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EVALUATING THE USE OF AMMONIUM OXALATES (AMOX) IN THE CONSOLIDATION OF LIMESTONE IN THE TEMPLE OF PTOLEMY XII (AULITS) IN ATHRIBIS, SOHAG

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Abstract:
The temple of Ptolemy XII (Aulits) in Athribis to the west of Sohag, Upper Egypt, was built from several types of limestone from the mountain range adjacent to the west of the temple site. This paper, on the one hand, explains the types of limestone used in the construction and engraving of the temple's architectural elements. It explores the manifestations of damage that affected these stones, such as cracks, stratigraphic separation, and granular disintegration. On the other hand, it discusses the evaluation results of ammonium oxalates used in treating the temple's stone using different techniques, such as defining the weight changes, visual appearance by USB stereo microscope (SM), important physio-mechanical. Abrasion resistance was determined using a Standard wide wheel, and the hydrophobicity was evaluated by an Automated contact angle meter. Finally, a Quanta 250 SEM-EDX was used to investigate the morphological features and elemental composition of the samples. The results proved that ammonium oxalates gave the best results in most experimental tests, where, it achieved sufficient consolidation target for temple's stone and success in maintaining the general appearance of the treated experimental samples, Furthermore, the results of physical and mechanical properties measurements showed an improvement of the average physical properties of the samples after being treated with ammonium oxalates. In addition, it showed an improvement in abrasion resistance rates and an increase in hydrophobicity of limestone samples after the treatment process Finally, SEM examination results illustrated the good diffusion of ammonium oxalates and their success in binding the separated calcite grains while maintaining the natural pores of the experimental limestone samples. Thus, it was applied as a consolidant of limestone in this temple.

1. Introduction

Although stones have been considered one of the most stable materials used in structures and buildings since prehistoric times, it is well known that they are subjected to natural weathering and deterioration, potentially leading to dangerous, severe, and sometimes rapid consequences [1]. According to many researchers [2-8] Although stones have been considered one of the most stable materials used in structures and buildings since prehistoric times, it is well known that they are subjected to natural weathering and deterioration, potentially leading to dangerous, severe, and sometimes rapid consequences. So, to provide a realistic evaluation of conservation treatments, it is essential to carefully determine the factors responsible for the observed effects whether they result from the parameters of the consolidation procedure or the properties of the substrate itself [9]. The choice of an effective consolidant for carbonate stones is a key objective in cultural heritage conservation, particularly when aiming to slow down or stop aging effects. From a specialized point of view, the consolidation of limestone using inorganic consolidants has gained increasing relevance in recent years, as reported by Bracci, et al [10]. In this context, several types of these consolidants were used to consolidate limestone buildings, including lime water, $Ca(OH)_2$ [11], barium hydroxide solutions [$Ba(OH)_2$] [12], barium hydroxide $Ba(OH)_2$ [13] and ammonium oxalate [$(NH_4)2C_2O_4$] [14].

1.1. *Ammonium oxalate*

Ammonium oxalate ($C_2H_2O_4.2H_3N$) is an odorless solid that sinks and mixes slowly with water [15]. It is a salt consisting of two ammonium cations (NH_4^+) and one oxalate anion ($C_2O_2^{-4}$) [16]. It is an extremely rare natural organic mineral derived from guano [17], and is typically acidified to pH 3 using oxalic acid [18]. *AmOx* is characterized by its completely colourless form and is produced according to the following reaction: $CaCO_3+(NH_4)\ 2C_2O_4 \rightarrow CaC_2O_4+ 2NH_3+H_2O+CO$ [19,20]. According to Soliman [19] the natural transformation of calcium carbonate (CaOx) into calcium oxalates (AmOx) with appropriate properties for stones by providing treated and resistant surfaces motivated conducting several experiments to obtain calcium oxalates through the interaction of calcium carbonate with free oxalates ions. Testing the double replacement reaction using ammonium oxalates showed that the ammonium oxalates were suitable for producing synthetic CaOx that began from the reaction with AmOx. This protective role has been further supported by several specialists [21-25].

1.2. *Workability of ammonium oxalate as a consolidant of archaeological limestone*

AmOx has been widely used in the field of archaeological conservation as one of the most effective inorganic treatments due to its favorable properties. It promotes the formation of a passivating layer of calcium oxalate monohydrate (whewellite) that covers the stone surface, as reported previously by Matteini [24] and Dreyfuss [25]. The workability of *AmOx* is attributed to its slightly acidic pH (below 7), when dissolved in deionized water, along with its significantly lower solubility compared to calcium carbonate. Because of this resin, a protective layer of calcium oxalates (CaOx) is formed, and ammonium carbonate (AmCr) is generated as a secondary byproduct that decomposes spontaneously. Thus, the reaction is typical and highly dependent several factors, including temperature, concentration, the surface of the reactant, and the flow dynamics of the liquid carrying the reagent [26-29].

