

## Synthesis and Application of Nano and Micro-silica particles to enhance the Mechanical Properties of Cement Concrete

Dalal Abdul Latif Rajab Al Balushi<sup>1</sup>, Geetha Devi<sup>2,c</sup>, Soleen Jabr Ahmed<sup>3</sup>, Nawal Said Almawali<sup>4</sup>

<sup>1,2</sup> Department of Mechanical & Industrial Engineering, Caledonian College of Engineering, Oman

<sup>3,4</sup> Department of Built & Natural Environment, Caledonian College of Engineering, Oman

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### Abstract

The purpose of the current research was to evaluate the influence of nano and micro silica on compressive strength properties of cement concrete. Silica nanoparticles of size around 350 nm are synthesized by Stober's method using Tetra Ethyl Silicate (TEOS), Ethanol and ammonia solution. The synthesized silica particles are characterized using Dynamic Light Scattering (DLS), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). Four different batches of sixteen cubes each of dimension 150 mm x 150 mm x 150 mm were prepared by mixing the cement with nano and micro silica, Ultra pozzolana, fine and coarse aggregates by incorporating 0.8% CF 615. The first batch composed of cement, fine aggregate and coarse aggregate and 0.8% CF 615 was considered as a reference batch. Same mix design of proportion 1:1.4:2.1 was followed for remaining mixes by replacing 1% of cement by nano silica, micro silica and Ultra pozzolana. The compressive strength tests of the above mixes are measured using Universal Testing Machine and the results were compared with the control mix. The results demonstrated that the sample were prepared using Ultra pozzolana with 0.8% CF 615, was found to have better strength of 37.45 N/mm<sup>2</sup> compared to other samples.

*Keywords: Compressive strength, cement concrete, nano-silica, micro-silica, scanning electron microscopy, stober's method, Universal Testing Machine.*

### 1. Introduction

Nanotechnology deals with the application of materials and structures at nano scale and has a great influence on construction field. The application of nanotechnology in building materials has received much attention due to its improved mechanical properties, durability and sustainability. The strength of cement concrete can be increased by mixing the blending material at varied proportions. A variety of blending materials are applied in construction field, of which the most widely used material to improve the performance of cement concrete are silica [1-3]. Silica nanoparticles play an important role in strengthening of concrete due to its chemical composition with the presence of calcium silicate hydrate C-S-H the primary component responsible for strength and other properties in cementitious systems, lies in the

<sup>c</sup> Corresponding Author: Geetha Devi, Email: [gdmdevi@gmail.com](mailto:gdmdevi@gmail.com)

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few nanometers range [4 - 6]. Several studies are done on the enhancement of mechanical properties such as compressive strength, durability and resistivity in cement concrete. Previous studies focused on the workability and strength of cement concrete by the partial replacement of cement with nanosilica was effective [7-11]. Commonly used admixtures in cement concreting are fly ash, silica fume, ground blast-furnace slag, and metakaolin [12-16].

The contribution of nanotechnology in enhancing the strength, durability and workability of building materials can affect the hydration kinetics of cement and addition of nano materials improve the performance of cement [17-22]. Excess dosage of cement results in increased emission of CO<sub>2</sub> which leads to greenhouse effect. In order to avoid the greenhouse effect and save the environment, a better method to reduce the cement content in concrete mixes is the use of amorphous silica in powder form with a particle size range of 0.1 to 0.5 μ. The past investigations revealed that silica fume was an excellent pozzolanic material in producing High Performance Concrete (HPC). The microstructural characteristics of mortar specimen in presence of silica nanoparticles showed improved performance. This is due to nucleation of hydration products on silica nanoparticles further promotes and accelerates cement hydration. Also addition of colloidal silica resulted in acceleration of C<sub>3</sub>S dissolution and rapid formation of C-S-H phase in cement paste [23, 24].

The partial replacement of cement in the mix by Nano and micro silica particles can reduce the consumption of cement leading to cut down the cost and decrease carbon dioxide gas emission so it is called eco-concrete. Nano silica particles can develop the mechanical and physical property of cement concrete, enabling to produce high performance concrete for extreme construction. In addition to nano silica other additives such as ultra pozz and micro silica are employed in order to compare the results.

The present study aims to investigate the effect of addition of nano and micro silica to increase the compressive strength of cement concrete and comparing with other type of pozzolanic materials.

