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Original scientific paper

EFFECT OF NANO-SILICA ADDITION ON PHYSICO-MECHANICAL PROPERTIES AND DURABILITY OF CONCRETE

Adnan Mujkanović[1], Anesa Bitić[2], Marina Jovanović[1], Nevzet Merdić[3], Amel Zahirović[4], Amna Karić[1]

[1]The University of Zenica, Faculty of Metallurgy and Technology,
[2]Ludwig-Maximilians-Universität München,
[3]HeidelbergCement, Cement plant Kakanj,
[4]The University of Zenica, Institute "Kemal Kapetanović"

ABSTRACT

This paper presents the results of an experimental investigation into the effect of nano-silica addition on the mechanical properties and durability of concrete. Three mixes of concrete containing 0%, 2%, and 4% nano-silica by weight of cement were tested. The slump of fresh concrete as well as compressive strength, dynamic modulus of elasticity at 2, 7, 28, 90, and 180 days, and flexural strength at 2, 28, and 90 days of hardened concrete were determined. As for indicators of concrete durability, the total volume of permeable voids, water absorption, the rate of water absorption, and the freeze-thaw scaling resistance were determined. Test results showed that the incorporation of nano-silica in concrete resulted in an improvement of all mechanical properties of concrete, as well as concrete durability parameters that are tested.

Keywords: nano-silica; concrete; durability; strength; porosity; water absorption

Corresponding Author:
Adnan Mujkanović,
Faculty of Metallurgy and Technology
Travnička cesta 1, 72000 Zenica, B&H
Tel.: +387 32 401 831; fax: +387 32 406 903
*E-mail address: adnan.mujkanovic@unze.ba

1. INTRODUCTION

Concrete is the most widely used construction material in the world and is applied in different environmental conditions. However, concrete has a substantial carbon footprint, as the production of cement, its main ingredient, involves the emission of large amounts of CO_2. The CO_2 emission from the concrete production is directly proportional to the cement content used in the concrete mix [1]. Reducing the cement content and/or enhancing the concrete durability parameters can reduce its negative impact on the environment. Properties of hardened concrete, including durability, can be enhanced through the incorporation of a relatively small number of nanoparticles that can modify concrete microstructure. Recently, particular attention has been given to nano-silica (nS), which has noticeably higher reactivity than micro-silica. Compared to other nanoparticles, nS has better performance in concrete. Besides the pore-filling effect and nucleation effect which accelerates the hydration of cement, nS had high pozzolanic activity and could react with $Ca(OH)_2$ in cement composites to generate more calcium silicate hydrate (C-S-H gel). This leads to modifying the internal C-S-H structure and achieving a denser and more compact microstructure [2]. Nano-silica is typically prepared by the neutralization of sodium silicate solutions with acid where silica monomers are allowed to condense to colloidal particles and aggregates. Nano-silica is commercially available in both powder and colloidal forms.

Although the modification of the concrete properties by nS addition has been subjected to intensive study worldwide, it is still not enough for confident use of this addition, and nS in concrete is not yet commonly applied [3]. Water to cement ratio, cement content, physical state, and dispersion of nS into the concrete could significantly influence the nS performance in concrete. Nano-silica production costs are higher than production costs of other mineral additions. There is also an issue with the increased water demand for mixtures containing higher amounts of nS [4,5]. The literature survey showed that controversial results were obtained regarding the nS effect on mechanical properties. While some researchers [6,7] observed a significant increase in strength, others reported only moderate improvement [8-11] or even there was no gain in mechanical strength [12]. On the other hand, a majority of researchers claim improvement of concrete durability by incorporating nS in concrete. For these reasons complete and systematic experimental studies to assess the properties of nS incorporated concrete are needed. In this study, the effects of different dosages on concrete consistency, compressive and flexural strength, dynamic modulus of elasticity, the total volume of permeable voids, water absorption, the rate of water absorption, and the freeze-thaw scaling resistance were determined. As the dry powders of nS particles are difficult to be dispersed in concrete, nS was added into concrete in the form of a slurry.

