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Research Article

Setting Time, Compressive Strength, and Photon Attenuation Properties of Cement Mortars Produced with Nano-SiO_2

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Setting time

Abstract: In this study, initial and final setting time, compressive strength, and photon attenuation properties of cement mortar samples produced with 0.5%, 1%, 2%, and 4% nano-scale SiO_2 addition were investigated. For this purpose, cement mortar samples were prepared and cured for 1, 2, 7, 28, and 90 days. Increases in compressive strength values were observed in the early curing ages, with a slight decrease of 3.17% and 0.33% for low nano-SiO_2 rates (0.5% and 1%) in later ages. In addition, these results were evaluated with Scanning Electron Microscope (SEM) images. In the samples added 4% nano-SiO_2, there was a 13.3% and 9.09% reduction in the initial and final setting times, respectively. Furthermore, mass attenuation coefficients were compared for different cure ages. It was aimed to determine the effect of the curing time on the photon attenuation property by adding nano-SiO_2 to the concrete.

Nano-SiO_2 ile Üretilen Çimento Harçlarının Priz Süresi Basınç Dayanımı ve Foton Zayıflatma Özellikleri

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Anahtar Kelimeler
Basınç dayanımı,
Çimento harcı,
Kütle zayıflatma katsayısı,
Nano-SiO_2,
Priz süresi

Öz: Bu çalışmada, %0.5, %1, %2 ve %4 nano boyutlu SiO_2 ilavesi ile üretilen çimento harcı numunelerinin priz başlangıç ve bitiş süreleri, basınç dayanımları ve foton zayıflatma katsayıları incelenmiştir. Bu amaçla çimento harcı numuneleri hazırlanmış ve 1, 2, 7, 28 ve 90 gün kür edilmiştir. Erken kürleme yaşlarında basınç dayanımı değerlerinde artışlar gözlenirken, ileri yaşlarda ve düşük nano-SiO_2 eklemelerinde (%0.5 ve %1) basınç dayanımları %3.17 ve %0.33 oranında azalmıştır. Ayrıca bu sonuçlar Taramalı Elektron Mikroskobu (SEM) görüntüleri ile değerlendirilmiştir. %4 nano-SiO_2 ilave edilen numunelerde priz başlangıç süresinde %13.3, priz bitiş süresinde ise %9.09 oranında azalma olmuştur. Ayrıca farklı kürlenme yaşları için kütle zayıflatma katsayıları karşılaştırılmıştır. Çalışmada kür süresinin, betona Nano SiO_2 eklenmesi ile foton zayıflatma özelliğine yapacağı etkinin belirlenmesi hedeflenmiştir.

1. Introduction

Concrete, used three tons per year by each person, is the most consumed material after water (Gagg, 2014). Due to cement production constituting 5-7% of the total CO_2 emissions in the world, reducing CO_2 emissions and energy costs has become an important issue for researchers (He et al., 2019). In order to reduce the CO_2 release and energy consumption in the production of concrete, researchers have investigated materials that can partially replace cement to improve properties (e.g., fly ash, silica fume, granulated blast furnace slag) or alternatives to it, such as geopolymer (Singh et al., 2013).

Nanotechnology is the understanding, controlling, and restructuring of a substance at the nanometer level (less than 100 nm) to produce materials with new features and functions (Sanchez & Sobolev, 2006). Nano concrete is defined as concrete produced with nanomaterials or the addition of nanomaterials of less than 500 nm (Norhasri et al., 2017).

According to Givi et al. (2010), nano-SiO_2 addition in concrete increases the compressive flexural and tensile strength for all curing ages. Nano-SiO_2 of 15 nm and 80 nm size can be replaced by 2% maximum cement, and the optimum replacing percentage is 1% and 1.5% for the above sizes, respectively. The smaller size (15 nm) is better for enhancing early age strengths with respect to the larger size (80 nm), which gives good results in 90 days. In their study, Ltifi et al. (2011) used nano SiO_2 with an average size of 9 nm at 3 and 10% ratios in cement mortar mixtures. In their study, in which they examined the setting time and strength of 3, 7, 14, and 28 days, they reported that Nano SiO_2 made the cement paste thicker, accelerated the hydration process, and increased the compressive strength. Another study on cement replacement by nano-SiO_2 showed improved compressive strength and that the optimum content is 5% (Behfarnia & Salemi, 2013). On the other hand, Nik & Omran (2013) investigated the compressive strength of self-compacted concrete using fibers and nano-SiO_2. They reported that a 4% nano-SiO_2 percentage is appropriate. According to Hou et al. (2015), nano-SiO_2 increases the hydration process at early ages, and at later ages, the hydration rate decelerates because of the compact form of pozzolanic products by nano-SiO_2. Jankovic et al. (2016) reported that while 2% of nano-SiO_2 compressive and flexural strengths increase, between 2–5% decrease. According to Givi et al. (2011), 15-nm-sized nano-SiO_2 can be used up to 2% to replace cement to obtain low water permeability and initial and final setting times. In their studies, Fallah & Nematzadeh (2017) reported that 2% nano-SiO_2 improves mechanical properties.

