivoli, pp. 547-554.
- Boschetti C, Corradi A, Baraldi P, Raman characterization of painted mortar in Republican Roman mosaics, *Journal of Raman Spectroscopy*, 39 (8) (2008) Special issue. In: Baraldi P, Tinti A, editors. *Raman spectroscopy in art and archaeology*, 1085-1090.
- Doehne E. and Price C.A. (2010) *Stone Conservation, an Overview of Current Research*, Getty Conservation Institute, U.S.A. 158 pp.
- Elert K, Rodriguez C, Pardo E.S, Hansen E. and Cazalla O. (2002) Lime Mortars for the Conservation of Historic Buildings. *Studies in Conservation*, 47, 62-75.
- Elert K., Rodriguez C., Pardo E.S., Hansen E. and Cazalla O. (2002) Lime Mortars for the Conservation of Historic Buildings. *Studies in Conservation*, 47, 62-75.
- Branda F, Costantini A, Luciani G, Piccioli C. (2000), A Note on Incrustations on a Mosaic Floor Fragment from Punta Epitaffio in Baia. *Studies in Conservation*, Vol. 45, No. 3 pp. 211-213
- Galli S, Mastelloni M, Ponterio R, Sabatino G, and Triscari M. (2004). Raman and scanning electron microscopy and energy dispersive X-ray techniques for the characterization of colouring and opaquening agents in Roman mosaic glass tesserae. *Journal of Raman Spectroscopy* 35: 622-27.
- Getty Conservation Institute and Israel Antiquities Authority. (2003). *Illustrated Glossary: Mosaic in Situ Project: Definitions of Terms Used for the Graphic Documentation of in Situ Floor Mosaics*. Los Angeles, Calif.: Getty Conservation Institute. Available at www.getty.edu/conservation/publications/pdf_publicationsmosaicglossary.pdf.
- Leslie K, Freestone I, Lowry D, Thirlwall M. (2006) the provenance and technology of Near Eastern glass: oxygen isotopes by laser fluorination as a complement to strontium, *Archaeometry* 48 (2) 253-270.
- Lucas A. and Harris J. R. 1962–99. *Ancient Egyptian Materials and Industries*. London: Dover Publications.
- Menicou, M., C. Fiori, and M. Macchiarola. (2003) Examples of deterioration following preservation works on mosaics in situ. In *Mosaics Make a Site: The Conservation in Situ of Mosaics on Archaeological Sites: Proceedings of the VIth Conference of the International Committee for the Conservation of Mosaics, Nicosia, Cyprus, 1996*, ed. D. Michaelides, Rome: ICCM, 225–33.

- Michalski, S. (1993) Relative humidity: A discussion of correct/ incorrect values. In ICOM Committee for Conservation 10th Triennial Meeting: Washington, DC, 22-27 August 2003: Preprints, ed. J. Bridgland, Washington, D.C.: *ICOM Committee for Conservation*, 624-29.
- Mao Y. Lead-alkaline glazed Egyptian faience (2000) Preliminary technical investigation of Ptolemaic period faience vessels in the collection of the Walters Art Gallery, *JAIC* 32 (2) 185-204.