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Degradation Processes of Egyptian Faience Tiles in the Step Pyramid at Saqqara

Fatma S. Madkour[a], Mohamed K. Khallaf[b*]

[a]*Conservation Dept. Faculty of Fine Arts, Minia University, Minia, 61519, Egypt*
[b]*Conservation Dept. Faculty of Archaeology, Fayoum University, Fayoum,63521 , Egypt*

Abstract

The tomb of King Djoser (2667-2648 B.C) at Saqqara known as the Step Pyramid was the first Egyptian pyramid tomb and the earliest stone building of its size in the world. Some of the walls of the substructure of the Step Pyramid and the so-called "Southern Tomb" were covered with panels of blue-green faience tiles. The present work aims to study causes of decay the faience tiles which coated Southern Tomb of Step Pyramid. Investigations and analytical techniques including scanning electron microscopy combined with energy-dispersive X-ray microanalysis (SEM/EDX), X-ray diffraction (XRD), X-ray florescence (XRF) and polarizing microscopy were used to study the chemical and mineralogical composition of different samples from bodies of faience tiles, stone supported walls and binding mortars.

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Keywords: Degradation; faience; step pyramid; mortars

1. Introduction

Faience, which consists of a ground quartz or quartz sand body coated with a soda-rich glaze, was first produced in both Egypt and the Near East as early as 4th millennium BC. This material was used to produce bowls and tiles as well as small objects such as amulets, beads, rings and scarabs (Tite et al., 2007). In Egypt, the earliest surviving use of Egyptian blue faience tiles was possibly in the painting from tomb 3121 at Saqqara which is dated to the reign of Ka-sen who was the last king of the 1 st Dynasty

* Corresponding author. Tel.: +202 01006781355; fax: +202 0846333178.
E-mail address: m_kamal555@yahoo.com.

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(2900 BC) (Hatton. et al....., 2008). However, the use of Egyptian blue frit in Egypt only became widespread by the 4th Dynasty (2600 BC) and continued through until the Ptolemaic-Roman period (Tite et al....., 2007; Hatton et al....., 2008). Comparison of the microstructures of the ancient faience samples suggest that frit cakes are the primary product and that these were ground to produce the powder, and then moulded to shape and refried to produce the vessels and beads (Hatton et al....., 2008). Egyptian blue frit is a multicomponent material that was produced by firing a mixture of crushed quartz or sand, with small amount of lime, a copper compound and alkali flux (natron or plant ash) to a temperature in the range 850-1000 oC. (Hatton et al....., 2008; La Delfa et al....., 2008). The earliest Egyptian pyramids were step pyramids. (Fig. 1). During the Third dynasty of Egypt, the architect Imhotep built Egypt's first step pyramid, the Pyramid of king Djoser at Saqqara by building a series of six successively smaller mastabas (an earlier form of tomb structure), one on top of another (fig. 2.(a)). Faience tiles were used to cover some of the walls of the substructures of the Step Pyramid and the so-called " South Tomb" (fig. 2.(b)), (fig. 3 a,b). The faience tiles suffer from many deterioration phenomena such as disintegration from stone supported walls, cracks, decay of mortars, crystallization of salts, missing parts from glazed layer and loose and fall of many faience tiles from the walls. The rising damp in the supported walls and crystallization of soluble salts consider the main deterioration factors. The moisture can migration into and within a building in variety ways, depending on whether it is in the liquid or vapor state (Bajare and Svinka, 2000) The transformation of moisture involving variety processes, such as absorption, evaporation, diffusion and capillarity as well as the surface tension of the liquid (Oliver, 1997; Charola, 2000). Failure of faience tiles is most commonly water related where the ceramic units are highly susceptible to glaze cracking, spilling and material loosing (Wilson, 1984). Different analytical methods use to obtain useful information about determination the chemical compositions and microstructures of the faience tiles in order to obtain information about the raw materials used to produce the frit (Hatton. et al....., 2008; Tite et al....., 2009). X-ray diffraction analysis (XRD), X-ray florescence (XRF) and scanning electron microscopy (SEM) coupled with EDX microanalysis were used to study the changes in microstructure and composition that have occurred in faience tiles as a result of weathering, in addition to study the different deterioration aspects.

