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A Comparative Analytical Study of Some Deterioration Products Formed on the Sandstone Reliefs of Edfu and Kom Ombo Temples

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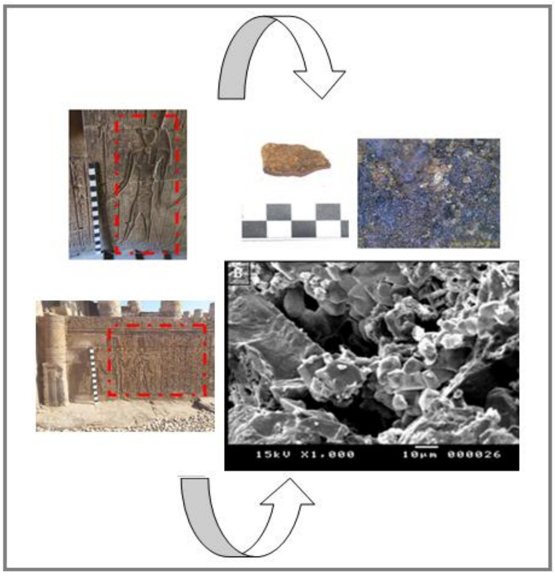
HIGHLIGHTS

- The weathering mechanisms affecting both Edfu and Kom Ombo temples were studied and evaluated.
- Optical and polarized light microscopes, scanning electron microscopy equipped by EDX unit (SEM-EDX) and X-ray diffraction analysis (XRD) were used to study the stone and the formed deterioration products.
- Among the common deterioration products that were previously detected on Egyptian monuments, new products were reported.

GRAPHICAL ABSTRACT



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ABSTRACT

This research aims to evaluate the main chemical and mineralogical characteristics of some deterioration products formed on the stone reliefs of Edfu and Kom Ombo temples, Upper Egypt. It is well known that characterizing the deterioration products and their possible origin helps in determining the most appropriate materials and methods for conservation. Optical microscope, polarizing light microscope and scanning electron microscope equipped by EDX unit (SEM-EDX) were used to characterize the stone and the formed deterioration products from the mentioned temples.

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The results confirmed that the sandstone grains are of small to medium sized rounded quartz. A notable percentage of iron oxides, which works as a cementing material for the secondary pores of stone, was also detected. X-ray diffraction analysis of the formed deterioration products on the sandstone reliefs of Edfu temple showed the presence of halite (sodium chloride, NaCl) and niter (potassium nitrate, KNO_3) salts. While the formation of some phosphate-based minerals was reported on the reliefs of Kom Ombo temple together with a notable stone surface pigmentation. The formation mechanisms of the mentioned products were proposed and discussed.

1. Introduction

The ancient monuments are exposed to many deterioration factors which result in many weathering forms including chemical and aesthetic damages that eventually affect the inner structure and the outer appearance of the surface. Most of the stone monuments, mainly in Upper Egypt, have been quarried of sandstone blocks as essential building materials. Many of the monuments in Upper Egypt are suffering from a serious damage due to various deterioration factors [1], such as the dramatic variations of the climatic conditions, the agricultural drainage, the low-level location of some sites and the surrounding urban sewage. Also, the geological setting itself is considered one of the main deterioration factors affecting the studied sites which geologically comprises four stratigraphic formations: Edfu, Esna shale, Armant and Issawia Formations [2]. The studied temples were constructed mainly on silt and clayey soil through the River Nile alluvial plains. According to El-Gohary [3], the groundwater level in the studied area is formed mainly from supplies of the irrigation waters, the urban sprawling, and the salinity index is relatively high. The soil characteristics, types of the contained salts and their proportions are importantly counted as threats to the archaeological sites. Another serious effect is induced by the water suction pressure migrating through the building foundations (which ranges between 4 to 5 meters from the ground surface) and the evaporation rate on the stone surface, which is associated to the relative humidity and air temperature [4].

As a common deterioration product on the monuments walls, soot layers can possibly result from atmospheric pollutants, from car exhausts and the products of traditional ovens, together with the human activities inside the archaeological sites. These layers are very thin and their color ranges from pale gray to dark gray and black. They also contain mixtures of dirt accumulations, oily matters and salts [5]. These factors combine with each other in attacking the sandstone monuments, causing disfiguration and disintegration in their external appearance and internal structure [6]. The crystallization process of salts is a dangerous deterioration factor that attacks sandstone monuments due to exerting hydration and crystallization pressures. In addition, this leads to a deterioration process widely known as 'alveolization' [7], which results from the crystallization of crystals inside the pores of stones. The growth of salt crystals depends basically on several factors, including the nature and porosity of stone, the salt type and saturation of the saline solution in addition to the surrounding microclimate.

The studied temples are exposed to extreme weather conditions (e.g. high temperature, dry air, groundwater, etc.), which contribute in forming salt minerals on or beneath the stone surface [8]. The continuous supply of salt ions and the repetition of evaporation and crystallization cycles affect the bonding between the grains and allow the complete fall of some parts of the stone blocks [9-12].

The present study contributes in understanding the weathering mechanisms and the resulted deterioration forms in Edfu and Kom Ombo temples, Upper Egypt. The approach was based on widely used analytical methods including optical microscope (OM), polarized light microscope (PLM), scanning electron microscope equipped by EDX unit (SEM-EDX), and X-ray diffraction analysis (XRD). The obtained results will improve our knowledge about the weathering mechanisms and the formed products affecting the studied sites and will suggest a possible hypothesis for their formation.

1.1. The studied sites

Edfu temple is one of the most important ancient Egyptian temples. It dates back to the 10^{th} year of the reign of Ptolemy III (237 BC), while the construction of the main building was completed during the reign of Ptolemy IV, in 212 BC. [13]. The temple was officially opened in 142 BC.

Kom Ombo temple is located about 97 km to the north of Aswan (about 165 km to the south of Luxor). The temple was dedicated to the gods *Sobek*, and *Haroeris*, and was built in the era of Ptolemy VI (180–145 BC), but the majority of buildings was completed by other kings [14].

Several deterioration forms were observed on the wall reliefs of both temples as shown in Figs. 1 and 2.

1.2. Meteorological data

The climate of Egypt slightly differs from the Mediterranean climate in the northern coastal area to the hot desert climate in the Upper Egypt. Aswan region is rated between the arid desert and the semi-arid zones. The weather of this area shows an average maximum air temperature of 41°C. The distribution of maximum temperature has significantly varied over the last three decades and the reports revealed that the average of the relative humidity (RH%) in Aswan is found to be 66.1±1.2% and 25.2±8.2% [15].

