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DECAYING PATTERNS OF QUEEN KHENUT TOMB IN SAQQARA, GIZA, EGYPT

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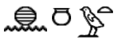
Abstract:
Archaeological monuments of Saqqara region are affected by cracking, weathering and man-made actions that have resulted in different types of deterioration in the long run. Accordingly historical masonry tombs in Saqqara require a full characterization of the materials used for their construction, before any restoration processes. The assessment of the mechanical and physical characteristics of the building materials is based on visual observation, sampling of the building materials used and laboratory testing of the samples (bulk density, porosity, water absorption, optical microscope, polarizing microscope (PM), XRD, SEM, compressive strength, …etc.). The present study is concerned with the restoration and reconstruction of a masonry tomb in Saqqara (Tomb of Queen Khenut), which over the years suffered significant damage due to various deterioration actions. Limestone used in the construction of historical buildings in Saqqara is classified as carbonates and salts with carbonate fragments stone. The stone is composed of carbonate cuttings; each of them is broclastic limemud stone to weak stone. This structure generally presents heterogeneous granular texture; with a very rare matrix and low cementation level between the internal components, causing a fair development of intergranular porosity. The values of bulk density are ranging from 2.22 to 2.33 gm/cm^3. Otherwise, the results of porosity reveal remarkable differences between the different samples. Also the stone has low compressive strength values (The uniaxial compressive strength values varied from 96 to 104 Kg/cm^2), due to the defects in its internal structure and decaying products. Finally, a damage description was carried out to identify the decaying forms. It is the basis for the quantitative rating of limestone damage by means of damage forms and products. Limestone in Queen Khenut's Tomb exhibits the need for restoration and preservation procedures.

1. Introduction

Saqqara is an open historical and architectural book telling the Egyptian history and civilization through time. Historically, Saqqara is the only archaeological cemetery in Egypt that includes tombs and other monumental buildings from the prehistory until Greek-Roman times [1-4]. It is located to the South of Giza pyramids. It is in the higher region of Giza governorate between the historical cemetery of *Abu-Rawash* in the north and *Miidum* in the south [4-8]. Queen *Khenut*  is an ancient Egyptian monarch. She is the legal wife of king *Onas* the famous king of the fifth dynasty in the old kingdom of Egypt. Her tomb is located to the north of the king *Onas* pyramid [9-11]. The tomb is in remains and suffers many deterioration phenomena fig. (1-a). The general layout of queen *Khenut's* tomb is shown in fig. (1-b). It represents the general architectural and historical characteristics of the fifth dynasty tombs. It was constructed of relatively small blocks of limestone connected with gypsum mortar. Its architecture, in general, is characterized by magnificence, simplicity and greatness. queen *Khenut's* tomb was constructed by having the main walls to be the main axis from north to south. However, the tomb construction did not show any visible attention to the north direction [10]. The structure has a rectangular 24×28 m. plan and 3.60 m. high. The main load bearing system of the tomb comprises 1.50 m. thick limestone bearing walls. The tomb consists of a main small entrance leading to two parallel halls linked by parallel paths in the same shape and dimensions. The second hall looks at six parallel chambers. In the first hall, there is a small path leading to the courtyard. On the opposite wall of the courtyard, there are remains of four fake doors, fig. (1-b). The original masonry walls of the tomb were constructed of large rectangular blocks of limestone, but the additional materials (rebuilt works in the next era) were small pieces of limestone, fig. (1-c). By the time, the tomb suffered many damage phenomena due to the act of many significant damage factors. Structural faults, man-made actions, earthquakes, weathering actions and crystallization pressure are the main damage factors, which caused the loss of many construction elements, fig. (1-c). Most of the archaeological masonry buildings in Saqqara are vulnerable to different weathering damage phenomena. Limestone weathering in particular at Saqqara tombs is the most visible deterioration phenolmenon. The study aims at studying the limestone decaying patterns in the royal tombs in Saqqara. It is concerned with the in situ investigation, petrographical characterization, decaying patterns and products, cracking, and damage rate of the limestone used in the royal tombs in Saqqara (Queen *Khenut's* tomb).