1.3. *Case study (Temple architectural elements, deteriorated features and weathering products)*

The Athribis area (Sheikh Hamad) is located on the western bank of the Nile, close to and extending into the rocky slopes of the Western Desert, at the edge of the cultivated land. It lies at latitude 26º-28º north and longitude 31º-39º east [30]. The temple of Ptolemy XII (Aulits), fig. (1) is the most significant archaeological site in Athribis, Sohag, upper Egypt. It is situated approximately 18-20 km west of Sohag, on the west bank of the Nile Valley [31]. It is dedicated to the goddess Repit. The long axis of this temple is oriented NNW–SSE. and is dedicated to the goddess Repit. The temple's long axis is oriented NNW–SSE, according to Keb-easy [32]. The excavation of the temple occurred in three phases. The first phase was conducted by Petrie in 1907-1908 [33]. The second phase was carried out by the Egyptian Supreme Council of Antiquities (SCA) between 1983-1998. The third phase began in 2003, led by an Egyptian-German team, which contributed significantly to further important discoveries [34]. The temple was constructed from different types of limestones sourced from the nearby mountains to the west. Its architectural elements – including walls, columns, cornice, in addition to other architectural elements- were built from regional yellowish, porous, homogeneous or cavernous soft limestone. On the other hand, the temple roof was constructed from light to grey layered limestone with hard inclusions in a splintery matrix [35]. The temple has suffered collapse, because of historical earthquakes. According to Leisen, et al. [36], at least 3 types of limestone were used in its construction, each differing in technical and artistic characteristics. These stones were quarried from multiple mountains bordering the western part of the temple [21]. From a specialized point of view, the weathering behaviour of these stones depends on their individual characteristics. Many developed soft, sandy surfaces with scales and exfoliation following the bedding planes as attested by Fitzner et al. [37]. Several deterioration forms affected the limestones including cracks, layer separation, and granular disintegration. They resulted from various factors, such as fluctuations of the environmental conditions such as air temperature (AT), relative humidity (RH) and windblown particles (WB). In addition to the negative impact of materials and procedures of the previous restoration interventions. These included the use of black Portland cement mortar applied directly onto the limestone in some areas. Manifestations of limestone deterioration in the temple are shown in fig. (2-a & b). Additionally, EDX and XRD analyses of stone samples collected from the temple confirm that the main building material used in the temple is limestone, fig. (3-a & b).

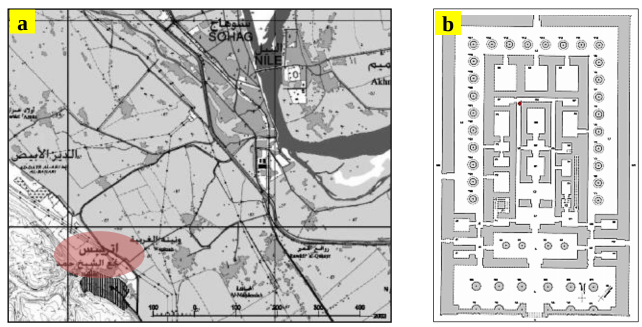


Figure (1) **a.** map of temple area, **b.** plan of Aulits temple

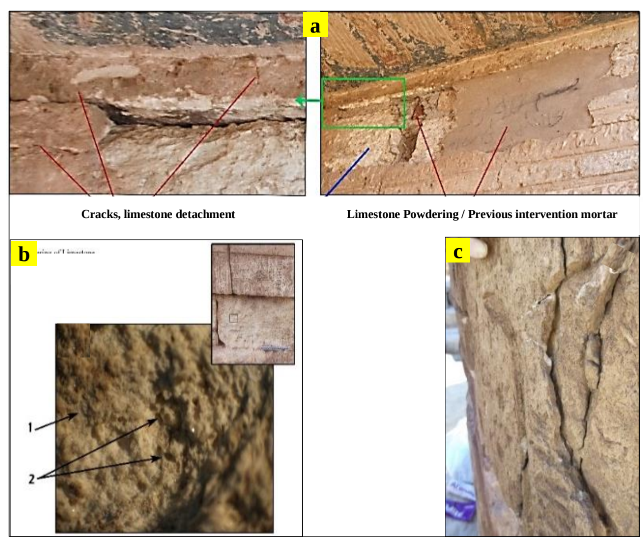


Figure (2) **a.** the damaging effect of the previous restoration's cement mortar on the limestone of the temple, **b.** stereotactic microscopy (SM) of a stone sample from the bottom of the western wall of corridor L3 shows the disintegration of the stone, **c.** the stratigraphic separation in the stone stand in column Y20.

