

Original article

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Influence of Activation Methods on the Structural and Technological Characteristics of Nanomodified Cement Compositions

Natalia O. Kopanitsa¹ , Olga V. Demyanenko¹ , Anzhelika A. Kulikova^{1*} , Svetlana V. Samchenko² ,
Irina V. Kozlova² , Nadezhda A. Lukyanova² 

¹ Tomsk State University of Architecture and building, Tomsk, Russia

² National Research Moscow State Civil Engineering University, Moscow, Russia

* Corresponding author: e-mail: lika.panda.19@gmail.com

ABSTRACT: Introduction. We studied the effect of nanosized silicon dioxide (SiO₂) on the structural and technological characteristics of nanomodified cement compositions introduced together with activated mixing water. **Materials and research methods.** Activation of mixing water was carried out by means of magnetic field and ultrasonic action. For investigation the capability to maintain their properties for a long time, the stability of silica suspensions in activated water was studied. For finding out the effect of activated silica suspensions on the structure and properties of composite materials based on cement, the physical and mechanical properties of the studied compositions were explored. X-ray and differential thermal analysis of the hardened activated nanomodified cement paste were also carried out. **Results and discussion.** The positive role of the suspension of silicon dioxide in activated water was associated with a decrease in the microheterogeneity of the hardened cement paste, ensuring the stability of its physical and mechanical characteristics. Based on the above mentioned observations, a mechanism was proposed for more efficient incorporation of nanosized silicon dioxide into cement hydration processes both due to chemisorption with Ca(OH)₂ in the hardening cement paste and due to the topological effect of nanoparticle localization in defects and ultramicrovoids of a crystallizing disperse system. **Conclusion.** The results show that suspensions of silica in activated water can maintain their properties for a long time. Graphical dependencies are shown, indicating the effectiveness of the use of activated silica suspensions in the production of cement composites. This quality makes it possible to obtain repair compounds with the required properties during construction work for various purposes.

KEYWORDS: cement compositions, nanomodifiers, magnetic treatment, ultrasonic treatment, mixing water.

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INTRODUCTION

Currently, much attention is paid to the development of nanotechnologies in various fields of science and technology. Promising nanodispersed materials include nanocomposites based on silicon compounds [1–3]. The effect of nanomodification on the structure, phase composition, and properties of materials is being actively studied [4–6, 24–28]. The introduction of nanotechnology

elements into the system is accompanied by intentional molecular engineering, neoformations, nanostructures, nanosystems and nanoobjects [7, 8].

Most frequently, nanosized silica particles are considered as growth stimulants in biotechnology for the development of the agro-industrial complex [9, 10].

For the construction industry, nanosized silicon compounds are also of interest [11–14]. For example, it was shown in [15] that the introduction of nano-SiO₂ and

nano- Al_2O_3 into cement at low relative humidity (60% relative humidity) and low air pressure (50 kPa, 60 kPa, 70 kPa) contributes to compaction of the microstructure, increasing the degree cement hydration and improved flexural and compressive strength, in contrast to samples that do not contain nanocomponents.

The purpose of this research is to establish the influence of activation methods on the structural and technological characteristics of nanomodified cement compositions by activating the mixing water under the influence of a magnetic field and ultrasonic action.

The analysis shows that the processes of hydration and structure formation of cement stone can be “activated” by affecting the mixing water. This leads to intensification of the processes of structure formation, modification of the structure and properties of composite materials. In [16, 17], changes in the structural, optical, kinetic, magnetic, and other physicochemical properties of the studied cement systems are noted. The external influences (chemical, physical) of the field on the components of a disperse system is considered both at the microlevel are considered: from the standpoint of the rearrangement of electron shells, and at the macrolevel, where structural macroscopic formations are modeled, as well as when using various kinds of nanoadditives at the level of formation of nanostructures in composite materials [18, 19]. Despite the promise of using activated water, it has not yet been widely used in construction technologies [20, 21]. The effect of magnetic activation of mixing water on the properties of composites with a mineral matrix and on the course of their structure formation processes is associated with a change in the properties of the treatment object itself, as well as with the intensity and degree of structure restructuring, as well as with a change in the degree of cement hydration in solid and liquid phases. In the technology of repeated magnetic activation of mixing water, regardless of its type, additional oxygen is introduced into the treatment object and a magnetic field is applied simultaneously, which stimulates the course of processes with a change in the energy saturation of water during treatment and its holding after activation. The treatment of a liquid by a rotating magnetic field enhances its effect on the molecules as a result of the orientational action, which reduces to their combination into complexes. In liquids with polar molecules, the orientational polarization is more pronounced. The electrostatic field is a special case of the electromagnetic field, therefore, the phenomena occurring in the liquid under the influence of this field will be similar, because the magnetic moments of the electrons interact with the external field. The paper presents comparative data on the assessment of the influence of different nano- SiO_2 content of the modes of cyclic magnetic activation of water and cement slurry on the strength characteristics of cement stone and the water demand of cement paste.

MATERIALS AND RESEARCH METHODS

Portland cement CEM I 42,5N from the Topkinsky cement plant GOST 31108-2020 was used as a binder.

The chemical and mineralogical compositions of Portland cement clinker are shown in Table 1.

Silicon dioxide obtained by the plasma-arc method was used as a modifying additive [22, 23]. To obtain a nanopowder, diatomite from the Kamyshlovskoe deposit of the Sverdlovsk region in Russia was used as a raw material. Characteristics of nano-modifier silicon dioxide (SiO_2) is given in Table 2, micrograph of nano- SiO_2 and size distribution curve of SiO_2 nanoparticles are shown in Figures 1 and 2, respectively.

As can be seen from Figure 1, SiO_2 nanoparticles with a polydisperse size distribution have a characteristic spherical shape and are presented in the form of agglomerates.

Based on the data of micrographs, the size distribution of nanoparticles (for at least 1000 particles) was estimated using the iTEM software (Olympus, Japan). The diagram presented in Figure 2 shows that the nanoparticles of the resulting powder have a size distribution in the range from 10 to 300 nm, but their largest number (82% vol.) is in the range up to 100 nm. The distribution peak falls on particles 11–20 nm in size (13% vol.) [24].

