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Diagnosis of construction stone state as a step in the conservation plan for Cairo Citadel Aqueduct

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Abstract

The aqueduct of the Cairo Citadel is one of the significant Ayyubid structures in Cairo, which dates back to 914 AH - 1508 AD, it was built for the purpose of transporting the Nile water to the mountain citadel with a length of 2.2 km., Unfortunately, the aqueduct of the Cairo Citadel is exposed to damage and extinction for many natural and human reasons. As a first stage in the restoration process, it is necessary to assess the existing state and make the proper diagnosis of this significant monument, it is necessary to identify the type and condition of the building stones used in the construction process and to achieve this goal some stone samples were collected for testing of physical and mechanical properties, using polarizing microscope (PLM), scanning electron microscope coupled with Energy dispersive X-Ray unit (SEM-EDX), X-ray diffraction (XRD), and X-ray fluorescence (XRF).

The study showed that the type of stone used for construction is fossiliferous limestone (Biomicrite) very fine to fine-grained. Micro-fossils and fossil fragments are present in significant amount scattered in the very fine-grained matrix, a tiny amount of clay mineral are present. The stone samples show 11.68% water absorption, 2.02 g/cm3 Bulk density 23.62% apparent porosity and 9.65 MPa compressive strength. Stone samples suffers from black hard crust, micro-cracks, crystallization of salts, in addition to microbial infection with Halophilic fungi growth. Based on the present study results and studying the surrounding environmental conditions, an appropriate restoration plan can be developed.

Key words: Cairo Citadel Aqueduct, Biomicrite limestone, deterioration aspects, investigation and analysis.

1. Introduction

The Cairo Citadel Aqueduct in the ancient Egypt region dates back to the Ayyubid era (Attia and Hassan, 2002, 4) and was rebuilt in the Mamluk era, and was used to transport water to the Cairo Citadel (Citadel of Saladin). After the use of that wall to transport water, during the reign of Khedive Ismail, the transportation of water was halted, it was neglected, and earthquakes led to a severe tendency in some of its arches, which it was then seen demolished to ward off the danger of its collapse at any moment. Those parts or arches have been reconstructed in the modern era with the same original materials as the antiquity, and in the same original form. (Mustafa, 2009, 112-113) (Fig. 1) Beginning at Fustat (along the coast of the Nile- Fom Al- Khalige), the Cairo Citadel Aqueduct ran to the Citadel of Salah al-Din al-Ayyubi. (Figure 2)

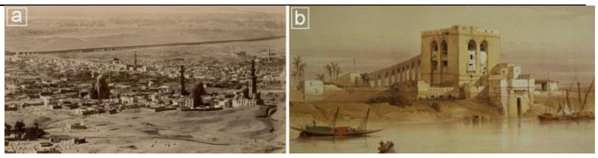

Fig. 1(a) View of the aqueduct (the dark line in the distance) passing through the Qarafa cemetery region on its way to the Citadel, in 1890[1] (en.wikipedia.org), (b) An 1838 illustration from the Fumm al-Khalig water intake tower [2](en.wikipedia.org)

1 https://en.wikipedia.org/wiki/Cairo_Citadel_Aqueduct, 26/6/2022- 19:53

2 https://en.wikipedia.org/wiki/Cairo_Citadel_Aqueduct, 26/6/2022- 19:53

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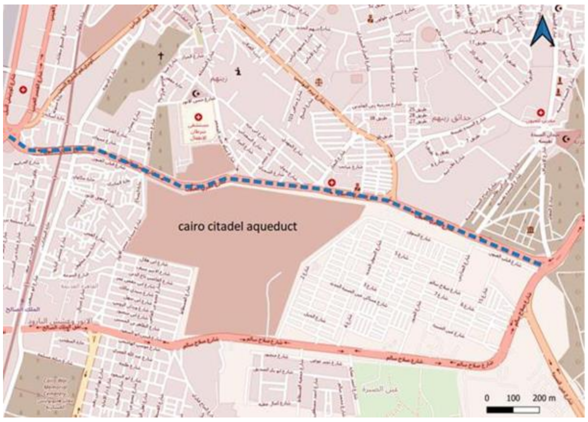

Fig.2. A map showing the location of the Cairo citadel aqueduct from the Nile to the Salah Salem St. (QGIS program)