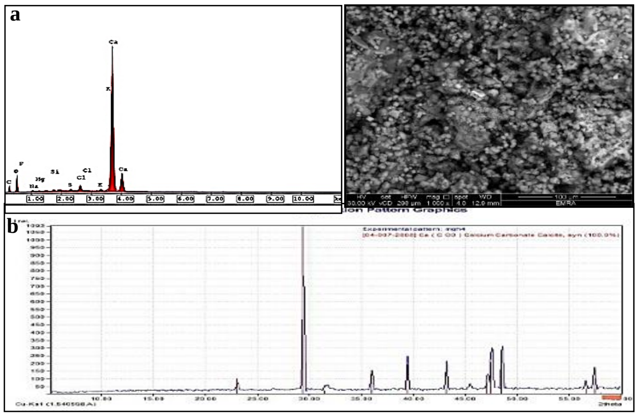


Figure (3) **a.** EDX pattern and SEM photomicrograph of a stone sample shows the main component, i.e., Ca and impurities of other elements, the damaging effect of the previous restoration's cement mortar on the limestone of the temple, **b.** XRD pattern shows mineralogical composition of deteriorated stone sample in calcium carbonate (calcite).

2. Experiments

2.1. *Artificial aging process*

Experimental methods based on laboratory simulations of limestone deterioration serve as a base for identifying stone deterioration manifestations. These simulation experiments take into consideration the relationship between the strength index and artificial conditions, helping to confirm the role of the rotational process from which the results were obtained [38]. This step aims to elucidate the chemical reactions involved (i.e., the degradation mechanism) and their physical consequences [39]. In this study, artificial ageing processes -representing three dominated deterioration factors in the study area- were adapted for studying the deterioration behaviour of limestone buildings before consolidation. Furthermore, the same ageing cycles were then repeated following consolidation with *AmOx* to assess their effects and compare the results before and after treatments and ageing cycles.

2.1.1. Wetting and drying

According to BS EN 14066 [40] both treated and untreated experimental samples were subjected to successive cycles of thermal and wet ageing cycles based on the following protocol: **a)** The samples were tempered at 105 °C. for 24 hours, then their weights were recorded. **b)** They were then immersed in water for two hours, placed in a desiccator for two hours, and left at room temperature for 14 hours, after which their weights were re-recorded. **c)** The samples were immersed in water for a period of approximately 2 hours-sufficient to achieve full saturation. This process was repeated for 30 consecutive cycles. It has been reported that a single day of exposure to a temperature of 60° C is equivalent to 14 days of natural weathering, while exposure to 70° C corresponds to 27 days. Moreover, exposure to a temperature of 100° C for a day corresponds to 157 days [4,41].

2.1.2. Salt weathering

Salt ageing cycles were performed in accordance to the European Standard [42] following these steps: **a)** Samples were dried in a drying oven for 24 hours at a temperature of 60°C, and then their weights were recorded. **b)** They were immersed in a 14% NaCl solution for two hours and covered to prevent water evaporation. **c)** After removal, the samples were allowed to dry at room temperature for 10 minutes. **d)** They were placed in a drying oven at 105°C for 18 hours. This process was repeated for 15 cycles with an interval of 2:4 hours

2.1.3. Acid resistance

This test was conducted as follows: **a)** The samples were dried at 105°C for 18 hours, then kept at room temperature for 6 hours, and their weights were recorded. This step was repeated for 15 cycles with a 2:4 hours interval. **b)** The samples were then sprayed with 20% diluted HCl and placed in a drying oven for 24 hours at a temperature of 60°C. This procedure was repeated for 5 cycles. The aged process was applied to both untreated (reference) and treated samples to study the efficiency of the consolidant in protecting limestone against the acid effect.

2.2. *Treatments*

2.2.1. Materials

As previously mentioned, ammonium oxalate $(NH_4)_2C_2O_4$) is a colourless, odourless, non-volatile, toxic, and combustible solid under standard conditions. It was selected as a consolidant in our case study due to its favorable properties, tab. (1)

Table (1) properties of Ammonium salt of oxalic acid (*After: www.chemistrysources.com*, 2024) [43].

