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STUDY THE EFFECT OF NANO MATERIALS ADDITION ON SOME PROPERTIES OF CEMENT MORTAR

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Abstract: This study involves nano-materials addition and interaction with cement mortar behavior for many mortar samples under variable curing time with constant water to cement ratio (W/C = 0.5). In this research some properties such as (hardness and water absorption test), were affected by adding small ratios of nano-particles (SiO_2 or Al_2O_3) as replacements to the Ordinary Portland Cement (OPC) / type (I). The percentages of nano materials additives replacement on the mixture of mortar include (1, 2, 3, 4 and 5%) for both nano materials with constant (W/C) ratio. Also the amount of the fine aggregate used was three times the amount of cement. The results showed that, the hardness of the mortars for both nano materials gives better properties than mortar without nano materials in all tests. Best enhancements in properties for mortars with nano silica were achieved with (3%) additives while were achieved with nano alumina at (2%) additives.

Keywords: *Hardness, Water absorption, nano-Al_2O_3, nano-SiO_2.*

دراسة تاثيرات اضافة المواد النانوية على بعض الخواص لمونة السمنت

الخلاصة: تتضمن الدراسة اضافة المواد النانوية وتداخلاتها مع سلوك مونة الاسمنت لكثير من العينات تحت تاثير اوقات متغيرة للمعالجة مع نسبة ثابتة للماء وبنسبة (W/C = 0.5). في البحث الحالي بعض الخواص الميكانيكية(اختبارخاصية الصلادة) والفيزياوية (اختبار امتصاص الماء) ، تأثرت من خلال اضافة نسب صغيرة من جسيمات نانوية (SiO_2 or AL_2O_3) وكأضافات الى الأسمنت البورتلاندي العادي (OPC) نوع (I). النسبة المئوية للمواد النانوية المضافة الى خليط من عينات مونة الاسمنت كانت (1، 2، 3، 4، 5%) لكلا المواد النانوية المضافة ، كما استخدم الركام الناعم بثلاثة أضعاف كمية من الاسمنت المستخدم. اضهرت النتائج تحسن وارتفاع صلادة مونة الاسمنت لكل العينات الحاوية على مواد نانوية بالمقارنة مع العينات بدون المواد النانوية المضافة ولكل الأختبارات. تم الوصول الى افضل تحسن في الخصائص للعينات المضاف اليها النانو السيليكا عند نسبة (3%) بالمقارنة مع العينات المضاف اليها النانو الومينا والذي تحقق بنسبة (2%) مواد نانوية مضافة.

1. Introduction

Recently nano technology is being used in many applications and it has received increasing attention in building materials. Significant number of works dealing with the use of nano particles in cement based materials to be used in constructions [1]. However, there is a limited knowledge about the mechanism by which nano particle affects the properties, setting times, consistency, workability, rheological, micro structural, mechanical properties etc of cementitious, mixes. Furthermore, the literatures appear to

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be contradictory about the influence of nano particles on the development of such materials (building materials) [2].

The effects of nano additives like (nano-Fe_2O_3) and (nano-SiO_2) on cement mortars properties must be experimentally studied. The experimental results will shows the affected properties measured (for example: at the (7^{th}) day and (28^{th}) day) of the cement mortars mixed with the nano-particles compared with of a plain cement mortar. The (SEM) study of the microstructures between the cement mortar mixed with the nano-particles and the plain cement mortar showed that the (nano-Fe_2O_3) and (nano-SiO_2) filled up the pores and reduced ($CaOH_2$) compound among the hydrates. These mechanisms explained the supreme mechanical performance of the cement mortars with nano-particles [3].

The mechanical properties and permeability of (porous concrete containing (Nano-SiO_2)-(PCNS)) were studied, researchers studied also (plain porous concrete (PPC)) and (porous concrete containing (Micro-SiO_2)-(PCMS)) as control materials. Results for (PCNS) specimens cured for (28 day) showed higher compressive and flexural strengths comparing to those of (PPC) with the same water–cement ratio (W/C).

It was demonstrated that the (Nano-SiO_2 (NS)) was more effective than (Micro-SiO_2 (MS)) in enhancing (porous concrete (PC)) flexural strength. Samples with (5% NS) by weight of binder presents the highest mechanical properties where the flexural and compressive strength of (porous concrete (PC)) were enhanced by (56% and 48%), respectively. Experimentally results showed that the permeability behavior of (PCNS) and (PCMS) decreased, results appeared that (Nano-SiO_2) (NS)) and (Micro-SiO_2) (MS)) could both reduce water penetration through (porous concrete (PC)); however, (Micro-SiO_2 (MS)) was found more effective for equal nano particles and material voids [4]. Casting of ferro cement elements reinforced with (nano-SiO_2 (NS)) particles were investigated and specimens were tested to determine their mechanical properties, durability and microstructural properties of interfacial transition zone (ITZ). The amounts of replacement ratios of (nano-SiO_2) (NS)) particles were low with respect to cement in (Ordinary Portland Cement (OPC)) mortar mixture.