2. Materials and methods

2.1. Materials

A group of samples was collected from highly weathered areas on the stone walls of each temple. The deterioration products were removed gently using a metallic scalpel and kept in plastic tubes. Three samples were collected from Edfu temple including stone, soot layers and salt encrustations. As for Kom Ombo temple, four samples including stone and surface bio-encrustations (e.g. bird droppings), were collected.

Table 1 represents the codes and description of the studied samples.

2.2. Methods

2.2.1. Microscopic examination

The microscopic and petrographic characteristics of the studied stone samples were evaluated through optical and polarizing light microscopes (BD-PL 135) Model 2010, at the laboratories of Abu Qir Higher Institutes at Alexandria. The samples were examined at magnifications between 40x and 100x.

2.2.2. Morphology and elemental analysis (SEM-EDX)

Scanning electron microscope with an EDX analyzer was used for the examination of surface morphology of samples and their chemical content. A JEOL JSM-5400LV scanning microscope and Oxford ISIS Link EDX Detector 'HIGH VACUUM', at a voltage of 15 kV, were used. Investigations were performed at the microscope unit, Faculty of Science, Assiut University.

2.2.3. X-ray diffraction analysis (XRD)

For determining the main mineral phases, the samples were analyzed by a Philips analytical X-ray diffractometer type PW 1710, with Cu tube anode, generator tension 40 kV and generator current 30 mA. Phase identification was obtained through a software "x'pert high score version 2003".

3. Results and discussion

3.1. Visual examination of samples

Visual examination, also with the aid of magnifying lenses, helps in identifying some properties of the building materials and their nature. The stone samples of Edfu temple appeared as fine-sized grains with a white color tone, while some of them showed a yellowish-beige tone. Few stone samples have a color homogeneity while the majority have dashed dark color veins (see images in Table 1). The visual observation on the stone samples of Kom Ombo temple revealed slightly coarser granules than those of Edfu temple. Their general color is earthy and some stone blocks have dark veins, most probably of iron oxides patches, as confirmed by the upcoming XRD method.

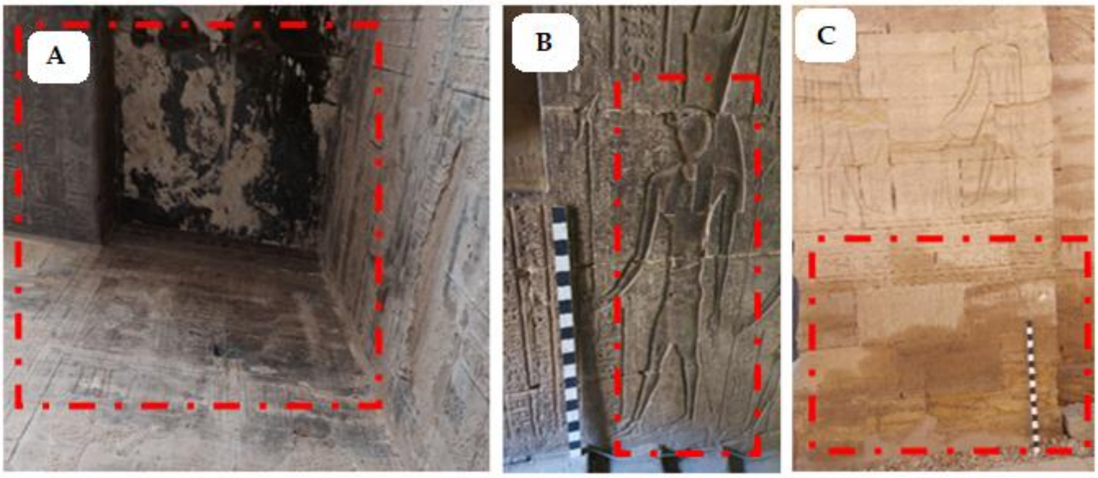


Fig. 1. Some deterioration forms on the wall reliefs of Edfu temple, A, B) Soot damage on the ceiling and temple walls, C) Moistened walls with adhered soiling materials.

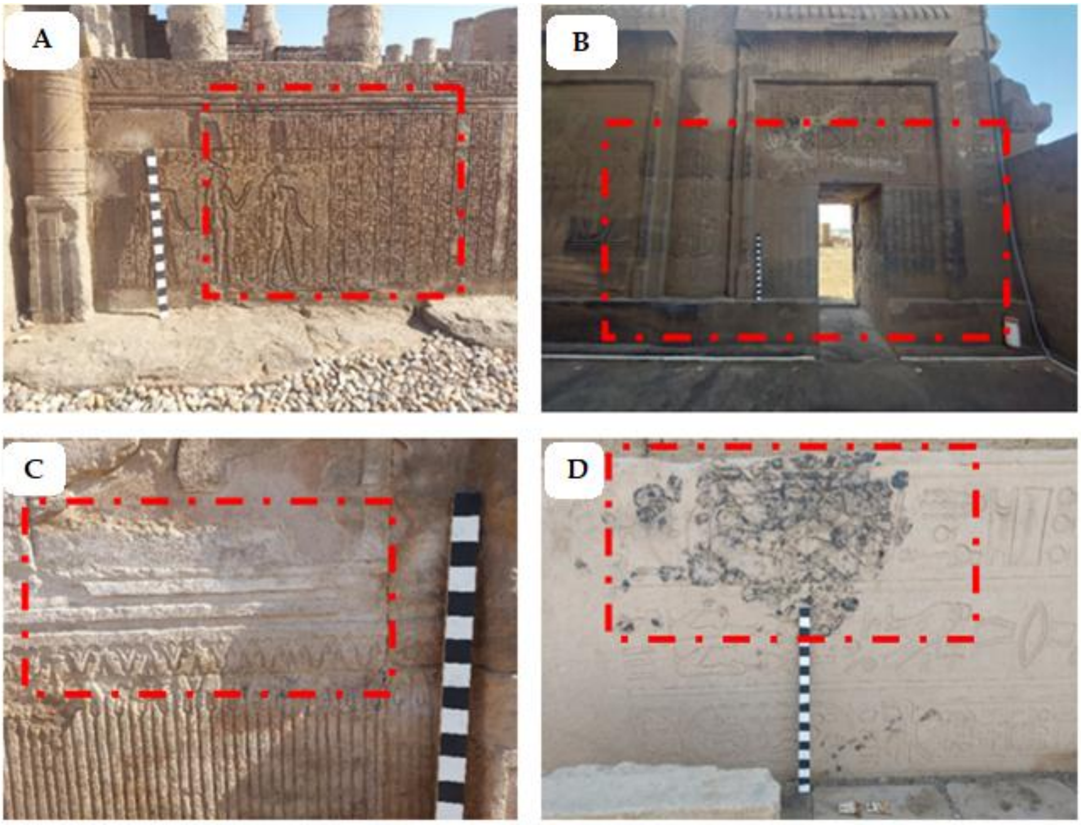
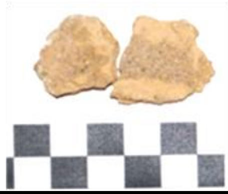


Fig. 2. Different deterioration forms on the walls of Kom Ombo temple, A, B) Moistened walls with adhered soiling materials, C) Salt efflorescence on the walls, D) Accumulation of soot layers.

