ORIGINAL PAPER

EVALUATING SOME OF CONSOLIDANTS APPLIED ON GRANITIC ROCK ART AT ASWAN QUARRIES, UPPER EGYPT

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Abstract. *This document Granite quarries in Aswan, south of Egypt, have many rock paintings and engravings. This rock art in Aswan's granite quarries is exposed to many weathering factors that threaten its survival. To protect these arts, the research focused on a comparative study to evaluate the application of certain reinforcing materials such as Paraloid B44, Paraloid B48, Estel 1000 and SILRES® BS OH 100 to strengthen crumbling rock. Examination and analysis methods such as polarized microscopy (PM), scanning electron microscopy and analysis with energy-dispersed X-ray unit (SEM-EDX) and X-ray diffraction analysis (XRD) have been used to determine the chemical and mineral composition of the rock and the degradation and damage to rocky art. These treatments were evaluated after artificial aging by scanning electron microscopy. The results show that treatment with Estel 1000 is suitable for studied rock paintings and engravings.*

Keywords: *Rock art, Granite, Aswan, Aging, Estel 1000, SEM.*

1. INTRODUCTION

Rock art is considered the oldest form of art, it is divided into rock painting and petroglyphs applied on rock surfaces [1]. Rock art is a significant source that provides valuable archaeological data [2]. It documents the cultural, social, economic activities, military events and religious relationships. In addition, data on exchanging patterns of trade and communication between different ancient peoples are also recorded [3]. Egypt is famous with its rock art sites spread across the desert lands and the quarries areas such as the granite quarries at Aswan. Kelany et al. [4] divided the granite quarries at eastern bank of Aswan to three main groups (Fig. 1); the Northern quarries; such as the Unfinished obelisk (S–1), the Fatimid cemetery (S–2), and three main quarry areas (S3, S4 and S5). The later quarries were used during the New Kingdom and Greco-Roman period. The Central quarries lie south of the Northern quarries and include five identified quarries (S6–S10); the Hatshepsut quarry (S8) and the Karor quarry are the most famous in this group. While the Southern quarries are the richest groups in the rock art and archaeological evidence related to the ancient quarrying, and contain the Shallal quarry (Ramses II statue quarry) (S13), the Shisah and Tlaina quarries. Also, Klemm and Klemm [5] identified islands of Saluga, Elephantine and Sehel as the main quarries for quarrying granite in ancient Egypt which contain also extensive rock art.

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1.1. THEMES OF ROCK ART AT ASWAN GRANITE QUARRIES

The rock art at studied area contains different and various themes such as: mining and quarrying [6], human scenes such as hunting and human handprints, animals scenes [7] such as cows, elephants and giraffes [8], dolphins and fishes [9], snakes, birds scenes such as ostrich (Fig. 2a), geometric scenes such as circles, stars and spiral figures, graffiti such as hieroglyphs, hieratic, Greek and Coptic[10], the themes of trade exchange, military expeditions (Fig. 2b), building roads and the excavation of canals in ancient Egypt [11], scenes of offerings of the kings to the gods, worshiping the kings and the priests to gods such as Khnum, Satis, Anukis, Amun, Sobak and Anubis or worshiping the priests to the kings [12, 13] in addition to official and personal inscriptions.

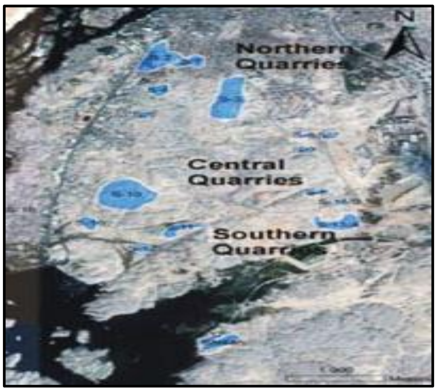

Figure 1. Map of Aswan quarries from Google Earth [4].

