ISSN 2075-8545 (online)

Nanotechnologies in construction
Нанотехнологии в строительстве
2026; 18 (3): 307–316

Nanobuild.ru

CONSTRUCTION MATERIALS SCIENCE

Original article
https://doi.org/10.15828/2075-8545-2026-18-3-307-316

High-strength cement-based repair and grouting compositions modified with nanodispersed silica

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ABSTRACT

Introduction. The development of high-strength repair and grouting compositions for restoring concrete structures is a relevant task. The aim of the research is to study the effect of long-term stored nanodispersed silica (NS) on cement stone properties. **Methods and Materials.** Portland cement CEM I 42.5 N and sol-gel NS (storage: 1 day – 1.5 years) were used. Surface acid-base properties – indicator method; hydration kinetics – non-isothermal calorimetry. Four compositions: control, NS, NS + PC type S, NS + PFM. Compressive strength – up to 28 days; SEM, XRD, DTA. **Results.** NS surface has a polyfunctional acid-base spectrum. Long-term storage does not degrade activity: strong acid centers (pKa = –4.4) increase from 7.42 to 260–265 mg eq/g; strong basic centers (pKa = 8.8) remain at 515–628 mg eq/g. NS stored for 1.5 years intensifies heat release (max ~25.5 °C). Compressive strength increase by day 28: NS – 14.5%; NS + PFM – 16.7%; NS + PC type S – 18% vs control. SEM, XRD, DTA confirm portlandite binding and dense low-basic C–S–H matrix formation (min. weight loss on heating – 9.1% for NS+PFM). Discussion. Bifunctional NS surface provides a synergistic effect: basic centers adsorb Ca^{2+} ions, initiating nucleation; Lewis acid centers coordinate OH^- and H_2O, activating them. Complex NS + surfactants improve nanoparticle distribution and intensifying pozzolanic reaction. **Conclusion.** NS activity remains after 1.5 years of storage. Complex NS+plasticizers provide a synergistic effect. Developed compositions are recommended as class R3 (B35) repair compounds and grouting mixtures for critical structures, including collector repair, equipment grouting and anchor bolt installation.

KEYWORDS: nanodispersed silica, cement stone, repair compound, grouting mixture, structure formation, cement hydration, pozzolanic activity, compressive strength, acid-base sites, surfactants, scanning electron microscopy, X-ray phase analysis, differential thermal analysis

SOURCES OF FUNDING FOR THE SCIENTIFIC WORK THAT RESULTED IN THE PUBLICATION: The work was realized within the framework of the implementation of the state task of the Ministry of Science and Higher Education of the Russian Federation No. FZWN-2026-0005 using equipment of High Technology Center at BSTU named after V.G. Shukhov.

FOR CITATION:
Bukovtsova A.I., Kuzmin E.O., Antonenko M.V., Strokova V.V. High-strength cement-based repair and grouting compositions modified with nanodispersed silica. *Nanotechnologies in Construction.* 2026;18(3):307–316. https://doi.org/10.15828/2075-8545-2026-18-3-307-316. – EDN: NTYCYM.

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Высокопрочные ремонтные и подливочные составы на цементной основе, модифицированные нанодисперсным кремнеземом

