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STRUCTURE FORMATION OF C-S-H FROM THE POSITION OF MICROMECHANICS OF COMPOSITE MEDIA

Abstract: the creation of an environmentally friendly building material to protect the human environment can only be carried out from the position of a transdisciplinarity approach, taking into account modern achievements in geomimetics and micromechanics of composite media. A wide range of basalt-fiber-reinforced concrete based on composite binders has been developed, which have increased characteristics of impermeability and durability under extreme operating conditions. The nature of the influence of the composition and manufacturing technology of cement composites on the pore structure of the composite has been established, which has a positive effect on the characteristics of gas, water and vapor permeability. High early strength was obtained, which allows the use of materials for operational repair and construction in emergency situations. The positive influence of the composition of the developed composite on the performances has been proved. The water resistance of the modified composite provides a water pressure of 2 MPa for 148 hours, which corresponds to the W18 grade (for the control sample – W8), the frost resistance grade – F300. It was found that the water absorption of the modified concrete samples was lower than that of the control sample, which is explained by the decrease in the pore structure index λ by 28.4 times, and the average pore diameter by 3.05 times. The total pore volume of the modified concrete was lower and decreased with increasing dose of nanosilica.

Keywords: composite, Portland cement, ash and slag mix, mechanical properties, impact strength

Introduction

A concrete is produced more than any other synthetic material on Earth. The current world production of cement is 3.5 billion tons, which is enough to produce more than 30 billion tons or 1.5 m³ of concrete per capita per year [1]. There is no other material that could replace concrete in the near future to meet the needs of society for housing, infrastructure, etc. However, concrete has an uncertain future due to its significant environmental impact, which accounts for 5-10% of global CO₂ emissions. It is now emerging that the micromechanics of composite media may be the discipline that enables the development of, among other things, the environmentally friendly green concrete of the future.

The creation of an environmentally friendly building material can be carried out only from the position of a transdisciplinarity approach, taking into account modern achievements in geomimetics [2-4]. Applying the laws of similarity, the principles of technogenic metasomatism and micromechanics of composite media when designing a cement composite, the components are linked together, compacting the structure due to the control of the pore space, and, accordingly, the durability of the material increases [5-8].

The fundamental idea of the proposed approach is that it is possible to evaluate the nanostructure of a material based on microporemechanics [9-11]. In the article, this approach is illustrated by an example of application to calcium silicate hydrates (C-S-H), the binder phase of cement-based materials [12-14]. For this class of materials, CSH exists in at least three structurally different, but similar in composition forms: low density (LD), high density (HD) and ultra-high density (UHD) [15-17]. These three forms differ only in the packing density of particles less than 5 nm in size [18-20]. The existence and mechanical significance of these phases can be confirmed by nanoindentation [21-23]: it was found that LD CSH and HD CSH are uniquely characterized by a set of material properties that do not depend on the proportions of the mixture, the type of cement, etc. Instead, they represent the intrinsic unchangeable properties of the material [24-26].

The relationship between these mechanical properties of the C-S-H phase and its packing density has been established, showing that the C-S-H phases exhibit a unique nano-grained (nanogranular) morphology with packing densities that are very close to the ultimate packing densities of spheres; namely, the random close-packed limit (RCP) or maximally random jammed state (MRJ) $\eta = 0.64$ [27-29] for the LD C-S-H phase; and ordered packing of

two types: hexagonal or face-centered cubic, equal by Gauss's theorem, $\eta = \pi/\sqrt{18} = 0.74$ [30-32] for the HD C-S-H phase. In this model, it is assumed that the porosity of the C-S-H phase gel is not constant, it depends on the volumetric proportions LD C-S-H (f_{LD}) and HD C-S-H (f_{HD}):

$$\varphi_0 = 1 - (0.64 \times f_{LD} + 0.74 \times f_{HD}) \quad (1)$$

In the nanoindentation technique, the hardness H and the indentation modulus M , determined from the load-depth curve (P - h) in accordance with classical analysis tools [33-35], are representative of the particle properties (particle rigidity m_s , hardness h_s , coefficient of friction μ) and packing of their particles (packing density η), as well as some morphological parameters (denoted by η_0):

$$H = \frac{P}{A_c} = h_s \times \Pi_H(\mu, \eta, \eta_0) \quad (2)$$

$$M = c \frac{(dP/dh)h_{max}}{\sqrt{A_c}} = m_s \times \Pi_M(\nu, \eta, \eta_0) \quad (3)$$

where A_c is the assumed contact area, Π_H and Π_M are dimensionless functions, and ν is Poisson's ratio. Linear and nonlinear micromechanics provide a convenient way to define these functions [36-40].

