ORIGINAL PAPER

ASSESSMENT OF DETERIORATION PROCESS IN EXPOSED CARVED LIMESTONE FALSE DOOR OF ABUSIR ARCHAEOLOGICAL AREA

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Abstract. *Carved limestone false door dating back to the late Sixth Dynasty or the First Intermediate Period in Abusir archaeological area suffers from many physiochemical and mechanical deterioration factors, which lead to various deterioration phenomena, such as distort the carvings, decorations, the disappearance of paint, cracks in different depths, and salt calcification. It was found in the fill of Shaft 5 in the tomb AS 79 at Abusir South, Excav. No. 9/AS79/2015 and located in the storeroom of Czech Excavation in Abusir area. The current work aims to study the type of limestone which the false door is made of, and to know if there were colors on the false door or not, also to know if there were deterioration mechanisms that affect the carved limestone false doors of Abusir to evaluate the deterioration ratio in stone structure and the effects of surrounding environmental factors on stone. The investigation and characterization processes of archaeological limestone samples were carried out by polarizing microscopy (PLM), optical microscopy (OM), scanning electron microscopy (SEM-EDX) micro-analysis system, X-ray diffraction (XRD), X-ray fluorescence (XRF), and Fourier transform infrared spectroscopy (FTIR). The results revealed that the limestone material of the false door belongs to micrite limestone with very fine grains and needs to be conserved.*

Keywords: *false door; limestone; Abusir; deterioration; microscopy; XRD; XRF; FTIR.*

1. INTRODUCTION

The archeological area of Abusir is one of the most important archaeological sites in the cemetery of Memphis. It is located to the south of the pyramids of Giza and represents a part of the area of Saqqara and away 3 kilometers from the Step pyramid. The archaeological area contains many historic buildings such as, Temples, Tombs, and Mastabas which contains many architectural elements, such as the false doors [1-5].

The false door idea considers one of the most important architectural elements that were found at the construction of Mastaba, Tombs, and Royal tombs. These doors have been started in the third dynasty [6-7]. The false door is the link between the dead and the living, as well as to receive food, sacrifices, and supplications when visiting the family of the deceased

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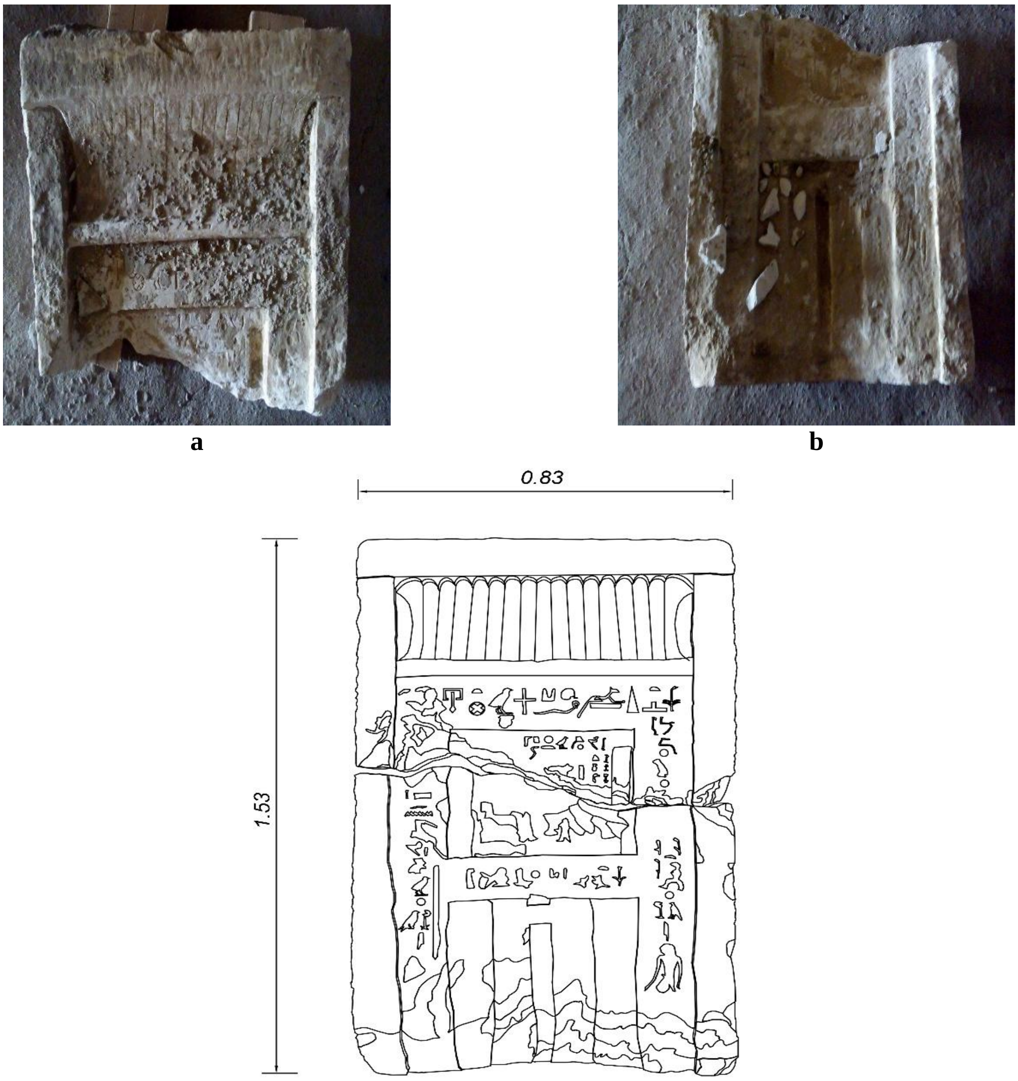
[8-9]. The place of the false door was often found in the western wall in the tombs, where the ancient Egyptian believed that the other world existed in the western side [10-12]. This type of objects has not been studied in-depth and need more studies on the stone structure and its deterioration phenomenon. The false doors mainly suffer from many deterioration phenomena as a result of the effect of surrounding environmental deterioration factors, and the deterioration takes the form of salt efflorescence, powdering, the disintegration of the stone surface and bonding material in stone structure, in addition to deterioration the pigments and pictorial layers (Fig. 1 - a, b, c). The current work focus on the study of archaeological false door dating to the late Sixth Dynasty or the First Intermediate Period /at Abusir area. The owner of this false door was a certain Khai. It was found in the fill of Shaft 5 in the tomb AS 79 at Abusir South, Excav. No. 9/AS79/2015 and it was removed from its original position probably in ancient times. The false door made from White limestone and the decoration in sunk relief. Typology of FD: three-jamb doors with a rim and additional frame, T-shaped panel, narrow apertures. On the panel, the owner is depicted seated on the chair in front of the offering table with schematized bread loaves [13]. Currently, this object located in the storeroom of Czech Excavation in Abusir area. This research focuses on determining the deterioration mechanism through the investigation and analysis mentioned above.



a b

c

Figure 1. The studied false door before the conservation process: (a) part 1 (photo H. Hakem); (b) part 2 (photo H. Hakem); (c) the architectural documentation of the false door (drawing M. Galal).

