Evaluation Calcium Hydroxide Nano Particle for Consolidation of Mud Brick

Sameh Rohayem[1], Hamada Sadek[2] and Prof. Mohamed Abd El Hady[3]

1- Ministry of Antiquities, Egypt
2- Conservation Department, Faculty of Archaeology, Fayoum University, Egypt.
3- Prof. of Conservation of Historical Buildings, Faculty of Archaeology, Cairo University, Egypt

Abstract

The earth structure expose to deterioration factors such as wind which cause erosion of the blocks components, several studies have been conducted on the use of traditional consolidats in earthen architecture in order to prolong the mudbrick stability. This study taking the advantages of nanoparticles such penetration and optimizing the internal cohesive of masonry particles to apply in Earth structure in Egypt. This study will evaluate the use of calcium hydroxide nanoparticles on experimental samples. Various analytical techniques used in the study, scanning electron microscopy and microscopy, Atomic Force Microscope, X-ray Diffraction. The results of the analysis showed that of calcium hydroxide improved the characterization of the studied samples.

1. Introduction

Nanotechnology has been known in the ancient civilizations, since people in ancient worlds have used nanomaterials for different purposes but are unaware that they practice nanotechnology. Several natural materials are characterized by Nano-size, such as clay minerals, luster particles etc. One of the most prominent examples used in ancient Egyptian civilization, using of black ink made of carbon nanotubes in writing[1].

A variety of analytical techniques are used to synthesize nanomaterials and control their internal structure by restructuring and arranging the atoms and their components molecules, thus ensuring access to unique and nanomaterials in terms of dimensions[2][3], This micro-cause some unique properties that are not available in normal state of the materials, making scientists describe these (nanoparticles) as the essential materials of the 21st century.[4][5][6]

The most important characteristic of nanomaterials from conventional materials is that nanoparticles have a very large surface area compared to their size. It is known that the smaller the size of the material, the greater the surface area to the size, the surface area of a body, External to this body. This means that the surface area of the material relative to its size is inversely proportional to its size.[7][8]

Calcium Hydroxide $Ca(OH)_2$ has been used for several years in the consolidation of carbonate materials. This type of consolidation is based on the deposition of calcium hydroxide into the pores. After solvent evaporation, Calcium Hydroxide is converted into Calcium Carbonate, by Carbonization by Carbon Dioxide (CO_2), Although the resulting carbonate material has compatibility and homogeneity with the components of carbonate materials, it is accompanied by some disadvantages, such as Low Penetration, and Incomplete Conversion into Calcium Carbonate[9].

---

(1) فتح الله الشيخ و محمود موسى: قصة النانو تكنولوجيا حاضرها و مستقبلها، المكتبة الأكاديمية ، الطبعة الأولى ، 2009 ص 21 - 23.

(2)Filipponi, L., and Sutherland, D., Introduction to Nanoscience and Nanotechnologyies، Interdisciplinary Nanoscience Center (iNano), Aarhus University, Denmark, 2010, p.4.

(3)Bennet, D., Nanotechnology: Ethics and Society, CRV press, Taylor and Francis Group, U.S.A ،2008 ،P. 9-10.

(4)Manoharan, M., Research on the frontiers of Material Science: The impact of nanotechnology on new material development, Technology in Society, Vol. 30, 20 ،08pp. 401-404.

(5)Andrew, D. M., Nanotechnology: AResearch Strategy for Addressing Risk, Woodrow Wilson ،2006 ،p.7.

(6)Michel, F. A. and Paulo, J.F. and Daniel, L. S., Nanomaterials: Nanotechnologies and Design، Elsevier Ltd, 2009, pp.18-89.

(7)Chandravathanam, S., Nanomaterials: A sojourn, N. C.for Catalysis Research, 2006, p.31.

(8)Michael, F. A. and Paulo, J. F. and Daniel, L. S., op. cit, 2009, pp. 182-189.`

(9)Daniele, V. et al., The Nanolimes in Cultural Heritage Conservation: Characterization and Analysis of Carbonatation Process, Journal of Cultural Heritage 9, 2008, p. 294.