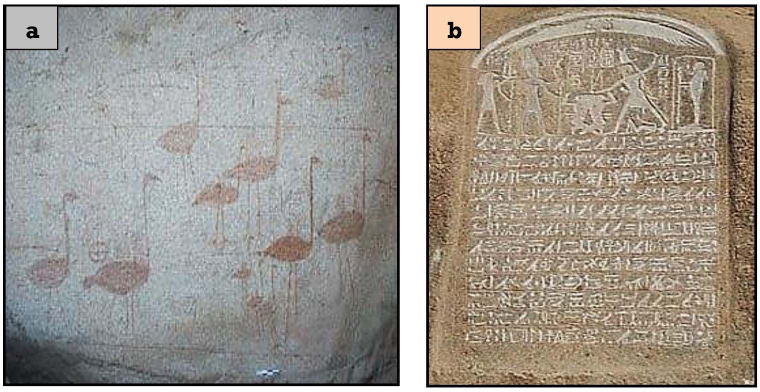

Figure 2. Themes of studied rock art: a) Ostrich paintings; b) Military scene of Amenhotep III.

2. GENERAL OVERVIEW OF CONSERVATION PROBLEM OF ROCK ART AT GRANITE QUARRIES

It is worth to mentioning that the place or natural environment of rock art coupled with the artistic quality and cultural importance often determine the scope and realization of the art because of the fact that it has been preserved at the place where it was carried out. The environmental (extrinsic) agents that affect rock art weathering in the granite quarries at Aswan are solar radiation, extreme temperatures [14, 15], wind, rains, moisture, salts [16, 17], microorganisms and higher plants [18] in addition to earthquakes, mass wasting [19] and geological structures such as faults, joints and places of intrinsic weakness [20].These factors coupled with the internal properties of the rock motivate and encourage both physical and chemical weathering processes of the granite rock art [21].The anthropogenic or human agents play fundamental role in the weathering of rock art at Aswan through various, multiple forms and methods such as; the application of the water and chalk on rock art during photography which known as physical enhancement [22] in addition to utilize rubbing, tracing and stippling techniques for archaeological documenting process [23-25], reuse rock art sites as temporarily necropolis [26], public parks and gardens, building modern houses on the rock art panels, abuse such military camps, modern activities such as quarrying and mining [27, 28], looting and cutting the rock panels for selling and also applying inappropriate treatments on the deteriorated rock art [29]. Another significant issue related to human-induced causes of rock art damage is bad social behaviors resulting from chaotic tourism visiting such as touching, cutting parts of the panels as souvenirs and vandalism through scribbling, chiseling and scratching the rock art [30], coating the rock art panels with synthetic paints and varnish [31].The previously mentioned agents cause weathering and deterioration rock art in different phenomena (Fig. 3) such as fissures, cracks and fractures, exfoliation, contour scaling, granular disintegration [32] also, the breakdown and toughening the exterior surface of the stone [33] discoloration, fading, and aesthetic disfigurement [34] in addition to changing the physical properties and chemical alterations of the granite rock art [35]. The extensive field visits to the studied rock art sites revealed that, the conservators frequently used group of conventional consolidants for strengthening the weathered rock art without any experimental tests to be sure of their appropriateness to the both, the rock art and the ambient environmental conditions, that issue caused additional damage to the rock art.

Figure 3. Deterioration phenomena of studied rock art: a) exfoliation; b) discoloration due to rains and inappropriate treatment; c) aesthetic disfigurement due to synthetic paints and chalk.

3. MATERIALS AND METHODS

3.1. PETROGRAPHIC EXAMINATION

Polarizing microscope (Olympus BX51 TF Japan, under 40X magnifications) was utilized to investigate the mineralogical composition of the untreated samples.

3.2. MORPHOLOGICAL FEATURES OF THE SAMPLES

The microstructure of the untreated, treated and treated aged samples, were observed by scanning electron microscope (SEM JEOL JSM 6400) attached with the energy dispersive X-ray spectrometer (SEM-EDX), this investigation was conducted to identify the fundamental elements of the studied samples and determine the morphology of the samples in addition to investigate the distribution, penetration and the behavior of the consolidants on treated and treated aged samples.

3. 3. X-RAY DIFFRACTION (XRD) INVESTIGATION

Determination of the mineral composition of the studied samples was performed by X-ray diffraction patterns, using a diffractometer type Philips PW 1840, operated at 35 kV, using a Cu Kα radiation wavelength of 1.540598 Å to identify the mineralogical, chemical composition and the deterioration features of the granite.

3.4. CONSOLIDANTS

The previous studies argued the use of the tested products (Table 1) in the conservation of the stones, it is worth to mentioning that studies recommended also that, testing these products on samples of stones before applying them in the field, the current study testes four consolidants which used frequently in Aswan quarries (Paraloid B44, Paraloid B48, Estel 1000 and SILRES® BS OH 100) [36-39] for studying them experimentally and assessment their effects on the treatment process through procedure accelerated artificial aging tests on treated granite samples and utilizing the scanning electron microscope (SEM) to examine the studied samples after each test.