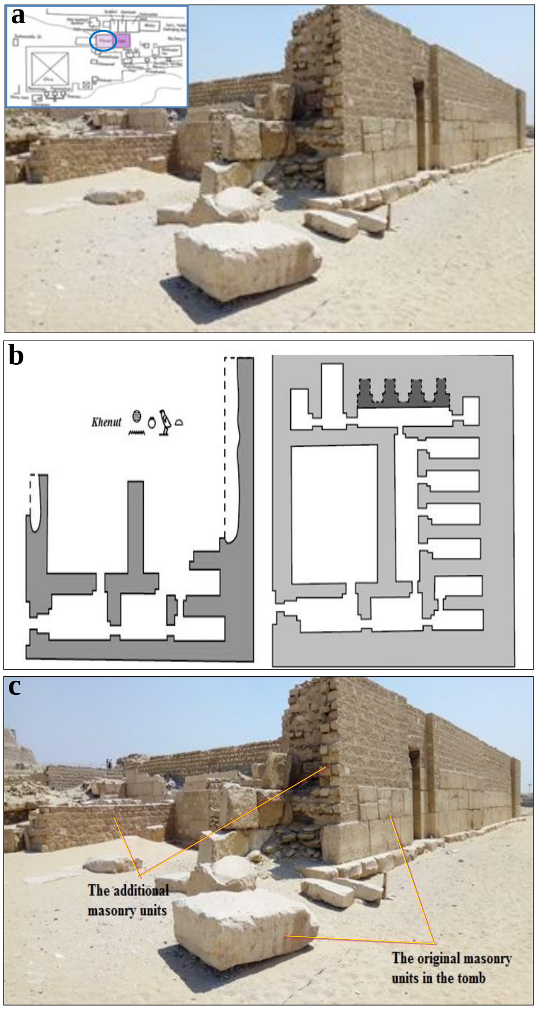


Figure (1) Shows **a.** the located and current situation of queen *Khenut* tomb **b.** the two similar and adjacent toms where, queen Khenut tomb (left) and queen Nebt tomb (right), **c.** the building materials used in the tomb

2. Decaying Patterns of Limestone in Queen Khenut's Tomb

2.1. *Mineralogy setting*

Eocene and middle Eocene mainly provided limestone blocks for building most of the historical monuments in Giza and Saqqara [11-13]. However, lots of studies indicated that the classifications and mineralogical compositions of limestone used in historical monuments in Cairo, Giza and Saqqara were almost pure calcite ($CaCo_3$), it was the predominated carbonate minerals. Dolomite [$CaMg(Co_3)_2$] and ankerite [$Ca(Mg,Fe)(Co_3)_2$] represented subordinately further carbonate minerals. At the same time, it has small amount of quartz and low content of salts (halite and gypsum) [12-14]. The petrographical characterization of Saqqara and Giza limestone according to its carbonate components showed micrite (microcrystalline carbonate), sparite (coarsely crystalline carbonate) and bioclasts (fossils fragment). Accordingly, the limestone in Saqqara can be classified into fossiliferous micrite, sparse biomicrite, poorly washed biosparite and packed biomicrite [15,16].

2.2. *Decaying patterns*

Dissolution, micro cracks and cracks are the most visible damage forms in the limestone used in the construction of monumental buildings in Saqqara region. Queen *Khenut's* tomb is a clear example that suffers from cracking, bleeding and weathering decaying in the constructional masonry units. From the visual survey of limestone in queen *Khenut's* tomb revealed a variety of characteristic decay patterns like; cracking, exfoliation, bleeding and delamination due both internal and external factors, fig. (2). Otherwise, exterior surfaces haven't shown visible salt crystallization (efflorescence) in many places. Salt crystallization beneath the surface (sub-fluorescence) or within the pores (crypto-florescence) is barely visible in the exfoliation pieces. The cycle of heating and cooling during the day and night has caused deformation and cracks due to dimensional changes in the limestone. The decaying patterns in queen *Khenut's* tomb are not uniform in damage rate on all limestone blocks. Many of them are in a fully bad situation, suggesting that the limestone blocks have different mineralogical and mechanical properties.

