



ORIGINAL ARTICLE

High-performance cement mortars-based composites with colloidal nano-silica: Synthesis, characterization and mechanical properties



Behnam Behnia^a, Hossein Safardoust-Hojaghan^b, Omid Amiri^{c,d},
Masoud Salavati-Niasari^{b,*}, Ali Aali Anvari^a

^a Department of Mining Engineering, University of Kashan, Kashan, Iran

^b Institute of Nano Science and Nano Technology, University of Kashan, Kashan, P.O. Box 87317-51167, Iran

^c Faculty of Chemistry, Razi University, Kermanshah 6714414971, Iran

^d Department of Chemistry, College of Science, University of Raparin, Rania, Kurdistan Region, Iraq

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Abstract In recent years, nanotechnology has found great application in the concrete and cement mortars industries. In this study, silica nanoparticles have been prepared in acidic solution via simple, low-cost, and efficient sol-gel methods. The effect of surfactant and calcination on the morphological properties of silica nanoparticles was investigated. The prepared silica nanoparticles are characterized via X-ray diffraction (XRD), Transmission Electron Microscopy (TEM), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and dynamic light scattering (DLS) analysis. Prepared silica nanoparticles are introduced to cement mortars at different dosage and their effects on compressive and flexural strengths were studied. Results showed that 4% (%wt) have the highest improvement in cement mortars quality. The presence of 4% contents of silica nanoparticles in cement mortars increases the compressive strength by 10.54% in three days, 12.35% in one week, and 15.04% in 28 days. Flexural strengths are increased to 23.25% in three days, 9.45% in one week, and 18.40% in 28 days.

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1. Introduction

Nanoparticles have an ultrafine size, which makes them show unique physical properties physical and chemical properties different from those of the conventional materials (Lau and Hui, 2002; Khan et al., 2019; Baig et al., 2021). Since nanoparticles possess unique properties, they have gained increasing attention and have been applied in a variety of industries to fabricate new materials (Bissessur and Narain, 2020;

* Corresponding author.

E-mail address: salavati@kashanu.ac.ir (M. Salavati-Niasari).

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Peters et al., 2016). The idea of using nanostructures developed since the late twentieth century and has affected different fields including the cement and concrete industry (Li et al., 2004; Son et al., 2018; Xu et al., 2018; Song et al., 2018; Rong et al., 2020; Zhuang and Chen, 2019). More attention has been attracted to these nanostructures that can act as a replacement to cement mortars in concrete or as an additive, although using nanomaterials in concrete and cement mortars industries faces a range of challenges: (1) the difficulties in the nanomanufacturing involves scaled-up, reliable and cost-effective manufacturing of nanomaterials; (2) the practical methods for field application have not been established; (3) incompetent of based material utilization; (4) there is no standards for mix-design yet (Raki et al., 2010; Saloma et al., 2015; Zhang et al., 2019).

Nanomaterials are classified into four main groups according to the number of dimensions, which are not restricted to the nanoscale range: (1) Zero-dimensional (0-D) such as quantum dots and fullerenes (2) One-dimensional (1-D) such as nanotubes, nanowires, and nanorods (3) Two-dimensional (2-D) such as graphene and nanosheets (4) Three-dimensional (3-D) such as nanoporous materials (Tiwari et al., 2012; Ngô et al., 2014). Till now, various nanomaterials from all four main groups have been applied in concrete and cement mortars such as; carbon nanotubes (Ubertini et al., 2014; Tzileroglou et al., 2017), silica nanoparticles (Mahdikhani et al., 2018; Li et al., 2018; Sobolev et al., 2009; Santos et al., 2015), TiO₂ nanoparticles (Yang et al., 2018), Fe₂O₃ nanostructures (Nouri, 2018; Siang Ng et al., 2020), Al₂O₃ nanoparticles (Szumigala and Polus, 2015) and organic/inorganic nanocomposites (Kaish et al., 2018). Among various applied nanostructures, major research efforts have been focused on the influence of nano-silica on cement paste. The result showed that nano-silica has a considerable effect on the early-age and long-term strength of cement paste, mortar, and concret (Ehsani et al., 2017; da Silva Andrade et al., 2018; Singh et al., 2015; Flores et al., 2017). In contrast to the above-mentioned nanostructures which have been applied, SiO₂ nanoparticles is a pozzolanic material. So, silica nanoparticles play a dual role: it promotes the workability of cement paste as a filler, it also has a positive influence on the mechanical properties of cement paste via the pozzolanic reaction (Arel and Thomas, 2017).