No.	Topics	Factor
1	Apprevated name	AmOx
2	Structure	Monohydrate
3	Solubility in water	5.20 g/(100 ml) (25°C)
4	Appearance	white solid crystals
5	pH	6,5 in 4% aqueous solution
6	Molecular weight/Molar mass	124.1 g/mol^{-1}
7	Density	1.5 g/cm^3 at 18.5°C
8	Melting point	70°C
9	Specific gravity	1.5 at 65.3°F (USCG, 1999) - *Denser than water; will sink*

2.2.2. Sample preparation

Historically, the limestone used in the construction of the Temple of Ptolemy XII (Aulits) was sourced from the same plateau located above the site. Thus, the samples used in this study consisted of limestone blocks obtained from the same plateau. These were divided into four groups, prepared as follows: **a)** the samples were cut into regular cubes measuring 3×3×3 cm to test physical properties such as density, porosity and water absorption. **b)** regular cube samples measuring 6×6×6 cm were used for compressive strength tests. **c)** Tiles measuring 15×10×2 cm were used in the abrasion resistance tests. **d)** Reference samples were used for conducting various analysis and examinations, especially SEM, colour change test, and hydrophobicity.

2.2.3. Consolidation process

A 5% ammonium oxalate (*AmOx*) solution was prepared by adding 50 g of *AmOx* in a liter of distilled water in a glass container. The solution was stirred thoroughly with a glass rod, and allowed to sit for three days, with daily shaking for 5 minutes to ensure complete mixing, taking into account that it should be shaken well before use. The prepared *AmOx* solution was applied to the limestone samples using three different techniques: brushing, spraying, and immersion. Applications were performed in successive cycles until the samples reached saturation. Each application was carried out before the previous layer had fully dried to ensure complete saturation of the stone. Following treatment, the samples were left at room temperature for one month to allow for drying, polymerization and stabilization, at which point the treated cubes reached a constant weight.

2.3. *Investigation and analytical methods*

Weight changes in the samples were measured to estimate the consolidant material remaining, according to Soliman [19]. The visual appearance of the samples —including deteriorated, treated, and aged surfaces—was examined using the *VehoVMS-004 Deluxe USB* microscope, which enabled high resolution analysis of fine surface details. Key physio-mechanical properties of both treated and untreated samples were assessed before and after ageing processes at the Housing and Building National Research Center in Cairo as follows: density [44], porosity [45], water absorption [46] and compressive strength [47]. Furthermore, abrasion resistance was determined using the BS EN14157 Standard Wide wheel method according to the European specifications for stone testing [48]. According to El-Gohary [4] the hydrophobicity of the treated samples was evaluated by measuring the static water contact angle using a *Drop master DM-701* fully automated contact angle meter. Finally, *Quanta 250 SEM* was used to investigate the morphological features of the samples. In addition, the attached *EDX unit* was used for elemental analysis of the same samples.

3. Results

3.1. *Consolidant remining materials after consolidation and weight lost after aging*

Our study results proved that (AmOx) achieved the sufficient consolidation target through weight increase (uptake) in all consolidated samples, with an average 4.04 **%**. Furthermore, the weight average of aged samples was decreased about 0.12 % as listed in tab (2).

Table (2) average of (*5 samples*) defining consolidant remining material within the samples (after treatment and weight lost after aging

Sample	Weight before consolidation	Weight after consolidation	Weight After aging	Remaining material			
				g	*%*	*g*	*%*
Ava 5 S	55.83	58.09	58.02	2.26	4.05	2.19	3.92

3.2. *Surfaces' appearances (USB digital microscope)*

Examination of the sample defined point by the USB digital microscope showed that the general appearance of the experimental limestone samples was affected by the artificial and laboratory weathering cycles. Consequently, local yellowing and salt crystals appeared on the surface with erosion in some weak surface areas. The examination also showed the success of ammonium oxalates in maintaining the general appearance of the treated experimental samples, fig. (4).

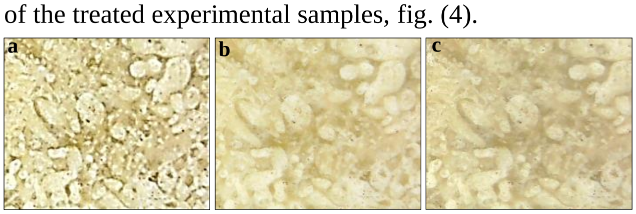


Figure (4) surfaces' appearance; **a.** un-treated, and the approx. same point of the sample, **b.** after treatment, **c.** after aging.

