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Morphological, chemical and mineralogical characterization of deterioration products from the tomb of Kheruef (TT192), (Luxor, Egypt)

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Abstract

The present paper aims at characterizing some deterioration products formed on the decorated walls of the tomb of Kheruef (TT192), El-Assasif district, El-Qurna necropolis (Luxor), Upper Egypt. Mainly, the deterioration phenomenon occurring in the area is closely related to the crystallization of salts. The local supply of salt ions usually comes from the abundance of soluble salts in the bed-rock and ground layers. The morphology and the microanalysis of the contained mineral phases in the damaged layers were carried out using scanning electron microscopy (SEM) equipped with an energy dispersive X-ray detector (EDS). The mineralogical composition of the samples was determined using X-ray powder diffraction analysis (XRPD), while the petrographic examination on the prepared thin-sections was carried out using polarized light microscopy (PLM). The results show that the limestone consists of fine-grained calcite crystals embedded in a micritic matrix rich in quartz and fossils (e.g. *foraminifera*). Also, the results allowed the determination of the main deterioration mechanisms affecting in the tomb. Based on the results of these analyses, different salt minerals were identified as halite (NaCl), gypsum ($CaSO_4 \cdot 2H_2O$), and bassanite ($CaSO_4 \cdot 0.5H_2O$). The obtained results will be used in the conservation-restoration interventions of the tomb.

Key words: Tomb of Kheruef, Luxor, Salts, SEM-EDS, XRPD, Petrographic examination.

Introduction

Historical background

The necropolis of ancient Thebes is located on the western bank of the Nile River of the city of Luxor (about 650 km south of Cairo, Egypt). The well-known Nobles tombs of Thebes are scattered along the eastern slopes of the Theban Mountain between the Queen's Valley and the King's Valley. There are around five hundred private tombs - there are at least 415 cataloged tombs, designated TT for Theban Tomb - belonging to officials of the New Kingdom (c.1570.1070 B.C.) (Kamil, 1976). The private tomb of Kheruef (Kheruf), TT 192 in the Assasif necropolis, is the largest such private tomb on the West Bank of

Luxor (The Epigraphic Survey, 1980). The tomb was first explored by the German Egyptologist Adolph Erman in 1885. In the 1940s, Alan Gardiner also worked the tomb and then, after it was robbed in the 1940s, the Egyptian Department of Antiquities in association with the Epigraphic Survey of the University of Chicago cleared, recorded, and finally published their results in 1980. Kheruef, also called Senaa, was "Steward of the Great Royal Wife, Tiye", "Royal Scribe" and "First Royal Herald" during the reign of Amenhotep III (1388-1348 B.C.) and Amenhotep IV (Akhenaten, 1360-1343 B.C.). The tomb complex is very large, as befits a man in his exalted position, but was unfinished at the time of his death (he was never buried in the tomb) and most of the inner rooms of the structure are closed off. Kheruef's tomb is entered down a staircase and passage which leads to a large open court leading to several other later tombs (http://egyptsites.wordpress.com/2009/02/07/tomb-of-kheruef-tt192/).

Figure 1 shows a map of the West bank area at Luxor, and the arrow refers to the location of the tomb investigated in this work.

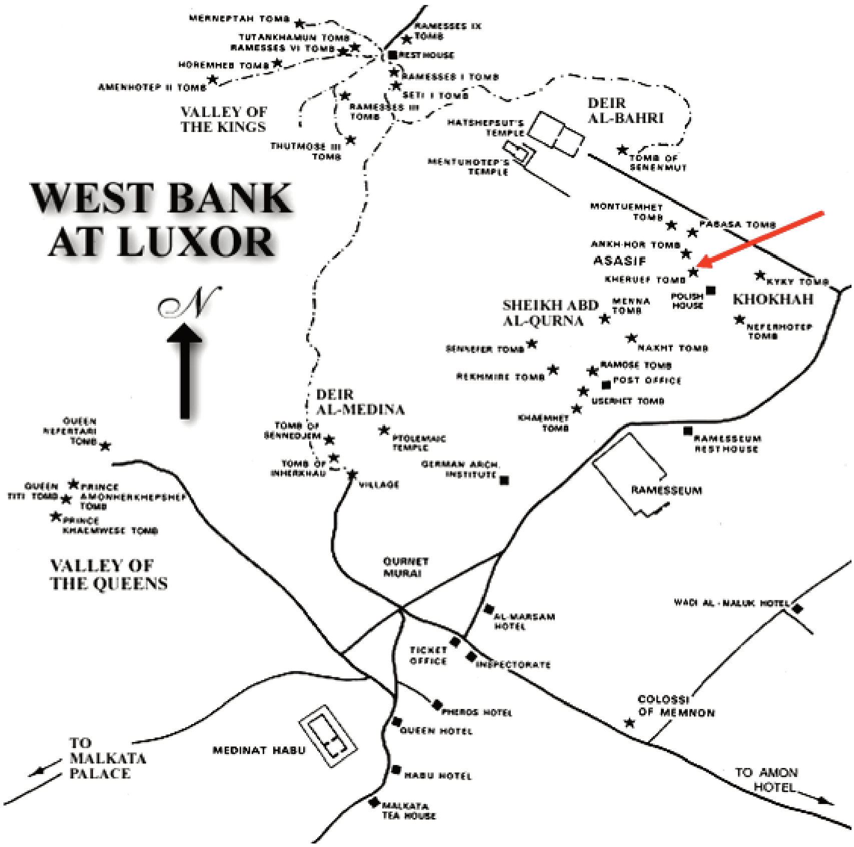


Figure 1. A map of the West bank area at Luxor, and the arrow refers to the location of the tomb investigated in this work (after: http://www.touregypt.net/map18.htm).