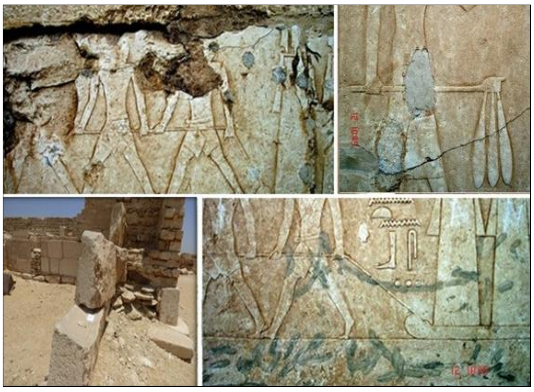


Figure (2) Shows decaying patterns in building materials "queen *Khenut's* tomb"

3. Materials and Methods

Limestone in queen *Khenut's* tomb has suffered many deterioration phenomena as a result of hard action of environmental and manmade factors. The following scientific methods and analytical techniques have been used to inspect not only the effect of internal factors, but also the external factors that have affected the building materials in order to recognize their deterioration mechanisms and forms [17-22].

3.1. *Sampling*

Fully and semi-deteriorated limestone samples (i.e. having visible and slightly visible decaying patterns) were taken and from the walls' courses and fallen stone blocks, fig. (3).

Figure (3) Shows the samples collected from the tomb

3.2. *Experimental methods*

The experimental methodology of the collected samples depended on one hand, on determining the physical properties such as bulk density, porosity and water adsorption. On the other hand, it relied on determining the chemical composition using X-ray diffraction (XRD) using a Panalytical X, pert pro PW 3040/60, X-Ray Diffractometer, with nickel-filtered Cu radiation (λ= 1.540Å, 45kV and 40 mA. Digital microscope (DM) model Dino Capture 2.0 with Version 1.5.12 was used to demonstrate the petrographic features of stone samples. Polarizing light microscope (PLM) was used to define the petrographic characteristics of the samples, Furthermore, Scanning electron microscope (Philips Xl 30 SEM) at acceleration 30 k voltages) was used to determine the morphological features of the investigated samples. Otherwise, uniaxial compressive tests were done to define the mechanical properties of the examined samples. The collected samples were machined to form equal side cubes (5 cm×5 cm×5 cm). All of the previous tests helped define the chemical composition, mineralogical components, decay patterns and products and the causes of the deterioration of limestone units

4. Results

4.1. *Physical properties results*

The majority of the inorganic building materials, including limestone, are porous materials [23-25]. Generally, the physical properties of limestone vary significantly depending on the way they were created, the sedimentary environment and the changes brought about by digenesis [25-28].

4.1.1. Bulk density

Bulk density is important in quantitative studies of inorganic building materials. The statement of bulk density consists of drying and weighing a per unit volume of the stone sample. We aim to achieve a substantial issue from bulk density assessment, i.e. the greater the density, the less pore space for water movement, the bulk density of the collected samples are listed in tab. (2).

Table (1) Bulk density values of the limestone samples

Samples	Sample properties		Bulk density
	Dry weight W (gm)	*Sample volume V (cm³)*	ρ_c (gm/cm³)
1	283.2	127.4	2.22
2	285.7	125	2.28
3	286.3	122.5	2.33
Density Ave.			**2.27**

4.1.2. Porosity

Physically, the porosity of limestone varies considerably depending on the degree of its compaction and inner structure. Typical values (by volume) are 0.1:2% for marble, 0.1:30% for limestone, 15:40% for chalks, and up to 50 %for marls [26]. Table (2) shows the porosity of limestone used in queen *Khenut's* tomb (cubic samples).

Table (2) Porosity values of the limestone samples

Samples	Sample properties			Porosity
	Dray weight D (gm)	*Sat weight W_s (gm)*	*Sample volume V (cm³)*	*P %*
1	283.2	299.6	127.4	12.8
2	285.7	300.6	125	11.9
3	286.3	301.7	122.5	12.5
Porosity Ave.				**12.4**

4.1.3. Water absorption

Absorption is the operation by which water is absorbed by porous materials. Absorbency is the consequence of two effects: Porosity and permeability. In the field of identifying the decaying of stone buildings, porosity and permeability are the most important physical properties [27-28]. Water absorption of porous materials mainly depends on many parameters such as the amount of water offered, the distribution and pore structure, the size and the orientation of the pores, as well as the density and moisture content. The water absorption values of the limestone cubic samples (5×5cm) obtained from the tomb and results are listed in tab. (3).