Hassanzadeh & Sadat Kiai (2018) stated that adding nano-sized SiO_2 to concrete improves nuclear properties and can be helpful in radiation shielding. In the study, they reported that the average relative difference between the linear attenuation coefficient for specimens with nano-SiO_2 material between simulation and theoretical and experimental results was 6.4% and 5.5%, respectively. They also reported that when nano-SiO_2 was increased up to 1.5% and $MnFe_2O_4$ temperature was increased to 673 K, the "μ" value increased at all energies and the photon dose rate decreased to 9.2% and 3.7% for $MnFe_2O_4$ and SiO_2 at 0.511 and 1.274 MeV gamma ray energies, respectively. According to Elsharkawy & Sadawy (2016) adding nano-sized SiO_2 to concrete by up to 1.5% increased the total γ-ray attenuation coefficient μ for all energies. They applied a 28-day cure for the samples. Zaghloul & Elwan (2017) state that nano SiO_2 in concrete mix is effective in increasing γ radiation attenuation and reduces the required thickness for shielding. In this study, cure periods were applied as 7 and 28 days. They reported that they used 1%, 3%, 5% and 7% instead of cement in mixtures and that 3% nano-silica improved the physical, mechanical and radiation shielding properties.

In this study, nano-sized SiO_2 was replaced by cement to investigate compressive strength, setting time, and photon attenuation propertiesof cement mortars. Considering past studies above, a nano-SiO_2 content rate of 0–4% was chosen for a mixture. In addition, the results were interpreted with images taken by scanning electron microscopy (SEM).

The main aim of the study is to determine the effect of the curing time of the cement mortar on the photon attenuation property of the samples added nano-SiO_2. In past studies on photon attenuation of nano SiO_2, concrete curing times were generally taken as standard. This study aims to reveal the especially effect of curing time (1, 2, 7, 28, 90 days) on this property. In addition, since the most important mechanical property of concrete is compressive strength and nano-SiO_2 is known to affect the setting time of concrete, the variation of these two parameters was also investigated.

2. Material and Methods

2.1. Materials

The mixture was prepared using CEM I 42.5 Portland cement, CEN-Standard Sand, and tap water. In the mixtures, except for the control group, nano-sized Silica SiO_2 (30 nm average size) and superplasticizer were used. The chemical and physical properties of the cement and nano-silica are given in Table 1, Table 2 and Table3 respectively. A scanning electron microscope (SEM) view of the nano-SiO_2 is given in Figure1.

Table 1. Chemical analysis of the cement

Material	Percentage (%)
SiO_2	20.78
Al_2O_3	5.10
Fe_2O_3	3.15
CaO	60.67
SO_3	2.79
Heat loss	2.10
Na_2O	0.35
K_2O	0.80
Free CaO	1.48

Table 2. Physical analysis of the cement

Physical Properties	
Fineness 45 µm test sieve residue (%)	1.5
Specific gravity (gr/cm^3)	3.09
Specific surface (cm^2/gr)	3739

Table 3. Physical properties of Nano-SiO_2

Average Size (nm)	Purity (%)	Colour	Shape	Bulk Density (gr/m3)	True Density (gr/m3)
30 nm	99.9	White	Almost spherical	~ 0.063	~ 2.2 – 2.6

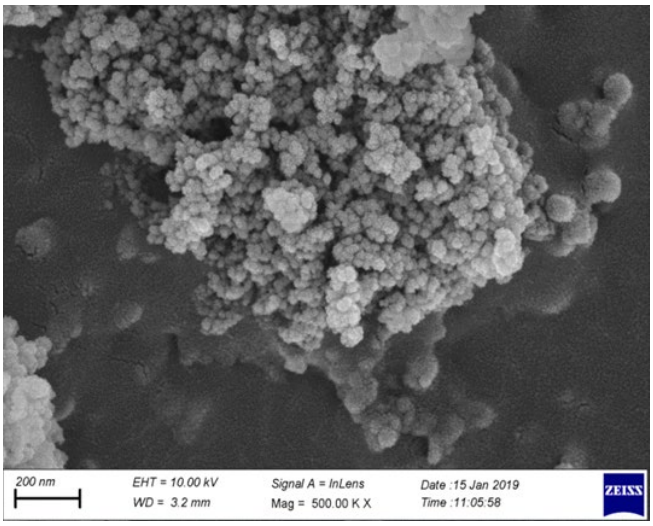


Figure 1. SEM view of the nano-SiO_2.