Over time, through nanotechnology, researchers have been able to eliminate the disadvantages associated with the application of calcium hydroxide in the consolidation and also to improve their properties by preparing the calcium nanoparticles and dispersing them into Non-Solvent aqueous (eg alcohol), Nanoparticles of calcium hydroxide are prepared by chemical deposition in a saturated aqueous solution with the reaction material (calcium chloride, sodium hydroxide) at high temperature, then dispersion the deposit in alcoholic solution. The addition of Sodium bicarbonate ($NaHCO_3$) to the solution helps complete the carbonization process(1)(2)(3). This work aims to evaluate Calcium Hydroxide in consolidation of mudbrick blocks, the study focusses on preparing experimental samples and use Calcium hydroxide to strengths the sample and use multi analytical techniques and microscope to evaluate the efficiency of Nano calcium hydroxide in mud brick monuments.

2. Materials and methods

2.2 Materials

2.2.1 Mudbrick samples

After studying the components of the bricks in the tomb, which is the subject of the study using various analytical techniques, samples were prepared in cubes (5 cm × 5 cm × 5 cm), and for use in the experimental study as shown in fig.1.

(1)Dei, L. et al., Nanotechnology in Cultural Heritage Conservation: Nanometric Slacked Lime Saves Architectonic and Artistic Surfaces from Decay, Journalof Cultural Heritage 7, 2006, pp. 110 : 115.
(2)Doehne, E. and Price. C., Stone Conservation: An Overview of Current Research, Paul Getty publications, Second Edition, Los Angeles, 2010, pp. 37 : 38.
(3)Giorgi, R. et al., A new Method for Consolidating Wall Paintings based on Dispersions of Lime in Alcohol, Studies in Conservation 45, 2000, pp. 154 : 161.

Fig.1 mudbrick samples (5x5x5) cm prepared accordance to one of the pyramids builders tombs.

2.2.2 Calcium Hydroxide Nanoparticles

The calcium Nano hydroxide produced by IBZ-Salzchemice GmbH & Co.KG, a German product under the name CaloSiL, a ready-to-use product, was used for micro-particles of calcium hydroxide $Ca(OH)_2$ suspended in an alcohol such as ethyl alcohol Alcohol, isopropanol and other alcohol(1). The calcium hydroxide was applied the mudbrick cubes by brushing until the complete penetration of the consolidant into the cube pores, then samples left for 48 hours and repeat the process of consolidation for three consecutive cycles. In the application process, it was observed that each session should be applied before the complete drying process of the consolidation material to obtain the full degree of saturation of the calcium hydroxide, and then leave the treated samples in the room temperature covered with polyethylene sheets for one month to allow the process to complete polymerization.

(1) https://ibz-freiberg.de/en/products .

2.3 Methods

2.3.1 Optical Microscopy

Optical microscopy is a quick way to identify grains. There are a variety of methods for the examination of materials by optical microscopy. The samples can be examined by transmitted light, reflected light, and through the stereomicroscope, on a three-dimensional image(1).In this study, samples of mudbricks were examined by optical microscopy: Stereo Microscope Type Discovery V 20 Zeiss.

2.3.2 SEM/EDX

The scanning electron microscopy has the ability to study the sample surface without samples preparation. The scanning electron microscopy can give a magnification force of up to 100,000 times. It can also obtain a detailed 3D image (2), and determine the penetration of consolidant into the samples. The model used in this study is Quanta-200.

2.3.3 X-Ray Diffraction (XRD)

X-Ray Diffraction is one of the most important and most common methods of analysis in the field of studying the crystallized archaeological material. This method can accurately identify the components of the archaeological material(3)(4) .

An X-ray diffraction device was used is Panalytical X, pert pro PW 3040/60.

2.3.4 Atomic Force Microscope (AFM)

Atomic Force Microscope (AFM) is a device used in the field of nanotechnology to study the topography of surfaces with Nano-particles(5). AFM provides several advantages over conventional microscopy techniques, AFM explores the sample

---

(1)Stuart, B.H; Analytical techniques in materials conservation, John Wiley & SonsLtd, England, 2007, PP.80-82.
(2)Stuart, B.H.; Op. Cit., PP.91-92.
(3)Stuart, B.H.; Op. Cit., PP.230-234.
(4)Toney, M. F., X Ray Diffraction, in Encyclopedia of materials characterization, Butterworth-Heinemann, U.S.A, 1992, pp. 198 - 212.
(5)https://ar.wikipedia.org

and makes measurements in three dimensions X, Y, Z, allowing 3D imager(1). Recently, physicists at the University of Osaka in Japan have been able to use the AFM to identify the chemical structure and determine the type and location of each atom on the three-dimensional pattern of the surface of matter. These scientists have discovered that interactions are an atomic imprint to distinguish atoms using an AFM microscope(2). Surface studies can be provided detailed information on chemical reactions on the surface, such as damage(3). A device has been used; Model: Wet - SPM9600 (Scanning Probe microscope) Shimadzu made in Japan, Non-Contact mode.