Table (3) Water absorption values of the limestone samples

Samples	Sample properties		Water sorption
	Dray weight D (gm)	*Sat. weight W_s (gm)*	%
1	283.2	299.6	5.79
2	285.7	300.6	5.22
3	286.3	301.7	5.38
Water Absorption Ave.			**5.46**

4.2. *XRD results*

The mineralogical composition of limestone and weathering products phases were recognized by XRD proved that they are occurring in two groups. The 1st group composed of Calcite (CaCo3) as a main component of limestone. The 2nd one includes Ankerite [$Ca(Mg,Fe)(Co_3)_2$, Gypsum ($CaSO_4.2H_2O$) and Quartz (SiO_2) as different weathering products, all of these minerals are shown in fig. (4).

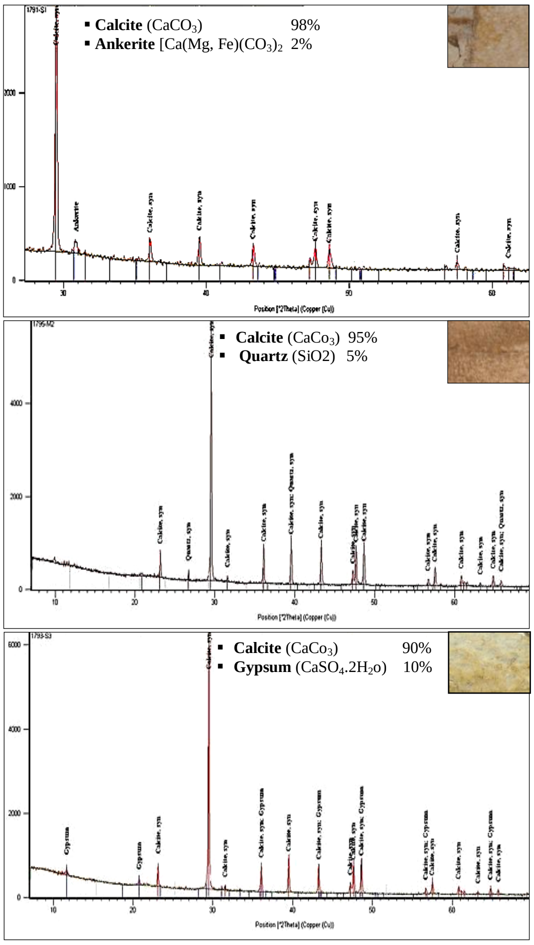


Figure (4) Shows XRD patterns of investigated limestone samples

4.3. *DM and PLM results*

4.3.1. Digital microscope (DM)

Through using DM, digital image capture systems and the image analysis software it could be noted that the investigated samples show some deterioration features such as caves and micro-crack, fig. (5)

Figure (5) Shows DM photographs showing the caves and micro-cracks distributed in the surface of limestone samples

4.3.2. Polarizing Microscope (PLM)

PM investigation used to determine the petrographic features of the limestone used in the construction of queen *Khenut's* tomb proved that limestone; frequently consists of coarse grained stone, with presences numerous, rounded and tightly packed fossils and seashells of various in shapes and sizes, fig. (6). These fossils are generally; joined together with microsparitic calcite cement [15,29].

Figure (6) Shows the results obtained from the investigated samples under PM (X-25).

4.4. *SEM/EDX test results*

Microstructural features of the investigated fragments using SEM proved that they have highly decayed surfaces. In addition, they include some inner cracks and small fissures between grains, fig. (7-a). Furthermore, presence of some small caves and micro-cracks around and between grains, fig. (7-b). EDX analytical results proved that chemical composition of limestone and decaying products used in queen *Khenut's* tomb includes C, Ca, Si, O, Fe, Al, Mg & S, as listed in tab. (4)