2.2. Method

2.2.1. Mixture preparation

All the mixtures were prepared according to TS-EN 197-1 (TSE, 2012) (Table 4). According to this standard, the mix proportions were constant. For the mixing process, a cement mixer in accordance with TS-EN 197-1 (TSE, 2012), shown in Figure 2 (a), was used. In this respect, water was put in the mixing vessel, and cement was subsequently added. The mixer was started immediately at low speed for 30 s. The sand was added continuously for 30 s. The mixer was set to high speed and mixed for 30 s. The mixer was stopped after 1 min 30 s. The mortar that adhered to the walls and the base was scraped off with a rubber scraper, and then mixing continued at high speed for 60 s. This process was repeated for the mixtures with nano-silica addition. However, to disperse the nano-silica nicely, water, nano-silica, and superplasticizer were mixed at high speed (120 rpm) for 2 min before adding this mixture to the main mixture. After the mixture process, a fresh mortar was moulded in standard cement moulds (40x40x160 mm in size), which were fixed to a vibrating device (Figure 2(b)). Fresh mortar was placed in the moulds (Figure 2(b)) in two layers. Each layer was vibrated for 60 falls, and after finishing the surface, the layers were put in a moisture cabin (Figure 2(d)) for 24 hours. As seen in Figure 3, demoulded hardened mortar prisms were put in the curing water at 20 ± 1ºC for 1, 2, 7, 28, and 90 days.

In the trial mixtures, it can be observed that the nano-silica addition reduces the workability of fresh mortars. This situation is consistent with the literature by Ltifi et al. (2011). In order to increase workability, a superplasticizer was used. The amount of superplasticizer was determined by trial and error method.

Table 4. Mixture proportions

Mixture	Sand (%)	Cement	Water	Nano-sized SiO_2 by weight (%)	Nano-Sized Silica (gr)	Superplasticizer (gr)
M0	1350	450	225	0	0	0
M0.5	1350	447.75	225	0.5	2.25	0.45
M1	1350	445.5	225	1	4.5	1.35
M2	1350	441	225	2	9	2.70
M4	1350	432	225	4	18	7.20

Figure 2. Mixture process.

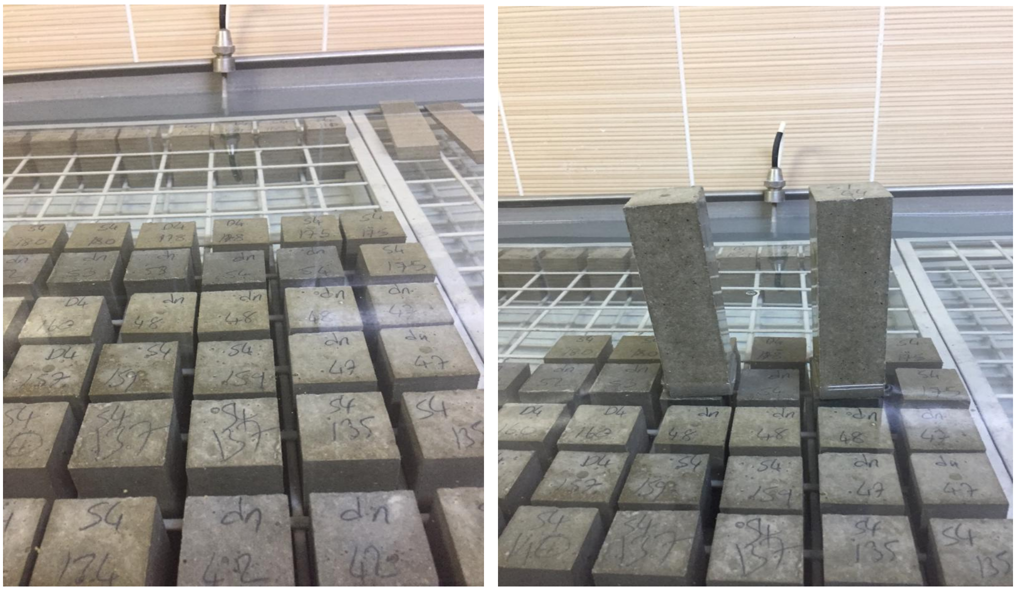


Figure 3. Curing of the samples.

2.2.2. Setting time experiment

For control samples, 500 gr of cement was measured and weighed to a sensitivity of 1 gr. Next, 125 gr. of water was weighed and placed in a mixing bowl. As seen in the Figure 2(a) the mixer was immediately run at low speed for 90 s. At the end of 90 s, it was stopped for 15 s; meanwhile, the cement paste which had adhered to the inner walls of the mixer container was peeled off and added to the mixture. This process was repeated for each nano addition percentage of replaced cement. The amount of superplasticizer, determined by trial and error, was added to the mixtures with nano addition. The mixer was rerun at low speed for 90 s. The total working time of the mixer was 3 min. Initial and final setting times were determined by a vicat tool (Figure 4) according to needle penetration depths. Penetration depths were determined by measuring in 10-minute periods.

Figure 4 Setting time experiment (Using Vicat Tool).

2.2.3. Compressive strength experiment

For compressive strength determination, as seen in Figure 5, a standard press was used. The load was increased smoothly (2400 ± 200 N/s) until the prism broke. The breaking load was saved, and the compressive strength values were calculated by dividing the force into the cross-section area.

