

The Effect of Using Hybrid Nano Materials on the Different Properties of Cement Mortar

Mostafa A. Mahdi (a), Saaid I. Zaki (b), Hossam A. Hodhod (c)

Abstract— The aim of this work is to study the effect of nano materials on the properties of mortar, the experimental program included three parts: a- two types of Nano Silica, locally produced NS1 and imported NS2, b- Nano clay (NC) and c- Hybrid nano particles (NS1 & NC). In each part, mortar was used with different percentages of nano particles. Compressive strength and drying shrinkage tests were applied in each part on the cured and uncured samples. Thermal conductivity and Scanning Electronic Microscope (SEM) tests were conducted on the optimum mortar samples. Finally, feasibility study about the cost of using nano materials in the construction was carried out. The results showed that the compressive strength improved in the mortar mixtures in the cured condition, the optimum percentages was 1% for NS1, 1% for NS2, 5% for NC, and 5% (0.5%NS1 & 4.5%NC) for hybrid nano particles. The drying shrinkage and thermal conductivity increase with adding nano silica and hybrid nano particles, while they decrease when adding NC. The SEM micrographs show that adding nano particles improved the mixture's microstructure by filling the voids in the cement matrix. The mixture with nano clay was more economic to be used than nano silica because its price was much cheaper.

Keywords: Nano Silica, nano clay, hybrid nano materials

1 INTRODUCTION

Previous researches concerning the effect of hybrid nano materials on the drying shrinkage of cement pastes, mortars and concretes are so limited, as most of the following previous researches deals mainly with the dispersion issue and the influence of hybrid nano materials, NS and NC on the properties of fresh and hardened states.

M.A. Mahdi [1] studied the Effect of Using Nano Materials on the Properties of Cement Paste and Mortar, He found that the compressive strength improved in the mixtures in the cured condition, the optimum percentages was 1% for nano silica, 5% for nano clay, and 5% (0.5%NS&4.5%NC) for hybrid nano particles. The drying shrinkage and thermal conductivity increase with adding NS and Hybrid, while they decrease when adding NC.

A.M. Mohamed [2] studied the Influence of Nano Materials on Compressive Strength of Concrete; he added nano silica, nano clay or both together with different percentages to the mixes. He found that, NS is more effective than NC and wet mix gives higher efficiency than dry mix. Adding NS and NC together in the mix had a remarkable improvement appearing in concrete compressive strength than using the same percentage of one type. The optimum percentages were 3% nano particles consisting of 25% NS and 75% NC for compressive strength.

I. Sobhy [3] studied the effect of using nano silica, nano clay and composite of them on the performance of concrete mixes

and found that using nano particles improves the mechanical properties of concrete mixes. The optimum percentage of NS and NC was 5%, and for hybrid was 2.5%NS & 2.5%NC. Dry mix gives the best dispersion of nano particles and less weak points of the inert materials in the mix compared to wet mix.

M.S. Morsy, et al [4] studied the Hybrid Effect of Carbon Nanotube and Nano-Clay on Physico-Mechanical Properties of Cement Mortar; they found that adding 6% NC in cement mortar increases compressive strength by 18%. The addition of CNTs (up to 0.02%) to NC cement mortar improves the compressive strength of the composites with 11% higher than mix containing 6% NC, while the addition of CNTs by 0.1% decreased the compressive strength.

H. Yang [5] studied the bending tensile Strength and Shrinkage Property of Nano Silica Powder Concrete; He found that when proper content of NS is added to concrete, bending tensile strength can be improved but this leads to higher shrinkage rate, easily producing early crack.

A. Sadrmomtazi, et al [6] studied the effect of Polypropylene fibers on the mechanical and physical properties of mortars containing nano-SiO₂. Compressive, flexural strength and shrinkage were measured. They found that adding NS to fiber reinforced cement composites improved the mechanical characteristics of mortars, but increases shrinkage rate.

Tobón J.I, et al [7] Analyzed the Performance of Portland Cement Blended with Nano Silica and Silica Fume, measuring the physical properties cement type III replaced with 1, 3, 5 and 10% NS and silica fume in percentages of 5, 10 and 15%. They found that 5% NS improves in compressive strength with 10% after 3 days of curing, while 5% and 10% NS improve in compressive strength with 10% and 80% respectively after 28 days of curing.

• Authors:

(a) Assistant Lecturer in Faculty of Engineering, Misr University for Science and Technology, Giza, Egypt.

(b) Professor in Housing and Building National Research Center HBRC, Cairo, Egypt.

(c) Professor in Faculty of Engineering Cairo University, Cairo, Egypt.

• Co-Authour Email: mostafa.senousy@must.edu.eg

J.S. Belkowitz1, et al [8] investigated Nano Silica in the Cement Hydration Process determining the compressive strength. They found that different sizes of silica improve some properties. When the silica particle size increases, the rate of early pozzolanic reaction decreases. Both nano and micron size silica particles were added at the same concentration, but the nano silica was more effective.

A. Sadrumontazi, et al [9] studied the effect of adding nano-SiO₂ and silica fume (SF) to cement mortar. They found that the optimum nano-SiO₂ percentage ranges between 5% and 7%. Adding nano-SiO₂ increased drying shrinkage and decreased the water absorption of cement composites by the pozzolanic effects and filling the pores, this led to reduction of permeability more than when SF is used.

L. Senff, et al [10] studied adding Amorphous nano-silica particles (0–2.5 wt%) to cement pastes and mortars. They found that NS modified the characteristics of fresh mortars. The presence of NS decreased the amount of lubricating water available in the mixture and yield stress increases considerably.

A. Hakamy, et al [11] investigated the Characteristics of Hemp Fabric Reinforced Nano clay-Cement Nano composites; they found that, the optimum content of NC was 1%. The HF-reinforced nano composites containing 1wt% NC decreased the porosity (15.5%) and also increased the density (5.3%), flexural strength (26.2%) and fracture toughness (24.9%). Adding more than 1% NC to the HF-reinforced cement composites affects negatively the fracture toughness and the flexural strength.

K. Patel [12] studied the use of Nano clay aiming to constitute blended cement mortar with fractional increase in mechanical strength. They found the compressive strength was improved by 300% with 1% NC and by 290% with 2% NC for seven day testing. At the age of 28 day the improvement reached 310% with 1% NC and 200% with 2% NC.

M.S. Morsy, et al [13] studied the effect of Nano-clay on mechanical properties and microstructure of ordinary Portland cement mortar. NC was added with percentages of 0, 2, 4, 6 and 8% by weight of cement. The samples were first cured at 100% relative humidity for 24 hours and then cured in water for 28 days. The enhancement in compressive was 7% at 8% NC.

The purpose of this research is to study the effect of adding nano silica, nano clay and hybrid nano particles with different percentages to the mortar to study their effect on compressive strength and drying shrinkage under different curing conditions. Scanning Electron Microscope and thermal conductivity tests were also performed on the optimum samples.

2 EXPERIMENTAL WORK

In this work the effect of adding different percentages of nano materials on the properties of mortar was examined under different curing conditions.

2.1 Materials

2.1.1 Cement

Ordinary Portland cement (OPC) (CEM1 52.5 N) was used during the study, obtained from Beni-Suef Cement Factory in Egypt. The chemical analysis of the cement is shown in Table 1.

2.1.2 Chemical Admixture

High performance superplasticizer (S.P) (Glenium ACE 30) was used which is an aqueous solution of modified Polycarboxylates, obtained from BASF Chemicals Company in Egypt.

2.1.3 Nano Powders

Nano powders are two nano silica types (NS1 & NS2) and nano clay.

2.1.3.1 Nano Silica

The first type one of nano silica (NS1) is produced locally in nano materials laboratory on Beni-Suef University; it is characterized by 38 nm of the mean particle size and 110 m²/g of surface area, it consists of pure silica. Fig. 1 shows the Transmission Electron Microscope (TEM) micrograph of NS1 with magnification 200 kx.

The second type two of nano silica (NS2) is imported from Sigma-Aldrich Company in Germany; it is characterized by 24 nm of the mean particle size and 220 m²/g of surface area, it consists of pure silica. Fig. 2 shows the TEM micrograph of NS2 with magnification 150 kx.