3.3. *Physio-mechanical properties of investigated samples*

The physical properties — density, porosity and water absorption — of the experimental limestone samples were determined and measured. The results of these measurements showed minor changes following the ageing cycles. However, they showed improvement in the average physical properties of the samples after being treated with ammonium oxalates. Furthermore, the average compressive strength of experimental limestone samples was determined and measured by measuring it before and after laboratory ageing cycles and after treating the samples with ammonium oxalates. The results showed low compressive strength after different ageing cycles. They also showed improved compressive strength after treatment and consolidation with ammonium oxalates, tab. (3).

Table (3) Average of (*5 samples*) different properties of un treated and treated aged samples

Properties	Samples state		
	Before consolidation	*Aafter consolidation*	*After aging*
Density (γd) g/cm^3	2.06	2.22	2.15
Porosity (ρ)%	24.55	8.63	8.97
Water Absorption (ϵ) %	11.38	3.75	3.91
Compressive Strength (σ_c) g/cm^3	152.77	207.81	206.73

3.4. *Abrasion resistance*

Abrasion resistance is one of the important indicators of the hardness and bonding strength between the stone particles. It is a well-known method for evaluating the relative efficiency and effectiveness of the treatment [49]. The average abrasion resistance of the experimental limestone samples was calculated by measuring before and after laboratory ageing cycles and after treatment with ammonium oxalates. The results showed a decrease in the abrasion resistance rates after exposure to various artificial ageing cycles and improved abrasion resistance rates after treatment and consolidation with ammonium oxalates, tab. (4).

Table (4) Average of (5 samples) of abrasion % resistance of treated samples

Sample	Samples state		
	Before consolidation	*After consolidation*	*After aging*
Ava 5 S	25	18	17.3

3.5. *Hydrophobicity*

Surface hydrophobicity was evaluated by measuring the contact angle of water with the surface of the material. The low angle of contact means that the material is hydrophilic. The water contact angle means that the material is hydrophobic [50,51]. Hydrophobicity of the experimental limestone samples was assessed via contact angle measurements. Untreated and aged samples displayed low contact angles, indicating a hydrophilic nature. Post-treatment samples showed a notable increase in contact angle values, signifying improved hydrophobic behavior due to ammonium oxalate application, tab. (5) & fig. (5).

Table (5) Average of (5 samples) of hydrophobicity % of the treated samples

Sample	Samples state		
	Before consolidation	*After consolidation*	*After aging*
Ava 5 S	31.8°	57.9°	54°

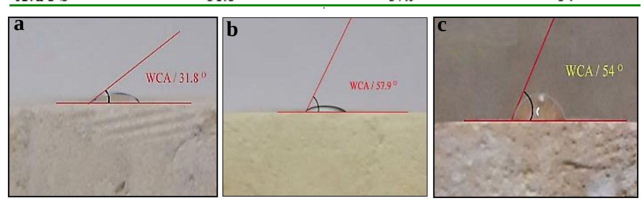


Figure (5) the water contact angle of the sample surface; **a.** un-treated **b.** treated **c.** aged

3.6. SEM examination

SEM examination revealed clear structural differences before and after laboratory ageing cycles and after the treatment with ammonium oxalates. The treated samples showed effective penetration and binding of calcite grains by ammonium oxalates, while preserving the natural pore structure, fig. (6).

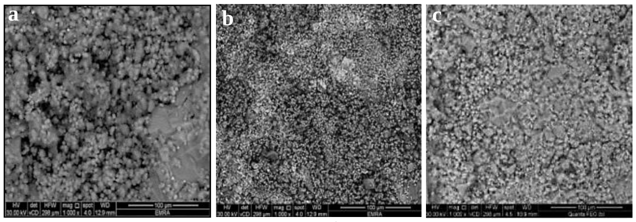


Figure (6) SEM photomicrographs of limestone samples; **a.** un-treated (1000-x), **b.** treated (1000-x), **c.** aged

4. Discussion

Stone decay has been widely discussed due to the diverse and complex causes associated with it. Sleater [52] emphasized where recognizing the causes of stone decay is the first step in its preservation and elimination of these causes. The poor quality of limestones and inappropriate past restoration procedures and materials had a major role in the damage of the Aulits temple, as mentioned above. The effectiveness of various commercial materials was determined by laboratory test programs. According to several researchers [53-56], conservation procedures that include appropriate interventions and the materials used must be laboratory tested and evaluated. Following the comparative testing of five consolidants, ammonium oxalates (*AmOx*) were selected as the most suitable material for application at the temple site [19]. The current study was conducted to re-evaluate Soliman's results under a broader range of conditions to confirm its effectiveness in situ. According to the above-mentioned results, AmOx demonstrated promising consolidation performance for the temple limestone, for the reasons discussed below.