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АННОТАЦИЯ

Введение. Обоснована актуальность разработки высокопрочных ремонтных и подливочных составов на цементной основе, модифицированных нанодисперсным кремнеземом (НК), для повышения долговечности восстанавливаемых конструкций. **Методы и материалы.** Использован портландцемент ЦЕМ I 42,5 Н и золь-гель НК с разными сроками хранения (1 сутки – 1,5 года). Кислотно-основные свойства поверхности оценены индикаторным методом, кинетика гидратации – неизотермической калориметрией. Исследованы четыре состава: контроль, НК, НК+ПК тип S, НК + ПФМ. Прочность на сжатие определяли до 28 суток. Микроструктуру и фазовый состав изучали методами РЭМ, РФА и ДТА. **Результаты.** Установлено, что поверхность золь-гель синтезированного НК характеризуется полифункциональным кислотно-основным спектром. Длительное хранение не приводит к деградации активности: концентрация сильных кислотных центров (pKa = –4,4) возрастает от 7,42 до 260–265 мг-экв/г, а сильных основных центров (pKa = 8,8) сохраняется на уровне 515–628 мг-экв/г. Методом неизотермической калориметрии подтверждено, что образец НК, хранившийся 1,5 года, не только сохраняет, но и интенсифицирует тепловыделение при гидратации цемента. Физико-механические испытания показали, что к 28 суткам твердения прирост прочности на сжатие составил 14,5% для состава с индивидуальным НК, 16% – для НК + ПФМ и 18% – для НК + ПК тип S по сравнению с контролем. Данные РЭМ, РФА и ДТА подтвердили, что комплексное введение НК с ПАВ способствует связыванию портландита, формированию плотной низкоосновной гидросиликатной матрицы и повышению термической стабильности материала (минимальная потеря массы при нагреве – 9% для состава с ПФМ). **Обсуждение.** Бифункциональная поверхность НК обеспечивает синергетический эффект: основные центры сорбируют ионы Ca^{2+}, инициируя нуклеацию, а кислотные центры Льюиса участвуют в координации гидроксильных групп и молекул воды, активируя их. Комплексные составы НК с ПАВ улучшают распределение наночастиц и интенсифицируют пуццолановую реакцию. **Заключение.** Впервые доказано сохранение активности НК после 1,5 лет хранения за счет формирования бифункциональных центров. Комплексные составы НК с пластификаторами обеспечивают синергетический эффект. Разработаны ремонтные составы класса R3 (B35) и подливочные смеси для ответственных конструкций, включая ремонт коллекторов, подливку оборудования и установку анкерных болтов.

КЛЮЧЕВЫЕ СЛОВА: нанодисперсный кремнезем, цементный камень, ремонтные составы, подливочные смеси, структурообразование, гидратация цемента, пуццолановая активность, прочность на сжатие, кислотно-основные центры, поверхностно-активные вещества, растровая электронная микроскопия, рентгенофазовый анализ, дифференциально-термический анализ

ИСТОЧНИКИ ФИНАНСИРОВАНИЯ НАУЧНОЙ РАБОТЫ, РЕЗУЛЬТАТОМ КОТОРОЙ СТАЛА ПУБЛИКАЦИЯ: Работа выполнена в рамках реализации государственного задания Минобрнауки РФ № FZWN-2026-0005 с использованием оборудования Центра высоких технологий БГТУ им. В.Г. Шухова.

ДЛЯ ЦИТИРОВАНИЯ:
Буковцова А.И., Кузьмин Е.О., Антоненко М.В., Строкова В.В. Высокопрочные ремонтные и подливочные составы на цементной основе, модифицированные нанодисперсным кремнеземом. *Нанотехнологии в строительстве.* 2026;18(3):307–316. https://doi.org/10.15828/2075-8545-2026-18-3-307-316. – EDN: NTYCYM.

INTRODUCTION

In modern construction materials science, one of the most promising areas is the development of highly effective repair and grouting compositions capable of ensuring durability and reliability of concrete and reinforced concrete structures under renovation [1–3]. These materials must comply with higher standards: high compressive strength (at least 25 MPa for class R3 according to EN 1504-3), adhesion to concrete ⩾1.5 MPa, shrinkage resistance, and the ability to achieve early strength gain [4, 5]. A key factor determining the overall properties of these systems is the dense and uniform microstructure of the cement matrix [6, 7].

Traditional methods of modifying cement composites do not always achieve the required level of structural optimization at the nanoscale. Therefore, nanoscale additives capable of specifically influencing the hydration and structure formation processes of cement stone are attracting increased attention from researchers. Among these, nanodispersed silica (NS) occupies a special place. It acts not only as an effective pozzolanic component, binding portlandite to form additional low-basic calcium hydrosilicates (C–S–H), but also as a nucleation center, accelerating the crystallization of new formations [8, 9].