Characteristics of nanomodifiers based on SiO_2 used in the work (Ts – nanosized particles; T – finely dispersed particles) are presented in Table 3.

When conducting experimental studies for mixing cement, water was used that meets GOST 23732-2011. The silicon dioxide nanomodifier was introduced into the water before it was activated in an amount of 0,01–0,05% by weight of the cement.

Table 1
 Portland cement clinker and its main components

Chemical analysis of Portland cement, %		Mineralogical composition of clinker (main components of the phase, %)	
CaO	61.9	C_3S	68.9
SiO_2	19.8		
Al_2O_3	4.6	C_2S	12.6
Fe_2O_3	3.0		
MgO	3.6	C_3A	6.0
SO_3	2.8	C_4AF	11.4
R_2O	0.9	CaO	1.1

Table 2
Main characteristics of the nanomodifier (SiO₂)

Indicator name	Indicator value
Appearance	Ultrafine powder, gray
Mass fraction of oxides, %, not less than	
SiO ₂	95.0
Fe ₂ O ₃	0.11
Al ₂ O ₃	0.17
CaO	0.25
R ₂ O	1.9
MgO	1.0
P ₂ O ₅	1.0
SO ₃	0.6
Mass fraction of water, % no more than	3.0
Mass fraction of losses on ignition, % no more than	1.06
Specific surface, m ² /g	38
Average particle size, nm	40

To activate mixing water by means of a magnetic field, an installation was used, which is a rack with magnetic funnels successively attached to it (Figure 3).

Treatment of water mixing cement paste was carried out in a magnetic funnel in the field of a permanent magnet. One cycle of water treatment was considered

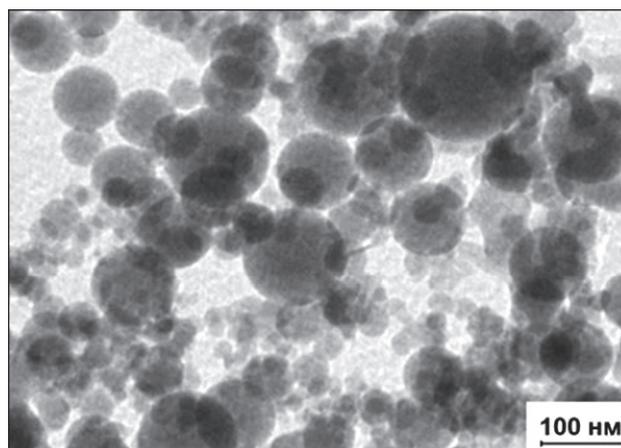


Fig. 1. Micrograph of SiO₂ nanopowder

Table 3
Characteristics of nanomodifiers

Characteristic	Specific surface, m ² /g	Average particle size, nm
Ts10	10	45.00
Ts38	38	37.12
Ts59	59	48.34
T84	82.9	43.71
T90	92	41.50
T110	111	24.50
T140	140	22.61

