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DETERIORATION AND CONSOLIDATION OF SOME POTTERY VESSELS IN TEL AJRUD, SUEZ, EGYPT

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Abstract

Many of excavated archaeological pottery from Tel Ajrud in Suez had suffered from different phenomena of deterioration.This kind of pottery needed a consolidation process for improving physical, mechanical properties and durability of various deterioration factors. Some archaeological pottery samples were treated to evaluate some traditional and nano-silica consolidants. Pottery samples were diagnosed using Polarized Microscope, Scanning Electron Microscope with Energy Dispersive of X-Ray and X-Ray Diffraction Analysis. Some consolidation materials such as Paraloid B67, Wacker BS28, Nano Silica, a mixture of Nano-Silica and Wacker BS28 at a rate of 1: 1, Wacker 290Lwere laboratory evaluated. Different experiments, tests and examinations had proved poor physical structure of pottery. They also proved success and efficiency of one of selected consolidants (First: a mixture of Nano Silica and Wacker BS28 at a rate of1:1, concentration 1 %, Second: Nano Silica, concentration 1 %).These consolidants improved physical and mechanical properties. It showed resistance to thermal, light and salt accelerated artificial aging. Therefore, the research recommends using one of two consolidants for strengthening archaeological pottery excavated from Tel Ajrud in Suez.

***Keywords**: Pottery; Consolidation; Paraloid B67; Wacker BS28; Wacker 290L; Nano Silica; Artificial Ageing*

Introduction

Tel Ajrud was located in west Suez, archaeological area was 50 acre, dating back to Ottoman age. It was station of ancient pilgrimage routes. It contained foundations of towers of citadel dating back to Sultan Muhammad ibn Qalawun. Sultan Qansouh al-Ghouri ordered establishment of Ajrud castle in Suez in 1509 AD for providing pilgrims with water and supplies. This castle resembled castles of Ayyubid era. Its architectural design was similar to Nakhl castle in north Sinai. It was a rectangular courtyard 30 × 11m, its four corners contained semi-circular towers. The entrance was located in western side of citadel. Mission of Supreme Council of Antiquities carried out excavations at archaeological site from 1992 to 2009. Excavation revealed thousands of pottery objects dating back to Ottoman era [1].

Archaeological pottery vessels excavated from tel Ajrud in Suez was poor physical structure due to burial in soil for long periods. It caused various deterioration manifestations such as peeling, cracking, surface deformation and poor physical structure [2]. Pottery often needs to improve physical and mechanical properties by strengthening internal structure using one of selected consolidants [3]. Silicon consolidants were one of the best consolidants applied

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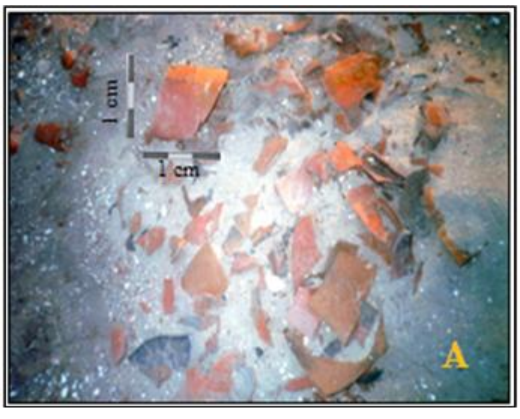
in strengthening of archaeological pottery from soil [4]. Success of consolidants depends on nature of archaeological pottery, firing temperature, chemistry of consolidants and their application methods. It has a great ability to penetrate the pores [5]. It should not cause future damage to pottery after treatment [6]. It must be achieved protection and stability of pottery against surrounding environmental factors [7]. Consolidants should not be used depending on trade name [8]. It is necessary to know chemistry of consolidants especially their properties, their application methods and their operating conditions [9]. It must be experimented before applying to choose the best material [10]. They must be stable and unchanged in new environmental conditions [11].

Recently, Nanotechnology was used in various fields [12]. Nano was derived from an ancient Greek word "dwarf" [13]. Traditional consolidants had been developed by controlling in shape and size of granules ranging from 1: 100 nm [14]. They were classified according to their forms to carbon nanotube or nano-fiber [15]. It was characterized by extraordinary thermodynamic properties [16]. Nanomaterials were used extensively in preservation, consolidation and cleaning of archaeological objects [17]. Nano zinc oxide was used in cleaning of archaeological materials that had suffered from presence of soiling, soot and stains [18]. Nano lime "calcium hydroxide" was used in consolidating of limestone [19]. Or mural painting [20].Nanomaterials were added to silicon consolidants to improve their physical and mechanical properties [21].This study is one of the first studies for evaluating some traditional and nano-silicon materials used in strengthening excavated archaeological pottery from Suez. The research will recommend the best selected consolidants materials after their laboratory evaluation to strengthen pottery of Agrud in Suez.

Materials and Methods

Materials

Four pottery samples were selected from pottery fragments of Tel Ajrud in Suez. Ten samples of manufactured pottery cubes were prepared from Tel Ajrud in Suez as in Figure 1.