2. MATERIALS AND METHODS

2.1. Materials

Ordinary Portland cement type CEM I 52.5 N was used in this study. A commercial slurry of colloidal nS (Levasil CB22 by Nouryion Chemicals) containing 30% solids and has a density of 1.3 g/cm^3 was used. The surface area of nS is 220 m^2/g and the average particle size is 12 nm [13]. Crushed limestone with a maximum particle size of 16 mm that complies with the requirements of EN 12620-1 [14] was used as aggregate in the concrete mixes. A polycarboxylate-based high-range water-reducing admixture (HRWRA) and air-entraining agent formulated from modified naturally occurring and synthetic surfactants, both conforming to EN 934-2 [15] were used as additives.

2.2. Mix proportions

Proportions of the reference mix and the mixes containing 2% and 4% nS by weight of cement are listed in Table 1. As nS was introduced into mixes in the form of a slurry, the same water-binder ratio in each mix is obtained by reducing the added water by the amount contained in the nS slurry.

Table 1. Mix proportions of the concrete

Material		0% nS	2% nS	4% nS
Cement (kg/m^3)		400	392	384
nS (kg/m^3)		-	8	16
Agreggate (kg/m^3)	0-4 mm	885	885	885
	4-8 mm	355	355	355
	8-16 mm	530	530	530
Water (kg/m^3)		176	176	176
HRWRA (kg/m^3)		3,2	3.2	3.2
Air entraining agent (kg/m^3)		0,4	0.4	0.4

2.3. Mixing procedure

First, the fine and coarse aggregates were added to the mixer, followed by dry-mixing with cement for 120 s. Then, nS slurry and around 75% of the total amount of water are slowly added and mixed for another 120 s. Finally, the remaining mixing water and additives (HRWRA and Air entraining agent) were added to the mixer, during the consecutive mixing for 180 s. The whole mixing time was 7 min for all the batches.

2.4. Test methods

After mixing, the slump test was carried out on each mix in accordance with EN 12350-2 [16]. For testing hardened concrete properties cubes 100×100×100 mm and beams 100×100×400 mm were prepared. All specimens had been demoulded after 24 hours and then stored in water at a temperature of 20 °C until tests were conducted. The compressive strength of the cubes was measured following the procedure described in the norms EN 12390-3 [17] at 2, 7, 28, 90, and 180 days. Before the compressive strength test, on cubic samples, a non-destructive test was carried out to determine the velocity of the ultrasonic pulse by direct method via a pulse velocity test device [18]. The dynamic modulus of elasticity is calculated according to the equation:

$$E_{bd} = \frac{V^2 \cdot \rho \cdot (1+\nu)(1-2\nu)}{(1-\nu)} \qquad [Pa]$$

where V is ultrasonic pulse velocity in m/s, ρ is apparent density in kg/m^3, and ν is the Poisson ratio (0.2 for concrete).

After the compressive strength test (at 2 and 28 days), pieces of concrete were taken for SEM analysis. They are immersed in isopropanol, washed by dietilether, and dried at 40°C. SEM analysis is performed on a microscope Tescan Mira 3 (20 keV).

The flexural strength test was carried out on the beams according to the norm EN 12390-5 [19] at 2, 28, and 90 days. At the curing age of 28 days, water sorptivity was measured (ASTM C1585-13) [20], as well as density, permeable voids, and water absorption (ASTM C642-06) [21]. ASTM C642-06 was used to determine the total amount of water absorption and volume of permeable voids. The samples at age 28 days were placed in an oven at a temperature of 105 °C for 48 hours and the oven-dry mass of each specimen was measured. After that, the saturated mass of samples was determined by immersing them in the water at 20 °C for 72 hours. Finally, the saturated mass was determined by immersion in boiling water. Samples were covered with tap water and boiled for 5 hours. The water absorption and volume of voids were calculated using oven-dry mass, immersed apparent mass, and immersed and boiled saturated mass values. The rate of water absorption (sorptivity) was obtained by using the procedure described in ASTM C1585-13. The schematic procedure of the test is shown in Figure 1.