Table 1. The consolidation products used in the study.

Consolidant used	Solvent	Composition	Chemical family
Paraloid B44	3% in acetone	MMA Copolymer	Acrylic Resin
Paraloid B48	3% in acetone	MMA Copolymer	Acrylic Resin
Estel 1000	diluted in white spirit 1:15	Tetraethyl Orthosilicate	Organosilicone
Silres® BS OH 100	diluted in white spirit 1:15	Tetraethoxy Silane	Ethyl Silicate

3.5. APPLICATION OF TREATMENTS

Granite blocks were collected from the Aswan quarries, then these blocks were cut as small cubic samples of 5 cm side length, after that the samples were washed with deionized water and cleaned with tooth brushes to remove the soil deposits, and then they were dried in an oven at 105 ˚C for 24 hours when the samples achieved a constant weight. Prior to the treatment process, the studied samples were brushed by acetone to open the porous of the stone. The application of the selected consolidants was performed by brush at room temperature and pressure to simulate the consolidation process in the site, and was repeated three times with interval 24 h between each application. Then the treated samples were left about three weeks to dry off and allowing for the polymerization process to take place. Some of the treated granite samples were subjected to investigation methods while the other treated samples were submitted to the accelerated (artificial) aging tests followed by SEM investigation to identify the changes of the tested consolidants after each test.

3.6. ARTIFICIAL AGING TESTS

The aim of these tests is to evaluate the effect of tested treatments, and identify the degree of their stability and their durability on stone strength after subjection to accelerated (artificial) aging tests, simulating the natural conditions of the studied rock art, and these tests include both artificial thermal aging and artificial salt weathering.

Thermal aging

This test was chosen to simulate the natural conditions affecting in the rock art sites at Aswan especially the high degrees of air temperature and solar radiation. This test was conducted according to Franzoni et al. [40] as follows: the samples were submitted to 30 cycles, each cycle consists of 2 hours of immersion in water at room temperature, followed by 2 hours of drying at room temperature, 18 hours of drying in an oven at 105 °C, and completed by 2 hours of cooling in room temperature.

Salt weathering (soundness test)

The purpose of this test was to determine the damage mechanism caused by sodium sulphate on the treated samples. This salt was selected to use as it is less aggressive [41], and more abundance than magnesium sulphate in the study area. According to [42-44], the samples were immersed for 4 hours in a saturated 10% sodium sulphate solution (Na_2SO_4) at 22 °C, removed from the solution, and allowed to dry for 12 hours, and then were placed for 8 hours in an oven at 105 °C until dry. The previous procedures were repeated for 15 cycles.

4. RESULTS AND DISCUSSIONS

4.1. PETROGRAPHIC EXAMINATION

The investigation of studied samples under the polarizing light microscope (PLM) (Fig. 4) showed that the granite samples consists essentially of quartz, plagioclase (albite) and sodic feldspars (microcline and orthoclase), in addition to biotite, as main constituents. The accessory minerals represented in zircon, hornblende, allanite and iron oxides. The petrographic study has identified the deterioration of granite is due to both physical and chemical weathering. The physical deterioration appeared through the presence of elongated crystals of minerals, corrosion of the grains bounders and long cracks due to the internal stress by high heat induced at the site. On the other hand, the chemical deterioration represented by transformation of both kind of feldspars to clay minerals particularly sericite and kaolinite, alteration of mafic minerals such as biotite and hornblende to iron oxides which lead to staining of the mineral grains with reddish or chocolate brown colours.

