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EXPERIMENTAL TESTS USED FOR TREATMENT OF DISINTEGRATED GRANITE IN VALLEY TEMPLE OF KHAFRE – EGYPT

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Abstract

The temple Khafre in Giza plateau was built with huge stones of limestone, which is covered with red granite. Most of this granite was taken from the outside. This granitic casing has been affected by many exogenous and endogenous deterioration factors, which led to the weakness of the surface layer and the fall of large parts of it and causes of a severe damage of the stone materials. In this study we documented the weathering of these granite casing using field recording and laboratory analysis to evaluate their conservation state. Weathering and deterioration aspects noted through light optical microscope (LOM), polarizing microscope (PM) and scanning electron microscope (SEM). The mineralogical characterization was performed using X-ray powder diffraction (XRD) and Energy dispersive analysis (EDX). Physical and mechanical properties of Granite were determined. The present study suggests that the most suitable treatment methods are composed of co-polymer (Acryloid B66, diluted at 5% in trichloroethylene + Wacker VB132 (Tetra Ethoxy Silane) or Estel 1000.

Keywords: *Khafre temple; Granite; Deterioration; Consolidation; Acrylic; Ethoxy Silane*

Introduction

Valley Temple of Khafre was part of the funerary complex in Giza plateau and it`s the entrance to the pyramid group. It is a pyramidal temple full of immortality is due to the architectural and richness of raw materials used. The temple was built with huge stones of limestone, which is covered with red granite. Most of this granite was taken from the outside. The granite cover of the interior walls and the 16 granite columns still bear witness to the great architecture of the ancient Egyptian during the Old Kingdom Era and the use of solid stones in the early period of its history. This granitic coating has been affected by many exogenous and endogenous deterioration factors, which led to the weakness of the surface layer and the fall of large parts of it and causes of a severe damage of the stone materials. The characteristics of the deterioration of granitic casing and columns takes the following forms: loss of a lot of parts from granitic casing (Fig. 1a) and mechanical exfoliation, detachment and falling of granitic surface, disintegration of grains, degradation of the minerals characteristic by a spontaneous granule of the granite stone (Fig. 1b). Separation of some of these grains and separation of the scales of the surface layer because of the detachment and loosing of the bonds that tie the grains of the surface area and those grains of the layer. Different type of cracks; macro, micro and wide deep cracks takes the form in all surfaces (Fig. 1c). Some parts in columns are particularly damaged and all surface was covered with numerous and different layers of dirties, deposits of

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dust, stains and numerous cobweb nests (Fig. 1d), due to the accumulation of airborne deposits from different sources. Soluble salts within the stone causing efflorescence on the surface and/or sub- efflorescence. The salt crystallization lead too loss of cohesion between grains and causes the spalling, current detachment of individual grains (Fig. 1e). Very dense layers of biological colonization accumulated on the granitic surface and accumulation of black to dark-colored crust were observed tracing the surface by brown, green and white stains of microorganisms, which caused the discoloration of stone surface (Fig. 1f). The aim of this paper is to examine the characteristics of studied granite and the weathering process acting on evaluate the efficiency of the various hydrophobic products in laboratory to treatment for the conservative treatment of the weathered granitic casing in Valley Temple of Khafre in pyramids plateau.

Fig. 1. Extensive deterioration features of granitic casing of Valley Temple;
a - loss of parts from granitic casing, **b -** Mechanical exfoliation and loss of a lot of parts from granitic casing, **c -** different type of cracks and numerous cobweb nests, **d -** Different layers of dirties, deposits of dust, stains, **e -** efflorescence salts on the surface, **f -** Very dense layers of biological colonization accumulated on the granitic surface.

Materials and Methods

Samples

The samples under investigation were carefully acquired and isolated from a variety of zones in the granite casing at Valley Temple of Khafre.

Petrographic examination

Thin sections were prepared from the studied samples, then Petrographically examination and photography was carried out under Olympus BX51 TF Japan petrographic microscope attached with digital camera under magnification 20 up to 40X.