2.1.3.2 Nano Clay

Nano clay (NC) is montmorillonite clay (OH₄ Si₈ AL₄ O₂₀n) was obtained from Middle East Mining Investments Company in Egypt (MEMCO) modified with quaternary ammonium salts (sodium calcium aluminum silicate) that is in crystalline state which is characterized by large length according to its thickness ratio. The nano clay used in this research was in an amorphous state. Thermal activation which was performed converted the nano clay crystalline state to amorphous state; it reduced the grain size of nano clay. The clay was exposed to 800 °c temperature for two hours. The chemical and physical properties of NC are shown in Table 1 & 2 respectively. Fig. 3 shows the TEM micrograph of NC with magnification 300 kx.

2.2 Mix design and preparation of specimens

Standard mortar was prepared according to ES (2421-2006) [14] to determine the standard water - cement ratio, which was found in this study 39.8% for all mixes with S.P and 49.7% for mix M1 without S.P. superplasticizer changed from (2 to

2.5) % of cement to maintain the same water-cement ratio for all mixes.

2.2.1 Mix design

Mortars were prepared, mixed, casted, cured and tested in the Material Laboratory in the Faculty of Engineering, Cairo University, nine mortar mixes were designed and shown in table 3, from which it can be noticed that mix M1 and M2 were control mixes with and without S.P, while mixes M3, M4 and M5 were with different percentages of nano silica (1%, 3%), mixes M6 and M7 were with different percentages of nano clay (5%, 7%), mixes M8 and M9 were with different percentages of hybrid nano materials (5%, 6%) as weight percentage of cement content.

2.2.2 Dispersion techniques of nano particles

To achieve perfect dispersion of nano particles to enhance the base matrix, two dispersion techniques are used in this study: a- Sonication, b- Stirring by vane motor.

2.2.2.1 Sonication

Water bath sonicator shown in Fig. 4 has been used. Nano particles were mixed with half amount of the water and superplasticizer.

This technique was applied with nano silica and the period of sonication was 5 minutes, while it was 10 minutes when nano clay was used, the temperature was 40° c. Table 4 shows the specifications of water bath sonicator.

2.2.2.2 Stirring by vane motor

The second technique of dispersion (Nano particles mixed with half amount of the water and super plasticizer) was dispersed by the vane motor shown in Fig. 5; the period was 2 minutes, until the mixture was homogeneous. Table 5 shows the specifications of vane motor.

2.2.3 Mixing Procedure

The cement and fine aggregate were dry mixed in a rotary mixer for thirty seconds; half amount of the water and SP needed for the mixture were added to the previous blend and were mixed thirty seconds. The ready mixed liquid (The other half of the water and SP amount and the nano particles) that resulted from the nano mixing technique was added gradually and mixed for additional three minutes.

2.2.4 Casting and Curing Procedure

The mixtures were casted in steel mold contains 3 cube of dimensions (50x50x50)mm for compressive strength, and a mold contain 5 prisms of dimensions (25x25x285)mm was used for shrinkage measurements.

After one day from casting, samples have been demolded. There were two curing systems carried out: a- cured in tap water, b- uncured in open air. All these samples were tested after 3, 7 and 28 days.

2.3 Testing Procedures

2.3.1 Compressive Strength

The compressive strength of mortar cubes was determined using SHIMADZU 500 KN Universal machine in Material laboratory in the Faculty of Engineering, Cairo University.

2.3.2 Drying Shrinkage

Drying shrinkage has been calculated according to [15], [16], using the samples demolded from the prisms by measuring the change in their length using the Micrometer. The samples extracted from the prisms were 25x25x285 mm with two nails fixed at their two ends; the length between their internal edges was 250 ± 2.5 mm. After demolding the samples, the change in their length from the two edges of the nails has been measured (L_0). Then cured samples have been submerged in tap water for only forty eight hours, while uncured samples were left in the open air. After lifting the cured samples from the water, they have been left in the open air till the date of testing. Change of their length has been measured at ages: 3, 7, 14, 21 and 28 from the age of casting (L_f). Ages of measuring have been the same for the uncured samples (L_f).

The percentage of linear drying shrinkage ($S\%$) was measured for each sample (cured and uncured) at each age as follows:

$$S\% = \frac{L_0 - L_f}{L} \times 100\% \quad [15]$$

Where,

- $S\%$: Drying shrinkage strain.
- L : The effective length of the testing samples which is 250mm according to [15].
- L_0 : The length between the two nails edges after 24 hours.
- L_f : The length between the two nails edges at each age.

At each age, the average of the drying shrinkage percentage ($S\%$) has been calculated for the six (cured and uncured) samples, in addition to measuring humidity and temperature.

2.3.3 Thermal Conductivity

The thermal conductivity (K -value) has been operated in the physics laboratory of the Housing and Building Research Centre and was calculated using one sample 50x50x50 mm at age 28 days from the optimum mortar mixtures. For this purpose, an insulating box (shown in Fig. 6) has been used in addition to a Scanning thermometer of 12 channels (shown in Fig. 7) which used a software program to read and store data, Digi-Sense Cole, Parmer Instrument Company, Model 92000-00 by Barnant Company Barruington, USA.

The insulating box characterized by a small window with a small piece of glass fixed to it to collect the heat emitted from the sun to reach one side of the sample, then, this heat passes through the opposite side of it.

K -value can be measured through the following equation:

$$K = q \frac{\Delta x}{\Delta t} \quad (\text{watt/m.k}) \quad [17]$$

Where,

- K : Thermal conductivity. (watt/m.k)
- q : Heat transferred per unit area. (watt/ m^2)
- Δx : The thickness of the sample = 0.05m.
- Δt : The temperature difference. (Kelvin)

A Heat Flow Sensor (CE Concept Engineering, USA) is used to measure q by milli volte units, and then it is converted to watt/ m^2 by multiplying to a factor attached to the sensor

and thermocouple is used to measure Δt .

Thermal resistance (R-value)

Thermal resistance is the ability of a material to resist the flow of heat.

$$R = \frac{\Delta x}{K} \quad (\text{m}^2 \cdot \text{K} / \text{watt}) \quad [17]$$

Thermal transmittance (U-value)

The reciprocal of thermal resistance, this measures the amount of heat transmitted per unit area of a particular thickness per unit temperature difference between inside and outside environments.

U-value calculation formula:

$$U = \frac{1}{RSI + RSO + R} \quad (\text{watt} / \text{m}^2 \cdot \text{K}) \quad [17]$$

Where,

- RSI: Thermal resistance of internal surface= 0.123 (m².K/watt)
- RSO: Thermal resistance of external surface=0.055 (m².K/watt)
- R: Thermal resistance of the material.

The definitions and the procedures of thermal conductivity test were taken from [17].

Table 1. Chemical analysis of materials

| % | CEM I 52.5 | NS1 | NS2 | NC |
|--------------------------------|------------|------|------|-------|
| SiO ₂ | 19.8 | 99.1 | 99.1 | 61.24 |
| Al ₂ O ₃ | 5.5 | --- | --- | 20.89 |
| CaO | 63 | --- | --- | 0.16 |
| MgO | 1.2 | --- | --- | 0.22 |
| Fe ₂ O ₃ | 3.4 | --- | --- | 1.06 |
| Na ₂ O | 0.64 | --- | --- | 0.71 |
| K ₂ O | 0.19 | --- | --- | 1.61 |
| SO ₃ | 3 | --- | --- | 0.17 |
| L.O.I | 2.5 | --- | --- | 13.12 |

Table 2. physical properties of nano particles

| Nano Type | Colour | Bulk Density (g/cm ³) | Mean Particle Size (nm) | Surface Area (m ² /g) |
|-----------|-------------|-----------------------------------|-------------------------|----------------------------------|
| NS1 | White | 0.24 | 38 | 110 |
| NS2 | White | 0.16 | 24 | 220 |
| NC | Light Cream | 0.19 | 25 | 330 |

Table 3. Mixing proportions of mortar mixtures with and without nano particles

| Mix | Mix type | Nano Dispersion Technique | Cement (Kg/m ³) | Sand (Kg/m ³) | Water (cm ³ /m ³) | S.P (cm ³ /m ³) | Nano weight (Kg/m ³) | W/b |
|-----|----------|---------------------------|-----------------------------|---------------------------|--|--|----------------------------------|-----|
| | | | | | | | | |