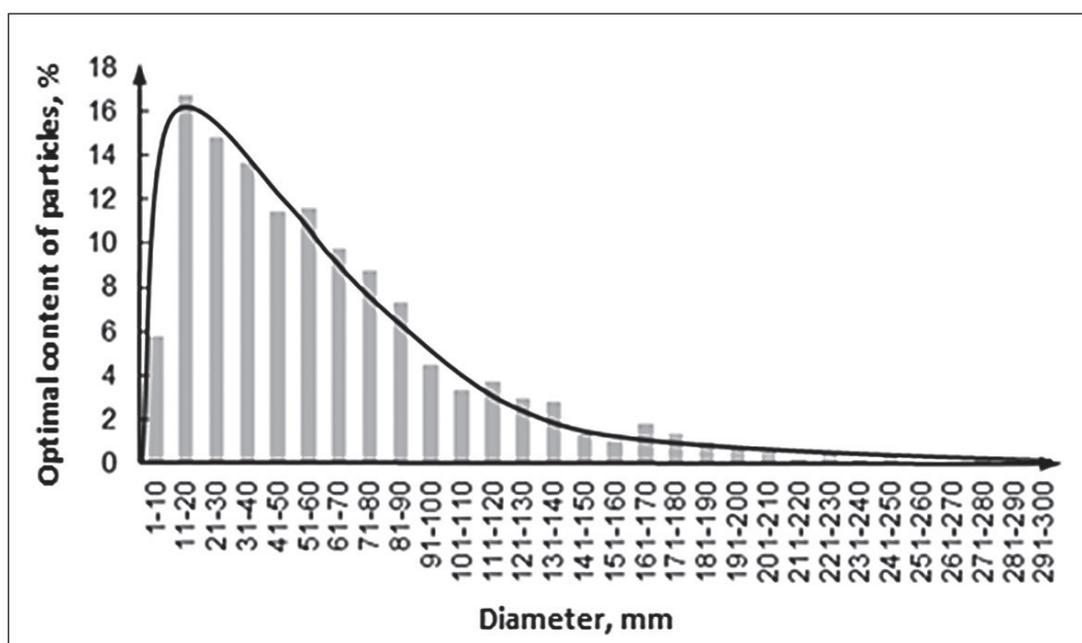


Fig. 2. Size distribution diagram of SiO₂ nanoparticles

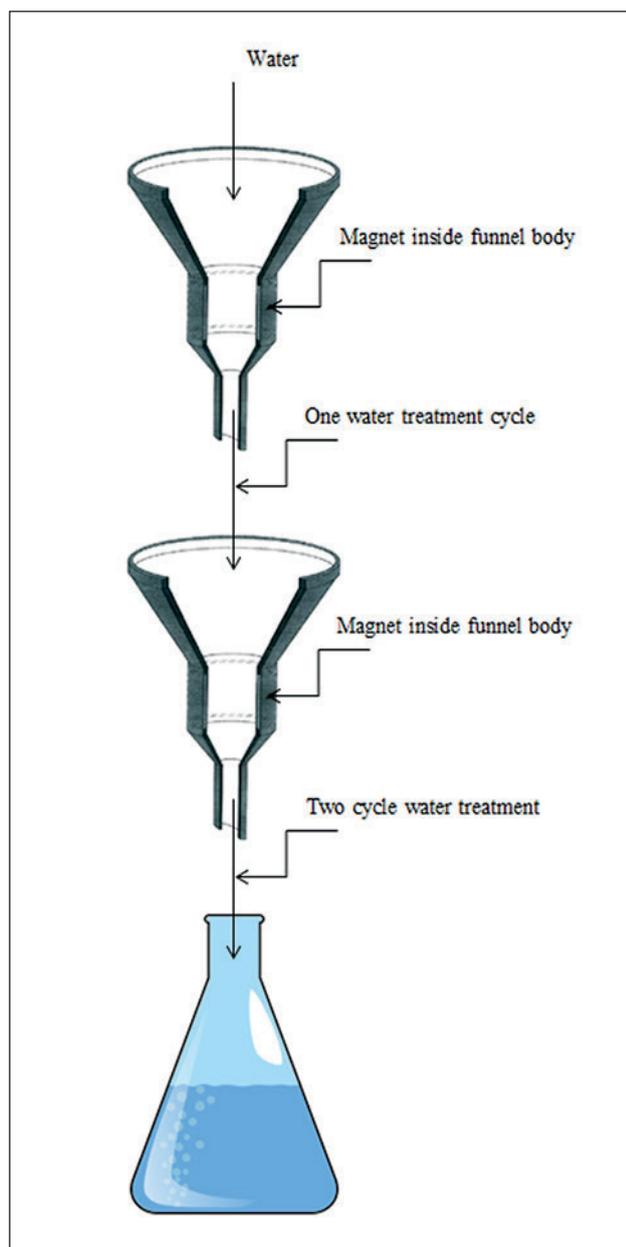


Fig. 3. Scheme of magnetic activation of mixing water

the passage of water through one funnel equipped with a magnetic field.

After reaching the required number of cycles of magnetic water treatment, the cement was mixed with mixing water and a nanomodifier until a cement paste of normal density was obtained.

Mixing water activation by ultrasonic treatment was carried out in an ultrasonic bath YAXUN YX-3560 (30W/50W). Specifications: power: 30/50 W; operating frequency: 42 kHz; timer: 1–30 minutes (auto-off). Mixing water was treated for 3 minutes at a power of 50 watts. The suspension of the silica nanomodifier prepared in this way was used to obtain a cement paste of normal density.

The studies were carried out on cube samples $20 \times 20 \times 20$ mm in size, made from cement paste of normal density, hardening under normal conditions ($T = 18\text{--}20^\circ\text{C}$, $W = 90\text{--}100\%$). The compressive strength of the samples was evaluated at 28 days of curing. For comparative analysis, modified compositions and control Portland cement were investigated. At least 5 samples were prepared for each composition. The strength value was determined as the arithmetic mean value of 5 samples, the coefficient of variation was not more than 5%.

The hydration products of the hardened cement paste were determined by X-ray phase analysis and derivatographic analysis.

X-ray phase analysis was performed on an XRD-6000 diffractometer (Shimadzu, Japan) using $\text{CuK}\alpha$ radiation. The analysis of the phase composition was carried out using the PCPDFWIN and PDF-4+ databases, as well as the POWDERCELL 2,5 full-profile analysis program.

The features of phase transformations in the cement system and the change in the mass of chemically bound water were determined by derivatographic analysis using an STA 449 F3 Jupiter device.

RESULTS AND DISCUSSION

Obtaining and stabilization of suspensions of silicon dioxide nanomodifier.

To establish the aggregative stability of the silicon dioxide (SiO_2) nanomodifier, the prepared solutions were poured into cylinders with a volume of $V = 100 \text{ cm}^3$ and the process of their sedimentation was observed. During the experiment, already 50–70 minutes after dispersion, particles with a content of 0,05% of the nanomodifier began to settle, after 90 and 180 minutes, respectively, 0,03% and 0,01% of the particles of the nanomodifier. Consequently, sedimentation proceeds most rapidly in mixing water containing 0,05% nanomodifier silicon dioxide (SiO_2), and mixing water containing 0,01 and 0,03% nanomodifier is the most aggregatively stable. The dependence of the deposition rate of the nanomodifier silicon dioxide (SiO_2) on their concentration is shown in Figure 4.

As can be seen from the presented data (Figure 4), the stability of systems at low concentrations of the nanomodifier (from 0,01 to 0,03%) is commensurate, and in systems with a concentration of these particles from 0,04 to 0,05%, where mixing water was used activated by magnetic fields are more stable. In systems treated with ultrasonic treatment, they begin to settle after 50 minutes, compared to 70 minutes fixed for systems activated by magnetic field.

Such stability of water-silicon dioxide (SiO_2) nanomodifier systems, activated by magnetic field, may be due to the increased effect on both water molecules and