2. MATERIALS AND METHODS

2.1. POLARIZED LIGHT MICROSCOPY

Thin section of collected archaeological carved limestone samples were examined for identification of limestone minerals using polarized light microscopy (PLM), which use to identify the samples refraction coefficient, shape and colour of the crystals, as well as internal disconnect and colour change [14], model a Zeiss Axio Imager A1m with an Axio Cam (Carl Zeiss Microscopy GmbH, Jena, Germany) under 100X magnifications in plane-polarized light.

2.2. OPTICAL MICROSCOPY

Upright microscopy using a Zeiss Axio Imager M1 (Carl Zeiss Microscopy GmbH, Jena, Germany), with an Axio Cam (200-400×) was used to investigate and identify the type of limestone. In addition, it helps to identify the aspects of damage within the samples and helps to recognize the visibility of fossils clearly [15].

2.3. STEREO MICROSCOPY

Preliminary morphological observation of the limestone samples was carried out using stereo microscopy, a Zeiss Stereo Discovery V20 (Carl Zeiss Microscopy GmbH, Jena, Germany), equipped with Axio Cam MRC5, light Optika B-383PL equipped with digital camera 4083-B9, under 10x and 20x magnification [16, 17].

2.4. SCANNING ELECTRON MICROSCOPY

The microstructure of the archaeological limestone samples was investigated by scanning electron microscopy (SEM) A Quanta 3D 200i (Thermo Fisher Scientific Inc. (FEI Company), Hillsboro, Oregon, USA), equipped with EDX micro-analytical system. This examination was performed to detect the element contents of collected archaeological limestone samples. Images were acquired in backscattered mode (SSD, LFD, BSED), the accelerating voltage was 25 kV in the magnification range of 80 to 3000x [15, 18, 19].

2.5. X-RAY DIFFRACTION

The identification of the mineral composition of the archaeological carved limestone samples was conducted by X-ray diffraction patterns. The limestone samples were analyzed by X-ray diffraction using X-ray Diffractometer System PW3040–Analytical Equipment–PANalytical pro model, Cu-target tube, and Ni filter at 40kV and 30MA. X’Pert High score software was used for identifying the components of the limestone [20, 21].

2.6. ATTENUATED TOTAL REFLECTION – FOURIER TRANSFORM INFRARED SPECTROSCOPY

Fourier transform infrared spectroscopy was performed using Vertex 70 spectrometer (Bruker AXS Inc, Billerica, MA, USA), equipped with an attenuated total reflection (ATR) system (the range of spectrum 4000-400 cm^{-1}; range with a resolution of 4 cm^{-1}; sample scans 20). This technique was used to identify the changes of molecular structure occurring in the samples because of weathering processes [22-29].

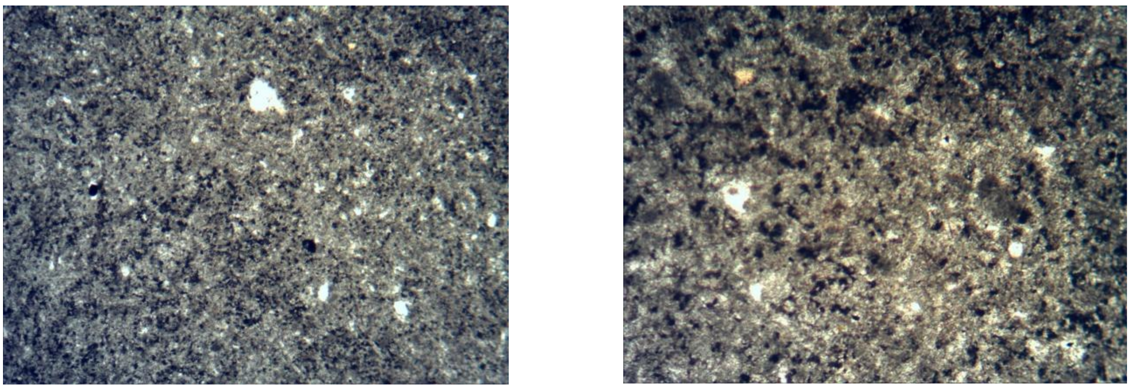
2.7. X-RAY FLUORESCENCE

X-ray fluorescence shows the elemental composition surface to identify the painted layer on the stone sample. X-Ray fluorescence analysis was carried out with a portable system, Niton™ XL3t XRF analyzer (Thermo Fisher Scientific Inc., Waltham, MA, USA), which includes X-ray tube with Ag anode, 50 kV and 0-200 μA max, metal and mining modes, spot diameter 3 mm and duration of exposure 60 seconds [16, 21, 22, 30-32].

3. RESULTS AND DISCUSSION

3.1. POLARIZED LIGHT MICROSCOPY

Thin section analysis for a limestone sample was sectioned and mounted on a microscopic slide. Polarizing microscope (PLM) observation of limestone showed that the limestone sample consists mainly of fine-grained calcite crystals, and limestone belonging to micrite limestone with very fine grains (Fig. 2a), and the microscopic examination showed that the limestone was deteriorated, and there is the disintegration of calcite crystals was observed from the presence of quartz crystals, silica minerals, and high ratio of halite and gypsum (Fig. 2b).



a b

Figure 2. Thin section of archaeological limestone samples (100x): a) the limestone sample consists of fine-grained calcite crystals which belonging to micrite limestone; b) showed the limestone was deteriorated.