Table 1. Codes and description of the studied samples.

The studied temple	Sample code	Sample description	Sample image
Edfu	T1S1	Sandstone fraction with a beige color tone from fall-en stone blocks	
	T1S2	Damaged layer covering a stone fragment from the main façade	
	T1S3	Salt deposits from the col-umns hypostyle	
Kom Ombo	T2S1	Sandstone fraction with coarse grains and earthy color tone from highly weathered stone blocks	
	T2S2	Stone fragment rich in iron oxide patches from the lower stone blocks of the temple	
	T2S3	Salts and bird residues from the walls of the col-umns hypostyle	
	T2S4	Accumulation of biologi-cal deposits from the walls of the columns hypostyle	

3.2. Microscopic examination

Fig. 3 represents the microscopic images recorded on the studied samples. Fig. 3 (A - C) represents useful information about the main characteristics of the samples collected from Edfu temple, while Fig. 3 (D - F) shows the observations recorded on the samples of Kom Ombo temple. The results of the microscopic examination of a stone sample collected from Edfu temple (sample T1S1) shows medium-sized granules with a high content of iron oxides (Fig. 3A). Another sample represents the accumulation of soot layers on the temple walls (sample T1S2), which cause disfiguration and clogging the surface's pores (Fig. 3B). The image in Fig. 3C shows crystallized salts on the surface (sample T1S3), which lead to stone flaking. The microscopic examination on the stone sample of Kom Ombo temple (sample T2S1) shows coarse sized and rounded/eroded-edge mineral granules (Fig. 3D). Also, the sample shows a high percentage of the iron oxides cementing material. The examination of sample (T2S2) represents high amounts of iron oxides which disfigure the appearance of stone (Fig. 3E), and bird residues (sample T2S3) formed on the stone surface are clearly observed (Fig. 3F).

3.3. Petrographic examination

Photomicrographs of thin-sections prepared on the sandstone samples, under cross-polarized light, are given in Fig. 4 (images A and B are for samples of Edfu temple at magnifications of 40 and 100x, respectively, while the images C and D are for Kom Ombo temple samples at magnifications of 40 and 100x, respectively). The petrographic observation of the sandstone samples showed fine microcrystalline quartz grains with a sharp roundness. This suggests that the stone type used in constructing both temples belongs to a siliceous quartz arenite [16], which will be confirmed later through the high amount of quartz measured by the XRD analysis of the sandstone samples. In this sandstone type, the quartz cement overgrowth also occurred, and high amounts of dark brown iron oxides patches, which occur as a cement material for the secondary stone pores, are observed [17].

3.4. Morphology and elemental analysis

Table 2 shows a representative EDX analysis (Atomic and Element concentrations %) performed on some deterioration products.

3.4.1. SEM-EDX results of Edfu temple samples

The scanning electron micrographs (SEM) of the studied stone samples showed the distribution of fine-grained quartz and the volume-distribution of the pores (Fig. 5A). SEM micrographs of some weathering products from Edfu temple show crystals of clay minerals which work as micro weathering phenomena while in another micrograph, noticeable salt crystals are observed between the pores causing a deep pitting of the stone grains (Fig. 5B). The large pores of the sample indicate that the cement of sandstone was exposed to many weathering factors. While the morphology of a damaged fragment covered with a thin soot layer is shown in Figure 5 C, D. EDX micro analysis of the sample indicated the chemical arrangement for a damaged sandstone sample collected from Edfu temple (Fig. 6). The element concentrations were as follows: (Si, 43.50%), (Al, 7.64%), (Ca, 14.5%), (Fe, 13.29%), (P, 2.25%), (Cl, 7.90%), (S, 5.15%), (K, 3.85%), and (Ti, 1.92%). The detection of potassium can probably be attributed to clay minerals or potassium-based salts. Also, the concentrations of calcium and sulfur are most likely due to Ca-sulphate phases, while phosphorus is highly attributed to biological processes.

3.4.2. SEM-EDX results of Kom Ombo temple samples

Based on the investigations by the scanning electron microscope (SEM), the stone microstructure shows high porosity and disintegration (Fig. 7 A, B). In the given SEM micrographs, a dense occurrence of salt crystals (Fig. 7 C, D), which cover the pores of the stone, is observed. Most of them appear as large euhedral cubic crystals. As a result of salt crystallization, exfoliation and the complete disintegration of stone blocks occur [18].