Ratios were (1%, 2% and 3% (nano-SiO_2) (NS)), water to binder ratio (0.35, 0.4 and 0.5) and sand to binder ratio (2 and 2.5). The results have shown that the cement mortars containing nano particles have reasonably higher strength, low water absorption and denser (ITZ) compared to those of the (OPC) ferro cement mortars. [5]. The influence of other nano particles on mechanical properties and durability of concrete had been investigated by adding constant content of (nano-ZrO_2 (NZ), nano-Fe_3O_4 (NF), nano TiO_2 (NT) and nano-Al_2O_3 (NA)) to concrete mixtures. Mechanical properties have been investigated through the compressive and indirect tensile strength and durability has been investigated through chloride penetration test and concrete permeability.

Results of this study showed that nano particles can be very effective in improvement of both mechanical properties and durability of concrete. Results of this study seem to indicate that the (nano-Al_2O_3) is most effective nano-particle of examined nano materials in the improvement of mechanical properties of high performance concrete [6].

Two different phases for the use of waste ground ceramic as a pozzolan in concrete were investigated. Concrete samples with (10–40%) of ground ceramic powder substitution were made. Then study the simultaneous effect of using (0.5–1%) of ((nano-SiO_2) (NS)) and from (10% to 25%) of ground ceramic powder were determined. In all cases, compressive strength and water absorption tests were performed. Results showed that adding ground ceramic up to (20%) does not have a significantly negative effect on the compressive strength of concrete. Furthermore, using any amount of ground ceramic in the concrete reduces its water absorption capacity.

Using ((nano-SiO_2) (NS)) and pozzolan simultaneously leads to improved compressive strength and reduced water absorption capacity. Therefore, ((nano-SiO_2) (NS)) can improve the effects of ground ceramic powder on the properties of concrete [7]. Two different types of nano silica (NS) applied in self-compacting concrete (SCC) were studied, both having similar particle size distributions (PSD), but produced through two different processes: fumed powder silica and precipitated silica in colloidal suspension. The influence of nano silica on (SCC) were investigated with respect to the properties of concrete in fresh (workability) and hardened state (mechanical properties and durability).

Additionally, the densification of the microstructure of the hardened concrete were verified by (SEM) and (EDS) analyses. The obtained results demonstrate that nano silica used in (SCC) can improve its mechanical properties and durability. Considering the reactivity of the two applied nano silica, the colloidal type showed a higher reactivity at early age, which influenced the final (SCC) properties [8]. The mechanical properties and the durability were studied for self-compacting concrete (SCC) containing alginate in variety values with artificial stone resin, micro and nano silica.

The values of (0.5 and 1%) alginate, (10%) micro silica, (0.5%) nano silica and (0.5%) artificial stone resin were used. Artificial stone resin was used as the super plasticizer. Properties of hardened (SCC) such as compressive, split tensile, flexural strength and water absorption were assessed and represented graphically. In general, the use of alginate improved the performance of (SCC) in fresh state and also avoided the use of viscosity modifying admixtures. Adding nano silica to samples increased (SCC) and both (workability and the concrete split tensile strength) decreased in (0.5%) alginate and in all mixes receptacle (0.5%) alginate. Adding micro silica to alginate increases of the split tensile strength while adding nano and micro silica decreases the values of water absorption [9].

2. Experimental Work

2.1. *Materials*

There are many materials which are used to prepare specimens, these materials consist cement, fine aggregate, water and nano materials.

2.1.1. *Water*

The water is used for all physical tests and mortars specimens' preparation from mixing and curing stages and it was used for all the mechanical tests that had been carried out.

2.1.2. *Fine aggregate*

Fine aggregates generally consist of natural sand or crushed stone with most particles smaller than (5 mm). The most desirable fine-aggregate grading depends on the required of work. Fine-aggregate grading within the limits of (ASTM C 33) is generally satisfactory for most concretes**.** AL-Ekadir in Karbala region sand was used as fine aggregate. It was tested to determine the grading and other physical properties. The sand used in this study is according to the standard specifications.

2.1.3. *Cement*

The type of cement was used in all mixes in this research is the Ordinary Portland cement (type I). The chemical analysis of the cement composition used is listed in table (1). The cement concordance with the Iraqi specification (No.5/1984) ordinary Portland cement (type I) from (Al Mass Iraqi cement factory), the test in the Table (1) was achieved in (National Centre for Construction Materials (NCCMLR) Lilaboratory and Rresearch.

