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Study of Compress Strength and Time Setting of Concrete by Additives of Silica Fume and Nano Silica

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In the present study effects of the silica fume and nano-SiO_2 on cement mortars is investigated. Silica fume size is in range of 0.1-0.5 micron and nano-silica is in particle form with 98.31 % of SiO_2 in nano scale. The properties of cement mortar are studied at three water/cement ratios of 0.30, 0.35 and 0.50 and three contents of silica fume particles per cement of 5, 10 and 15 %. For evaluation of nano-silica effects on the cement mortar four water/cement ratios of 0.25, 0.30, 0.35 and 0.50 and four contents of nano-SiO_2 particles per cement of 5, 10 and 15 % are used. The experimental results show that the compressive strengths of mortars with nano-SiO_2 particles are higher than the corresponding mortars containing silica fume for two times of 7 and 28 days. The setting time of mortars with nano-SiO_2 particles are less than the corresponding mortars containing ordinary portland cement and micro silica.

Key Words: Cement, Nano-SiO_2, Silica fume, Setting time, Compressive strengths.

INTRODUCTION

Cement is an inorganic, non-metallic substance with hydraulic binding properties and is used as a bonding agent in building materials[1]. Using the mineral additives in cement-based materials has been grown in the recent years, due to the technical advantages in the final product. The durability and mechanical properties of high performance concrete are mainly dependent on the gradually refining structure of hardened cement paste and the gradually improving paste aggregate interface incorporating additives and admixtures[2-5].

The nano scale particles can result dramatically improvement of different properties in comparison with the conventional grain-size materials of the same chemical composition[6,7]. There are few reports on mixing of nano particles in cement-based building materials. Li *et al.*[6] investigated cement mortars with nano-SiO_2 or nano-Fe_2O_3 to explore their super mechanical and smart (temperature and strain sensing) potentials. Studies have shown that the hydration heat indicates the activation of pozzolanic materials. SiO_2 is one of the cement component indicates a suitable hydration reaction. The hydration reaction is as follows:[8]

$$2SiO_2 + 3Ca(OH)_2 \rightarrow 3CaO{:}2SiO_2{:}H_2O$$

$$Si\text{-}O\text{-}Si + H_2O \rightarrow Si\text{-}OH + Si\text{-}OH$$

SiO_2 has many different possible crystalline structures, which can easily form amorphous materials (*i.e.* materials with no long-range order). Chemical properties of SiO_2 such as hygroscopicity (tendency to react with ambient water) vary tremendously depending on the structure. SiO_2 as three forms are added to the cement mortar: micro-silica, ash-silica, nano-silica in the cement industry due to the increasing effective surface area and high consistency with the cement structure.

The additive of silica, which is the major component of a pozzolan, reacts with calcium hydroxide formed from the hydration of the calcium silicates[9,10]. The rate of pozzolanic reaction will be proportional to the amount of surface area available for reaction. The pozzolanic activity of ash was greatly improved by adding nano-SiO_2[11], which led to an increase in the short and long-term of compressive strength of high volume fly ash concrete[12,13]. Therefore, it is plausible to add nano-SiO_2 of a high purity (99.9 %) and a high Blaine fineness value in order to improve the characteristics of cement mortars[4,5,14]. Using a transmission electron microscope (TEM), it was observed that the particle shapes of cement-based composite materials, with nano-SiO_2 and silica fume, have a high specific surface area[15]. The compositions and microstructure of the cement-based material were also analyzed by X-ray diffraction (XRD) and SEM[16]. The mechanical properties of the mortar were more improved by adding the nano-SiO_2 and silica fume in comparison with the Portland cement[17,18].

The main purpose of the current study is to investigate the influences of some additives such as silica fume and nano silica in compress strength and time setting of cement mortars. Silica fume in range of 0.1-0.5 micron and in powder form with SiO_2 ranging from 85 to 93 % has been used. In addition, nano-silica in the range of 30-80 nm with 98.31 % of SiO_2 is used. These materials are also utilized as the partial replacement for cement or as additives when special properties are desired. The experimental results of compressive strengths of mortars with nano-SiO_2 particles, silica fume and micro silica and setting time of them are compared with each other.