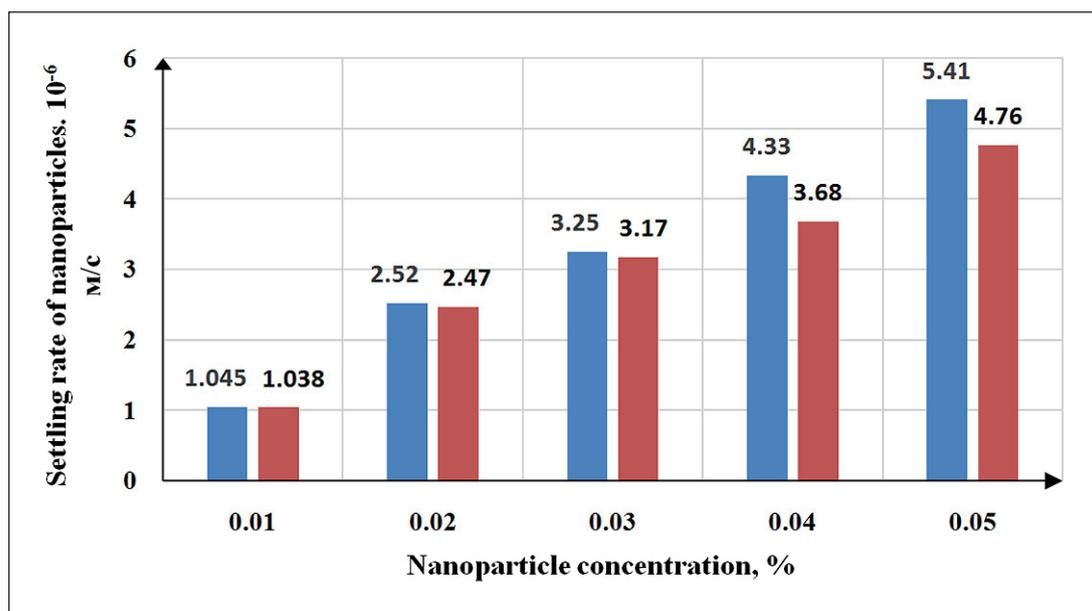


Fig. 4. Dependence of the particle settling rate on the concentration of silicon dioxide nanomodifier: 1 – Activated mixing water by magnetic field (MF); 2 – Activated mixing water by means of ultrasonic influence (USI)

SiO₂ nanoparticles as a result of orientational action, which reduces to their combination into aquacomplexes [SiO₂ · nH₂O].

Ultrasonic action on the system water – nanomodifier silicon dioxide (SiO₂) contributes to the dispersion of SiO₂ conglomerates, and water dipoles, surrounding individual particles with an active surface, also form aqua complexes that can act as a substrate for the growth and nucleation of crystal hydrates.

Thus, it can be concluded that the activation of systems water – nanomodifier silicon dioxide (SiO₂) both by magnetic field and by ultrasonic action are effective methods for obtaining activated mixing water containing nanosized particles.

Study of the effect of activated mixing water with silicon dioxide nanomodifier (SiO₂) on the properties of cement paste.

Experimental studies were carried out to assess the influence of structural characteristics of nano-SiO₂, methods for obtaining nanosized particles, a rational ratio in the “cement-nanoadditive” system, conditions for the uniform distribution of nanoparticles in the volume of cement paste and the stability of the obtained characteristics.

The studies were carried out on cube samples 20×20×20 mm in size, made from cement paste of normal density with different ratios of additives. The content of the nano-SiO₂ additive varied from 0,01 to 0,05% by weight of the cement. For research, silicon dioxide nanomodifiers with different values of specific surface from

10 to 140 m²/g were used. To determine the rational way to introduce additives, three options were investigated:

- Method 1 – the additive was pre-mixed with mixing water;
- Method 2 – the additive was mixed with cement until homogeneous, after which it was closed with water;
- Method 3 – the additive was mixed with mixing water, the suspension was subjected to ultrasonication, after which it was added and mixed with cement.

After molding, the samples hardened under normal conditions (T = 18–20°C, W = 90–100%). The compressive strength of the samples was evaluated at 28 days of curing. At least 5 samples were prepared for each composition. The strength value was determined as the arithmetic mean value of 5 samples, the coefficient of variation was not more than 5%. The results of the conducted studies are presented in table 4.

Analysis of the data presented in Table 4 shows that all types of nano-SiO₂ studied in the work provide an increase in the strength of the cement stone in 28 days of hardening from 2 to 38% compared to the control sample. The significance of the effect depends on the modifiable factors. An increase in the specific surface of nano-SiO₂ from 10 to 38 m²/g affects the increase in strength by 32% compared to the control sample, but a further increase in the specific surface does not lead to a significant increase in the strength of the cement stone, which is associated with an increase in the water demand of the binder, due to which further studies were carried out on nano-SiO₂ (Ts38). The analysis of the obtained results, depending on the method of introducing the additive, shows the

Table 4
Characteristics of Cement Stone with Nano-SiO₂

Sample number	Sample	Additive content, % by weight of cement	Water cement ratio	R ²⁸ , MPa		
				With mixing water (1 way)	Dry (2 way)	Ultrasonic processing (3 way)
1.	Control	0.00	0.270	68.1	68.1	72.2
2.	Cement+Ts-10	0.01	0.275	68.3	68.8	74.0
3.	Cement+Ts-10	0.02	0.275	74.1	75.3	76.0
4.	Cement+Ts-10	0.03	0.275	76.0	76.5	81.0
5.	Cement+Ts-10	0.04	0.280	77.0	76.8	82.1
6.	Cement+Ts-10	0.05	0.280	76.7	77.0	83.0
7.	Cement +Ts38	0.01	0.270	67.0	74.0	79.0
8.	Cement +Ts38	0.02	0.265	74.2	81.0	79.8
9.	Cement +Ts38	0.03	0.267	83.0	90.0	95.0
10.	Cement +Ts38	0.04	0.270	84.0	93.7	95.3
11.	Cement +Ts38	0.05	0.275	85.1	94.0	96.1
12.	Cement +Ts59	0.01	0.275	69.0	69.9	84.0
13.	Cement +Ts59	0.02	0.280	73.0	71.0	85.0
14.	Cement +Ts59	0.03	0.285	71.0	71.8	86.6
15.	Cement +Ts59	0.04	0.290	67.0	71.9	87.0
16.	Cement +Ts59	0.05	0.290	71.0	72.6	87.1
17.	Cement +T84	0.01	0.275	76.0	77.0	77.0
18.	Cement +T84	0.02	0.280	75.0	81.0	77.1
19.	Cement +T84	0.03	0.280	64.9	83.0	81.0
20.	Cement +T84	0.04	0.285	77.0	83.9	80.1
21.	Cement +T84	0.05	0.290	78.0	84.0	81.9
22.	Cement +T90	0.01	0.280	64.6	77.0	73.0
23.	Cement +T90	0.02	0.285	72.3	77.2	77.0
24.	Cement +T90	0.03	0.285	75.2	77.3	83.8
25.	Cement +T90	0.04	0.285	75.0	79.0	84.1
26.	Cement +T90	0.05	0.290	76.0	81.0	85.0
27.	Cement +T110	0.01	0.265	66.0	68.0	82.0
28.	Cement +T110	0.02	0.265	70.0	72.0	83.0
29.	Cement +T110	0.03	0.265	68.0	73.0	86.0
30.	Cement +T110	0.04	0.275	65.0	69.0	86.6
31.	Cement +T110	0.05	0.275	64.9	67.0	86.8
32.	Cement +T140	0.01	0.270	68.0	71.0	84.0
33.	Cement +T140	0.02	0.270	69.0	73.0	85.4
34.	Cement +T140	0.03	0.270	69.9	73.3	88.0
35.	Cement +T140	0.04	0.280	71.1	74.5	89.0
36.	Cement +T140	0.05	0.285	73.0	74.9	89.1
37.	Cement +SiO _{2pl}	0.01	0.270	64.0	69.0	71.0
38.	Cement +SiO _{2pl}	0.02	0.270	67.0	64.0	69.0
39.	Cement +SiO _{2pl}	0.03	0.270	82.0	87.0	90.0
40.	Cement +SiO _{2pl}	0.04	0.270	85.0	88.0	93.5
41.	Cement +SiO _{2pl}	0.05	0.280	86.0	89.0	94.1