Geological background

Figure 2 shows a geological map of the studied area. The tomb of Kheruef is built of stone types belonging to the limestone formations of Thebes Mountains. These Mountains are composed of 350 m thick Eocene marls and limestones overlying the 60 m thick Esna shale Formation (Said, 1962). The lower levels of the Theban Formation are composed of slightly clayey, sub-chalky limestone, which serves to enclose a few bedrock layers of flint nodules and becomes more massive at greater depths (Guillaume and Piau, 2003).

Deterioration factors

All the materials used in the construction of wall paintings undergo degradation when exposed to aggressive environments. The rate and symptoms of such process are influenced by a number of factors, including the properties of the material itself, the natural factors and human actions. The climatic conditions of the area play an important role to enhance several deterioration mechanisms affecting the monuments found in the West bank of Luxor. The monthly average of air temperature at the studied area ranges between 12 and 32 ºC, the maximum relative

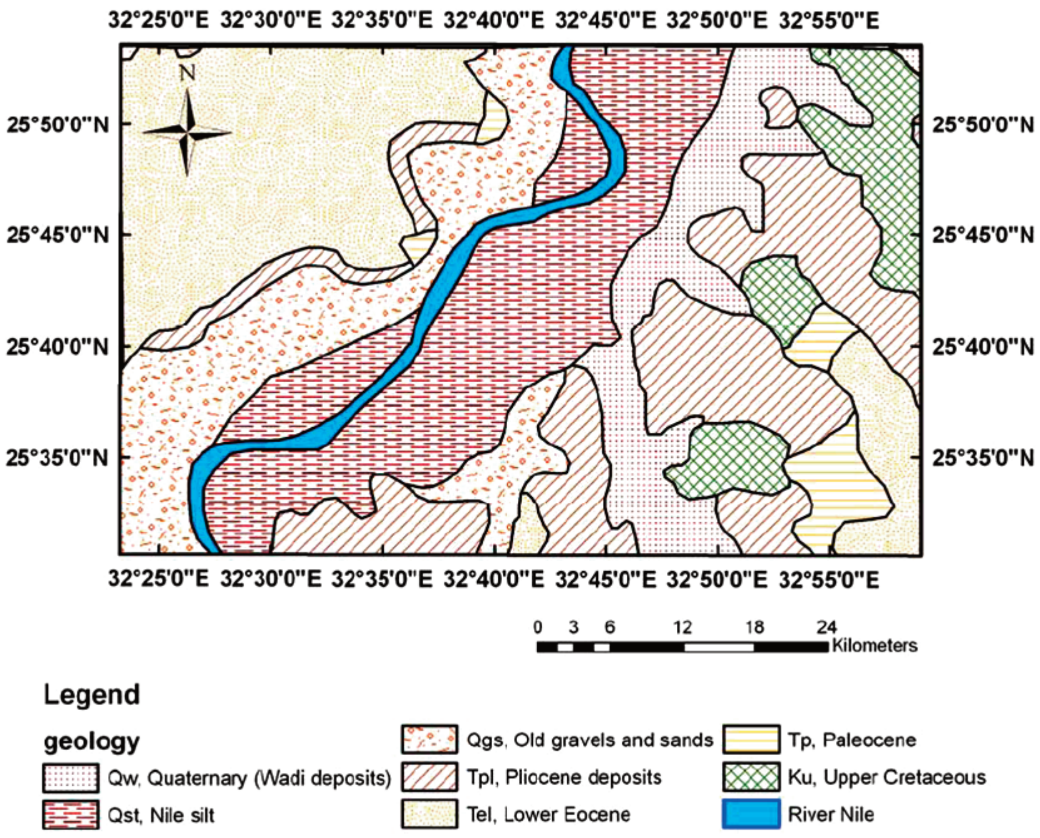


Figure 2. A geological map of the studied area (after: Geological Survey of Egypt).