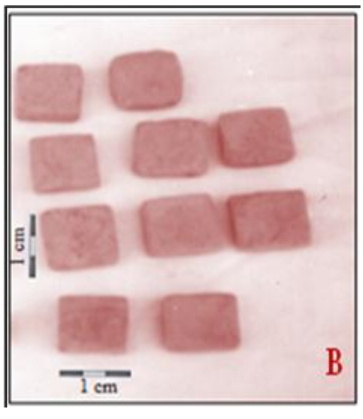


Fig. 1. Study samples: A. archaeological pottery samples; B. Manufactured pottery cubes

Pottery samples were strengthened using the following selected consolidants: Paraloid B67dissolved in trichloroethylene 3%, Wacker BS28, Nano Silica, a mixture of Nano-Silica and Wacker BS28 at a rate of 1: 1, concentration 1% and Wacker 290L. These consolidants were applied by Immersion method [22, 23].

Methods

Methods of Examinations and Analyzes

Pottery specimens were examined to assess their condition, poor physical structure and nature of damage due to burial environment, weather changes. Lenses from 4 - 6× magnification were used [24]. Pottery was examined by Polarized Microscope. It was Olympus BX51 TF japan under magnification 4× up to 40×. Thin-sections were prepared. Examination was conducted at polarized microscope laboratory at Geology Department of Faculty of Science at Cairo University. Samples were also examined and analyzed by Scanning Electron Microscope with Energy dispersive X-ray unit SEM-EDX. It was JEOL JSM-840 and SEM Quanta 200 FEG, XTE 325/D8395. Operating conditions were 20kV and 1×10^{-9}A. The examination and analysis were carried out in Ministry of Antiquities. X-Ray Diffraction analysis XRD was used. It was Philips. Operating programs was X'Pert Graphics', identify by Philips programs. Diffraction pattern was 0:60 2θ. Operating conditions were 40 MA, 45 kV. Radiation source was Cu-K α radiation. The analysis was carried out at X-ray diffraction laboratory in Ministry of Industry in Cairo.

Method of Consolidation of pottery samples

The pottery samples were consolidated by immersion about 8 hours for 3 days. Paraloid B67dissolved in trichloroethylene. Its concentration was 3%. Selected traditional silicon consolidants were used in their nature solution, Nano Silica was 1% concentration.Mixture of Nano-Silica and Wacker BS28 in a rate of 1: 1 at 1% concentration [25].

Tests and examinations of consolidated pottery samples:

a. Determination of physical and mechanical properties [26];
b. Examination of consolidated samples by Scanning Electron Microscope;
c. Accelerated artificial ageing (thermal - light by U.V- salt weathering) [27];
d. Examination of consolidated pottery samples by Scanning Electron Microscope after artificial ageing processes

Results

Methods of Examinations and Analysis

Visual examination showed that pottery specimens of Tel Agrud in Suez suffered from different deterioration phenomena such as breaking, gaps, crystallization of salts, peeling of slip layer and poor physical structure.

Polarized microscope played an important role in identifying petrographic structure, mineral components [28], tempers, surface treatments and firing process [29]. Examination of the pottery sample showed angular quartz grains, sub-round quartz grains, rutile, calcite, biotite, and coarse pottery fabric as shown in figure 2A. Examination of another part of the same sample showed presence of quartz, pyroxene and calcite as shown in figure 2B.

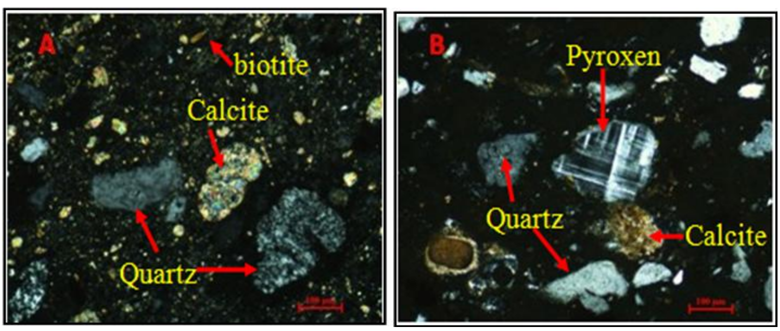


Fig. 2. Petrography micrograph of pottery sample in Tel Agrud in Suez:

A. quartz grains, calcite and biotite; B. quartz grains, pyroxene and calcite (10×-CN)

Scanning Electron Microscope showed morphology of surface of archaeological pottery, shape of granules, pottery fabric and its elemental composition. It showed heterogeneous quartz granules, clay minerals, some gaps and crystallization of salts, as shown in figure 3.