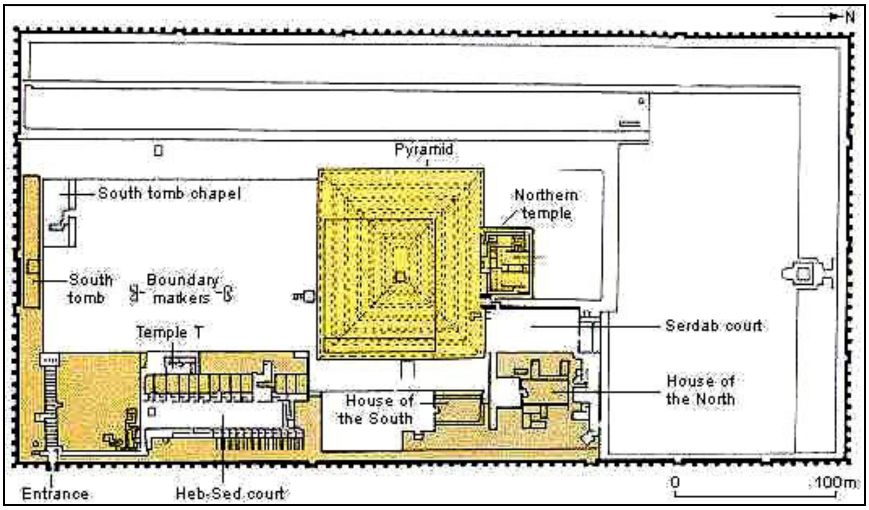


Fig. 1. Shows the complex of King Djoser at Saqqara.

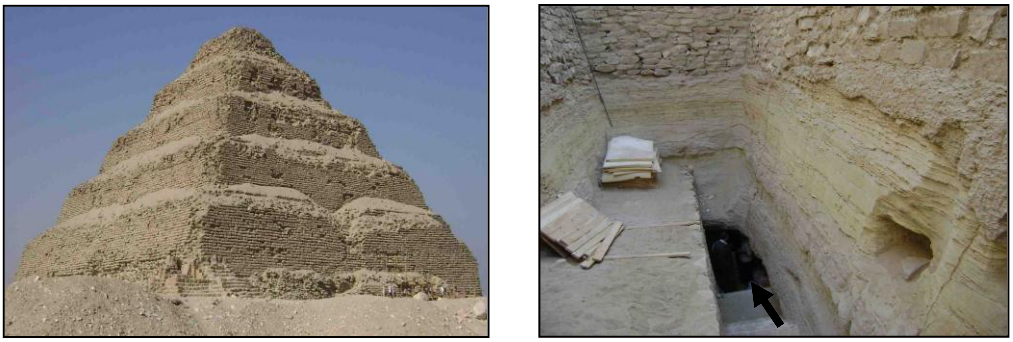


Fig. 2. (a) Shows the step pyramid at Saqqara; (b) Shows entrance of South tomb of King Djoser at Saqqara

Fig. 3. (a); (b) Shows faience tiles cover the walls of the south tomb at step pyramid

2. History of the Step Pyramid and the Faience Tiles

Across the Great Court of the Pyramid Complex of Djoser (2667 - 2648 BC), the second king of the 3rd Dynasty, stands the Step Pyramid. It is believed to have been created by one man, called Imhotep. This complex represents the first major work in stone. That is, unless there are other works that have yet to be found . On the Pyramid, most of the outer casing is gone. In some places the core masonry has disappeared as well. It is obvious there were different stages of construction. (Lehner, 1997). The eastern side gives the best picture, but it can be seen from the northern and southern side as well . The original structure was an underground burial chamber. This chamber was rare in that it was square; most mastabas were rectangular. The royal tomb is 28m underground with a vertical shaft leading to it. The entrance was sealed with 3 pieces of granite stone. The face of the mastaba was a fine Tura limestone. The superstructure of the Step Pyramid is six steps and was built in six stages, as might be expected with an experimental structure. (Siliotti,A., 1997). The South Tomb has been likened to the satellite pyramids of later Dynasties, and has been proposed to house the ka in the afterlife. Current evidence suggests that the South Tomb was finished before the pyramid. The symbolic king's inner palace, decorated in blue faience tiles, is much more complete than that of the pyramid. Three chambers of this substructure are decorated in blue faience tiles to imitate reed-mat facades, just like the pyramid. (Martin. I., 2001). There are differences in dimensions of faience tiles but on average the dimensions of faience tiles are : 10 cm length, 6 cm width and 6 mm thickness.

3. Description of Faience Tiles Degradation

The faience tiles suffer from high degradation including failure and fragile of the binding mortar which used to fix the faience tiles (fig. 4 a, b) The glazed surfaces in some tiles are subjected to high fragile and flaking off resulted in detachment from bodies of tiles, in addition to changing of the faiences colors (fig. 5 a, b) because of high moisture and crystallization of salts in the supported walls (fig. 7). Colored Glazes suffer from crazing and spalling which appears as small blisters on the surfaces in addition to some tiles subjected to cracking (fig. 8). Furthermore failure and fragile of the binding mortar between the tiles. All previous deterioration factors resulted in high moisture in the walls, salt crystallization and finally human negligence of the maintenance these faiences which due to fall of many of them from the supported walls (fig. 9 a, b).