Even though some researchers have been investigated the effect of nano-silica on cement mortars but this work has been focused on the engineering of nano-silica to obtain considerable results. For this, the first step, silica nanoparticles were synthesized via a simple and fast sol-gel method. The effect of calcination and surfactant on the size and morphology of prepared nano-silica were investigated. The Prepared silica nanoparticles were characterized by XRD, TEM, SEM, EDS, and DLS analysis. Finally, prepared silica nanoparticles

were introduced to cement mortars at different dosage and their effects on compressive and flexural strengths were studied.

2. Experimental

2.1. Materials and methods

Ethanol, sodium orthosilicate, sulfuric acid, and sodium dodecyl sulfate (SDS) were purchased from Merck and all the chemicals were employed with any further purifications. X-ray diffraction (XRD) patterns analysis was done by a Philips-X'pertpro, X-ray diffractometer employing Ni-filtered Cu K α radiation. Nicolet Magna-550 spectrometer in KBr pellets was applied for recording Fourier transform infrared (FT-IR) spectra. Morphological properties of products were investigated via scanning electron microscopy (SEM) obtained on LEO-1455VP equipped with energy-dispersive X-ray spectroscopy. Philips EM208 transmission electron microscope was applied for recording TEM images of prepared samples.

2.2. Synthesis of SiO₂ nanoparticles

A mixture was prepared with sulfuric acid and ethanol (S/E) in equal amounts, then, 5 g of sodium orthosilicate was added in 60 ml of distilled water and added drop-wise to the S/E mixture. After aging for 4 h, it was centrifuged and washed with distilled water and dried at 60 °C for 10 h. Finally, the obtained powder was calcined at 500 °C for 2 h. The preparation procedure is shown in Fig. 1. For the SDS-based procedure, the SDS was dissolved in distilled water with sodium orthosilicate. Other steps were quite similar to the method mentioned.

2.3. Preparation and characterization of cement-based pastes

The chemical composition of cement was listed in Table 1. The used cement was exposed to ambient air for the minimum time possible. When it has to be kept for more than 24 h between sampling and testing, it was stored incompletely filled and airtight containers made from a material that does not react with cement. Deionized water was used for validation testing. The proportions by mass were considered one part of the cement, three parts of CEN Standard sand, and one half part of water (water/cement ratio 0.50). Each batch for three test specimens consists of (450 \pm 2) g of cement, (1350 \pm 5) g of sand and (225 \pm 1) g of water. Weigh the cement and water by means of the balance. When water is added by volume it shall be dispensed with an accuracy of \pm 1 ml. Each batch of mortar mechanically using the mixer was mixed. The silica

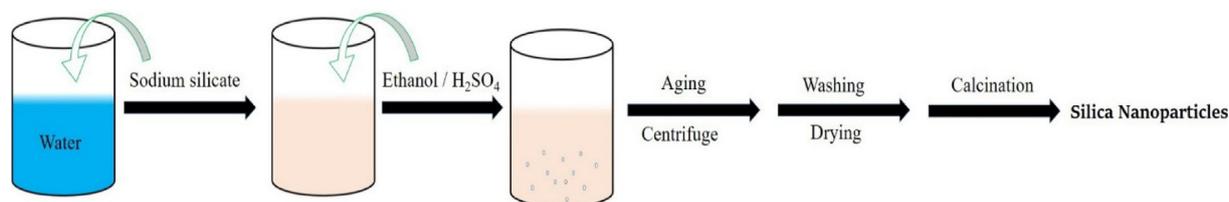


Fig. 1 Schematic procedure of silica nanoparticles preparation.

Table 1 Chemical and physical properties of testing cement.

Constituents	(%)Content
SiO ₂	21.20
Al ₂ O ₃	4.09
Fe ₂ O ₃	4.99
CaO	63.59
MgO	1.14
SO ₃	1.98
K ₂ O	0.61
Na ₂ O	0.29
L.O.I.	2.43
CL ⁻	0.02
I.R.	0.32
SiM	2.33
AIM	0.82
L.S.F.	92.3
C ₃ S	57.3
C ₂ S	18.0
C ₃ A	2.4
C ₄ AF	15.2
CaSO ₄	3.4
Density (g/cm ³)	3.15

nanoparticles were stirred with the mixing water at high speed for 1 min. After the mixing stage, the effect of different dosages of silica nanoparticles on the workability and flow of various mortars was studied (Table 2). The timing of the various mixing stages refers to the times at which mixer power is switched on/off and shall be maintained within ± 2 s. The mixing procedure was done as follows:

- Water and the cement were placed into the bowl with care to avoid loss of water or cement;
- Immediately the water and cement were brought into contact start the mixer at a low speed whilst starting the timing of the mixing stages.