humidity (RH %) reaches 50% in December and 29% in August. The most significant environmental process occurring in the site is the crystallization of salts. Crystallization of salts in building materials produces a destructive effect, as under favorable conditions, some salts may crystallize or re-crystallize to different hydrates, which occupy a larger space and exert additional pressure producing cracking, powdering, and flaking (Rodriguez-Navaro and Doehne, 1999; Pel et al., 2002). According to Kotulanová et al. (2009), there are several potential sources of inorganic salts in wall paintings. Perhaps most obviously, are the materials used for the artwork itself; stones, bricks, plaster, and mortar, can all contain inorganic salts. Salts can be transported in porous building materials only if they are dissolved in water, and they may crystallize on surface of the stone with various forms and habits, or below the surface producing different pressures on the inner matrix (Arnold and Zehnder, 1987). According to Marey Mahmoud (2010), the biggest hazard to the area is the flood water that penetrates into the structures. Furthermore, the deterioration becomes more effective in the presence of swelling clay minerals and soluble salts in the geological structure. In the Valley of the Kings, the storms caused the flooding of several tombs, the tomb of Bay was the most heavily hit, inspectors measured 1.40 m of water in its lower chambers. The tomb of Setnakht (KV 14), the tomb of Seti II (KV 15), the tomb of Amenhotep II (KV 35), and the tomb of Horemheb (KV 57), among the others, received smaller amounts of rain and debris (El-Bayomi, 2007). Adsorption of gases and vapors in the internal surface of pores and capillaries plays a fundamental role in the decay process. Adsorption causes the molecules of water vapor and airborne pollutants to penetrate the internal micro cavities. There, condensation occurs before the environmental relative humidity reaches the 100%, so that, many physio-chemical processes may occur, such as the chemical weathering of carbonate stones and lime mortars which leads to the formation of gypsum crystals inside the carbonatic rocks (Del Monte and Sabbioni, 1984; Camuffo and Valcher, 1986; Ausset et al., 1999). The main objective of this study was to characterize some salt encrustations formed on the walls of the tomb of Kheruef (TT192), El-Assasif district, El-Qurna necropolis (Luxor), Upper Egypt. In this work, SEM–EDS, XRPD and the petrographic examination were utilized to study the samples.

Figure 3 shows the decorations and deterioration aspects on the walls of tomb of Kheruef (TT192) (a): the salt layers cover the painted bass releifs of the tomb, (b): hard crusts are observed on the surface, (c, d): macro-cracks spread on the surfaces.