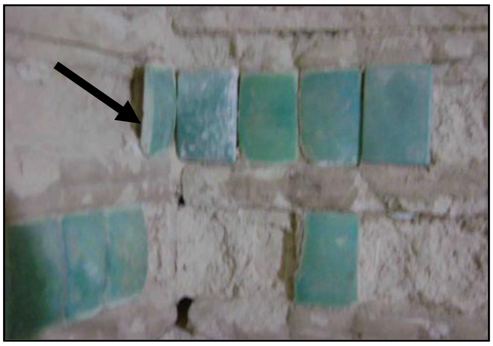



Fig. 4. (a); (b) Shows fragile and flaking of some faience tiles

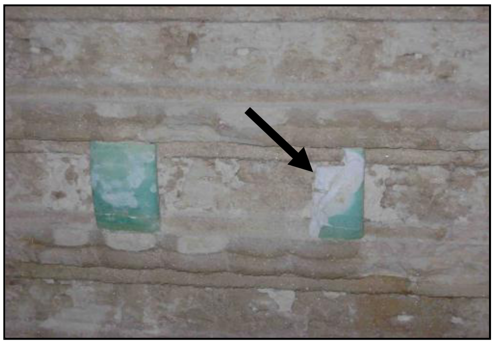
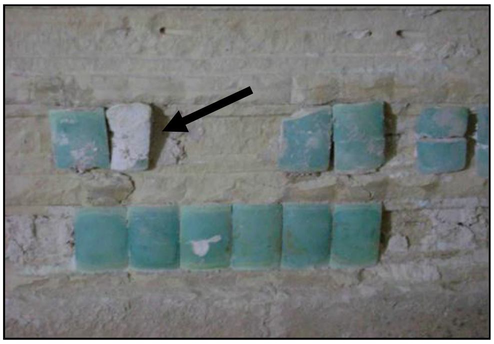



Fig.. 5. (a); (b) Shows missing of glazed layers and cracks in faience tiles

4. Materials and Methods

Different analytical techniques were performed to characterize the faience tiles, mortars and stone walls to study the various types of degradation of these tiles. X-ray diffraction analysis (XRD) was used to identify the mineralogical characterization of mortars and stone walls (Hein et al....., 2004; Drebushchak et al....., 2005). X-ray diffractometer (Philips, PW 1840) with Ni-filtered CuKα radiation at operating conditions of 40 kV/30 mA and a scan speed of 2° (2θ)/min. was used for this purpose. Elemental analysis for the major and trace concentrations for samples of faience tiles were determined by X-ray florescence (Papachristodoulou et al....., 2006) (XRF; S4 XPLORER, Bruker at Department of

Mineralogy and Petrology, University of Granada was used). A representative 5 g powder sample from each locality was pressed into an aluminum holder for disk preparation. ZAF correction was performed systematically. Raw materials which used in the manufacturing and Chemical composition of the faience tiles were determined by using scanning electron microscopy (SEM) (Iordanidis et al....., 2009; Neira et al....., 2009).In addition to different samples of mortars and supported stone walls were examined by using a Variable Pressure Scanning Electron Microscope (VPSEM) Leo 1430VP coupled with EDX microanalysis INCA 350 version 17 Oxford Instrument (Department of Mineralogy and Petrology, University of Granada).

Fig. 6. (a) Shows high moisture and crystallization of salts in the supported walls; (b) Shows crazing and cracking on the surfaces of faience tiles.

Fig. 7. (a); (b) Shows fall of many faience tiles from the supported walls

5. Results and Discussion

5.1. Polarizing Microscope (PLM)

Different samples of faience tiles and limestone supported walls were examined using polarizing microscope. Cross section in faience tiles shows that the components of faience tiles are mixture of crushed quartz or sand which with small amount of lime. The quartz was appeared as angular grains and also we can see the pores and cracks in the body matrix which appear in black color. A copper compound and alkali flux (natron or plant ash) appeared through a blue glazed layer which penetrated through porous bodies of the faience (fig. 8.). Thin sections of limestone supported walls show that limestone consists mainly of fine-grained calcite crystals (biomicrite) besides presence of iron oxides, quartz, clay minerals and fossils. (fig. 9).