2.4 Artificial aging

Artificial aging aims to study the behavior of treated samples under the influence of specific conditions, as they correspond to the environmental conditions surrounding the original building walls, especially open environments, which are characterized by variable weather factors, especially changes in temperature and humidity.

The aging cycles conducted as follow:-

- The samples were dried in a furnace for 24 hours at a temperature of 105 ° C and recording their dimensions and weights.

- The treated mud samples were immersed in the water for three hours, then left to dry in the room atmosphere of 8 hours.

- Then enter the oven temperature of 70 º for 4 hours, and this method was repeated ten consecutive cycles.

3. Results and discussion

3.1 Optical Microscope

The investigation of mud brick samples shows that a high percentage of sand, as shown in Figure 2. Thus, sand was added to the clay in high amount to optimize the mudbrick blocks.

(1)Bhatt.P.M.; and others; Low temperature Atomic Force Microscopy-A Review Paper ,Journal of Mechanical and Production Engineering (JMPE) Volume 2, Issue 2, Sep 2012, PP 109-110 .
(2)http://www.syr-res.com
(3)Stuart, B.H.; Op. Cit., PP.100-101.

Fig. 2 sample 1 of the bricks in the pyramid area (X5)

3.2 SEM/EDX

The results of the examination and analysis revealed the presence of some cracks and the degradation or loss of some of the cohesive in the first sample fig.3,4. The investigation of the second sample showed the presence of some elements such as carbon C and titanium Ti and the proportion of iron Fe higher than the secondfig.(5,6)