There is a 2.2 kilometres wall between the waterwheel structure at the mouth of the Gulf and the citadel walls, and the thickness of the wall itself ranges from 2.5 to 3 m, and the rest of the space is filled with broken stone, mortar and some bricks (El Brambly, 2008: 5, 25-26). The land, along with other arches, was completely blocked, there are some dilapidated parts in the body of the Cairo Citadel Aqueduct, and there are only the foundations of the pillars, as a result of traffic accidents through the collision of its contracts with heavy transport vehicles, and other parts have been completely removed to establish a road network, and the metro line - the Helwan Railway- (Attia, and Hassan, 2002: 6). This first occurred after the wall was finished under Sultan Ghouri rule. all its arches were built as pointed stone arches Al-Wasiya Sabil and the current Salah Salem Street, but there were several restoration works on the wall that led to a change in its shape (El Brambly, 2008: 6) (El-Kasaby and Farag, 2015). (Fig. 3)

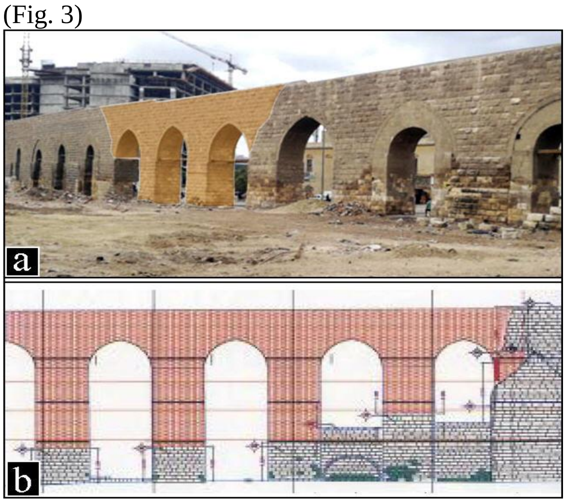

Fig. 3 a. The current condition of the Cairo citadel aqueduct, (b) sketch shows some newly completed arches

The Ayyubid structures built with limestone are of a similar nature, in terms of the geological composition, and the damage they suffer from, due to the similar conditions of their existence and the factors affecting them. Egyptian limestone is considered to be vulnerable to weathering compared with other limestone; the arid climate of Cairo only serves to exacerbate the deterioration of the naturally susceptible Egyptian stone. Deterioration processes, generally detrimental to any stone's ability to reduce the integrity of the masonry structure, are particularly harmful in such an inherently weak limestone (McCormack, 2001, 9) (Fitzner et al., 2002).

The two factors responsible for the Egyptian limestone deterioration are the high content of soluble salts and the clay content. Water acts as a medium that facilitates the movement of salts into and within the stone. Once inside the stone, salts can gradually deteriorate it as they are crystallized from evaporating water; the extent of the harm depends on the stone's porosity., and the type of salts present within the stone (McCormack, 2001, 6-7). These soluble salts are the primary cause of stone deterioration due to their repeated dissolution and crystallization cycles (Moon, 2002, 8-9). Salts move through a porous body of stone when dissolved in water. The presence of water in the pores can originate from capillarity, infiltration, condensation, and hygroscopicity (Moon, 2002, 9). Salt crystallization can damage stone building and make a serious damaged not only for stone. (Saleh, 2005, 69).

It has been observed that the stones of the Ayyubid walls of Cairo suffer from the blooms and crystallization of salts, which form scales of different thickness, in addition to many fine cracks. (McCormack, 2001, 12-16). These are the same problems experienced by the limestone used in the construction of Cairo Citadel Aqueduct.

According to importance of Cairo citadel aqueduct is a unique monument as well as subjecting it to many different manifestations of damage, and in line with the state’s plan to preserve it, our responsibility is to make a scholarly contribution to the procedures of archaeological documentation in order to aid the development of the neighbourhood., and to diagnose its condition, in order to find an appropriate treatment and maintenance plan for it.