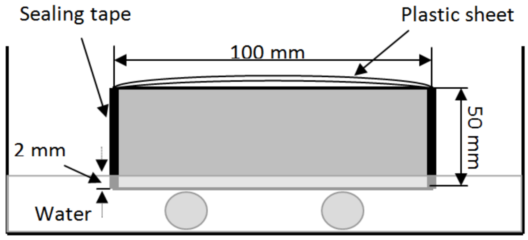


Figure 1. Schematic of the procedure in accordance with ASTM C 1585-13

The test was conducted on the specimens of 100×100×50 mm size after 28 days of curing. This test method consists of preconditioning samples, followed by exposing the bottom surface of the sample to liquid water and measuring the increase in mass resulting from water absorption. The samples were preconditioned using an oven able to maintain a temperature of 50±2 °C and a desiccator. The relative humidity is controlled in the desiccator at 80±0.5 % by a saturated solution of potassium bromide. All surfaces of the specimen except the top and bottom were sealed to prevent moisture ingress. Only the bottom surface of the sample was left exposed for water penetration. The samples were placed over the water and the level of the water was maintained at 2 ± 1 mm. The change in weight after the certain intervals was recorded and the normalized absorbed fluid volume (i) was calculated as:

$$\mathrm{i} = \frac{\mathrm{m_t}}{(\mathrm{a} \cdot \rho)}$$

where m_t is the change in specimen mass at time t, *a* is the area of the specimen exposed to the fluid, and ρ is the density of the absorbed fluid water. Then, the calculated absorption value at each time is plotted against the square root of time ($t^{1/2}$) to investigate the slope of its linear trend, sorptivity. This index is determined in two stages; initial and secondary absorption due to the absorption time. The freeze-thaw scaling resistance of concrete was tested in accordance with the norm CEN/TS 12390-9 on samples cut from cubes 100´100´100 mm. The test cubes were cured for 28 days at 20 °C in the water bath and were then exposed to conditioning under ambient conditions of 20±2 °C and 65±5% relative humidity for 7 days. Test samples were sawed from different samples of concrete cubes. A 3% de-icing salt solution was poured over the surface of the test sample before exposure. The liquid height was maintained at 3 mm throughout the test. Then, samples were exposed to freezing and thawing in the temperature range from -20 °C to +20 °C. After each 7, 28, and 56 cycles the scaled material was collected from the surface of each sample. This material was dried to constant mass and weight to the nearest 0,1 g. After that, the new quantity of 3% NaCl solution was placed on the sample surface, and samples were returned to the freezer. Scaled material is cumulatively added until 56 cycles [7]. Photos of the upper surface of the samples were taken, and the mass losses were weighted on each sample after 7, 28, and 56 cycles.

3. RESULTS AND DISCUSSION

The slump of fresh concrete mixes versus the amount of nS is presented in Figure 2. The data illustrates the direct relation between the nS amount and the workability of fresh concrete; with the addition of nS, the slump of fresh concrete decreases linearly.

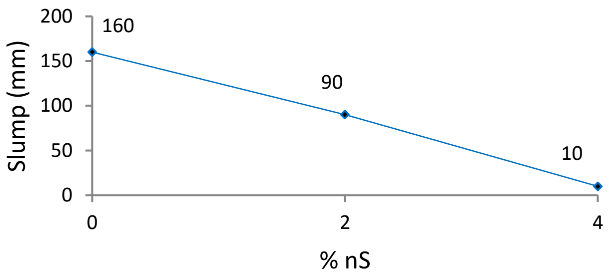


Figure 2. Slump of concrete mixes at different nS additions

The slump reduction is explained by the fact that the specific surface area of nS particles is larger than that of cement, and it absorbs more water thus reducing the amount of water needed for the fluidity of the concrete mixture. These results are consistent with the majority of previous studies that reported a significant decrease in the workability of concrete containing nS [4,5,22,23]. However, some researchers claimed that only a mild reduction in workability is observed in concretes containing up to 3% of nS by weight [24,25].
To clarify the effect of nS addition to the concrete microstructure, an SEM analysis was carried out. The analyses were performed on concrete samples at the age of 2 and 28 days and the micrographs are shown in Figure 3. It is evident from the images that the incorporation of nS densified the microstructure, and decreased the number, as well as the size of capillary pores, which is consistent with the remarkable reduction of sorptivity of nS containing concrete (Figure 3). This can be attributed to both the filling effect and the pozzolanic reactivity of nS particles [26]. Micrographs shown in Figure 3 reveal that the 4% dosage of nS had a stronger effect compared to the 2% dosage in refining the microstructure structure of the concrete. There was no observed agglomeration of nS particles, even at dosages of 4%.
Porosity (volume of permeable voids) and water absorption of hardened concrete determined in accordance with ASTM C642-06 are presented in Figures 4a and 4b respectively. The measurements confirm that both porosity and water absorption of

