TECHNICAL PAPER



Influence of nano silica on durability properties of concrete

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Received: 14 December 2021 / Accepted: 15 February 2022 / Published online: 9 March 2022 © Springer Nature Switzerland AG 2022

Abstract

This study focuses on determining water permeability, water absorption, chloride permeability, and compressive strength of nano-silica added concrete. Nano-silica was utilized as a cement replacement material (0–3 wt %) to design and cast two different grade concrete mixes. The microstructure development of concrete was studied using scanning electron microscope images and energy-dispersive X-ray spectroscopy. The results specify that adding 3% nano-silica to concrete enhances its microstructure by creating a denser calcium silicate hydrate gel and lowering calcium hydroxide crystals. After 56 days, the compressive strength of concrete mixes (M 30 and M 40 grade) containing 3% nano-silica was 13.14% and 16.92% greater than the control mix (0% nano-silica). Compressive strength is reasonably high in nano-silica added concrete mixes, but they have low water absorption, water, and chloride permeability. Thus, nano-silica can develop concretes with lower water absorption, water permeability, and chloride permeability.

Keywords Concrete · Microstructure · Durability · Nano-silica

Introduction

After water, concrete is the second most often used substance, and it is primarily utilized to construct various structures. The increasing usage of concrete has an unfavorable effect on the environment. Durable concrete contributes to environmental sustainability by conserving resources, minimizing waste, and reducing repair. Depending on the exposure conditions and the desired qualities, different types of concrete require different levels of durability. The longterm durability of concrete is greatly improved by adding various supplementary cementitious materials [1]. Recently, the significant ability of nanomaterials to enhance concrete properties has prompted increased efforts from researchers to investigate its multiple applications. Different researchers have used nano oxides in cement composites to study various properties [2-11]. Among these, nano-silica has grabbed the interest of many researchers, not only because it has a chemical composition comparable to cement but also because it can improve the various properties of cement composites. Even though concrete containing nano-silica has attracted significant interest in the previous decade, its durability has garnered less attention. The inclusion of nano-silica in concrete was discovered to improve its resistance to water penetration [12]. Jalal et al. [13] found lower water absorption, capillary absorption, and chloride ion concentrations in concrete mixes produced with micro and nano-silica. Compressive strength, water penetration resistance, and chloride ion penetration resistance improved in concrete containing 0.3 percent nano-silica [14]. Rao et al. [15] noticed a rise in water absorption of self-compacting mortars produced with nano-silica. The addition of colloidal nano-silica to normal and high-performance mortars hinders the transport of both water and chloride ions [16]. Impact resistance was increased, and abrasion loss was lowered by 29.82% and 52.21%, respectively, for M 30 and M 40 nano-silica mixed concrete [17]. Most of the above-said investigations checked the impact of specific percentages of nano-silica on a few specific concrete durability properties. Concrete's longterm durability has yet to be studied in detail when fly ash and nano-silica are combined. Compressive strength, water

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absorption, water and chloride permeability of concrete containing fly ash and nano-silica were investigated in this study, and the results of the examination are listed below.

Materials

Concrete was made with commercially available ordinary portland cement (53 grade) that met the requirements of IS 269:2015 [18]. Fly ash confirming the requirements of IS 3812:2013 was used [19]. Nano-silica particles ranging in size from 5 to 40 nm were employed to partially replace cement by weight (1%, 2%, and 3%) in all concrete mixtures. Locally available coarse and fine aggregate confirming the requirements of IS 383:2016 were used [20]. The workability of all concrete mixes was controlled using a Polycarboxylate-based chemical superplasticizer. Concrete mixes were made with locally accessible potable water. M 30 and M 40 grade concrete mixes were designed using the mix design technique outlined in IS 10262:2009 [21]. In the designation of each mix, M 30 or M 40 stands for a grade of concrete, and NS0, NS1, NS2, NS3 stands for varying (0, 1, 2, 3%) nano-silica content.