Scanning electron microscope (SEM-EDX)

Mineralogical composition, pore structure and percentage were characterized. Scanning electron microscopy (SEM JEOL JSM 6400) coupled with energy dispersive microanalysis (EDS).

X-Ray fluorescence (XRF)

Geochemical analysis of the samples consisted of XRF analysis were determined, XRF measurements were performed in situ nondestructive analysis. XRF spectrometer for elemental analysis (JSX-3222), equipped with end window type X-ray tube, tube voltage 5 to 50Kv (in 1Kv steps), tube current 0.01 to 1.0mA, using Rh anode as target, window Be, 127µm thick.

X-ray diffraction (XRD)

Mineralogical investigation was carried out through X-ray diffraction analysis (XRD). A Philips PW 1840 diffractometer with a Ni-filtered Cu Ká radiation (ë = 1.54056Å). Diffraction patterns were recorded in a 4.005°, 59.775° 2è and angular range with a 0.030°: 0.05° step.

Study of physical and mechanical properties of limestone:

Granitic samples were taken from the removal of fragments around Valley Temple of Khafre and subjected to the same weathering, and then were cut into cubes samples (4cm^3), slabs (7×7×3.5 thickness), columns (20L×4h×4t) (fig.2). The granite samples were cleaned dried at (105ºC) for at least 24 hours to reach constant weight, and left to cool at room temperature to evaluate the physical properties (bulk density, porosity and water absorption) and mechanical properties (compressive strength, tensile strength, losing by Abrasive).

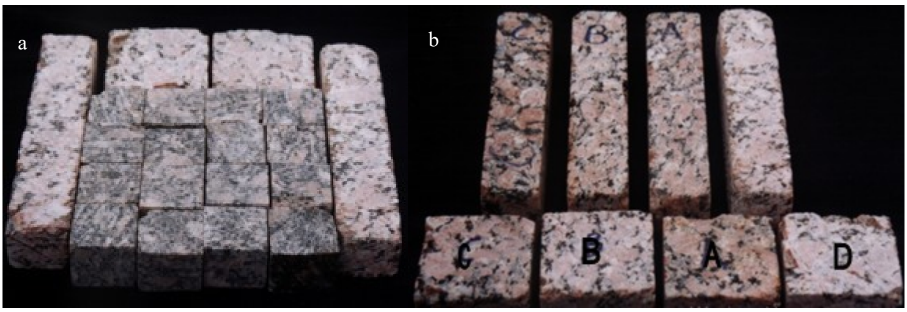

Fig. 2. the prepared granite samples to evaluate physical and mechanical properties

Results and Discussions

Petrographically characteristics of studied granite samples

General observation from the petrographic study for the samples revealed that, the rock is medium to coarse grained, dark grey in color and characterized by Leuco Granite. The rock under investigation composed essentially of Quartz, Quartz occurs as rounded to subrounded and faceless granules (Fig. 3a), Plagioclase occurs as euhedral to subhedral and frequently appeared cloudy due to conversion to clay minerals, Plagioclase is subject to slight to moderate alteration to sericite and kaolinite, Seriticised plagioclase occurred frequently and was it split by micro-cracks which iron stained (Fig. 3b and c). Potash feldspar (Microcline) occurs as microcline crystals showing cross hatching twinning and partly kaolinized and serietized (Fig. 3d), Muscovite with prismatic to full crystalline facets grains presented as accessories (Fig. 3e). Mafic minerals are represented by and hornblende, hornblende is represented by euhedral to subhedral; hornblende shows pale brown to mild-green pleochroism (Fig. 3f). Cracks are common in hornblende grain with presence internal grains filled with secondary quartz. Chlorite and clay minerals are secondary constituents.