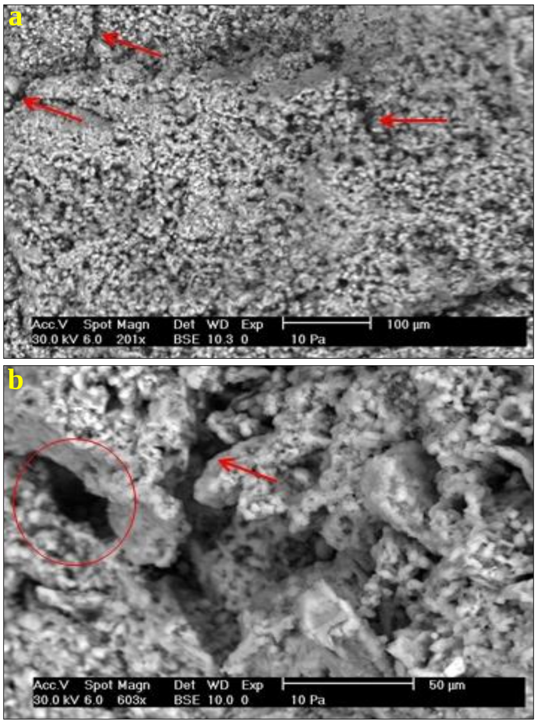


Figure (7) Shows photomicrographs **a.** inner cracks and small fissures between grains **b.** small caves and micro-cracks around and between grains

Table (4) SEM/EDX analytical results of the investigated samples

Elemental ratios	Investigated samples	
	S_1	S_2
C	23.83	17.00
Ca	25.07	15.21
Si	2.25	3.55
O	46.52	51.26
Fe	-----	1.69
Al	0.53	1.66
Mg	1.63	0.72
S	0.18	8.91
Total	100	**100**

4.5. *Compression test results*

The obtained results values of compressive strength test given in tab. (5) affirmed that the compression values are varied due to the effects of dominated environmental parameter in the study area.

Table (5) Compressive strength and failure load of limestone specimens

Specimens	Type	Dimensions (cm)			Weight	Failure load	Compressive strength
		Side a	*Side b*	*Height*	(Kg)	(KN)	(Kg/cm^2)
A_1	Cube	5.2	5.1	5.1	0.298	2.50	104.22
A_2	Cube	5.3	5.2	5.1	0.296	2.31	96.34
A_3	Cube	5.3	5.0	5.0	0.295	2.43	101.47
Compressive strength Ave							**100.68**

5. Discussion

In situ observations carried out on queen *Khenut's* tomb and adjacent tombs illustrated that most of the decay patterns of the building materials were cracking, desolation, exfoliation and scaling. The methodology to identify the decay patterns on the limestone used in the masonry buildings in Saqqara were carried out comprising in situ investigation and laboratory analysis. The laboratory analysis included physical properties assessment (density, porosity and water absorption), mineralogical investigations (XRD, optical examination, Microstructural features (SEM)) and compression strength tests to state the decay phenomena in the limestone used in queen *Khenut's* tomb. To identify the petrographical characterization, deterioration factors and decaying forms affecting the limestone used in queen *Khenut's* tomb, the following qualitative and quantitative test techniques were employed.

5.1. *Physical properties*

Laboratory studies carried out to identify the physical properties of limestone in queen *Khenut's* tomb differ significantly, see tabs. (1, 2 & 3). The limestone samples show approximate values of bulk density, ranging from 2.22 to 2.33 gm/cm^3. Otherwise, the results of porosity reveal remarkable differences between the different samples, tab. (1). The average value of porosity is 12.4%. [13] investigated the porosity values of limestone used in historical buildings in Cairo that rated from (6.4%) to (27%). Thus, it can be concluded that limestone in queen *Khenut's* tomb (lay in the middle zone of porosity values of limestone in Cairo. The absorption values obtained in the limestone specimen tests of queen *Khenut's* tomb showed a high absorption rate (the average value of water absorption was 5.46 %). In contrast, the high ratio of water absorption value of the limestone did not contain salt minerals due to the absence of moisture content in Saqqara quarter.

5.2. *Mineralogical compositions*

According to the XRD patterns of the tested samples presented in fig. (4-a,b,c) it could be concluded that limestone used in queen *Khenut's* tomb was mainly derived from carbonate rocks (crystalline to microcrystalline limestone). Moreover, calcite ($CaCO_3$) appeared as a major mineral of limestone (up to 98%) used in queen *Khenut's* tomb**.** The presence of gypsum ($CaSO_4.2H_2O$), quartz (SiO_2) and ankerite [$Ca(Mg, Fe)(CO_3)_2$], as minor and trace minerals, mostly refer to the deterioration forms and products in the limestone, tab. (6). These compositions indicated that the observed detachment of stone crusts, cracking and subsequent loss of limestone materials resulted from soiling, black and whitish crusts and salt deposits in the pore space of limestone units.