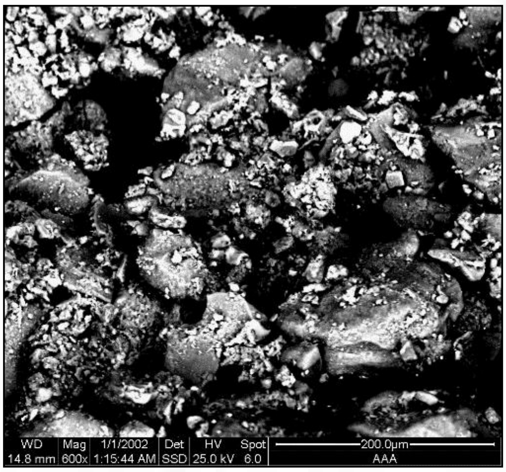


Figure (3) of sample 1 of the bricks in the pyramid area

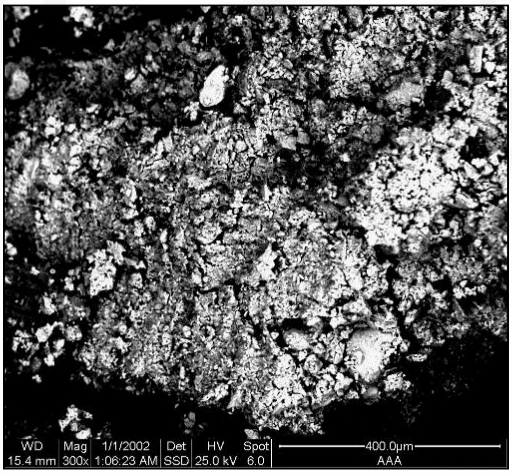


Figure (4) of sample 2 of the bricks in the pyramid area

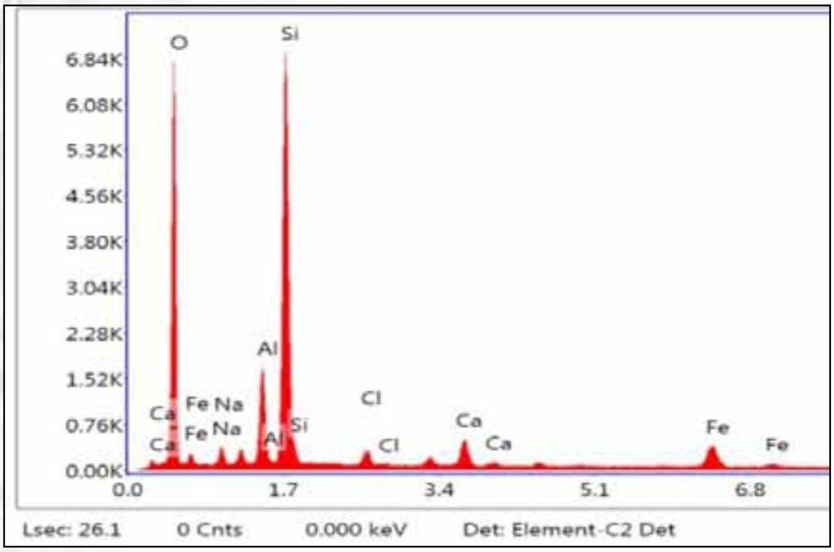


Figure (5) First sample analysis pattern using SEM supplied with EDX.

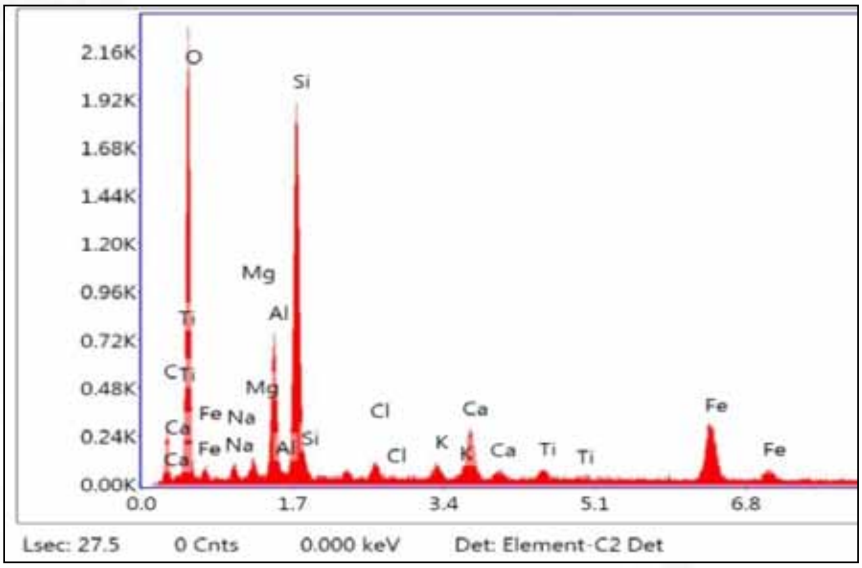


Figure (6) Second sample analysis pattern using SEM supplied with EDAX.

3.3 X-Ray Diffraction Analysis(XRD)

The first sample:A study of the X-ray diffraction pattern of the sample showed that quartz (SiO_2) present in high concentration 85% and Albite ($NaAlSi_3O_8$) and calcite ($CaCO_3$) present in 15%, as shown in fig.7.

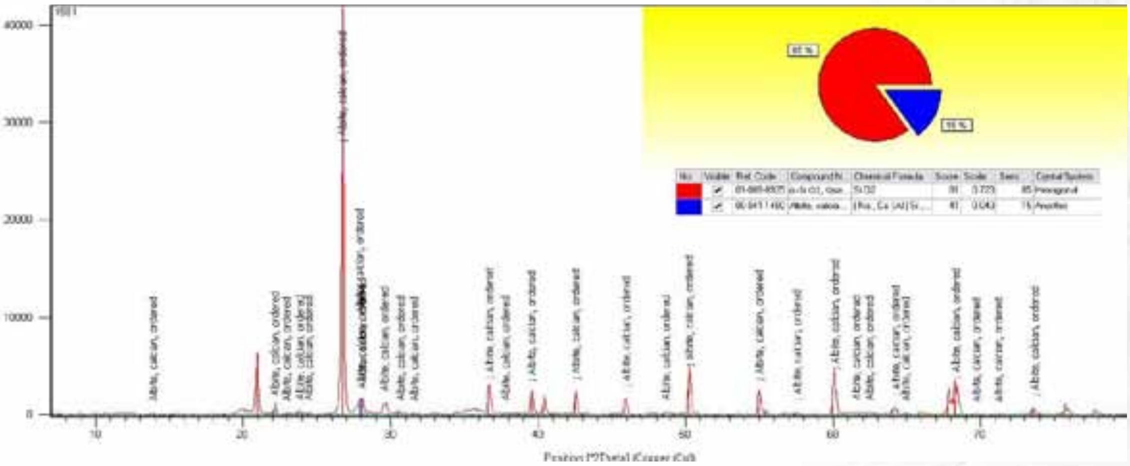


Fig.7XRD- pattern for the first sample

The second sample: X-ray diffraction pattern shows that quartz (SiO_2) found at con centration of 69% and albite ($NaAlSi_3O_8$) was 31%, as shown in Figure 8.