Testing methods

Initially, 150 mm size concrete cube samples were produced. After 24 h, they were taken out and cured with normal potable water. The cube test was performed on the samples at 3, 7, 28, and 56 day intervals following IS 516:1959 [22]. The DIN 1048-Part 5:1991 depth of penetration method was used to determine concrete's permeability [23]. Rapid Chloride Permeability Test (RCPT) was carried out on samples as per ASTM C 1202:2012 [24]. Water absorption of concrete was determined as per BS 1881-122:1983 [25]. The microstructure of nano-silica added concrete was studied using scanning electron microscope (SEM) images and Energydispersive X-ray spectroscopy (EDX). Concrete's strength and durability were studied concerning nano-silica content using the analysis of variance (ANOVA) test.

Results and discussion

Concrete microstructure

The microstructure of concrete is described as the microscopic details of its macrostructure. Typically, the microstructure of concrete results from the quality and quantity of concrete ingredients, mixing, placing, compacting, and curing procedures. SEM and EDX observations have been reported here for nano-silica added concrete. Figures 1 and 2 illustrate the microstructure morphology of M 30 and M 40 grade concrete mixes. According to Figs. 1a and 2a, most of the added fly ash particles were replaced by hydrates due to a chemical reaction with calcium hydroxide in a 0% nano-silica added concrete mix. Pozzolanic reactions of fly ash intensified with time, increasing the quantity of calcium silicate hydrate (C-S-H) gel. The produced C-S-H gel existed in clusters, and these clusters are connected with calcium hydroxide (CH) crystals. According to Fig. 1a, the reaction completely consumed all fly-ash particles.

In M 40 grade concrete mix containing 0% nano-silica, even though the hydration product of the chemical reaction between cement and fly ash is to be connected, an unreacted particle of fly ash has been observed, which acted as filling material. This explanation agrees with Bapat (2013), who reported that fly ash particles larger than 45 μ m could be considered inert and predominantly act as filler [26]. Figures 1b and 2b show the microstructure of concrete containing 3% nano-silica, which is different from what has been seen earlier. The microstructure of nano-silica mixed concrete is dense, compact and the number of pores, CH crystals were minimum. Also, micro cracks were absent in such concrete.

Typically, C–S–H gel is formed during the cement's hydration and pozzolanic reaction. Three major chemical

Fig. 1 Scanning electron microscope image for M 30 grade nano-silica concrete



(a) For 0% nano-silica concrete (M 30)



(b) For 3% nano-silica concrete (M 30)





(a) For 0% nano-silica concrete (M 40)

(b) For 3% nano-silica concrete (M 40)

elements, Ca, Si, and O, are required to produce C–S–H gel, and thus such gel is distinguished by its Ca/Si ratio. As per a previous study, the Ca/Si ratio of cement-based binders differs from 1.2 to 2.3 and changes in the range of 0.7–2.4 for supplementary cementitious materials [27]. Ca/Si ratios are obtained from EDX spectra were valid indicators of calcium silicate hydrate gel and calcium hydroxide content in hardened concrete. The compressive strength and its respective Ca/Si ratio of nano-silica added concrete are shown in Table 1.

Table 1 reveals that concrete with 3% nano-silica has shown the lowest Ca/Si ratio and highest compressive strength that 56 days. Additionally, it is well recognized that the strength increases when nano-silica is included in cement composites. However, this is usually attributed to filler effect, microstructure refinement, and pozzolanic reaction. According to the EDX report, lowering the Ca/Si ratio increases compressive strength in nano-silica mixed concrete. The examination's outcome is consistent with the previous researcher's findings [28].

Figures 3 and 4 show the EDX spectra of concrete mixes. The presence of a low Al peak in the EDS spectra indicates the presence of a small amount of ettringite.