Purpose of the work. Controlling the packing density of calcium silicate hydrates from the point of view of the micromechanics of composite media.

Materials and Methods

Experimental studies were carried out on modern high-tech equipment of the Russian Academy of Sciences and leading Russian and foreign universities.

The following raw materials were used:

- for the composite binder (CB): Portland cement CEM 32.5N, an aluminosilicate additive (ASA) obtained by purifying an ash and slag mixture, hydrothermal nanosilica in two forms (sol and powder);
- for the concrete mix: superplasticizer Pantarhit PC 160 (SP), quartz sand (S), and basalt fiber.

From the point of view of the law of similarity, basalt microfiber has similar deformation characteristics with cement paste (linear coefficients of thermal expansion $8 \cdot 10^{-6}$ and $10 \cdot 10^{-6} \text{ } ^\circ\text{C}^{-1}$, respectively). This enhances the adhesion of the CSH-gel to the fiber surface and increases the shear stress of the cement matrix relative to the fiber surface, and thus a synergistic enhancement of the action of the different-scale combination "nanosilica – basalt microfiber" is manifested.

Results and Discussion

The modified composite binder, sand and basalt fiber in dry form were mixed until homogeneous, then a homogeneous mixture of water, nanosilica sol and SP was poured in and mixed (Table 1).

Table 1

Experimental proportions of fiber-reinforced concrete

Mix ID*	Cement	ASA	SiO ₂ **	Water	SP	Sand	Fiber
Ref-1-2	100	-	-	40	1.1	300	1,5
Ref 1-3	100	-	-	40	-	300	1,5
Ref-3	70	30	-	40	1.1	300	1,5
001SAF	69.99	30	<u>0.086</u> <u>0.01</u> <u>0.08</u>	39.72	0.33	300	1,5
1SAF	69.00	30	<u>8.6</u> <u>1.0</u> <u>8.0</u>	31.74	1,1	300	1,5
2SAF	68.00	30	<u>17.2</u> <u>2.0</u> <u>16.0</u>	23.87	1,55	300	1,5
3SAF	67.00	30	<u>25.8</u> <u>3.0</u> <u>24.0</u>	15.88	2,2	300	1,5

Continuation of table 1

2SACF	68.00	30	<u>17.2</u> <u>2.0</u> 16.0	23.87	1,55	300	1,5
3SACF	67.00	30	<u>25.8</u> <u>3.0</u> 24.0	15.88	2,2	300	1,5
001PAF	69.99	30	0.01	39.80	0,33	300	1,5
1PAF	69.00	30	1	39.34	1,1	300	1,5
2PAF	68.00	30	2	39.07	1,55	300	1,5
3PAF	67.00	30	3	38.68	2,2	300	1,5
2PACF	68.00	30	2	39.07	1,55	300	1,5
3PACF	67.00	30	3	38.68	2,2	300	1,5
1LCF	79	-	<u>0.086</u> <u>0.01</u> 0.08	23.74	1,1	300	1,5
2LCF	78	-	<u>86</u> <u>1.0</u> 8.0	15.87	1,55	300	1,5
3LCF	77	-	<u>17.2</u> <u>2.0</u> 16.0	7.88	2,2	300	1,5
4LCF	68	-	<u>25.8</u> <u>3.0</u> 24.0	11.87	1,55	300	1,5

Note:

* LCF - low cement + fiber; SAF - sol + ASA + fiber; PAF – nanopowder + ASA + fiber; SACF and PACF – complex additive SiO_2 + SP.

** For compositions of the SAF and LCF type, in the SiO_2 column: the first line shows the consumption of sol, the second – the consumption of solid SiO_2 nanoparticles in the composition of the sol, and the third – the consumption of the sol

The structure-forming role of the filler is most pronounced with an increase in the interaction surface, these conditions are implemented in fine-grained concrete using sand from the Razdolnenskoye field based on the CB, which, due to a highly developed surface, allow intensifying the processes of structure formation and accelerating hardening at an early age, as well as densifying the structure.