Table (1): The chemical composition of ordinary Portland cement (OPC).

Oxides Composition	*Oxide content%*	*Limits of Iraqi specification No.5/1984*
SiO_2	20.26	-
Al_2O_3	5.50	-
Fe_2O_3	2.19	-
CaO	61.39	-
MgO	1.99	<5.00
SO_3	2.7	<2.8
Free CaO	1.12	-
Loss on Ignition	3.2	<4.00
Insoluble Residue	0.73	<1.50
Lime Saturation Factor	0.94	0.66-1.02

2.1.4. *Nano materials*

Two types of nano materials are used. The first type is nano silica particles (SiO_2) and other type is nano alumina particles (Al_2O_3). Both have high purity approach to (99.9%) and particle size rounded range between (15 to 20 nanometer). Nano Shell Company is the source of nano particles improved from (Arrege Alfrat Company).

Table (1): The main properties of nano-materials replacement.

No.	*Material*	*Particles size (nm)*	*Purity percentage%*	*type*	*Surface area (m^2/gm)*
1	Silica	15-20	99.5	S	170-200
2	Alumina	20	99.9	γ	230-400

2.2. *Preparation of mortar mixing*

The suitable mortar mixing was prepared by using cement-sand ratio of (1:3) with (W/C) ratio of (0.5%). The blended cement mortar was prepared using ordinary Portland cement that was partially replacements in (1, 2, 3, 4 and5%) by nano- particles. The ingredients were homogenized on an electric mixer to assure complete homogeneity. The mortar pastes were molded into (20 mm) cubes for water absorption according to ASTM C642-2004, (20 mm) cubes for hardness. The samples were kept in molds at (100%) relative humidity for (24 hours) and then cured in water for (3, 7, 14, 21, 28, 60 and 91 days). The mixtures are shown in figures (1-a) and (1-b).

Figure (1): The mixture of cement mortar (a) dry state (b) with nano-materials addition.

2.3. *Testing*

2.3.1. *Surface Hardness strength test*

Shore hardness method with type (D) for brittle materials was used as a (Non Destructive) test. The used device was (HPE II) which it is digitalized for decimal precision. The force can be applied on the specimen through the help of a patented force grip which is on the center of the instrument body. The final result of hardness recorded was the average of six measuring operations according to (ASTM D2240).

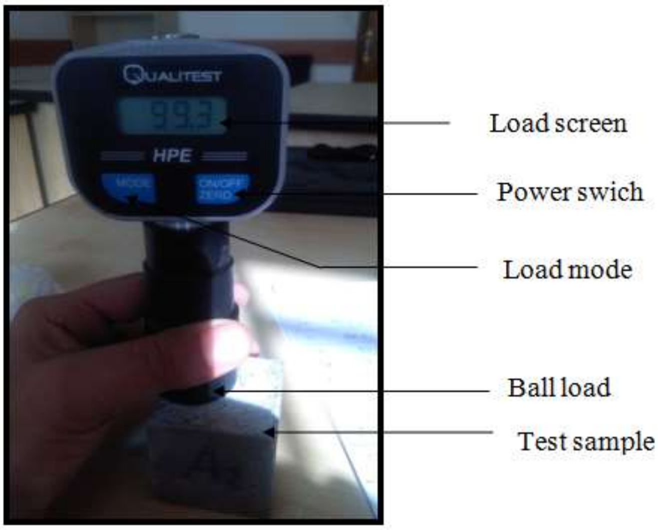

Figure (2): Surface Hardness Device.

2.3.2.*Water absorption*

One of the most important tests for specimen was water absorption. This test was done accordance to ASTM C642.The procedure of this test consist of, the samples were taken and dried in oven with temperature of (105°C ± 5) for 24 hours, and then the samples was taken out and weighed .Then, the samples was immersed in water for 24 hours, then removed, surface dried with a cloth and finally weighed again. This test was achieved at curing time of (3, 7, 14,21,28,60 and 91day). The degree of oven was used to drying samples reach to (3000°C).

2.4. *Result and Discussion*

2.4.1. *Water absorption*

One of a most important test to measuring mortar or concrete durability is called water absorption. The total amount of required water absorption that will give an indicator for mortar or concrete durability which water fill and enter into the voids of mortar and saturates the matrix pores. Figures (3 and 4) can illustrate a comparison for the results of water absorption test between the control mortar and specimen contain replacement additions.