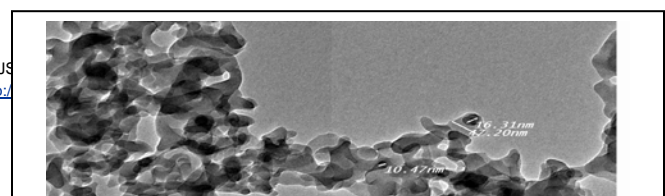
USER

Table 4. Specifications of water bath sonicator

| | |
|--------------------------|------|
| Volume (L) | 6 |
| Power supply (V) | 220 |
| Frequency (Hz) | 50 |
| Power consumption (watt) | 200 |
| Timer (Minute) | 1-30 |

Table 5. Specifications of vane motor

| | |
|--------------------------|-----------|
| Speed range (rpm) | 1500-2800 |
| Frequency (Hz) | 50 |
| Power consumption (watt) | 600 |



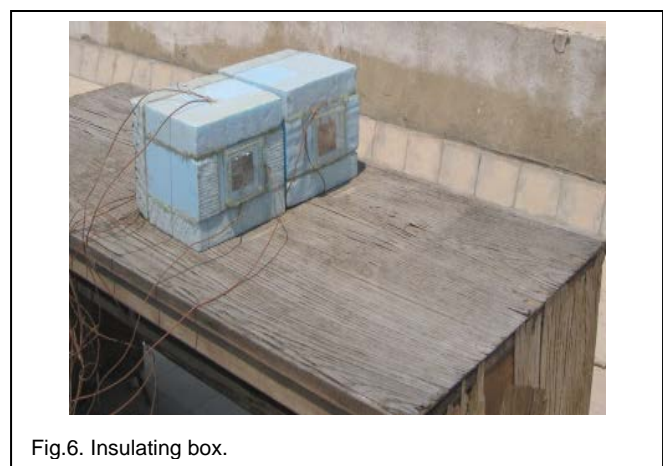
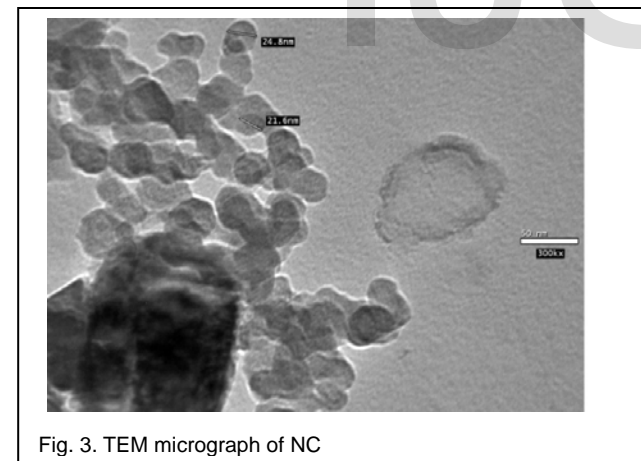
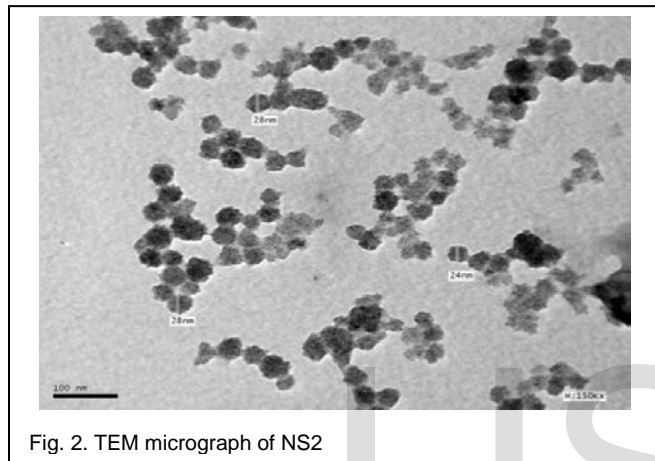




Fig.7. Scanning thermometer.

3 ANALYSIS AND DISCUSSION OF TEST RESULTS

3.1 Compressive strength

Table 6 shows the compressive strength test results of mortar mixtures with and without nano particles at the ages of 7, 14 and 28 days for both cured and uncured mixtures.

3.1.1 The effect of addition superplasticizer (S.P) on compressive strength

Fig. 8 shows that when S.P was added to the mortar, the enhancement in the workability and compressive strength at all ages was clear in the cured and uncured samples compared to the mixtures without S.P. this can be attributed to the reduction in water cement ratio. The percent of improvement in compressive strength for control cured and uncured mortar mixture with S.P are 17.86% and 17.63% respectively at 28 days compared to control mixes.

3.1.2 The effect of adding NS1 and NS2 on compressive strength

Fig. 9 shows the development of compressive strength for control mortar mixes with S.P and mixes with different percentages of nano silica, it can be seen that the addition of 1%NS1- locally produced - using stirring dispersion technique to mixes increased the compressive strength compared to control ones for cured samples at all ages, the improved percentages of compressive strength reached 16.6%, 7.1%, and 17.5% at 7, 14, and 28 days respectively, while the uncured samples with 1% NS1 showed a decrease in the compressive strength compared to the uncured control mixture with S.P.

The increase in the compressive strength can be attributed to the efficiency of nano particles in promoting pozzolanic reaction, packing the voids and consuming calcium hydroxide crystals forming additional calcium silicate hydrate (C-S-H).

When the NS1 percentage was increased to 3%, using stirring dispersion technique, there was an increase in the compressive strength at the ages 7 and 14 days, while it was decreased slightly at the age 28 in the cured and the uncured conditions compared to the control mixture.

This means, there is a certain limit after which any increase in the NS percentage leads to a decrease in the compressive strength, this can be attributed to the agglomeration caused by

the nano silica particles when adding excessive amount as the

Table 6. Compressive strength of mortar samples with and without nano particles

| Mix | Mix Description | Nano Mixing Procedure | Compressive Strength (MPa) | | | Curing Conditions |
|-----|--------------------|--------------------------------------|----------------------------|---------|---------|-------------------|
| | | | 7 days | 14 days | 28 days | |
| M1 | Control without SP | --- | 28.96 | 33.05 | 37.8 | Cured |
| | | | 25.58 | 28.43 | 34.43 | Uncured |
| M2 | Control with SP | --- | 29.7 | 38.18 | 44.55 | Cured |
| | | | 28.38 | 34.1 | 40.5 | Uncured |
| M3 | 1% NS1 | Stirring | 34.63 | 40.88 | 52.34 | Cured |
| | | | 28.49 | 34.06 | 39.02 | Uncured |
| M4 | 3% NS1 | Stirring | 34.22 | 40.5 | 43.63 | Cured |
| | | | 30.33 | 38.88 | 39.83 | Uncured |
| M5 | 1% NS2 | Sonication | 35.1 | 47.74 | 59.6 | Cured |
| | | | 33.08 | 45.36 | 47.25 | Uncured |
| M6 | 5% NC | Sonication | 31.41 | 41.69 | 52.7 | Cured |
| | | | 29.43 | 31.05 | 35.32 | Uncured |
| M7 | 7% NC | Sonication | 31.62 | 36.83 | 37.31 | Cured |
| | | | 29.89 | 33.59 | 33.75 | Uncured |
| M8 | 5% Hybrid | Stirring for NS1 & Sonication for NC | 33.75 | 44.55 | 58.16 | Cured |
| | | | 30.24 | 35.78 | 41.49 | Uncured |
| M9 | 6% Hybrid | Stirring for NS1 & Sonication for NC | 32.13 | 38.34 | 44.69 | Cured |
| | | | 25.92 | 30.05 | 38.39 | Uncured |

high surface area increases attracting these particles to each other forming weak clogs, these clogs fill the voids of the mortar mixture preventing the filling effect of nano particles decreasing the mixture strength. The other possible explanation is that the mixing water is not enough to coat this large percentage of the nano silica particles causing defects in the hydration process decreasing compressive strength, thus mixing with 3%NS1 needs more percentage of S.P compared to control mixture in order to enhance workability.

As a result of adding 1% NS1 to mixture which is the optimum percentage, this percentage has been applied for NS2 particles -imported- to mixture using sonication dispersion technique. Fig. 9 also shows that the addition of 1%NS2 to mixes increased the compressive strength compared to control ones for cured and uncured samples at all ages.