3.2. OPTICAL MICROSCOPY

Examination of a thin section of limestone sample under upright microscopy revealed to the presence of some differant fossils (Figs. 3a-c) and displayed the aggregates of opaque brown and some remains of colors (Fig. 3d).

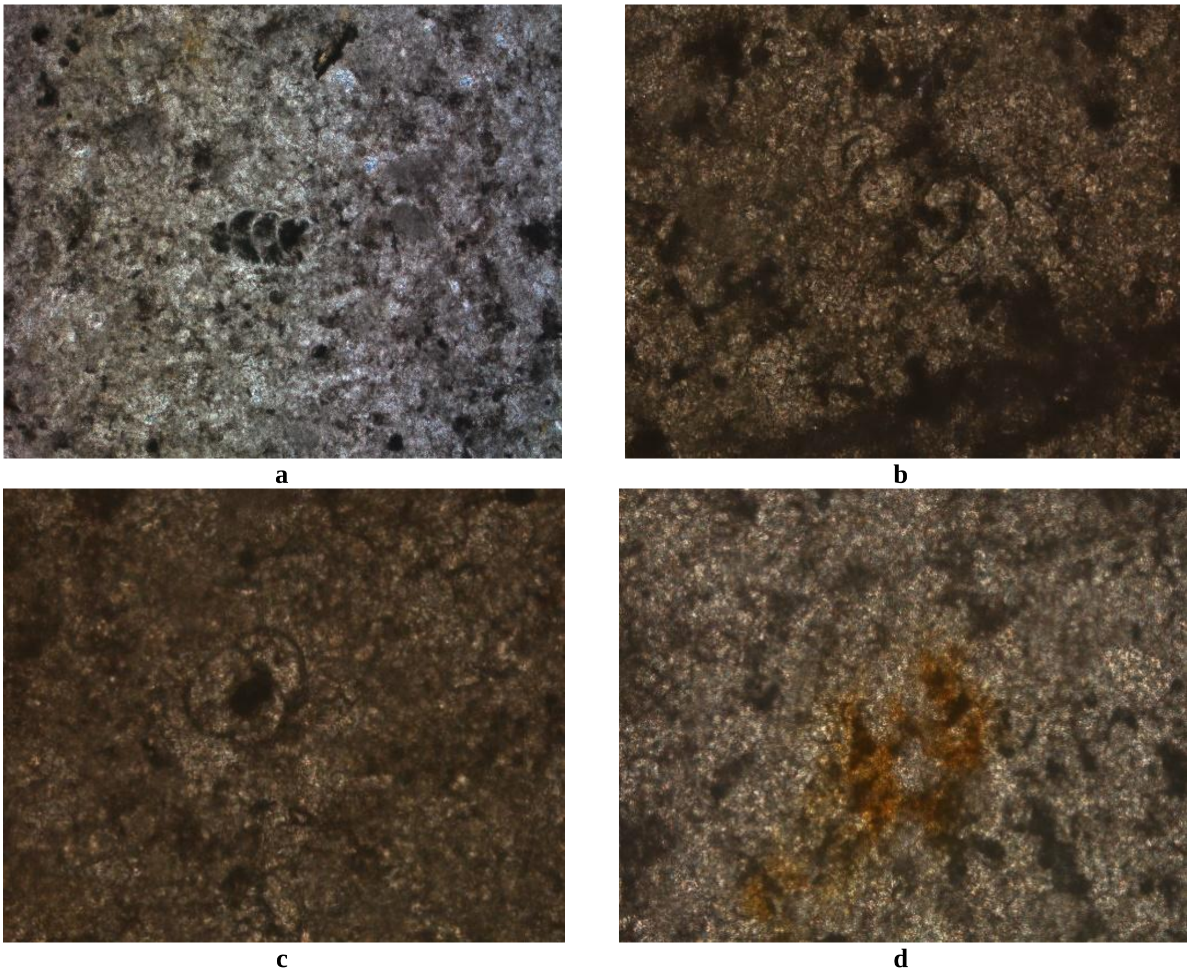


Figure 3. Thin section of archaeological limestone sample under upright Microscopy (200x – 400x): a, b, c) the limestone sample consists of some differant fossils; d) the limestone sample consists of remains of colors.

3.3. STEREO MICROSCOPY

The investigation of the limestone sample under stereo microscopy revealed that the samples are suffering from deterioration (Fig. 4a), by salt efflorescence (Fig. 4b), and disintegration of the binding material of the stone surface (Fig. 4c), in addition to the presence of some fine cracks and voids in some areas in stone surface (Fig. 4d).