1. Materials and Methods

To characterize the building stones used in the construction of the Cairo citadel aqueduct, some samples were collected from partially separated areas of the walls, and falling below, The samples description are shown in the (Table 1). At the National Research Centre laboratories, 4 cubic (3X3X3 cm) samples were examined in accordance with ASTM C97/C97M - 2015 and ASTM C170/C170M - 2015 to assess physical and mechanical properties (SOILTEST, INC. 86 ALBRECHT DRIVE LAKE BLUFF, IL 60044 U. S. A., Model CT- b200- 8), The Polarizing Microscope (PLM) leitz orthoplan, Camera: leica MC 190 HD at (The Egyptian Mineral Resources Authority laboratories (EMRA)) was used for the petrographic study, Scanning Electron Microscope coupled with Energy dispersive X-ray unit (SEM- EDX) SEM Model Quanta FEG 250, and type of sample a solid sample coated with gold. X-ray Diffraction (XRD) BRUKER AXS D8 ADVANCE Diffractometer, type of sample was powder, Copper (Cu) anode, Angle (2θ) from 15–60°, with a step of 0.2° min−1, 20 M A- 35 Kv, Analysis software by Match3+PDF4 2015. X-ray Fluorescence (XRF), Axios Sequential WD XRF Spectrometer, type of sample was powder, 4kW output at 160 mA, Analysis software by ASTM E 1621& ASTM D 7348, the SEM, XRD and XRF analysis were carried out in National Research Center laboratories, Giza.

Table (1): The samples description (codes, dimensions, and color).

Sample code	Length (cm)	Width (cm)	Thickness (cm)	color	Sample Photo
L1	**04.5**	**1.5 - 3**	**0.5 - 1**	**Yellowish, the tool marks of sculpting tools appear on the surface and the effect of air pollution in the form of a thin black layer**	
L2	**12.0**	**3 - 6**	**1.5 - 2**	**yellowish white with dark black crust on the stone surface**	
L3	**03.5**	**0.5 - 1.5**	**0.5 - 0.75**	**yellowish white**	

2. Results

2.1. Physical and Mechanical Properties

Four samples from the same type of archaeological specimen stone, were prepared in the form of cubes with the dimensions 3X3X3 cm, these samples were used to determine both water absorption and bulk density.

- Procedure: According to ASTM C97/C97M – 2015

a. Calculate the water absorption for each specimen as follows equation:

Absorption, weight % = ((B –A) /A) × 100 (eq. 1)

Where:

A = weight of the dried specimen, (g), and

B = weight of the specimen after immersion, (g).

The average water absorption of all samples is 11.86% (Table 2)

b. Calculate the bulk density for each specimen as follows equation:

Density= weight/ volume (gm/ cm3) (eq. 2)

Which calculates as follows:

Dry the specimens for 48 h in a ventilated oven at a temperature of 60 ± 2°C. At the 46th, 47th, and 48th hour, weigh the specimens to ensure that the weight is the same

Determine the three dimensions of the cubes (Table 2-2), and then calculate their volum

Substitution in the previous equation (eq. 2)

The average bulk density of all samples is 2.02 gm/ cm3 (Table 2)

c. Calculate apparent porosity for each specimen as follows equation:

Ø %= bulk density × water absorption % (eq. 3)

The average apparent porosity of all samples is 23.62% (Table 2)

- Procedure: According to ASTM C170/C170M– 2015

a. The compressive strength of each specimen was calculated as follows,

C = W/A (eq.4)

Where:

C = compressive strength of the specimen, MPa

W = total load, N, on the specimen failure

A = calculated area of the bearing surface in mm^2

b. Round each individual result to the nearest 1 MPa

The sample's average compressive strength is determined as 9.65 MPa, which is the average compressive strength ratio over all samples. (Table 3)

Table 2: Physical properties of stone samples

Sample No.	Dry wt. (g)	SSD wt. (g)	Water absorption (%)	Bulk density (g/cm^3)	Apparent porosity (%)
1	**9.4222**	**10.4746**	**11.17**	**1.9787**	**22.10**
2	**11.1558**	**12.4750**	**11.83**	**2.0368**	**24.09**
3	**13.8573**	**15.5324**	**12.09**	**2.0289**	**24.53**
4	**10.7251**	**11.9734**	**11.64**	**2.0433**	**23.78**
Average			***11.68***	***2.02***	***23.62***

*SSD wt.: Weight of soaked and surface-dried specimen in air

Table 3: Mechanical properties of stone samples

Sample No.	Length, (mm)	Width, (mm)	Area, (mm^{2})	Load (kN)	Compressive strength (MPa)
1	**19.32**	**15.61**	**301.5852**	**2.8**	**9.28**
2	**21.34**	**18.32**	**390.9488**	**3.9**	**9.98**
3	**21.77**	**17.03**	**370.7431**	**3.5**	**9.44**
4	**18.68**	**15.13**	**282.6284**	**2.8**	**9.91**
Average					***9.65***

2.2. Petrography

Two thin-sections were taken prepared from the samples obtained from Cairo Citadel Aqueduct limestone, in order to be investigated with the polarizing microscope (63x) the sample result showed as follows (Mackenzie,Guilford,1986):

- The sample is very fine to fine-grained. Microfossils (Foraminifera, gastropod) and fossil fragments are present in significant amount scattered in the very fine-grained matrix. Many fine pore spaces are present in the sample. Micro-cracks and rare amounts of quartz, iron oxides and opaque minerals (Figure 4, a-f).