Figure 5. Compressive strength experiment.

2.2.4. Imaging by scanning electron microscopy

Microstructures of the samples were examined using Scanning Electron Microscopy (Zeiss Sigma 300).

2.2.5. Radiation shielding experiment

Hardened samples were exposed to radiation by a ^{60}Co radiation source that emits 1250 keV photons as shown in Figure 6(a), the photon transmissions were measured using a farmer-type ion chamber (0.6 cc PTW^{TM}), and a as seen in Figure 6(b) Unidos Electrometer was used for dose readings Without sample (I_0), readings were done first, and then readings with samples were made (I), using the formula (1)

$$I = I_0 e^{-\mu x} \quad (1)$$

'μ' (photon attenuation coefficient) values were calculated.

Mass attenuation values were calculated by dividing the photon attenuation coefficient into density as 'μ/ρ'.

Figure 6. a) Radiation experiment (I-Radiation source, II-Collimator (5cmx5cm), III-Lead collimator with 1cm^2 opening, IV-Phantoms) b) Unidos E electrometer (I) and Control panel of the radiation source (II).

3. Results and Discussion

3.1. Setting time

Nano-silica content between 0.5 and 2% did not cause significant changes in start and finish setting time. However, 4% nano-silica content significantly decreased initial and final setting time, 13.33% and 9.09%, respectively (Table 5, Figure 7). This result is consistent with Ltifi et al. (2011) and Qing et al. (2007).

Table 5. Setting time results

	M0 (min)	M0.5 (min)	M1 (min)	M2 (min)	M4 (min))
Setting Time (Start)	225	220	225	230	195
Setting Time (Finish)	275	280	285	285	250

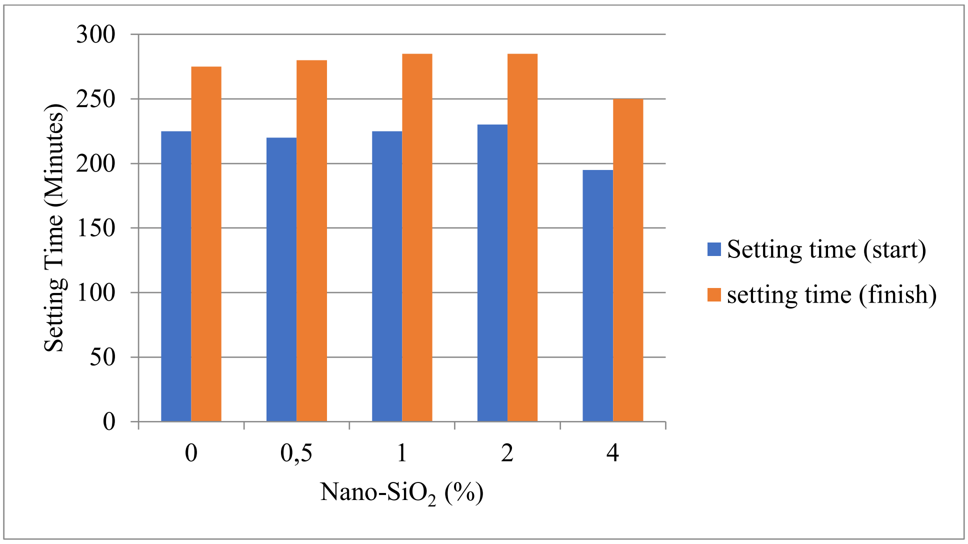


Figure 7. Setting time variation with Nano-SiO_2 content.

3.2. Compressive strength results

Compressive strength test results and change rates according to control samples are given in Tables 6 and 7. The graphical representation of the results is shown in Figure 8.

Table 6. Compressive strength results

Curing age(day)	M0 (MPa)	M0.5 (MPa)	M1 (MPa)	M2 (MPa)	M4 (MPa)
1	14.03	15.38	16.40	16.73	15.98
2	27.28	32.30	31.73	33.53	34.40
7	44.48	45.23	47.55	48.40	53.15
28	53.70	54.93	56.78	59.50	63.45
90	60.25	58.10	60.05	64.43	64.55

Table 7. Compressive strength difference versus control samples (%)

Curing age(day)	M0 (%)	M0.5 (%)	M1 (%)	M2 (%)	M4 (%)
1	-	9.63	16.93	19.25	13.9
2	-	18.42	16.32	22.91	21.25
7	-	1.69	6.91	8.83	19.51
28	-	2.28	5.73	10.80	18.16
90	-	-3.57	-0.33	6.93	7.14