4.1. *Weight changes*

The weight increase (WI) uptake at the end of the consolidation process is defined by the difference in weight of the samples. Table (1) proves that the amount of solid phase precipitated in the stone pores after drying of the specimens was increased by 4.04 %. This obtained result was in good agreement with the expectation, considering the high open porosity of the limestone estimated at 24.55 % before ageing and 29.85 % after. This is consistent with findings by Pesce, et al. [57]; Ban et al. [58] and De Gennes, et al. [59] who noted that the amount of the participated consolidant in the stone pore network depends essentially on four main factors: liquid height, surface tension, features of the stone fabric and inversely to the pore radius [60]. On the other hand, weight loss of ~0.12 % was observed after the ageing process, attributed to the decohesion of the samples due to the effects of thermal ageing [5,61,62], and dissolution processes attributed to acid etching affecting both grain boundary widening and etch pits, as attested by Viles & Moses [63] and Van den Eynde, et al. [64] in their studies. This ratio was re-evaluated after 1 month recording (3.91), 3 months recording (3.59 %) and 6 months recording (3.64 %), which, confirms that the product was completely cured and dried within the pore of stone samples. This process can be attributed to the positive role of AmOx in the consolidation process, which agreed with Gemelli, et al. [65] in his case study.

4.2. *Surfaces' appearance*

Maintaining the original visual appearance of treated surfaces is a fundamental principle in the field of monument conservation, as endorsed by international conservation charters and conventions. Any repair work should not detract from the appearance of the existing stonework. In this context, treated areas must be carefully integrated and remain harmony with adjacent original surfaces as attested by Delgado Rodrigues [66]. Based on fig. (2) it can be asserted, on the one hand, that the application of *AmOx was successful* in treating sample surface. The treatment effectively filled surface voids and resulted in a smoother overall appearance of the surface after complete drying, without the formation of salt efflorescence as argued by Tavares, et al. [67] in their case study, fig. (2-b). This outcome may be attributed to the effects of wetting and drying mechanisms - particularly when the treated surface of the test area maintained a consistent visual appearance without efflorescence as noted in our present case. On the other hand, a noticeable darkening of the surface was observed following the artificial ageing cycles, fig. (2-c), and can be accurately attributed to artificial ageing, and is expected to diminish with time as argued previously by Wheeler & Newman [68,69]; Nishiura [70].