It is important to note that not only the concentration, but also the morphology of nanosilica particles (spherical, aggregated, gel-like) significantly affects the hydration kinetics and final properties of cement composites [21].

It is known that the use of NS in combination with surfactants allows for a synergistic effect due to improved nanoparticle dispersion and additional plasticization of the mixture [10–12]. Similar approaches using complex nanodispersed systems including carbon nanotubes and microsilica also demonstrate significant strengthening of the cement matrix [13, 14]. However, the greater part of the existing studies are devoted to the use of freshly synthesized or commercial NS. The issue of the evolution of the chemical activity of nanodispersed silica during long-term storage (more than 1 year) remains insufficiently studied, which has important practical significance for the industrial production and repair materials logistics. As shown in [15, 16], the surface properties of nanoparticles can be significantly transformed over time; however, no systematic studies have been conducted on the effect of storage periods of sol-gel NS on its activity in cement systems.

Furthermore, the literature extensively presents studies on the modification of cement composites with hydrothermal nanosilica [17], mineral fibers [18], high-strength fiber-reinforced concrete [20], and their combinations [19]. However, the combined effect of long-term stored nanosilica in combination with various types of surfactants on the structure formation processes of cement stone has not been studied sufficiently. There are no data on the kinetics of cement hydration upon the introduction of nanosilica with different storage periods, nor on the nature of the transformation of the microstructure and phase composition under the influence of such additives.

The aim of this work is to study the evolution of the surface activity of nanodispersed silica during long-term storage (up to 1.5 years) and to evaluate its effect on the properties of cement stone when added individually and in combination with plasticizing additives. Based on the obtained results, it is planned to develop formulations for high-strength repair mortars of class R3 (B35) and grouting mixtures for critical structures. To achieve this goal, the following objectives were addressed:

To study changes in the acid-base spectrum of the NS surface depending on storage time (from 1 day to 1.5 years) using an indicator method.

To evaluate the effect of NS with different storage periods on the hydration kinetics of cement paste using non-isothermal calorimetry.

To study the physical and mechanical properties (compressive strength) of cement stone modified with individual NS and its surfactant-containing compositions (PC type S, PFM) at different curing times.

To analyze the microstructure, phase composition, and thermal behavior of the modified cement stone using scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential thermal analysis (DTA).

MATERIALS AND METHODS

The following materials were used for the study:

Portland cement CEM I 42.5 N (AO Sebryakovcement), complying with GOST 31108-2020, with a normal cement paste consistency of 26.2% and setting times of 145 min and 215 min.

Nanodispersed silica (NS) synthesized by the sol-gel method from tetraethoxysilane (TEOS) using the surfactant-stabilizer Span 83 [11]. Samples with different storage periods were studied: 1 day, 1 week, 1 month, and 1.5 years.

Surfactants: polycarboxylate superplasticizer type S (PC type S) and PFM-NLK plasticizer (PFM). The surface properties of nanodispersed silica were characterized using an indicator method that allows for the identification and quantitative determination of Brønsted and Lewis acid-base sites based on the adsorption of indicators with specified pKa values. The effect of the modifier on the kinetics of cement stone structure formation was assessed using non-isothermal calorimetry by continuously monitoring the temperature of the cement paste under adiabatic conditions. Four series of samples were prepared to study the physicomechanical properties: a control (without additives), one with the addition of 1.5% nanodispersed silica (NS), one with a complex NS + PFM additive, and one with an NS + PC type S additive. Three samples were molded from each series for testing at 1, 3, 7, 14, and 28 days of curing. Compressive strength was determined according to GOST 5802-86, after which fragments of the destroyed samples were placed in isopropyl alcohol to immediately stop hydration. The structure and phase composition of the cement stone were studied using a combination of methods: scanning electron microscopy (SEM) on a TESCAN MIRA 3 LMU device, X-ray phase analysis (XPA) on an ARL 9900 spectrometer, and differential thermal analysis (DTA).