Materials and methods

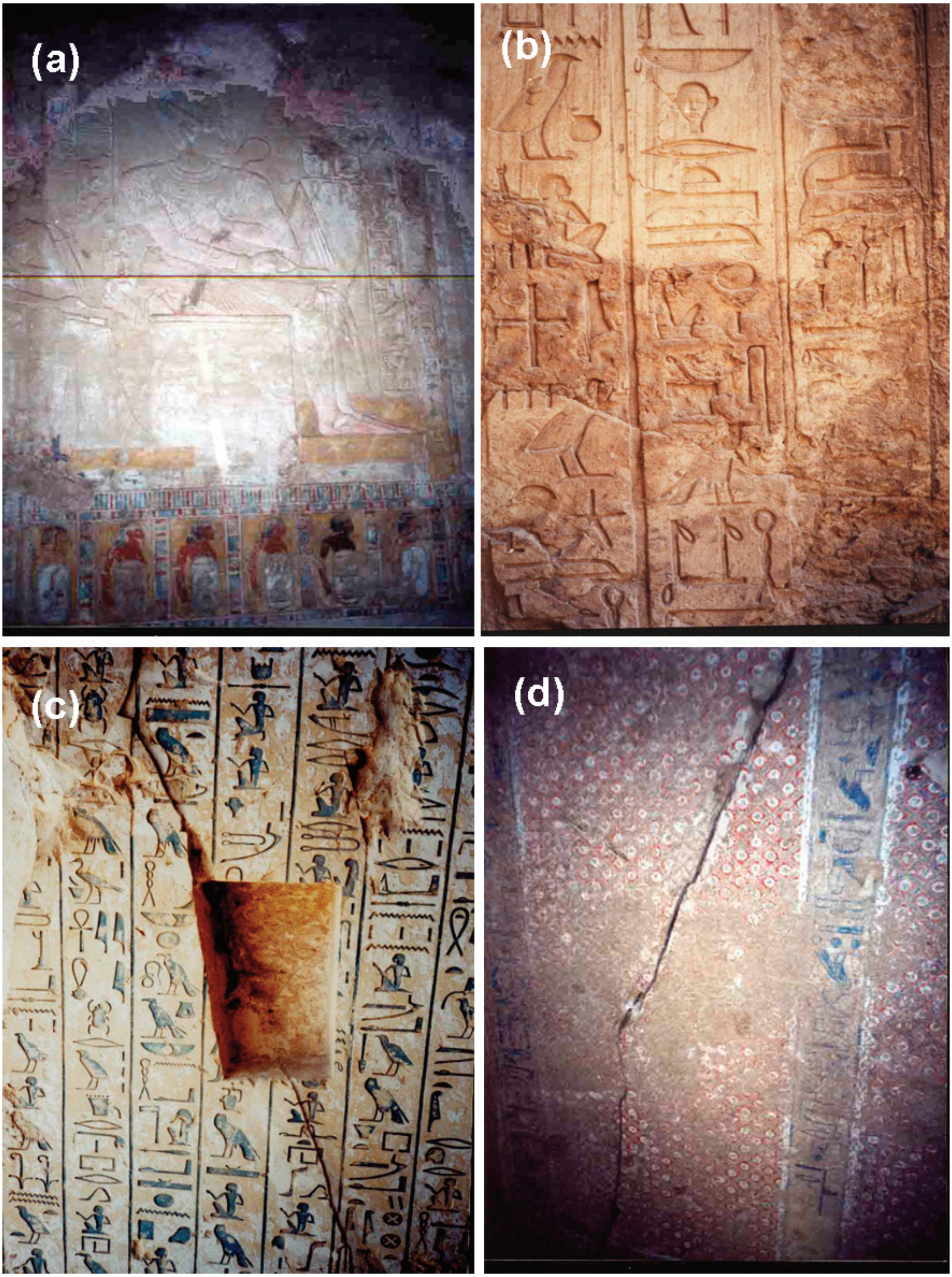
Sampling

The observation of the damaged walls led to diagnose the main weathering forms such as blistering, disintegration of stone, and forming of different crusts on the walls. Specific material data about the geological structure, the metrological data of the region, and the history of conservation projects of the studied tomb were collected in order to clarify the deterioration phenomena affecting the tomb. Micro-samples of the different crusts were carefully collected using two methods: firstly, fragments were removed with a scalpel, especially from the friable crusts; secondly, few powders have been scraped off from the hard crusts.

Analytical techniques

Scanning electron microscopy with an EDS microanalysis detector (SEM-EDS).

The microstructure and the morphological features were determined by a JEOL JSM-840A scanning electron microscope in the backscattered electrons mode (BSE). The microanalysis was carried out using an energy



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dispersive X-ray (EDS) Oxford ISIS 300 micro analytical system, with a detection limit of less than 1% depending on the element. The accelerating voltage was of 20 kV, the probe current of 0.5-45 nA, the working distance of 20 mm, and the counting time of 60 s real time. The matrix correction protocol was ZAF correction being provided on-line. The samples were coated with carbon, using a Jeol JEE-4X vacuum evaporator. Analyses in the backscattered electrons mode (BSE) on polished cross-sections were used for elementary semi-quantitative chemical study of the layers.

X-ray powder diffraction analysis (XRPD)

In order to determine the mineralogical composition of the damaged layers, the collected samples were ground into powder in an agate mortar and were studied by XRPD using a Philips PW1710 diffractometer with Ni-filtered CuK_α radiation on randomly oriented samples. Step size: 0.01° 2θ, 3–63° 2θ angular range, and 0.02° 2θ/sec scan speed. The XPowder analytical software was used for the semi-quantitative determination of the mineral phases by automated processing of the diffraction data.

Polarized Light Microscopy (PLM)

The prepared thin-sections were observed using a Leitz Orthoplan polarizing microscope in order to characterize their structure, texture, grain size and mineralogical association.

Results

The EDS microanalysis and the mineralogical composition determined by XRD method of the studied samples are reported in Tables 1 and 2, respectively.

Petrographic examination

The photomicrographs obtained on polished thin-sections of limestone samples collected from the bed-rock of Theban Mountain are given in Figure 4. The microscopic observations show the fine-grained calcite embedded in a micritic matrix rich in amorphous silica and fossils (e.g. *Globigerina sp*.) (Figure 4a). Foraminifera, large grains of quartz can be observed (Figure 4b). Typical planktonic foraminifera embedded in a biomicritic matrix are clearly seen in Figure 4c. Some veins in the matrix were re-filled with sparry calcite grains (Figure 4d).

Morphology and microanalysis

Although both operating modes (secondary electron “SE” and backscattered electrons “BSE”) are complementary, when working with patinas and crusts, the latter mode usually provides more information than the former. Therefore, the different grey range, a consequence of the atomic numbers of the chemical elements constituting the minerals, let us distinguish the different layers with different elemental composition (Vazquez-Calvo et al., 2007). Analyses in the backscattered electrons mode (BSE) on polished cross-sections are used for elementary semi-quantitative chemical study of the layers of the patina. SEM analysis in the secondary electron mode (SE) on unpolished sections or pieces is used for microscopic observations of the microstructure and the texture of the damaged layers. Figure 5 displays some of the SEM and BSE micrographs obtained on the damaged layers. The SEM investigation of the external surface of the hard crusts (Figure 5a) shows gypsum crystals embedded in clayey clusters and crystallization of salts mainly of halite (sodium chloride). The BSE image obtained on a cross-section of the sample (Figure 5c) shows an irregular and heterogeneous layer which is partially detached from the stone substrate below. This layer is composed mainly of crystals of gypsum scattered within the layers. SEM investigation of a salt layer (Figure 5b) shows the crystallization of different salts, with waxy and hollow-faced halite covering the stone surface. The BSE image obtained on a cross-

Table 1. Qualitative SEM-EDS analysis (compound %) of the weathered layers.

Sample/Oxide	Na_2O	MgO	$A1_2O_3$	SiO_2	SO_3	K_2O	CaO	TiO_2	Fe_2O_3	C1
Hard crusts	1-12	1-2.5	2-7.5	6.3-8.9	17.4-37	1.2-13.8	15.5-32	0-1.5	1.9-7.9	1.7-11.6
Salt layers	11-37	0-1.5	1.2-6.4	7.2-28	7.9-19	1.4-6.1	6.2-28.4	0-2.3	0-4.7	8.6-34.7
Damaged stone	1.6-8	0-6.5	0-4.3	3.2-14.2	10-43.5	24.7-52	0-1.5	0-2.10	-	3.3-6.6

Table 2. The mineralogical composition of the studied samples by XRPD.