## **2. Materials and Methods**

### **2.1 Materials**

The raw materials used are Tetraethyl Ortho silicate (TEOS) (98% purity), Ethanol and Ammonia solution, NH<sub>4</sub>OH, HCl, Ordinary Portland Cement (Fineness of 350 m<sup>2</sup>/kg), course aggregate (20 mm size) and fine (< 5 mm) aggregate, potable water, Admixtures (CF 615 and cure WH), and Ultra-pozz (Size 0.1μm to 5μm).

The equipment's used for the characterization of silica samples are Scanning Electron Microscope (SEM-Heraeus Fresco 17), Fourier Transform Infrared Spectroscopy (FTIR- Cary 600 Series), Dynamic Light Scattering (DLS- Zeta sizer Nano ZS), UTM Machine, and Micro Centrifuge.

### **2.2 Synthesis of silica nanoparticles**

Silica nano particles are prepared using stober's method by mixing 6 ml of tetra ethyl ortho silicate, 100 ml ethanol, 4 ml ammonia solution and 8 ml distilled water at room temperature. The synthesis reaction was carried out at room temperature and reaction time was kept at 8 hours. After reaction, the particle suspensions are washed three times using distilled water and centrifuged to remove any unreacted materials attached on the surface of silica particles. The resulting silica particles are dried at 423 K to remove the moisture content. The

particles are characterized using scanning electron microscope (SEM); dynamic light scattering (DLS) and Fourier transform infrared spectroscopy (FTIR).

## 2.3 Characterization techniques

### 2.3.1. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy was employed to determine the surface morphology and shape of the silica particles. The characterization was carried out at an operating voltage of 30 kV and a magnification of 5000X. The sample for SEM analysis is prepared by dropping, a known amount of silica suspension on to a glass plate and allowed to dry at room temperature (25°C). Finally, the sample was coated with a thin layer of gold to prevent charging effect.

### 2.3.2. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to find the functional groups present in the synthesized nanoparticles. The bonding between the atoms and the presence of functional group on the surface of the particles are analyzed using Fourier Transform Infrared (FTIR) Spectroscopy. The characterization was performed in powder form at room temperature.

### 2.3.3. Dynamic Light Scattering

Dynamic light scattering (DLS) is method to measure the size of Nano particles by estimating the diffusion of the particles in suspension form. The Brownian motion of particles or molecules in suspension causes laser light to be scattered at different intensities. Analysis of these intensity fluctuations yields the velocity of the Brownian motion and hence the particle size using the Stokes-Einstein relationship.

## 2.4. Preliminary Test

### 2.4.1. Sieve Analysis

Sieve analysis was used to study the particle size distribution of fine aggregate of sizes 5 mm, 2.36 mm, 1.18 mm, 600  $\mu$ m, 300  $\mu$ m, 150  $\mu$ m and coarse aggregates of sizes 20 mm, 10 mm and 5 mm. Table 1 shows the size distribution of fine aggregates.

Table 1: Size distributions of fine aggregates.

Sieve Size (mm)	Mass Retained (g)	Cumulative Mass (g)	Percentage Retained Cumulative %	Percentage Passing %
5	39.5	39.5	39.5	96.05
2.36	378.0	417.5	41.75	58.25
1.18	187.1	604.6	60.46	39.54
600	137.6	742.2	74.22	25.78
300	160.9	903.1	90.31	9.69
150	48.0	951.1	95.11	4.89
Pan	48.0	999.1	99.91	0.09

### 2.4.2. Preparation of concrete mix

Concrete mix with proportion 1:1.4:2.1 was prepared by mixing cement, fine and coarse aggregate, with water-cement ratio of 0.33. The above blend is mixed with CF 615, admixture by adding 0.8% of cement. The proportion of mix design was calculated according ACI 21. Each batch mix was prepared as per the compositions presented in Table 2.

Table 2: Composition of concrete mixes.

Ingredients, (kg)	Batch 1 control Mix	Batch 2 Control Mix + 0.8% CF 615	Batch 3 Mix 1% Pozzolan + 0.8% CF 615	Batch 4 Mix 1% MS + 0.8% CF 615	Batch 5 Mix 1% NS + 0.8% CF 615
Cement	8.18	8.18	7.58	7.58	7.58
Gravel, (size 20 mm)	17.2	17.2	17.2	17.2	17.2
Sand	11.75	11.75	11.75	11.75	11.75
Water	2.68	2.68	2.68	2.68	2.68
Nano-Silica	-	-	-	-	0.60
Micro-Silica	-	-	-	0.60	-
Ultra-Pozz	-	-	0.60	-	-
Admixture	-	0.065	0.065	0.065	0.065

### 2.4.3. Slump test

The consistency, workability, or fluidity of fresh concrete was determined using slump test. After preparing the concrete mix, the mix was filled in the slump cone in three layers each compacted 25 times followed by lifting the slump cone upwards, and overturned and the slump value was measured. The slump value was found to be within the permissible range of normal concrete of 75 mm to 100 mm.