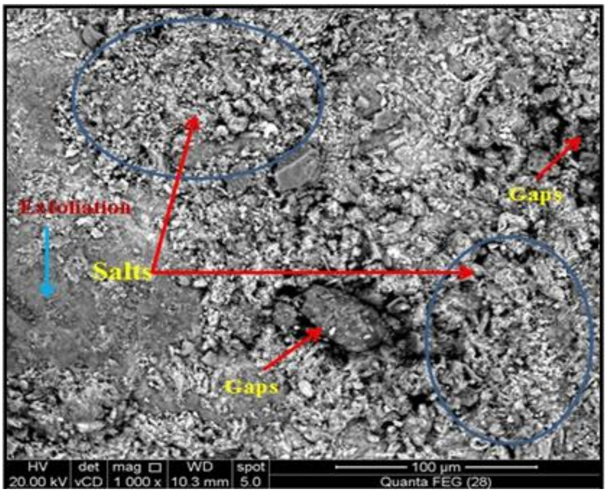
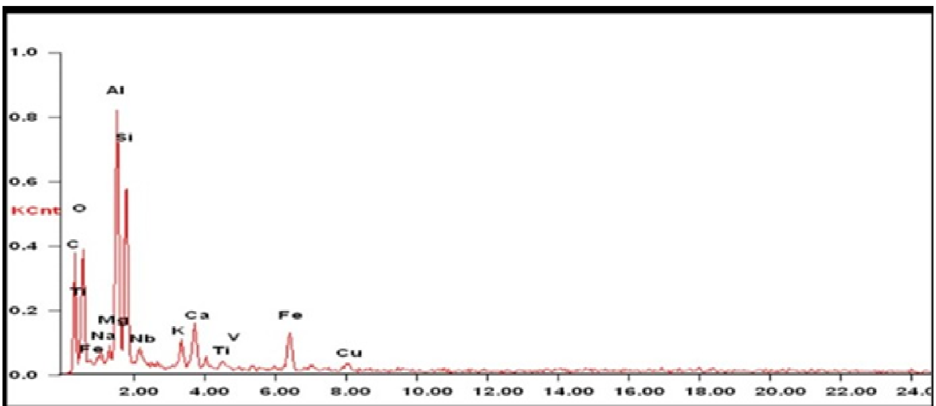


Fig. 3. SEM photomicrograph of pottery sample of Tel Ajrud, Suez

SEM-EDX

Results of analysis of pottery sample of Tel Ajrud in Suez showed presence of carbon, sodium, magnesium, aluminum, silica, nybalium, vanadium, potassium, calcium, titanium iron and copper as shown in figurer 4.



C	O	Na	Mg	Al	Si	Nb	V	K	Ca	Ti	Fe	Cu	total
10.35	8.54	1.14	2.86	31.51	29.59	0. 75	0.24	1.40	10.87	0.26	1.93	0.56	100%

Fig. 4. EDX pattern of pottery sample of Tel Ajrud, Suez

XRD

Pattern of XRD contained quartz (SiO_2), magnetite (Fe_3O_4), albite ($NaAlSi_3O_{10}$) as shown in figure 5.

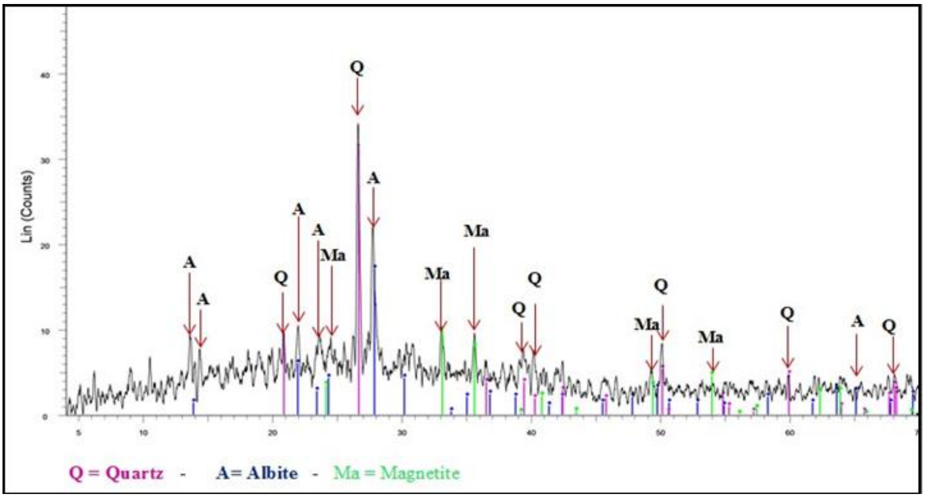


Fig. 5. XRD pattern of pottery sample of Tel Ajrud, Suez

Determination of physical and mechanical properties of consolidated pottery samples of Tel Ajrud in Suez

Degree of Water Absorption

Degree of water absorption of consolidated pottery samples was determined by Building Materials Research Center, Ministry of Industry, Egypt. Determination of water absorption degree of consolidated pottery samples and untreated sample ranged from 4.88% as in mixture of Nano-Silica and Wacker BS28 up to 22.40% as in Paraloid 67. Percentage of reducing water absorption degree was 85.73% for the mixture but percentage of reducing water absorption degree was 34.52% for Paraloid 67. Between these two values was difference in degree of water absorption and its percentage of consolidated pottery samples compared to untreated pottery that recorded 34.21% as shown in figure 6.

