

## DAMAGE ASSESSMENT AND NANO TREATMENT OF THE SHARIA JUDGE TOMB AT THE FATIMID CEMETERY, ASWAN - EGYPT

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#### Abstract:

This paper focuses on the environmental hazards affecting the building materials at the Sharia Judge tomb in Aswan, abreast with finding out the appropriate Nano consolidant, to achieve the afore-mentioned purpose; the ambient environmental factors were studied and the deterioration products were noticed, building materials were studied using SEM-EDX, XRD and FTIR. Through SEM investigation; it was found that the plaster layers had a significant deterioration in addition to the spread of gaps, separations, cracks, and lack of interconnection between the grains. The analyses by EDX and XRD proved that the two layers of the plaster consist of calcium carbonate  $\text{CaCO}_3$  and quartz  $\text{SiO}_2$  with a percentage of halite  $\text{NaCl}$ , whitewash from gypsum  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  with a percentage of halite. Through FTIR analytical results it was found out the type of organic media used. It turned out that the media was from animal glue. The study aims to consolidate the layers of plaster that suffer from complete or partial separation by applying the consolidation with calcium nano hydroxide mix with  $\text{TiO}_2$  nanoparticles in different concentrations to consolidate the layers of the separated plaster on models similar in composition to the composition of the original plaster layers. The results indicated that the mixture consisting of calcium nano hydroxide mix with  $\text{TiO}_2$  nanoparticles 1:2 was the best for preserving the plaster layers of the tomb of the Sharia judge in the Fatimid cemetery in Aswan.

#### Keywords:

Sharia judge cemetery

Plaster layers

SEM-EDX

XRD

Nano-calcium hydroxide

$\text{TiO}_2$  nanoparticles

### 1. Introduction

Aswan city is located on the eastern side of the Nile at a latitude of  $22^\circ$  north of the Tropic of Cancer, fig. (1-a). It rises 85 meters above sea level [1]. Muslim historians called Aswan the name of the gap and sometimes the guarded gap of Aswan [2]. The area of the Fatimid (297-576 AH./ 910-1171 AD. [3,4]. Cemetery in Aswan is several kilometers, fig. (1-b). The architectural activity in building tombs and mausoleums in Aswan reached its peak, especially in the Abbasid period (132-656 AH.) [5,6], which preceded the advent of Ahmed Ibn Tulun not long before and then took a period of his rule [7].

Aswan was subjected to torrential rains in 1886, which led to the destruction of most of these tombs and shrines [8]. The urban development of the city also led to the demolition of many of these domes in order to widen the streets and create new streets, especially in the southern part of the city. Then, it became challenging to determine the true owner of the cemetery or even to date it accurately. Thus, history became limited only to tracing the architectural development of these tombs [9]. The Italian Monnier de Villar [10] studied the cemetery according to tombstones - with their text and the history mentioned

in them- that were collected. He analyzed their elements and architectural planning, numbered them, and fixed them on a map. Then, Creswell [11] dealt with the cemetery, relying in many places on Monnier's research [12].

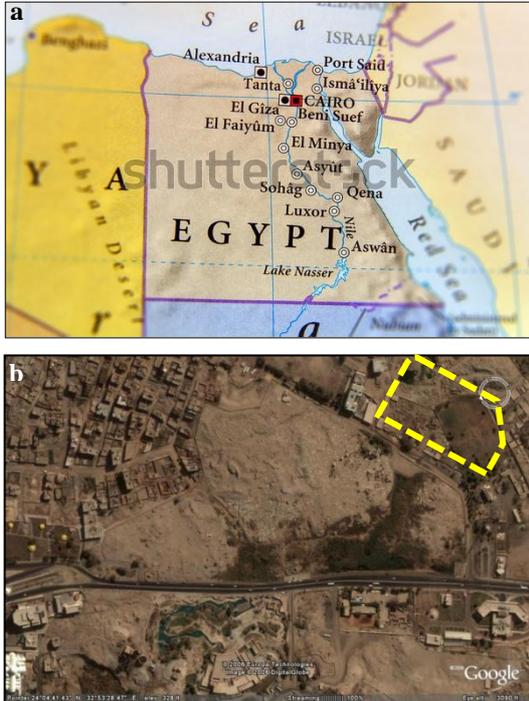


Figure (1) Shows **a**, Egypt map showing the location of Aswan city (After: Google map, 2022) **b**, the location of the Fatimid cemetery in Aswan