0% nS 2% nS 4% nS

2 days

28 days

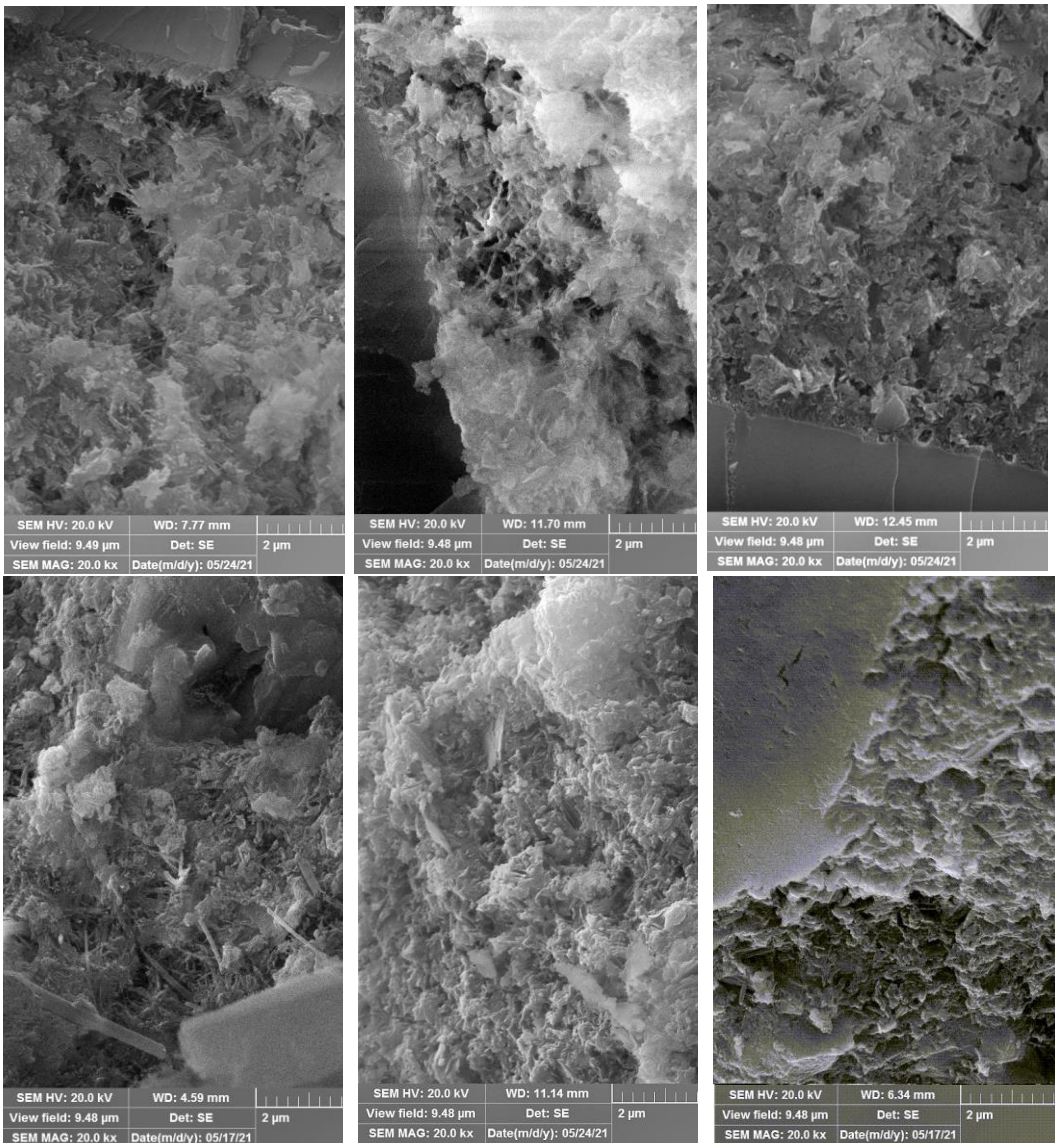
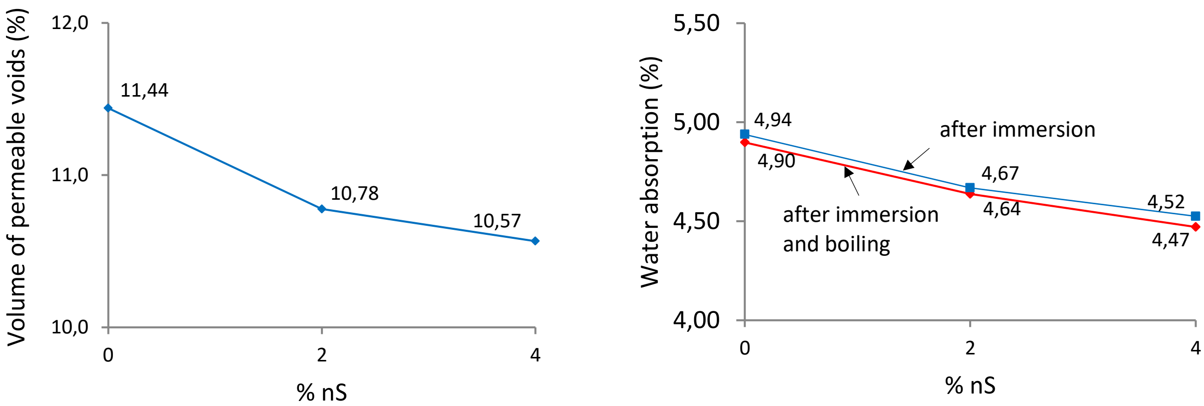