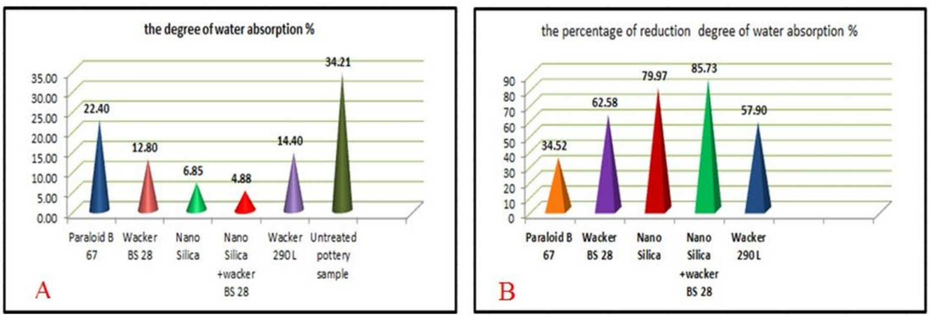


Fig. 6. Physical properties A: water absorption degree,
B: percentage of reducing water absorption degree of consolidated pottery samples

Degree of Apparent porosity

Degree of apparent porosity of consolidated pottery samples was determined by Building Materials Research Center, Ministry of Industry, Egypt. Determination of apparent porosity degree of consolidated pottery samples and untreated sample ranged from 9.22% with percentage of reducing a apparent porosity degree 81.64% as in sample treated by a mixture of Nano-Silica and Wacker BS28 up to 38.35%, with percentage of reducing apparent porosity degree 22.66% as in sample treated by Paraloid 67. Between these two values was difference in apparent porosity degree and its percentage of consolidated pottery samples compared to untreated pottery that recorded apparent porosity 50.24% as shown in figure 7.

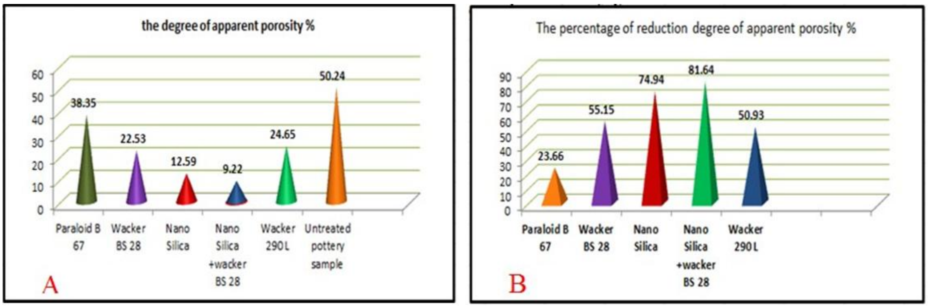


Fig. 7. Illustrates physical properties A: apparent porosity degree,
B: percentage of reducing apparent porosity degree of consolidated pottery samples

Degree of Compressive Strength of Consolidated Pottery Samples

Degree of compressive strength of consolidated pottery samples was determined by Building Materials Research Center, Ministry of Industry, Egypt. The test was carried out by placing pottery sample in center of device. The device was operated. The load occurred vertically until the sample was crushed. Reading of the indicator was recorded in kg/cm^2.

The difference in determination of degree of compressive strength of consolidated pottery samples and untreated sample ranged from 69.27kg/cm^2 with a percentage of increasing compressive strength degree 71.03% as in sample treated by Paraloid 67 up to 185.70kg/cm^2 with a percentage of increasing compressive strength degree 358.51% as in sample treated by a mixture of Nano-Silica and Wacker BS28. Between these two values was difference in compressive strength degree and its percentage of consolidated pottery samples compared to untreated pottery that recorded compressive strength degree 40.5kg/cm^2 as shown in figure 8.

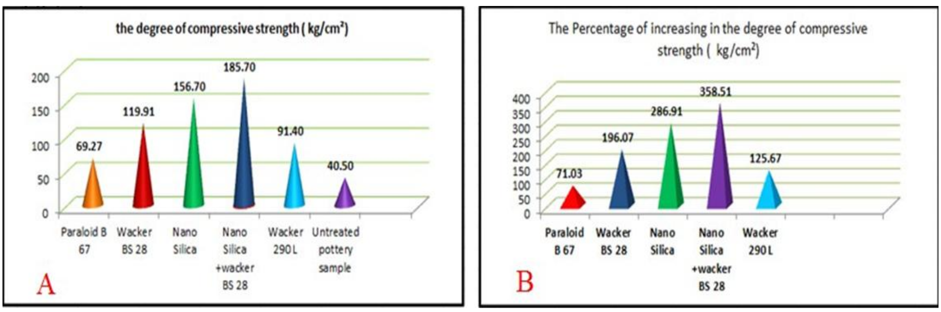


Fig. 8. illustrates mechanical properties A: compressive strength degree,
B: percentage of increasing compressive strength degree of consolidated pottery samples