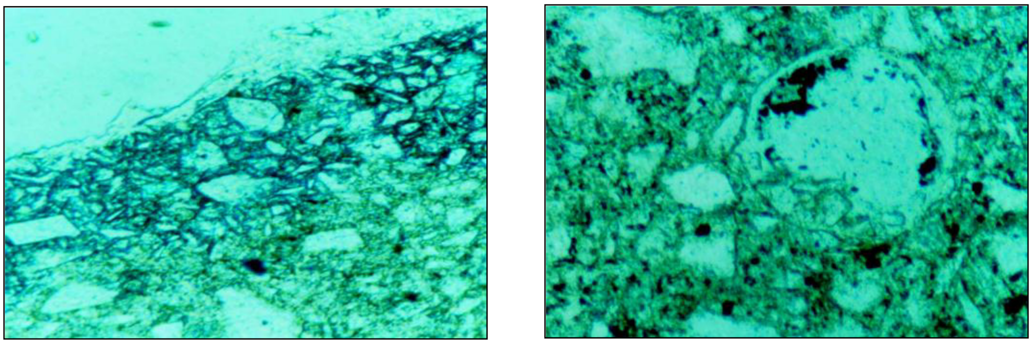


Fig. 8. Cross section photomicrographs showing mixture of crushed quartz which appeared as angular grains and also we can see the pores and cracks in the body matrix which appear in black color

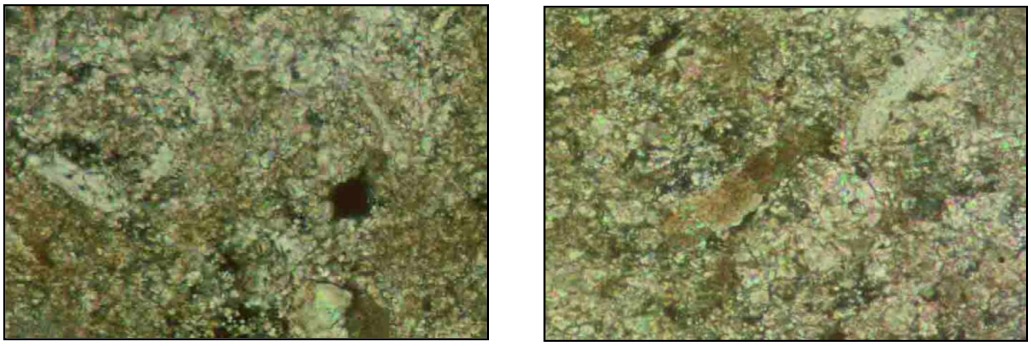


Fig. 9. Thin section photomicrographs showing iron oxides, clay minerals, fossil and grains of quartz in a mass ground of fine- grained calcite. 36X (C.N).

5.2. X-ray Diffraction (XRD)

5.2.1. Bodies of faience tiles

X-ray diffraction results of faience tiles samples shows that, sample consists of quartz mineral and calcite mineral in addition to halite mineral as a salt. (fig. 10. (a)).

5.2.2. Supported limestone walls

X-ray diffraction data of supported walls sample shows that, sample consists mainly of calcite, dolomite, halite, quartz and gypsum. (fig. 10. (b)).

5.2.3. Mortars

Data of X-ray diffraction of building mortar shows that, sample consists mainly of calcite, quartz, gypsum, in addition to traces of halite as a salt. (fig. 11. (a)). Fixing mortar of faience tiles consists mainly of calcite, quartz, gypsum besides to halite mineral as a salt. (fig. 11. (b)).

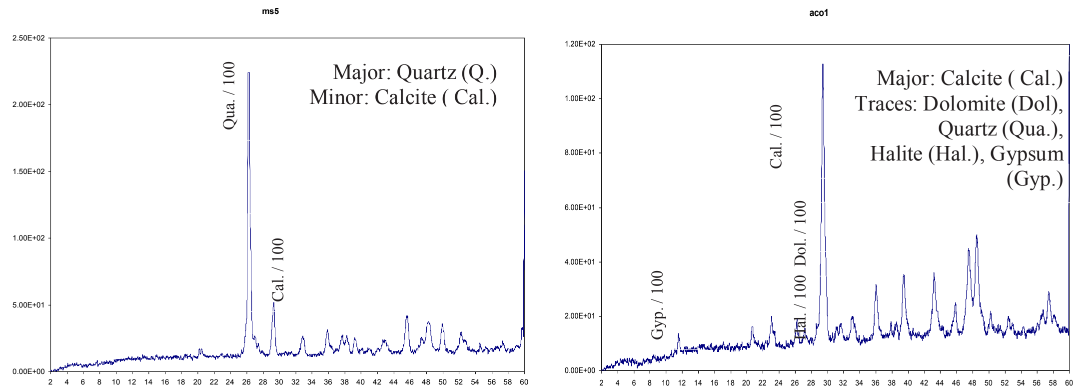


Fig. 10. (a) Shows XRD pattern of bodies of faience tiles; (b) Shows XRD pattern of supported limestone walls.