### 2.5. Casting and curing of concrete samples

Cube moulds are cleaned and lubricating oil are spread into the internal surfaces of the cubes to avoid water absorption and stickiness of the fresh concrete. Concrete samples are casted into the moulds in three layers; each layer will be compacted by 25 times in order to remove air voids. The surface of the samples should be pressed and levelled. The above samples are allowed to stay in the moulds for 24 hours. The concrete cubes are demoulded and kept for curing for 28 days. Figure 1 (A& B) represents the casted concrete cubes.



Figure 1 (A&B). Casting and Levelling of Concrete Samples.

## 2.6. Compressive Strength Test

The compressive strength of the specimens is tested using Universal Testing Machine (UTM) by placing the specimen between two plateau and apply the load in increasing manner. The compressive strength is calculated by dividing the maximum load (P) (attained from the test) by the cross sectional area of the cube (A). The compressive strength is calculated using eq (1).

$$\sigma = \frac{P}{A} \quad \dots\dots\dots (1)$$

## 3. Results and discussion

### 3.1. Synthesis of silica Particles

The micro and nano silica particles are synthesized by stober's method and the particles obtained was found to be intact, mono dispersed and spherical in shape as indicated in the scanning electron microscopy images shown in Figures 2 and 3.

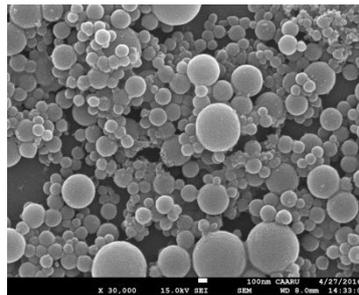


Figure 2. SEM micrograph of micro silica particles.

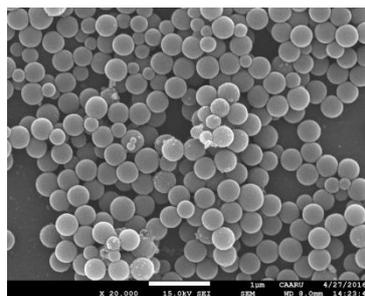


Figure 3. SEM image of nano silica particles.

### 3.2. Determination of size of silica Particles

The size of synthesized silica particles are determined using dynamic light scattering method. From the experimental results, it was observed that a single peak in Figure 4 indicates the particles are of same size and the average particle sizes are around 340 nm.

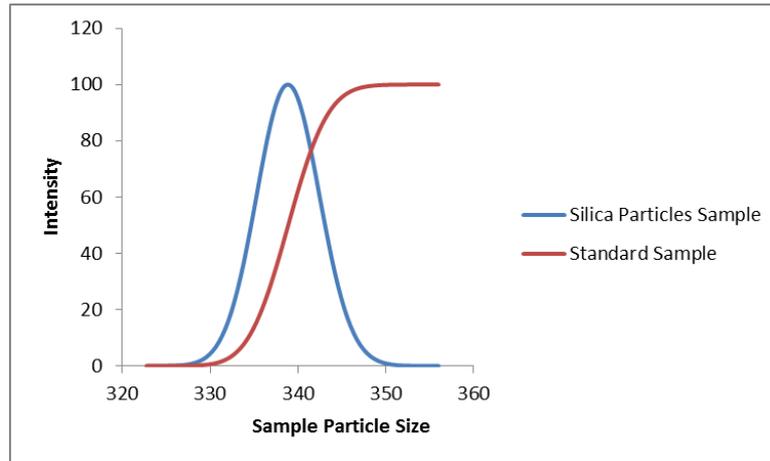


Figure 4. DLS of silica particles.

### 3.3. Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier Transforms Infrared spectroscopy characterization of the silica particles are shown in Figure 5. The FTIR image shows the functional groups present on the surface of the nano silica particles. The broad peak corresponding to the wave number  $3433.2\text{ cm}^{-1}$  represent the O-H group, peak at  $2000\text{ cm}^{-1}$  represent  $-C\equiv C-$  group, sharp peak at  $1635\text{ cm}^{-1}$  represent presence of C=O group.