Architecturally, the cemetery is a square whose sides are 2.70 m. Its walls were built of mud. In the middle of the eastern side is a doorway with a pointed arch and in the middle is an apse of a mihrab erected with an arch. As for the other three sides, an opening with a pointed arch is in the middle of each side. The square is topped by an octagon with concave sides to show its edge in the form of horns, followed by the smooth dome body [12]. The Fatimid cemetery in Aswan includes ancient domes with unique architectural styles, fig. (2-a). However, the cemeteries generally suffer from the fall of plaster layers, whether in the form of small or large parts, fig. (2-b) as well as various sources of moisture. They also suffer from weeds, trees, and plants, which are irrigated with water, causing a rise in the content of the groundwater or gathering of grou-

ndwater inside cemeteries whose floors are lower than the surface of the earth, dome of Sidi Abdel Aal, fig. (2-c). The Sharia Judge cemetery is considered the oldest Islamic cemetery in Aswan and one of the oldest Islamic cemeteries in Egypt, as the construction of some tombs dates to the first Hijri century. It suffers from human damage, represented in the blotting of its walls in blue, cracks, and joints, fig. (2-d). It also suffers from falling and eroding layers of plaster, fig. (2-e). This study aims to preserve pigmented and non-pigmented plaster layers in the studied tomb, enforce these architectural treasures that extend for more than four centuries, draw the attention of specialists and officials of the antiquities of Islamic Aswan, and pave the way for further studies that help preserve this unique, diverse architectural heritage because of its archaeological and architectural importance of these tombs.



Figure (2) Shows **a**, some different forms of domes in the Fatimid cemetery in Aswan, **b**, fall off of small and large pieces of plaster, **c**, fall of the plaster layers, as well as the trees and plants **d**, accumulation of saline groundwater in the tomb of Sidi Abdel Aal, **e**, blue pigment that distorts the walls and covers the floor of the Sharia judge's cemetery, **f**, fall of the plaster layers with the pigments above from the tomb of the Sharia judge.

## 2. Materials and Method

### 2.1. Study materials

Some plaster layers and pigment layers from the separate parts of the tomb were collected as samples for examination and analysis.

### 2.2. Study methods

Gemini Zeiss-Ultra 55 Scanning Electron Microscopy attached to Energy Dispersive X-ray Spectroscopy (SEM-EDX) was used in the KTH department of materials and Nano physics, Stockholm, Sweden for investigating the morphological features of the samples after treatment. In addition to determine the elemental composition of the plaster layers before treatment. According to El-Gohary [13] Philips PW 1710 X-Ray diffraction with control unit anode material CU 40 K.V, 30 M.A, Optics was performed to define the mineralogical composition of the samples and weathering products. Within the same context, Nicolet 6700 FTIR spectrophotometer analysis was used to identify the media used with the pigment layer according to Cosentino [14].

## 3. Results

### 3.1. SEM-EDX analytical results

The morphology of the plaster samples of the Sharia Judge Tomb in the Fatimid cemetery examined using SEM-EDX. EDX. A constructed sample was found to be composed of calcium 74.43%, quartz 20,36%, and chlorine 5.21%, fig. (3-a). The sample of whitewash was found to be composed of calcium 43.23%, sulfur 45.12%, halite 2.42%, and sand 9.23%, fig. (3-a).

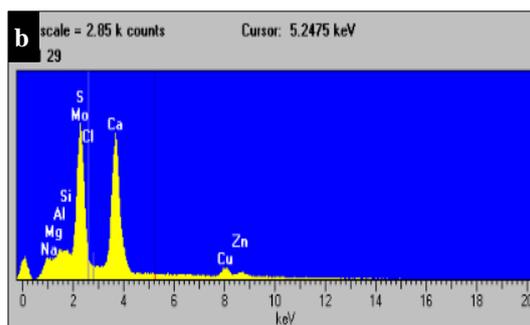
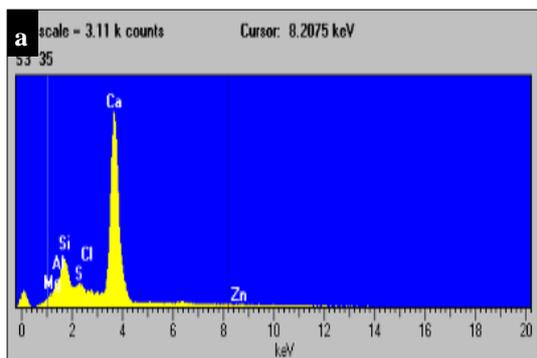