ISSN 2075-8545 (online)

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RESULTS

1. Evolution of nanodispersed silica activity during storage

An indicator method was used to study changes in the surface acid-base properties of synthesized nano-dispersed silica depending on its storage duration. Four samples with different storage periods after synthesis were examined: sample 1 (1 day), sample 2 (1 week), sample 3 (1 month), and sample 4 (1.5 years). The resulting acid-base spectra of surface site distributions are presented in Figure 1.

Note on the figure: The abscissa axis shows the pKa values of the indicators, characterizing the strength of the centers; the ordinate axis shows q pKa (mg-eq/g), the concentration of the adsorbed indicator, proportional to the number of centers of the corresponding strength.

The obtained data demonstrate complex nonlinear dynamics of changes in the surface condition. The key conclusion is the absence of a monotonic decrease in the overall chemical activity. A vivid rearrangement of the acid-base spectrum is observed, indicating the processes of relaxation and transformation of surface silanol grouPC (Si–OH) and active Lewis sites. The concentration of strong acidic sites (pKa = –4.4) increases from 7.42 mg-eq/g for sample 1 to 260–265 mg-eq/g for samples 2 and 3, and by 18 months decreases to 185 mg-eq/g, remaining an order of magnitude higher than the initial value. The concentration of strong basic sites (pKa = 8.8) remains high in all samples (515–628 mg-eq/g). Thus, the natural aging process does not lead to an irreversible loss of reactivity of the material.

To confirm these findings, a study of the effect of NS on the hydration kinetics of cement paste was conducted using non-isothermal calorimetry (Fig. 2, 3). All studied compositions were added with 1.5% NS by weight of cement.

Sample 4, stored for 1.5 years, demonstrates not only the retention but also an intensification of heat generation: the highest absolute temperature is reached (approximately 25.5 °C), and the main exothermic peak zone is the most pronounced and long-lasting. This is consistent with the results of the indicator method, which revealed a high concentration of both strong basic and strong acidic sites in sample 4. The bifunctional nature of the surface ensures a synergistic effect: the basic centers sorb calcium ions, initiating nucleation, while the Lewis acidic sites participate in the coordination of hydroxyl grouPC and water molecules, activating them.

2. Effect of modifiers on physical and mechanical properties

After confirming the hypothesis that the activity of nanodispersed silica does not decrease over time, ce-

