

Effect of Nanosilica on Mechanical and Microstructural Properties of Cement Mortar

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ABSTRACT

Recently, nanomaterials (such as nanosilica) are receiving special attention because of their ability to improve the performance of concrete compared with traditional mineral admixtures. In this work, a number of specimens were prepared to study the behavior of cement mortar containing nanosilica. The cement replacement by nanosilica of 3% and 5% by weight of cement was used. The mechanical and microstructural properties of the specimens were investigated. An experimental result from literature was utilized to predict the transport of chloride into reinforced concrete structures modified with nanosilica using COMSOL multiphysics commercial package. The experimental results show that cement mortars containing nanosilica have higher strength than ordinary portland cement mortars. Also, the SEM images confirmed the improvement in the microstructure of mortar with nanosilica.

KEYWORDS: Cement mortar, Nanosilica, Compressive strength, Microstructure.

INTRODUCTION

Worldwide, a huge amount of cement is produced annually. Large amounts of energy is consumed during its production and it is also one of the main contributors to carbon dioxide emission (1 ton of cement produces approximately 1 ton of CO₂). Thus, there is a critical need in the concrete industry to minimize the amount of cement used. The main task is to produce impervious concrete within reasonable cost and with less cement (Aly et al., 2011).

The use of supplementary cementitious materials in cement-based materials has grown in recent years. When a suitable amount is used as a partial replacement for cement, the performance of cement-based materials can be improved through changes in

the fresh and hardened states, the modifications in physical, mechanical and chemical properties, as well as microstructural properties and hydration.

Due to the innovative use of materials in nanometer scale, nano-technology has attracted substantial attention of researchers in recent years. If used properly, nanomaterials can result in significant improvement in the properties of the cement-based materials (Park et al., 2007).

The mechanical behavior of cement-based materials exists in a nano- scale and a micro-scale. Consequently, the molecular structure of cement-based materials can be altered with the use of nanotechnology; and subsequently the bulk properties of the material will be improved. Also, durability, mechanical properties and sustainability of the materials can be enhanced using the new innovation. In concrete industry, nanotechnology can be utilized to produce high-

performance, durable and cost-effective cement-based materials. Use of nanomaterials in cement-based materials has three major advantages. Firstly, development of high-strength concrete can be achieved for particular applications. Secondly, similar strength of concrete can be obtained by reducing the amount of cement used in concrete and thus decreasing the environmental impact and cost of construction materials. Finally, the construction periods can be reduced, because with less curing time, a high-strength concrete may be produced (Morsy et al., 2011).

In recent years, special types of nano-materials have drawn the attention of some concrete researchers (Ltifi et al., 2011; Senff et al., 2010; Nazari and Riahi, 2010; Belkowitz and Armentrout, 2010; Doroganov, 2012; Mondal et al., 2010; Barluenga et al., 2012; Rodriguez et al., 2013; Hou et al., 2013; Ozyildirim and Zegetosky, 2010; Ibrahim et al., 2012; Kim et al., 2010) because of their remarkable physical, mechanical, electrical, chemical and thermal properties, as well as exceptional performance in polymer and cement-based materials. Since nanotechnology in concrete is a relatively new research area, not much study has been conducted yet to investigate the properties of concrete containing nanomaterials.

This study is aimed at: 1) investigating the mechanical and microstructural properties of cement mortars prepared with nanosilica. 2) predicting the transport of chloride into reinforced concrete structures using COMSOL multiphysics commercial package.

Experimental Program

Materials

ASTM C 150 Type I Portland cement was used in this study. The chemical composition of Portland cement used in the preparation of mortar specimens is shown in Table 1. The fine aggregate used in this study was dune sand. The specific gravity of the fine aggregate was 2.56, and the absorption was 0.4%. Table 2 shows the grading of the dune sand used in the study.