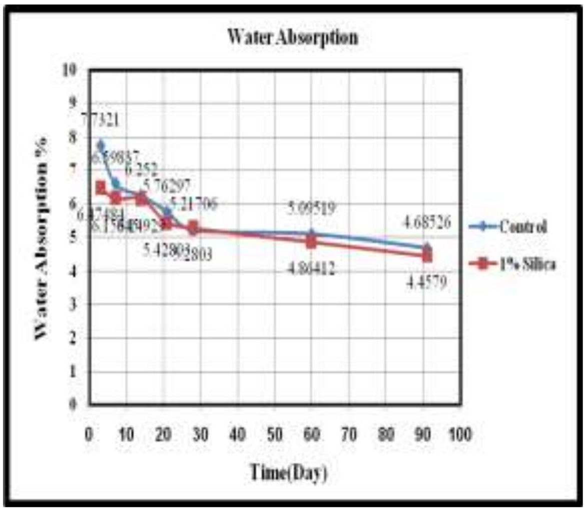

Fig.3: Water absorption for (1% nano Silica) addition.

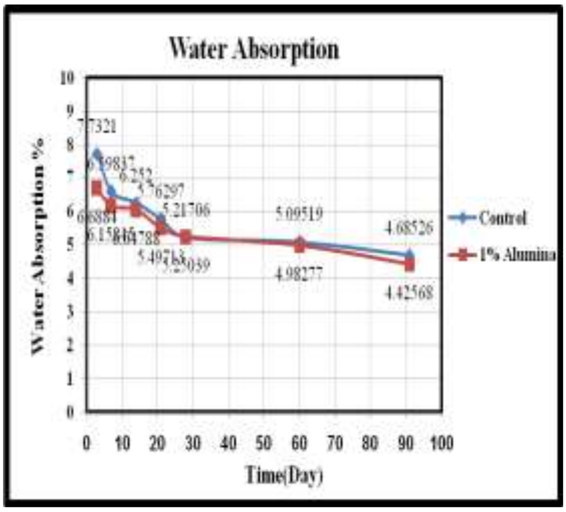

Fig.4: Water absorption for (1% nano Alumina) Addition.

It was found that water absorption decreases in specimens contain (1% nano silica) and specimens contain (1% nano alumina) compared with control samples.The results give the indication that, when the amount of silica nano particles increases it will cause a decrease in water absorption. This behavior can be seen in figures (1 and 2). When the amount of nano silica (SiO_2) reaches (2%) as seen in figure (5) water absorption decreased more than for (2%) nano alumina (2% Al_2O_3) and control. As seen in figures (5 and 6), water absorption values is less than control samples. The water absorption of (2%) nano alumina (2% Al_2O_3) can be seen in figure (4).

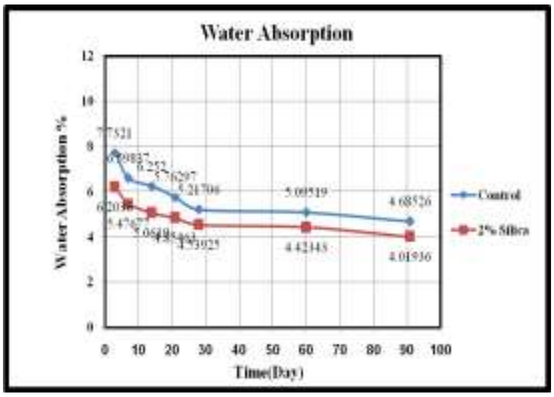

Fig.5: Water absorption for (2% nano Silica) addition.

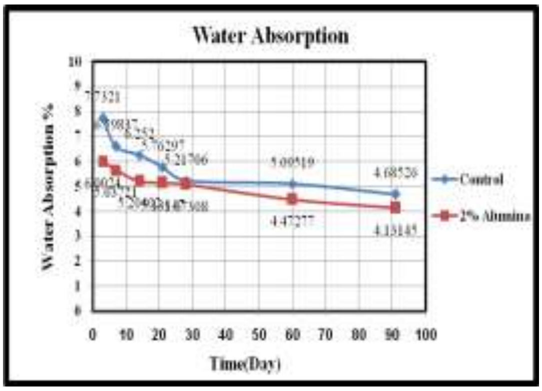

Fig. 6: Water absorption for (2% nano Alumina) addition.

When the amount of nano silica increases to ((3%) nano-SiO_2) replacement, water absorption reaches its maximum value, this is illustrated in figure (7), nano alumina effect is shown in figure (8) for different curing time.

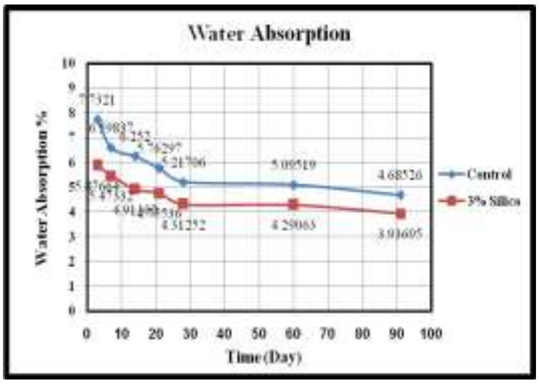

Fig.7: Water absorption for (3% nano Silica) addition.