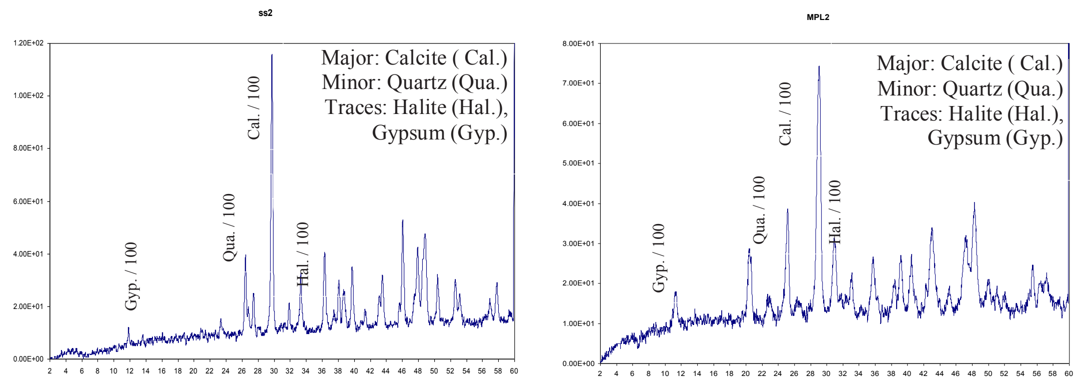


Fig. 11. (a) Shows XRD pattern of building mortar.; (b) Shows XRD pattern of faience tiles mortar.

5.3. X-ray Florescence

The major and trace elements composition of the samples from the body of faience were determined by X-ray florescence spectroscopy. The results obtained (table 1,2) showed the body contains high ratio of silicon oxide SiO_2 (91.19 %), water H_2O (5.5%). The alkaline oxides (Na_2O, MgO) are .73%, .23 % respectively whereas calcium oxide CaO is (.88 %). The result showed also that the body contains a few percent each of aluminum oxide (.34%) and iron oxide (.27%).

Table 1. and Table 2. Show the XRF analysis results for body of faiences

Compound Formula (Table 1)	Concentration (%)
H	0.6155
O	54.28
Na	0.542
Mg	0.139
Al	0.181
Si	42.63
P	0.0593
S	0.0285
Cl	0.516
K	0.0616
Ca	0.633
Ti	0.0199
Mn	0.0043
Fe	0.193
Ni	0.00609
Cu	0.0725
Sr	0.00508
Zr	0.00218

Compound Formula (Table 2)	Concentration (%)
H_2O	5.5
Na_2O	0.731
MgO	0.23
Al_2O_3	0.342
SiO_2	91.19
P_2O_5	0.136
SO_3	0.0712
Cl	0.516
K_2O	0.0742
CaO	0.886
TiO_2	0.0332
MnO	0.0055
Fe_2O_3	0.276
NiO	0.00774
CuO	0.0908
SrO	0.00601
ZrO_2	0.00294

5.4. Scanning Electron Microscopy coupled with EDX

Many samples including samples from body of faience tiles, mortars and supported walls were investigated and analyzed by scanning electron microscopy coupled with EDX microanalysis. Investigation by SEM of the body sample showed the erosion and disintegration of the faience by the salt crystallization (fig. 12a) whereas EDX analysis of the same location contains the elements of each chloride (Cl) and sodium (Na) with high ratio. This indicates that the faiences deteriorated by the crystallization of sodium chloride salt, in addition to leaching of alkaline compounds from the core of faience to the surfaces (fig. 12b). The result analysis of the colored surface showed the presence of elements silicon (Si), copper (Cu) (fig. 13a, b, c) and this indicate that a copper compound was used to obtained the turquoise blue color in an oxidizing atmosphere (Hatton et al....., 2008; Tite et al....., 2008). The presence of sodium, magnesium and calcium elements indicate that weathering process was occurred according to (Oakley 1989; Huisman et al....., 2008; Tite et al....., 2009) by action of water where the alkali ions (Na+ and K+) are replaced by hydrogen ions (H+) (fig. 14d). In addition to, several different degradation processes occur within a porous ceramic bodies resulting to the presence of water (Skibo and Schiffer, 1987). The mortar used to fix the faience tiles have been analyzed and the results showed that it contains mainly elements of chloride (Cl), sodium (Na) and this indicate that this mortar contains high ratio of sodium chloride salt NaCl (fig. 14a, b, c, d). Regarding to the kind of building mortar used, the results were proved that it was consisted of lime and quartz (as aggregate), in addition to few amounts of sodium, aluminum, potassium and iron, (fig. 15a, b). At very high relative humidity (RH) lime mortars become weak mechanical properties and low internal cohesion as well as high porosity, and this making lime mortar susceptible to damage caused by salt crystallization (Elert et al....., 2002). The supported wall analysis showed that it is limestone where it contains mainly calcium (Ca) and few contents of silicon (Si) and iron (Fe), (fig. 16a, b, c).