It can be concluded that the best percentage of NS1 and NS2 is 1%, using stirring and sonication dispersion techniques respectively for cured samples, while the uncured samples of 1%NS2 is higher compared to the uncured and cured control mixtures.

3.1.3 The effect of adding NC on compressive strength

Fig. 10 shows the development of compressive strength for control mortar mixes with S.P and mixes with different percentages of nano clay, it can be seen that the addition of 5%NC using sonication dispersion technique to mixes increased the compressive strength compared to control ones for cured samples at all ages, the improved percentages of compressive strength reached 5.8%, 9.2%, and 18.3% at 7, 14, and 28 days

respectively, While the uncured samples with 5% NC showed a decrease in the compressive strength compared to the uncured control mixture.

The increase in the compressive strength can be attributed to a- the efficiency of nano particles in promoting pozzolanic reaction, packing the voids and consuming calcium hydroxide forming additional calcium silicate hydrate (C-S-H) b- the effect of nano clay which behaves as a filler that improves the microstructure, and as a self curing action when it is mixed into cement, and after it is hardened, nano clay will slowly release this water to an unhydrated cement during the critical early conditions of curing, assisting in more complete hydration of cement.

Fig. 10 also shows that, by raising the NC percentage to reach 7% using sonication dispersion technique, there was an increase in the compressive strength at the age 7, while it was decreased at the ages 14 and 28 in the cured and the uncured conditions compared to the control mixture; this is due to the same reasons mentioned before when adding excess amounts of NS1 mixtures.

3.1.4 The effect of adding Hybrid nano materials on compressive strength

Fig. 11 shows the development of compressive strength for control mortar mixes with S.P and mixes with different percentages of hybrid nano materials, using suitable dispersion technique for each nano particle, it can be seen that the addition of 5% Hybrid to mixes increased the compressive strength compared to control ones for cured samples at all ages, the improved percentages of compressive strength reached 13.64%, 16.7%, and 30.55% at 7, 14, and 28 days respectively, while the uncured samples with 5% Hybrid showed a slight increase in the compressive strength compared to the uncured control mixture.

The increase in the compressive strength can be attributed to a- the efficiency of nano silica and clay in promoting pozzolanic reaction, packing the voids and consuming calcium hydroxide forming additional calcium silicate hydrate (C-S-H) b- the effect of nano clay alone behaves as a filler that improves the microstructure, and as a self curing action when it is mixed into cement, and after it is hardened, nano clay will slowly release this water to an unhydrated cement during the critical early conditions of curing, assisting in more complete hydration of cement.

Fig. 11 also shows that, when the hybrid nano materials percentage was increased to 6%, there was a decrease in the compressive strength at all ages for the cured and uncured samples compared to the control mixture; this is due to the same reasons mentioned before when adding excess amounts of NS1 mixtures.

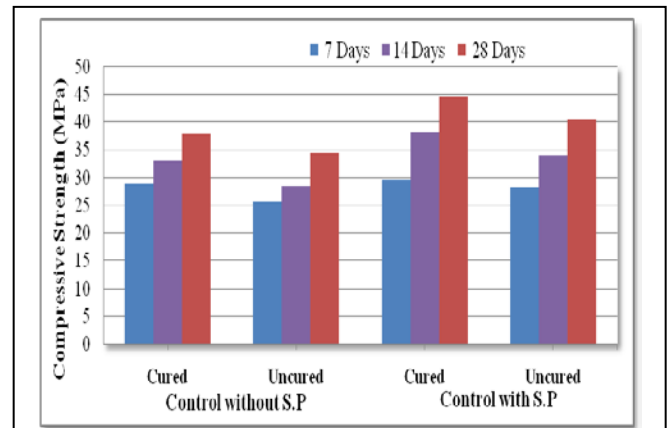


Fig.8. Development of compressive strength for control mortar mixes with and without S.P at different ages.

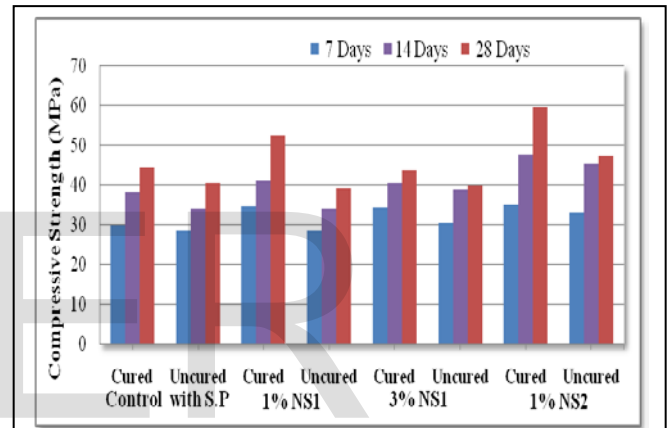


Fig.9. Development of compressive strength for control mortar mixes with S.P and mixes with different percentages of nano silica.

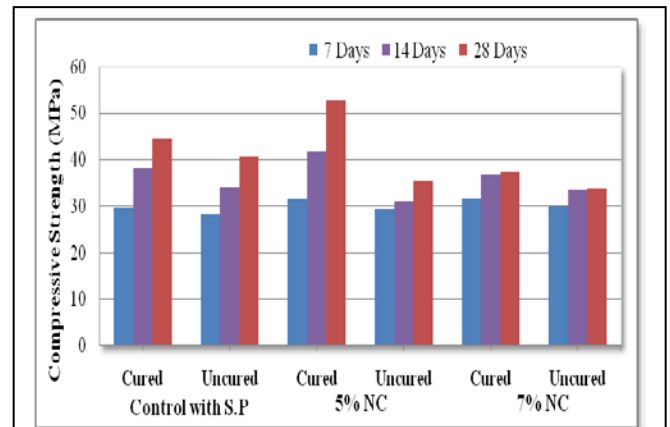
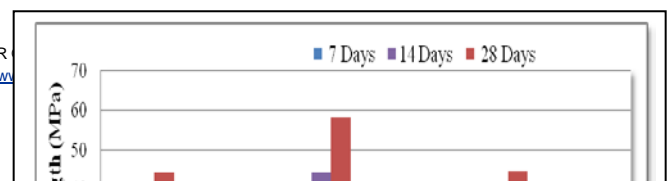


Fig.10. Development of compressive strength for control mortar mixes with S.P and mixes with different percentages of nano clay.



3.2 Drying Shrinkage

Table 7 shows the drying shrinkage test results of mortar mixtures with and without nano particles at the ages of 3, 7, 14, 21 and 28 days for both cured and uncured mixtures.

In the procedures of measuring drying shrinkage, temperature and humidity were measured at each age as shown in Fig. 12 and 13 respectively.

Table 7. Drying shrinkage of mortar samples with and without nano particles

| Mix | Mix Description | Nano Mixing Procedure | Drying Shrinkage (%) | | | | | Curing Conditions |
|-----|--------------------|--------------------------------------|----------------------|--------|---------|---------|---------|-------------------|
| | | | 3 days | 7 days | 14 days | 21 days | 28 days | |
| M1 | Control without SP | --- | -0.131 | 0.0314 | 0.0830 | 0.0843 | 0.0875 | Cured |
| | | | 0.0392 | 0.0588 | 0.0892 | 0.0931 | 0.0941 | Uncured |
| M2 | Control with SP | --- | -0.245 | -0.049 | 0.0333 | 0.0373 | 0.0412 | Cured |
| | | | 0.0108 | 0.0333 | 0.0510 | 0.0510 | 0.0510 | Uncured |
| M3 | 1% NS1 | Stirring | -0.278 | 0.0252 | 0.0412 | 0.0513 | 0.0578 | Cured |
| | | | 0.0235 | 0.0544 | 0.0583 | 0.0676 | 0.0686 | Uncured |
| M4 | 3% NS1 | Stirring | -0.243 | 0.0208 | 0.0478 | 0.0628 | 0.0690 | Cured |
| | | | 0.0259 | 0.0561 | 0.0686 | 0.0769 | 0.0784 | Uncured |
| M5 | 1% NS2 | Sonication | -0.184 | -0.004 | 0.0424 | 0.0526 | 0.0545 | Cured |
| | | | 0.0260 | 0.0358 | 0.0500 | 0.0578 | 0.0578 | Uncured |
| M6 | 5% NC | Sonication | -0.167 | 0.0020 | 0.0265 | 0.0353 | 0.0353 | Cured |
| | | | 0.0052 | 0.0157 | 0.0328 | 0.0373 | 0.0373 | Uncured |
| M7 | 7% NC | Sonication | -0.144 | 0.0020 | 0.0319 | 0.0402 | 0.0402 | Cured |
| | | | 0.0065 | 0.0183 | 0.0353 | 0.0412 | 0.0412 | Uncured |
| M8 | 5% Hybrid | Stirring for NS1 & Sonication for NC | -0.196 | 0.0039 | 0.0373 | 0.0451 | 0.0510 | Cured |
| | | | 0.0196 | 0.0373 | 0.0529 | 0.0549 | 0.0608 | Uncured |
| M9 | 6% Hybrid | Stirring for NS1 & Sonication for NC | -0.157 | 0.0157 | 0.0386 | 0.0471 | 0.0539 | Cured |
| | | | 0.0216 | 0.0392 | 0.0549 | 0.0608 | 0.0647 | Uncured |