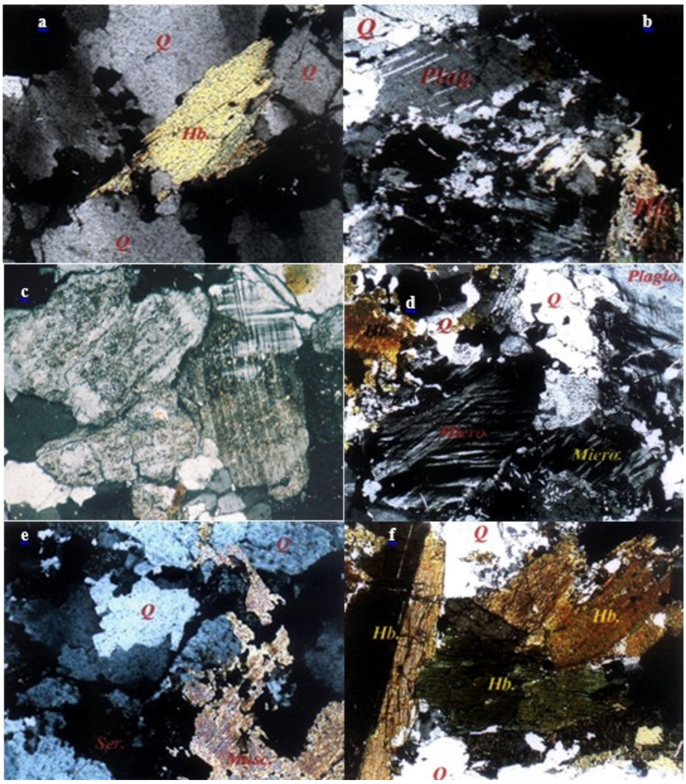

Fig. 3. Petrographic view of granite samples shows:
a - texture of quartz grains; **b,c** - altered plagioclase to clay minerals;
d - microcline with cross hatching with Inclusions and altered Plagioclase,
e - Muscovite with prismatic to full crystalline facets grains; **f** - euhedral to subhedral Hornblende.

SEM-EDX examination

SEM micrographs of the untreated granite samples (Figs. 4a-f) show the patches of the fine-grained quartz mineral, obliteration of the characterized igneous textures, fractures in the minerals, the volume and distribution of the pores. SEM micrograph revealed the intra-crystal microcracks inside feldspar and quartz, such splits added to expand porosity causing the granite end up penetrable. The SEM studies show that, the major deterioration phenomena in granite sample is exfoliation and micro pitting SEM micrograph showed that, some of the constituent minerals of granite have been altered into clay minerals due to different weathering factors. Also, the effect of salt weathering on samples was observed, which concealed the distinctive shapes of the mineral formation of the granite and notes the caves resulting from the minerals breakdown of the granite components. It is also noted that there is a network of microcracks and cracks between the granules of the samples. This is evidence of the severe deterioration of these granite stones. In addition to the presence of salts gypsum and halite which led to the destruction and destruction of crystals of granite minerals.

Elemental Analysis by XRF

The chemical analysis was performed by using X-ray fluorescence, and the results are summarized in figure 5 and table 1.

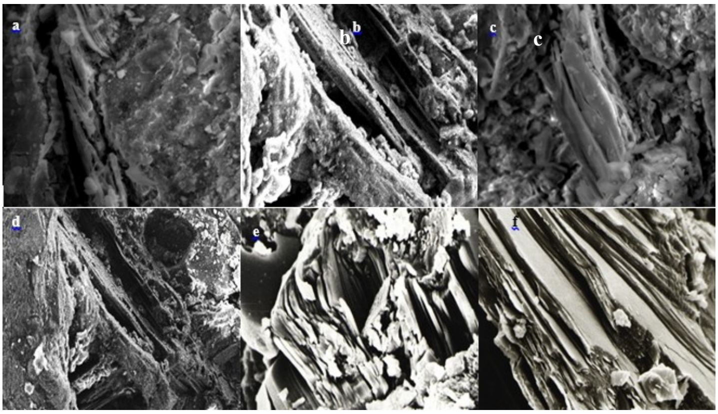

Fig. 4. SEM photomicrographs of the granite samples shows deformation of internal structure of granite, micro exfoliation and pitting, disintegration, Gypsum and halite salts