In addition, the time to the nearest minute, as 'zero time' was recorded. After 30 s of mixing, the sand was added steadily. Then the mixer was switched to high speed and continue mixing for an additional 30 s. The test specimens were 40 mm \times 40 mm \times 160 mm prisms. The specimens immediately after the preparation of the mortar were molded. The mold gently was lifted from the jolting table and removed from the hopper. Immediately the excess mortar was struck off with the metal straightedge, held almost vertically but inclined in the direction of striking. This striking off was repeated to procedure with the straightedge held at a more acute angle to

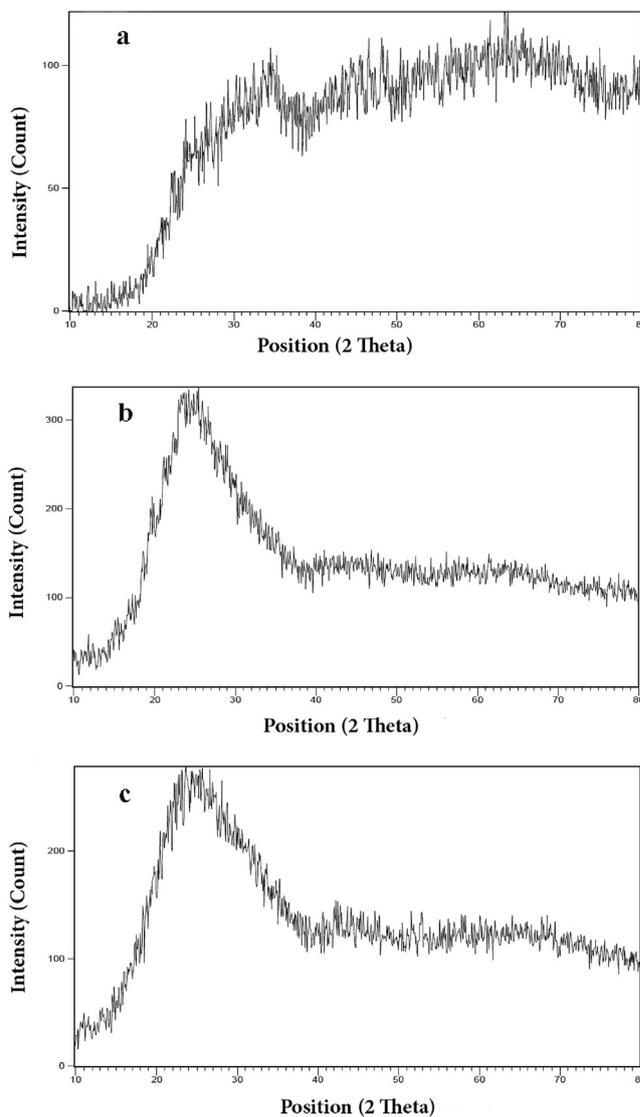


Fig. 2 XRD pattern of prepared silica nanoparticles a) before calcination b) after calcination without surfactant c) after calcination in the presence of SDS as a surfactant.

smooth the surface. Demoulding was Carried out, for 24 h tests, not more than 20 min before the specimens are tested. The marked specimens were submerged without delay in a convenient manner, either horizontally or vertically, in water at (20.0 ± 1.0) °C in the containers. With horizontal storage,

Table 2 Different applied experimental conditions.