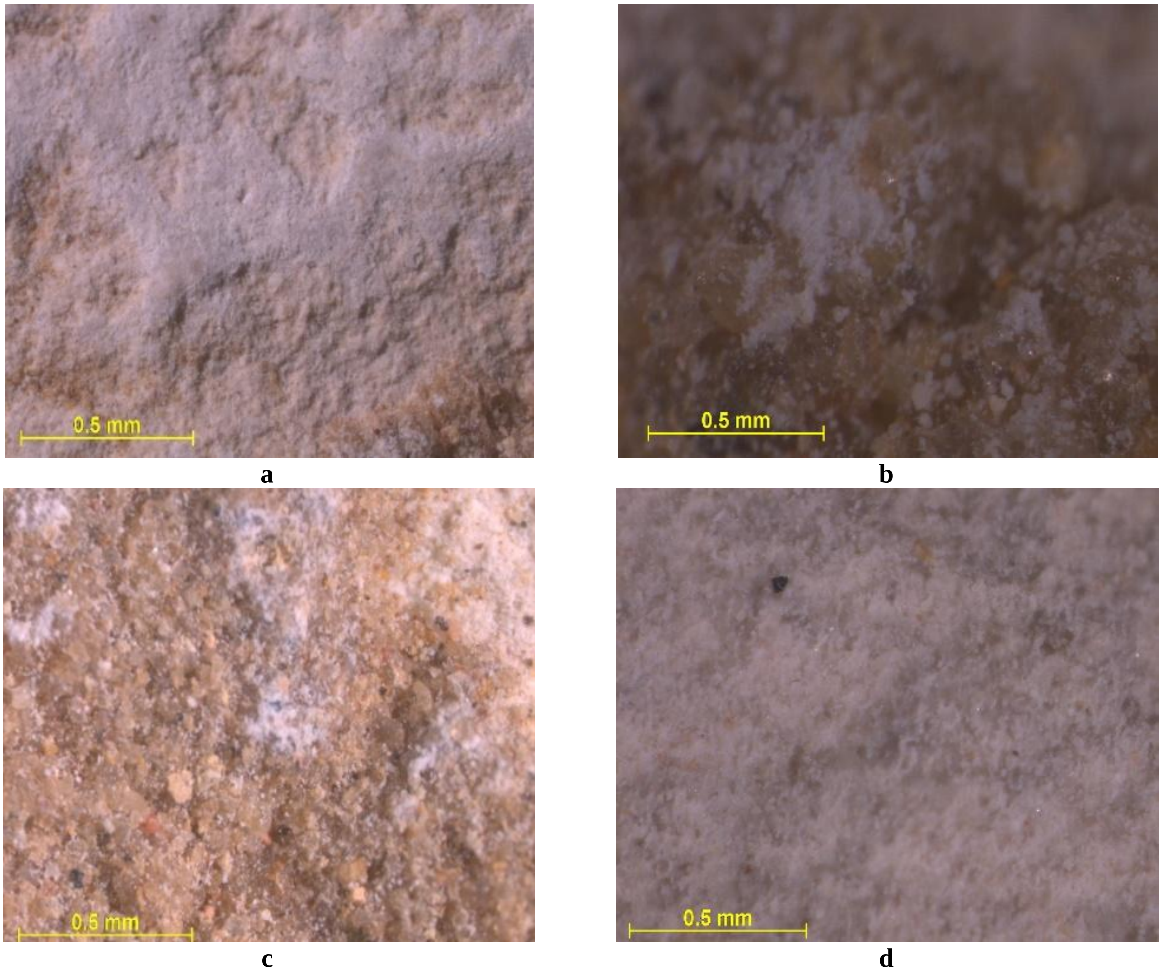
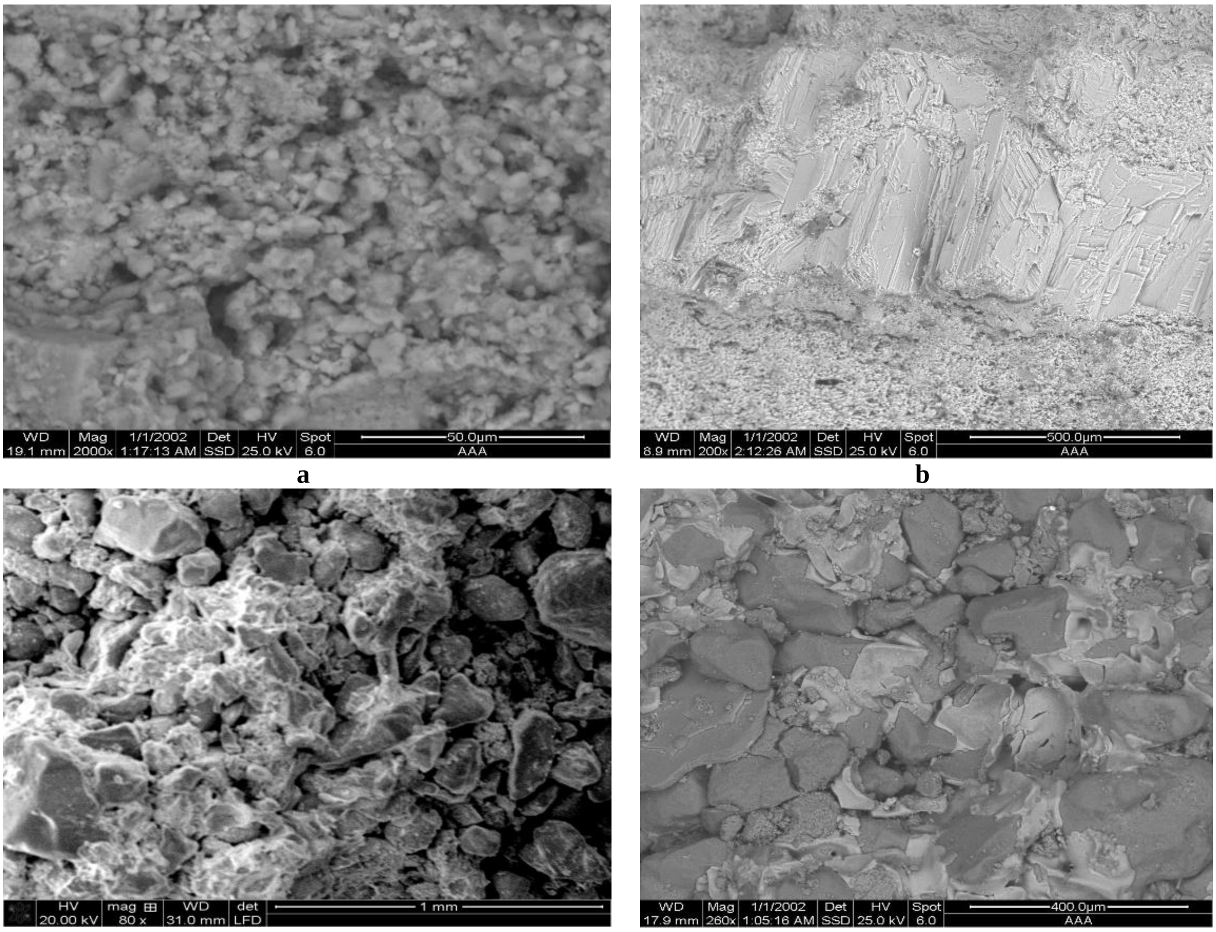


Figure 4. Stereo microscopy investigation of archaeological limestone sample: a) show the deterioration features of the studied sample; b) salt efflorescence; c) disintegration of the binding material of the stone surface; d) the presence of some fine cracks and voids in some areas in stone surface.