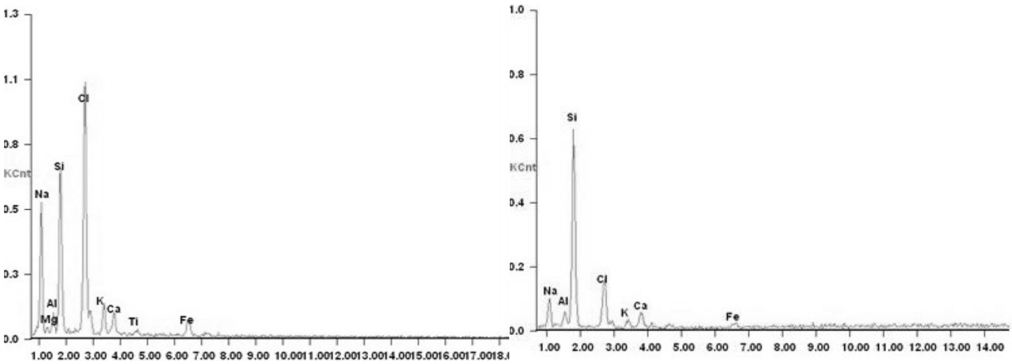

Fig. 5. Shows elemental composition by X-ray fluorescence.

Table 1 Chemical analysis in weight per cent of the weathering granite samples collected from the Valley temple

Main constituents	Concentration (wt%)
Na_2O	13.32
Al_2O_3	05.81
SiO_2	65.27
Cl_2O	10.23
K_2O	01.20
CaO	03.08
Fe_2O_3	01.09

Results of Mineralogical Analysis by XRD

X-ray diffraction analysis (Figs. 6a-d) indicates that most abundant phase in the granite samples is quartz (SiO_2), K-feldspar- Orthoclase ($KAlSi_3O_8$), Na-feldspar Albite, mica group "Muscovite" ($KAl_2(AlSi_3O_{10})(F,OH)_2$ and Biotite $K(Mg,Fe)_3(AlSi_3O_{10})(F,OH)_2$ and traces amount of Hematite (Fe_2O_3), Kaolinite ($Al_2Si_2O_5(OH)_4$). Also XRD analysis showed salts as halite ($NaCl$) and gypsum ($CaSO_4.2H_2O$).