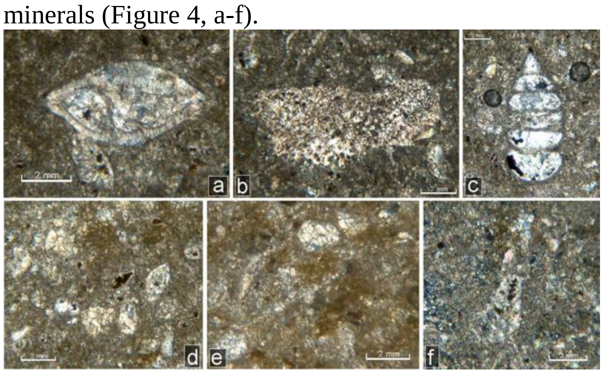

Fig. (4 a-f). Recrystallized Nummulites and Gastropods embedded in dense micro-sparry calcite groundmass.

2.3. SEM- EDX result

The small (less than 0.5 cm) sample is studied in a high vacuum by mean of an electron beam accelerated at around 20 kV. A scanning electron detector and amplifiers produce a displayed picture with a very good depth of field this picture can be photographed. Scanning electron microscope (SEM) was used to investigate the morphology of the stone surface and the samples show same results such as:-

The limestone surface is deteriorated, pore spaces are present in different places in the samples, salt crystals varied in size are noticed, a tiny amount of clay mineral are present. On the other hand, microbial infection is appear (Figure 5 a-d), (Figure 6 a-c), and (Table 4).

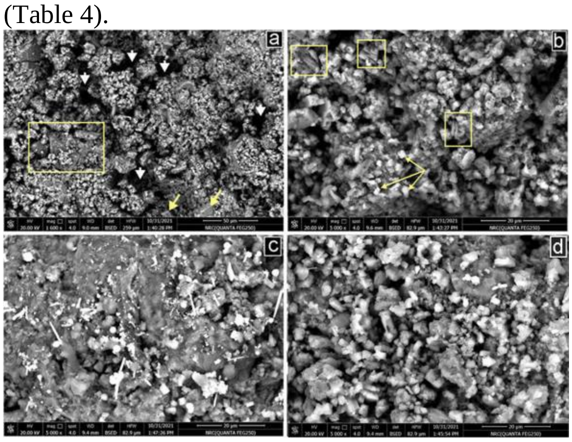

Fig. (5). (a). White arrow shows gaps scattered on the limestone surface, the rectangle shows granular disintegration and yellow arrow shows salt crystal (b). Clay minerals appear inside the rectangle shape and yellow arrow shows salt crystal (c). Microbial infection appears to grow on the limestone surface at high salinity, Halophilic fungi, the hyphae of fungi was clearly shown (d) Surface granular disintegration and salt crystal spread on the limestone surface.

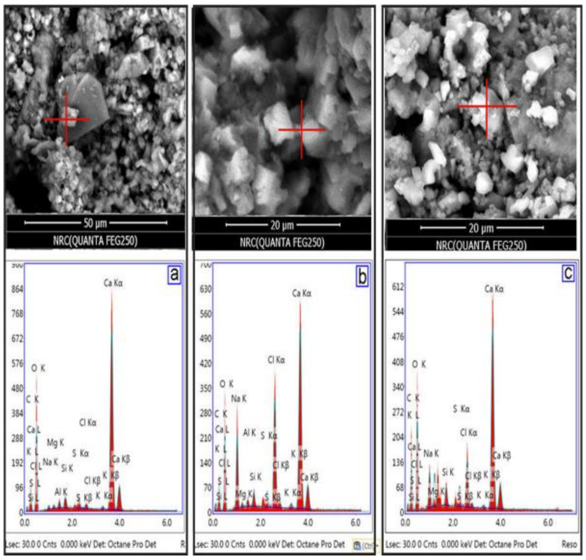

Fig. (6). (a) micro-sparry calcite crystals, (b) halite crystals are present (c) salt crystals and roses

2.4. XRD Results

The samples for X-ray Diffraction were collected to detect the actual statues of the Cairo citadel aqueduct limestone. The samples were tested by the XRD in order to determine the mineralogical composition of stone samples. The results of XRD had been shown that:

Stone samples mainly composed of Calcite $CaCO_3$ as a major component and traces of Halite NaCl, Kaolinite, $Al_2Si_2O_5(OH)_4$ and Hematite, Fe_2O_3 (Table 5)

2.5. XRF Result

The samples for X-ray Fluorescence were collected to detect the actual statues of the Cairo citadel aqueduct stones. The samples were tested by the XRF in order to determine the chemical elements of stone samples, (Table 6).