Table 1. Chemical composition of Portland cement

Constituent (wt %)	Type I cement
Silica (SiO ₂)	19.92
Alumina (Al ₂ O ₃)	6.54
Ferric oxide (Fe ₂ O ₃)	2.09
Lime (CaO)	64.70
Magnesia (MgO)	1.84
Silicate (SO ₃)	2.61
Potassium oxide (K ₂ O)	0.56
Sodium oxide (Na ₂ O)	0.28
Tri calciumsilicate (C ₃ S)	55.9
Dicalcium silicate (C ₂ S)	19
Tricalcium aluminate (C ₃ A)	7.5
Tetracalciumaluminoferrite (C ₄ AF)	9.8

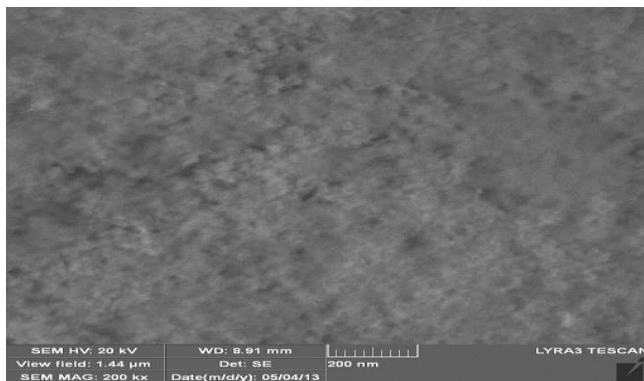
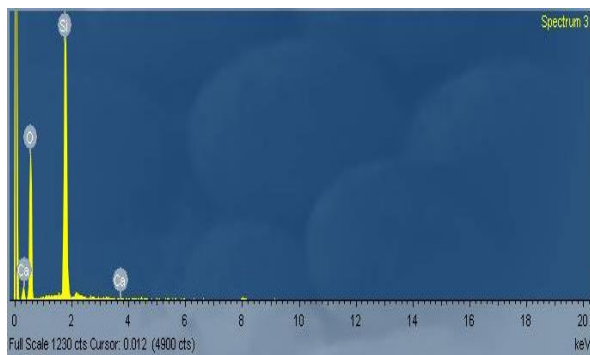
Table 2. Grading of the fine aggregate used in the study

ASTM Sieve #	Sieve opening	% passing
4	4.75 mm	100
8	2.36 mm	100
16	1.18 mm	100
30	600 μm	76
50	300 μm	10
100	150 μm	4

A commercial nano-silica by the commercial name VK-SP 15 was used. The chemical and physical properties of the used nano-silica as supplied by the manufacturer are provided in Table 3. Also, the scanning electron micrographs (SEM) and the energy dispersion analysis (EDAX) are shown in Figures 1 and 2, respectively. The superplasticizer used in all the mixes was Glenium 51®. It is a new generation polycarboxylic-based ether hyperplasticiser. Its technical data is shown in Table 4, as obtained from the manufacturer.

Table 3. Physical and chemical properties of nanosilica

Appearance	White powder
SiO ₂ content (%)	99.8
Average particle size (nm)	15±5
Specific surface area (m ² /g)	250±30
PH	7

**Figure (1): SEM image of nanosilica****Figure (2): EDAX of nanosilica****Table 4. Technical data of Glenium 51**

Appearance	Brown liquid
Specific gravity @ 20°C	1.08±0.02 g/cm ³
pH-value @ 20°C	7.0±1.0
Alkali content	≤ 5.0
Chloride content	≤ 0.1 %

Mix Proportions and Casting of Test Specimens

For all the mixes, a cement:sand ratio of 1:2.75 and a water: cementitious materials ratio of 0.4 were used. The cement replacement by nanosilica of 3% and 5% by weight of cement was used. The amount of superplasticizer used in the mixes was calculated as a proportion of the weight of the cement plus nanosilica. In all mixtures, the dosage of superplasticizer used was adequate such that bleeding or segregation was avoided. The summary of mix proportions for cement mortars is presented in Table 5. Preparation of OPC mix proceeded as follows: cement and fine aggregate were mixed individually in the mixer. A homogenous mortar was obtained with all the constituents mixed together with the addition of potable water and with a superplasticizer mixed uniformly with the constituents to enhance workability. For the cement mortar containing nanosilica, as the nano-materials are difficult to disperse uniformly due to their high surface area, the mixing was done as follows: Firstly, the nanosilica was stirred with the mixing water at a high speed for 5 min. using a sonicator machine. Then, the cement was added into the mixer and mixed at medium speed; sand was then added gradually. The superplasticizer was added and stirred at a high speed. The moulds (50 x 50 x 50 mm) were oiled and the mortar was then poured into the moulds and the mortar was consolidated by vibrating the molds over a vibrating table. The test specimens were demolded after 24 hours of casting. All the specimens were then cured for a period of 28 days in water tanks under laboratory conditions.