EXPERIMENTAL

The Portland cement of Ilam Cement Industry (Ilam Province, Iran) that produces the best quality of the cement in the world is used. The specific surface area (Blaine fineness), chemical analysis and density are measured by ISIRI-390 standard, ISIRI-2931 and ASTMC188-95, respectively. It is found that silica fume has a specific surface area of 19.2 m^2/g as well as nano silica powder (Nanoseruse company; Tehran, Iran) has solids in the range size of 30 nm-80 nm, average specific surface area of 80 m^2/g and density of 1.16 g/cm^3. Chemical compositions and physical properties of cement and silica components are listed in Table-1.

TABLE-1
CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF CEMENT AND COMPONENT SILICA

Items	Chemical composition (%)		
	PC	Silica fume	Nano-silica
SiO_2	21.40	93.0	98.310
Al_2O_3	5.60	1.2	0.076
Fe_2O_3	2.40	0.7	0.293
CaO	66.10	0.4	0.391
MgO	1.85	0.8	0.050
SO_3	0.13	0.7	0.185
Li_2O	0.85	3.3	0.100
CaO_{free}	1.00		
Physical Properties			
Density (g/cm^3)	3.15	2.41	1.16
Average particle size (d_p)	13 µm	0.1 µm	30-80 nm
Specific surface area (m^2/g)	0.38	19.2	80

PC = Portland cement

The superplasticizer admixture was a lignosulfonic acid sodium salt based (Sigma-Aldrich Company-USA and Merck Company-Germany) with molecularity between 5200 and 8000 g/mol. A commercial sand is composed by four size fractions (1.2, 0.6, 0.3 and 0.15 mm) that each of them containing 25 wt % and it is used as aggregation in the mortar. Accordingly, silica fume used in this study is the powder with 93 % of SiO_2 and particle size of 0.1-0.5 micron to perform a comparison between silica fume and nano-SiO_2 in terms of compressive strength of cement mortar.

Cement Portland and superplasticizer: The Bogue composition of the clinker and the cement powder shown in Table- 2. The used superplasticizer was a commercial lignosulfonic acid sodium salt (liquid solution, water content of 98 % and molecularity of 5200).

Silica fume: Silica fume has been used either as a partial replacement for cement or as an additive when special properties are desired[19,20]. A maximum content of silicon dioxide (SiO_2) is a property that most frequently is specified. Accordingly, the used silica fume is the powders form with 93-95 % of SiO_2, a particle size of 0.1-0.5 µm and 19.2 m^2/g Blaine fineness. Scanning electron micrographs of silica fume is shown in Fig. 1.

TABLE-2
PHASES AND CLINKER MODULE OF CEMENT PORTLAND

Phase	Clinker (%)	Cement Portland
C3S	56.10	60.07
C2S	27.25	21.26
C3A	11.13	11.38
C4AF	1.52	0.97
Module		
SIM	4.83	4.81
ALM	9.04	14.06
LSF	90.68	93.11