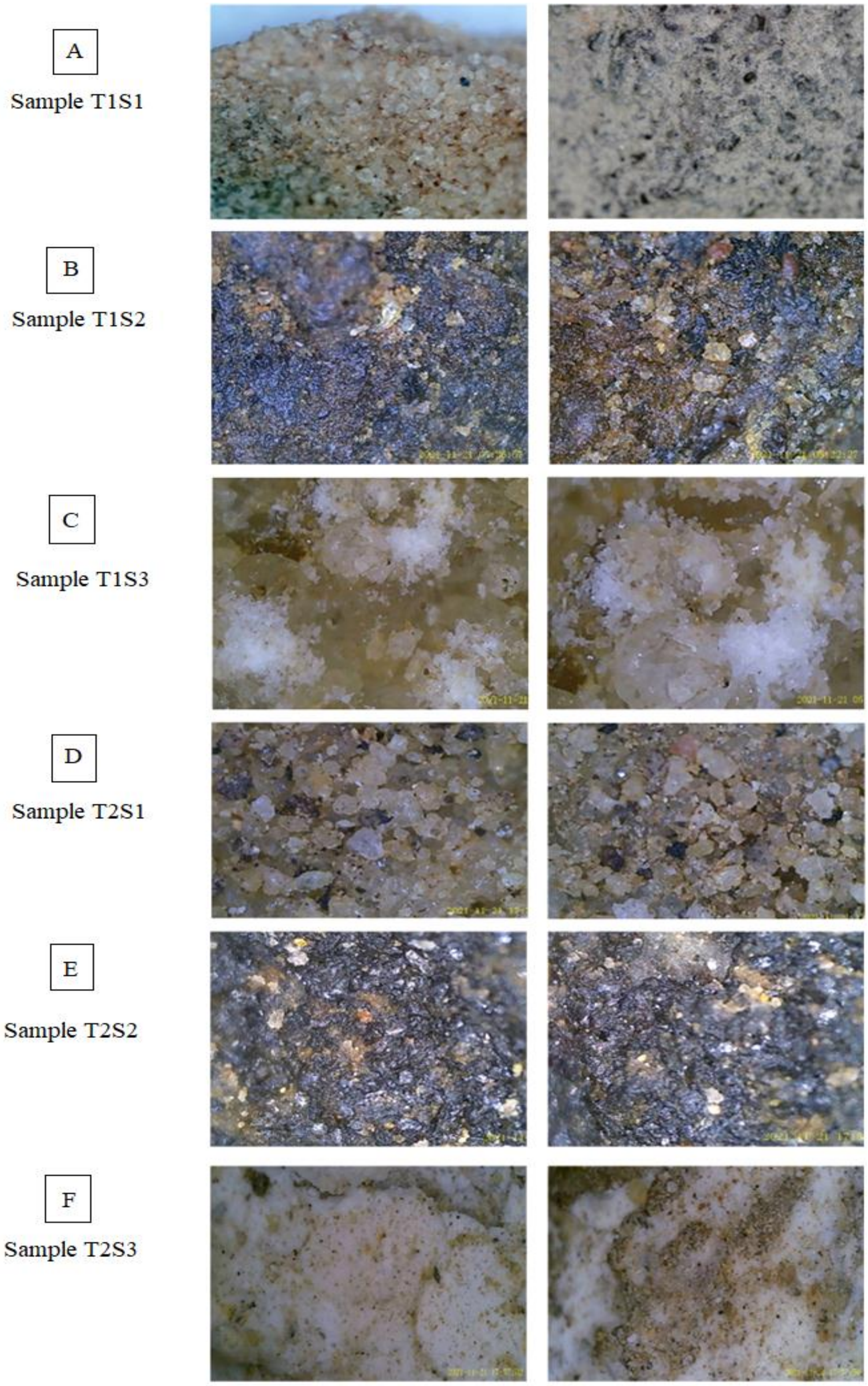


Fig. 3. Microscopic examination on the studied samples: A) Sandstone sample of Edfu temple, B) The soot layers covering the walls of Edfu temple, C) Salt deposits, D) Sandstone sample of Kom Ombo temple, E) Iron oxide damage, F) Bird droppings on the stone matrix.

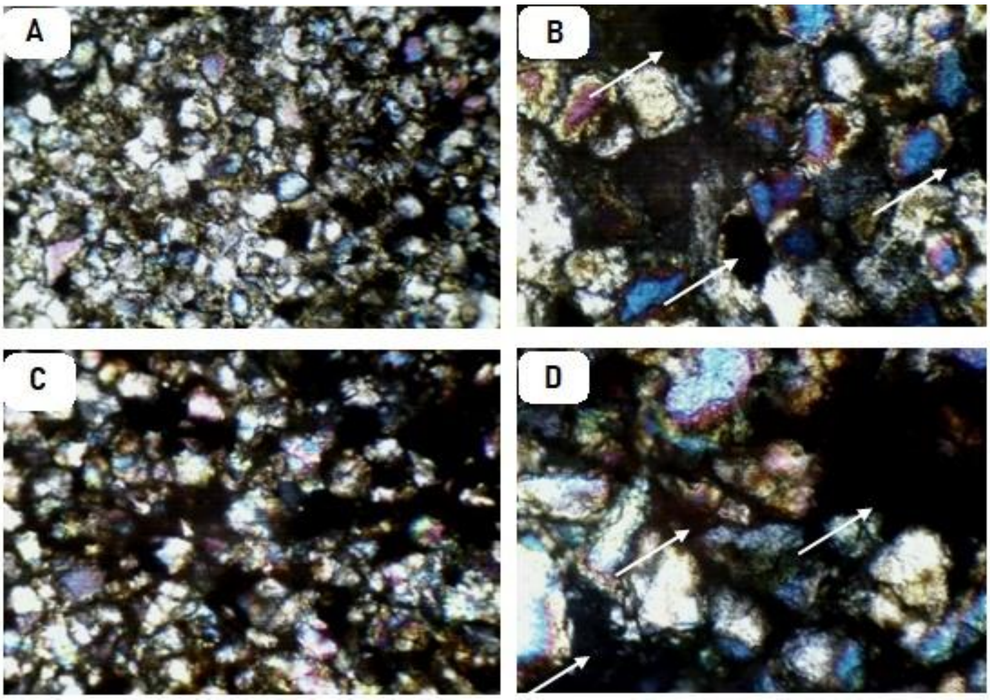


Fig. 4. Photomicrographs in cross-polarized light of thin-sections prepared from the stone samples: A) From Edfu temple with magnification of 40x, which shows medium-sized grains with a sharp roundness, B) The same sample under 100x magnification which shows a high content of iron oxides, C) Stone sample from Kom Ombo temple at 40x magnification which shows coarse rounded-edge quartz grains, D) A high percentage of iron oxides is contained in the section.

Table 2. Representative EDX analysis on some deterioration products.