The naming convention adopted for the mixes is as follows: OPC stands for ordinary portland cement and NS for nanosilica. All the work has been conducted in the Concrete Lab, Civil Engineering Dept., King Fahd Univ. of Petroleum and Minerals (KFUPM), Saudi Arabia.

Table 5. Mix proportions of the specimens

Specimen designation	Water (g)	Cement (g)	Sand (g)	Nanosilica (g)	SP (g)
OPC	240	600	1650	-	7.2
NS3	240	582	1650	18	15
NS5	240	570	1650	30	21

Experimental Tests

Compressive Strength

Compressive strength test was conducted according to ASTM C 39 after 7 and 28 days of curing. The test was conducted in a hydraulic machine with a load capacity of 3000 kN, the loading rate was kept at 1.5 kN/s for all the tests until the specimen failed. The maximum load was noted. The compressive strength was calculated by dividing the failure load by the cube cross-sectional area.

Scanning Electron Microscopy (SEM)

SEM was performed on the cement mortar specimens containing nanosilica in order to study the effect of nanomaterials on the mortar morphology. The following procedure was followed:

- 1) Fractured samples were obtained from the mortar specimens and coated with gold.
- 2) Using SEM machine (JEOL SEM JSM-5800 LV), the images of the mortar samples were captured. Images were carried out at 10,000 magnification using secondary electron mode.

RESULTS AND DISCUSSION

Compressive Strength

The summary of compressive strengths for OPC and nano-silica mixes is shown in Figure 3. The Figure shows the compressive strengths at ages of 7 and 28 days of curing. The reported values of compressive strength are averages of three specimens prepared from each mix. It can be observed that the compressive strength in mortars containing nanosilica is higher than

that of control cement mortars. It was observed that there is a significant increase in the compressive strength after 7 days of curing but after 28 days of curing, the increase in compressive strength is not that significant. The average percentage increase in compressive strength for 3% nanosilica replacement was 51%; while it was 92% for 5% nanosilica replacement after 7 days of curing. After 28 days of curing, the average percentage increase in compressive strength for 3% replacement was 17% only; while it was 35% for 5% replacement. The strength development of the mortars containing nanosilica may be attributed to pozzolanic reaction and filler effect. Also, nanosilica would fill pores thereby increasing the mortar strength. Hence, the addition of nanosilica to cement mortars improves their strength characteristics.

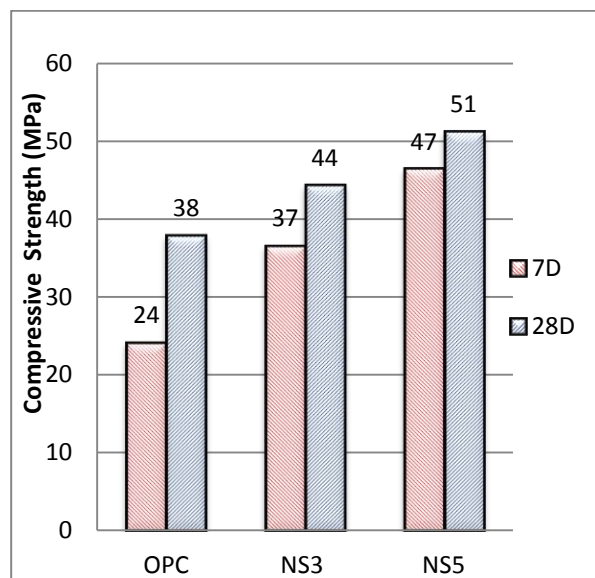


Figure (3): Compressive strength of the mixes for 7 and 28 days

Effect of Nanomaterials on Mortar Pore Structure

To verify the mechanism predicted by the compressive strength test, SEM examinations were performed. Figures 4 and 6 show the SEM images of cement mortar containing 3% and 5% replacement of cement by nanosilica, respectively. The images

confirmed a dense and compact formation of hydration product, unlike porous plate-like structure of OPC as reported in literature (Gemelli et al., 2001) (Figure 8). Further, the interfacial layer between the sand particles and the paste is dense. Figure 5 and 7 show the EDAX for NS3 and NS5 after 28 days, respectively. Also, the EDAX results confirmed the high contents of calcium (Ca) and silica (SiO₂) in the cement mortars, thereby confirming the high quantity of C-S-H formed in the mortar specimens with nano-silica.