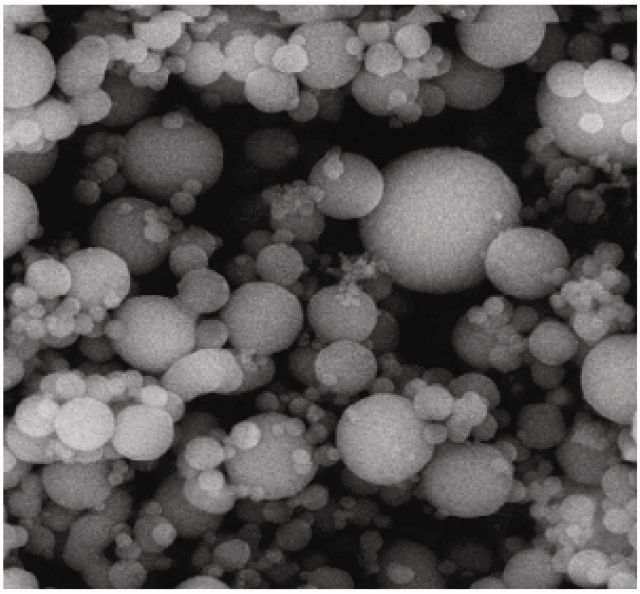
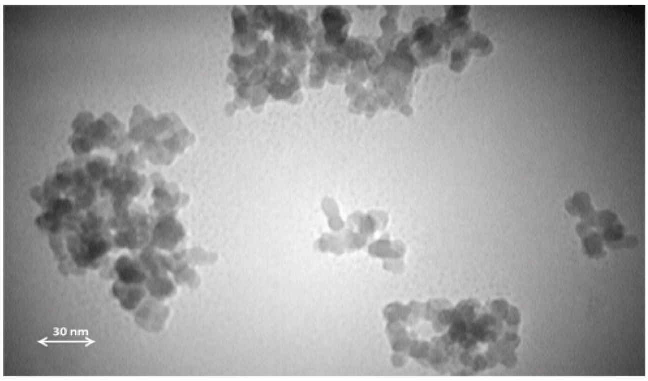


Fig. 1. Sample of SEM photograph of silica fume

Nano silica: Nano-SiO_2 used in the current research is in particle form with 98.3 % SiO_2 and a particle size range of 30-80 nm. The nano-silica has the density of 1.16 g/cm^3 and specific surface area of 80 m^2/g. Two arbitrary of scanning electron micrographs of the aggregated nano-SiO_2 is shown in Fig. 2.



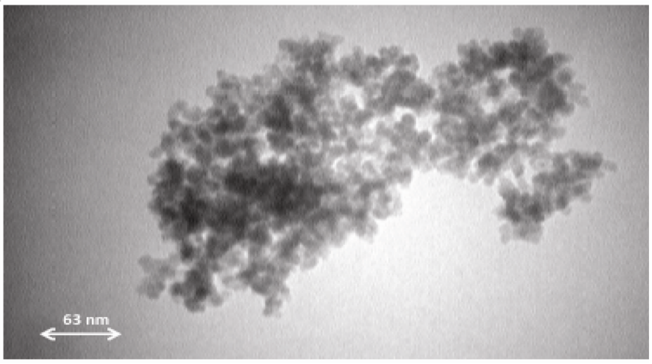


Fig. 2. Sample of SEM photograph of nano-SiO_2

Sand: The commercial sand used in this study is from sand and Blast Ilam company composed by four size fractions (0.2, 0.4, 0.6 and 1 mm), each of them corresponding to 25 wt % of mortar is used as aggregation in the mortar.

Preparation of paste specimen: Cement pastes incorporating nano-SiO_2 or silica fume are prepared at a standard consistency using a planetary mixer similar to others[21,22]. Five different water/cement ratios of 0.20, 0.25, 0.30, 0.35 and 0.45 and three contents of nano-SiO_2 particles per cement of 5, 10 and 15 % are considered in the experiments. The compressive strength of cement mortar with the silica fume as an additive is evaluated at water/cement ratio of 0.45 in three contents of silica fume per cement of 5, 10 and 15 % for comparing with the mortar containing nano-SiO_2 particles similar to Toutanji and El-Korchi[23] work. For the whole pastes, superplasticizer ratio of 0.8 % is used. The cement with nano-silica or silica-fume is fully mixed under the condition of dry process before hand. The additives are completely mixed to the cement in dry condition, after that water is added till the mixture achieves the mentioned water/cement ratios. The cement mortars are mixed in a rotary mixer. The mixing procedure is as follows:

The nano-SiO_2 particles or silica fume are mixed with the mortar at high speed of the mixer for 1 min. Then the mixing is continued at a medium speed for one anymore minute. Along the mixing in a medium speed, sand is added into the mixture gradually. The superplasticizer is added and stirred at high speed for additional 30 s. Moreover, mixing process is allowed to rest for 90 s and then it is continued for additional 1 min.