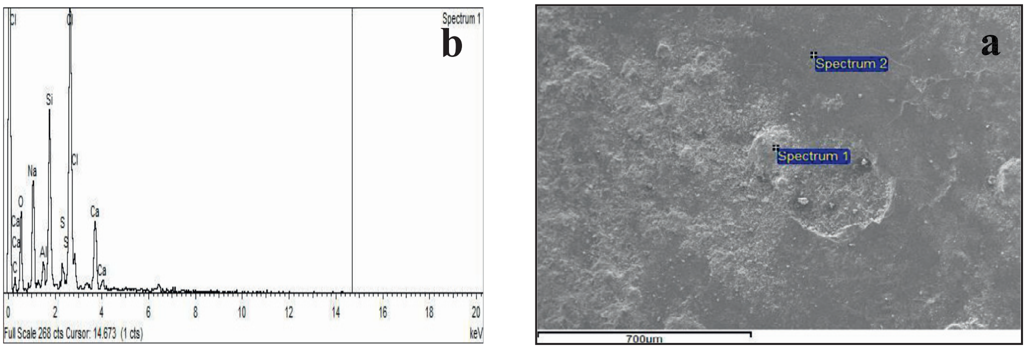


Fig. 12. (a); (b) Show the SEM/EDX for the faience tiles

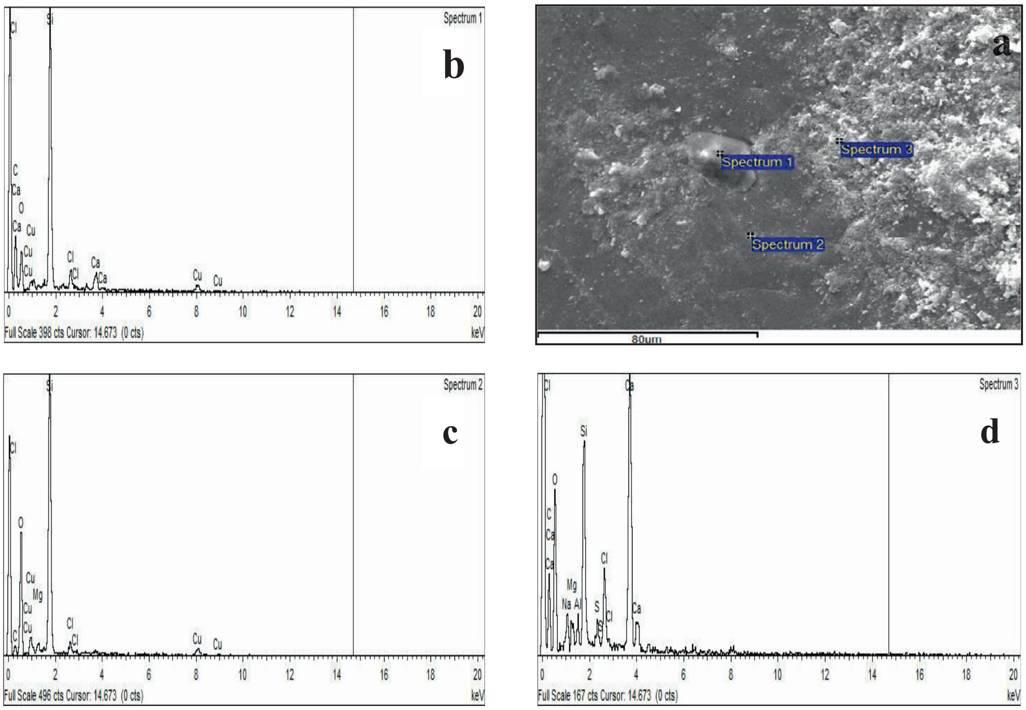


Fig. 13. (a); (b); (c); (d) Show SEM/EDX results of colored faiences surfaces

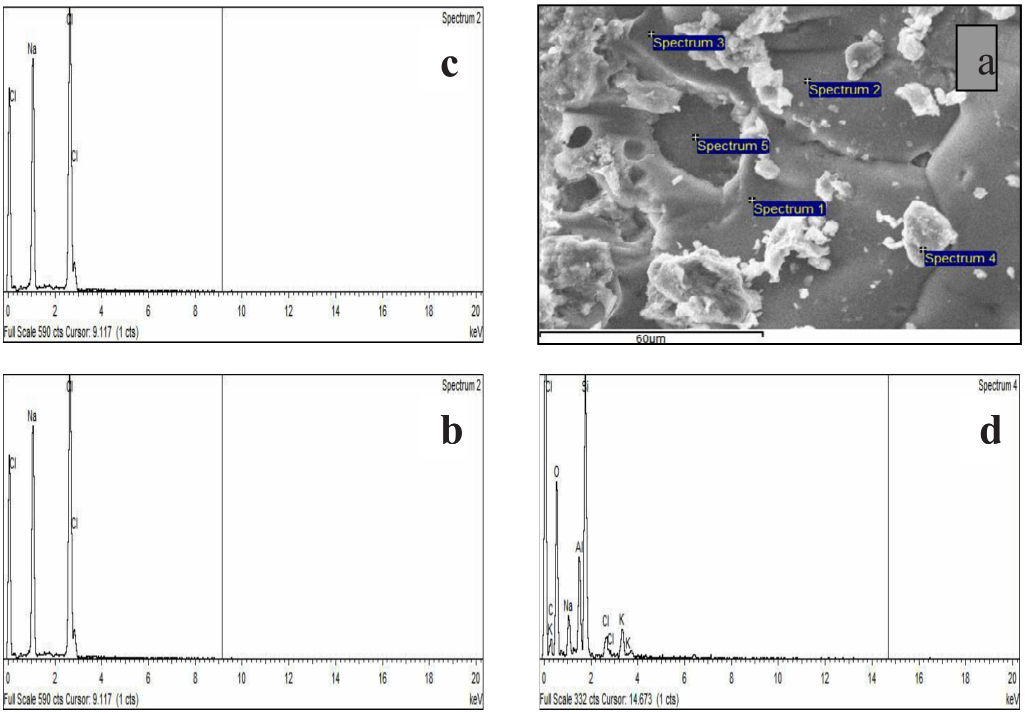


Fig. 14. (a); (b); (c); (d) Show SEM/EDX results of mortar

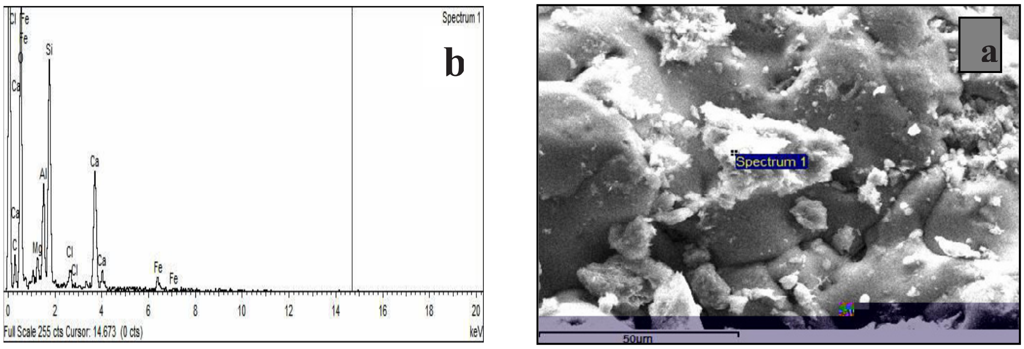
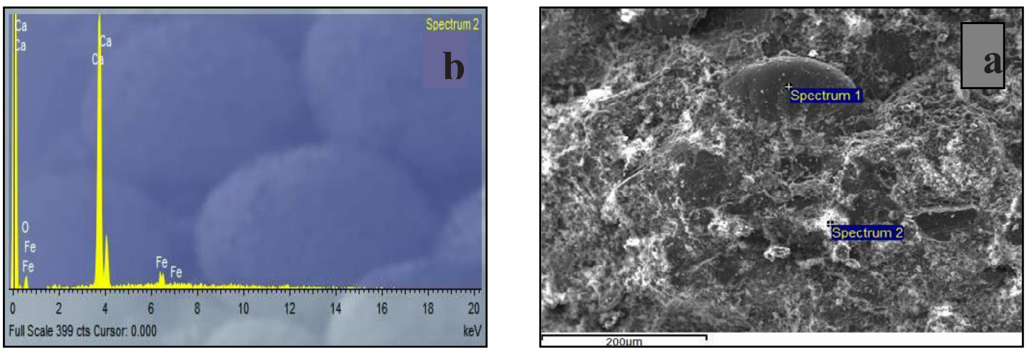


Fig. 15. (a); (b) Show SEM/EDX results of mortar.



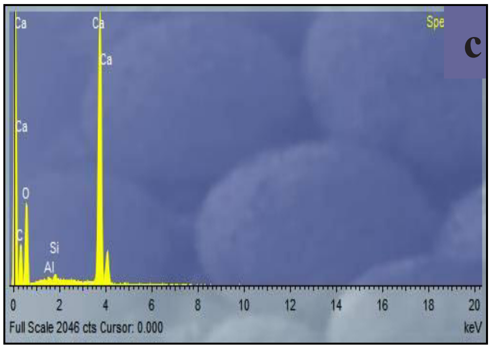


Fig. 16. (a); (b); (c) Show SEM/EDX results of supported walls

5.5. Causes of decay of faience tiles

The results showed that the faience tiles suffered from high degradation because of rising damp in the supported walls and crystallization of soluble salts. Failure of faience tiles is most commonly water related, where the units are highly susceptible to glaze cracking, spilling and material loosing (Wilson, 1984). Also when a glass or glaze is subjected to weathering by the action of water, the alkali ions (Na^+ and K^+) are replaced by hydrogen ions (H^+) and the glass network progressively breaks up. Thus, the silica glass structure is lost and replaced by amorphous layer so-called silica gel. In addition to leaching out of the alkalis, there are also some leaching out of the colorants which will no longer be present as ions but will have been deposited as fine amorphous or poorly crystalline compounds resulted in the change the chemical composition of faience surfaces. (Schreiner et al....., 1999; Huisman et al....., 2008; Tite et al....., 2009).