Sample	Silica nanoparticles (wt%)	Silica nanoparticles (g)	Water (ml)	Cement (g)	Sand (g)	Blaine (Cm ² /g)
SC-0	0	0	225	450	1350	3150
SC-1	1	4.5	225	450	1350	3150
SC-2	2	9	225	450	1350	3150
SC-3	3	13.5	225	450	1350	3150
SC-4	4	18	225	450	1350	3150
SC-5	5	22.5	225	450	1350	3150
SC-10	10	45	225	450	1350	3150
SC-15	15	67.5	225	450	1350	3150

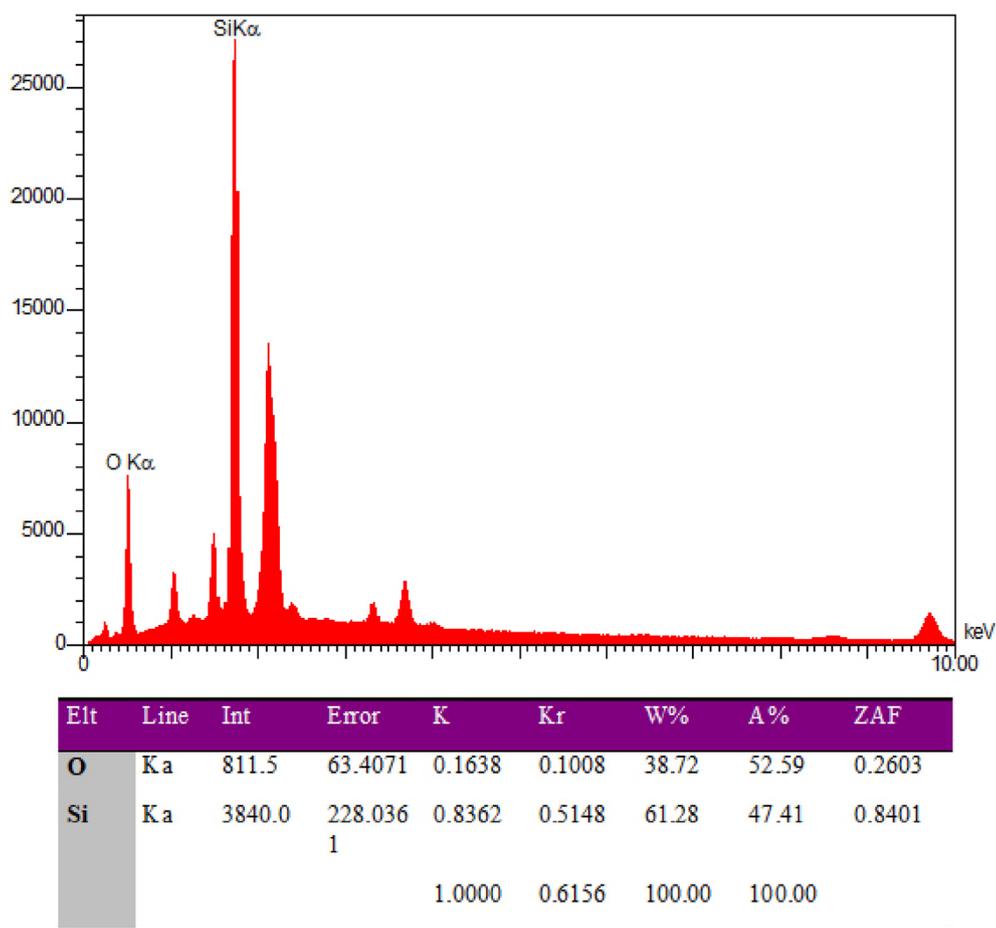


Fig. 3 EDS analysis of prepared silica nanoparticles at a calcination temperature of 500 °C in the presence of SDS.