3.2.1 The effect of addition superplasticizer (S.P) on drying shrinkage

Fig. 14 shows that, when S.P was added to the mortar, as a result of decreasing the water cement ratio, the compressive strength of the mixture was enhanced and the drying shrinkage decreased at all ages in the cured and uncured conditions compared to the mixtures without S.P. The percent of decreasing in drying shrinkage for control cured and uncured mortar mixture with S.P are 52.92% and 45.84% respectively at 28 days.

3.2.2 The effect of adding NS1 and NS2 on drying shrinkage

Fig. 15 shows the development of drying shrinkage for control mortar mixes with S.P and mixes with different percentages of nano silica, it can be seen that the addition of 1%NS1 using stirring dispersion technique to mixes increased the drying shrinkage compared to control ones for cured and uncured samples at all ages. Thus, hydration of cement accelerated for nano silica which acts as an activator, so the shrinkage increased.

When raising the NS1 percentage to reach 3%, using stirring dispersion technique, there was an increase in the drying shrinkage for cured and uncured samples at all ages compared to mixes with 1% NS1 using stirring method. This can be attributed to that the rate of hydration is directly proportional to the percentage of nano silica, the heat emitted from hydration increases the evaporation leading to higher rates of drying shrinkage.

Fig. 15 also shows that the addition of 1%NS2 using sonication dispersion technique to mixes increased the drying shrinkage compared to control ones for cured and uncured samples at all ages; this is due to the same reason mentioned before in 1% NS1 mixes.

3.2.3 The effect of adding NC on drying shrinkage

Fig. 16 shows the development of drying shrinkage for control mortar mixes with S.P and mixes with different percentages of nano clay, it can be seen that the addition of 5%NC using sonication dispersion technique to mixes decreased the drying shrinkage compared to control ones for cured and uncured samples at all ages.

This is can be attributed to the effect of nano clay which behaves as a filler that improves the microstructure, and as a self curing action when it is mixed into cement, and after it is hardened, nano clay will slowly release this water to an unhydrated cement during the critical early conditions of curing, assisting in more complete hydration of cement which contributes to decreasing drying shrinkage.

When NC was increased to 7% using sonication dispersion technique, there was a decrease in the drying shrinkage for cured and uncured samples at all ages compared to control mixtures and an increase compared to mixes with 5% NC.

When adding excessive amount of NC as the high surface area increases, this attracts these particles to each other forming weak clogs. These clogs fill the voids of the mortar mixture preventing the filling effect of nano particles leading to less decreasing in shrinkage.

3.2.4 The effect of adding Hybrid nano materials on drying shrinkage

Fig. 17 shows the development of drying shrinkage for control mortar mixes with S.P and mixes with different percentages of Hybrid nano materials using suitable dispersion technique for each nano particle, it can be seen that the addition of 5% Hybrid nano materials to mixes increased the drying shrinkage compared to control ones for cured and uncured samples at all ages.

When the Hybrid nano materials percentage was increased to 6%, there was an increase in the drying shrinkage for cured and uncured samples at all ages compared to mixes with 5% Hybrid.

This can be attributed to that the increase in drying shrinkage of NS1 overcomes the decrease in drying shrinkage of NC in hybrid mixtures, but hybrid mixtures were not higher than NS1 mixtures in shrinkage results.

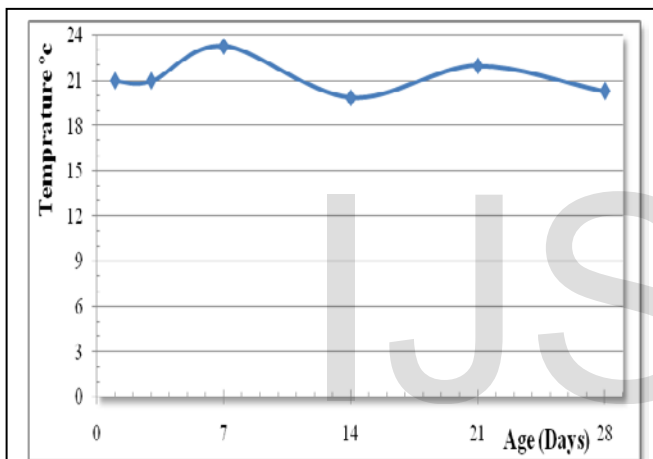


Fig.12. Variation of temperature during the age of testing drying shrinkage.

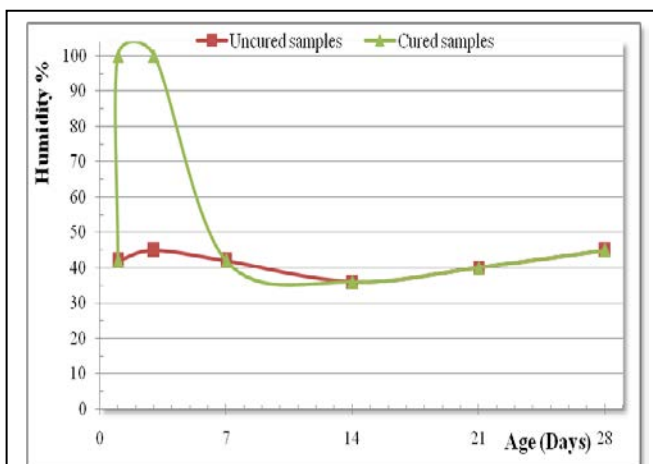


Fig.13. Variation of humidity during the age of testing drying shrinkage for cured and uncured samples.

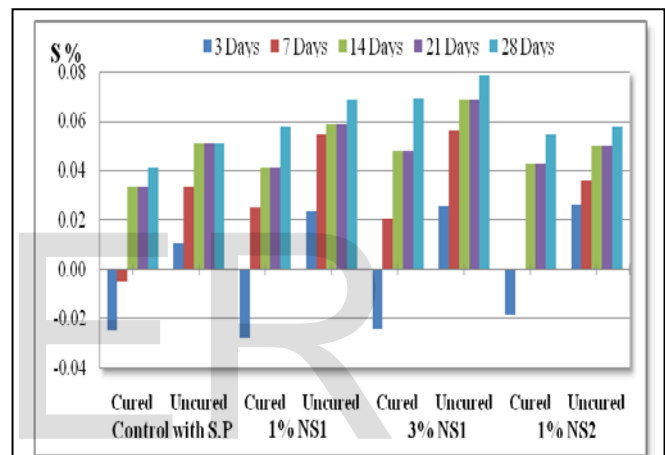


Fig.15 Development of drying shrinkage for control mortar mixes with and S.P and mixes with different percentages of nano silica.