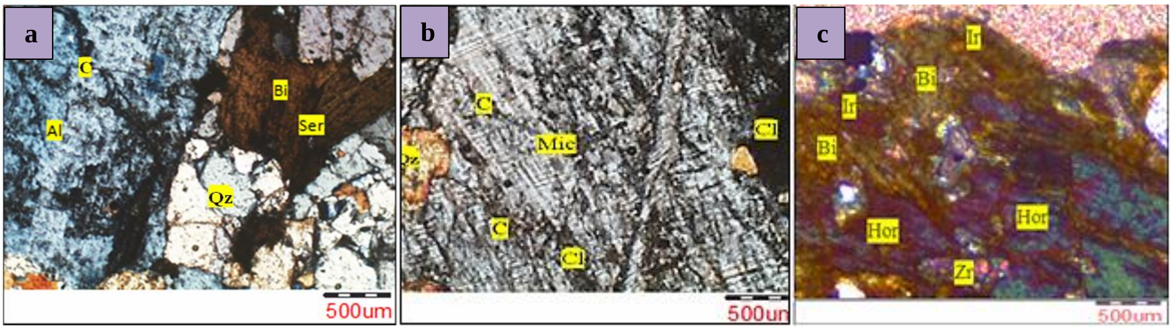

Figure 4. PM Photomicrographs of weathered granite samples.

4.2. INVESTIGATION BY SCANNING ELECTRON MICROSCOPE (SEM)

SEM micrographs of the weathered granite samples displayed several deterioration features such as, etching of the grain boundaries, corrosion pits in the feldspar grains, in addition to multiple gaps, voids, cracks, fissures and fractures generated by internal stresses due to the hydration and swelling of the secondary clay minerals [45-47]. Furthermore, the weathering of feldspar minerals and their alteration to clay minerals such as kaolinite booklets (Fig. 5a) due to the effect of chemical weathering predominant in the site. Also, the SEM micrographs showed the crystallization of sodium chloride crystals (halite) in typical cubic form (Fig. 5b) as a result of the salty solutions affecting the site. In the same manner, the groundwater bearing-soluble salts lead to form the calcium sulfate salts such as gypsum and anhydrite (Fig. 5c) which attributed to weathering of plagioclase.

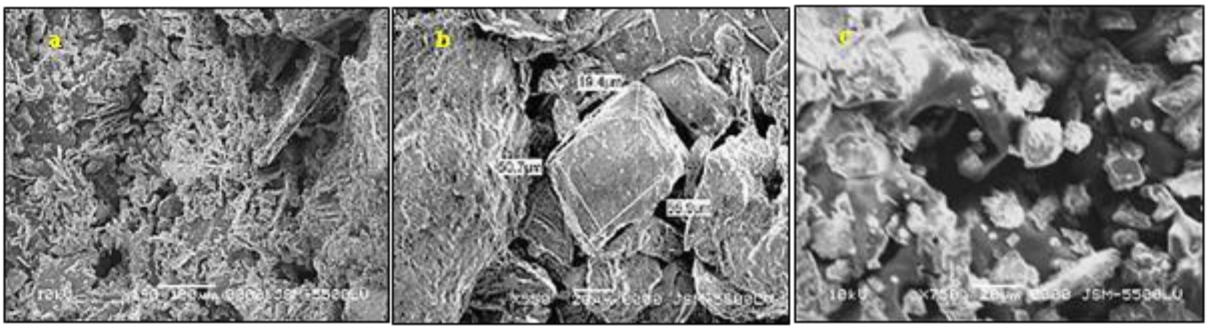

Figure 5. SEM Photomicrographs show the deterioration features of the studied samples.

4.3. XRD MINERALOGICAL ANALYSIS

The study of weathered samples by X-Ray diffraction analysis (XRD) (Fig. 6a-b) revealed that the granite rock consists mainly of quartz (SiO_2), albite ($NaAlSi_3O_8$), microcline ($KAlSi_3O_8$) and biotite $K(Mg,Fe)_3AlSi_3O_{10}(OH)_2$. The alteration products are represented in form of calcite ($CaCO_3$), anhydrite ($CaSO_4$) and high percent of halite (NaCl).

4.4. ENERGY DISPERSIVE X-RAY ANALYSIS (EDX)

The results obtained by energy dispersive X-ray analysis (EDX) for the deteriorated granite samples (Fig. 6c-d) are in accordance with the results obtained by XRD as following: the decrease of silica (Si) and iron (Fe) contents refer to the alteration process and hydration of iron oxides through changing ferrous to ferric ions which cause the staining and discoloration the granite.