Sample/Component	Gy	Ba	Q	An	Cc	Ha	Kf	Pl	Cl	G
Hard crusts	+++	+	+++	–	–	–	–	–	–	–
Thick salt layers	++	+++	++	–	–	–	–	–	–	–
Thin salt layer	++	+++	++	–	–	–	–	–	–	–
Damaged stone	–	–	++	++	+++	++	–	–	+	–
Soiling layer	–	–	+++	–	++	++	+	++	+	+

Gy=gypsum; Ba= bassanite; Q=quartz; An=anhydrite; Cc= calcite; Ha=halite; Kf= feldspar; Pl= plagioclase; Cl=clay minerals; G=graphite. – = absent; + = traces; ++ = minor constituent; +++ = major constituent.

section of the sample (Figure 5d) shows the fairly thick layer of salt lying on the stone substrate. The microanalysis spectrum of the salt layer (Figure 6a), obtained by EDS, shows quite high concentrations of chlorine and sodium and low concentrations of sulfur, calcium, silicon, aluminum, and titanium. The microanalysis spectrum of the hard crust (Figure 6b) shows that silicon, aluminum, sulfur, and calcium are the prevailing ions, with minor chlorine, potassium, iron, and sodium content. Traces of magnesium and titanium were also detected.

Mineralogical characterization

Representative XRPD patterns of the studied salt samples are given in Figure 7. XRPD analysis of the salt layers shows that they consist mainly of bassanite ($CaSO_4 \cdot 0.5H_2O$). Minor amounts of quartz (SiO_2), halite (NaCl) and gypsum ($CaSO_4 \cdot 2H_2O$) were also observed (Figure 7a). XRPD analysis of the hard crust samples (Figure 7b) shows that they consist mainly of quartz (SiO_2) and gypsum. Traces of bassanite were also found. XRPD analysis of damaged stone samples shows that they consist mainly of calcite ($CaCO_3$), with minor amount are halite, anhydrite ($CaSO_4$), and, quartz. Traces of clay minerals were also found. XRD data of a soiling layer covering the walls shows that the sample is consists mainly of quartz, with minor halite, calcite, plagioclase (albite, $NaAlSi_3O_8$). Traces of potassium feldspar (microcline, $KAlSi_3O_8$), graphite and clay minerals were also observed.

Discussion

The morphological, microanalytical and mineralogical analyses of the deterioration layers