4.3. *Physio-mechanical properties*

The average rate of change in key physical and mechanical properties of the stone after laboratory ageing cycles were as follows: density decreased by 4.18%, the average rate of change in porosity was 21.58%, the average rate of change in water absorption was 36.73%, the average rate of change in compressive strength was 27.85%, and the average rate of change in abrasion resistance was 12%, the average change in the rate of water contact angle with the surface was 9.14%. Density and porosity are interrelated parameters, because bulk density appears to be inversely related to the total cumulative volume and total open porosity of the material [71]. *Density* value is a key physical parameter related to the stone durability and compaction of building materials. As shown in the tab. (3) the average sample density of the samples increased by 7.76 % following the consolidation process compared to the untreated samples. Within this framework, the density increase is attributed essentially to the effectiveness of AmOx as a protective treatment that penetrates stone pores and covers the grains [72]. Upon solvent evaporation, AmOx contributes to the formation of a solid matrix that partially fills and uniformly coats the pores, thereby enhancing the compactness of the material [73]. Then, grants homogenous coverage of the pores that leads to partial closing of pores, this process that influenced by the open porosity and pore size distribution of the selected lithotype [4,74]. Vice versa, a slight decrease in average density by 0.64 % was observed after the ageing cycles. This reduction is likely due to the decaying and loss of minor parts of the samples during the ageing cycles and minor mineral etching with a parallel decrease of water within porosity. The results of this test after treatment as well as after ageing cycles are consistent with the results related to the quantity of consolidant retained in the sample, tab. (2) supporting earlier observations mentioned by El-Gohary [4,75]. Furthermore, the decreasing density may be due to some chemical relevance problems between the stone substrate and consolidant material during ageing cycles [4,76]. *Porosity* values and pore size distribution are key parameters used in this study to examine the efficiency of AmOx treatment over various time intervals. Based on tab. (2) and fig. (6) a significant reduction in average porosity was observed after consolidation, compared to the same samples both before and after ageing. Specifically, porosity decreased by about 64.80 % in consolidated samples, and 63.46 % after the ageing process. This result aligns closely with the observed reduction in consumption and the corresponding increase in the weight of (*AmOx*) within the treated samples, both of contributed to the decrease in total porosity. Similar values were reported by Delgado Rodrigues & Ferreira Pinto [77], who emphasized that limestones with porosity above 15 % can be classified as very porous and, consequently ranked among the most vulnerable lithotypes in terms of deterioration. These observations are also consistent with the results of De Clercq, et al. [78]; El-Gohary [75]; El-Gohary & Abo el Magd [79]. *Water absorption* is a key physical property that controls the penetration in the pores of the stone under the influence of some pressure and capillarity. This property can be modified by using protective treatments that partially fill the pores without completely blocking them, coat the pore walls and modify the surface tension of the pore walls by increasing the surface contact angle [72]. The test was aimed to check the hydrophobic effect induced by treatments, as well as the variation in porosity. Our tested samples, tab. (2) showed a significant enhancement in water absorption, with a reduction of 67.04 % before ageing and 65.64 % after ageing cycles. These results confirm the good workability of AmOx in reducing open pores of the stone and coating of stone grains as argued before by UNI EN [80], where, the water uptake tends to decrease as the amount of consolidant increases due to three main factors; reduced porosity, increased hydrophobicity induced by the consolidant [81]. In addition, the water can penetrate deep into the stone intergranular spaces. These beneficial properties are mainly attributed to the presence of AmOx which functions as both a strengthening and protective agent against environmental loading [82]. *Compressive strength* is one of the most important parameters for evaluating the efficiency of applied consolidants, as it directly reflects the material's ability to withstand mechanical stress before and after treatment and artificial ageing [83]. Through our study, the results clearly indicate that the structural integrity (strengthening) of the sample was improved after the consolidation process. The compressive strength increased by approximately 36.02 % in samples before ageing and 35.32 % after artificial ageing. The increased compressive strength value after consolidation and ageing processes compared to un-treated ones, tab. (3) can be attributed to the penetration of AmOx within the samples. The consolidant effectively reinforced the internal structure of the limestone samples, as argued by Slizkova, et al. [84], who also reported the beneficial impact of AmOx on limestone durability. However, a slight reduction in compressive strength was noted after ageing when compared to the consolidated ones, which is likely due to the direct effects of thermal and environmental stresses accompanied with heating, cooling, wetting and drying cycles. Despite this reduction, strength still remained higher than in untreated samples, as it was increased by ~ 0.52 % as reported previously by Dong, et al. [85] and Ge, et al. [86]. Likewise, many researchers showed that the compressive strength of rocks was reduced by saturation with water, and is influenced by grain size redistribution and pore structure alterations caused by consolidant interferences [87, 88].

4.4. *Abrasion resistance*

Wind-blown sand particles can act as natural abrasives leading to gradual surface degradation of the temple stones over time. Furthermore, airborne sand particles with a Mohs hardness of 7 can seriously damage the limestone, which has a lower hardness index of approximately 3, leading to surface erosion and granular disintegration, tab. (4). These observations are consistent with previous findings of several authors [89-93]. In this study, the application of AmOx with 7% (wt.) resulted in a notable improvement in abrasion resistance following consolidation and 7.7% (wt.) after artificial ageing when compared to non-consolidated samples. These results are in full agreement with other studies presented by Ferri et al. [94]; Stucchi et al. [95]. These results highlight the strong correlation between rock abrasion resistance and aggregate hardness, as noted by (Özvan & Direk [96] and, the total volume of grooves. In addition, the decrease in porosity after treatment contributes to enhanced abrasion resistance, due to the positive workability of consolidant in promoting grain compaction and partial pore closure. On the other hand, notable negative changes in abrasion values may also result from rock type and pore size distribution crystallization cycles as argued by Eren and Bahali [97].