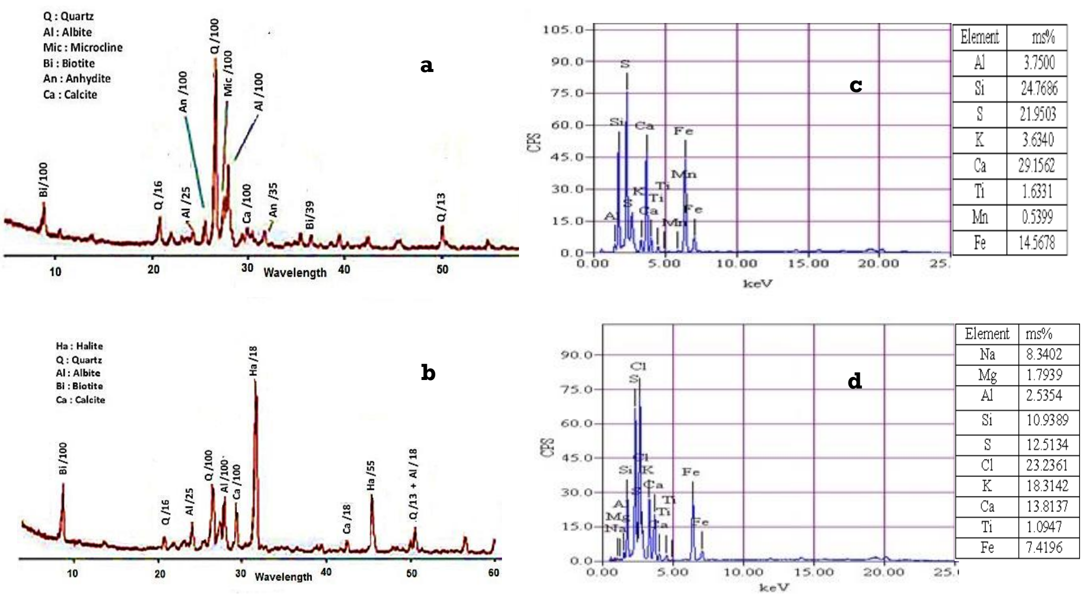

Figure 6. a-b) XRD patterns of weathered granite samples; c-d) EDX patterns of weathered granite samples.

The existence of potassium (K), aluminium (Al) and magnesium (Mg) are attributed to the main constituents in the rock such as albite, microcline and biotite. On the other hand, the presence of high percent of chlorine ions (Cl) from groundwater bearing soluble salts which react with sodium ions (Na) from weathered albite feldspar to form sodium chloride (NaCl). The high sulphur content (S) is attributed to the contaminated groundwater and air pollution from the traffic. These ions react with the high content of calcium (Ca) ions resulting from the chemical weathering of plagioclase feldspars to form phases of calcium sulphate (anhydrite or gypsum) which cause the destruction of the granite.

4.5. EVALUATION STUDY OF THE TREATED GRANITE SAMPLES AFTER ARTIFICIAL AGING TESTS BY SEM

Samples treated with PARALOID B-44

The microscopic examination showed that Paraloid B-44 has formed dense layers of polymer, which spreads on the surface in the form of clusters and nodules distributed between the mineral grains and covered them heterogeneously that led to form several fine holes that resemble the spongy appearance on the treated surface (Fig. 7a). After the thermal aging, Paraloid B-44 has been damaged and the resulting consolidation polymer has been cracked and the damage takes the form of caves and deep gaps due to the inability to resist high temperature (Fig. 7b). After cycles of salt weathering, very severe damage, fragmentation in the polymer layer, several gaps and cuts were formed due to the growth of saline crystals between the mineral grains (Fig. 7c).

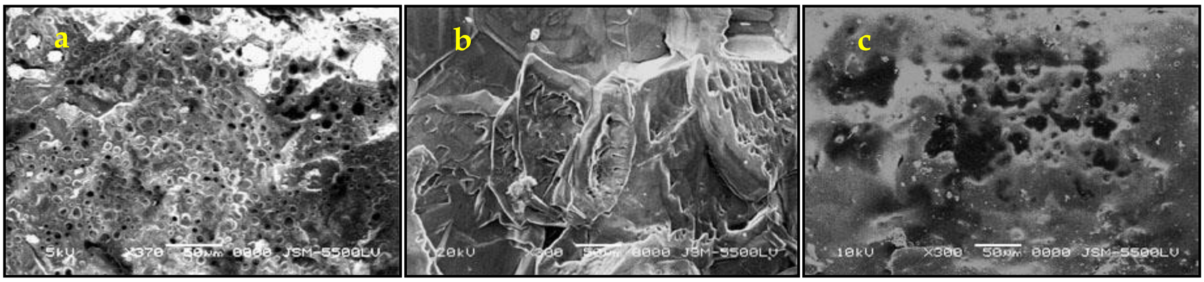

Figure 7. SEM of granite samples treated with Paraloid B 44: a) initial; b) after thermal ageing; c) salt ageing.