3.4. SCANNING ELECTRON MICROSCOPY

SEM investigation of the archaeological limestone sample showed a high porosity of limestone and includes a high ratio of calcite, quartz, iron oxides (Fig. 5a), and gypsum (Fig. 5b). The disintegration of calcite crystals in some areas (Fig. 5c) is due to degradation by physical weathering and salt crystallization (Fig. 5d). The total EDX analysis of the limestone samples in (Fig. 6a) showed that sulfur (S), calcium (Ca), quartz (Si), are the dominant elements; iron (Fe), magnesium (Mg), aluminum (Al) were observed (Table 1). In Fig. 6b the total EDX of other points in archaeological limestone showed that silicium (Si), chloride (Cl), sodium (Na) are the dominant elements; calcium (Ca), aluminum (Al), magnesium (Mg), and iron (Fe) were observed as well (Table 2).



a b c d

Figure 5. SEM images of archaeological limestone samples: a) a high ratio of calcite, quartz, iron oxides; b) crystals of gypsum; c) the disintegration of calcite crystals in some areas; d) show the deterioration features of the studied sample.

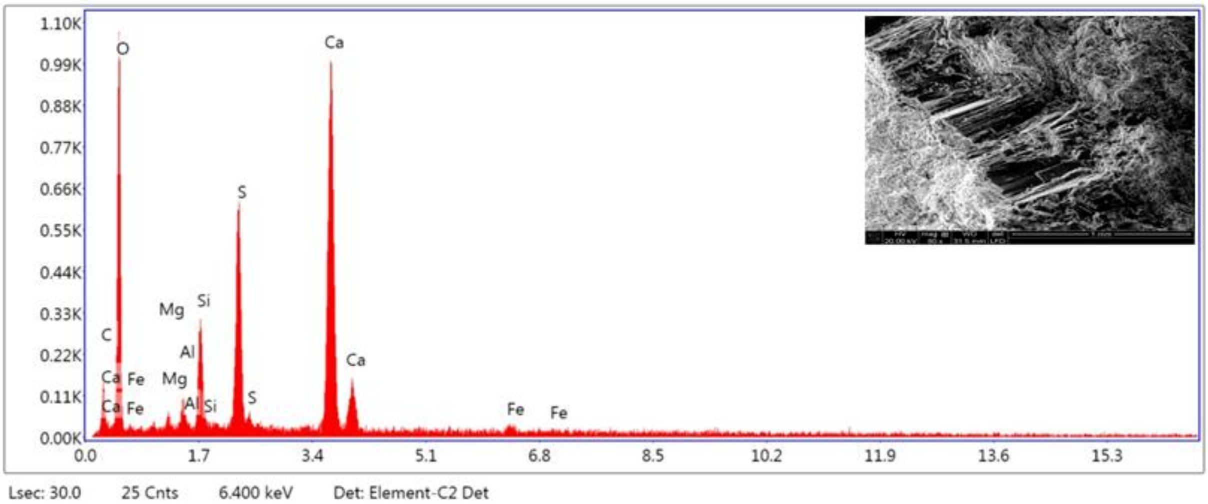


Figure 6a. EDX spot analysis from the side view of archaeological limestone samples.

Table 1. Quantitative SEM-EDX analysis (element %) of the side view of the studied sample.

Element	C	O	Mg	Al	Si	S	Ca	Fe
Weight [%]	4.73	58.76	0.55	1.02	3.57	8.51	22.45	0.41

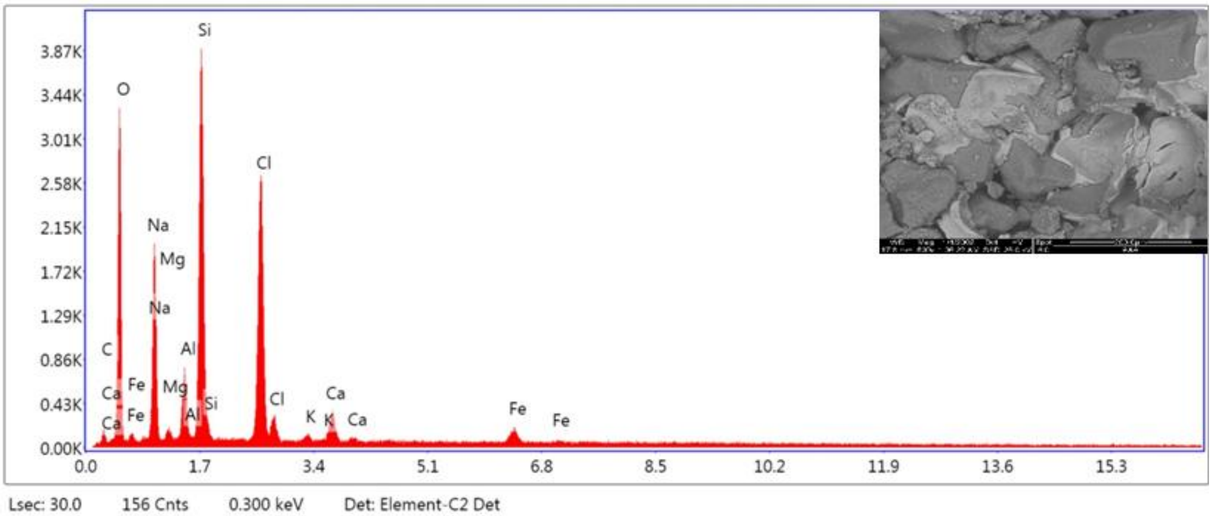


Figure 6b. EDX spot analyses from the surface of archaeological limestone samples.

Table 2. Quantitative SEM-EDX analysis (element %) of the side view of the studied sample.

Element	C	O	Na	Mg	Al	Si	Cl	K	Ca	Fe
Weight [%]	1.23	39.91	16.98	0.53	3.47	18.14	15.38	0.25	2.16	1.96

3.5. X-RAY DIFFRACTION

The XRD analysis of the archaeological limestone samples (Table 3) showed that the major mineral is calcite ($CaCO_3$), quartz (SiO_2), with a trace amount of halite (NaCl), as shown in (Fig. 7a). The XRD analysis of the gypsum samples picked from the archaeological limestone showed that the major mineral is gypsum, with trace amounts of calcite and quartz (Fig. 7b). The XRD analysis of the deteriorated archaeological samples fallen from the stone surface showed that it consists essentially of calcite ($CaCO_3$), and quartz (SiO_2) as a major mineralogical constituent, with trace amounts of the following minerals: gypsum ($CaSO_4 \cdot 2H_2O$), and dolomite ($CaMg(CO_3)_2$ (Fig. 7c).