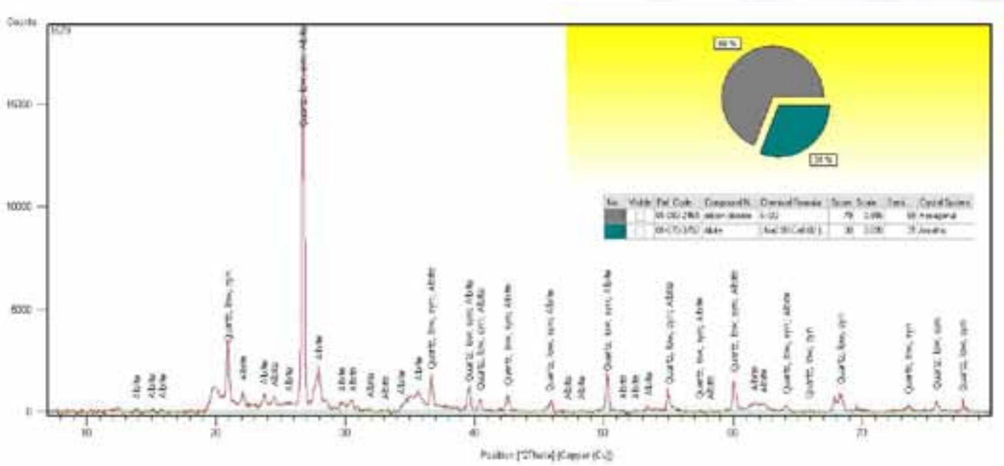
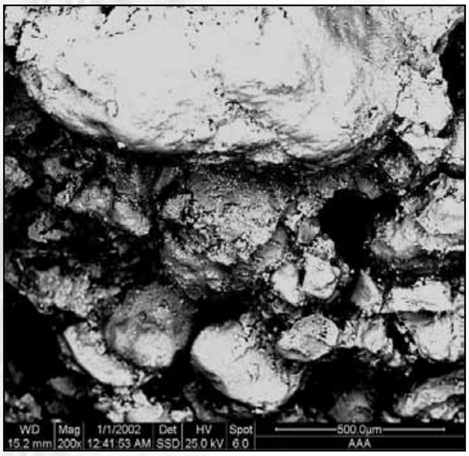


Figure (8)XRD pattern for the Second sample

3.4 Treated samples

Scanning electron microscope (SEM)

The imaging (SEM) showed CaLoSiL penetration and diffusion in a homogeneous manner.



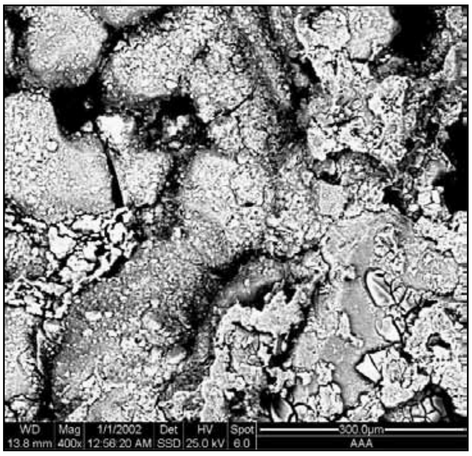
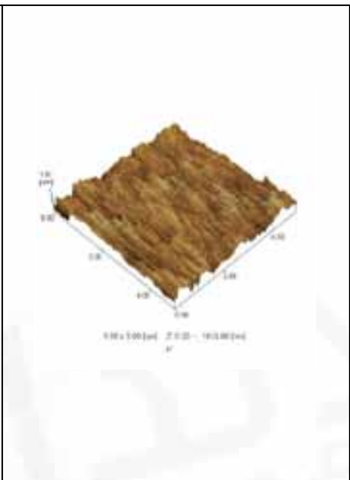


Figure (9) mud brick untreated – after artificial aging.

Figure (10) mud brick treated with CaLoSiL after artificial aging.