Samples treated with PARALOID B-48N

SEM examination showed that Paraloid B-48N formed very thick layers of consolidation polymer look like large islands, which lead to the excess coating of the grains and forbid the breathing of the stone (Fig. 8a).

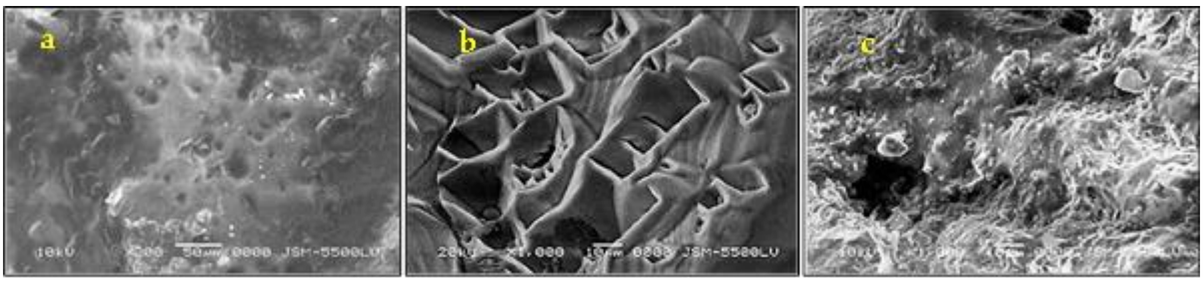

Figure 8. SEM of granite samples treated with Paraloid B 48: a) initial; b) after thermal ageing; c) salt ageing.

After the thermal aging, the consolidant damaged through the formation of surface cracks and cavities, in the form of caves or several deep grooves due to shrinkage by high heat (Fig. 8b).After the salt weathering, the consolidant was unable to resist the salt aging cycles and turned into a brittle film that lost the ability to bind the mineral grains and the damage appeared in the form of cracking, fragmentation, deep gaps due to the growth of heavy salt crystals (Fig. 8c).

Samples treated with ESTEL 1000

The examination of Estel 1000 by SEM showed that the polymer has precipitated on the surface of the stone in a good form, and coated the mineral grains, penetrated uniform in the spaces and voids between them, that led to form the network structure that linked the grains very well (Fig. 9a).After the thermal aging, the consolidant still maintains its previous properties prior to the thermal test, and it showed high resistance to the effect of high heat (Fig. 9b). After the salt aging, the consolidation material showed good stability to salt growth and forbids the formation the salts (Fig. 9c).

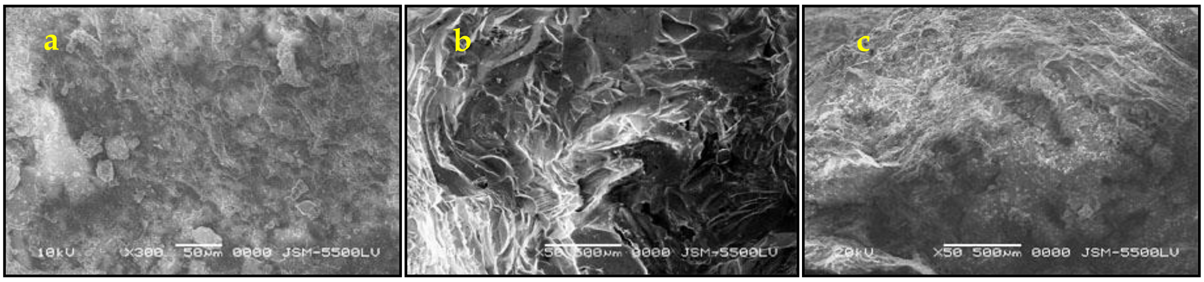

Figure 9. SEM of granite samples treated with Estel 1000: a) initial; b) after thermal ageing; c) salt ageing.