Table 3. X-Ray Diffraction data.

Compounds [%]	Samples		
	S. 1	S. 2	S. 3
Calcite	21.0	23.0	67.7
Quartz	77.0	3.0	21.2
Gypsum	-	74.0	8.1
Halite	2.0	-	-
Dolomite	-	-	3.0

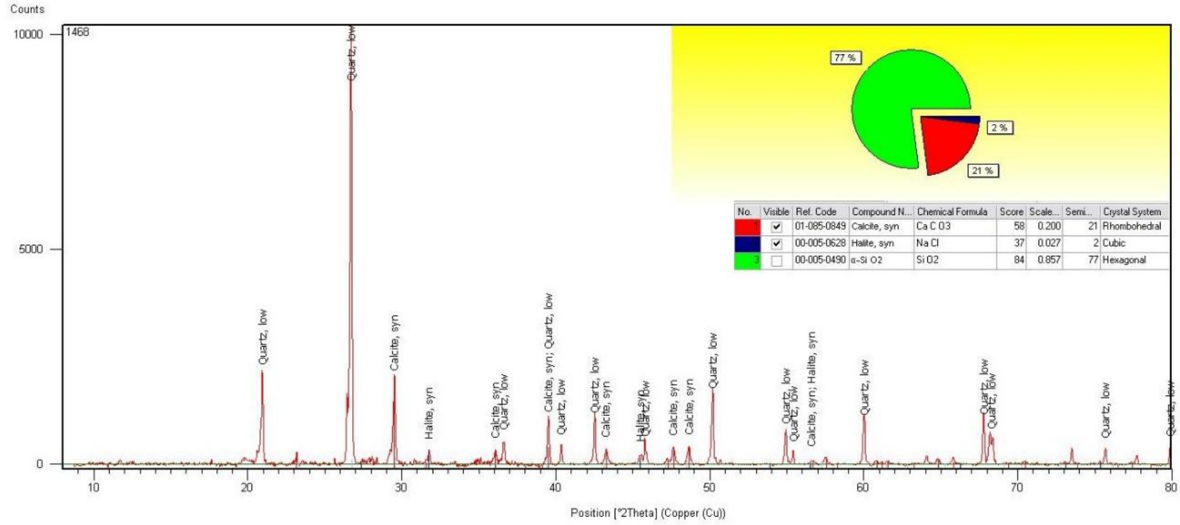


Figure 7a. XRD pattern of archaeological limestone samples, picked from the stone surface.

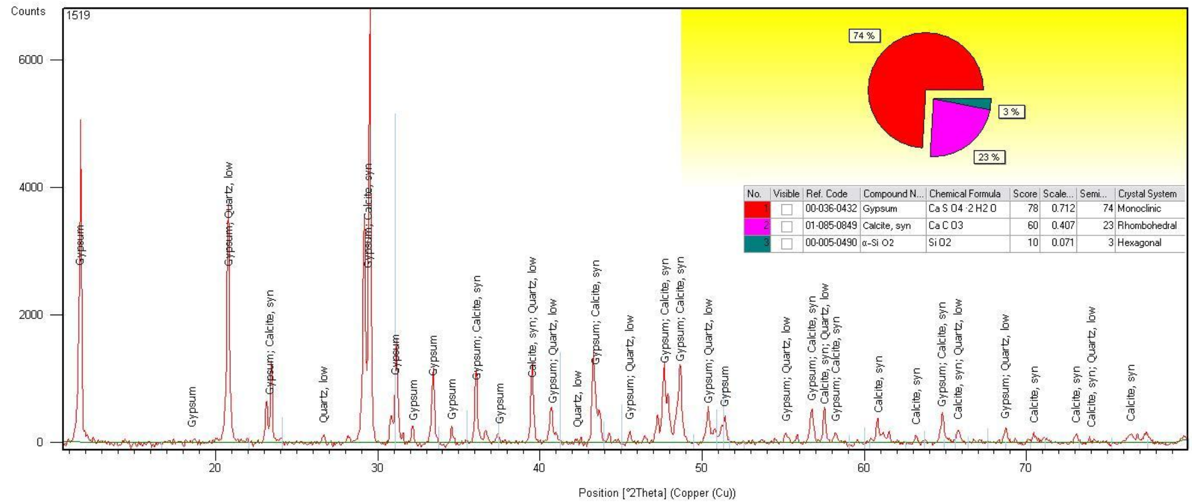


Figure 7b. XRD pattern of gypsum samples, picked from the side view of archaeological limestone false door.

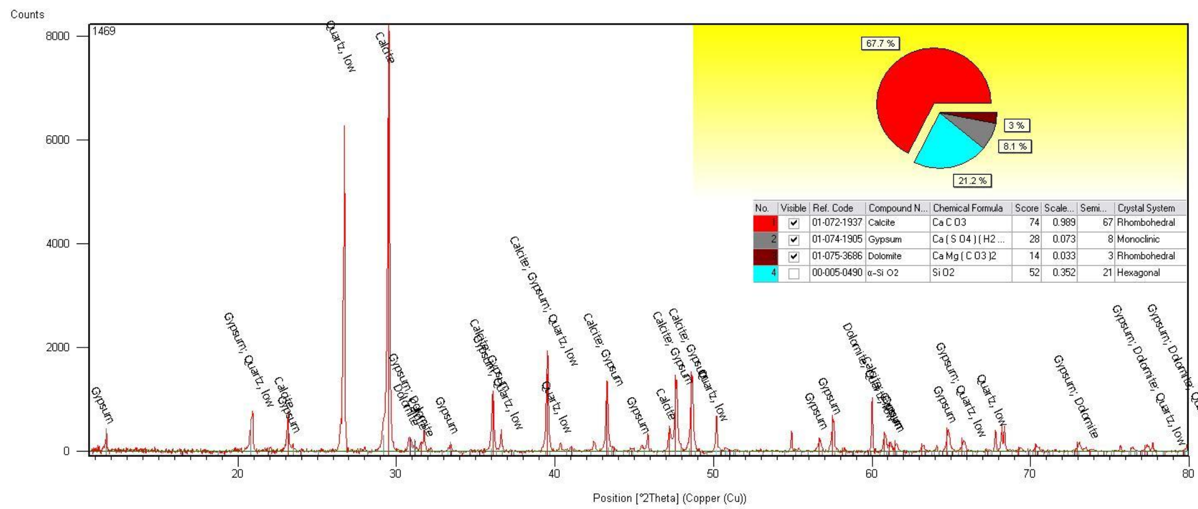


Figure 7c. XRD pattern of the deteriorated archaeological samples fallen from the stone surface.