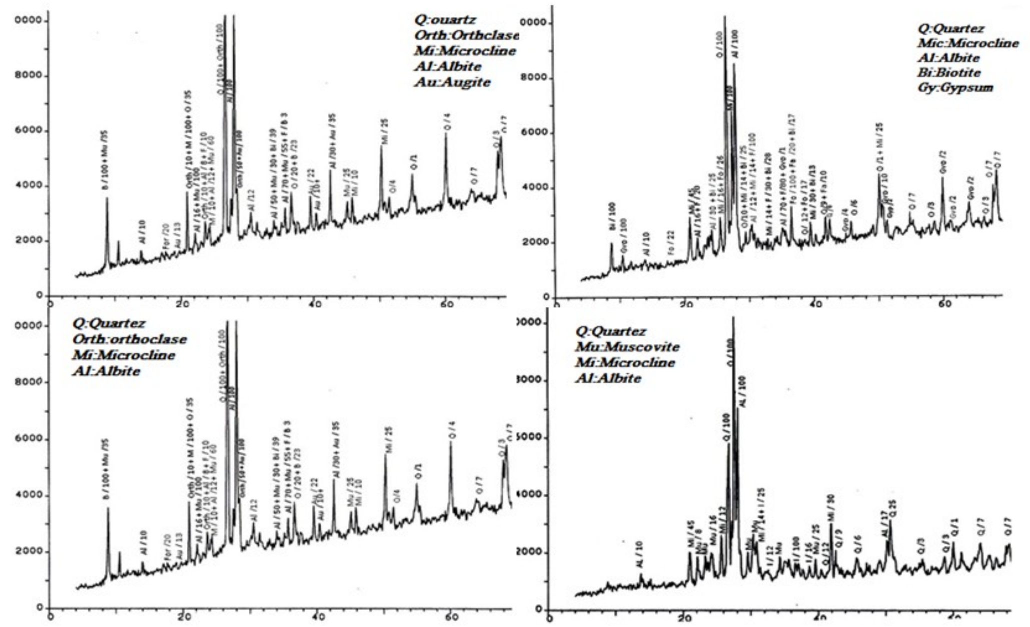

Fig. 6. XRD patterns of the granite samples from Khafre temple

Physical and mechanical properties

The physical properties (bulk density, water absorption, porosity) detected that the granite in Khafre temple is characterized (table 2) by high porosity (4.17%), which gives the stone a high water absorption rate (1.55%) and its consequent ability to absorb water with salt solution and this is the main cause of deterioration. The bulk density of the samples gives 2.67g/cm^3.

Mechanical properties (Compressive strength, indirect tensile strength, and abrasive test) were determined for untreated samples (Table 3), the results showed that the strength of granite in the facade characterized by poor mechanical characteristics (by 760kg/cm^2 Dry and 690kg/cm^2 Wet, the tensile strength is 12.7m, the losing by abrasive is 14.18g. The changes of physical and mechanical properties of the stone were a result of weathering process.

Table 2. Physical and mechanical properties for untreated samples

Physical properties	**Untreated Samples**	
Bulk density (g/cm^3)	2. 67	
Water absorption (%)	1.55	
Porosity (%)	4. 17	
Mechanical properties	**Untreated Samples**	
Compressive strength (g/cm^2)	Dry,	Wet
	760	690
Tensile strength N/mm^2	12. 7	
Losing by Abrasive (g)	14. 8	

Treatment

The field study and lab analysis show that, the granite casing at Khafre temple suffered from a lot of deterioration factors as disintegrating, the granules lost their cohesion, mechanical exfoliation; so, the use of chemical "preservatives" to stabilize stone deterioration must be done.

Deteriorated stone can`t be saved without effective consolidation, for this purpose, four products of consolidation polymers were tested, one of Acrylic resin (Paraloid B-66), two of organosilicic type (Estel 1000, Wacker BS OH 100) and co-polymer based on acrylic resin + silicone (Wacker VB132 + 5% Acryloid B-66) as shown in table 3.

The water repellent silicone treatment aims to eliminate or reduce capillary absorption of water in driving rain, prevent wet absorption of reactive gases, reduce damage effects from frost, diminish biological growth and the amount of particulate fixation preventing the soiling, also ethyl silicate helped by the catalyst, reacts with atmospheric water, producing silica gel (SiO_2-aqua) as a final product of polycondensation.

Table 3. shows the consolidant polymers & there solvents

Acryloid B-66	An isobutyl methacrylate polymer. It is an alkyd-compatible acrylic dissolved in trichloroethylene (5%),
Estel 1000	A ready-to-use consolidant product based on ethyl silicate in white spirit D40 solution; it is particularly suitable for the consolidant and treatment of weathered silicate stone.
Wacker BS OH 100	SILRES BS OH 100 is a solventless, ready-to-use product for the consolidation of stone, based on Tetra Ethoxy Silane
Wacker VB132 & 5% Acryloid B-66	Acrylic resin + Ethyl silicate (TEOS)

Application of the consolidant to the stone samples:

The samples were cleaned, dried at (105ºC) and the samples which were treated with organosilicon were wetted and left in the air until the water content was 0.2g. This procedure favors the polymerization of organosilicon products [1].

$H_2SiO_3 + H_2O \rightarrow SiO(OH)_4$ [1], $Si(OH)_4 \rightarrow SiO_2 + 2H_2O$ [2].

Stone samples were impregnated through capillary suction for 1 hour to half way; the same treatment was repeated after 24 hours. After treatment, all the samples were stored at the atmospheric circumstances of the room for at least 30 days coated with polyethylene to allow the polymerization process to take place.

The same properties were measured on samples after the treatment to evaluate the effects of polymers on improvement of granite properties.

For testing successful extension for consolidation materials to protect the granite surface and granting it mechanical power to struggle the sandy wind, the abrasion resistance test was determined using Bohme abrasion wheel 1006, some granite samples were cut into square slabs (7 x 7 × 3.5cm thickness), mechanical brushing had been applied to clean the samples, drying in an oven at 105ºc for 24 hours and then weighed. The slabs were consolidated with chosen polymers and after 30 days the slabs samples were put in testing machine with Corporundum.