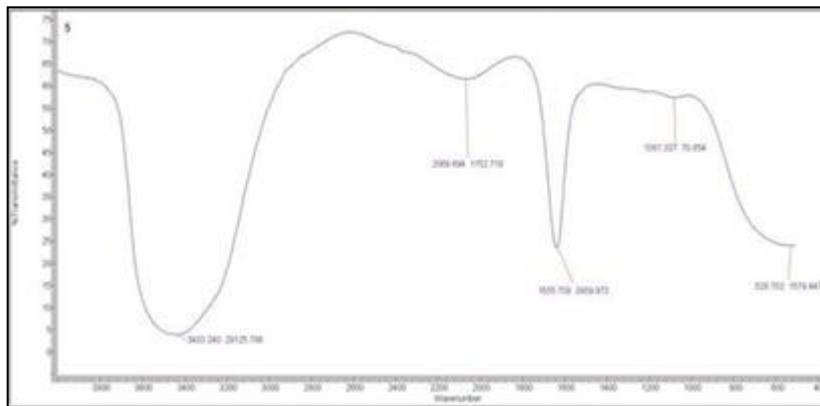


Figure 5. FTIR characterization of silica particles.

### 3.4. Slump test results

From the slump test it was observed that; for normal mix the type of slump formed is True slump as displayed in Figure 6. Figure 7 shows the slump test results of 0.8% CF 615, 1% Nano-Silica and the slump formed is a Collapse slump presented in Figure 8, whereas Figure 9 designates the sample consisting 1% Micro-Silica, 0.8% CF 615 and the type of slump formed is true slump, and finally for the 1% Pozzolan, 0.8% CF 615 the type of slump formed is true slump as it shown in Figure 10.



Figure 6. Slump test result for normal mix.



Figure 7. Slump test result for 0.8% CF 615.



Figure 8. Slump test result for 1% nano-silica, 0.8% CF 615.



Figure 9. Slump test result for 1% Micro-Silica, 0.8% CF 615.



Figure 10. Slump test result for 1% Pozzolan, 0.8% CF 615.

### 3.5. Compressive strength test results

From the compressive strength test of each batch as indicated in Table 3 it was observed that maximum compressive strength was obtained as 37.45 N/mm<sup>2</sup> for batch consists of Ultra-pozz and 0.8% CF 615 compared to the reference mix strength with an increase of 17.4%. The maximum compressive strength obtained was due to the fineness of Ultra-pozzolane sample, which has highest surface area available for hydration process there by contributes the enhancement of compressive strength.

Table 3: Average compressive strengths of each batch.

Batch Type	Average Compressive Strength, N/mm <sup>2</sup>
Reference mix.	30.95
Admixture 0.8% CF615, cure WH	32.26
Nano-Silica and 0.8% CF615, cure WH	-
Micro-silica and 0.8% CF615, cure WH	30.94
Ultra-pozz and 0.8% CF615, cure WH	37.45

It was observed that addition of ultra-pozz to the concrete can give the maximum compressive strength compared to other batches. Furthermore, the compressive strength increased by addition of admixtures by 4.2% whereas there was no change in compressive strength in the case of batch consisting of Micro silica. The test failed in the batch made up of nano silica at same percentage. This may be due to the fact that the amount of the nano particle added to the concrete mix is more than the optimum amount, which cause the failure. When the content of silicon powder is more, the new mixed concrete becomes so sticky.

## 4. Conclusions

Silica nanoparticles of size around 340 nm were successfully prepared by stober's protocol using tetra Ethyl Ortho Silicate (TEOS), Ethanol, ammonia and water. The surface morphology and size of the synthesized Nano particles were confirmed using Scanning Electron Microscopy and Dynamic Light Scattering Method.

It was observed that the synthesized particles are of uniform size with spherical shape and mono disperse in nature without any aggregation. These particles were applied for the strengthening of cement concrete of different batches by replacing 1% of cement by nano silica particles, 1% micro silica, 1% Ultra pozzolana and the compressive strengths was maximum for the sample prepared using 1% Ultra pozzolana.

The test results indicate that the batch prepared by incorporating Nano silica in the concrete mix collapsed during in the slump test that indicates the failure of the test. The compressive strength test could not be performed due the collapse of specimen in the curing tank which failed curing.

The percentage compositions of nano silica in the mixture influence the failure of the test. It may require less amount of silica to pass the slum test. The research is currently concentrated on the optimization of the nanoparticles to get better compressive strength of the specimen by varying the percentage of silica particles.

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