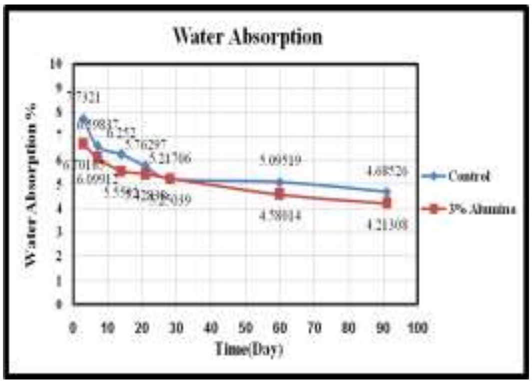

Fig. 8: Water absorption for (3% nano Alumina) addition.

The materials at (3%) nano silica (SiO_2) gives the optimum behavior of water absorption This can be attributed to the pozzolanic reaction of the nano silica that continues with age and consumes the calcium hydroxide to produce additional calcium silicate hydrate. This product fills in pores, which caused the previously mentioned high air content values in the fresh state and results in a refinement of the pore size distribution. This leads to a lower permeability and a higher resistance to absorption. So, it can be concluded that the utilization of nano silica has a positive effect on filling the voids in the mortar matrix and hence reduces the absorption.

while for nano alumina (Al_2O_3) is at (2%) which gives the highest value, this is because the parameters that controls the decrease on water absorption is the capacity of filling process, activating of pozzolanic reactions and the hydration rate of the cement, all these will create decrease and the porosity and the materials become more dense than control specimen. Smaller nano-particles react faster than the nano-particle size, the more the heterogeneous nucleation sites gives lower water absorption; therefore, the nano particles must be dispersing as possible as to give the required improvement for the mortar properties in order to prevent the agglomeration. Figures (9) and (11) represented the water absorption of nano silica (SiO_2) addition at (4 & 5%) respectively.

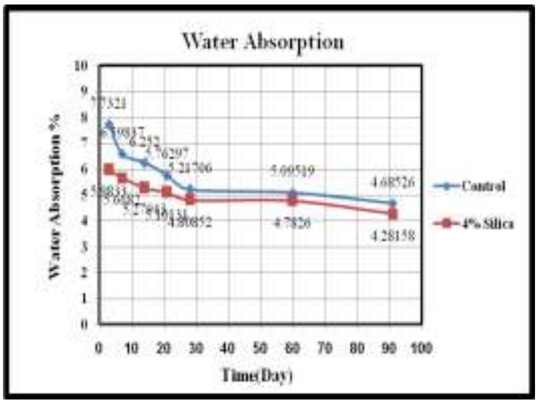

Fig.9: Water absorption for (4% nano Silica) addition.

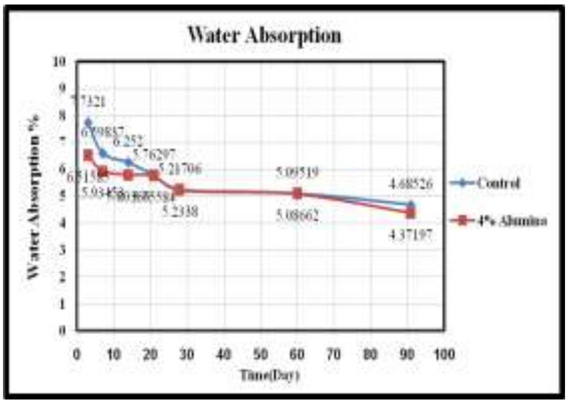

Fig. 10: Water absorption for (4% nano Alumina) addition.

Figures (10) and (12) represents of nano alumina (Al_2O_3) replacement addition at (4 & 5%) respectively. These illustrate the water absorption which begins to increase and near to control specimens. Defects may be generated during the dispersion of nano-particles that causes weak area.

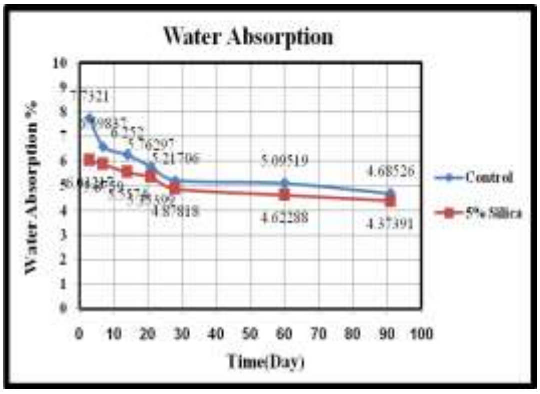

Fig.11: Water absorption for (5% nano Silica) addition.