Compressive strength

Compressive strength values at 3, 7, 28, 56 days for concrete mixes produced with different nano-silica percentages appear in Table 2. The compressive strength of all concrete mixes increases as the nano-silica level increases. The early age compressive strength of mixes M 30 NS1, M 30 NS2, M 30 NS3 was improved by 5.01%, 19.96%, 28.54% after 3 days and 19.70%, 24.20%, 30.27% after 7 days. Similarly, compressive strength of mixes M 40 NS1, M 40 NS2, M 40 NS3 was improved by 8.46%, 10.41%, 12.42% after 3 days and 5.02%, 7.24%, 9.20% after 7 days. The compressive strength of mixes M 30 NS1, M 30 NS2, M 30 NS3 was improved by 5.42%, 11.11%, 16.03% after 28 days and 4.17%, 9.41%, 13.14% after 56 days. Similarly, compressive strength of mixes M 40 NS1, M 40 NS2, M 40 NS3 was improved by 1.92%, 5.76%, 8.07% for 28 days and 3.87%, 11.98%, 16.92% after 56 days. Additionally, it was discovered that when compared to the control mix, the rate of increase in compressive strength is faster at 3 and 7 days than at 28 and 56 days. The apparent early strength enhancement of nano-silica modified concrete is due to rapid cement hydration and completion of the pozzolanic reaction at young ages. However, as curing time increases, the nanosilica content steadily decreases, resulting in a decrease in the effect of the later stage improvement.

Table 3 summarizes the ANOVA results obtained when the compressive strength of concrete mixtures was considered. The p value is less than 0.05 for both concrete mixtures, indicating that the mean compressive strength of all concrete mixes containing various concentrations of nanosilica varies significantly.

The compressive strength of concrete is increased primarily due to nano-silica acting as a filler and contributing to the pozzolanic process. In nano-silica material, the concentration of silanol atoms is more on the surface, making it

Table 1	Compressive strength
and Ca/	Si ratio for concrete

Mix	Ca/Si ratio	Compressive strength (N/mm ²)	Mix	Ca/Si ratio	Compressive strength (N/ mm ²)
M 30 NS0	1.238	33.78	M 40 NS0	1.432	42.07
M 30 NS1	1.188	35.19	M 40 NS1	1.224	43.70
M 30 NS2	1.139	36.96	M 40 NS2	0.905	47.11
M 30 NS3	0.824	38.22	M 40 NS3	0.468	49.19





Fig. 3 EDX spectra for M 30 grade concrete

hydrophilic and chemically reactive. Because of such nature, nano-silica particles act as nucleation sites for cement hydration reaction growth and accelerate cement hydration reaction. Nano-silica in concrete chemically reacts with other concrete ingredients and produces $H_2SiO_4^{2-}$ which reacts with Ca²⁺ to produce C–S–H gel and fills all the gel pores. In conventional cement paste, free portlandite content increases up to ninety days. In nano-silica added cement paste, it increases till seven days and decreases till ninety days [29]. As the amount of nano-silica in cement paste increases, the amount of calcium hydroxide in the paste decreases, resulting in a high-quality C–S–H gel [30, 31]. Cement hydration produces CH crystals which are placed in the Interfacial Transition Zone (ITZ). Nano-silica reacts with these crystals and converts them into calcium silicate hydrate gel. A gel of this nature establishes a perfect bond between the cement paste and the aggregate, resulting in an improvement in the mechanical properties of the concrete mixture. The study's findings align with other researchers' findings [7, 32, 33].

Water permeability

Figure 5 shows the depth of water penetration into concrete with varying nano-silica levels at 56 days.

Compared to control mixes, concrete produced with nano-silica demonstrated a significantly lower water penetration depth after 56 days. The outcome shows that nanosilica added concrete has a dense structure with fewer pore spaces resulting in lower water permeability. The average depth of water penetration has reduced by 24%, 38%, and 49% with 1%, 2%, and 3% nano-silica, respectively, in M 30 grade concrete compared to conventional concrete. For M 40 grade concrete same has been reduced by 15%, 71%, and 72% with the mixing of 1%, 2%, and 3% nano-silica, respectively. As shown in Table 4, for both the concrete mixes, i.e., M 30 and M 40 p value is less than 0.05. So, there is a significant difference between the mean depth of water penetration for four concrete mixes produced with varying nano-silica content.