Figure (3) Shows EDX analysis of **a.** constructed sample composed of calcium, quartz, and halite, **b.** white lye sample contains calcium, sulfur, halite, and quartz.

### 3.2. XRD analytical results

Six different samples were analyzed from the cemetery of the Sharia Judge, and the results were - according to JCPDS 1967 [15,16]- as follows: sample (1) is a coarse plaster sample, had 43.6% calcium carbonate  $\text{CaCO}_3$  (lime) according to card no. 96-702-0140, 49.2%  $\text{SiO}_2$  (sand) in card no. 96-901-0145, and 7.2% sodium chloride  $\text{NaCl}$  (halite salt) in card no. 0630-900-96, fig. (4-a). Sample no. (2) is a whitewash sample- had calcium sulfate dihydrate  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (gypsum) by 98.2 % in card no. 96-500-0040, and  $\text{NaCl}$  (halite) sodium chloride at 1.8% card no. 96-900-6388, fig. (4-b). Sample no. (3) is a pigment sample composed of 92.9% calcium sulfate dehydrate; card. 96-500-0041, 5.1% sodium chloride  $\text{NaCl}$  (halite) card no. 96-900-6378, 1.1% hematite  $\text{Fe}_2\text{O}_3$  at card. 96-900-2161, and 0.9% goethite  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  card 96-434-4129, fig. (4-c). Regarding the sample (4), it is a pigment showed contains 94.3% calcium sulfate dehydrate; card 96-500-0041, and 5.2% sodium chloride  $\text{NaCl}$  (halite); card 96-900-6378, in addition to 0.5% goethite  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  by card no. 96-900-3081, fig. (4-d). Sample (5), is a blue pigment similar to the pigment of the blue washing flower distorted the walls and covered the cemetery floor. It had 100% ultramarine  $\text{Na}_6\text{Al}_4\text{Si}_6\text{S}_4\text{O}_{24}$  appeared at card no. 96-101-0317, fig. (4-e). Sample (6); it is a pigment of the blue washing flower showed 100% ultramarine card no. 101-96-0317, fig. (4-f).