Physical and Mechanical properties of treated samples:

The treated parts of all samples got darker. The effect was a little more intensive in case of increased humidity. The degree of penetration was assessed by low magnification scanning electron microscope. Bulk density, Water absorption and Porosity of the treated and untreated samples were determined (fig.7).

Compressive strength was determined. The untreated samples had an average value of 730 kg/cm^2, the samples treated with TEOS + 5% Acryloid B-66+ Wacker VB132 gave the highest value 785 kg/cm^2.

Indirect Tensile strength measuring (Table 4) show that the average values for treated and untreated sample, the value for the untreated sample was 12.7N/mm^2 whereas those for the treated samples with Acryloid B-66+ Wacker VB132 gave the highest value 15.80N/mm^2.

Losing by abrasive measuring (table 5) show that the average values for treated and untreated sample, the value for the untreated sample was 14.8g whereas those for the treated samples with Acryloid B-66+ Wacker VB132 gave the low value 4.30g losing.

Table 4. The mechanical properties to untreated & treated granite samples

Mechanical properties	**Untreated**		**B-66**	**Estel 1000**	**Wacker BS OH 100**	**VB132+B -66**
Compressive strength (g/cm^2)	Dry	490	695	655	560	630
	Wet	467	589	542	535	592
Tensile strength N/mm^2	12. 7		14. 2	13. 35	13. 90	15.80
Losing by Abrasive (g)	14. 8		6.5	7.20	7.10	4.30

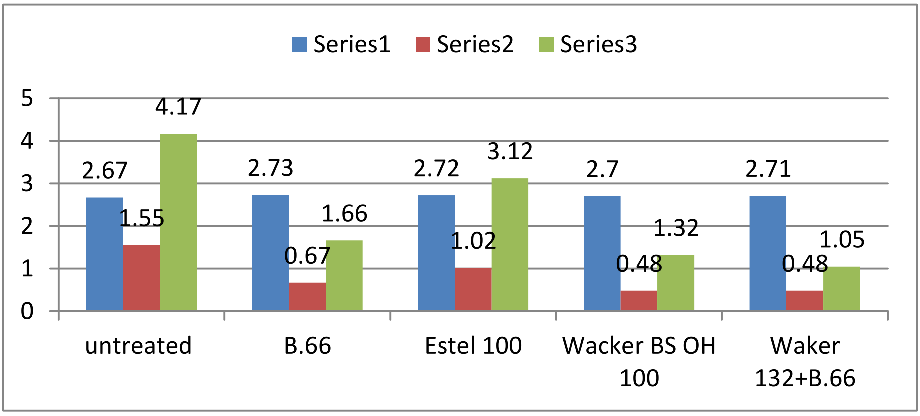

Fig. 7. shows Physical properties of untreated and treated granite samples

Scanning electron microscopy (SEM) study of treated granite samples

SEM micrographs of the granite sample treated with Acryloid B-66 (Fig. 8a) shows unsuccessful results because a thin layer of the polymer has been formed but this network structure has failed to fill fine cracks. The sample treated with Estel1000 (Fig. 8b) shows that the massive coating was deposited around granite grains, the polymer was found on the surface of the granite grains. The sample treated with Wacker BS OH100 (Fig. 8c) shows the coating of the consolidant material on granite grains and the sporadic network of the polymer was observed. The sample treated with Wacker BS OH132 and after that treated with 5% Paraloid B-66 (mixture between the silicon resin & the acrylic resin) (Fig. 8d) shows perfect penetration, the network structure of consolidant can be seen dispersed between the grains and through the pores.