Water permeability refers to how pores in concrete are connected. Pore spaces allow water to readily travel between them in high permeability materials, whereas in low permeability materials, pore spaces become separated, and water becomes trapped. Concrete water permeability is connected to the w/c ratio, water absorption, hardened concrete porosity, and compressive strength. The presence of more free water in concrete results in the formation of pores that are not filled with hydration products. In such cases, the permeability of concrete is more when such



Fig. 4 EDX spectra for M 40 grade concrete

Mix	3 days (N/mm ²)	7 days (N/mm ²)	28 days (N/mm ²)	56 days (N/mm ²)
M 30 NS0	10.37	14.67	30.07	33.78
M 30 NS1	10.89	17.56	31.70	35.19
M 30 NS2	12.44	18.22	33.41	36.96
M 30 NS3	13.33	19.11	34.89	38.22
M 40 NS0	14.89	26.52	38.52	42.07
M 40 NS1	16.15	27.85	39.26	43.70
M 40 NS2	16.44	28.44	40.74	47.11
M 40 NS3	16.74	28.96	41.63	49.19
	Mix M 30 NS0 M 30 NS1 M 30 NS2 M 30 NS3 M 40 NS0 M 40 NS1 M 40 NS2 M 40 NS3	Mix 3 days (N/mm²) M 30 NS0 10.37 M 30 NS1 10.89 M 30 NS2 12.44 M 30 NS3 13.33 M 40 NS0 14.89 M 40 NS1 16.15 M 40 NS2 16.44 M 40 NS3 16.74	Mix 3 days (N/mm²) 7 days (N/mm²) M 30 NS0 10.37 14.67 M 30 NS1 10.89 17.56 M 30 NS2 12.44 18.22 M 30 NS3 13.33 19.11 M 40 NS0 14.89 26.52 M 40 NS1 16.15 27.85 M 40 NS2 16.44 28.44 M 40 NS3 16.74 28.96	Mix 3 days (N/mm²) 7 days (N/mm²) 28 days (N/mm²) M 30 NS0 10.37 14.67 30.07 M 30 NS1 10.89 17.56 31.70 M 30 NS2 12.44 18.22 33.41 M 30 NS3 13.33 19.11 34.89 M 40 NS0 14.89 26.52 38.52 M 40 NS1 16.15 27.85 39.26 M 40 NS2 16.44 28.44 40.74 M 40 NS3 16.74 28.96 41.63

water leaves the pore due to evaporation or any other reason. Nano-silica in concrete reduces water permeability and increases concrete durability in five ways.

- Nano-silica chemically reacts with water and CH through • pozzolanic activity to make extra cementitious compounds. Leaching does not occur when CH is chemically coupled with nano-silica.
- The conversion of CH to C-S-H gel decreases channels • and void spaces, thereby reducing permeability.
- The filler effect of nano-silica reduces the water penetra-• tion depth of concrete containing nano-silica. Nano-silica

particle size is very small and could quickly fill in the pores of cement paste making it dense and durable.

As nano-silica is used in combination with superplasti-٠ cizer, it dramatically reduces the water demand (15-30%) of concrete. With the reduction of water, the internal voids bleed channels are less in concrete, which makes concrete more durable

The outcomes in this investigation concur with the past studies conducted on nano-silica added concrete [14, 34].

Table 3 Analysis of variance (ANOVA) results for compressive strength

	Between groups	Within groups	Total
Compressive streng	th (M 30 grade conci	rete)	
Sum of squares	34.339	0.258	34.597
df	3	8	11
Mean square	11.446	0.032	
F	354.743		
Significance	0.000		
Compressive streng	th (M 40 grade conci	rete)	
Sum of squares	93.347	0.194	93.541
df	3	8	11
Mean square	31.116	0.024	
F	1285.776		
Significance	0.000		

Rapid chloride permeability test (RCPT)

The electrical resistivity of the concrete being tested is reflected in the RCPT, which is an indirect method of assessing concrete permeability. The average values of charge passed for nano-silica added concrete are compared with typical ASTM C 1202:2012. Table 5 indicates the charge passed in Coulomb for concrete containing varying nanosilica content.

From the test readings of charge passed values for nanosilica added concrete samples of M 30 and M 40 grade, it is seen that the values of charge passed are in the range of 1196–1658 coulombs. The above values represent low chloride ion permeability as per ASTM C 1202. Table 6 shows ANOVA results obtained after considering total charge passed values of nano-silica added concrete. For both the concrete mixes p value is less than 0.05. So, there is a significant difference between the charge passed values for four concrete mixes produced with varying nano-silica content.