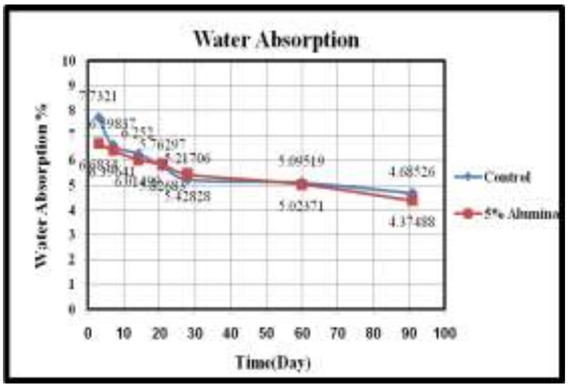

Fig. 12: Water absorption for (5% nano Alumina) addition.

2.4.2. *Hardness analysis*

In this test, samples of mortar were prepared with suitable scale and then tested. The investigation of hardness test is concerned with the development of materials surface. Small samples of cement mortar with and without nano materials additives tested. Figure (13) explain the amount of ((1%) nano-silica (SiO_2)) addition compared with control specimen. The value of hardness increased for about up to (3%) at (28 days). With ((1%) nano alumina (Al_2O_3)) the surface hardness value is increase for about (1%) than in control ordinary cement, this can be seen in figure (14). As seen in figures, the nano silica mortar remain more than both nano alumina and control samples.This nano materials makes the mortar more denser with low in voids. Increasing in the values of hardness can be seen figures (15) and (17) for (2 and 3%) nano-silica addition respectively.

The resin of such result is the material became extreme in pozzolanic characteristic of silica nanostructures and (Calcium-Silicate-Hydrate) forms as results of dense gel.

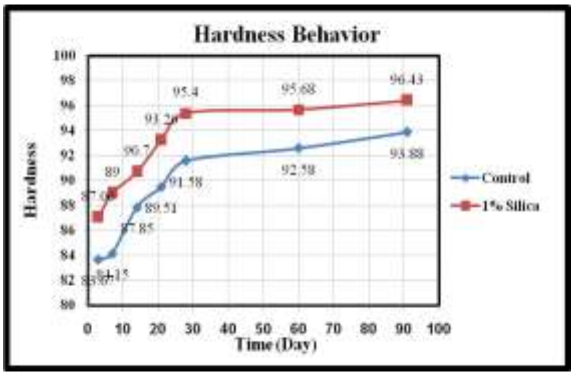

Fig.13: Surface hardness for (1% nano Silica) addition.

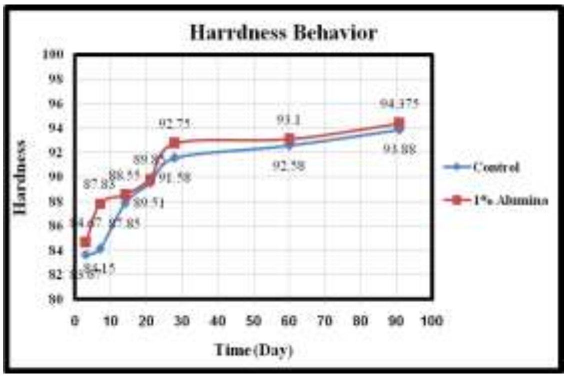

Fig. 14: Surface hardness for (1% nano Alumina) addition.

For ((2%) nano alumina addition), as seen in figure (16). The values of hardness increased for (2%) nano alumina replacement addition compared with control ordinary cement. This is because of decreasing of the voids size which becomes less. However, nano particles cause a reduction in permeability, because nano particles in paste of cement will make the materials particles packed together. When the amounts of nano-alumina reach to 3% the surface hardness is to decrease because the formation of more pores in the cement mortar and hence weakens the strength of the hardened matrix. In particles that produce stronger cement paste-fine. In addition, the excess of free water pores will contain non-reacted nano alumina particles.

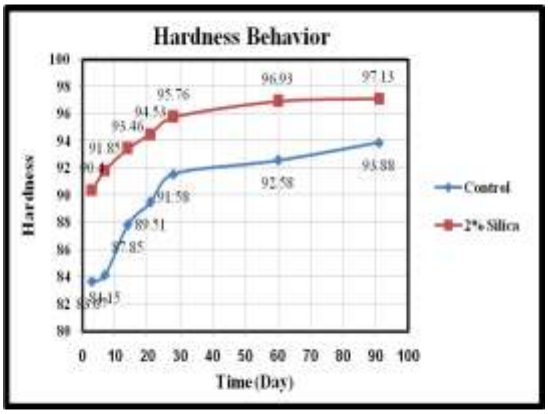

Fig.15: Surface hardness for (2% nano Silica) addition.