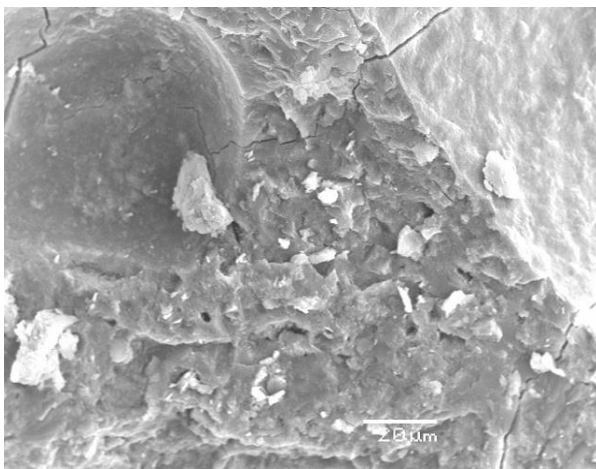


Figure (4): SEM image for NS3 after 28 days

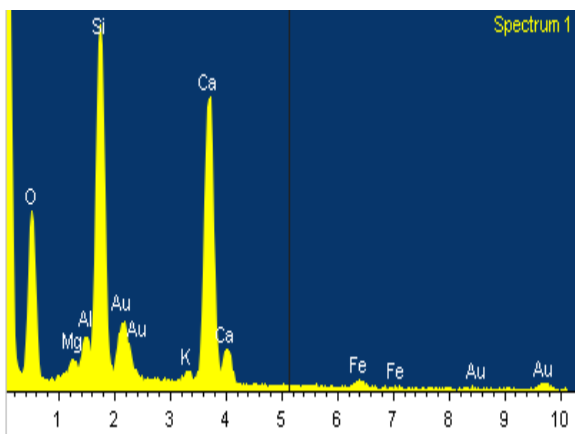


Figure (5): EDAX for NS3 after 28 days

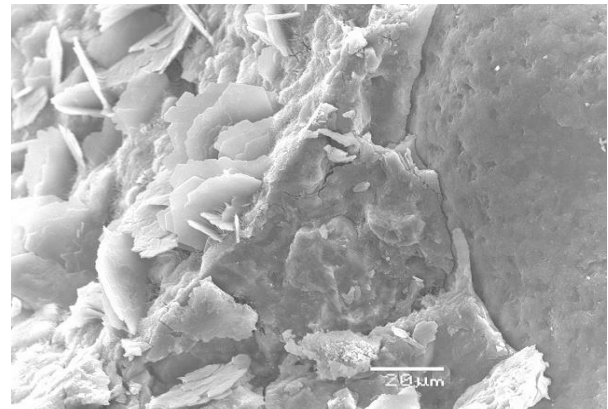


Figure (6): SEM image for NS5 after 28 days

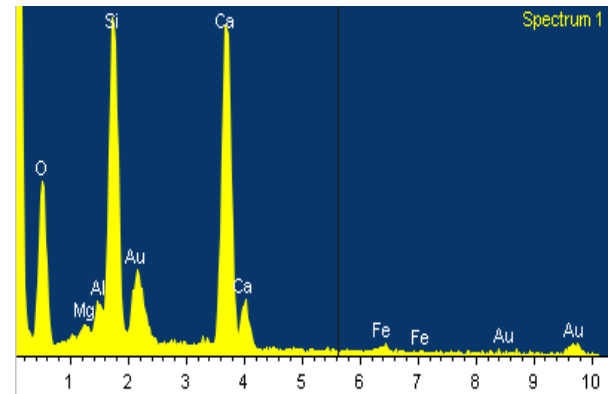


Figure (7): EDAX for NS5 after 28 days

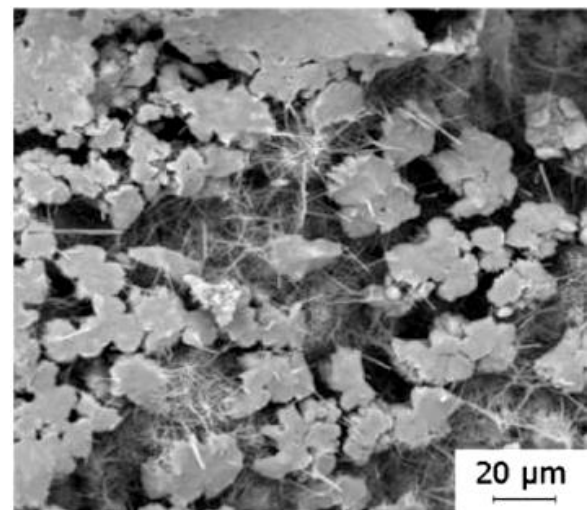


Figure (8): SEM image for plain cement mortar after 28 days (Gemelli et al., 2001)

Prediction of Chloride Transport in Reinforced Concrete

In order to predict the transport of chloride in reinforced concrete (RC) structures, COMSOL finite element software was used to simulate the chloride diffusion in concrete modified with nanosilica. The coefficient of chloride diffusion obtained in the experiment conducted by Chen et al. (Chen et al., 2012) (i.e. $3.18 \times 10^{-11} \text{ m}^2/\text{s}$) was used in the simulation.

Input Parameters Used in the COMSOL Software

Figure 9 shows the RC beam modeled in the COMSOL software. The dimensions of the concrete beam are 425 x 1800 mm, the domain of reinforcement is 15 x 1800 mm and the concrete cover domain is 60 x 1800 mm. Hence, the total depth of the beam is 500 mm. A free chloride concentration of 0.3 mol/m^3 was applied to the top and bottom of the beam. Also, a force of 100 N was applied at the center of the beam. Table 6 shows the plane stress input parameters.