greatest efficiency when mixing the additive with cement (increase in the strength of the modified cement stone is 38%), as well as with ultrasonic treatment of an aqueous suspension – up to 41% compared with control samples. With the first method of adding the additive, it was noted that nano-SiO₂ particles agglomerate, which prevents their uniform distribution throughout the volume of the mixture and does not give a significant effect of increasing strength. It should also be noted that ultrasonic exposure to an aqueous suspension with nanosized silicon dioxide does not significantly increase the strength of the cement stone, but the technology for producing mixtures becomes more complicated.

The optimal content of nano-SiO₂ additive in cement compositions is 0,03% by weight of cement [22, 24]. From the results presented in Table 4, it can be seen that the increase in the strength of cement stone in 28 days of hardening is 28% for composition 4 (additive content 0,03%), 29,2% for composition 5 (additive content 0,04%) and 30,7% for composition 6 (additive content 0,05%).

Features of phase transformations in the cement system and the change in the mass of chemically bound water, in the temperature range from 20 to 1000°C, were determined by derivatographic analysis (DTA). Figures 5–6 show comparative derivatograms of the control and modified samples of cement stone with TG and DSC curves.

On the derivatogram (Figure 5) of the control sample of cement stone, by the 28th day of hardening, the first peak of the endoeffect is identified at 109°C with a weight

loss of 8,63% due to the removal of free, and then, weakly bound water, the peak area is 366 J/g; the peak of the endo effect at a temperature of 100–120°C corresponds to the dehydration of C₂SH₂, C₂S₃H₂; endoeffect (peak 460°C) with a weight loss of 2,59% refers to the dehydration of CaOH₂, calcium hydrosilicates of the C₂SH(B), C₂SH₂ type peak area 76,59 J/g; the endoeffect (peak 696°C) with a weight loss of 3,29% is associated with the dehydration of highly basic calcium hydrosilicates: tobermorite-like phases, C₂SH(C), C₂SH₂ and CaCO₃ decarbonization.

In the modified cement stone sample, the derivatogram (Figure 6) shows a shift of endoeffects towards higher temperatures; the first peak of the endoeffect, at 115°C, can presumably be explained by the transition of weakly bound water into chemically bound water. An increase in the peak area to 380.6 J/g, compared with the control sample, indicates the formation of a larger amount of nanostructured hydrated compounds. Endoeffects in the range of 440–490°C with a mass loss of 3.16% are responsible for the removal of water associated with partial surface hydration of the original mineral, and endoeffects at a temperature of 700–800°C with a mass loss of 3.91% are associated with complete dehydration calcium hydrosilicate type CSH(I). An increase in the intensity and area of the peaks indicates an increase in the number of hydrate neoplasms.

Comparative X-ray phase analysis of the hydration products of the control cement and cement with Ts38

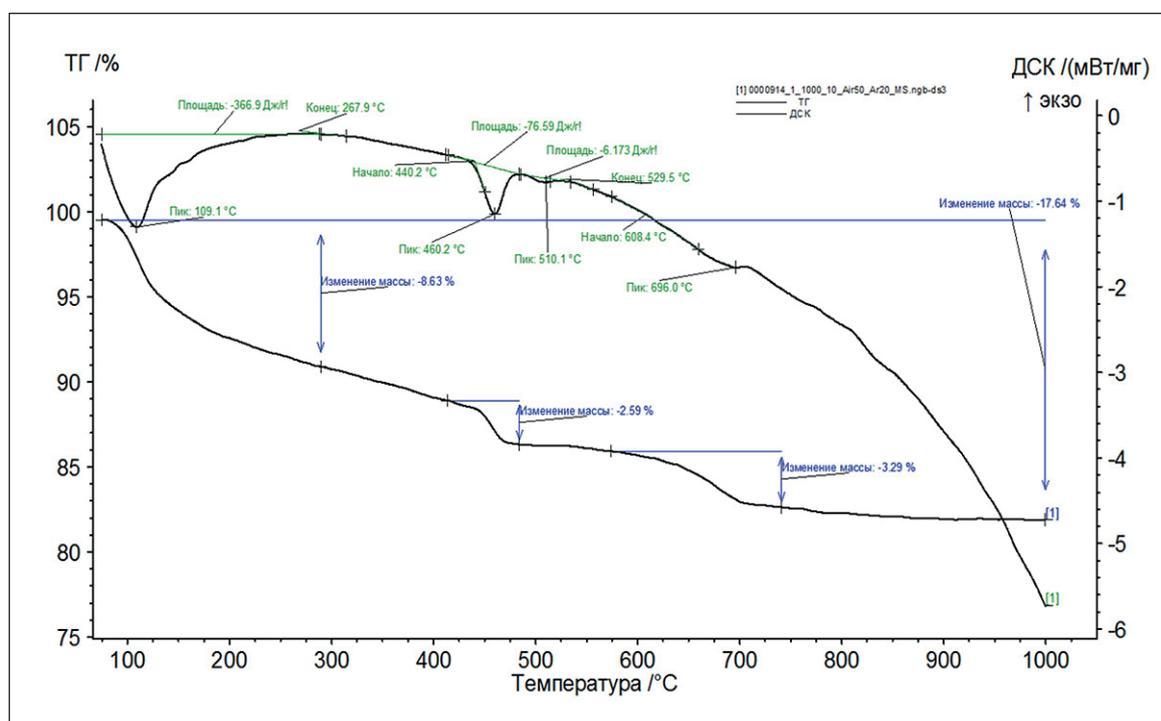


Fig. 5. Derivatogram of the control sample of cement stone in 28 days of hardening