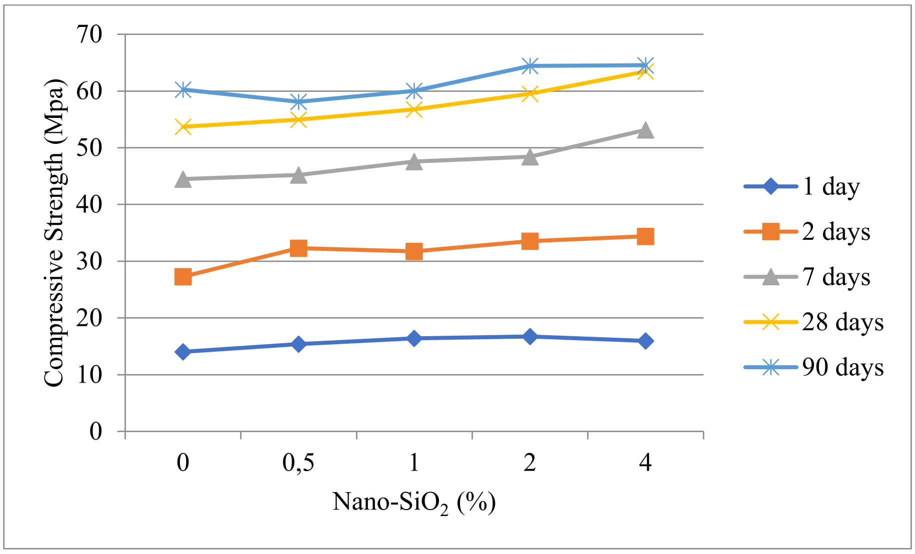
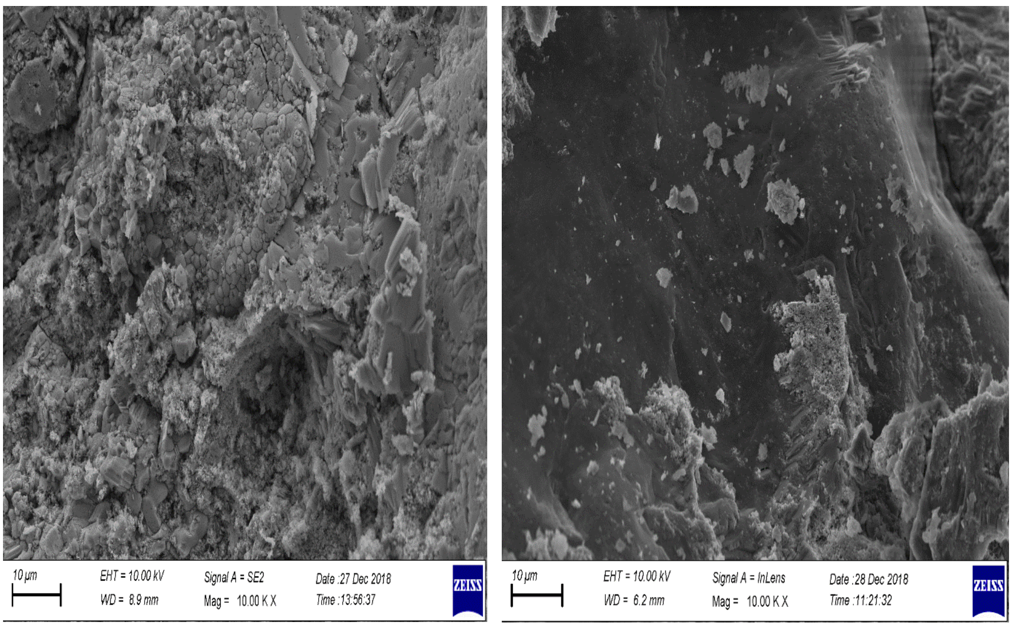


Figure 8. Compressive strength results.

When the results were examined, the following was found:

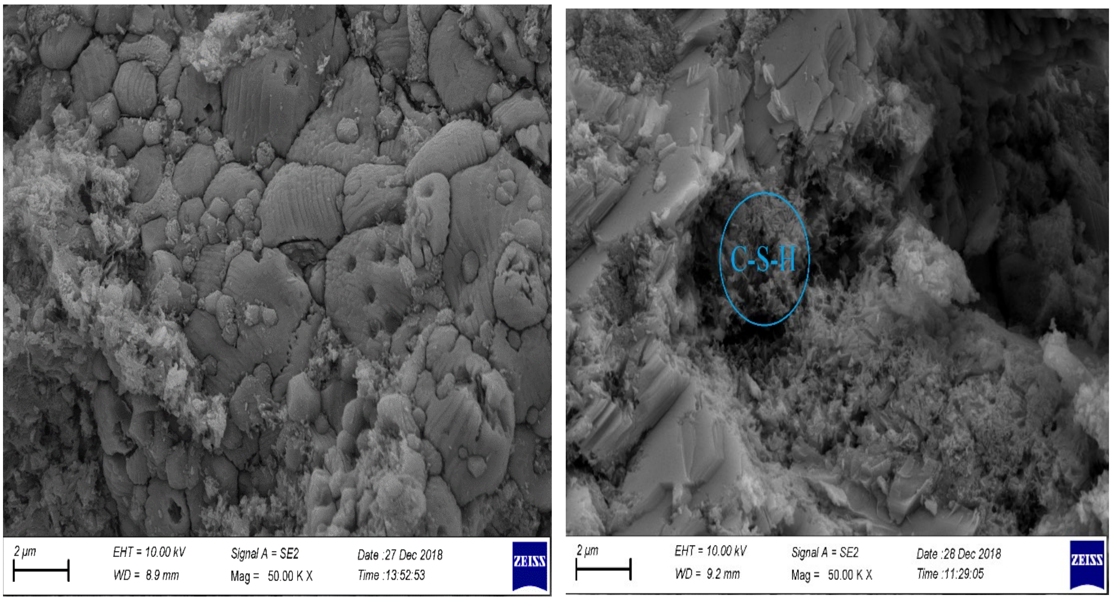
The mortars containing 0.5, 1, 2, and 4% nano-silica increased their compressive strengths in early curing ages (1 and 2 days) compared with control samples. The highest increase investigated was in the samples that contained 2% nano-silica with a 2-day curing age; the increase was 22.91% (Table 6, Table 7, Figure 8). When the 10000 and 50000 magnification SEM images of the samples cured for two days are examined (Figure 9), it is seen that the samples containing 2% Nano-SiO_2 have less voids and denser microstructure and have more calcium silicate hydrate (C-S-H) which could be due to high pozzolanic activity and the filler effect of the nano-sized SiO_2,which causes increasing in compressive strength (El-Gamal et al., 2017; Nik & Omran, 2013; Zhang et al., 2017; Wu et al., 2017; Shih et al., 2006; Behfarnia & Rostami, 2017; Beigi et al., 2013; Ltifi et al., 2011).

For early ages, in the 4% nano addition, the rate of increase of compressive strength was lower than for the 0.5%, 1%, and 2% samples.



a) 0% Nano-SiO_2 b) 2% Nano-SiO_2

Figure 9. SEM images at 10000 magnification of samples cured for two days.

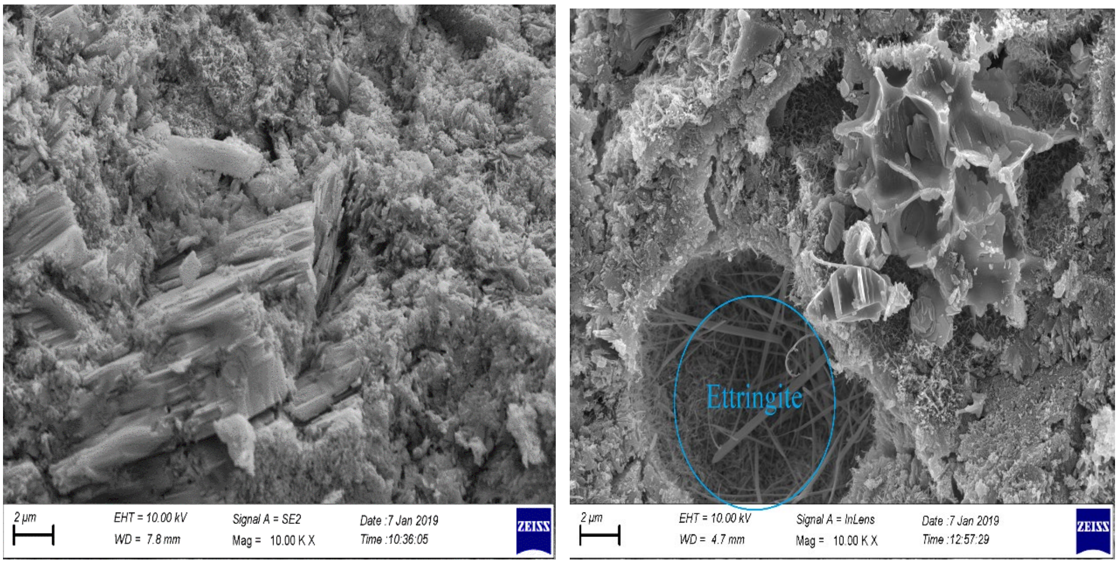


a) 0% Nano-SiO_2 b) 2% Nano-SiO_2

Figure 10. SEM images at 50000 magnification of samples cured for two days.

For the 7- and 28-day curing ages, increasing nano silica content from 0.5 to 4% increased the compressive strength values. The highest increase was found in samples with 4% nano content cured for seven days: 19.51 % (Table 6, Table 7).