The effect of increasing the compressive strength of cement composites modified with sol and basalt microfiber (Figure 1) increased with an increase in the SiO_2 dosage from 0.01 to 3.0% of the cement mass, with particular manifestation at an early age.

Fig. 1. Dependence of the flexural strength of the samples on the dosage of the sol of SiO₂ nanoparticles wt. %

The synergistic effect of two materials of different scales can be estimated by comparing the absolute value of the increment in flexural strength with a dosage of SiO₂ nanoparticles in an amount of 3 wt. % for the composition of SF groups (sol + fiber) relative to the control in group S (modification with sol) with a simple sum of absolute increments of flexural strength due to the independent modifying action of SiO₂ nanoparticles and microfiber: at the age of 1 day is 2.8 MPa (relative contribution 82.9% of SiO₂ nanoparticles, 7.1% of microfiber) and 1.2 MPa (2.33 times more); 7 days age is 4.1 MPa (48.8% – SiO₂ nanoparticles, 51.2% of microfiber) and 3.2 MPa (1.28 times), 28 days is 8.0 MPa (41.25% of SiO₂ nanoparticles, 58.75% of microfiber) and 6.2 MPa (1.29 times higher), for the 3SFC composition is 9.3 MPa (49.5% of SiO₂ nanoparticles, 50.5% of microfiber) and 6.7 MPa (1.38 times higher). The relative contribution of nanoparticles to the synergistic effect is higher at an early age (1 day) due to a relatively larger increase in the volume of CSH gel at this stage of alite hydration and a relatively larger contribution of the pozzolanic reaction.

SiO₂ nanoparticles accelerate the kinetics of C₃S hydration and participate in the pozzolanic reaction, increasing the volume fraction of calcium silicate hydrates CSH (I) with a low Ca / Si ratio, and ultimately promoting an increase in the volume of the CSH gel. In addition, SiO₂ nanoparticles increase the bulk packing density η of nanogranules in the HD phase of the CSH gel, as a result of which the mechanical characteristics of the phases - M, H increase. the fact that within the volume of a denser HD-phase the adhesion of the CSH-gel to the microfiber surface is enhanced, the shear stress of the CSH-gel relative to the fiber surface increases, and, thus, a synergistic enhancement of the action of the different-scale combination "SiO₂ nanoparticles and basalt microfiber" is manifested.

The effect of the increment in compressive and flexural strength, referred to the dosage of SiO₂ nanoparticles, decreased according to power-law dependences, which can be approximated with a high statistical factor R^2 for SF cement composites modified with sol and basalt microfiber, for an age of 28 days by the equations:

$$\Delta R_{\text{com}}^{28} / [\text{SiO}_2] = A / [\text{SiO}_2]^z, A=25.379, z=0.62, R^2 = 0.9832$$

$$\Delta R_{\text{flex}}^{28} / [\text{SiO}_2] = A / [\text{SiO}_2]^z, A=27.699, z=0.66, R^2 = 0.9892$$

Power-law dependences of type $A / [\text{SiO}_2]^z$ for $\Delta R_{\text{com}} / [\text{SiO}_2]$ and for $\Delta R_{\text{flex}} / [\text{SiO}_2]$ for compositions of SF, PF groups with ages of 1 and 7 days were found by approximating experimental data with high statistical factors R^2 .

According to the results presented, there is a high early strength. In particular, for samples with a sol of nanosilica, an almost doubling of the ultimate strength in compression is traced for 1 day at a dosage of SiO₂ in an amount of 3%. In addition, a monotonic increase in strength with an increase in the SiO₂ dosage is characteristic. In addition, a noticeable effect can be traced with the combined introduction of SiO₂ and SP, when they are premixed. Combinations of "fiber + sol" and "fiber + nanopowder" with a quadrupling of strength have a significant effect on the flexural strength. Successful approximations of the compressive strength and bending strength on the SiO₂ dosage are traced for compositions with the inclusion of sol and sol with fiber for different ages (1, 7, and 28 days) with a regular behavior of the coefficients in power-law dependences. For compositions with the inclusion of SiO₂ nanopowder and silica nanopowder with a fiber, the approximations are similar).