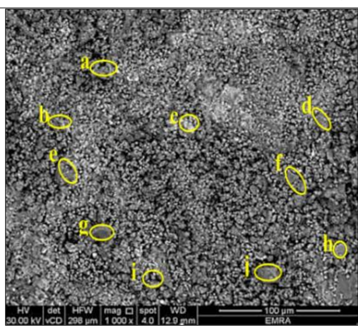
4.5. *Hydrophobicity*

Hydrophobicity and water absorption are linked parameters. Based on fig. (5) the water contact angle (WCA) on the surface was measured before and after exposure to artificial ageing cycles. The results indicate that the average rate of change in this property is favorable. Specifically, the WCA increased to 54° after treatment, compared to 35° before ageing and 31.8° after ageing. These changes are essentially attributed to an increase in surface hydrophobicity. Hydrophobicity is a parameter that controls the interfacial tension between the stone surface and water from one side, and the geometrical and stone porosity from the other side as argued by Charola [98] in her case study. Furthermore, it could be affirmed that there are positive results in WCA after treatment, where, it was notably increased by about 154.3 % compared to untreated surface, and 169.8 % compared to WCA after ageing cycles. This increase, which may be considered a degree of super hydrophobicity, is mainly attributed to the relationship between polarity and hydrophobicity characterized by the stone and the negative surface charges attracted by the positive end of water molecules. In addition, the effects of a polar head and a non-polar tail structure of consolidant material as described by Karapanagiotis & Hosseini [99], plays a significant role in enhancing hydrophobicity.

4.6. SEM examination

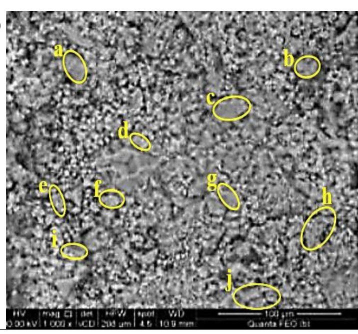
Through the evaluation of the SEM micrographs, fig. (6-a & 6-b), notable differences between non-consolidated and consolidated surfaces can be observed. The consolidated surface exhibited the deposition of relatively large crystals (~211 µm) as attested previously by Pintus, et al. [100] in their case study, fig. (7-a). Furthermore, the comparison between the treated and aged samples, fig. (6-b & 6-c) revealed evidence of surface swelling, attributed to the effects of the heat aged cycle. In addition, an increase in pore diameter likely due to the effects of NaCl crystal growth was noted. This increase in pore size can be a consequence of the interaction between the consolidant treatment and pre-existing salts already present in the stone, as well as the formation of new byproducts, as suggested by Dreyfuss & Cassar [101]. Despite this feature, it could be noted that the aged surface still demonstrates effective diffusion and binding of small particles, without complete pore closure, fig. (7-b)

a



Point	Area	Max	Min	Mean
A	39.657	128.400	67.000	**98.482**
B	79.309	158.710	68.00	**125.084**
C	64.893	200.841	135.045	**178.242**
D	78.309	148.710	67.00	**124.022**
E	86.525	130.242	23.563	**76.717**
F	46.867	211.167	22.000	**136.949**
G	61.288	173.000	70750	**129.040**
H	57.683	117.222	76.000	**103.239**
I	46.867	157.750	104.000	**134.231**
J	46.867	151.000	91.000	**123.077**

b



Point	Area	Max	Min	Mean
A	121.358	181.496	144.911	**159.640**
B	91.019	191.231	177.401	**183.731**
C	115.290	169.917	148.670	**156.545**
D	72.815	171.072	151.202	**161.482**
E	78.883	185.718	142.258	**165.073**
F	109.222	171.694	143.053	**160.176**
G	109.222	173.098	114.642	**158.601**
H	127.426	192.854	69.911	**146.450**
I	109.222	182.351	118.295	**152.725**
J	133.494	194.226	97.909	**147.667**

Figure (7) the deposition of crystals (a) after consolidation, (b) after aging

5. Conclusion

The results confirmed the efficiency of ammonium oxalates in the treatment and consolidation of limestone in the temple of Ptolemy XII (Aulits). The treatment proved to be efficient, easy to apply, and demonstrated no adverse effects on the limestone, either in the short or long term. It significantly improved the overall appearance and physical properties without introducing any negative side effects. Among the various consolidation methods tested, ammonium oxalate showed the best results for consolidating limestone and treating granular disintegration. A follow-up investigation conducted more than three years after the initial application in selected areas of the temple of Ptolemy XII (Aulits) demonstrated the long-term stability and durability of the treatment. Ammonium oxalate (AmOx) showed excellent consolidation performance and did not respond to the surrounding environmental conditions. Importantly, the treated surfaces retained their original color, with no noticeable changes over time. These findings support the use of ammonium oxalates as a safe, effective, and sustainable solution for the conservation and consolidation of historic limestone structures.

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