Porous faience tiles can also deteriorate due to the presence of soluble salts within the body itself where the salts dissolve and re-crystallization (Van Balen et al....., 1996). Crystallization of the salts results in tremendous pressures within the pores of the body or at glazed body interface where this caused a glazed layers to flake away at many faience tiles (Oakley and Jain, 2002). The binding mortars suffer from failure and fragile due to chemical alteration (Dotter, K. R.2010) resulting of high content of moisture and crystallization of salts where the mortar has always been the key to the survival of ceramic tiles. (Wilson ,1984; Ashurst, 1989). Limestone supported walls of faience tiles suffer from salts crystallization. Salts may be carried into the stone with groundwater by capillary rise, (Figueired, C., Et al..... 2010). or may be dissolved from the mortar joints, or result from chemical reaction between atmospheric pollutants (especially SO_2) and minerals (such as calcite ($CaCO_3$) in limestone). (Beck., K. Et al..... 2010).

As the water evaporates, the supersaturation of the pore solution increases until salt precipitates. (Shahidzadeh N., et al..... 2008) A change in temperature, generally a decrease, leads also to a supersaturation of the solution resulting in the crystallization of salts. (Rothert, E., et al..... 2007). If evaporation occurs on the surface of the stone, then the crystals form a harmless (but unattractive) deposit on the surface called 'efflorescence'. (Lubelli, B. et al..... 2007). However, if salts precipitate beneath the material surface (a phenomenon called subflorescence or cryptoflorescence), severe damage can be induced. (Espinosa-Marzal, et al...., 2010) Although efflorescence does not generally affect the coherence and endurance of building materials, it impairs the surface appearance, which can be significant in case of historical buildings. (Sghaier. N. et al..... 2009).During the initial period of decreasing drying rate, the meniscus penetrates progressively into smaller pores. As long as a continuous liquid film covers the pore walls from the meniscus up to the exterior surface, then liquid transport can proceed by flow of the film, causing evaporation to take place on the surface and forming efflorescence. (Espinosa-Marzal, R. M. et al...., 2010). At the same time some liquid evaporates within the unsaturated pores, which causes salt to precipitate as subflorescence. (Viles HA. 1994). The decrease in the moisture content, the pore clogging with crystals and the increase of the solution viscosity with concentration may retard the liquid (capillary) flow. (Figueired, C., et al..... 2010). If the capillary flow becomes slower than the evaporation rate then the film becomes discontinuous and the flow to the surface is partly interrupted. Thereafter, evaporation takes place only inside of the material, indicating the start of the second (more accentuated) decreasing rate period of drying. (Sass, O. et al..... 2010).

The results determined also the human deterioration phenomena due to the human negligence which observed on the careless of the faience tiles at the tomb. Because of negligence and absence of the periodical maintenance and conservation procedures, many tiles fall of from their supported walls and sometimes lost completely.

6. Conclusions

X-ray florescence for body of faience tiles showed that it contains high ratio of silicon oxide SiO_2 and low amounts of alkaline oxides (Na_2O, MgO). X-ray diffraction XRD results of fixing mortar of faience tiles showed that it consists mainly of calcite, quartz, gypsum, in addition to halite (sodium chloride) as a salt and this result confirmed with the results obtained by EDX for the same sample. EDX microanalysis of the faience tiles colored surfaces determined that the presence elements of silicon (Si), copper (Cu) which indicate that a copper compound was used to obtain the turquoise blue color in an oxidizing atmosphere. The results determined that the faience tiles suffer from high degradation including failure and fragile of the binding mortar, fragile and flaking off the glazed surfaces and fall of many faience tiles from the supported walls.

The binding mortars which used to fix faience tiles have always been the key to the survival of these tiles. These mortars suffer from failure and fragile due to chemical alteration resulting of high content of moisture and crystallization of salts mainly halite salt (sodium chloride). The rising damp in the supported walls and crystallization of soluble salts consider the main deterioration factors of faience tiles. The results confirm first of all the major role of water in the development of stone and faience tiles alteration. Finally this study will aid to carry out the conservation and maintenance works of these faience tiles at the future and will be useful for future researches of Egyptian faience tiles.

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