Figure 3. SEM images of concrete samples at different nS additions after 2 and 28 days of curing

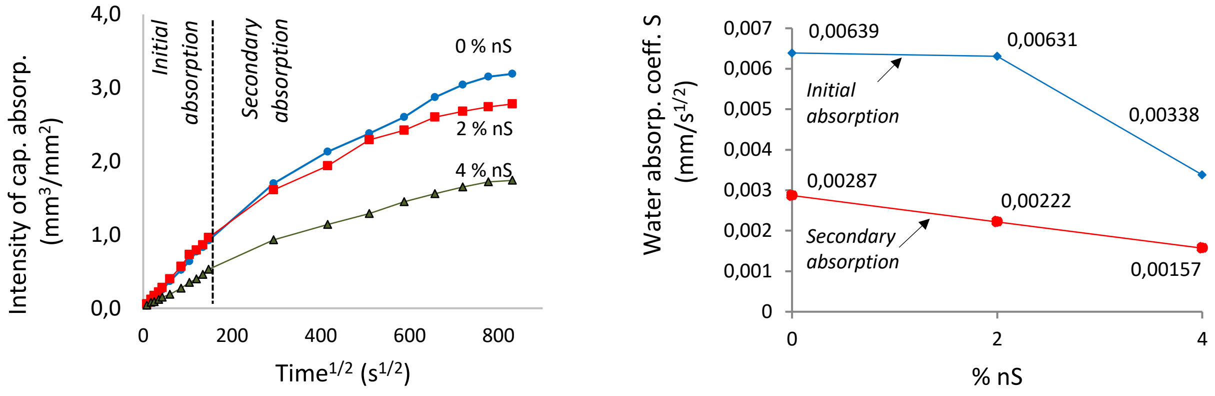


a) b)

Figure 4. Volume of permeable pores (a) and water absorption (b) of concrete at different nS additions

concrete decrease in the presence of nS in the concrete. The volume of permeable voids is reduced by 5.8%, and 7.6% with the addition of 2%, and 4% nS, respectively. Also, water absorption is reduced for 5.5%, and 9.5% with addition of 2%, and 4% nS, respectively. The volume of permeable voids in concrete is affected by many factors including compaction, curing, air entrainment, etc. Also, aggregate properties have a great impact on the volume of permeable voids of concrete. According to Shaikh et al. [27] volume of permeable pores in concrete containing recycled coarse aggregate was not significantly influenced by nS addition.

After the samples were conditioned for 18 days (3 days in a desiccator and 15 days in a sealed container), the test was performed following ASTM C1585-13, with results provided in Figure 5a. Figure 5b shows the calculated initial sorptivity and secondary sorptivity.