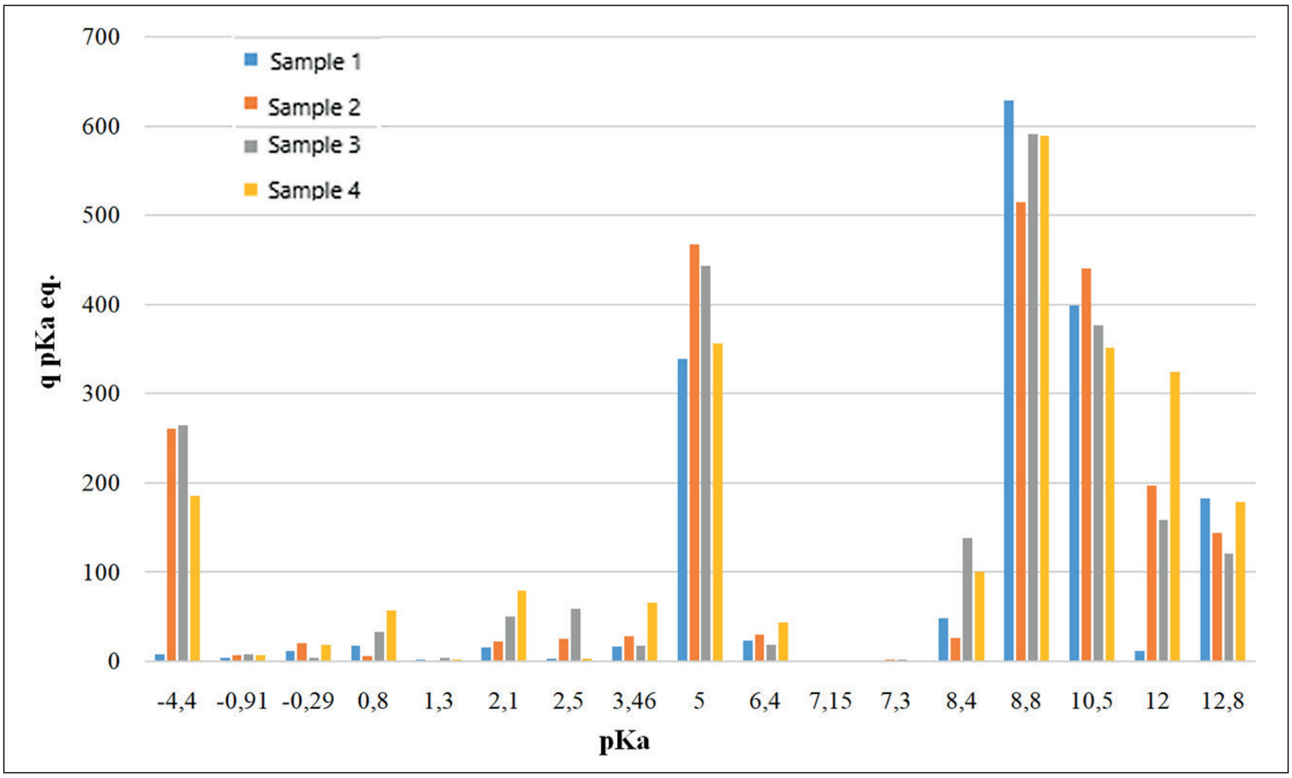

Fig. 1. Distribution of acid-base sites on the surface of nanodispersed silica depending on storage duration

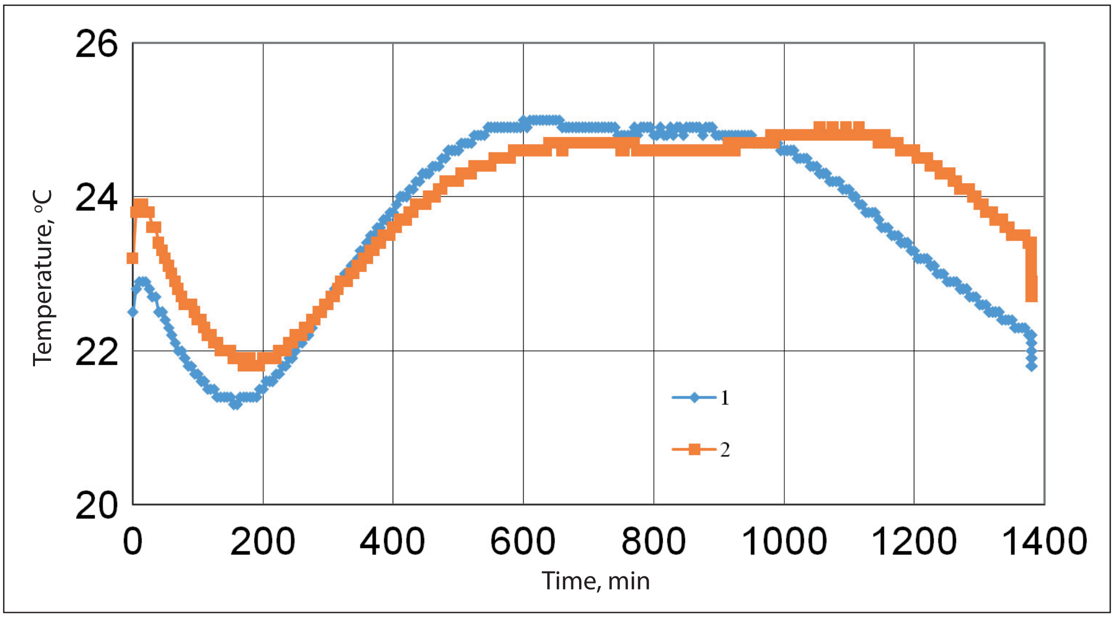

Fig. 2. Thermokinetic curves of heat release of cement paste with the addition of nanodispersed silica (samples 1 and 2)