3.6. FOURIER TRANSFORMED INFRARED SPECTROSCOPY

FTIR was used for identifying the limestone components, and the binding media. The components of limestone were identified as calcium carbonates by a comparison with a controlled samples based on characteristic absorption bands. The infrared spectra of inorganic carbonates consist of a CO_3^{2-} stretching band at 1367-1506 cm^{-1}, a band of medium intensity near 1122 cm^{-1}, a weak band at l097-l049 cm^{-1}, a band of O-C-O bending band at 875-794 cm^{-1}, and a band of variable intensity at 713-670 cm^{-1} (Fig. 8a). The binding media was identified as Arabic gum by a comparison with controlled sample which means there were signs of painted layer applied on the limestone false door. The infrared spectra consists of O-H stretching band at 3628-3408 cm^{-1}, C-H stretching band at 2924 cm^{-1}, O-H bending band at 1650 cm^{-1}, C-H bending band at 1480-1300 cm^{-1}, C-O stretching band at 925 cm^{-1}(Fig. 8b) [33].

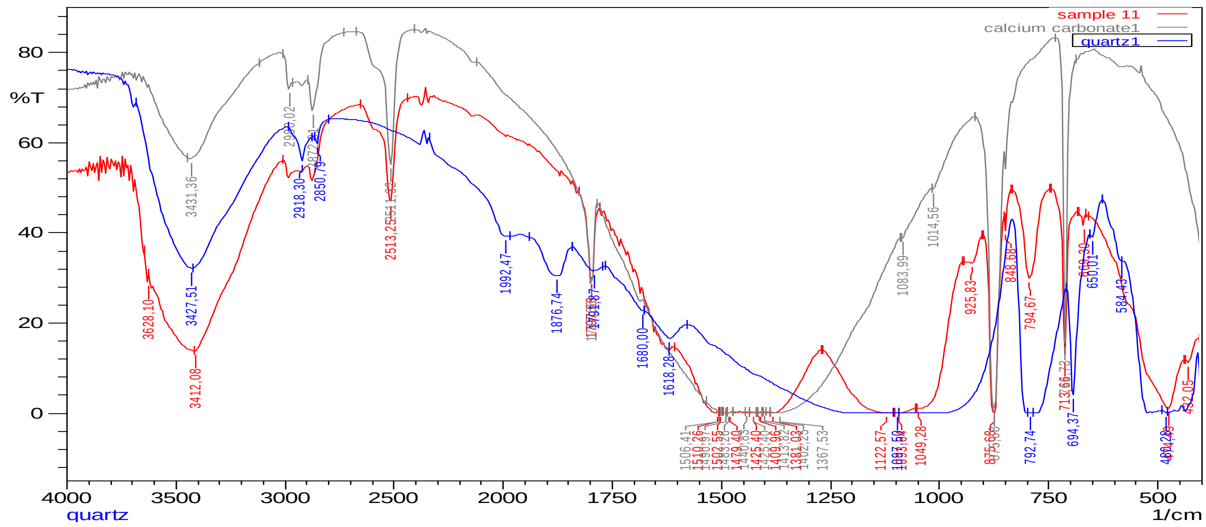


Figure 8a. FTIR-ATR analysis of deteriorated limestone samples picked from the surface.