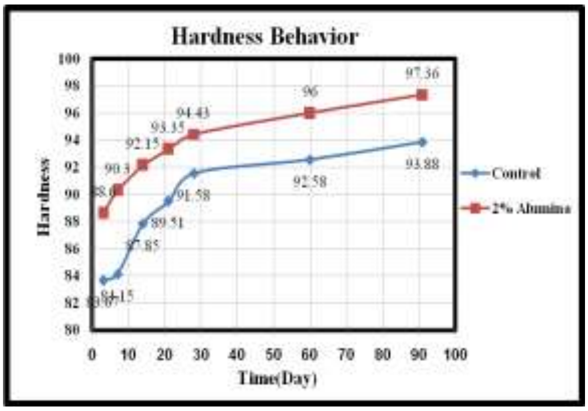

Fig. 16: Surface hardness for (2% nano Alumina) addition.

However, when the nano-alumina particles cannot be well dispersed, as the case of extensive nano-particles content, the agglomeration nano-particles create weak zone in the form of voids consequently, the homogeneous hydrate microstructure could not be formed, and low strength will be expected. Weak zone in the form of cavities will be produced due to unsuitable dispersing of nanoparticles.

With the increase of (nano silica (SiO_2)) particles quantity up to (3%), microstructure was improved completely and achieved better density.The high early enhancement in the strength is due to the filling effect of nano silica particles that reinforces the micro structure of the matrix. Moreover, the ultra high surface area of nano-Silica and hence particles accelerates the chemical reaction and hence contributes to the early enhancement in strength. Usage of more amounts of nano-silica particles more than (3%) will lead to make the cement paste matrix able to dense in a way that remarkable permeability reduction has been observed. So, the pozzolanic performance of silica nano particles is clearly in the case of mortar durability.

This was found in samples containing (nano silica (SiO_2)) particles and because of the agglomeration of nano-particles voids were create. Figures (19) and figure (21) illustrate the harness behavior at ((4 and 5%) nano-silica (SiO_2)) particles addition. Using nano alumina up to (5%) will cause the hardness behavior to decrease, these can be seen in figures (18), (20) and (22) represent with (3, 4 and 5%) nano addition respectively which effects on hardness behavior. These nano particles will cause uniformly distribute in cement mortar and due to agglomeration, weak zone appears in the cement mortar. However, when the particles of nano alumina cannot be in good dispersed, as the case of widening nano-particles content, the agglomeration nano particles make weak zones in the form consequently voids, the homogeneous structure during hydrate process could not be formed and also strength become low.

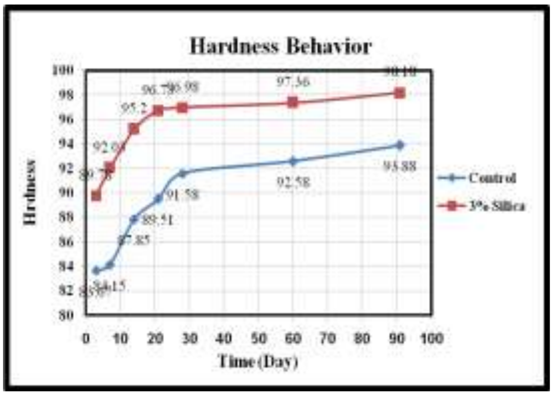

Fig.17: Surface hardness for (3% nano Silica) addition.

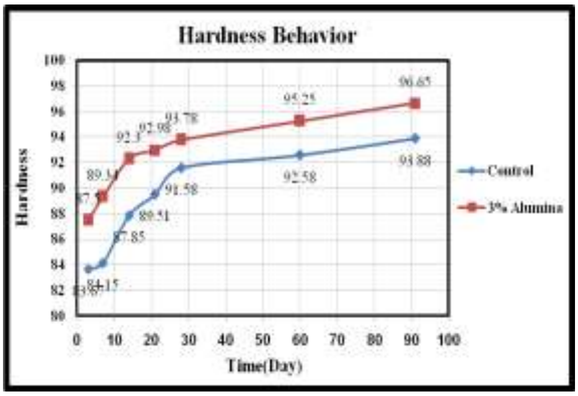

Fig. 18: Surface hardness for (3% nano Alumina) addition.

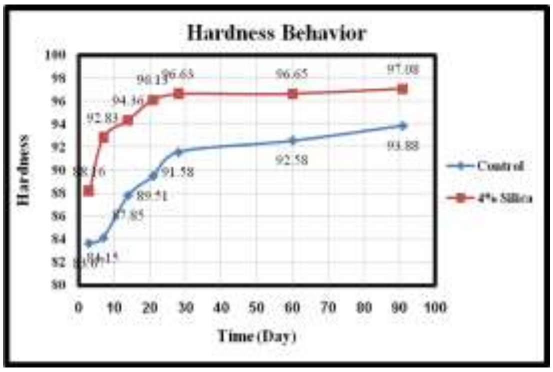

Fig.19: Surface hardness for (4% nano Silica) addition.