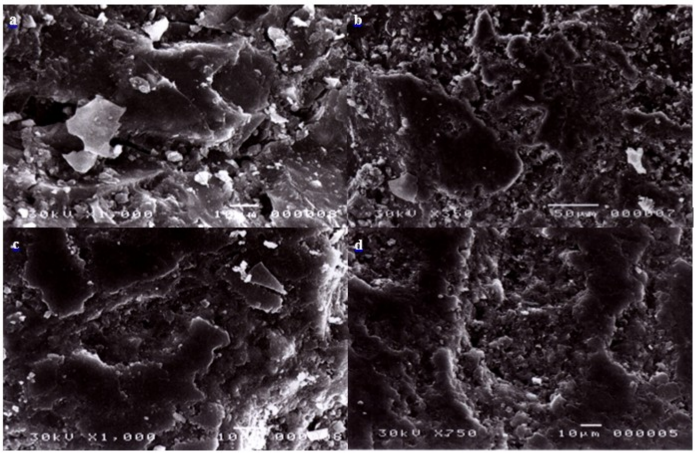

Fig. 8. SEM photomicrographs of the treated granite samples by different polymers; **a-** samples treated with B.66, **b-** samples treated with Estel 1000; **c-**samples treated with Wacker BS OH 100; **d-** samples treated with VB132+B-66.

The influence of ageing condition on the properties of treated granite samples

In order to obtain the most suitable polymer to give the best consolidation which can outlast in front of different weathering factors, the experimental condition used for the purpose of artificial weathering were far more severe than natural condition. Samples were saturated with water through capillary suction and left partially immersed for free evaporation of water from the surface. Other samples were treated with wetting – drying cycles. In case of wetting – drying tests the same deposits started to from on the surface of the stone after 15 cycles. Considerable concentration of salts and sulphuric oxides in the atmosphere of Giza plateau is responsible for sulphuric compounds and salts, the samples were saturated by capillary suction with diluted solution of sodium sulphate. The test was carried out according to (ASTM Designation C88 – 56 T). The results were determined after 15 cycles. Physical & mechanical properties were determined (Tables 6, 7 and 8) and the results were confirmed by S.E.M (Fig. 9a-d).

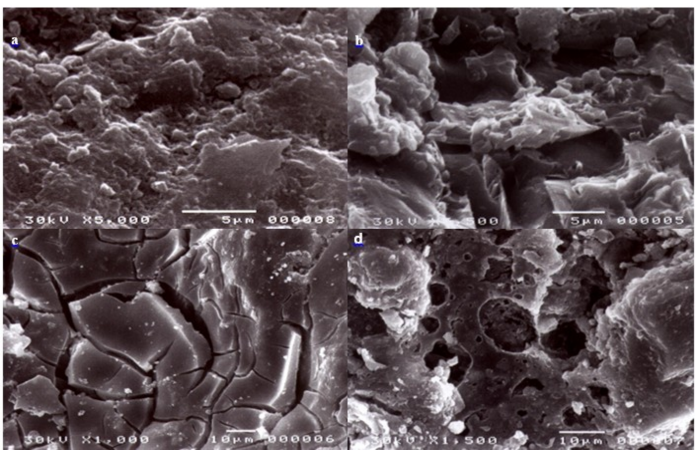

Fig. 9. SEM photomicrographs of the treated granite samples by different polymers after artificial weathering; **a-** samples treated with B.66, **b-** samples treated with Estel 1000; **c-**samples treated with Wacker BS OH 100; **d-** samples treated with VB132+B-66

Table 3. Physical properties to untreated & treated granite samples after thermal weathering

Physical properties	untreated	B-66	Estel 100	Wacker BS OH 100	B-66 +VB132
Bulk density (g/cm^3)	2.67	2.64	2.59	2.66	2.61
Water absorption (%)	1.55	0.57	1.23	0.72	0.55
Porosity (%)	4.17	1.86	3.28	1.74	1.52

Table 4. Physical properties to untreated & treated granite samples after salt crystallization weathering

Physical properties	untreated	B-66	Estel 100	Wacker BS OH 100	B-66 +VB132
Bulk density (g/cm^3)	2.67	2.60	2.56	2.63	2.59
Water absorption (%)	1.55	0.72	1.67	0.82	0.68
Porosity (%)	4.17	1.95	5.18	2.01	1.93