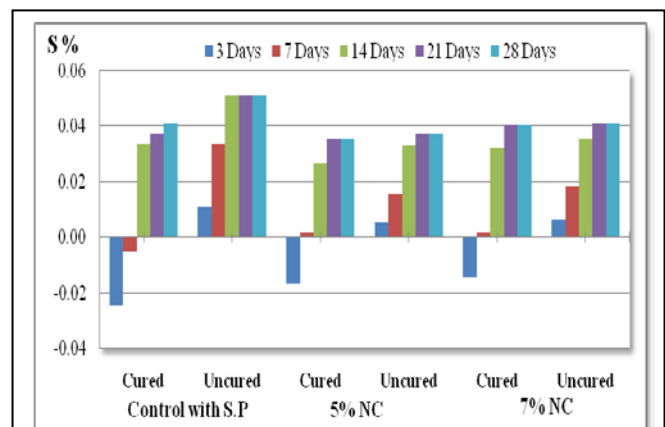
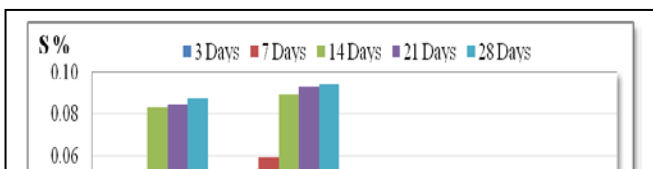
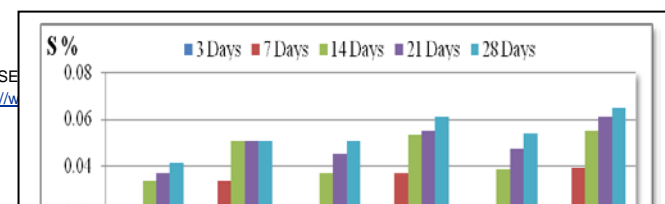


Fig.16. Development of drying shrinkage for control mortar mixes with and S.P and mixes with different percentages of nano clay.



IJSE
<http://www.ijse.in>



3.3 Effect of Dispersion Techniques

Sonication and stirring were the two dispersion techniques that had been used to reach well dispersion for nano particles in this study. From compressive strength test results, sonication was the efficient technique when the nano particle size is less than 30 nm and its bulk density is less than 0.2 g/cm³ as in the imported nano silica (NS2) and nano clay (NC). Stirring was the suitable technique when the physical properties changed by increasing the particle size more than 30 nm and bulk density more than 0.2 g/cm³ of nano material as in the local nano silica (NS1). In all cases the particle size did not exceed 100 nm.

3.4 Effect of Curing Conditions

From compressive strength results, cured condition was effective with all types of used nano particles on mixes which enhanced the rate of hydration. Uncured condition was only effective with imported nano silica (NS2) compared to the cured and uncured control mixture as presented in Fig. 9. This can be attributed to the small particle size of NS2 which fills the voids that enables the mix to maintain some of the mixing water. The reason of studying the uncured condition is to evaluate its acceptability when using nano materials.

3.5 Thermal Conductivity

Thermal conductivity was carried out on the cured mortar cubes after 28 days. Fig. 18 shows the difference of thermal conductivity (K-value) for control mixes with S.P and mixes with nano particles, it can be seen that the addition of 1%NS1 to mortar mixes increased the thermal conductivity compared to control ones. When raising the NS1 percentage to reach 3%, there was more increase in the thermal conductivity.

When 1%NS2 was added, there was an increase in the thermal conductivity compared to the control ones, but 1%NS2 mixture was not higher than 1% NS1 mixture in thermal conductivity results.

By adding 5%NC to mortar mixes, the thermal conductivity decreased compared to control ones. When raising the NC percentage to reach 7%, there was more decrease in the thermal conductivity.

This can be attributed due to that the thermal conductivity

of the cement as the physical property is lower than silica and higher than clay.

When 5% Hybrid nano materials was added to the mortar mixes, there was an increase in the thermal conductivity compared to control ones.

This can be attributed due to that the increase in thermal conductivity of NS1 overcomes the decrease in thermal conductivity of NC in hybrid mixtures, but hybrid mixtures were not higher than NS1 mixtures in thermal conductivity results.

Thermal transmittance's (U-value) calculation depends on the thermal conductivity of the sample, so the same effects occurred when nano NS1, NS2, NC, and Hybrid nano materials was added. Fig. 19 shows the difference of thermal transmittance (U-value) for control mixes with S.P and mixes with nano particles.

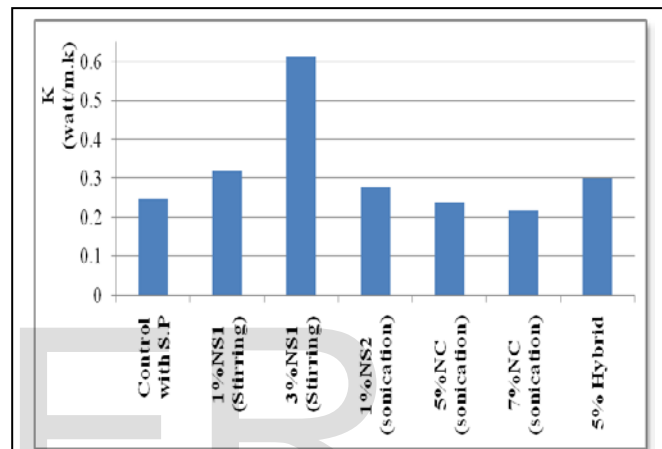


Fig.18. Difference in thermal conductivity (K-value) of mortar samples with and without nano particles.

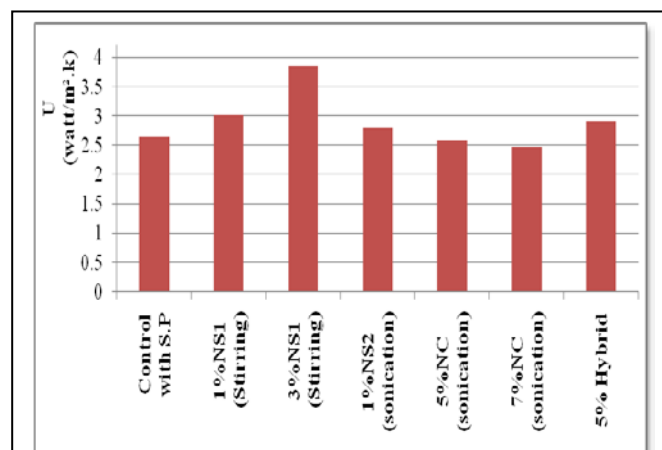


Fig.19. Difference in thermal transmittance (U-value) of mortar samples with and without nano particles.

3.6 SEM results

Scanning Electron Microscope was applied on specimens taken directly from the best mortar samples after testing compressive strength after 28 days to analyze their microstructure to compare them with the control mixture. The SEM samples were cut directly from the crushed cubes, their shape was reg-

ular and their surface was flat.

The best cured samples of cured mortar cubes at the age 28 days with and without nano particles used in SEM micrograph are as follows:

- 1- Control mixture with S.P.
- 2- 1% NS1 using stirring dispersion technique.
- 3- 1% NS2 using sonication dispersion technique.
- 4- 5% NC using sonication dispersion technique.
- 5- 5% Hybrid nano materials (0.5% NS1 & 4.5% NC).

Fig. 20 shows the microstructure of the first sample (without nano particles), many needles, calcium hydroxide crystals (CH) and large voids were clearly noticed leading to a loose structure. Calcium silicate hydrate C-S-H plates were also identifiable.

Fig. 21 shows the microstructure of the second sample (with 1% NS1). Addition of 1% NS1 resulted in filling most of the voids, reduced the needles and absorbed CH crystals. This led to an increase in the C-S-H concentration making it denser and more compact through bonding between its particles.

Fig. 22 shows the microstructure of the third sample (with 1% NS2). Addition of 1% NS2 resulted in the absence of the un-hydrated crystals and voids, the higher C-S-H concentration led to denser and more compact structure through bonding between its particles. This explains the superior compressive strength results of NS2 which were higher than NS1.

Fig. 23 shows the microstructure of the fourth sample (with 5% NC). Addition of 5% NC resulted in increasing the C-S-H concentration due to absorbing CH crystals making it denser and more compact through bonding between its particles, fewer voids were also noticed.

Fig. 24 shows the microstructure of the fifth sample (with 5% Hybrid). Addition of 5% Hybrid nano materials resulted in the absence of the un-hydrated crystals and voids, the higher C-S-H concentration led to denser and more compact structure through bonding between its particles. This explains its superior compressive strength results which were higher than NS1 and NC.