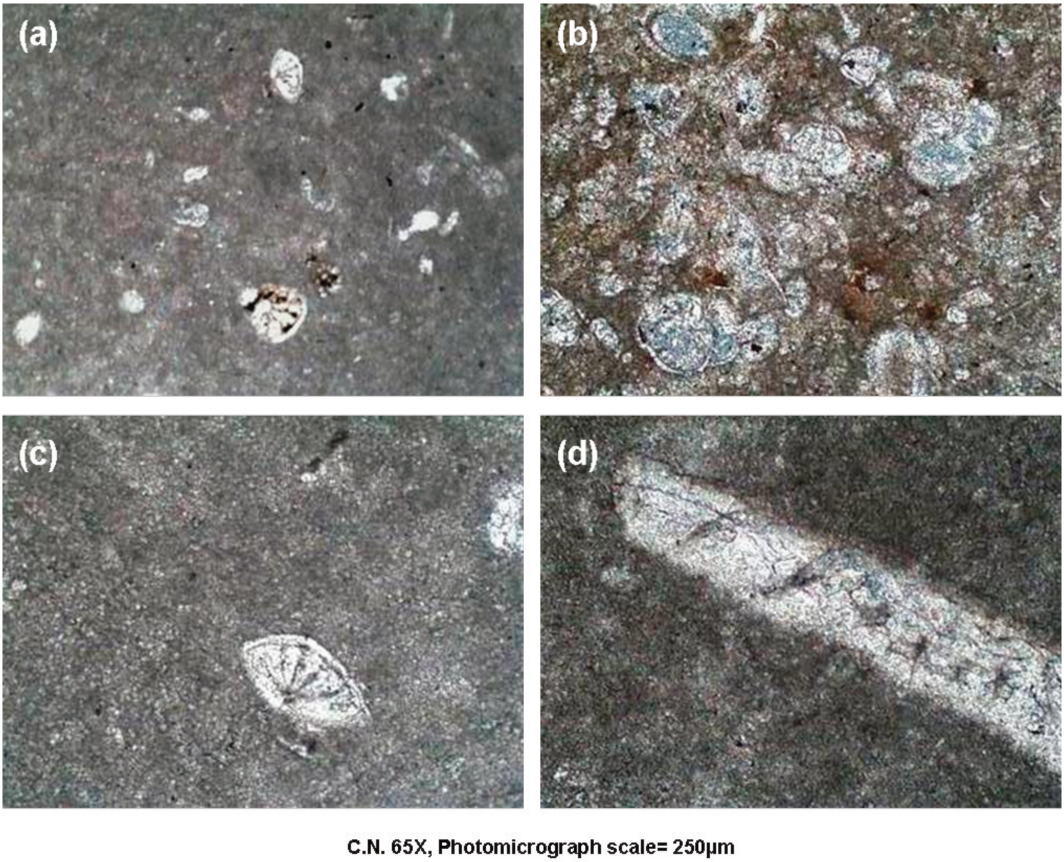

Figure 4. Photomicrographs (C.N., 65X) of thin-sections prepared on the limestone samples. (a): the fine-grained calcite embedded in a micritic matrix rich in amorphous silica and fossils. (b): Foraminifera, large grains of quartz. (c): Typical planktonic foraminifera embedded in a biomicritic matrix. (d): Some veins in the matrix were re-filled with sparry calcite grains.

show that the crystallization of different types of salts is the main reason of the damage process in the tomb. XRD revealed the occurrence of two calcium sulphates (the hemi hydrate phase bassanite and gypsum) and the soluble salt sodium chloride (halite). Gypsum probably comes from the bedrock or the stone itself, but the chemical transformation of the white pigments of calcium carbonates and the lime-based plasters used for the construction of the wall paintings into sulphates, provides another source of gypsum which will re-crystallize in form of different salt phases (bassanite and anhydrite) depending on the environmental conditions. Transformation of gypsum into bassanite or anhydrite occurs at relatively high ambient temperatures and/or high brine salinities (e.g., 45 ºC at a_{H2O} 0.88) (Mees and De Dapper, 2005). The dehydration of gypsum to hemihydrate requires lower relative humidity than the dehydration of gypsum to anhydrite. Although, gypsum also starts dehydration to

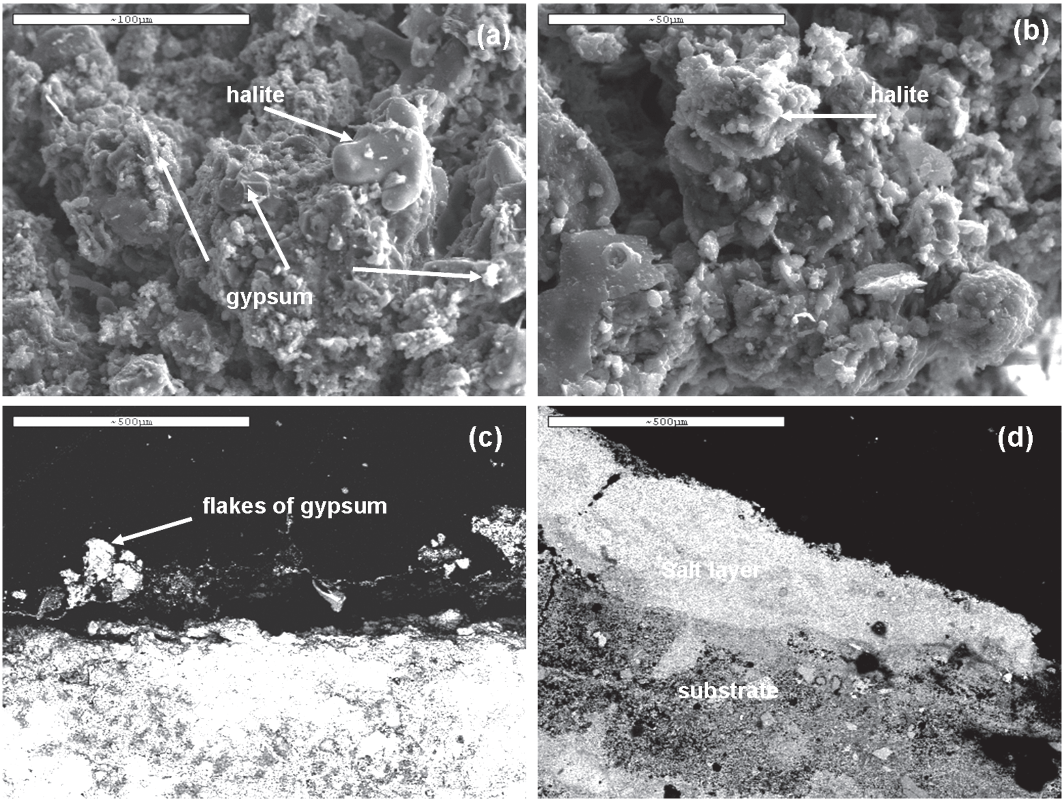

Figure 5. Some SEM and BSE micrographs obtained on the damaged layers. (a): SEM micrograph of the external surface of the hard crusts. (b): SEM micrograph of a salt layer shows the crystallization of different salts, waxy, and hollow-faced halite covers the stone surface. (c): BSE image obtained on a cross-section of the hard crusts. (d): BSE image obtained on a cross-section of the salt layer.