Element	Edfu Temple (Sample T1S2)		Kom Ombo Temple (Sample T2S2)	
	Atomic %	Element %	Atomic %	Element %
Al K	6.55	7.64	9.21	12.86
Si K	33.93	43.50	29.20	39.70
P K	2.80	2.25	3.40	3.70
S K	4.85	5.15	2.12	2.03
Cl K	8.30	7.90	3.40	3.45
K K	4.78	3.85	0.94	0.94
Ca K	15.8	14.5	3.67	3.57
Ti K	2.60	1.92	1.48	1.21
Mn K	-	-	3.78	2.68
Fe K	20.39	13.29	42.8	29.86
Total	100.00	100.00	100.00	100.00

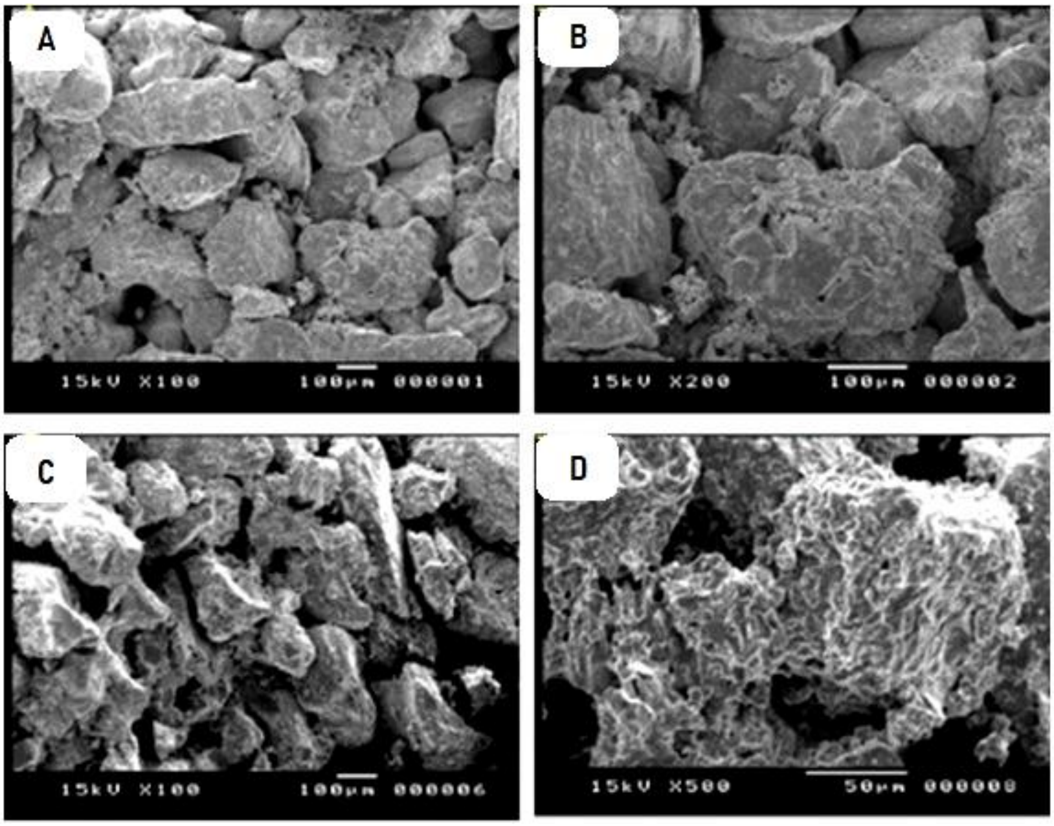


Fig. 5. Scanning electron micrographs (SEM) of a stone sample of Edfu temple (sampleT1S1): A) 100x, B) 200x, and Scanning electron micrographs of damaged and soot layers (sample T1S2) from Edfu temple C) 100x, D) 500x.

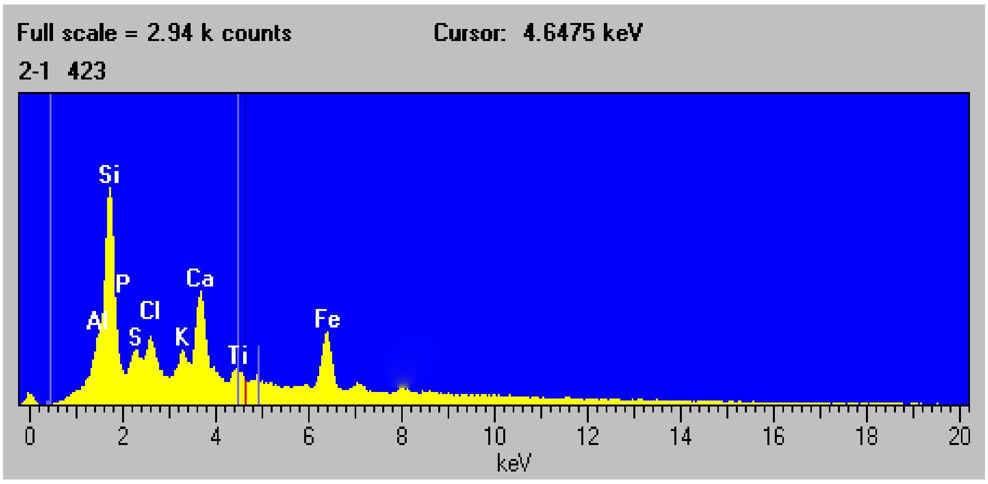


Fig. 6. An energy dispersive X-ray spectrum collected on a damaged layer from Edfu temple.