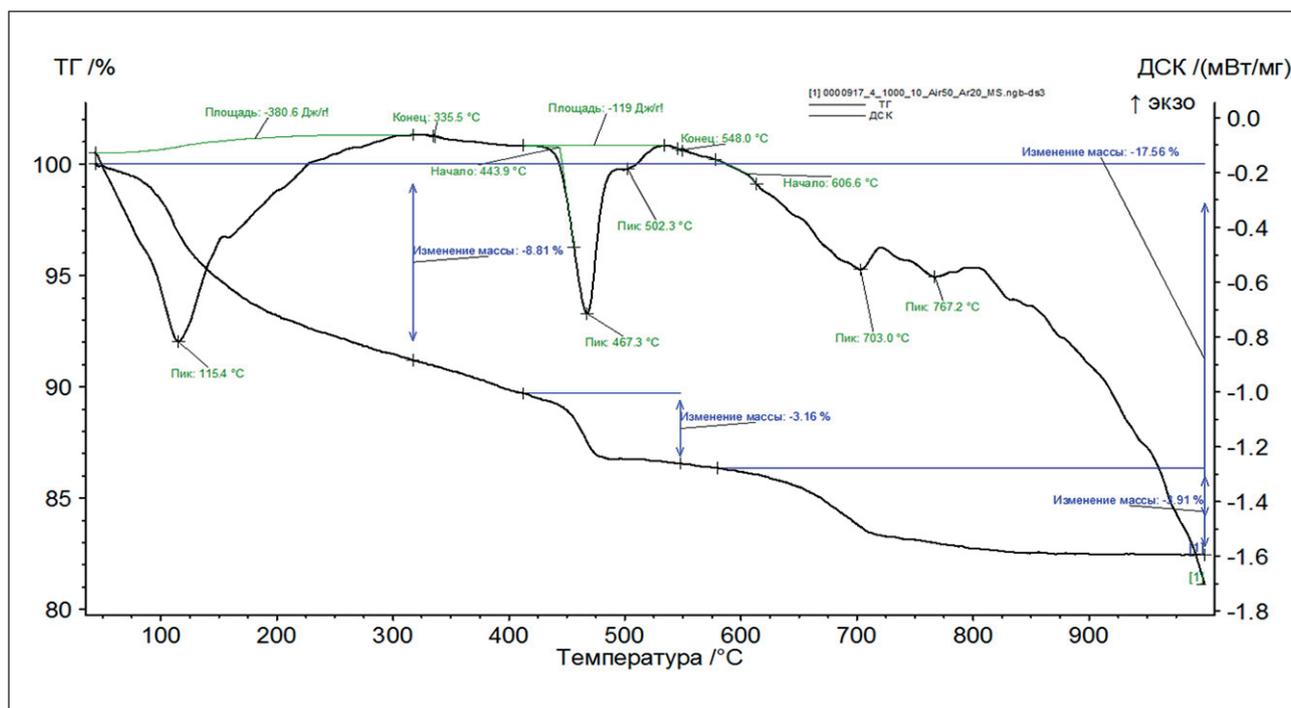


Fig. 6. Derivatogram of a sample of cement stone with the addition of nano-SiO₂ in 28 days of hardening

(Figure 7, 8) confirms the formation of new crystalline phases in the modified cement stone. According to the data of X-ray phase analysis, the introduction of nano-SiO₂ additive activates the binding of calcium hydroxide, contributing to an increase in the content of low-basic calcium hydrosilicates C-S-H ($d/n = 4.94; 2.92; 2.18; 2.06; 1.98; 1.82 \cdot 10^{-10}$ m), which leads to an increase in the strength of the cement stone. A significant decrease in the proportion of free calcium

hydroxide is confirmed on the diffraction patterns by an increased background in the region of small angles and a decrease in the intensity of the peaks of the Ca(OH)₂ crystalline phases ($d/n = 4.9; 2.63; 1.79; 1.48 \cdot 10^{-10}$ m), which is associated with hydration reactions of cement clinker minerals.

To assess the effect of cyclic magnetic activation of mixing water and cement paste with the addition of a nanomodifier, cube samples of 20×20×20 mm in size