Water resistance of heavyweight concrete modified with sol of SiO₂ nanoparticles in an amount of 1.0 wt. %, was determined according to the Russian standard GOST 12730.5. In the absence of water filtration on the opposite face of the cube sample with dimensions of 100 × 100 × 100 mm³ for 12 hours at a pressure of 1.0 MPa, the water resistance grade of the control unmodified samples corresponded to W8. Samples modified with SiO₂ nanoparticles at a dosage of 1.0 wt. %, showed the absence of filtered water at a pressure of 1.8 MPa, and corresponded to the W18 grade for water resistance. When modifying heavyweight concrete with SiO₂ nanoparticles, in accordance with the kinetic curves of water absorption, the average diameter and volume of capillary pores decreased.

In this study, cement composites of the group (SA and PA) and group (SF and PF) showed the same grade for water resistance depending on the dosage of SiO₂ nanoparticles, which depends on the average diameter and volume of capillary pores: at a dosage of SiO₂ nanoparticles 0.01-0.1 wt. % are W10-W12 (1.0-1.2 MPa), 0.25-1.0 wt. % are W14 (1.4 MPa), 2.0 wt. % is W16 (1.6 MPa), 3.0 wt. % is W18 (1.8 MPa) (Fig. 2).

Fig. 2. Dependence of the working pressure corresponding to the water resistance grade on the dosage of SiO₂ nanoparticles

Statistical correlations were found between the mechanical characteristics of R_{com}^{28} and R_{flex}^{28} and the filtration coefficient K_f (Fig. 3). The filtration coefficient K_f is defined as the ratio of the volume of water Q_f filtered through the sample cross section for a certain time τ to the pressure drop between the sample faces in the filtration direction ($P - 0.1$ MPa) multiplied by the dynamic viscosity of water μ at a temperature of 20°C, ρ_w is the density water, g is the acceleration of gravity and the distance between the sample faces δ : $K_f = (\mu \cdot \rho_w \cdot g \cdot \delta \cdot Q_f) / (P - 0.1) \cdot \tau$. With an increase in the dosage of SiO₂ nanoparticles from 0.0 to 3.0 wt. % in compositions S modified by sol, the K_f coefficient decreased from $1.36 \cdot 10^{-6}$ cm / s to $0.329 \cdot 10^{-6}$ cm / s. For the correlation ($R_{com}^{28} - K_f$) the polynomial function corresponded better, correlations ($R_{flex}^{28} - K_f$) - the linear function (Fig. 3):

$$R_{com}^{28}: y = 7.0 \cdot 10^{12} \cdot x^2 - 2.0 \cdot 10^7 x + 51.853, R^2 = 0.9624$$

$$R_{flex}^{28}: y = -1.0 \cdot 10^6 \cdot x + 5.6299, R^2 = 0.9342$$

Fig. 3. Statistical correlations between the mechanical characteristics of cement composites S and the filtration coefficient. a) $R_{com}^{28} - K_f$, b) $R_{flex}^{28} - K_f$

The frost resistance of cement composites was determined by the number of freeze-thaw cycles, after which the compression strength loss did not exceed 10% and the weight loss did not exceed 2% relative to the control samples, and there were no cracks and chips. The frost resistance grade corresponded to the number of cycles. With a dosage of nanosilica in an amount of 0.25 wt. % for the compositions of cement composites modified with SiO_2 nanoparticles and a combination of SiO_2 nanoparticles – basalt microfiber, the number of completed cycles increased from 200 to 300 (Fig. 4).

Fig. 4. Loss of compressive strength in freeze-thaw cycles

Fig. 5 shows the kinetic curves of water absorption of concrete samples. Fig. 5 and Table 2 that the relative rate of weight gain in the modified concrete samples was lower, and this difference in the relative rate of weight gain increased with raising SiO₂ dosage. The total pore volume of the modified concrete was lower and decreased with increasing SiO₂ dosage. The porous structure index λ and the pore homogeneity index α , calculated from the results of the water saturation kinetics $W_t(t) = W_{\max} \left[1 - e^{-(\lambda t)^\alpha} \right]$ according to the Russian standard GOST 12730-4 changed depending on the dosage of SiO₂; where $W_{\max}$ is the maximum change in concrete mass as a result of water absorption.