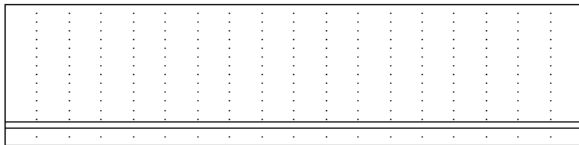


Figure (9): Geometry of the RC beam

Table 6. Plane stress input parameters

Subdomain	Young's Modulus (MPa)	Poisson's Ratio	Density (kg/m ³)
Concrete	30000	0.2	2400
Reinforcement Steel	200000	0.3	7850

Results from the COMSOL Software

Figure 10 shows the free chloride distribution in the RC beam. It can be observed that there is significant change in the chloride penetration in the compressive zone of the beam, while the tension zone is almost free from chloride.

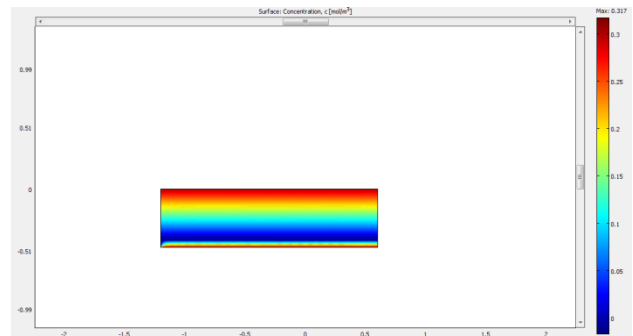


Figure (10): Free chloride distribution in the RC beam

CONCLUSION

In this research study, a number of cement mortar specimens were prepared to examine the effect of nanomaterials on mechanical and microstructural properties of mortar containing nanomaterials. The cement mortars were prepared by replacing cement with 3% and 5% nanosilica. Compressive strength and morphology of the mortar specimens prepared with nanosilica were studied. The results of compressive strength revealed a considerable improvement in the strength characteristics of cement mortars containing nanosilica. Also, the SEM images confirmed the improvement in the mortar microstructure of mortar with nanosilica. Also, the interfacial zone in the mortar specimens with nanosilica was very dense.

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