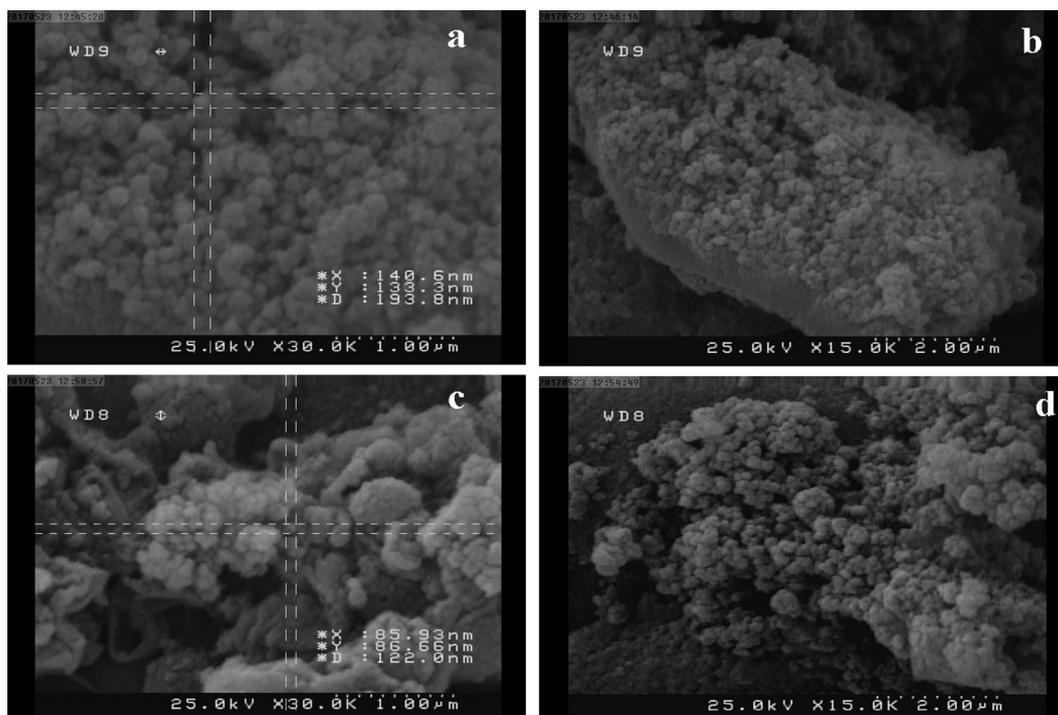


Fig. 4 SEM images of prepared silica nanoparticles a,b) in the absence of SDS c,d) in the presence of SDS.

vertical faces as cast vertical were kept. The age of specimens was calculated from zero time. Strength tests were carried out at different ages within the following limits: $3 \text{ d} \pm 45 \text{ min}$, $7 \text{ d} \pm 2 \text{ h}$ and $28 \text{ d} \pm 8 \text{ h}$. All tests were repeated 5 times and the mean of them was reported.

The consistency and the setting time of fresh pastes were done according to AASHTO standards by allowing a 1-mm Vicat needle.

3. Result and discussion

X-ray diffraction (XRD) was applied for the investigation of the phase structure and crystallinity of the prepared silica nanoparticles. Fig. 2 shows the XRD pattern of prepared silica nanoparticles. Fig. 2a shows an XRD analysis of the prepared samples without calcination. It is clear that without calcination, no silica NPs are formed. Fig. 2b represents the XRD pattern of prepared silica NPs in calcination temperature

of 500°C without using a surfactant. XRD pattern has a broad peak at $2\theta = 26^\circ$ which is attributed to the amorphous nature of synthesized silica NPs. As well as shown in Fig. 2b, silica NPs are formed without impurity. Fig. 2c shows the XRD pattern of prepared silica NPs in calcination temperature of 500°C at the presence of SDS as a surfactant. Amorphous silica NPs are formed without any impurity.

The characteristic peak in Fig. 2c is broader than Fig. 2b. According to the Scherrer equation, the main peak of the XRD pattern is broadening when the crystallite size is decreasing. It was predictable that the final crystalline size of silica NPs is decreased in the presence of SDS and results confirm the prediction.

Energy Dispersive X-Ray Spectroscopy (EDS) is a suitable technique for chemical analysis of the prepared samples. EDS analysis was applied to the sample prepared at a calcination temperature of 500°C in the presence of SDS. The obtained results are shown in Fig. 3. All results obtained from the