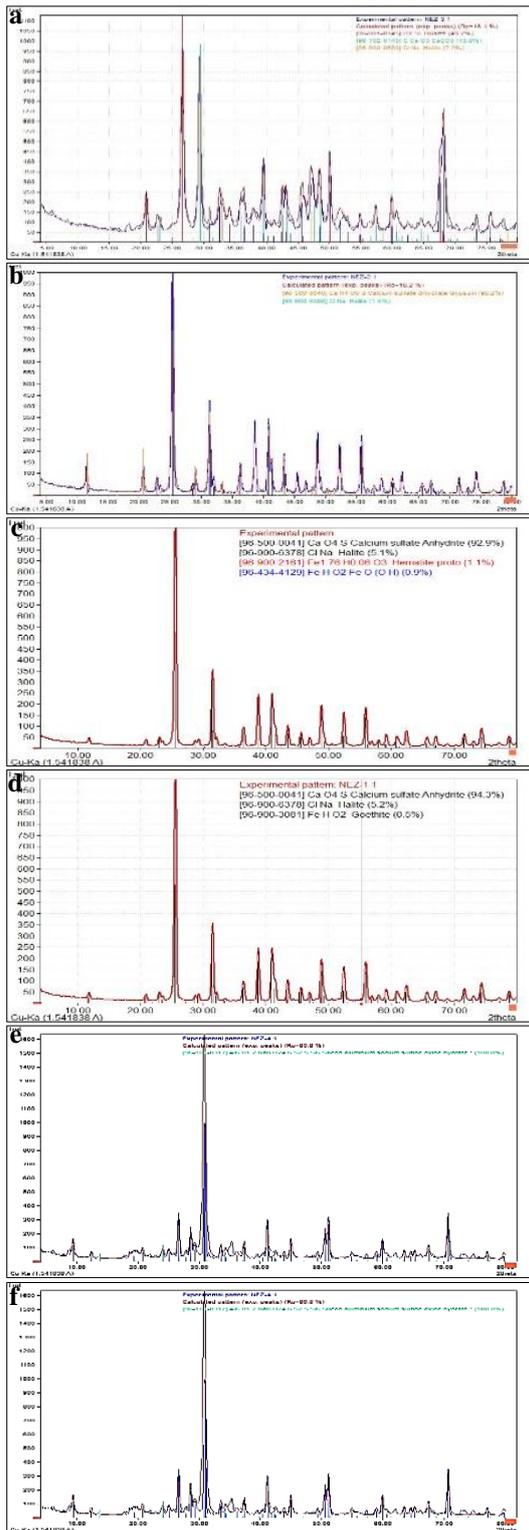


Figure (4) Shows X-ray diffraction patterns of **a.** constructed sample contains lime, sand, and halite, **b.** whitewash sample contains gypsum, halite, and salt, **c.** pigment sample contains gypsum, goethite, halite, and bixbyite, **d.** pigment sample contains gypsum, halite, and goethite, **e.** blue sample contains ultramarine blue, **f.** blue washing flower sample contains ultramarine blue.

### 3.3. FTIR analytical results

After matching the absorption areas of the archaeological sample with the absorption areas of the standard one of glue, it could be noted that it composed of glue. This result were close, according to Derrick, et al. [17], which indicating that the organic medium was animal glue, tab. (1) & fig. (5) Table No. (1) The convergence of the active groups in the tow samples.

Archaeological sample		Standar sample of Animal Glue	
Wavelength	Function groups	Wavelength	Function groups
3407 $\text{cm}^{-1}$ -3223 $\text{cm}^{-1}$	N-H stretching band	3400-3200 $\text{cm}^{-1}$	N-H stretching band
3010 $\text{cm}^{-1}$ -2911 $\text{cm}^{-1}$	C-H stretching bands	3100-2800 $\text{cm}^{-1}$	C-H stretching bands
1683 $\text{cm}^{-1}$	C=O stretching band	1660-1600 $\text{cm}^{-1}$	C=O stretching band
1614 $\text{cm}^{-1}$ -1517 $\text{cm}^{-1}$	C-N-H bending band	1565-1500 $\text{cm}^{-1}$	C-N-H bending band
1457 $\text{cm}^{-1}$	C-H bending band	1480-1300 $\text{cm}^{-1}$	C-H bending band
865 $\text{cm}^{-1}$ -677 $\text{cm}^{-1}$	N-H group	--	--

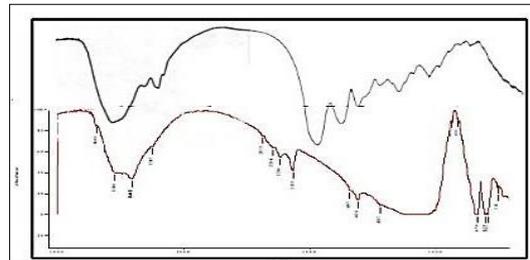


Figure (5) Shows FTIR pattern of the standard sample of animal glue and the archaeological pigment sample.

### 4. Application of the Consolidates to the simulated samples

The study was achieved upon the samples consolidated by calcium nano-hydroxide mixed with  $\text{TiO}_2$  nanoparticles, which were applied in different concentrations. It was carried out on samples to the high penetration and homogeneity of nanomaterials in the mineral composition [18]. The lime-based is a deep penetration consolidant because of the grain size [19,20]. Nanoparticle composites have attracted great interest in cultural heritage conservation [21]. Due to their unique multifunctional properties, nanoparticles have a role in producing hydrophobic surfaces [22]. Silane makes them more water-repellent [23], which can be used to protect stones [24]. Adding titanium dioxide and silica nanoparticles to polymers was also found to improve their mechanical properties [25]. The use of  $\text{TiO}_2$  nanoparticles to protect materials to increase the durability of these materials, especially in polluted environments, is encouraging [26]. The application of nano treatments on stones becomes a

useful tool for the realization of preserving and maintaining stone surfaces [27]. To reach the best results for reinforcing materials, they were applied several times after ensuring complete impregnation with an interval of two to three weeks to complete the polymerization process, fig. (6).