Fig. 5. Kinetics of water absorption W_t of samples modified with SiO₂ sol:
a) in the range of 0-12 days, b) in the range of 0-24 hours

Table 2

Water resistance characteristics of the developed composites

Sample	Filtration level / water pressure	Duration, hour: min	Grade
Ref-1-1	5 / 1.0 MPa	66:51	W10
001SAF	6 / 1,2 MPa	82:27	W10
1SAF	6 / 1,2 MPa	81:29	W10
2SAF	10 / 2.0 MPa	146:55	W18
3SAF	9 / 1,8 MPa	133:24	W18
3SACF	10 / 2.0 MPa	147:42	W18

The λ index decreased, and the α index increased with a raise in the SiO₂ dosage, which corresponded to a decrease in the average pore diameter and an increase in the homogeneity of the differential distribution of pore volume over the diameter. The following values of the parameters λ and α were established for different dosages of SiO₂:

Ref-1-1 – control sample, maximum water saturation (according to drying data) $W_{\max} = 6.28\%$, $\lambda = 1.50$, $\alpha = 0.30$;

001SAF – sample with addition of SiO₂ = 0.01%, $W_{\max} = 6.27\%$, $\lambda = 0.239$, $\alpha = 0.469$,

1SAF – sample with addition of SiO₂ = 1%, $W_{\max} = 5.72\%$, $\lambda = 0.080$, $\alpha = 0.706$,

2SAF – sample with addition of SiO₂ = 2%, $W_{\max} = 5.41\%$, $\lambda = 0.0542$, $\alpha = 0.824$. Taking into account that the porous structure index λ is proportional to the 3rd degree of the average pore diameter, at a dosage of SiO₂ = 3%, λ decreased by 28.4 times compared with the control sample, and the average pore diameter, respectively, decreased by 3.05 times. The relative weight loss during drying was lower for the modified concrete and decreased with increasing SiO₂ dosage. At the end of drying, the weight reduction for unmodified concrete was 6.25%, for modified concrete at a SiO₂ dosage of 0.01 wt. % is 6.2%, 2.0 wt. % is 5.7%, 3.0 wt. % is 5.4%. Four days after the start of drying, the weight reduction in unmodified concrete was 4.1%, with a SiO₂ dosage of 0.5 wt. % is 3.8%, 2.0 wt. % is 3.4%, 3.0 wt. % is 2.9%.

Thermal resistance is an important performance characteristic for runways and spaceport coatings. The number of thermo-shifts causing the destruction of the sample at different temperatures is given in Table 3. The increase in thermal resistance of the developed composites is explained by the influence of nanosilica on the formation of the concrete structure, especially in the interfacial transition zone between the binder matrix, sand grains and fiber. Heat resistance is increased due to the introduction of basalt microfiber. However, nanosilica turned out to have a stronger effect on thermal stability in the compositions SAF, PAF, and LC. With an increase in the dosage of nanosilica, the heat resistance parameters increased. Moreover, according to their dependence on the SiO₂ dosage, it can

be seen that a further increase in the dosage will lead to a significant increase in performance. These results cannot be explained by the influence of nanosilica alone; here the synergistic effect of the composite binder is traced, as well as the interaction of sand particles with the microfiber surface.

Table 3

Heat resistance of the developed composites			
Mix ID	Number of heat cycles before destruction		
	700°C	900°C	1100°C
001SAF	5	5	1
1SAF	10	8	3
2SAF	12	9	5
3SAF	15	12	7
001PAF	6	4	2
1PAF	9	9	3
2PAF	13	9	4
3PAF	14	13	8
1LC	8	7	3
2LC	11	8	4
3LC	14	11	6
4LC	16	13	8
Ref-1	4	3	1
Ref-1-1	8	6	2
Ref-2	5	4	0
Ref-3	7	6	1

The effect of the influence of SiO_2 nanoparticles is more pronounced on compositions 2LC, 3LC and, especially, 4LC, with a forced lower amount of binder. In these formulations, the microfiber-sand ratio was the same as in the SF and PF formulations.

To understand the complex effect of the composition and structure of the composite on its performance characteristics, it is necessary to compare the results for heat resistance with the results for flexural strength in the same compositions. Flexural strength correlates with crack resistance, both under the action of mechanical loading and under the action of stresses arising from temperature gradients.