Table 5. Mechanical properties to untreated & treated granite samples after salt crystallization weathering

Mechanical properties	untreated		B-66	Estel 100	Wacker BS OH 100	B-66 +VB132
Compressive strength (g/cm^2)	Dry	477	540	695	595	520
	Wet	435	410	442	435	390
Tensile strength N/mm^2	12. 7		12.25	13. 35	12.45	13.90
Losing by Abrasive (g)	14. 8		8.7	9.40	9.15	7.52

Results and Discussion

This study of the granite used in the Valley Temple of Khafre shows some of the reasons for the deterioration of the stone as; mechanical exfoliation, rock granular disintegration, detachment, falling of granite surface, the surface covered with dust and thin layer of sand, wild bees, nests, a lot of micro cracks are spread and accumulation of salts. These deterioration aspects due to the internal stresses resulting from temperature and relative humidity changes; the climate is typically Saharan, hot and dry with scanty winter rain and bright sunshine throughout the year. According to the bio-climatic provinces of Egypt, the area is hyper-arid with mild winters and hot summers [2], due to the arid climate of the region and continuous changes between day, night and seasonal changes in temperatures, which are considered very important participating factor in physical weathering. The surface layers of granite on the sunny sides often reach much higher temperature in July 35°C and relative humidity - 50% RH in December [3] and this causes serious deterioration and decomposition of granite stones, Chemical weathering processes, change ferrous iron to ferric ions of the minerals constituents, this alteration produces cracks and weakness which cause granular disintegration into minerals of granite and coloration [4]. Relative humidity variation and saline salts also plays a role in granite decomposition, many rocks show a significant strength decrease with increasing moisture content [5]. A more probable cause of this type of degradation is the infiltration of soluble salts into the rock pores and crystallization of the salts therein. Salts are the most powerful weathering agents. Efflorescing salts improve the esthetic value. Under natural conditions surrounding minerals, which are enough to crack most rocks common to granite [6], also the ground water assists in the weathering of Plagioclase and Biotite and other minerals.

Thin-section analysis showed the alteration of potassium feldspar minerals (orthoclase to kaolinite), this confirmed the role of chemical weathering like mainly moisture and air pollution (mainly SO_2 gas which its concentration in Giza plateau [7].

$$2KAlSi_3O_8 + 2H^+ + SO_4^{2-} + H_2O \rightarrow Al_2Si_2O_5(OH)_4 + 2K^+ + 4SiO_2 + SO_4^{2-} \quad [4]$$

Traces of sodium chloride (Halite) salts were found between grains which are considered most strongly influencing the level of stone degradation, the salt pressure crystallization of Sodium chloride between the grains cause main stone exfoliation.

All these deterioration factors lead to the wastage of a lot of part of the granite in Valley Temple of Khafre. It was necessary to study some consolidation products to choose one of them which give the best degree of penetration, make a linking between separated grains, and replace the natural grain cement destroyed by weathering on the stone's surface [8-10]. Some acrylic products, silicone and mixture of the both were evaluated as follow, study of physical & mechanical properties, and study with S.E.M. after artificial aging (included cycles of heating & cooling, salt weathering) showed that the (Wacker VB1321+Paraloid B-66) is the best material to consolidate the weakness of granite surfaces.

Conclusion

This study showed that the important causes of deterioration of granite in Valley Temple of Khafre due to environmental process property changes in temperatures and relative humidity between day and night .This phenomenon leads to disintegration and decomposition of granite, so a lot part of relief were removed.

Granite samples of studied area showed that: Bulk density = 2.54g/cm^3), Porosity = 4.87%, Water absorption = 2.4%, Dry – compressive strength = 148 kg/m^2. , Tensile strength = 11.5 kg/m^2, Losing by abrasive = 15.6g.

The Petrographic study showed that decomposition of K-Feldspars into Clay minerals, cleavage planes of Biotite.

To obtain the most consolidants, some acrylic products, silicone and mixture of the both were investigated before and after artificial aging. It showed that the paraliod B66 (diluted at 5% in trichloroethylene is the best material to consolidate disintegrated granite.

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