SEM

Scanning electron microscope revealed that Paraloid B 67 had spread, penetrated into the pores partially but failed to link granules together. It was also observed that it did not achieve homogeneity in linking and packing of granules as shown in figurer 9. Wacker BS28 had linked granules together. It deposited among the pores in a medium degree. It was also observed that it achieved almost complete homogeneity in form of cross-links as shown in figure 10. SEM Examination of consolidated pottery samples by Nano-Silica or a mixture Nano-Silica and Wacker BS28 showed that they linked and packaged the particles of pottery sample homogeneously. They achieved a perfect homogeneity especially a mixture nano-silica and

Wacker BS28 that spread inside the pores, linked granules and completely packaged them in all parts of the sample, as shown in figs. 11 and 12.

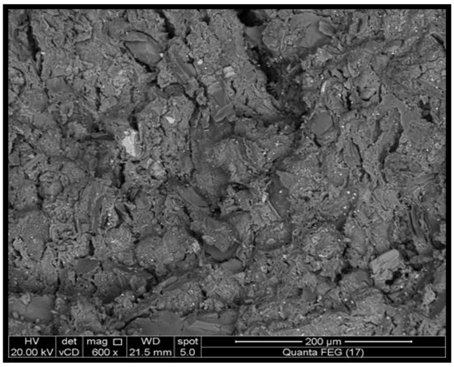


Fig. 9. SEM photomicrograph of treated pottery sample by Paraloid B67 (600×)

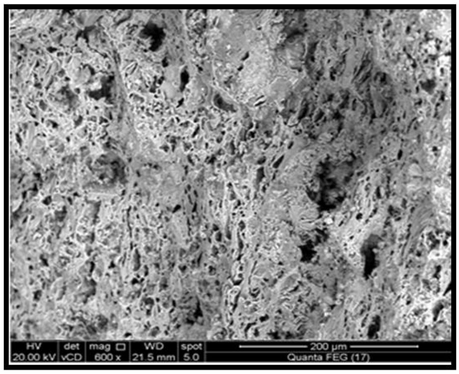


Fig. 10. SEM photomicrograph of treated pottery sample by Wacker BS28 (600×)

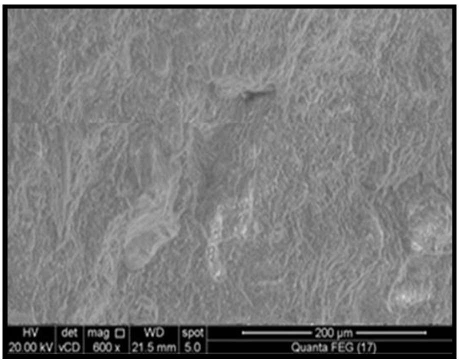


Fig. 11. SEM photomicrograph of treated pottery sample by Nano-Silica (600×)

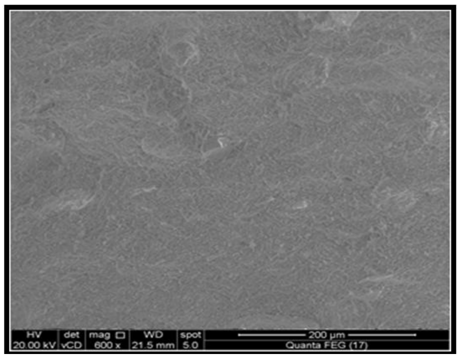


Fig. 12. SEM photomicrograph of treated pottery sample by mixture Nano-Silica and Wacker BS28 (600×)

It was revealed by scanning electron microscope of the consolidated pottery sample by Wacker 290 L that it partially spread into the pores. It did not fill all of the pores in the sample, and it partially linked and packaged the grains as shown in figure 13.

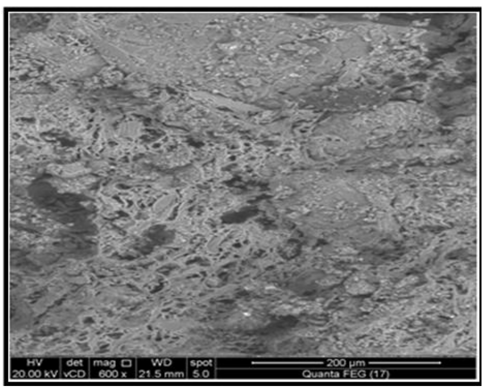


Fig. 13. SEM photomicrograph of treated pottery sample by Wacker 290L (600×)

Effect of Accelerated Artificial Ageing on Consolidated Pottery Samples

Thermal Artificial Ageing

Thermal artificial ageing was conducted for 30 days inside an electric furnace. Its temperature was set at 65°C for 15 days at a rate of 16 hours inside the furnace followed by 8 hours at room temperature and anther cycle for15days inside the furnace at 120°C [30**].** Consolidated pottery sample by a mixture Nano Silica and Wacker BS28 had recorded the highest degree of resistance to thermal artificial ageing but it suffered from weight loss about 0.24%. The weight loss increased up to 3.16% in consolidated sample by Paraloid B 67, compared to untreated pottery sample that recorded weight loss 24.80% as shown in figure 14.