Figure (6) Shows **a.** simulation samples, **b.** calcium nano hydroxide, **c.** applying the consolidant.

#### 4.1. Visual examination

The aim of this step is to determine the extent of the change in the appearance of the treated surface of the reinforced samples. All the concentrations that were tested were successful.

#### 4.2. SEM-EDX examination

The objective of the examination is to compare the untreated and treated samples according to Briffa & Vella [28] and El-Gohary & Al-Shorman [29]. Moreover, evaluate the extent of the consolidant's ability to bind the granularity of the plaster, the presence of the mineral composition in a homogeneous manner for the concentration materials, as well as the ability to distribute, sediment, and fill the gaps. In this regard, the untreated (*archaeological sample*) showed the granular formation of the surface, its interspaces, and the surface texture, fig. (7-a). Within the same context, the treatment effectiveness of the second sample (*consolidated with 2:1 calcium nano hydroxide mixed with TiO<sub>2</sub> nano particles*) showed the spread and homogeneity of the treated material on the plaster layer, fig. (7-b). The third sample (*consolidated with 1:1 calcium nano hydroxide mixed with TiO<sub>2</sub> nano particles*) showed good diffusion, good distribution and clear penetration into the pores, fig. (7-c). The fourth sample (*consolidated with 1:2 calcium nano hydroxide mixed with TiO<sub>2</sub> nanoparticles*) showed the spread of TiO<sub>2</sub> nano particles on the plaster layer and increased the penetration bet-

ween the pores, in addition to the presence of good connection between the mineral composition, fig. (7-d).

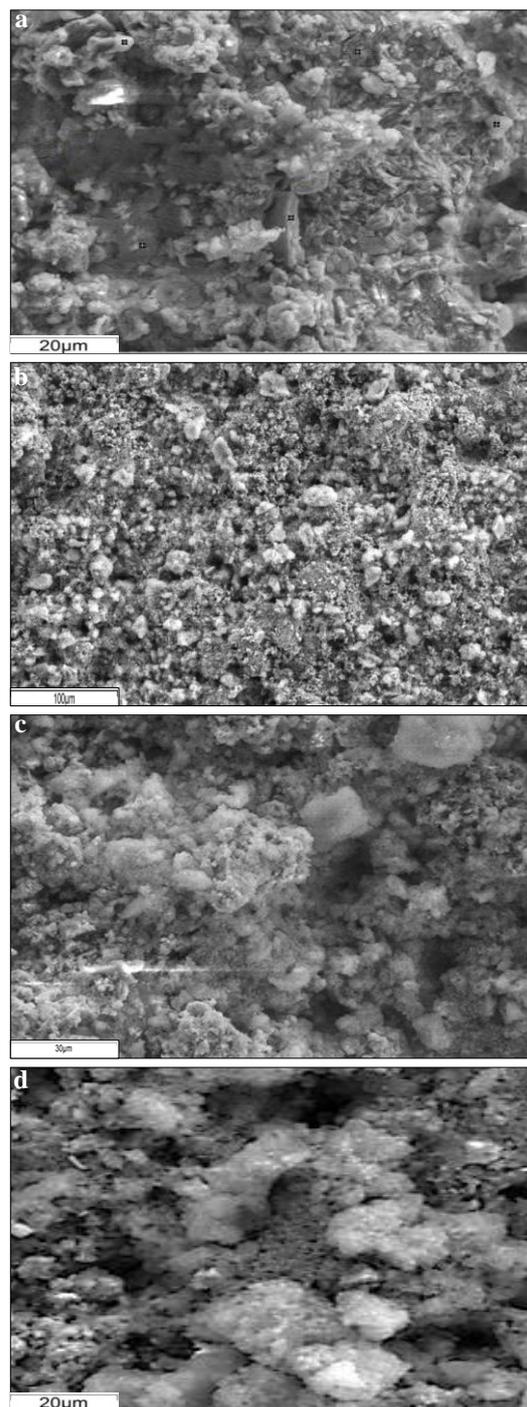


Figure (7) Shows SEM micrograph of **a.** morphology the lower layer of the Sharia Judge's tomb in the Fatimid cemetery, distribution of **b.** (2:1) nano calcium hydroxide mix with TiO<sub>2</sub> nanoparticles in treated plaster, **c.** (1:1) nano calcium hydroxide mix with TiO<sub>2</sub> nanoparticles of the treated plaster, **d.** (1:2) nano calcium hydroxide mix with TiO<sub>2</sub> nanoparticles treated plaster