3.5 Atomic Force Microscope (AFM)

Figure (11) and (12) show a study of the surface of the two samples. Figure (11) shows a weakness in the cohesion of the components and erosion of the surface of the sample and its impact on the damage factors, while the picture (12) shows the cohesion and bonding of the container and is the best case of the sample First.



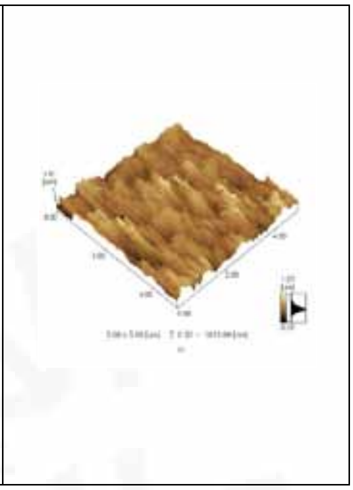


Figure (11) Three-dimensional images of the surface of the first sample surface.

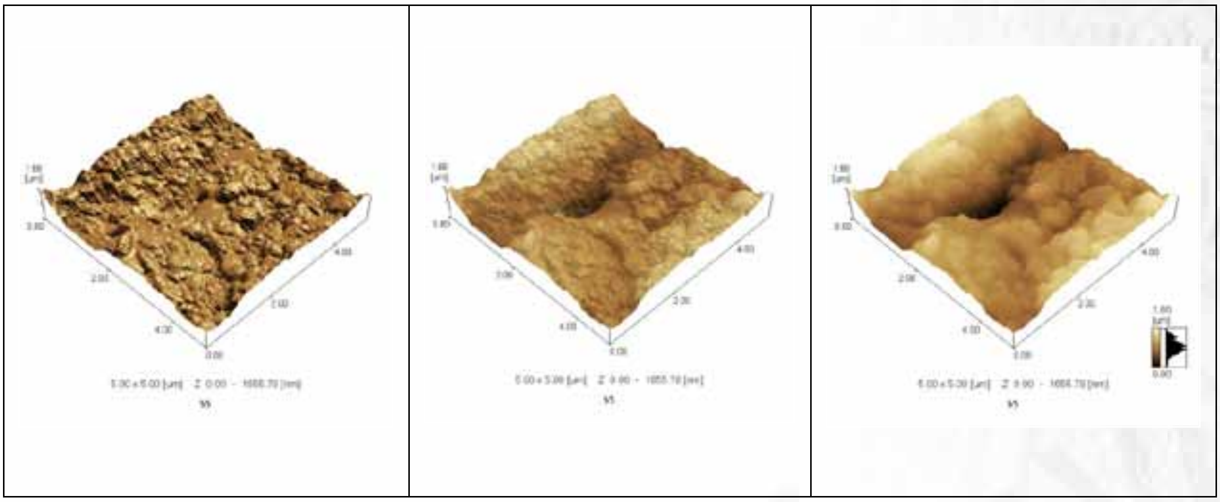


Figure (12) Three-dimensional images of the surface of the second sample surface.

3.6 Physical properties

3.6.1 Compressive Strength Tests

This property is defined as the ability of materials to withstand the load resistance and pressures in different directions before the material breaks down or turns into granules. The pressure resistance test was performed using a pressure machine of type ASTM D 3787.The average resistance to pressure for the untreated sample was 397 kg / cm3.The average resistance to pressure for the sample treated with CaLoSiL was 1385 kg / cm^3.

3.7 Submersion Test

This test is one of the important tests after chemical treatment, to ensure that the materials of the resistance to resist various sources of moisture. The resistance of the brick to the water effect was observed compared with the untreated sample that collapsed as soon as it was immersed in water, fig. 13 shows the resistance time of mudbrick block before its break comparing to untreated samples.