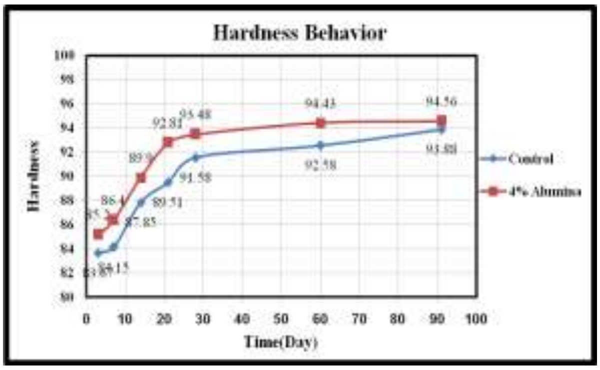

Fig. 20: Surface hardness for (4% nano Alumina) addition.

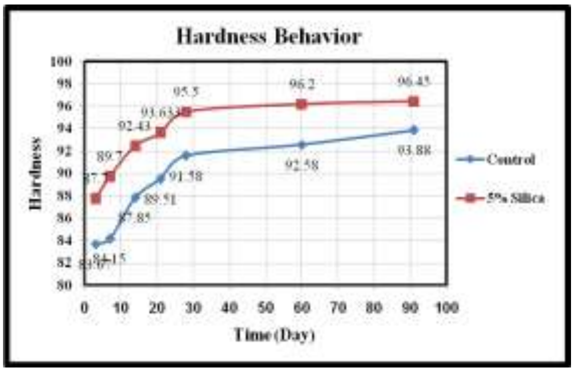

Fig.21: Surface hardness for (5% nano Silica) addition.

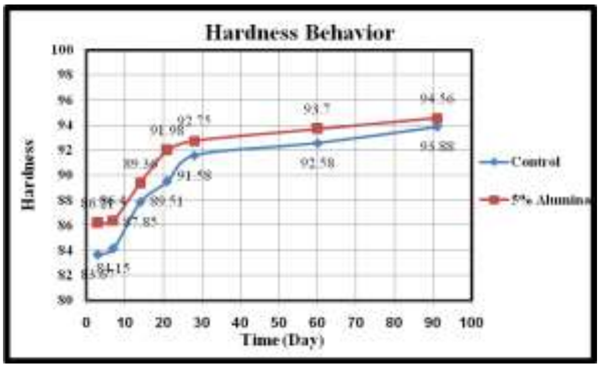

Fig. 22: Surface hardness for (5% nano Alumina) addition.

2.5. *Conclusions*

According to the results from the experimental tests, the following points can be concluded:

1. Application of both nano-particles (in low amount of replacement up to 1,2,3,4 and 5%) can lead to microstructural development due to their multi functional behavior in the matrix of cement-based materials. This characteristic can enhance both water absorption and surface hardness property of cement mortar specimens.
2. The silica nano-particles into the matrix of cement mortar that are prevention of growth of harmful crystals such as $Ca(OH)_2$ and, more production of C-S-H gel, micro and nano filling effect, and helping the hydration reaction to be developed due to nucleus-like action.
3. The hardness strength of mortar consist of nano silica is higher than mortar containing the same replacement of (nano-Alumina) at all curing ages.
4. Optimum (nano silica) replacement ratio improved the surface hardness was (6%) at (91 days) compared with control mortar and optimum (nano alumina) replacement ratio which improve the surface hardness was (4.5 %) at (91 days) compared with control mortar.
5. The water absorption was improved by (17%); the enhancement of water absorption by ((3%) nano-Silica-(SiO_2)) replacement at (91 days) above controls mortar and the water absorption was improved by (14%); the enhancement of water absorption by ((2%) nano alumina-(Al_2O_3)) replacement at (91 days) above controls mortar.
6. Nano particles improve water absorption resistance of all specimens containing both nano materials (nano silica and nano alumina) compared with control specimens.
7. The enhancement of surface hardness strength by nano silica replacement was achieved especially at (3%) addition while it was achieved by nano alumina addition at (2%) replacement at all curing time.
8. The enhancement of water absorption by nano silica was achieved at (3%) addition while it was achieved by nano alumina addition at (2%) at all curing time.

9. At all curing ages, hardness strength of mortar cubes cast contain (nano silica (SiO_2)) was higher than containing the same amount of (nano alumina (Al_2O_3)).

3. References

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