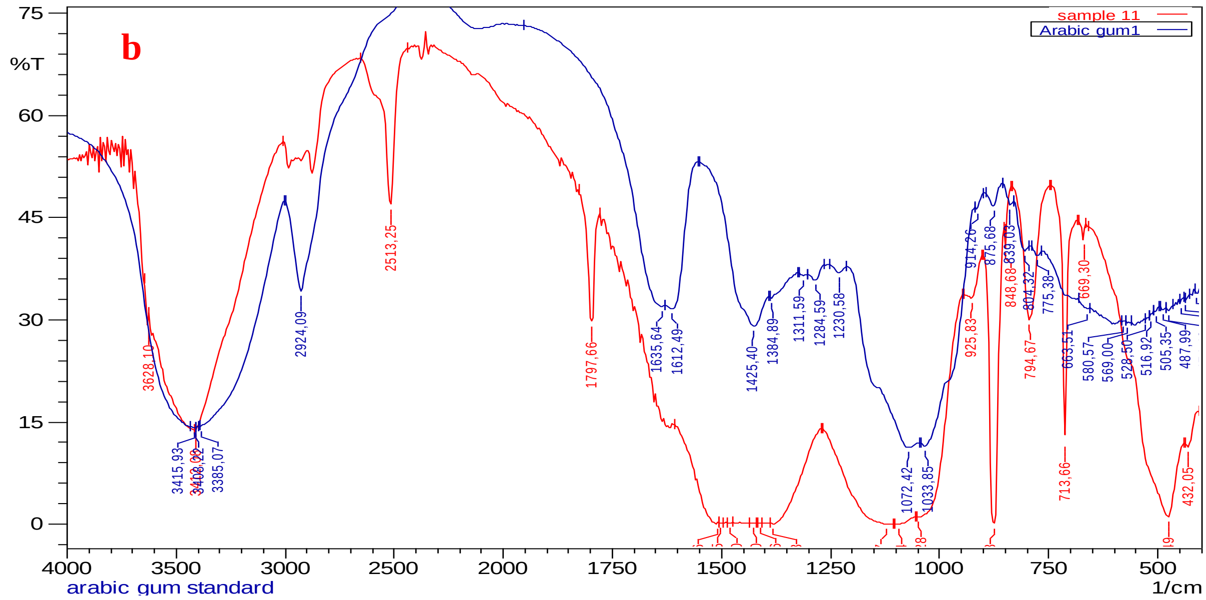
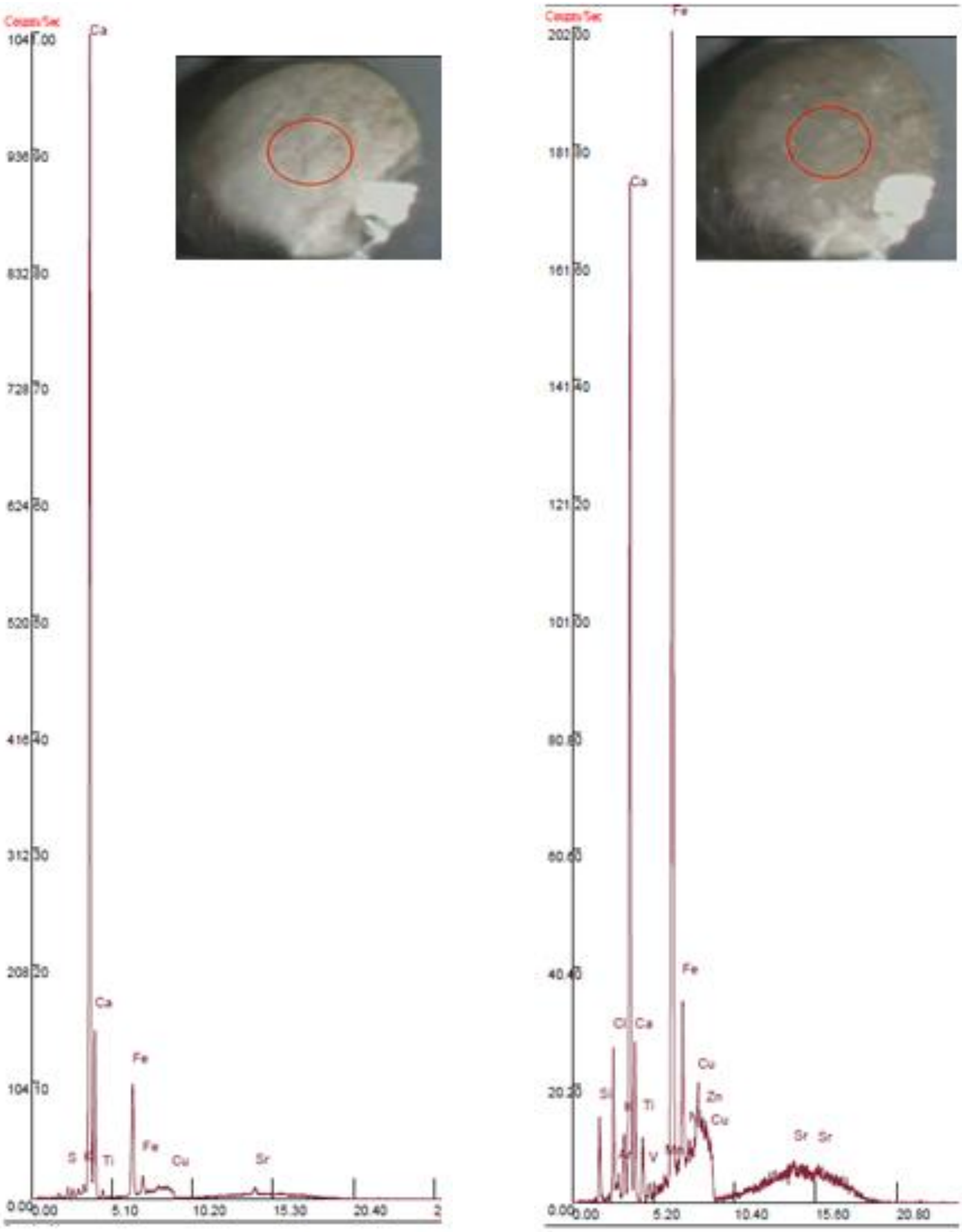


Figure 8b. FTIR-ATR analysis of deteriorated limestone samples picked from the surface.

3.7. *X-RAY FLUORESCENCE*

XRF results revealed the presence of (Ca), and high ratio of (Fe) as Main elements, Cu, Zn, and Sr as trace elements (Fig. 9a). Suggesting the use of hematite as Pigment on stone surface which used from the 5th dynasty till the roman times [34] (Fig. 9b), which showed that it consists essentially of (Fe), and high ratio of (Ca) as Main elements, Cu, Zn, Ti, Si and Sr as trace elements



a **b**

Figure 9. XRF analysis of deteriorated limestone samples picked from the surface: a) a high ratio of Ca (calcite); b) a high ratio of Fe (hematite).

4. CONCLUSIONS

The current work presented analysis of the deterioration effects on the carved limestone false door in Abusir archaeological area located to the south of the pyramids of Giza. The studied false door dating back to the late Sixth Dynasty or the First Intermediate Period. The analysis and investigation study indicated that the false door suffers from many physio-chemical and mechanical deterioration factors. The results of mineralogical composition showed that the sample consists of calcite, quartz, clay minerals, gypsum, iron oxides, and organic materials, in addition to that, the microscopic investigation indicated that, the limestone material of the false door belonging to micrite limestone with very fine grains. Upright microscopy showed some fossils and displayed the aggregates of opaque brown and some remains of colors, This corresponds to other results, such as the results of XRF, SEM-EDX. SEM result showed high porosity of limestone in addition to the disintegration of calcite crystals in some areas is due to degradation by physical weathering and salt crystallization. XRD study has identified that, the studied limestone composes mainly of calcite ($CaCO_3$), and quartz (SiO_2) as a major mineralogical constituent with trace amounts of the following minerals: halite (NaCl), gypsum ($CaSO_4 \cdot 2H_2O$), and dolomite ($CaMg(CO_3)_2$. FTIR analysis suggested The binding media was identified as Arabic gum by a comparison with controlled sample which means there were signs of painted layer applied on the limestone false door, This corresponds to other results, such as the results of XRF, SEM-EDX. XRF analysis suggested the use of hematite as a Pigment on the stone surface.

The study showed the importance of studying the deterioration mechanism of the limestone false doors in Abusir area. In addition to the importance of carrying an experimental study on the conservation materials which can be used in the conservation plane of those limestone monuments. The conservation study will be done in future work.

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