form the hemihydrate (bassanite) at 42 ºC (Carbone et al., 2008; Ballirano and Melis, 2009), and only in few instances has this salt been found in nature, such as in semiarid gypsum deposits in south Tunisia due to the extreme conditions in this area where temperatures can range up to 70 or 80 °C with very low humidity can explain this formation (Charola et al., 2007). As a result of the arid climate in the studied area and the very low humidity in summer months, similar reactions are expected. In addition, the presence of solutions of other salts such as halite (NaCl) which also was found in all the studied samples can lead to the dehydration of gypsum as a result of the lower water vapor pressure of these solutions. Because of its low mobility, gypsum tends to accumulate in large pores, and in an enhanced moisture retention thus facilitating gypsum re-crystallization and development of larger crystals (Steiger, 2003). Halite, sodium chloride was identified in all the studied samples. According to Wüst and Schülchter (2000), the source of the salts in the Thebes area is mainly the bedrock. Sodium chloride is a highly mobile and easy soluble salt (Leisen et al., 2008). Salts have long been known

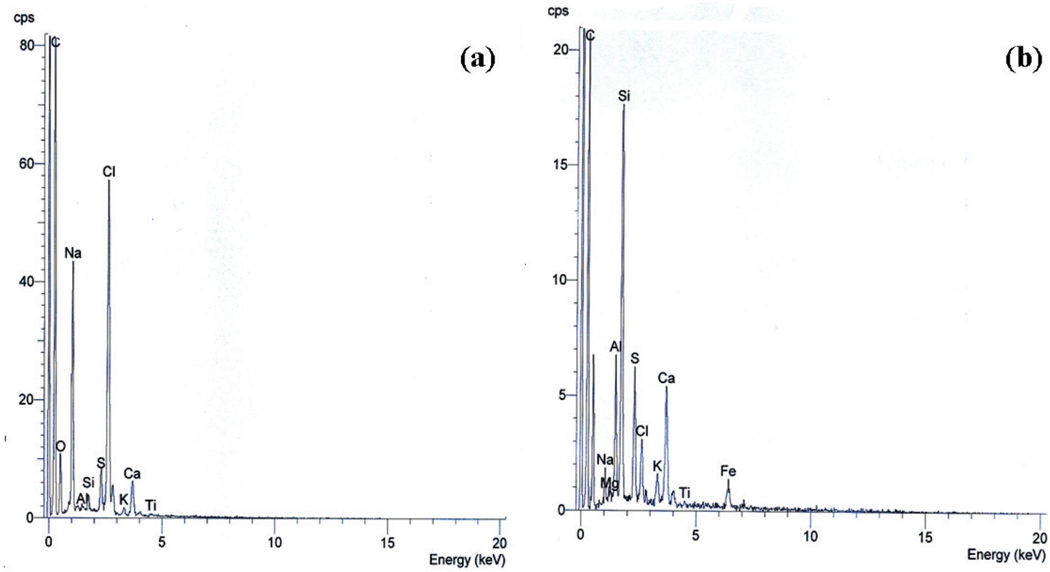

Figure 6. EDS spectra recorded on the damaged layers. (a): EDS spectrum obtained on the salt layer. (b): EDS spectrum obtained on the hard crust.

to damage porous materials, mainly through the production of physical stress resulting from the crystallization of salts in pores. Salts can also damage stone through a range of other mechanisms, such as differential thermal expansion, osmotic swelling of clays, and enhanced wet/dry cycling due to deliquescent salts. Surface soiling phenomena are caused by soot particles embedded in the black crusts onto the surfaces of buildings. Graphite was also detected in the black layer which plays a driving role in surface soiling and black crusts formation (Grossi et al., 2003; Sabbioni, 2003).

Conclusions

The application of SEM-EDS, XRPD and the petrographic examination allowed the characterization of the main deterioration layers formed on the wall of the tomb of Kheruef (TT192), El-Assasif district, El-Qurna necropolis (Luxor), Upper Egypt. The results showed that the deterioration processes of the tomb are mainly caused by salt crystallization. The decay can be observed through the accumulation of chloride and sulphate salts, particularly gypsum. The results have shown that the crystallization of soluble salts of sodium chloride (halite, NaCl), as the predominant salt affecting the site and the precipitation of two phases of sulphates (bassanite, $CaSO_4 \cdot 0.5H_2O$ and gypsum, $CaSO_4 \cdot 2H_2O$). The climatic conditions of the area enhance the crystallization of different salts inside pores of stone or in form of hard crusts. Based on the field study to the tomb, it was possible to observe the distribution of salts within the walls. High concentrations of sodium chlorides appear mainly in the higher and lower areas of the walls; while gypsum is mainly concentrated in the higher parts; it was suggested that the mobility of gypsum to these zones may have been enhanced by the presence of NaCl. According to Smith (1994), salts can cause