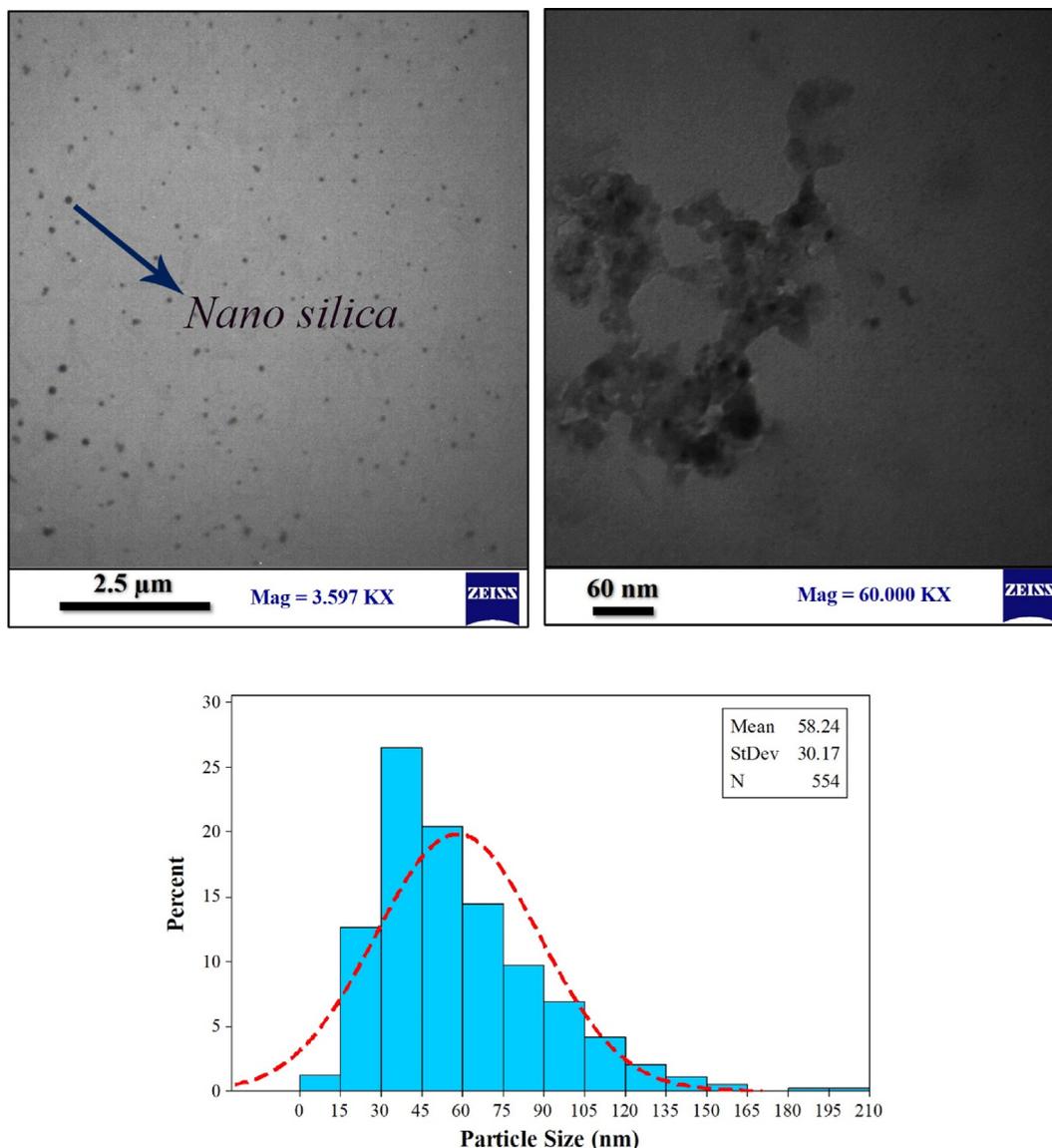


Fig. 5 TEM images and size-distribution histogram of silica nanoparticles with SDS.

spectrum and table of quantitative results confirm the formation of silica without any impurity and undesirable elements.

Morphology and particle size were investigated via a scanning electron microscope (SEM). Fig. 3 shows SEM images of prepared silica nanoparticles. Fig. 4a and Fig. 4b represent SEM images of prepared silica nanoparticles at the presence of SDS at two different magnifications and Fig. 4c and 4d show SEM images of prepared silica nanoparticles in the absence of surfactant. Particle size in the presence of SDS is less than in the absence of surfactant. SDS is an anionic surfactant that has a 12-carbon tail attached to a sulfate group. For its structure, it can prevent agglomeration while the formation of silica nanoparticles. Although, all prepared nanostructures have spherical morphology.

For better estimation of morphology and particle size, Transmission electron microscopy (TEM) was applied. Fig. 5 shows TEM images of prepared silica nanoparticles at the presence of SDS at two magnifications. TEM images confirm the spherical morphology of prepared silica nanoparticles. According to the size-distribution histogram, prepared silica NPs have mean diameters of 58.24 nm. The average diameter of synthesized particles was estimated at 51.55 nm according to dynamic light scattering (DLS) analysis that completely congruous with TEM results (Fig. 6).

The influence of nano-silica on the setting time of cement paste was investigated. For investigation, the sample SC-4 (4% nano-silica) was applied. Results indicated that the addition of nano-silica has a significant effect on the setting time. Setting time of blank (SC-0) was measured at 250 min. While setting time of cement paste accelerated to 220 min in SC-4 sample.