Test of compressive strength of cement specimens: Three prism specimens of 4 cm × 4 cm × 16 cm were made for compressive strength (according to EN196-1). All paste specimens, which are cured at 20 ± 2 ºC above 90 % relative humidity, are demolded after 24 h and then all specimens are immersed in water at 20 ± 1 ºC for 7 and 28 days. The prisms are tested in terms of compression strength by EN 1015-11.

Test of consistency and setting time of fresh pastes: The consistency and the setting time of fresh pastes were tested by EN 196-3[24]. The consistency is determined by putting the paste in a mould consisting of a steel ring (40 mm in height) on a sheet of glass and by determination of the plunger penetration depth applied on the top surface of the paste specimen. The initial and the final setting times are determined with the needle of the Vicat apparatus.

RESULTS AND DISCUSSION

Table-3 shows setting time of cement mortars with silica fume and ordinary Portland cement at water/cement ratio of 20 %. It can be seen that the initial time increases with increasing silica fume percentage, while the final setting time and differential time decreases with increasing silica fume percentage. The setting time of cement mortars with silica fume results are presented in the Fig. 3.

TABLE-3
SETTING TIME OF CEMENT MORTARS WITH SILICA FUME AND ORDINARY PORTLAND CEMENT (OPC) AT WATER/CEMENT OF 20 % RATIO

Sample	Initial time (min)	Final time (min)
OPC	168	263
Silica fume (5 %)	282	317
Silica fume (10 %)	305	339
Silica fume (15 %)	325	364

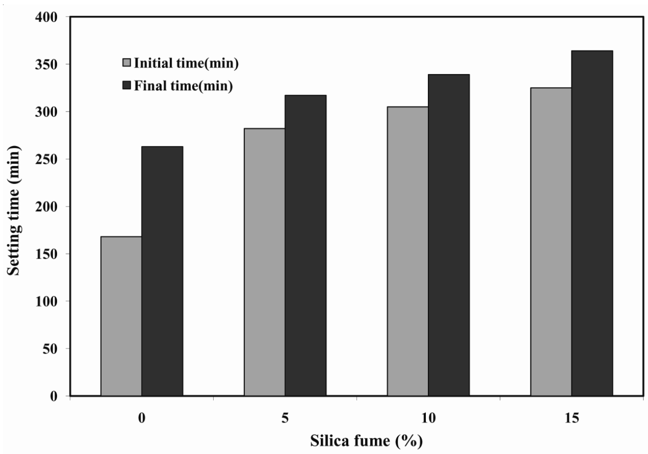


Fig. 3. Sitting time of cement mortar with silica fume (water/cement ratio of 20 %)

Table-4 shows setting time of cement mortars with nano-SiO_2 and ordinary Portland cement (OPC) at water/cement ratio of 20 %. The setting time in the two set of times, initial and final times, is shown in Fig. 4. It can be seen that the initial time decreases by increasing nano-SiO_2 percentage, moreover the final time decreases by increasing nano-silica and decreasing differential time.

TABLE-4
SETTING TIME OF CEMENT MORTARS WITH NANO–SiO_2 AND ORDINARY PORTLAND CEMENT (OPC) AT WATER/CEMENT RATIO OF 20 %

Sample	Initial time (min)	Final time (min)
OPC	168	263
Nano-SiO_2 (5 %)	128	184
Nano-SiO_2 (10 %)	109	167
Nano-SiO_2 (15 %)	98	146

Table-5 shows compressive strength of cement mortars with silica fume and ordinary Portland cement as a function of water/cement ratio. Moreover, Fig. 5 shows compressive strength of cement mortar with silica fume. It can be found that the compressive strength development of mortars mixture containing micro-silica particles are higher than ordinary

Portland cement that of the control cement mortars with the same water-cementations in the two case compressive strength at 7 and 28 days. The difference of strength development in mortars can be attributed to the pozzolanic reactions.