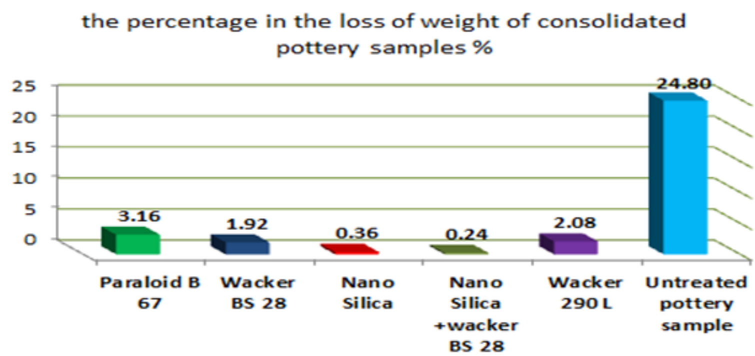


Fig. 14. The percentage of weight loss of consolidated pottery samples due to thermal artificial ageing

Light Artificial Ageing by Ultraviolet Radiation

This experiment was carried out at restoration laboratory at Research and Conservation Center at Ministry of Antiquities in Cairo Using a deuterium bulb coupled with an electrical stabilizer at room temperature. The results of exposure to ultraviolet radiation showed that consolidated pottery samples did not change their surface color, but they suffered weight loss. Percentage of weight loss of consolidated pottery sample by Nano Silica was 0.12% increasing up to 1.92%, as in consolidated pottery sample by Paraloid B 67, compared to untreated sample recorded weight loss 7.20% as shown in figure 15.

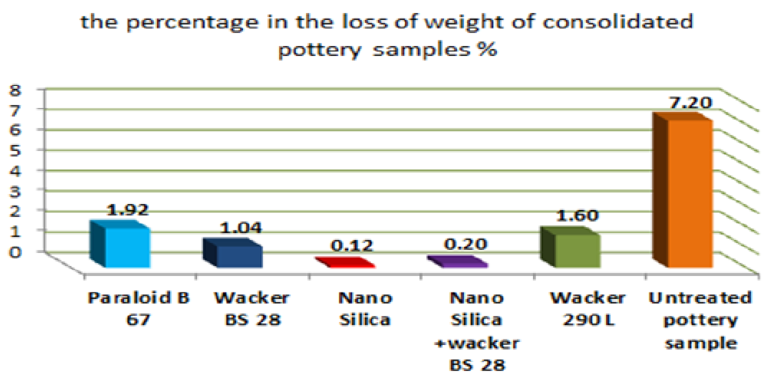


Fig. 15. The percentage of weight loss of the consolidated pottery samples due to light artificial ageing *by* ultraviolet radiations

Artificial Ageing by Salt Weathering

This experiment was conducted at Housing materials Research Center at Ministry of Industry in Cairo. Pottery samples were immersed for 4 hours in sodium chloride solution10%, and then they were subjected to 28 hours to air at room temperature. The samples were placed for 16 hours at 60°C inside an electric furnace [31]. Results of salt weathering showed that the samples were affected by sodium chloride 10%, in different rates. Consolidated pottery sample by Paraloid B67 had undergone very little change. Consolidated pottery samples by Nano Silica or mixture of Nano Silica and Wacker BC28 remained color unchanged. The consolidated pottery samples by Wacker BS28 or Wacker 290L were changed into dark color. Most consolidated pottery samples suffered from weight loss in different rates. Treated pottery sample by mixture of Nano Silica and Wacker BC28 had recorded the highest degree of resistance to salt weathering. However, it suffered from weight loss 1.60% increasing up to 6.52% as in consolidated pottery sample by Paraloid B67, compared to untreated pottery sample that had recorded weight loss 47.44%, as in figure 16.

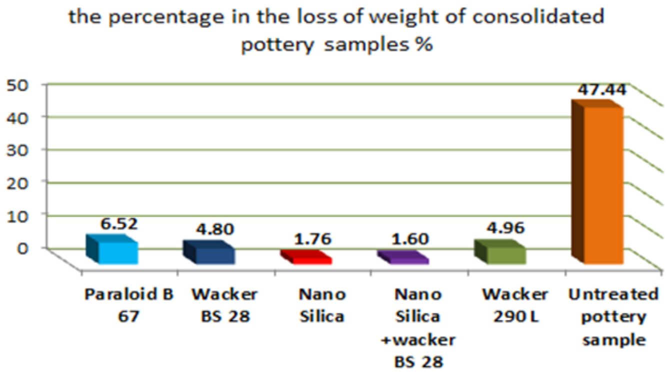


Fig. 16. The percentage of weight loss of consolidated pottery samples due to salt weathering

SEM analysis of consolidated pottery Samples after Artificial Ageing

It was showed by examination of scanning electron microscope" SEM" that consolidated pottery sample by Paraloid B67 was affected by *artificial ageing*, but it still spread partially in the pores. Some granules were detached as shown in figure 17A.