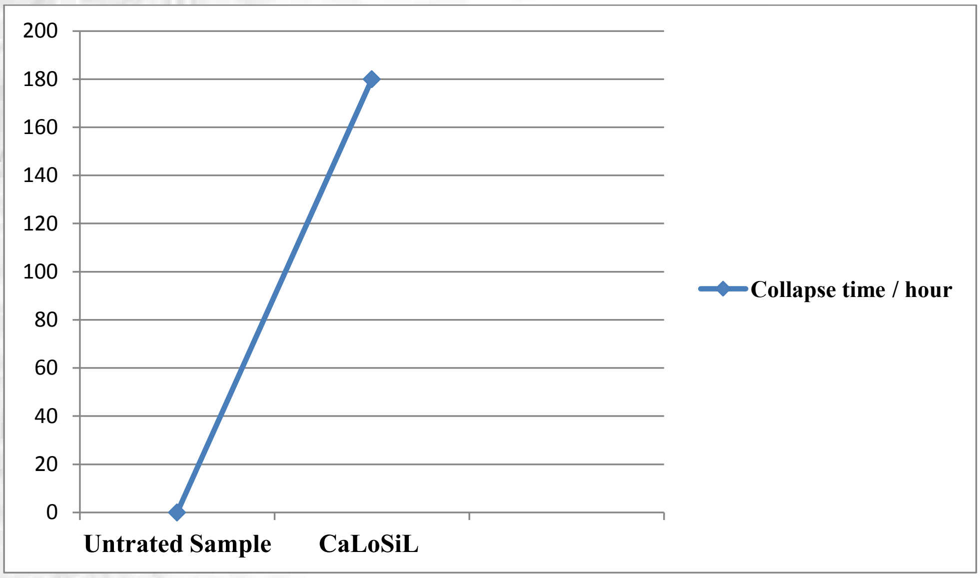


Fig. 13 the resistance time of treated and untreated samples inwater.

3.6.3 Chromatic changes after artificial aging

Chromatic changes in the sample after artificial aging were studied, and the results were recorded that there were no changes in samples color after consolidation and aging process.

3.6.4 Loss of Weight.

Samples after aging on treated brick samples was also evaluated by calculating the percentage of loss in the weight of samples before and after aging, the results was as follows that samples treated with CaLoSiL loss of weight average are.35%

4. Conclusion

The study of the physical and mechanical properties of the experimental samples showed the effectiveness of calcium nanohydroxide in improving the resistance of the mud bricks against the external factors and influences. The SEM investigation reveal the homogeneity of CaloSil in the test samples blocks.The treated mud bricks showed good resistance to the pressure, in addition to improving the resistance of the mud bricks to the effect of moisture, confirming the effectiveness and success of calcium Nano hydroxide.

REFERANCES

1- فتح الله الشيخ و محمود موسى : قصة النانو تكنولوجيا حاضرها و مستقبلها ، المكتبة الأكاديمية ، الطبعة الأولى ، 2009 .

2- Andrew, D. M., Nanotechnology: AResearch Strategy for Addressing Risk, Woodrow Wilson ،2006.

3- Bennet, D., Nanotechnology: Ethics and Society, CRV press, Taylor and Francis Group, U.S.A ،2008 .

4- Bhatt.P.M.; and others; Low temperature Atomic Force Microscopy-A Review Paper ,Journal of Mechanical and Production Engineering (JMPE) Volume 2, Issue 2, Sep 2012.

5- Chandravathanam, S., Nanomaterials: A sojourn, N. C.for Catalysis Research, 2006.

6- Daniele, V. et al., The Nanolimes in Cultural Heritage Conservation : Characterization and Analysis of Carbonatation Process, Journal of Cultural Heritage 9, 2008.

7- Dei, L. et al., Nanotechnology in Cultural Heritage Conservation: Nanometric Slacked Lime Saves Architectonic and Artistic Surfaces from Decay, Journalof Cultural Heritage 7, 2006.

8- Doehne, E. and Price. C., Stone Conservation : An Overview of Current Research, Paul Getty publications, Second Edition, Los Angeles, 2010.

9- Filipponi, L., and Sutherland, D., Introduction to Nanoscience and Nanotechnologyies، Interdisciplinary Nanoscience Center (iNano), Aarhus University, Denmark, 2010 .

10- Giorgi, R. et al., A new Method for Consolidating Wall Paintings based on Dispersions of Lime in Alcohol, Studies in Conservation 45, 2000.

11- Manoharan, M., Research on the frontiers of Material Science: The impact of nanotechnology on new material development, Technology in Society, Vol. 30, 2008 .

12- Michel, F. A. and Paulo, J.F. and Daniel, L. S., Nanomaterials: Nanotechnologies and Design، Elsevier Ltd, 2009.

13- Stuart, B.H; Analytical techniques in materials conservation, John Wiley & SonsLtd, England, 2007.

14- Toney, M. F., X Ray Diffraction, in Encyclopedia of materials characterization, Butterworth-Heinemann, U.S.A, 1992.

15- http://www.syr-res.com

16- https://ar.wikipedia.org

17- https://ibz-freiberg.de/en/products