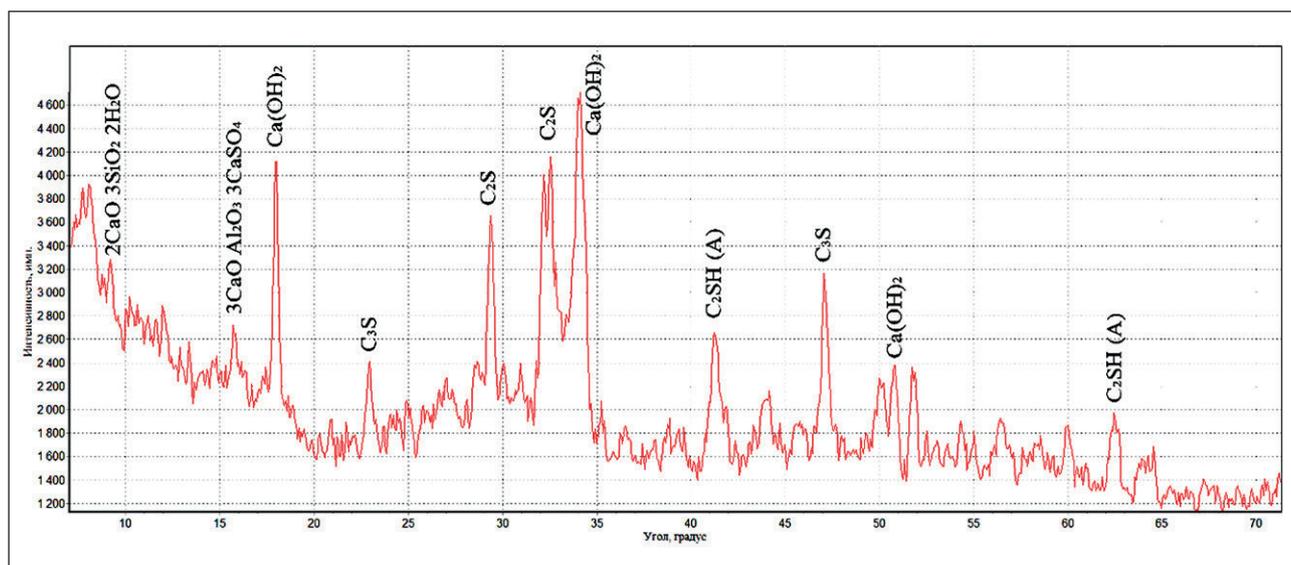


Fig. 7. Diffractogram of the control cement stone in 28 days of hardening

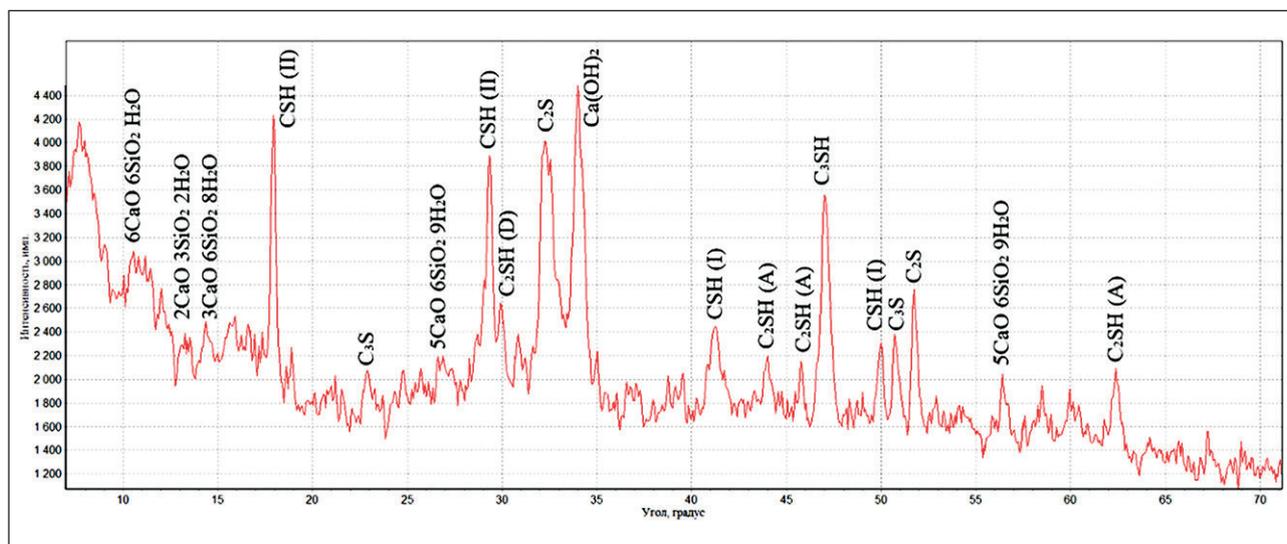


Fig. 8. Diffraction pattern of cement stone on the 28th day of hardening: cement + nano-SiO₂

were prepared. The number of cycles of magnetic treatment of mixing water was 5, 10, 15, 20, 25.

Data on the effect of cyclic magnetic treatment of water for mixing cement paste with the addition of a nano-modifier on the strength of cement stone are presented in Table 5.

Analysis of the data presented in Table 5 shows that with 10 cycles of water treatment for mixing cement paste, it leads to a change in the main physical and mechanical characteristics of the resulting composites. The strength of the nanomodified cement stone is increased by 38% (composition 3) compared to the control composition, without activation. At the same time, for each composition, the coefficient of variation was calculated, which for composition 3 is 4.75%, which proves the effectiveness of the developed method for activating mixing water on the uniform distribution of nanoparticles in the volume

of cement paste. The water demand of cement stone is reduced from 0.27 to 0.255.

Features of phase transformations in the cement system and the change in the mass of chemically bound water, in the temperature range from 20 to 1000°C, were determined by derivatographic analysis (DTA). Figure 9 shows the derivatogram of a nanomodified cement stone sample with activated mixing water.

On the derivatogram (Figure 9) of the modified cement stone, by the 28th day of hardening, the first peak of the endoeffect at 104°C is identified and is associated with the removal of water adsorbed by finely dispersed neoplasms; endoeffect (peak 460°C) with a weight loss of 1.32% and peak (508.1°C) refers to the dehydration of Ca(OH)₂, calcium hydrosilicates of the C₂SH(B) type, C₂SH₂ peak area 26 J/g; the endoeffect (peak 717°C) is associated with the dehydration of highly basic cal-

Table 5

Characteristics of nano-SiO₂ cement stone with magnetic treatment

Sample number	Sample	Additive content, % by weight of cement	Number of processing cycles	Water cement ratio	R ²⁸ , MPa	The coefficient of variation, %
K	C+W	0	0	0.27	68.1	6.25
1	(C+nano-SiO ₂)+W	0.03	0	0.27	87.0	6.9
2	(C+nano-SiO ₂)+W	0.03	5	0.267	89.1	6.1
3	(C+nano-SiO ₂)+W	0.03	10	0.255	94.2	4.75
4	(C+nano-SiO ₂)+W	0.03	15	0.26	91.4	5.1
5	(C+nano-SiO ₂)+W	0.03	20	0.265	93.6	5.36
6	(C+nano-SiO ₂)+W	0.03	25	0.26	93.7	4.99