Based on the obtained results, the different dosages of SDS-silica nanoparticles were introduced to cement mortars, and their effect on compressive and flexural strengths was investi-

gated. The compressive strength of cement mortars depends on different parameters such as water-cement ratio, cement strength, quality of cement mortars material, nature, and amount of additive (Aitein et al., 2019). In this study, all parameter is kept constant, but the amount of silica nanoparticles as an additive is changed. As well as shown in Fig. 7, after three days, the compressive strength of blank cement mortars without silica nanoparticles was 23.8 MPa. For the SC-1, the compressive strength increased 24.2 MPa after three days. For SC-2, SC-3, SC-4, SC5, SC-10 and SC-15, the

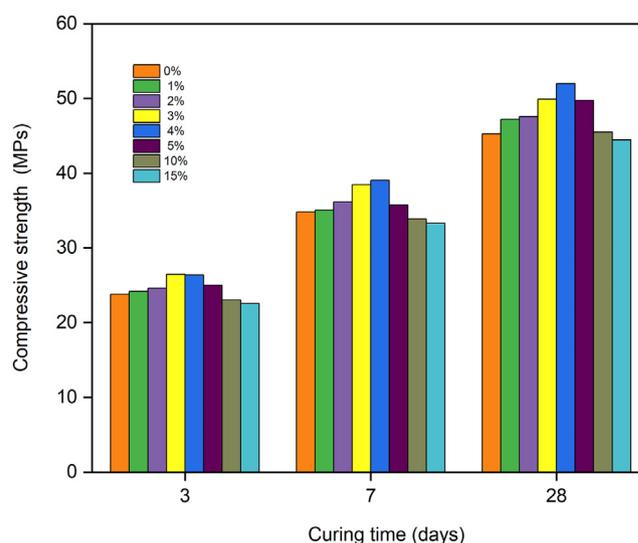


Fig. 7 Amount of compressive strengths of mortar at the different dosages of silica nanoparticles in different curing time.

Results

Z-Average (d.nm): 51.55

Pdl: 0.418

Intercept: 0.914

	Diam. (nm)	% Number	Width (nm)
Peak 1:	25	93.2	36
Peak 2:	98.1	7.8	97.2

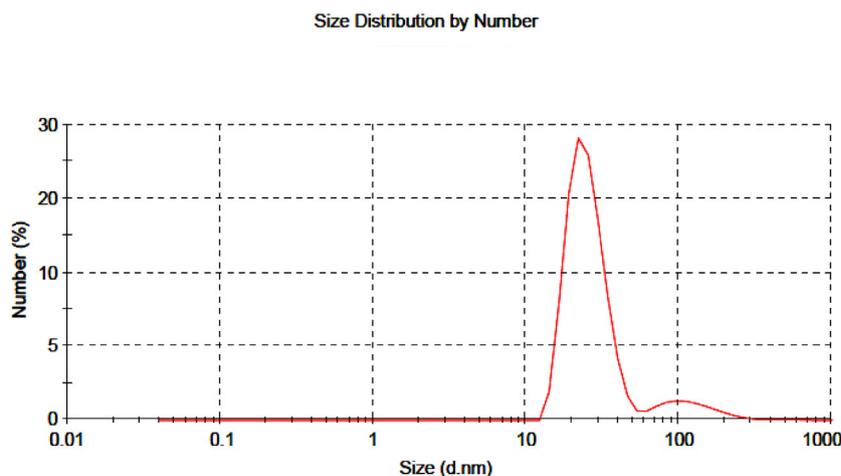


Fig. 6 DLS analysis of prepared silica nanoparticles with SDS at two magnifications.

Table 3 Compressive strengths and standard deviation of compressive test in the presence of different content of silica nanoparticles.

Sample	3-day		7-day		28-day	
	Strength(MPa)	Standard deviation	Strength(MPa)	Standard deviation	Strength(MPa)	Standard deviation
SC-0	23.8	0.61	34.8	0.53	45.2	0.75
SC-1	24.2	0.57	35.1	0.64	47.2	0.67
SC-2	24.6	0.78	36.2	0.59	47.6	0.53
SC-3	26.4	0.63	38.5	0.73	49.9	0.48
SC-4	26.3	0.53	39.1	0.61	52.00	0.56
SC-5	25.0	0.59	35.7	0.81	49.7	0.62
SC-10	23.1	0.83	33.8	0.74	45.5	0.73
SC-15	22.5	0.69	33.3	0.87	44.4	0.64