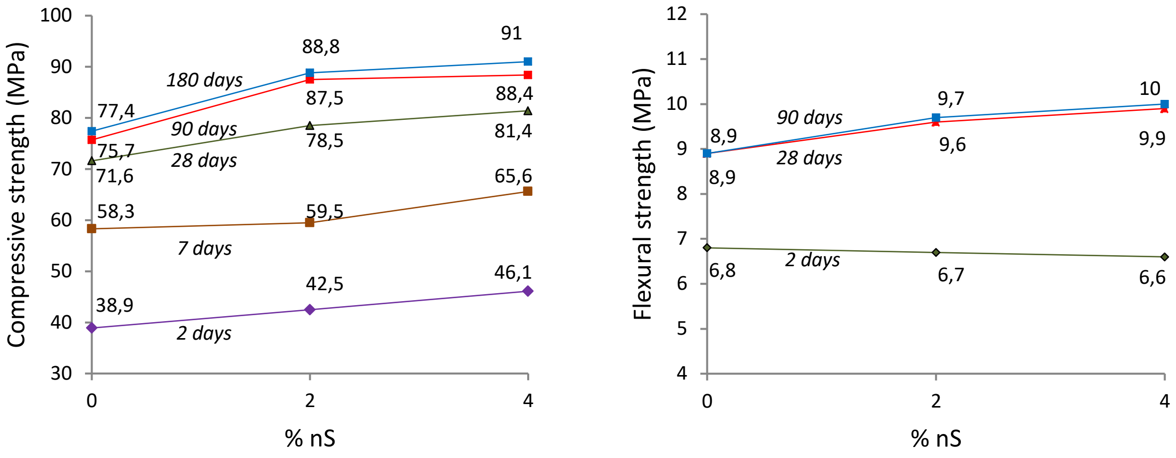


a) b)

Figure 5. Cumulative water absorption (a) and water absorption coefficient (b) of concrete at different nS additions

Results shown in Figure 5 indicate that sorptivity values decrease with the addition of nS to concrete. However, the addition of 4% of nS resulted in a much greater reduction of sorptivity than the addition of 2% nS. The lower sorptivity is explained by the refinement of the cement paste microstructure in the capillary range. The use of nS tends to disconnect continuous pores and subdivide larger pores into smaller ones. This produces a structure with a finer pore, which exhibits lower transport properties [26,28,29]. Zhang et al. [30] also stated that the large capillary porosity decreased with the increased dosage of nS.

The results of mechanical testing of hardened concrete are presented in Figures. 6 and 7. Results given in Figure 6a show that nS addition leads to the increase of compressive strength at all tested periods. The increase of compressive strength with the addition 2% of nS is 9.3%, 2.1%, 9.6%, 15.6%, and 14.7%, at 2, 7, 28, 90, and 180 days, respectively. The addition 4% nS results in compressive strength increase for 10.8%, 12.5%, 13.7%, 16.8%, 17.6% at 2, 7, 28, 90, and 180 days, respectively. According to studies [25,30-32], a notable gain in compressive strength can be achieved with the addition of nS in concrete. Jalal et al. confirmed that 2% nS in concrete increased compressive strengths by 22, 38, and 43% and respectively at 7, 28, and 90 days [25]. Studies by Elkady et al. [33] reported 43.5% gain in compressive strength at 3% nS dosage concrete at 28 days while 1.5 nS resulted in 17.5% strength gain compared with the control specimen. On the contrary, Quercia et al. [10] reported a lower increase in compressive strength with the addition of 3.8% nS: 3.7% at 3 days, 10.8% at 28 days, and 10.4% at 90 days.



a) b)

Figure 6. Compressive (a) and flexural (b) strength of concrete at different nS additions