The most probable mechanism for increasing thermal stability is an increase in the characteristics of the structure of the CSH gel. The introduction of SiO_2 nanoparticles leads, due to an increase in the amount of hydrates and the pozzolanic reaction, to a decrease in the total capillary porosity, a decrease in the λ index of the pore structure and, accordingly, a decrease in the average pore diameter. Also, the bulk density of the packing of particles increases, the modulus of elasticity and the hardness of the phases of the CSH gel increase. As a result, SiO_2 nanoparticles increase the flexural strength and mechanical coefficients of fracture toughness, including under the action of temperature gradients.

No visible effect was observed on the surface of the samples heated to 700°C. Large cracks and partial cracking of the samples were observed at 900°C. Samples lost integrity at 1100°C. The properties of concrete after exposure to temperature can be assessed by observing the color change of the concrete.

The degree and nature of chipping, flaking and weight loss varied. Chipping was considered negligible when only superficial corrosion occurred. However, cracks can affect the thermal stability and load-bearing capacity of a structural member if it subjects the fiber to rapid heating. Thus, the effects of flaking depend on the concrete application. For example, cracks, which in most structures can be considered minor, pose serious problems for concrete runways used for military aircraft.

The products obtained as a result of studies of materials for special-purpose road surfaces were protected by RF patents for invention:

- No. 2691035, priority date 07.12.2018 "Highway";
- No. 2665324, priority date 05.10.2017 "Conductive concrete".

Conclusions

1. Scientifically substantiated and experimentally confirmed the possibility of obtaining effective fiber-reinforced concretes on the developed modified composite binder, providing high strength properties ($R_{com} > 48$ MPa, $R_{flex} > 13$ MPa) and performances (water resistance grade – W18, frost resistance grade – F300, high heat resistance in the temperature range of 700-1100°C), which makes it possible to use these composites for runways of airfields and cosmodromes.

2. It was revealed that SiO₂ nanoparticles lead to: acceleration of C₃S hydration; to the appearance of an additional amount of calcium silicate hydrates CSH (I) due to the pozzolanic reaction; an increase in the volume of CSH-gel and a decrease in the porosity of the solid cement composite; acceleration of the polymerization of silicon-oxygen tetrahedra, an increase in the degree of their polymerization; a decrease in the Ca / Si ratio in the phases of the CSH gel; the formation of a more ordered structure of the CSH gel with an increased proportion of the HD phase with the bulk packing density of granules $\eta = 0.78$ and higher average values of M, H compared to the LD phase with $\eta = 0.66$. It has been established that the mechanical fracture toughness coefficients increase with a raise in the volume fraction of the HD phase, which has higher mechanical characteristics compared to the LD phase.

3. From the point of view of the law of similarity, basalt microfiber has similar deformation characteristics with cement paste (linear coefficients of thermal expansion $8 \cdot 10^{-6}$ and $10 \cdot 10^{-6} \text{ }^{\circ}\text{C}^{-1}$, respectively). This enhances the adhesion of the CSH-gel to the fiber surface and increases the shear stress of the cement matrix relative to the fiber surface, and thus a synergistic enhancement of the action of the different-scale combination "nanosilica - basalt microfiber" is manifested. It was found that with an increase in the dosage of SiO₂ nanoparticles, the nature of the destruction of cement composites changed: with a dosage of SiO₂ of 0.5 wt. % and higher, the number of cracks increased, their width decreased, and the tortuosity coefficient K_{curv} increased.

4. It was revealed that the effective increment in compressive and flexural strength, related to the dosage of nanosilica, decreases according to power-law dependences, which can be approximated with a high statistical factor R^2 for modified composites. High values of early strength were established, in particular, with a dosage of nanosilica in an amount of 3%, a doubling of the compressive strength at the age of 1 day is observed. In this case, the combination of microfiber – nanosilica with a quadrupling of this characteristic has a synergistic effect on the flexural strength.

5. The positive influence of the composition of the developed composite on the performances has been proved. The water resistance of the modified composite provides a water pressure of 2 MPa for 148 hours, which corresponds to the W18 grade (for the control sample – W8), the frost resistance grade – F300. It was found that the water absorption of the modified concrete samples was lower than that of the control sample, which is explained by the decrease in the pore structure index λ by 28.4 times, and the average pore diameter by 3.05 times. The total pore volume of the modified concrete was lower and decreased with increasing dose of nanosilica.

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