Table 4: The EDX weight percentage of elements in the sample

Element Wt%	C	O	Na	Mg	Al	Si	S	Cl	K	Ca	Total
Spot 1 (a)	11.85	50.54	01.21	00.54	00.82	01.18	01.46	00.68	00.43	31.30	100
Spot 2 (b)	16.16	34.85	11.42	00.56	00.73	01.08	01.31	09.06	00.55	24.28	100
Spot 3 (c)	24.04	38.63	05.41	03.13	00.00	01.28	01.50	03.58	00.26	22.17	100

Table 5. the result of X-ray diffraction analysis

Mineral	Chemical formula	Percentage %
Calcite	$CaCO_3$	95.147
Halite	NaCl	02.283
Kaolinite	$Al_2Si_2O_5(OH)_4$	02.283
Hematite	Fe_2O_3	00.285

Table 6: The XRF main constituents of sample

Main Constituents	(Wt. %)	Main Constituents	(Wt. %)	Main Constituents	(Wt. %)	Main Constituents	(Wt. %)
Na_2O	0.426	SiO_2	2.995	K_2O	0.323	SO_3	0.778
MgO	0.853	Cl	0.913	CaO	47.022	$Fe_2O_3^{tot}$.	0.887
Al_2O_3	1.463						
LOI (loss on ignition)		43.650					

According to percentage of calcium oxide (47.022%) the stone sample mainly composed of Calcite (CaCO3= ~ 84.05 %), also has quartz (SiO2) (2.9995%) and halite salts (NaCl) (Na_2O %= 0.426%), hematite (Fe_2O_3) (0.887%)

3. Result Discussions

By studying limestone samples taken from Cairo citadel aqueduct it was found that, **The physical properties** of limestone samples have average apparent porosity percentage is 23.62%, with average water absorption 11.68, and bulk density 2.02 g/cm^3, the results are close to the limestone samples constructed for the walls of Cairo, (Bourguignon, Elsa, 2000,82- 83). The limestone samples have average compressive strength value 9.65 MPa, which between 11.7 MPa for load-bearing masonry units (ES 1292-1 (2005), and ASTM C90/2003), and 3.45 MPa for non-load-bearing masonry units (ES 1292-2 (2005), and ASTM C 129/2003). This indicates the construction engineer's awareness of choosing a good type of building stone.

The petrography study showed that, the limestone samples are organic, carbonate sedimentary rock, fossiliferous limestone (Biomicrite) according Folk classification (Folk, R.L. 1962). The samples are very fine to fine-grained. Micro-fossils and fossil fragments are present in significant amount scattered in the very fine-grained matrix. Many fine pore spaces are present in the sample. The primary mineral ingredient of the stone is calcite with trace amounts of quartz, iron oxides and clay minerals. Calcite occurs as very fine-grained (micrite), anhedral crystals that represent the majority of the matrix of the sample. A significant amount of micro-fossils and shell fragments of different sizes and shapes are scattered in the matrix of the sample. Some micro-fossils are filled with recrystallized calcite. Quartz occurs as very fine to fine-grained, anhedral crystals scattered in the matrix. Iron oxides and clay minerals are scattered on the rock. Iron oxide stains may be seen in some areas of the stone. According to (N. Aly et al. 2015), the Helwan area quarry south of Mokattam, which is perhaps the most well-known limestone quarry in Egypt, is the location of the biomicrite to fossiliferous micrite (Mid-Eocene Mokattam Group) (Ahmed H., 2015), (Park a H.D., Shin G.H., 2009).