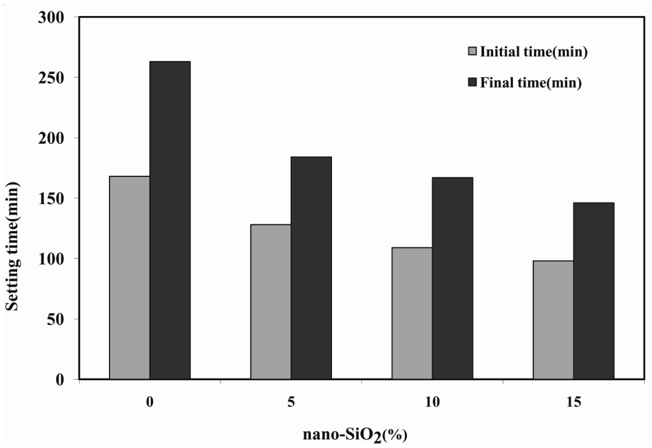


Fig. 4. Sitting of cement mortars with nano-SiO_2 (water/cement ratio of 20 %)

TABLE-5
COMPRESSIVE STRENGTH OF CEMENT MORTARS WITH SILICA FUME AND ORDINARY PORTLAND CEMENT (OPC) AS A FUNCTION OF WATER/CEMENT RATIOS

Silica fume (%)	Water/ cement (%)	Compressive strength (Mpa) 7 days	Compressive strength (Mpa) 28 days
OPC (0 % silica fume)	20	67.1	77.7
SF (5)	20	66.3	94.4
SF (10)	20	65.3	98.3
SF (15)	20	65.2	100.3
OPC (0 % silica fume)	45	17.1	23.5
SF (5)	45	22.5	34.2
SF (10)	45	23.1	35.7
SF (15)	45	25.8	36.2

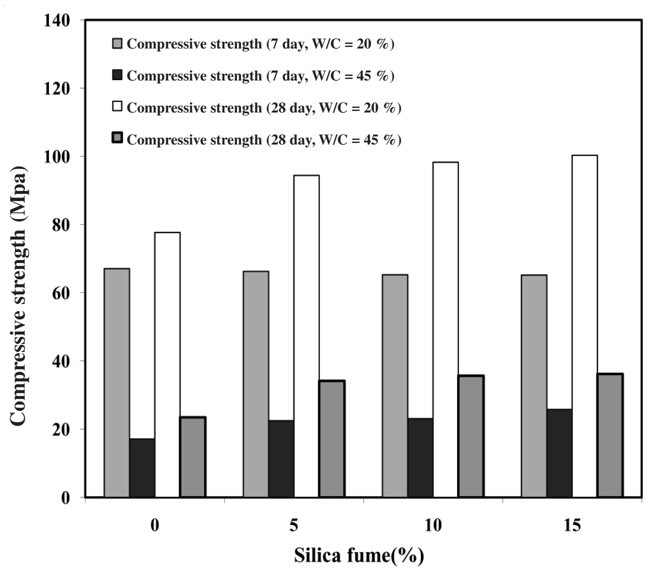


Fig. 5. Compressive strength of cement mortars with silica fume

As mentioned above, it implies that the nano-particles are more effective in pozzolanic reactions than silica fume. In addition, the nano-SiO_2 will fill pores and increases the strength more than silica fume. Therefore, adding nano-SiO_2 leads to improvement of cement mortar characteristics. Compressive strength of cement mortars with nano-SiO_2 and ordinary Portland cement as a function of water/cement ratios are summarized in Table-6. As can be seen in that Table-6, for the water/ cement ratios of 20-45 %, the strength of the mortar increases with increasing the nano-SiO_2 content from 5 to15 %.