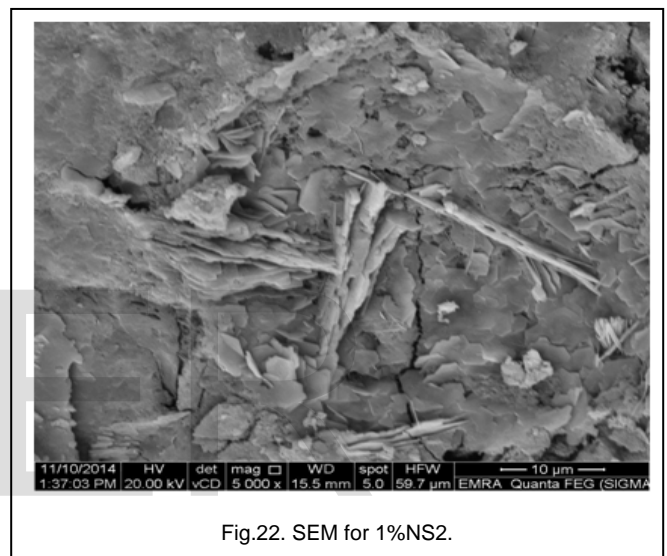


Fig.22. SEM for 1%NS2.

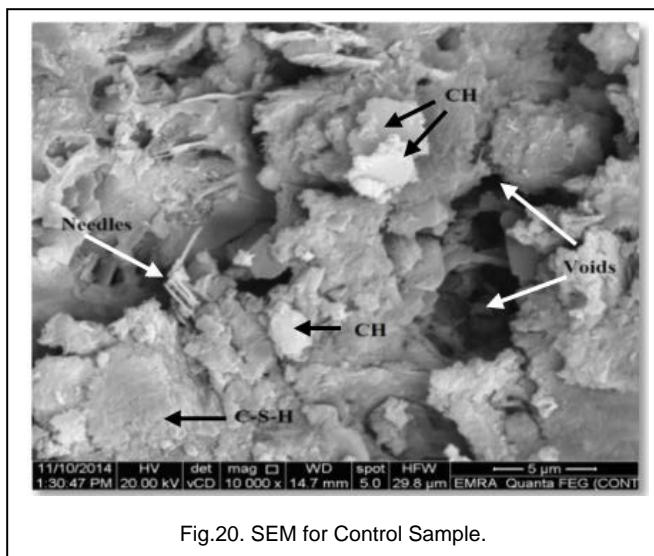


Fig.20. SEM for Control Sample.

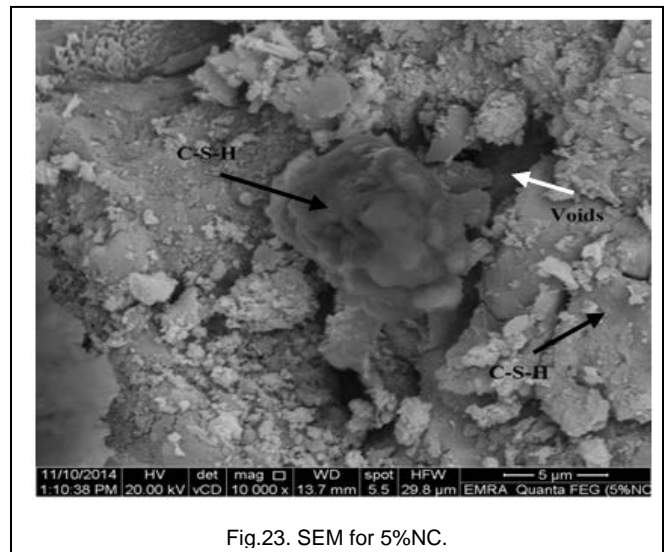
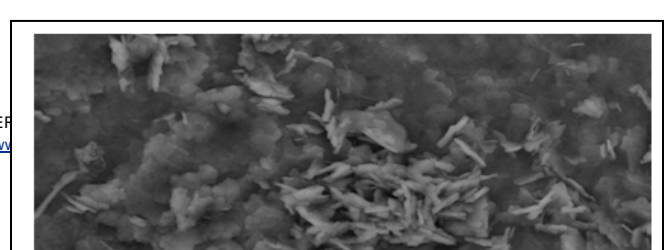
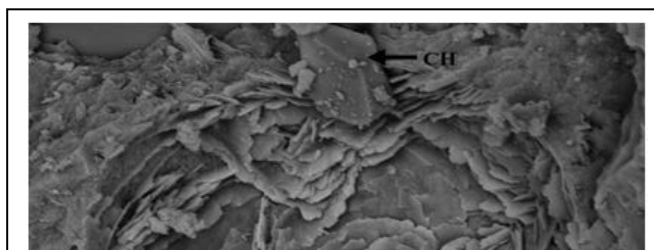


Fig.23. SEM for 5%NC.



3.7 Economic feasibility study of using nano materials in concrete construction

During this part economic assessment study for the optimum nano mortar mixes has been calculated and compared to the control mixture.

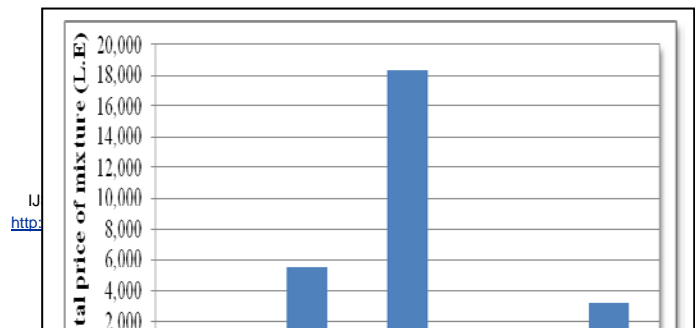
Table 8 shows the price difference between the used nano particles for 1Kg, which consequently affects on the total price. Fig. 25 shows the differences in the total price of 1m³ of the mixtures with and without nano particles.

The lowest cost was for the NC mixture followed by hybrid one. NS1 and NS2 mixtures were much higher in cost and it isn't economic to use them except for special uses. Fig. 26 shows the differences in the costs of the excess compressive strength unit.

There is another method to reduce nano silica's price significantly by producing it from silica fume using sonication and homogenizer apparatus [18].

Table 8. Economic feasibility of nano materials used in mortar mixtures

| Mix | M2 | M3 | M5 | M6 | M8 |
|-----------------------------|------------------|----------------|------------------|-----------------|-----------|
| Mix Description | Control with S.P | 1%NS1 Stirring | 1%NS2 Sonication | 5%NC Sonication | 5% Hybrid |
| Cement (Kg/m ³) | 515 | 509.85 | 509.85 | 489.25 | 489.25 |
| Price of | | | | | |



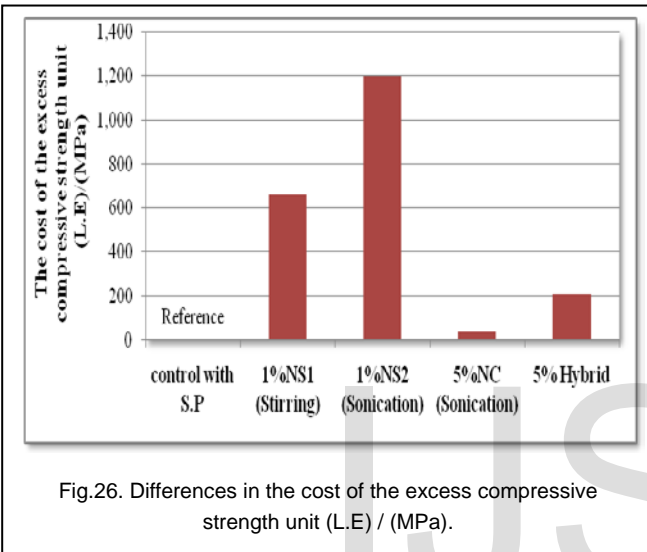


Fig.26. Differences in the cost of the excess compressive strength unit (L.E) / (MPa).