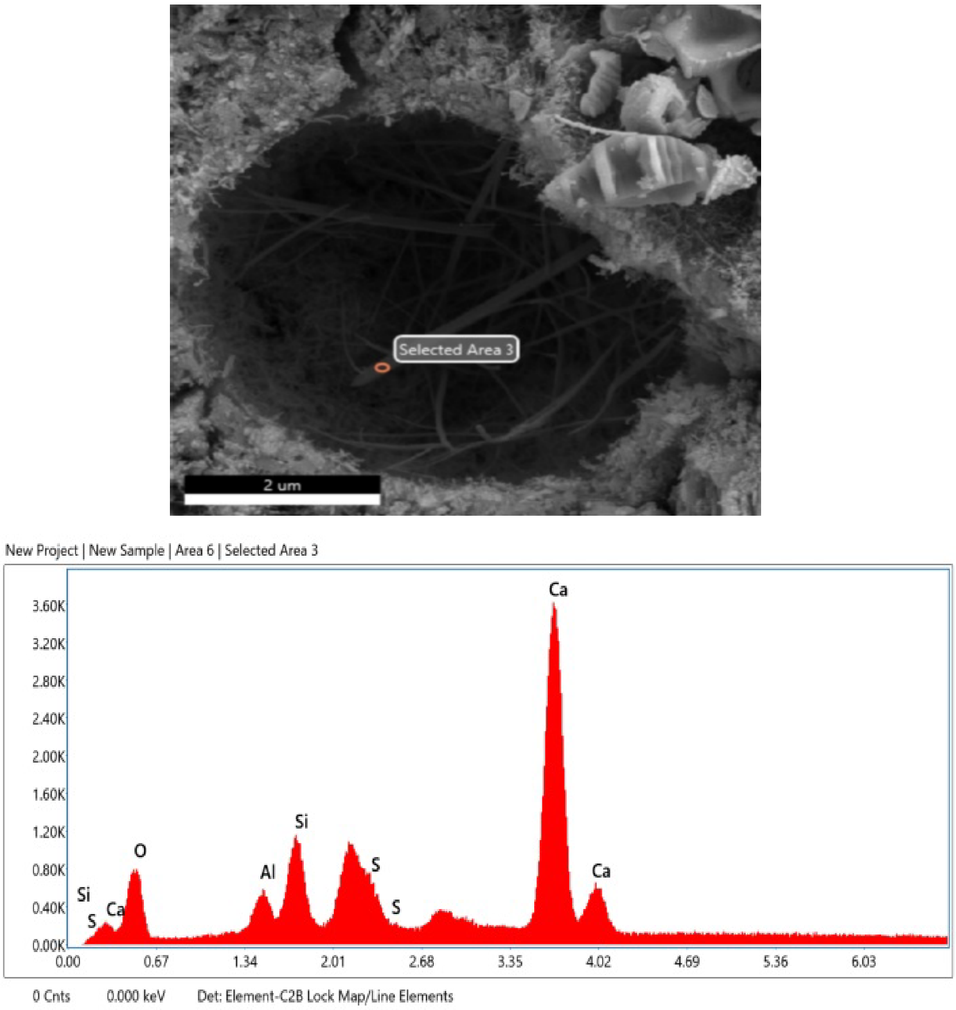
For the later curing age (90 days), few decreases in compressive strength were obtained for the samples containing 0.5 and 1% nano-silica: 3.57% and 0.33%, respectively. When the 10000 magnification SEM images of the samples cured for 90 days were examined (Figure 11), microvoids and ettringite formation were observed in the sample containing 0.5% nano-silica. This could be the reason for the decrease in compressive strength.



a) 0% Nano-SiO_2 (6-1) b) 0.5 % Nano-SiO_2 (7)

Figure 11. SEM images at 10000 magnification of samples cured for 90 days.

In the 2 and 4% nano-silica content samples, increased compressive strength values were found at 6.93% and 7.14%, respectively. The compressive strength values of later curing ages were increased by nano-silica, but they were lower than those of earlier ages (Table 6, Table 7).



Smart Quant Results

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
O K	10.59	21.69	205.58	11.26	0.0254	1.1635	0.2062	1.0000
AlK	2.47	3.00	137.44	6.02	0.0215	1.0192	0.8499	1.0048
SiK	5.27	6.14	296.16	4.20	0.0496	1.0391	0.8999	1.0069
S K	11.87	12.12	460.01	3.94	0.1160	1.0130	0.9526	1.0128
CaK	69.80	57.05	1160.69	3.48	0.6674	0.9674	0.9875	1.0009

Figure 12. EDX analysis result for the ettringite region.

Figure 12 shows the edx analysis of the region estimated to be ettringite. It is seen that there is 11.87% sulfur, 69.80% calcium and 5.27% silicon in the structure. This result supports the idea that the structure is ettringite.

3.3. Radiation shielding results

1. In the control samples, the mass attenuation coefficient values increased as the curing age increased. The highest values of mass attenuation coefficient were obtained for 90 days curing age at 2.67 %. The likely reason is that the curing of the concrete causes C-S-H formation, and the mass attenuation coefficient reaches higher values (Table 8, Table 9, Figure 13).