## 5. Discussion

Based on the above examinations and analyses, the Fatimid cemetery in Aswan includes a group of domes unique in architectural styles. However, it suffered from various factors and forces of deterioration [13,30,31], which appeared by the accumulation of aerosols and dust on the archaeological surfaces, which led to the blurring of the aesthetic appearance of the monument. It needed to be cleaned using safe materials, such as gel-based systems [32]. The cemetery is like all archaeological sites that often suffer from severe environmental changes [33,34], such as various sources of moisture, which cause weakness, building materials decomposition, cracks, gaps, and the fall of plaster layers because most of the tombs were built of mud bricks, which was not able to cope with moisture in any of its forms. The moisture also helped grow weeds, which often caused fires that endangered the area. The archaeological cemetery has been used for burial until now. The plaster in the Sharia Judge tomb consisted of two layers; the first was rough, showing quartz  $\text{SiO}_2$  (sand) with a proportion of components up to 50%, and calcium carbonate  $\text{CaCO}_3$  (calcite) in a large percentage, as well as sodium chloride salt  $\text{NaCl}$  (halite), which indicates the presence of salts, and the second layer is fine. It had less sand proportion and more calcium carbonate proportion. The whitewash consisted of aqueous calcium sulfate  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (gypsum) in a large proportion with a small proportion in sodium chloride. The pigment in the cemetery was pink and consisted of aqueous calcium sulfate, sodium chloride, hematite  $\text{Fe}_2\text{O}_3$ , and goethite  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ . The blue pigment that distorted the walls and covered the floor of the cemetery was the pigment of the blue washing flower ultramarine  $\text{Na}_6\text{Al}_4\text{Si}_6\text{S}_4\text{O}_{24}$ . It also turned out that the organic medium used to mix the pigment in the cemetery of the Sharia Judge was animal glue. Due to the practical aspect, the

naked eye and the SEM examinations before and after the consolidation process showed that the consolidation with calcium hydroxide nano material mixed with  $\text{TiO}_2$  nanoparticles consolidation at a concentration of (2:1) achieved a remarkable spread to fill the gaps and microcracks. The consolidation with calcium hydroxide nano material mixed with  $\text{TiO}_2$  nano particles consolidation at a concentration of (1:1) achieved a better and satisfactory spread. The consolidation with calcium hydroxide nano material mixed with  $\text{TiO}_2$  nanoparticles consolidation at a concentration of (1:2) spread widely and worked to bind the granules better. It was also able to fill the joints and cracks that reached the depth with good distribution and spread between the cracks and gaps resulting from the mineral disintegration and the weakness of the internal structure. It is still necessary to conduct several new studies by making a mixture of nano-calcium hydroxide and other materials [35,36] to improve the properties of the preservative materials based on the dispersion of nano-calcium hydroxide.

## 6. Conclusion

*The Fatimid cemetery in Aswan suffered from many deterioration factors, especially moisture. The fine plaster layer in Sharia Judge Tomb had less sand proportion and more calcium carbonate proportion. The whitewash was calcium sulfate dehydrate. The pink pigment consisted of calcium sulfate dehydrate, sodium chloride, hematite, and goethite. The blue pigment that distorted the walls and covered the tomb floor was the blue washing flower ultramarine. The organic medium used in the Sharia Judge Tomb was animal glue. The consolidation with calcium hydroxide nano mixed with  $\text{TiO}_2$  nano particles at (1:2) concentration worked much better than the other two concentrations.*

## 7. Recommendations

- Scientific documentation should be achieved for all cemetery tombs.
- The cemetery tombs must be restored and conserved, especially the deteriorated ones.

- The use of the cemetery in new burials must be stopped.
- The walls of the cemetery must be completed to protect it.
- The cemetery must be cleaned from weeds and trees.
- The archaeological awareness of the Aswan people must be enhanced and promoted about the value of this cemetery.

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