TABLE-6
COMPRESSIVE STRENGTH OF CEMENT MORTARS WITH NANO-SiO_2 AS A FUNCTION OF WATER/CEMENT RATIOS

Nano–SiO_2 (%)	Water/ cement (%)	Compressive strength (Mpa) 7 days	Compressive strength (Mpa) 28 days
OPC (0 % nano–SiO_2)	20	67.7	75.9
Nano–SiO_2 (5)	20	83.8	96.2
Nano–SiO_2 (10)	20	80.1	101.3
Nano–SiO_2 (15)	20	66.2	92.5
OPC (0 % nano–SiO_2)	25	55.23	73.6
Nano–SiO_2 (5)	25	61.4	78.4
Nano–SiO_2 (10)	25	77.8	98.7
Nano–SiO_2 (15)	25	64.3	91.2
OPC (0 % Nano–SiO_2)	30	51.0	65.4
Nano–SiO_2 (5)	30	52.4	69.5
Nano–SiO_2 (10)	30	56.2	82.6
Nano–SiO_2 (15)	30	61.2	80.4
OPC (0 % Nano–SiO_2)	35	38.8	45.6
Nano–SiO_2 (5)	35	39.1	48.2
Nano –SiO_2 (10)	35	40.2	51.4
Na no–SiO_2 (15)	35	49.8	61.3
OPC (0 % Nano–SiO_2)	45	17.1	23.5
Nano–SiO_2 (5)	45	28.4	41.3
Nano–SiO_2 (10)	45	29.1	43.4
Nano–SiO_2 (15)	45	37.2	51.3

The compressive strength of cement mortars with nano-SiO_2 and ordinary Portland cement as a function of water/ cement ratios are also presented in the Fig. 6. However, it must be noted that with increasing the content of nano-SiO_2, the dosage of superplasticizer should be added to the mortar to make sure the cement mortar not to be excessive dry. Higher amount of nano-SiO_2 may actually decrease the strength of composites instead of improving it. The result of this study has shown that 15 % nano-SiO_2 has no adverse effect on strength, but this percentage cannot be considered as the optimum value for maximizing strength. In the current study, the optimum content value of nano-SiO_2 is 10 % at water/ cement ratio of 20 %.

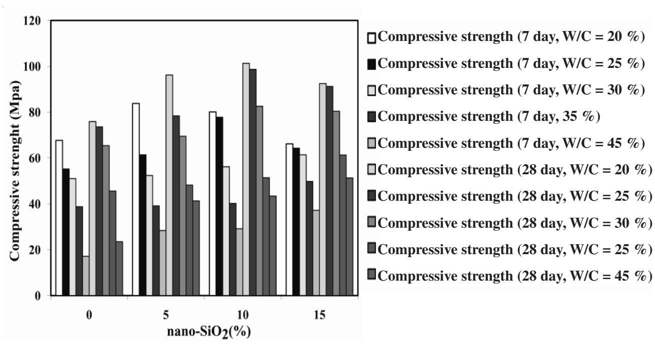


Fig. 6. Compressive strength of cement mortar with nano-SiO_2

Conclusion

In the current study, the influences of some additives of silica fume and nano-SiO_2 in compress strength and time setting cement mortars was investigated. The additives were silica fume SiO_2 in size range of 0.5-1 micron and nano-silica in particle form with 99.9 % of SiO_2 in nano scale. The properties of cement mortar were evaluated at various water-cement ratios. The experimental results showed that the initial time increases with increasing silica fume percentage, while the final setting time and differential time decreases with increasing silica fume percentage. In addition, the experimental results showed that the initial time decreases by increasing nano-SiO_2 per cent, moreover the final time decreases with increasing nano-silica and decreasing differential time. The experimental results indicated that the compressive strengths of mortars with nano-SiO_2 particles are higher than those of mortars containing silica fume and micro silica at both times of 7 and 28 days. The experimental results showed that the optimum value for content of nano-SiO_2 is 10 % at water/cement ratio of 20 %.

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