Table (6) Mineral compositions of limestone used in *queen Khenut's* tomb

Samples	Mineral Composition			
	Calcite	*Quartz*	*Gypsum*	*Ankerite*
1	98%	--	--	2%
2	90%	--	10%	--
3	95%	5%	--	--

5.3. *Surface and petrographic description*

DM investigation showed that, on one hand the surfaces of limestone suffered loss and detachment of materials making unequal volume cavies, fig. (5). On the other hand, it showed the structural discontinuities of the limestone used in the tomb. The loss of the stone materials mainly, due to the processes of salt crystallization, resulted from the cyclic action of the water and of the thermal radiation [30-31] and helped the development of materials detachments. Moreover, low cementation level and pitting features are observed on the stone surface, which could, in turn, enhance granular disaggregation processes. Within the same context, the petrographic features, fig. (6) illustrates that the limestone in the tomb is classified as carbonates and salts with carbonate fragments stone. In addition, the samples contain micrite matrix crowded by minute foramiaforal with a coarser shell that partly re-crystallized micro sparry calcite. Moreover, the stone is composed of carbonate cuttings; each of them is broclastic lime mud stone to weak stone. This structure generally presents heterogeneous granular texture; with a very rare matrix and low cementation level between the internal components, leading to a fair development of inter-granular porosity. Therefore, the cracks spread due to the incompatible and low cementation level in the internal components of the limestone used in the tomb. The microstructure features, fig. (7) illustrated that the inner micro-laminations lost continuity of the calcite grains and cohesion between inter-granular compositions. Also, the resulted data tab. (4) affirmed that the presence of Si-based compounds and Fe, Al, Mg state to clay minerals fill the micro-laminations of the samples. These minerals that enhance the micro-cracks inside the laminations within the stone body and the cracks on the stone surface.

5.4. *Mechanical properties*

The low values of the mechanical properties of the limestone, tab. (5) highlighted the weakness in the internal structure. The uniaxial compressive strength values varied from 96 to 104 Kg/cm^2, with ave. 100.68 Kg/cm^2. These variations are attributed essentially to the various values of compression between the investigated samples that showed a clear relationship between mechanical properties values and internal structure defects of the stone. Furthermore, the increasing of cavities within the stone structure play a negative role on the stone strength values, as they exhibited weakness and embrittlement of stone surfaces during the application of compression and tensile forces [32].

6. Conclusions

Limestone is the main building materials used in the construction of monuments in Saqqara. All the historical limestone buildings in Saqqara have been prone to weathering actions. Limestone in queen Khenut's tomb revealed a variety of characteristic decay patterns (cracking, exfoliation, bleeding and delamination) due to internal and external factors. The portion and amount of noncarbonate and clay minerals and components in addition to decay products had a significant effect on the deterioration of limestone used in queen Khenut's tom. The discontinuities of the internal structure with different amplitudes crossing the stones as irregular planes may accelerate the decay processes resulting in the limestone. Thanks to the absence of moisture sources, limestone of queen Khenut's tomb had suffered neither the black crust nor biological growth. The results of the laboratory tests and in situ investigation indicated that limestone used in queen Khenut's tomb suffer from a loss of surface component materials, cavities, and dissolution due to the critical action role of the dust particles in the air and small particles from air pollution. Limestone is the most building material that is often highly characterized by discontinuities-structure surface due to internal components of scales and shell-like structures. At various scales, this physical component is associated with material loss of the original stone. Limestone used in the construction of historical buildings in Saqqara has low compressive strength values due to the defects in its internal structure and decaying products. An increasing occurrence of cavities exhibit weak zones during the application of compression and tensile forces. Limestone decaying patterns in Saqqara historical tombs are initially alarming. This stresses the shortage of restoration and preservation procedures and the need to begin in the measures, e.g. cracks treatment and consolidation of weak *and loose stone material. Finally, environmental management and rehabilitation procedures must be considered.*

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