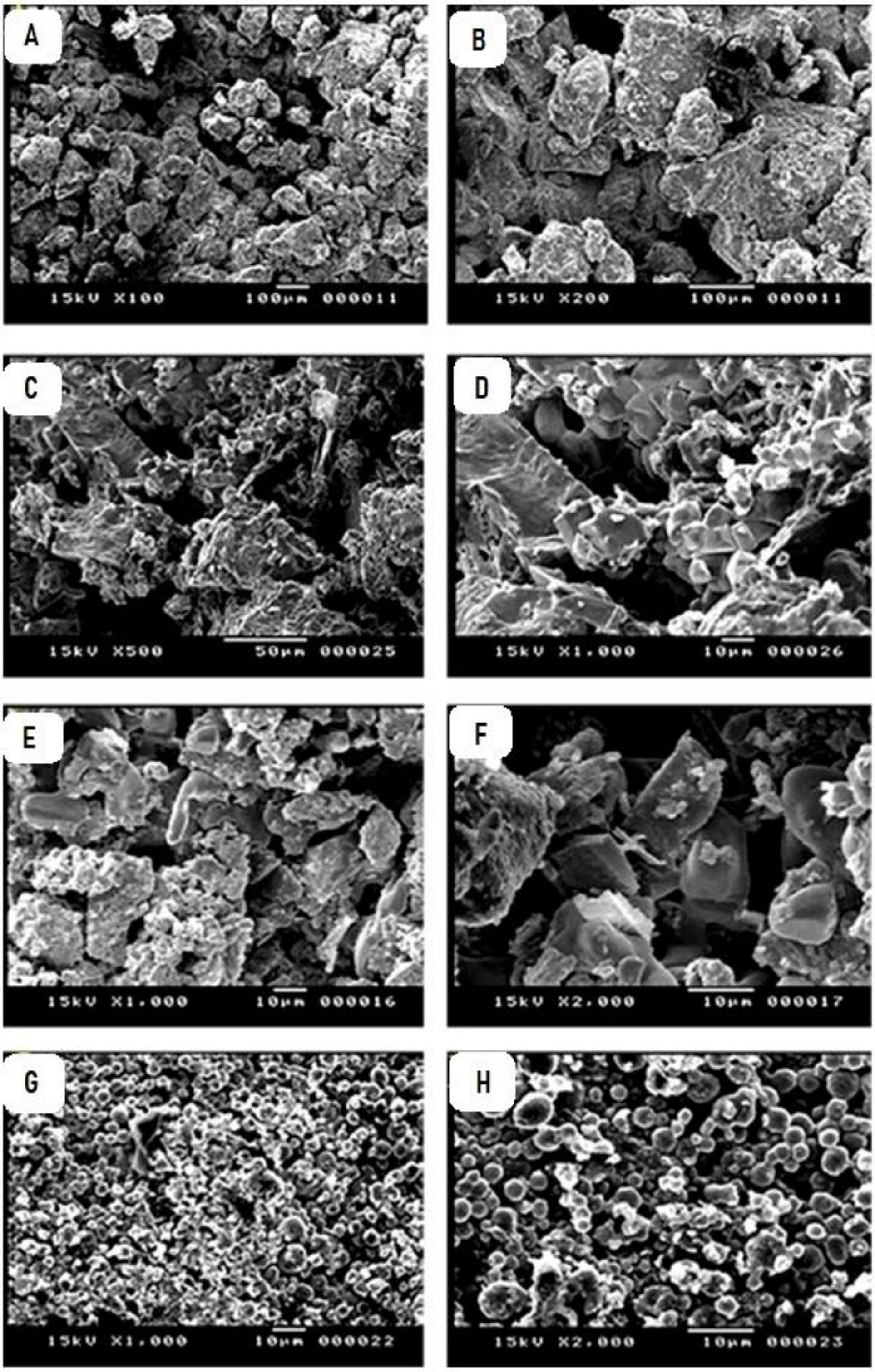


Fig. 7. Scanning electron micrographs (SEM) of a stone sample of Kom Ombo temple (sample T2S1): A) 100x, B) 200x; Scanning electron micrographs of salt accumulations (sample T2S3): C) 500x, D) 1000x, Scanning electron micrographs of iron oxides (sample T2S2): E) 1000x, F) 2000x; Scanning electron micrographs of bird droppings (sample T2S4): G) 1000x, H) 2000x.

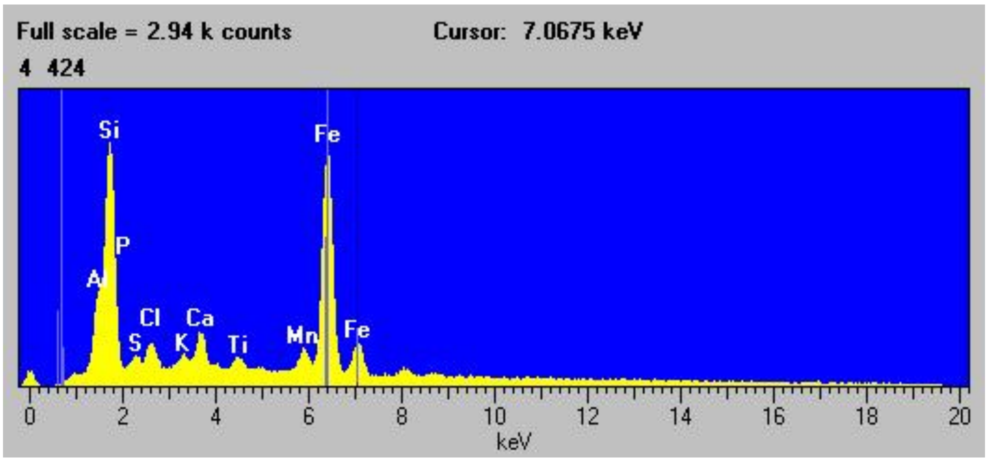


Fig. 8. An energy dispersive X-ray spectrum of a pigmented sandstone from Kom Ombo temple.

The morphological investigation indicated that the pores are affected significantly by the crystallization of salts, which exert aggressive pressures to the inner grains and pores. Fig. 7 (E, F) shows the presence of high amounts of iron oxides with large cubic salt crystals within the stone matrix. Moreover, several cracks of small dimensions are reported in addition to observed modification in the pore structure of stone. Fig. 7 (G, H) revealed the presence of spherical biological accumulations on the surface of the stone which is likely responsible for the phosphate-based minerals. An EDX spectrum of the sample (Fig. 8) indicated the detection of the following elements: Si (39.70%), Fe (29.86%), Al (12.86%), P (3.70%), Ca (3.57%), Cl (3.45%), Mn (2.68%), S (2.03%), Ti (1.21%), and K (0.94%).

In their study of sandstone degradation, Labus and Bochen [19] pointed out that the resistance of stone to weathering is strongly linked to the pore size distribution, the texture and the mineralogical composition of the stone components.

3.5. Mineralogical results of X-ray diffraction analysis (XRD)

Table 3 summarizes the main components obtained from the X-ray diffraction analysis of the studied samples.

3.5.1. Samples of Edfu temple

In this section, X-ray diffraction analysis was used to characterize the minerals contained in the sandstone samples as well as the deterioration products formed on the stone walls of Edfu temple (Fig. 9).

3.5.1.1. Sandstone fraction (Sample T1S1)

The XRD pattern of the sample showed a high percentage of quartz (SiO_2, 94%) (Fig. 9A). Quartz is the main component of sandstone and under microscope it appears as white or colorless crystalline grains [20]. Additionally, calcium silicate ($CaSiO_3$) was measured in the sample. The mineralogical analysis of sandstone samples revealed high occurrence of quartz which dominated the stone type as quartz arenite. Actually, this is in agreement with the petrographic observation on the stone samples. The sandstone samples contain a cementing material of iron oxide (hematite) together with few amounts of clay minerals (kaolinite type).

3.5.1.2. Damaged sandstone with soot layers (Sample T1S2)

X-ray diffraction analysis of the sample showed a major occurrence of graphite (71%). Besides, amounts of quartz (29%) were measured in the sample (Fig. 9B). The accumulation of soot layers on the walls of ancient Egyptian monuments is a very common deterioration aspect. Mainly in the Roman age, a large number of monuments in Upper Egypt was used as shelters. Regrettably due to the human activities inside the archaeological sites, fire hazards and consequently aesthetic damage in form of soot layers are produced. Soot is based mainly on fine carbon particulates with oily matters [21]. These oily matters require further analysis by Fourier Transform Infrared Spectroscopy (FTIR) and chromatographic techniques to determine their nature.