2. Adding 0.5% nano-Silica increased mass attenuation coefficient values at all curing ages compared with control samples by 0.6%, 1.24%, 1.16%, 0.002%, and 1.65%, respectively. This group obtained the most significant change at 90 days of curing age. However, the increase at 28 days is meager (Tables 8 and 9, Figure 13). In the literature, it has been reported that nano SiO_2 at rates such as 1.5% and 3% causes improvements in photon attenuation property of concrete (Hassanzadeh & Sadat Kiai, 2018; Elsharkawy & Sadawy, 2016; Zaghloul & Elwan, 2017). In this study, it can be said that the best result was obtained with 0.5% nano SiO_2 rate related to photon attenuation.

3. Adding 1% nano-SiO_2 increased 1, 2, 28, and 90 day samples by 0.12%, 0.42%, 0.004%, and 1.01%, respectively; however, there was a decrease of 0.16% for seven days.

4. Addition of 2% and 4% nano-SiO_2 decreased mass attenuation coefficient values for all curing ages, except for 1 and 2 days for the M2 group (Table 8, Table 9, Figure 13).

Generally, at a 1250 keV gamma energy level, even 0.5% nano-SiO_2 addition improved the mass attenuation coefficient; no linear relation could be found between the nano-SiO_2 rate and the mass attenuation coefficient. Similarly, except for control samples, there was no significant relation between the curing age and mass attenuation coefficient at 1250 keV gamma energy level.

Table 8. Photon attenuation coefficient (μ) of samples

Curing age(day)	M0 (cm^{-1})	M0.5 (cm^{-1})	M1 (cm^{-1})	M2 (cm^{-1})	M4 (cm^{-1})
1	0.091694	0.093115	0.091879	0.092682	0.091694
2	0.092126	0.094731	0.092867	0.093239	0.091079
7	0.092064	0.093487	0.092435	0.092311	0.089914
28	0.092867	0.092991	0.093363	0.092806	0.092435
90	0.092744	0.093735	0.093922	0.091202	0.092435

Table 9. Mass attenuation coefficient (μ_ρ) of samples

Curing age(day)	M0 (cm^{-1})	M0.5 (cm^{-1})	M1 (cm^{-1})	M2 (cm^{-1})	M4 (cm^{-1})
1	0.044665	0.044938	0.044725	0.045057	0.044221
2	0.044921	0.045475	0.045108	0.045106	0.044424
7	0.045058	0.045583	0.044989	0.044759	0.043276
28	0.045280	0.045383	0.045452	0.045082	0.045050
90	0.045864	0.046618	0.046321	0.044331	0.044887

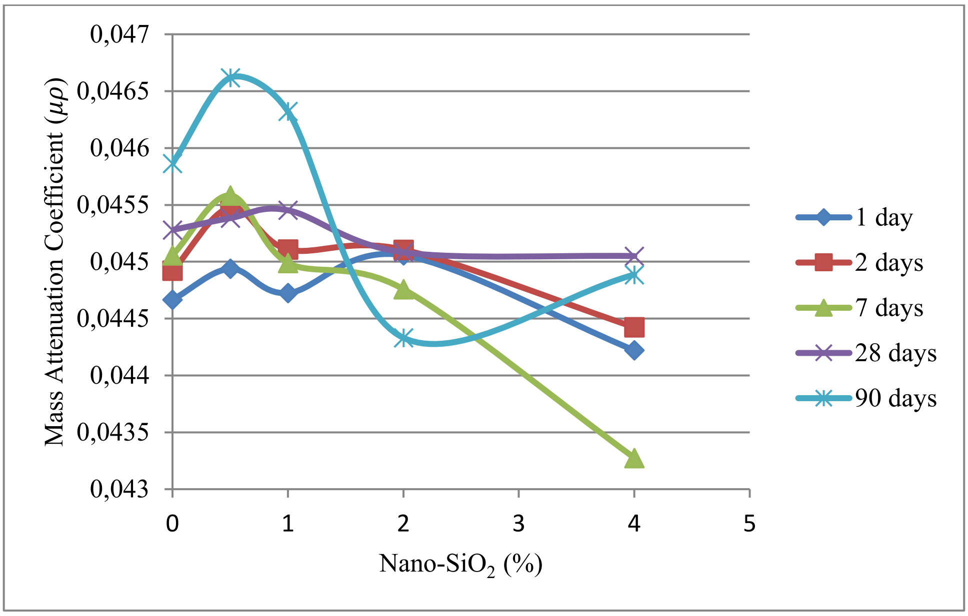


Figure 13. Mass attenuation coefficient versus nano silica addition rates.

4. Conclusion

The addition of 4% nano-silica decreases the setting time.

Even at low rates, nano addition increases the compressive strength values, especially in early curing ages. In later curing age (90 days) samples, low percentages (0.5–1%) of nano-silica decrease compressive strength at low rates, but 2% and 4% nano-silica addition increase the compressive strength with a rate less than for early ages.

The addition of 0.5% nano-silica improves the photon attenuation coefficient, but no linear relation can be found between the nano-SiO_2 rate and the mass attenuation coefficient.

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