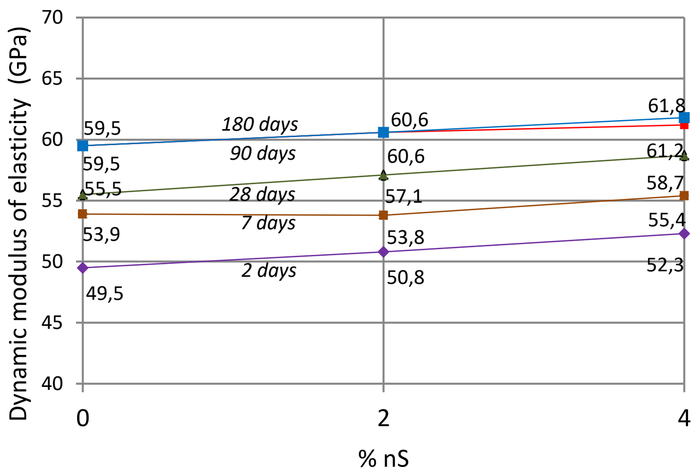


Figure 7. Dynamic modulus of elasticity of concrete at different nS additions

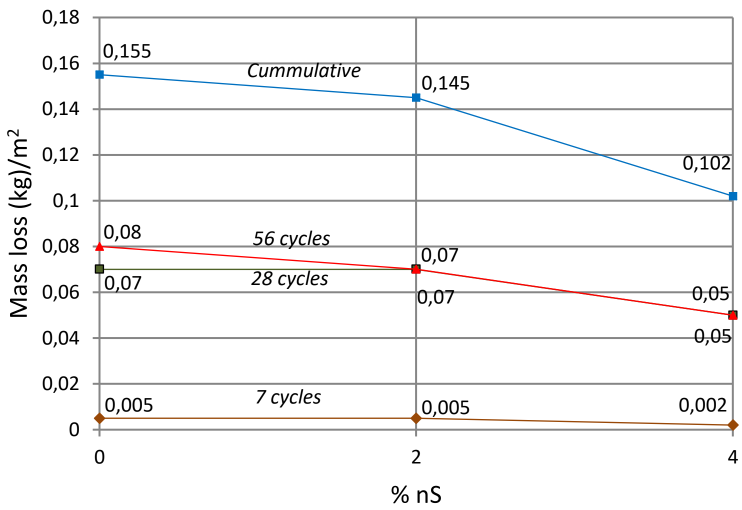


Figure 8. Mass loss in freeze-thaw and de-icing test at different nS additions

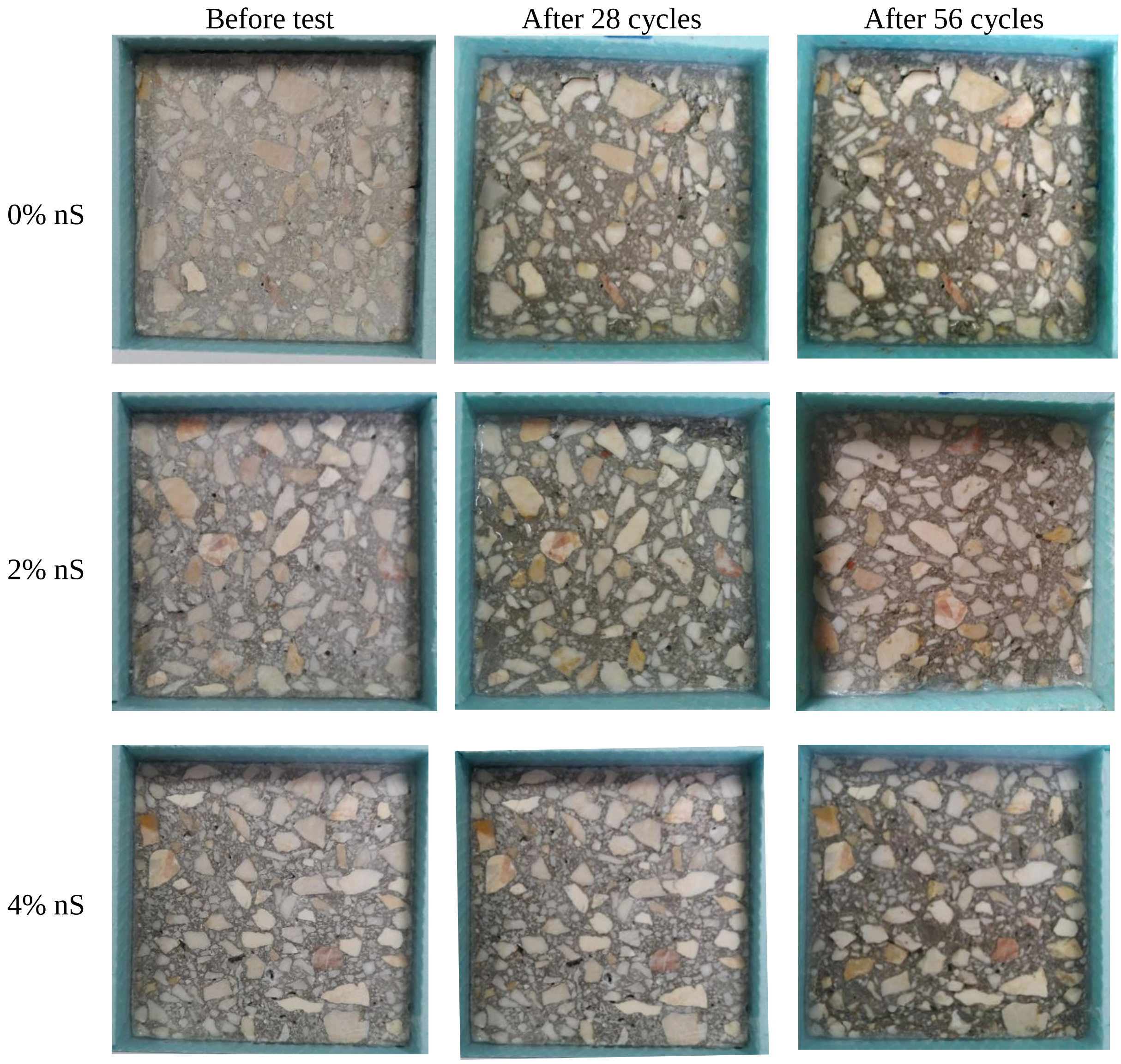


Figure 9. The surface of the samples after exposure to freeze-thaw and de-icing test at different nS additions

Flexural strength (Figure 6b) also increases with the increasing content of nS, with exception of results obtained at 2 days where is observed negligible loss in flexural strength in concrete mixes containing nS. The 28-day flexural strength is increased for 7.9% (2% nS) and 9.0% (4% nS) and the 90-day flexural strength is increased for 11.2% (2% nS) and 12.4% (4% nS). The gain in flexural strength is lower compared to the studies [25,26,34] and comparable to the gain reported in [35].

The influence of nS on the dynamic modulus of elasticity was also observed to be beneficial (Figure 7). The percentage increase of modulus is the highest in samples tested at 2 days (2.62% and 5.66% for nS content 2% and 4%, respectively), and decreased with the increased age of the samples. The slight increase in the elastic modulus of concrete with adding the nS particles up to 3% is also demonstrated in [35], while other studies [36,67] reported a higher increase in dynamic modulus in concrete containing nS. The improvement of mechanical properties of nS incorporated concrete can be explained by the increased formation of hydration product in the presence of nS particles, which act as a filler to increase the concrete density and an activating agent for cement hydration. In addition, nS possess high pozzolanic activity even at an early age. As a result, the high-density CSH phase is formed that densifies the interface between the aggregates and the hardened cement, resulting in a stronger bond between them. Consequently, the strength development process is enhanced from the beginning of hydration, and long-term mechanical properties are improved as well [35, 37-40].