Total charge passing values have increased as nano-silica in concrete increases. The increase in the charge passed values is mainly because of the change in the chemical composition of pore solution with the inclusion of nano-silica and superplasticizer. In ASTM C 1202, it is mentioned that concrete with dissociating admixtures may produce high values of RCPT cumulative charge passed due to the high ionic concentration of pore solution. Colloidal nano-silica particles show an enhanced ability to chemically adsorb and even disassociate various organic molecules due to increased surface area at the nanoscale. When nano-silica particles are added to concrete, they drastically alter the conductivity of the pore solution without appreciably altering the cement matrix's pore structure. RCPT tests measure chloride ions

 Table 4
 ANOVA results for depth of water penetration of nano-silica

 added concrete

	Between groups	Within groups	Total
Depth of water per	etration (M 30 grade	e concrete)	
Sum of squares	655.729	153.5	809.229
df	3	8	11
Mean square	218.576	19.188	
F	11.392		
Significance	0.003		
Depth of water per	etration (M 40 grade	e concrete)	
Sum of squares	683.156	332.293	1015.449
df	3	8	11
Mean square	227.719	41.537	
F	5.482		
Significance	0.024		



25 23.33 19.83 20 15 mm 10 6.83 6.5 5 0 M 40 NS0 M 40 NS1 M 40 NS2 M 40 NS3 Type of Mix

Fig. 5 Average depth of water penetration for concrete mixes

 Table 5
 Total charge passed in

 Coulomb for nano-silica added
 concrete

Mix	Charge passed (Coulomb)	Average charge passed (Coulomb)	Mix	Charge passed (Coulomb)	Average charge passed (Coulomb)
M 30 NS0	1233	1196	M 40 NS0	1283	1254
	1154			1219	
	1201			1260	
M 30 NS1	1209	1244	M 40 NS1	1232	1257
	1227			1253	
	1296			1285	
M 30 NS2	1313	1365	M 40 NS2	1581	1658
	1387			1770	
	1395			1623	
M 30 NS3	1238	1264	M 40 NS3	1621	1618
	1283			1552	
	1271			1681	

Table 6 ANOVA results for total charge passed values

	Between groups	Within groups	Total
Total charge passed	d (M 30 grade conci	rete)	
Sum of squares	45,548.250	12,550.000	58,098.250
df	3	8	11
Mean square	15,182.750	1568.750	
F	9.678		
Significance	0.005		
Total charge passed	d (M 40 grade conci	rete)	
Sum of squares	441,712	31,558.667	473,270.7
df	3	8	11
Mean square	147,237.333	3944.833	
F	37.324		
Significance	0.000		

movement and other ions movement, including OH^- ions which have greater mobility than chloride ions. The surface of silica nanoparticles is covered by silanol groups which mainly contain OH^- . Because of such groups, the concentration of hydroxyl ions in pore solution increases. This will increase the capacity of exchanging anions with permeating solutions. Hence the charge passed values increases.

The chloride binding ability of constituent materials also affects resistance to chloride ion penetration. The degree of chloride binding increases with C_3A content [35, 36]. The chlorides which are entered into concrete react with C_3A to form stable chloro complexes. Due to the high alumina in fly ash, such materials increase C_3A content [37]. In nano-silica, the alumina content is significantly less because of which total charge values increases. Although the charge passing values have increased, the compressive strength of concrete

Table 7 Water absorption of M30 grade concrete

	Water charaction	Watan abaamtian	07 no devotion in motor	07 and untion in motor
MIX	after 10 min (%)	after 24 h (%)	absorption after 10 min	absorption after 24 h
M 30 NS0	0.91	1.70		
M 30 NS1	0.86	1.12	5.15	34.25
M 30 NS2	0.63	0.95	30.15	44.03
M 30 NS3	0.58	1.08	35.66	36.59