3.5.1.3. Salt deposits (Sample T1S3)

The analysis of this sample showed a majority of halite (NaCl, 88%). Minor minerals of calcite ($CaCO_3$) in addition to potassium nitrate (KNO_3) were also detected (Fig. 9C). Salts of sylvite (KCl) and thenardite (Na_2SO_4) were previously measured in the same temple by El-Gohary [3]. The groundwater, mainly nitrate-rich, provides several dissolved salt ions that reach the temple walls by the capillary action [22]. Niter, along with other salt minerals, was previously reported on the sandstone walls of Thutmosis III festival temple at Karnak [23]. In a study of some deterioration products formed on sandstone structures at Kraków, Poland, Marszałek [24] claimed that the presence of niter salts is highly attributed to air pollution. However, Siedel [25] referred that nitrate salts usually crystallize near the lower blocks of walls and that they result from the effect of soils or the oxidation process of the organic nitrogen of animal waste or biochemical processes.

3.5.2. Samples of Kom Ombo temple

Fig. 10 represents XRD patterns of stone and deterioration products from Kom Ombo temple.

3.5.2.1. Sandstone fraction (sample T2S1)

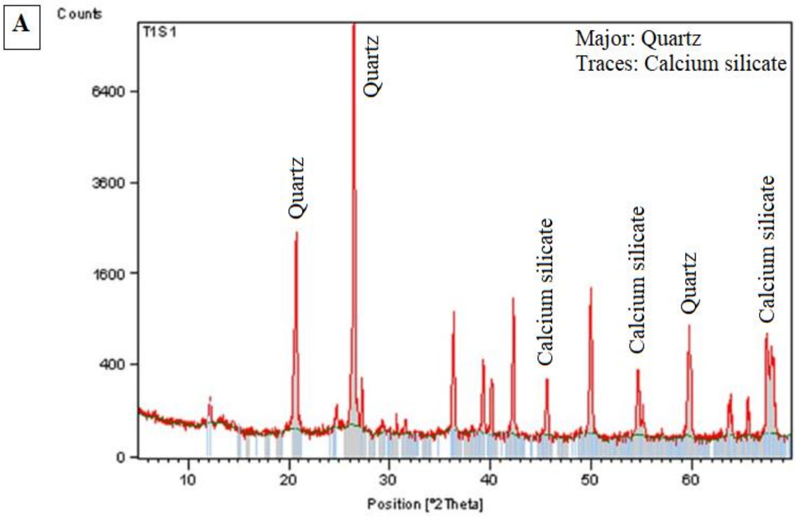
The analysis of this sample revealed quartz (97%) as the major component of the sandstone (Fig. 10A).

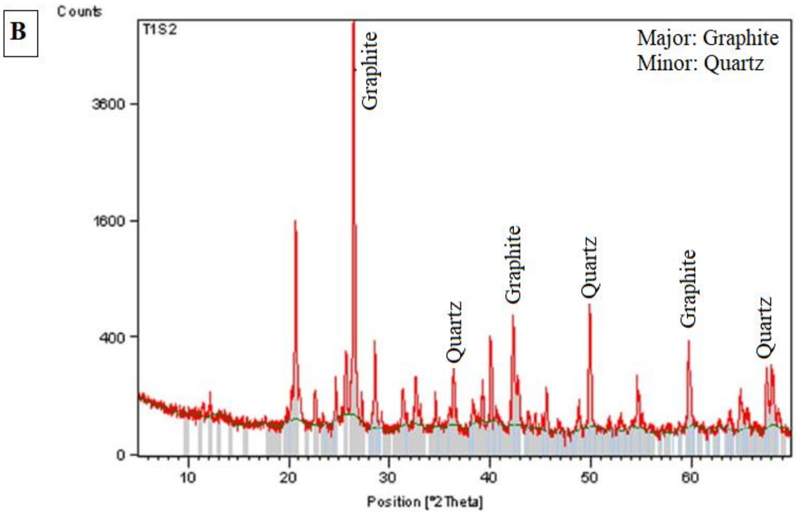
3.5.2.2. Sandstone rich in iron oxides (Sample T2S2)

This sample was taken from a sandstone block in the temple that showed a high percentage of iron oxides. XRD pattern of the sample revealed quartz (47%) and iron oxide mineral of hematite (Fe_2O_3, 41%) as the major components of the sample. Also, clay minerals of kaolinite type ($Al_2Si_2O_5(OH)_4$, 12%) were measured (Fig. 10B). The presence of a high amount of hematite in the sample is caused by the presence of iron-based cement material in the sandstones of Kom Ombo temple. Worthy to note that under variable conditions, the iron-based cementing material may mobilize and transform into the outer surface of stone causing notable aesthetic damages.

Table 3. Summary of the main components measured by XRD of the studied samples.

Temple	Sample code	Sample nature	The measured minerals
Edfu	T1S1	Sandstone	Quartz, Calcium silicate
	T1S2	Damaged layer with soot	Graphite, Quartz
	T1S3	Salt deposits	Halite, Calcite, Niter
Kom Ombo	T2S1	Sandstone	Quartz
	T2S2	Iron oxides	Quartz, Hematite, Kaolinite
	T2S3	Salt and bird residues	Quartz, Iron phosphate
	T2S4	Biological deposits	Quartz, Kaolinite





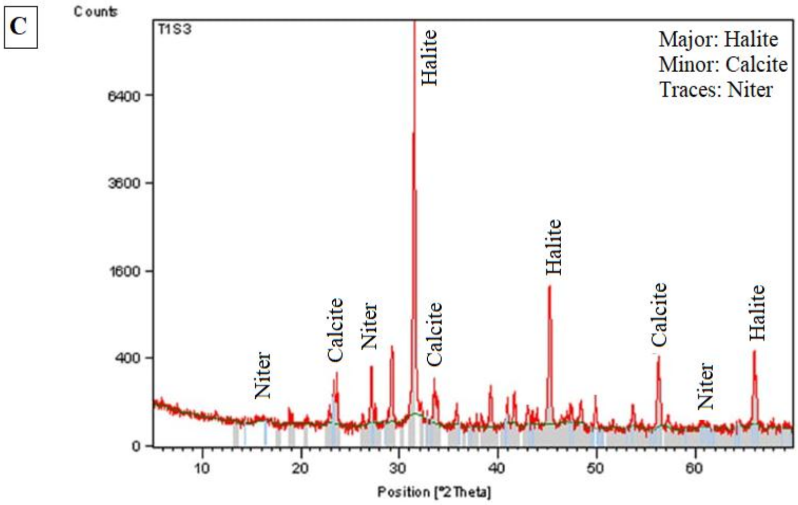


Fig. 9. X-ray diffraction patterns collected on samples of Edfu temple: A) Sample T1S1, B) Sample T1S2, C) Sample T1S3.