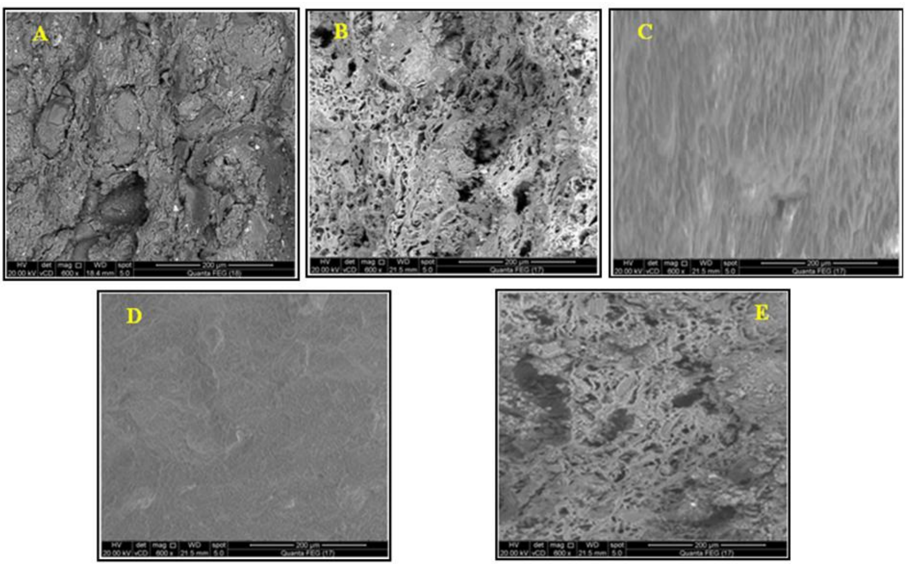


Fig. 17. SEM photomicrograph of consolidated pottery samples *after artificial ageing:*
A. Paraloid B67, B. Wacker BS 28, C./ Nano Silica, D. mixture of Nano Silica and Wacker BC28, E. Wacker 290L

Although consolidated pottery sample by WackerBS28 had exposed to *artificial ageing*, it was still good spreading into the pores, linking and package grains, as shown in figure 17B. SEM showed that consolidated pottery samples by Nano Silica or mixture of Nano Silica and Wacker BC28 retained their strength and cohesion of granules, linking and package grains as in figure 17C and D. Scanning electron microscope showed that consolidated pottery sample by Wacker 290L was affected by *artificial ageing*, but it was still partially spread in the pores. Some granules were detached as shown in figure 17E.

Discussions

Results of various examinations and analyzes of pottery samples excavated from Tel Agrud in Suez indicated that the samples suffered from poor physical structure due to low firing temperature degree. It was evidenced by presence of magnetite" Fe_3O_4", absence of firing minerals.

The analysis also proved presence of some salts such as carbonates and chlorides as halite due to burial in the soil.

The examinations and analysis had shown presence of alumina, potassium, magnesium, pyroxene, and mica "biotite". This indicated that the used clay was montomorillonite as it had a high magnesium ratio.

The tests confirmed poor physical and mechanical structure of pottery specimens being buried in the soil, which necessitated strengthening with one of the selected consolidants.

The results of experiments of physical properties of pottery samples treated with some selected consolidants had shown that most of consolidation materials did not affect surface color of treated pottery samples compared to untreated pottery samples except for WackerBS28 and Wacker290L samples that had exposed to a slight color change.

Laboratory study proved that all selected consolidants had improved physical properties in different degrees. The results of determination of water absorption and porosity degrees of pottery samples before and after treatment showed that a mixture of Nano Silica and Wacker BS28, Nano Silica, Wacker BS28, Wacker 290L and Paraloid B 67 achieved the best results respectively.

Determination of mechanical properties results proved that all selected consolidants also improved mechanical properties of treated samples compared to untreated samples, which linked and strengthened the granules achieving highly resistance to compressive strength. Mixture of Nano Silica and Wacker BS28, Nano Silica, Wacker BS28, Wacker 290L and Paraloid B 67 achieved the best results respectively.

The examination of scanning electron microscope (SEM) of treated samples showed that a mixture of Nano Silica and Wacker BS28, then Nano Silica, after that Wacker BS28, followed by Wacker 290L and finally Paraloid B 67 gave satisfying results respectively. Mixture of Nano Silica and Wacker BS28, then Nano Silica gave excellent results, but Wacker BS28, and Wacker 290 L were less efficient. While Paraloid B67 did not give any satisfied results.