Samples treated with SILRES®BS OH 100

By examining the treated samples by SEM, it was found that Silres®BS OH 100 coated the grains well and was deposited in the cracks and spaces between grains and penetrated partially into the rock, although, small gaps and holes were developed in the consolidation film (Fig. 10a). After the thermal aging, the consolidant still maintains the good coat and homogeneous distribution of the polymer film on the surface, around the grains and in the spaces between grains, that indicated the relative resistance of the consolidant to the effect of high heat (Fig. 10b).After the salt aging, SEM showed that a thin layer of the polymer still covers the grains well, leaving the mineral grain-boundaries visible and consolidant has failed to fill the cracks and gaps, some salt crystals were formed on the surface of the grains (Fig. 10c).

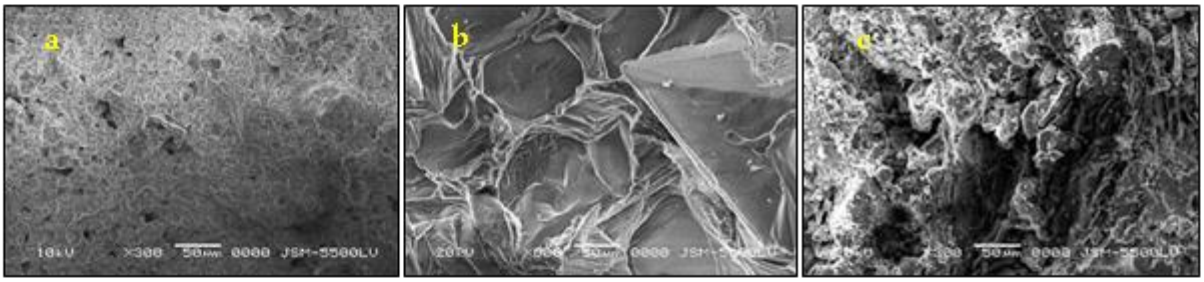

Figure 10. SEM of granite samples treated with Silres BS OH 100: a) initial; b) after thermal ageing; c) salt ageing.

5. CONCLUSIONS

During the last decade, the conservators in Aswan area used frequently the consolidation products; Paraloid B-44, Paraloid B- 48N, Estel 1000 and Silres® BS OH 100 to protect the granite rock art without investigation their negative effects on the treated rock art. The current study investigates the deterioration products of the studied rock art by PM, SEM-EDX and XRD. The petrography study showed that, the granite consists essentially of quartz, albite, microcline, orthoclase and biotite, as main constituents. Zircon, hornblende, allanite and iron oxides were represented the accessory minerals in the samples. PM identified the deterioration of granite due to both physical and chemical weathering, the chemical deterioration represented by transformation of both kind of feldspars to clay minerals particularly sericite and kaolinite. The alteration of biotite and hornblende to iron oxides lead to staining of the mineral grains with reddish or chocolate brown colours. The morphological study by SEM has cleared that, the deterioration of the studied rock art represented in etching, corrosion of the grains, existence the multiple gaps, cracks, fissures and fractures. SEM showed also that the alteration of feldspars minerals to clay minerals and the crystallization of sodium chloride and calcium sulfate salts such as gypsum and anhydrite. XRD study has identified that, the studied granite composes mainly of quartz (SiO_2), albite ($NaAlSi_3O_8$), microcline ($KAlSi_3O_8$) and biotite $K(Mg,Fe)_3AlSi_3O_{10}(OH)_2$. The alteration products represented in calcite ($CaCO_3$), anhydrite ($CaSO_4$) and high percent of halite salt (NaCl). EDX results showed that the granite is highly affected by chemical weathering through the decrease of silica (Si), alteration of the feldspars and hydration of iron oxides, in addition to the crystallization of sodium chloride and calcium sulphate.The results obtained have proved that, the success of Estel 1000 and Silres® BS OH 100 in good penetrating inside the fissures and voids and building up adhering bridges between them, well binding the friable mineral grains of the rock through formation networks structure, high stability to the effect of the high temperature (heat) in the thermal aging test, and good resistance to the salt weathering in spite of the second consolidant (Silres® BS OH 100) failed in the last test. Concerning the two other acrylic compounds it is necessary not to apply as consolidating materials on studied rock art, because of their ineffectiveness and bad performance against the artificial aging tests also, they are not miscible with the water in the pores of the stone and in the very arid areas like aswan exposed permanently to direct solar radiation which leads to breakdown in the Internal structure of acrylic consolidants causing supplementary damage of granite rock art. Finally the Estel 1000 is more appropriate one between the tested consolidation materials for the treatment of the studied rock art.

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