The average mass losses after 7, 28, and 56 freeze-thaw cycles of concrete samples exposed to de-icing salt are presented in Figure 8. The figure indicates a slower rate of mass loss in mixtures containing nS in all testing periods. After the freeze-thaw cycle tests, the surface layers of the concrete specimens had different degrees of damage, as shown in Figure 9. The surfaces of specimens with 0% and 2% nS spalled off moderately after 28 cycles, while the surface of specimen containing 4% remains almost intact. After 56 cycles all samples suffered mild surface spalling. From the results shown in Figures 8 and 9 can be seen that the highest freeze-thaw and de-icing salt resistance is observed in concrete containing 4% nS. These results correspond to the results of the sorption test, as well as mechanical tests. The pore structure refinement in the capillary range is arguably the main reason for the improvement of the frost resistance of concrete incorporating nS. The findings of other studies also confirmed the positive effect of nS on concrete frost resistance [41-43].

4. CONCLUSIONS

Based on the test results, it can be concluded that nS significantly modifies the consistency of fresh concrete, as well as the mechanical and durability properties of hardened concrete. The slump of fresh concrete decreases significantly with the addition of nS. The greater the nS content, the greater loss in the slump of fresh concrete mixes. A beneficial effect of nS on transport properties was observed by water absorption tests. The volume of permeable pores, water absorption, and sorptivity (rate of absorption) of concrete decreases with the addition of nS. The addition of nS resulted in a moderate increase in compressive strength, flexural strength, and dynamic modulus of elasticity for all curing ages. To achieve a higher increase in strength and modulus, certain mixture modifications that include higher dosages of superplasticizer are needed in concretes incorporated nS. SEM analysis showed that the microstructure's homogeneity and density of concrete increase, as the content of nS increases. This refinement of the pore structure in the capillary range resulted in improving the frost resistance of concrete incorporating nS. The test results confirmed that the mass loss and surface damage after cycles of freeze-thaw in the presence of de-icing salt decrease as the dosage of nS in concrete increases

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Conflicts of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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