SEM- EDX for samples appears that, gaps spread on the limestone surface due to dissolution of micro-fossils and shell fragments of different sizes and shapes. The salt crystals of halite appear in their cubic shape, which indicates the availability of a source of salt supply and the availability of appropriate conditions of ventilation, temperatures and time that help in the formation of the cubic crystal shape of halite salt. the hyphae of Halophilic fungi was clearly shown as a result of microbiological activity due to saline conditions on the stone building (Maisa M.A. Mansour 2017). In sedimentary contexts, the clay minerals (kaolinite) is present as a secondary altering mineral. To prevent the damaging effect of the crystallization of salts inside the limestone, different types of salt inhibitors such as 0.01 M polyacrylic acid and 0.05 M amino trimethylene phosphonic acid were used.(Saleh, M. et al., 2022)

Investigations using XRD and XRF demonstrated that the degraded stone portions contain two groups of minerals: calcite, a significant mineral, and quartz, halite, kaolinite, and hematite, which are trace minerals.

Here, it can predict the role of both halite salt and kaolinite, which absorb moisture, as they are hygroscopic materials, and accordingly, moisture and halite salt provide the high salinity appropriate environment for the growth of Halophilic fungi.

One of the most hazardous elements in the weathering of monumental stones across the world is salt deterioration. Understanding the mechanisms of salt weathering, the conservators may put effective treatment strategies in place to stop or at least postpone such degradation. Egyptians have used limestone in the construction of Cairo buildings from ancient times. Over time, these buildings were subjected to various weathering processes, including salt. The alternating processes of salt dissolution, crystallisation, and hydration are the mechanics of stone degradation. This process results in the formation of several geomorphologic features of stone degradation, such alveolus or honeycombs, Tafoni, or cavernous weathering. A dangerous cause of damage, salt crystal formation within the porous stone can lead to surface erosion or even structural failure.(Saleh, M. et al., 2022).

Subsurface water is one of the most important sources of moisture that threatens the cohesion of the archaeological stone, which is found in fluctuating levels around the stone foundation of the wall. Cairo University, upon a mandate from the Supreme Council of Antiquities of the Center for archeology and environment Engineering at the Faculty of Engineering - Cairo University (El Brambly, Hossam, 2008: 30)., has carried out pallet work, examined the soil, assessed the condition of the foundations of the Cairo citadel aqueduct in their current condition, monitored the subsurface water level, determined its sources and its pH value. It was found that the pH value ranges between acidity and alkalinity, which varies from one place to another depending on the source of the subsurface water. Among the most important sources of subsurface water and at the same time sources of soluble salts, especially chlorides and sulfates in the area, sewage and industrial drainage, especially for tanneries that was located around the Cairo citadel aqueduct for long periods of time before its transfer, and other industrial activities associated with it. (El Brambly, Hossam, 2008: 30- 40).

Also, the activity of microorganisms and its vital wastes have a destructive effect on the stones, as they reduce the cohesion of their granules, and raise the percentage of soluble salts in them, causing more separations, crusts and microcracks in the stone body. The accumulated garbage under the Cairo citadel aqueduct decades, dirt, soot and dust on the surface of the impact, and the moisture in the stone is a suitable environment for its reproduction and destructive activity. Among the most important sources of dirt are car exhaust, fires lit by residents under the Cairo citadel aqueduct's arches, and the remnants of industrial areas surrounding the Cairo citadel aqueduct. (Attia, Amr- Hassan, Ghada, 2002: 4-5)

It was noted that the limestone used in the construction of the Cairo citadel aqueduct contains iron oxides resulting from its rust, due to the high humidity in the stone, the activity of microorganisms, and the chemicals leaking with industrial wastewater inside the stone through the subsurface water, and rust foci lead to chemical changes in the composition of minerals stone components, causing the disintegration of stone grains, and the occurrence of minute cracks, causing damage to the stone.

4. Conclusions

The limestone built by Cairo citadel aqueduct was cutted from Helwan area quarry south of Mokattam, it is suffers from dirt caused by vehicle exhaust, neighboring industrial activities and arson. Also suffers from separations, fine scales and microcracks as a result of the crystallization of dissolved salts between its grains that return to the repeated dissolution and crystallization cycles. About the presence of iron minerals, which may have come from the stone itself, or carried by industrial wastewater, which accompanies the activities of tanning leather that were next to it. In addition to presence of microbiological activity on the surface and between the stone grains, which found a suitable environment was for their growth.

Therefore, the treatment and conservation program must include getting rid of dissolved salts, reducing the percentage of subsurface water, by drying its sources, such as changing the sewage network, transferring industrial activities adjacent to the monument, as well as discouraging microbiological activity, and finding a site around the monument that preserves it from various infringements. If there are cases of reconstruction of damaged parts of the Cairo citadel aqueduct, building stones must be brought from Helwan area quarry south of Mokattam.

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