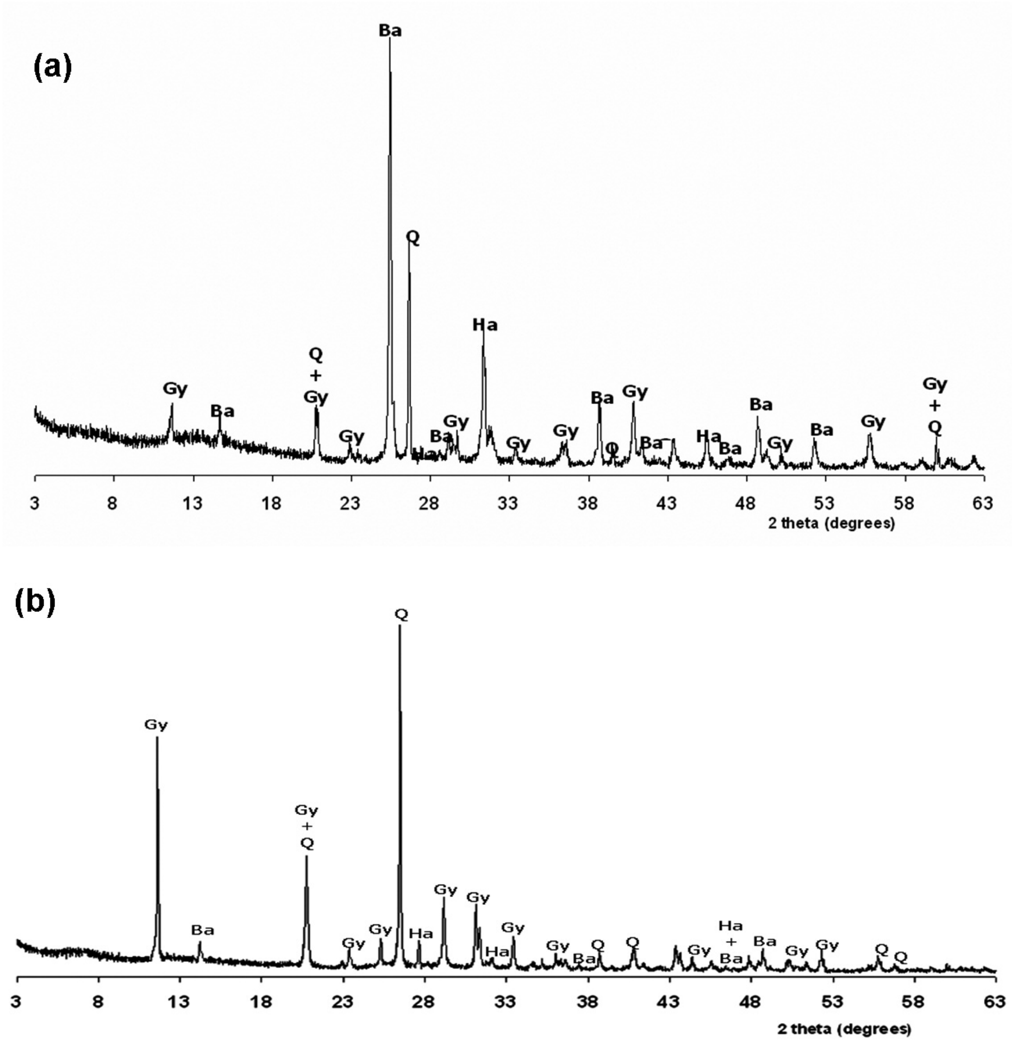

Figure 7. Representative XRPD patterns of the studied salt samples. (a): XRPD pattern of the salt layers. (b): XRPD pattern of the hard crust sample. The identified minerals were labelled as follows: Ba = Bassanite; Gy = Gypsum; Ha = Halite;Q = Quartz.

damage to stones and wall paintings through decay pathway includes differential thermal expansion, crystallization pressure, hydration pressure, through salt crystal growth and enhanced wet/dry cycling caused by deliquescent salts. The expanding salt crystals exert a pressure on the walls causing blistering, stone disintegration micro/macro cracks and the back weathering of the substrate.

In conclusion, the results obtained in this study could to be useful for establishing a conservation plan of the tomb. Wall paintings are an integral part of the building or structure. Therefore, their conservation should be considered together with the fabric of the architectural entity and surroundings. For consolidation of the deteriorated releifs, alkoxysilane-based formulations such as methyltrimethoxysilane (MTMOS) and tetraethoxysilane (TEOS) could be used for stone materials consolidation. Also, acrylic silane mixtures such as Raccanello E55050 improve the stone durability to salt weathering (Brus and Kotlik, 1996). After consolidation, cleaning and removal of the salt crusts took place using mechanical and chemical means. In order to desalinate the soluble salts in the porous matrix, wet poultices (saturated with distilled water) of Japanese tissue and cellulose pulps are used (Marey Mahmoud, 2010). For the hard crusts, the poultice known as AB57 could be useful, this poultice consists of: ammonium bicarbonate (30 g), sodium bicarbonate (50 gr), distilled water (100 ml), (carboxy methyl cellulose) (60 gr), and EDTA (ethylenediaminetetraacetic acid, 10%) (25 gm) (Mora et al., 1984). Uncontrolled public uses of monuments and sites with wall paintings can lead to their damage; this may necessitate the limitation of visitors and, in certain cases, involve temporary closure to public access. In many cases, the hourly capacity of monuments, or types of resources, can be increased by adding and restoring new areas or sites. In other cases, the capacity of areas can be increased by adding additional resources, such as additional tombs in West Bank areas (Luxor, Upper Egypt), this could be accomplished by opening new tombs in existing areas or by opening areas which do not now receive visitors (Abraham et al., 2000).

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