4 Conclusions

- The nano particles improve the features of the mixtures by promoting pozzolanic reaction, packing the voids, in addition to consuming calcium hydroxide crystals forming additional calcium silicate hydrate (C-S-H) which participate in increasing strength. This can only be achieved by using suitable dispersion technique that results in well dispersion.
- Nano Particles improve the mechanical properties at certain percentages. When there is an excessive amount, the particles form weak clogs as a result of the high surface area or the increase in the mixing water demand so it won't be able to enhance the hydration process forming C-S-H.
- Applying Nano particles with S.P improved the strength and shrinkage of the mixes because the nano particles interpenetrate polymer network causing the above improvements.
- Cured condition was effective with all types of used nano particles, since it enhanced the rate of hydration. While uncured condition was only effective with imported nano silica (NS2) compared to the cured and uncured control mixture because of the small nano particle size of NS2 which fills the voids that enables the mix to maintain some of the mixing water.
- The optimum percentage of NS1 in the compressive strength test was 1% using stirring dispersion technique, the improvement were 11.83% for cured mixes at 28 days compared to control cured samples.
- The optimum percentage of NS2 in the compressive strength

test was 1% using sonication dispersion technique, the improvement was 16.45% and 4% for cured and uncured mixes respectively at 28 days compared to control cured samples.

- The optimum percentage of NC in the compressive strength test was 5% using sonication dispersion technique, the improvement was 12.06% for cured mixes at 28 days compared to control cured samples.
- The optimum percentage of hybrid nano materials in the compressive strength test was 5% (0.5%NS1 & 4.5% NC) with using the suitable dispersion technique for each nano particle, the improvement was 14.7% for cured mixes at 28 days compared to control cured samples.
- The drying shrinkage decreases in the cured and uncured conditions with adding nano clay in the mortar mixtures compared to the control ones. This can be attributed to the effect of self curing action of NC when it is mixed into cement, and after it is hardened, nano clay will slowly release this water to unhydrated cement during the critical early phases of curing which assist in more complete hydration of cement which contributes to increase compressive strength and decrease drying shrinkage.
- The drying shrinkage increases in the cured and uncured conditions with adding nano silica and hybrid nano materials in mortar mixtures compared to control ones due to the acceleration of hydration of cement, since nano silica acts as an activator, so the drying shrinkage increased. In hybrid mixtures, the increase in drying shrinkage of NS1 overcomes the decrease in drying shrinkage of NC.
- The thermal conductivity increases with adding nano silica and hybrid nano materials in mortar mixtures compared to control ones, while it decreased with adding nano clay, due to the thermal conductivity of the cement as the physical property is lower than silica and higher than clay. In hybrid mixtures, the increase in thermal conductivity of NS1 overcomes the decrease in thermal conductivity of NC, but hybrid mixtures were not higher than NS1 mixtures in thermal conductivity results.
- The SEM micrograph of the control mixture shows that, needles, Ca (OH)₂ and voids were obvious, while in the best samples of NS1, NS2, NC and Hybrid micrographs, there were improvements. The best results can be shown in the NS2 and Hybrid micrographs, while there were still some needles, Ca (OH)₂ and voids that were obvious in the NS1 and NC micrographs. These micrographs came consistent with the compressive strength results.
- From the current economic assessment study of using nano materials in constructions, mortar with nano clay was economic to be used because its price was much cheaper than nano silica and not much more expensive than control mortar.

5 REFERENCES

- [1] M.A. Mahdi, "The Effect of Using Nano Materials on the Properties of Cement Paste and Mortar", M.Sc Thesis on Structural Department, Faculty of Engineering, Cairo University, Egypt, September 2015.
- [2] A.M. Mohamed, "Influence of Nano Materials on Flexural Behavior and Compressive Strength of Concrete", HBRC Journal, November

- 2014.
- [3] I. Sobhy, "Effect of Using Nano- Silica Particles on the Properties of Different Concrete Mixes", MSc. Thesis, Civil Department, Faculty of Engineering, Helwan University, Egypt 2012.
- [4] M.S. Morsy, S.H. Alsayed, M. Aqel, "Hybrid Effect of Carbon Nano-tube and Nano-Clay on Physico-Mechanical Properties of Cement Mortar", *Construction and Building Materials*, 25 (2011), PP (145-149).
- [5] H. Yang, "Strength and Shrinkage Property of Nano Silica Powder Concrete", Department of Civil Engineering, Shandong Jiaotong University, Jinan, Shandong, 250023, China, 2nd International Conference on Electronic & Mechanical Engineering and Information Technology (EMEIT-2012).
- [6] A. Sadrmomtazi and A. Fasihi, "Influence of Polypropylene Fibers on the Performance of Nano-Sio2-Incorporated Mortar", *Iranian Journal of Science & Technology, Transaction B: Engineering*, Vol. 34, No. B4, PP (385-395) Printed in The Islamic Republic of Iran, 2010 Shiraz University.
- [7] Tobón, J. I, Restrepo, O. J & Payá, J, "Comparative Analysis of Performance of Portland Cement Blended with Nanosilica and Silica Fume", *Dyna*, May 12th, 2010, 163, PP (37-46).
- [8] J.S. Belkowitz and D. Armentrout, "An Investigation of Nano Silica in the Cement Hydration Process Concrete Sustainability Conference", National Ready Mixed Concrete Association, 2012, PP (1-15).
- [9] A. Sadrmomtazi, A. Fasihi, F. Balalaei, and A.K. Haghi, "Investigation of Mechanical and Physical Properties of Mortars Containing Silica Fume and Nano-SiO₂", in the 3rd International Conference on Concrete and Development, Tehran, Iran, PP (1153-1161), April 2009.
- [10] L. Senff, JOA. Labrincha, V.M. Ferreira, D. Hotza, W.L. Repette, "Effect of Nano-Silica on Rheology and Fresh Properties of Cement Pastes and Mortars", *Construction and Building Materials* 23, 8 February 2009, PP (2487-2491).
- [11] A. Hakamy, F.U.A. Shaikh, and I.M. Low, "Characteristics of Hemp Fabric Reinforced Nanoclay-Cement Nanocomposites", *Cement & Concrete Composites*, 50 (2014), PP (27-35).
- [12] K. Patel "The Use of Nanoclay as a Constructional Material", Department Of Civil Engineering, L.D. College of Engineering Ahmedabad- Gujarat, India. *International Journal of Engineering Research and Applications*, Vol. 2, Issue 4, July-August 2012, PP (1382-1386).
- [13] M.S. Morsy, S.H. Al Sayed and M. Aqel, "Effect of Nano-clay on Mechanical Properties and Microstructure of Ordinary Portland Cement Mortar", *International Journal of Civil & Environmental Engineering IJCEE-IJENS* (2010) Vol: 10 No: 01.
- [14] ES Specification 2421-2006, "Cement-Physical and Mechanical Tests" Part 7, "Determination of Strength Prism Method".
- [15] ECP 203, "Drying Shrinkage of Cement Mortar Containing Portland cement", Egyptian Code for Design and Construction of Concrete Structures, Tests Guide, part 1 PP (62-67), HBRC, Cairo, Egypt, 2003.
- [16] ASTM C 157-04 "Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete ", ASTM inter, 2004.
- [17] Charlotte Baden-Powell, "Architect's Pocket Book", Third edition updated by Jonathan Hetreed and Ann Ross, 2008.
- [18] M.S. El- Feky, "Optimizing the Preparation and Utilization of Nano Silica to Enhance Concrete Properties and Behavior", Ph.D. Thesis on Structural Department, Faculty of Engineering, Cairo University, Egypt 2013.
- [19] S.I. Zaki, "Assessment of the Durability of Nano Silica / Fly Ash Cement Pastes Exposed to Normal and Aggressive Curing Conditions" Seventh International Conference On Nanotechnology in Construction (NTC 2015), Sharm El-Sheikh, Egypt, March 2015.
- [20] S.I. Zaki, and I. Sobhy, "The Use of Activated Nano Clay to Develop the Compressive Strength and Microstructure of High Performance Concrete" .CTU Conference, Concrete in the Low Carbon Era, the University of Dundee, UK, 9-11 July. 2012.
- [21] S.I. Zaki, K. S. Ragab and A.M. Rashad "The Use of Nano SiO₂ to Develop the Water Permeability and Microstructure of High Performance Concrete". International conference on Nano Technology for Green and Sustainable Construction 14-17, Cairo, Egypt, March 2010.
- [22] S.I. Zaki, K. Ragab, "How Nano Technology Can Change Concrete Industry". 1st International Conference On Sustainable Built Environment Infrastructures in Developing Countries, ISSN 2170-0095, Oran, Algeria, 12-14 October 2009, vol. 1, PP (407-414).