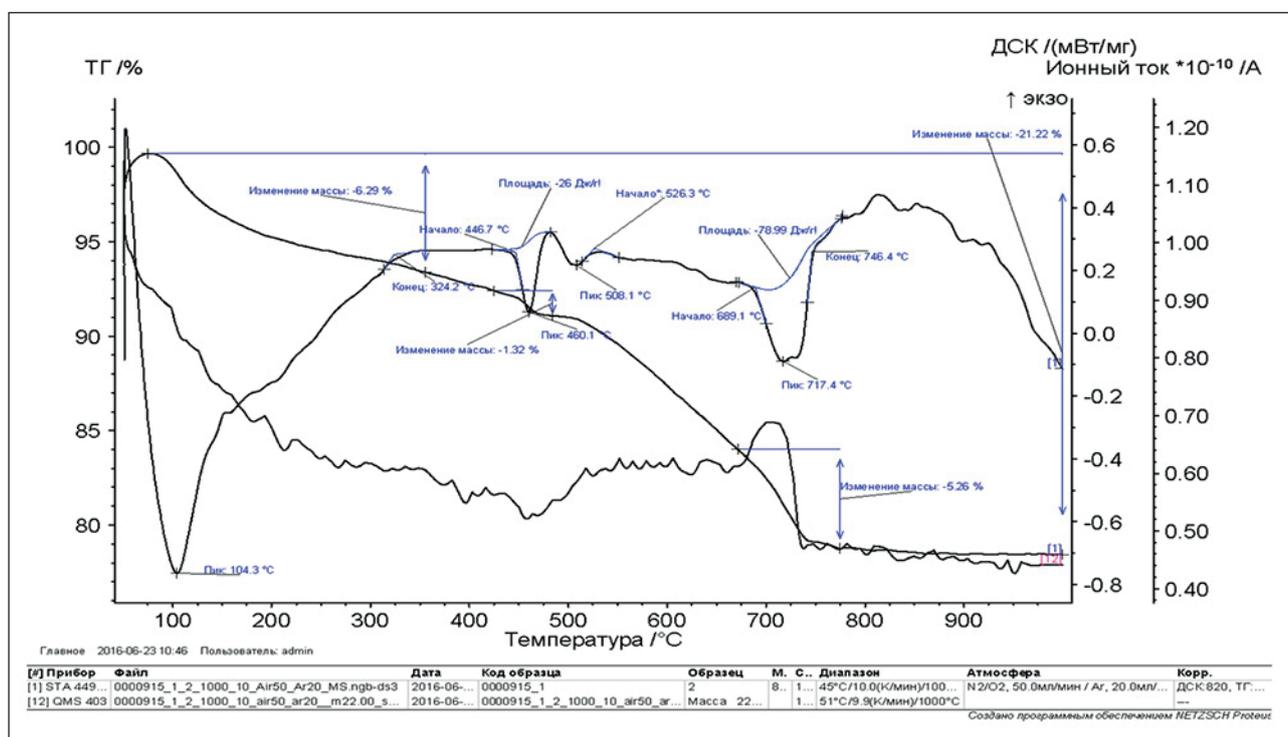


Fig. 9. Derivatogram of a sample of cement stone with the addition of nano-SiO₂ with activated mixing water

cium hydrosilicates: tobermorite-like phases, C₂SH(C), C₂SH₂.

CONCLUSION

The obtained results of experimental studies on the role of magnetic treatment of mixing water together with the use of nano-SiO₂ in composite materials based on Portland cement prove the effective possibility of con-

trolling the structural state and phase composition of the hardening cement stone with a significant increase in its strength. The magnetic activation of mixing water together with the additive improves the quality of the cement stone, ensuring the stability of the obtained characteristics. The established patterns of changes in the physical and mechanical parameters of the studied compositions indicate the effectiveness of the use of water activated by a magnetic field in the production of building materials.

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INFORMATION ABOUT THE AUTHORS

Natal'ya O. Kopanitsa – Dr. Sci. (Eng.), Prof., Tomsk State University of Architecture and Building, Faculty of Civil Engineering, Department of Building Materials and Technologies, Tomsk, Russia, kopanitsa@mail.ru, <https://orcid.org/0000-0002-0991-8550>

Olga V. Dem'yanenko – Senior Lecturer, Tomsk State University of Architecture and Building, Faculty of Civil Engineering, Department of Building Materials and Technologies, Tomsk, Russia, demyanenko.olga.v@gmail.com, <https://orcid.org/0000-0003-0391-808X>

Anzhelika A. Kulikova – Post-graduate Student, Tomsk State University of Architecture and Building, Faculty of Civil Engineering, Department of Building Materials and Technologies, Tomsk, Russia, lika.panda.19@gmail.com, <https://orcid.org/0000-0002-6723-0084>

Svetlana V. Samchenko – Dr. Sci. (Eng.), Prof., National Research Moscow State Civil Engineering University, Institute of Industrial and Civil Engineering, Department of Building Materials, Moscow, Russia, samchenko@list.ru, <https://orcid.org/0000-0002-3523-593X>

Irina V. Kozlova – Cand. Sci. (Eng.), associate professor, National Research Moscow State Civil Engineering University, Institute of Industrial and Civil Engineering, Department of Building Materials, Moscow, Russia, iv.kozlova@mail.ru, <https://orcid.org/0000-0001-8269-5624>

Nadezhda A. Lukyanova – Cand. Sci. (Eng.), associate professor, National Research Moscow State Civil Engineering University, Institute of Industrial and Civil Engineering, Department of Building Materials, Moscow, Russia, galcevanadezda@mail.ru, <https://orcid.org/0000-0003-2014-6739>

CONTRIBUTION OF THE AUTHORS

Natal'ya O. Kopanitsa – scientific leadership; setting goals and objectives of the study; development of research methodology; analysis of research results.

Olga V. Dem'yanenko – literature review; conducting the experimental part of the study; processing results; writing original text.

Anzhelika A. Kulikova – conducting the experimental part of the study; processing results; graphical and tabular presentation of the results.

Svetlana V. Samchenko – setting goals and objectives of the study; development of research methodology; analysis of research results; formation of final conclusions.

Irina V. Kozlova – conducting the experimental part of the study; processing and interpretation of research results.

Nadezhda A. Lukyanova – literature review; processing and interpretation of research results; text revision.

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