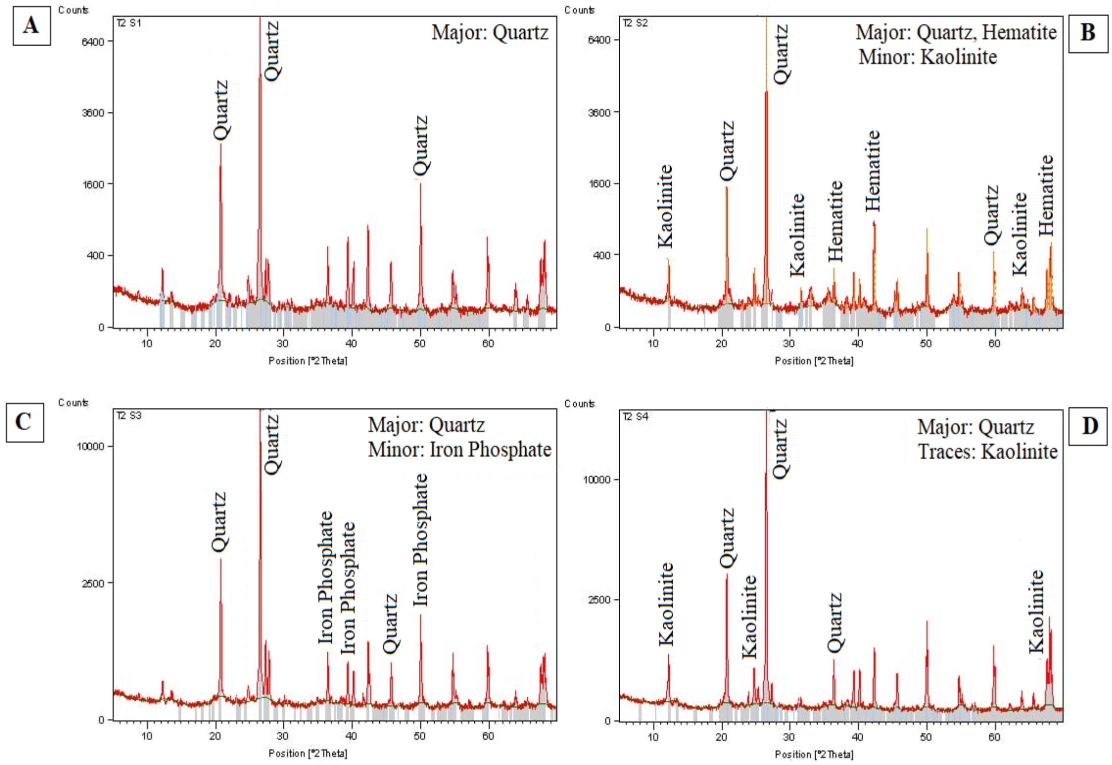


Fig. 10. X-ray diffraction patterns collected on samples of Kom Ombo temple: A) Sample T2S1, B) Sample T2S2, C) Sample T2S3, D) Sample T2S4.

3.5.2.3. Salts and bird droppings (Samples T2S3 & T2S4)

The sample (T2S3) was collected form highly damaged sandstone reliefs with accumulations of bird droppings. The analysis showed the presence of quartz (69%) and iron phosphate $Fe_2(PO_4)_3$, (27%) (Fig. 10C). The results of the X-ray diffraction analysis of sample (T2S4) indicated quartz together with kaolinite are the components contained in the sample (Fig. 10D). The presence of iron phosphate as by-products in sample (T2S3) is probably due to the effect of bird residues, eventually lead to flaking of the stone and closing of its pores [26, 27]. Under the normal environmental conditions, the asuricite crystals aqueous suspension of bird droppings is greatly insoluble. Moreover, a thin layer of urea and uric acids covers this suspension, and in rainy weather, the dissolution occurs and re-crystallization of white staining crystals is expected. In addition, other effects are associated to the deposition of bird droppings such as encouraging the biodeterioration process on the stone structures. The microbiological growth provides a large number of organic acids which react with the stone components. In a study of the biological damage of the mortuary temple of Medinet Habu at Luxor, Ahmed et al. [28] reported that the excrements of wild pigeons cause the decomposition of the sandstone structures due to an acidic reaction. These excrements contain several components, one of them is the phosphoric acid (H_3PO_4). This acid can react easily with stone and the iron-based cement material to form new deterioration products. Additionally, the bat guano resulting from the common bat colonies can provide another source of phosphate since its concentration reaches about 25% of the guano mass [29]. Further, the irrigation water from the cultivated lands may supply additional phosphate ions (PO_4^{3-}) that react with stone components, and consequently phosphate-based components are formed [30].

4. Conclusion

In the present approach, a number of widely used analytical methods was applied to study sandstone and weathering products formed on the wall reliefs of Edfu and Kom Ombo temples, Upper Egypt. The microscopic description of samples helped to maintain their grain characteristics. While the morphological aspects of samples together with their chemical content were routinely evaluated by scanning electron microscope. The stone types used in the temples of Edfu and Kom Ombo were identified as quartzitic and quartz-arenite sandstones. The quartz grains in the sandstone samples of Edfu temple showed a percentage of 94%, while it reached 97% in Kom Ombo temple. The identification of the deterioration products formed on Edfu temple showed the presence of halite and potassium nitrate salts. While unusual occurrence of iron phosphate was found on the walls of Kom Ombo temple. Thus, the phosphorus produced from the bird droppings reacts with the stone components, mainly the ferric cementing material, to produce such phosphate-based compounds. The environmental impacts affecting the studied sites, in combination with the biological effects of bird droppings, play an important role in the deterioration process and the formation of the aforementioned minerals. The deterioration process should be eliminated through the full control of the ground water penetration beneath the foundations of the studied temples. In a parallel way and taking into consideration the ecological prospective, immediate efforts should be directed to monitor and stop the occupation of the wild pigeons of several parts of the studied temples.

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