Table 8 Water absorption of M40 grade concrete

Mix	Water absorption after 10 min (%)	Water absorption after 24 h (%)	% reduction in water absorption after 10 min	% reduction in water absorption after 24 h
M 40 NS0	0.91	1.29		
M 40 NS1	0.90	1.21	1.47	6.20
M 40 NS2	0.63	1.11	30.40	13.95
M 40 NS3	0.37	1.10	58.97	14.47

Table 9 ANOVA results for water absorption values

	Between groups	Within groups	Total
Water absorption (M	A 30 grade concrete)		
Sum of squares	1.003	0.057	1.06
df	3	8	11
Mean square	0.334	0.007	
F	46.631		
Significance	0		
Water absorption (M	A 40 grade concrete)		
Sum of squares	0.071	0.015	0.086
df	3	8	11
Mean square	0.024	0.002	
F	12.964		
Significance	0.002		

has also increased. This is the primary reason a concrete mix containing fly ash and silica fume performs well in chloride permeability.

Water absorption

Tables 7 and 8 show the water absorption results of concrete samples after 10 min and after 24 h.

Table 9 shows ANOVA results obtained for water absorption values of nano-silica added concrete. For both the concrete mixes p value is less than 0.05. So, there is a significant difference between the mean water absorption values for four concrete mixes produced with varying nano-silica content.

Tables 7 and 8 show that increasing the nano-silica content reduced the water absorption of both concrete samples. After 10 min, the inclusion of 1%, 2%, and 3% nano-silica content to M 30 grade concrete resulted in a reduction in water absorption of 5.15%, 30.15%, and 35.66%, respectively, compared to the control mix. After 10 min, adding 1%, 2%, or 3% nano-silica to M 40 concrete reduced water absorption by 1.47%, 30.40%, or 58.97% compared to the control mix. The reductions in water absorption values for M 30 and M 40 mixes containing 1%, 2%, and 3% nanosilica were obtained as 34.25%, 44.03%, 36.59% and 6.20%, 13.95%, 14.47%, respectively, after 24 h. The results demonstrated that the addition of nano-silica refines the pore structure of concrete. Such a refined pore structure of concrete is desirable to improve the durability of concrete. Incorporating nano-silica particles into cement composites results in a dense, compact microstructure with few voids and microcracks, and hence water absorption values reduce. The findings align with the earlier research [13].

Conclusion

Nano-silica added concrete mixes were experimentally examined to assess strength and durability. The following conclusions are derived from the findings of the research:

- The addition of nano-silica in concrete has increased its strength and durability.
- SEM images show that the 3% nano-silica added concrete has a more homogenous, dense, and compact microstructure than conventional concrete. Nano-silica converts calcium hydroxide crystals into calcium silicate hydrate gel, reducing their quantity and size making ITZ denser. A dense binding paste matrix can be achieved using nanosilica particles as these particles fill all the pores of C–S–H gel and create a tight bond with it. In nano-silica, added concrete, compressive strength increases, and the Ca/Si ratio falls with increasing amounts of nano-silica.
- The compressive strength of the concrete containing 3% nano-silica was the highest. At 28 and 56 days, the rise in compressive strength of M 30 grade concrete was 16.03% and 13.14% with 3% nano-silica, whereas it was 8.07% and 16.92% for M 40 grade concrete.
- Nano-silica added concrete showed low chloride permeability as per ASTM C 1202, with 1196–1658 Coulomb charge passed values.
- The average depth of water penetration was reduced by 49% and 72%, respectively, by incorporating 3% nanosilica into M 30 and M 40 grade concrete.
- After 24 h, by incorporating 3% nano-silica into M 30 and M 40 grade concrete, the water absorption values were reduced by 36.59% and 14.47%, respectively.
- The present investigation on concrete indicates that using nano-silica and fly ash as cement replacement materials improves its strength and durability. Similarly, the effect of nano-silica and fly ash on higher-grade concrete mixes needs to be evaluated in the future, along with cost analysis. Future research should also consider the impact of other pozzolanic admixtures such as ground granulated blast furnace slag, micro silica, rice husk ash, and nano-silica. In this way, ternary mixed concrete can be developed using novel components, which could save money and help the environment at the same time.

Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

Ethical statement The authors have no relevant financial or non-financial interests to disclose.

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