compressive strength was obtained 24.6, 26.4, 26.3, 25.0, 23.1, and 22.5 MPa respectively. Results demonstrated that 3% and 4% of silica NPs have the highest improvement on the cement mortars. In 10% and 15%, the compressive strength of cement mortars diminished below the blank sample. Compressive strengths of cement mortars after one week and 28 days have been increased more than three days at related %wt. From the results of Fig. 7, it is clear that 4% is the optimum content of silica nanoparticles. The addition 4% contents of silica nanoparticles in cement mortars increase the compressive strength by 10.54% for three days, 12.35% for one week, and 15.04 % for 28 days. The standard deviation of measurements is also provided in Table 3. The compressive strength of cement mortars gradually decreases with further increase in silica nanoparticle contents (e.g. at 5%, 10%, and 15%). This can be related to the agglomeration of nanoparticles in the mix at high contents of silica nanoparticles. Nanoparticles have a high surface area to volume ratio and large curvature. For the presence of dangling bonds at the surface of silica nanoparticles, the agglomeration has occurred through the attractive van der Waals force between them (Ehsani et al., 2017).

The flexural strength tests is one of the main tensile strength test of cement mortars. Flexural strength test determines an un-reinforced cement mortars beam or slab to resist failure

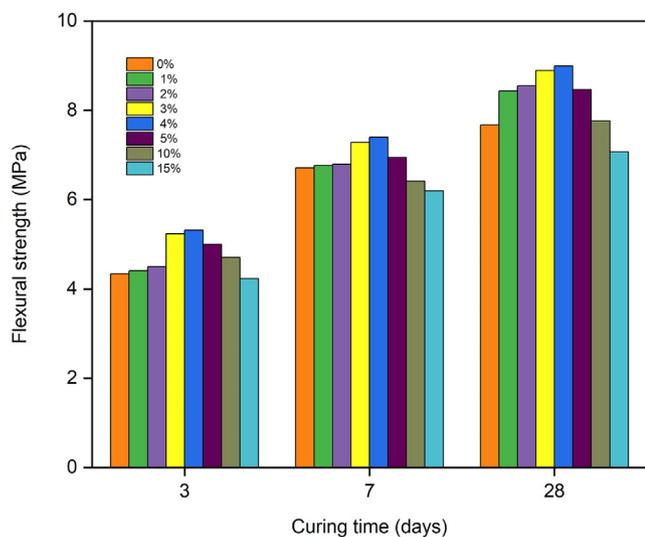


Fig. 8 Amount of flexural strengths of mortar at different dosage of silica nanoparticles in different curing time.

in bending. Fig. 8 show the effect of various silica nanoparticle contents on the flexural strengths of cement mortars on different days. Results showed that 4% is the optimum amount of silica nanoparticles. This improvement has occurred for all test days. Flexural strengths of SC-0 are 4.3, 6.7 and 7.6 MPa after three, seven, and 28 days while flexural strengths for the SC-4 are 5.3, 7.4 and 9.0 MPa after three, seven and 28 days. Surprisingly, the flexural strengths of SC-15 were decreased below the flexural strengths of SC-0 on all test days. Results indicate that adding 4% contents of silica nanoparticles in the same condition increase the flexural strengths by 23.25 % for three days, 9.45 % for seven days, and 18.4 % for 28 days.

4. Conclusion

In summary, silica nanoparticles were synthesized through a non-complicate and fast method. The morphology engineering was carried out via controlling the surfactant and calcination temperature. It is revealed that a prepared sample in the calcination temperature of 500 °C at the presence of SDS as a surfactant has a suitable size and morphology. The morphology engineering significantly enhanced the capability of nano-silica to improve the compressive and flexural strengths of cement mortars. This work also focused on optimizing the amount of nano-silica to achieve the highest mechanical properties. Addition of 4% (%wt) contents of silica nanoparticles in cement mortars increased the compressive strength with 10.75% for three days, 12.1% for seven days and 14.85% for 28 days and increased the flexural strengths by 22.61% for three days, 10.12% for seven days and 17.20% for 28 days. The compressive and flexural strength of cement mortars slightly decreased with further increase in silica nanoparticle contents. Also, by adding 4% silica nanoparticles (SC-4) to the cement paste, the setting time of the sample accelerated to 220 min from 250 min for SC-0.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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