Stability of treated samples by selected consolidants against accelerated artificial ageing factors was sufficient evidence for its success in treatment and maintenance. The results of experiments and tests of thermal artificial ageing showed that pottery samples treated with selected consolidants had achieved stability and resistance to thermal ageing compared to untreated samples. Mixture of Nano Silica and Wacker BS28, then Nano Silica, after that Wacker BS28, followed by Wacker 290L and finally Paraloid B 67 achieved the best results respectively.The treated samples showed good resistance to thermal ageing demonstrating success of selected consolidants in improving mechanical properties of samples especially a mixture of Nano Silica and Wacker BS28.

The results of experiments and tests of light artificial ageing by ultraviolet radiations showed that pottery samples treated with selected consolidants had achieve**d** stability and resistance to light artificial ageing in different rates. Mixture of Nano Silica and Wacker BS28, then Nano Silica, after that Wacker BS28, followed by Wacker 290L and finally Paraloid B 67 achieved the best results respectively.

The results of experiments and tests of artificial ageing by salt weathering showed that treated pottery samples with Nano Silica or a mixture of Nano Silica and Wacker BS28 did not exposed to any color change, but Wacker BS28 or Wacker 290L exposed to a slight color change. A mixture of Nano Silica and Wacker BS28, then Nano Silica, after that Wacker BS28, followed by Wacker 290L and finally Paraloid B 67 achieved the best results for resistance of artificial ageing by salt weathering respectively.

The examination by SEM for consolidated samples after accelerated artificial ageing showed that treated specimen by Paraloid B 67 had affected by artificial aging, but it was still partially in the pores. But some grains were separated. Wacker BS28 was still widespread in the pores. It linked and packaged granules despite exposure to artificial ageing factors. While samples treated with Nano Silica or a mixture of Nano Silica and Wacker BS28 remained their stability, strength and cohesion granules. They linked and packaged grains. The specimen treated with Wacker 290 L had affected by artificial ageing, but it was still partially in the pores, and some granules had separated.

Process of Consolidating excavated pottery lamp from Tel Ajrud in Suez

The research had shown poor physical and mechanical structure for pottery in Suez due to Manufactural defects, burial in soil and climatic changes. Consolidation process was one of the most important maintenance processes. It strengthened, linked and packaged the grains in a homogeneous rate [32]. Pottery lamp was documented by digital camera. It was mechanically and chemically cleaned. Salts were extracted. Assemblage of pottery lamp was done by Paraloid B72 at concentration 50% [33]. Strengthening was done by a mixture of Nano Silica and Wacker BS28 by 1: 1 respectively [34].It was strengthened as in figure 18. Pottery lamp should be museum displayed in National Suez Museum. Relative humidity must be 55:60%. Air temperature is 18 - 20°C, and light intensity is 300 lux [35].

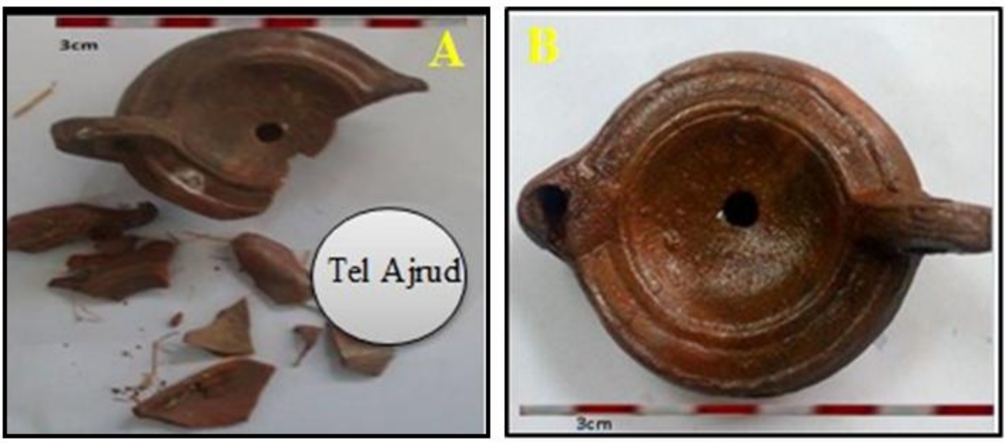


Fig. 18. The Pottery lamp A: before treatment, B: After treatment

Conclusions

The research proved that most of excavated archaeological pottery from Tel Ajrud in Suez suffered from poor physical structure and breaking phenomenon. This pottery needed a consolidation process that strengthens, links granules and improves physical and mechanical properties using one of selected consolidation materials. The research recommends using one of the following consolidation materials: first: mixture of Nano Silica and Wacker BS28, at a rate of 1:1, concentration 1%, second: Nano Silica, concentration 1% in strengthening of archaeological pottery of Tel Ajrud in Suez. These consolidants had improved physical and mechanical properties. It showed high more resistance to accelerated artificial aging. It had achieved deep penetration in pores of the samples. It had also strengthened, linked and packaged the granules homogeneously.

Acknowledgement

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