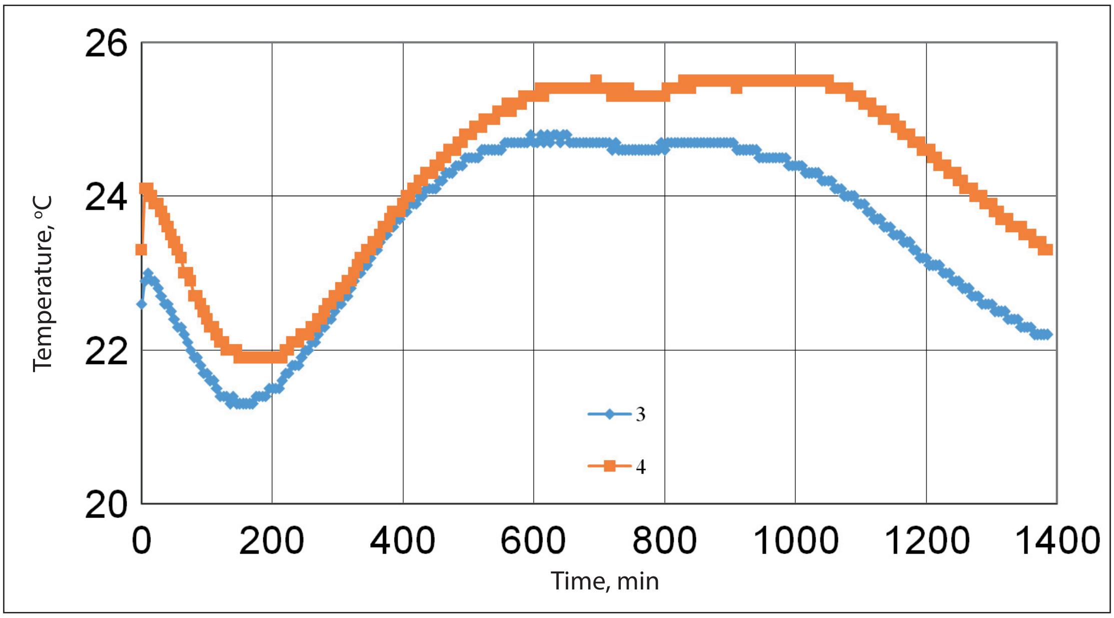

Fig. 3. Thermokinetic curves of heat release of cement paste with the addition of nanodispersed silica (samples 3 and 4)

ment stone samples with various additives were produced. Compressive strength values are presented in Table 1.

An analysis of the results shows that, in the early stages (1 day), a slight decrease in strength is observed for the sample with pure nanosilica added relative to the control, which may be due to a slowdown in the initial stages of hydration due to the adsorption of water molecules on the highly developed surface of the nanoparticles. However, by the 7th day, the samples with nanosilica excel the control composition. By the 28th day, all modified compositions show an increase in strength: 14.5% for the composition with individual nanosilica, 16.7% for the nanosilica + PFM composition, and 18% for the nanosilica + PC type S composition, compared to the control.

3. Microstructure and Phase Composition

To verify the conclusions regarding the nature of structure formation, a scanning electron microscopy analysis of the cement stone microstructure was performed (Fig. 4).

ISSN 2075-8545 (online)

Table 1. Results of compressive strength of samples, MPa

Day	Control	NS	NS + PFM	HS + PC type S
1	9.56	9.39	10.06	11.85
3	37.69	33.92	27.12	34.37
7	41.72	45.24	40.87	42.59
14	40.38	49.77	51.15	51.64
28	53.52	61.28	62.45	63.24

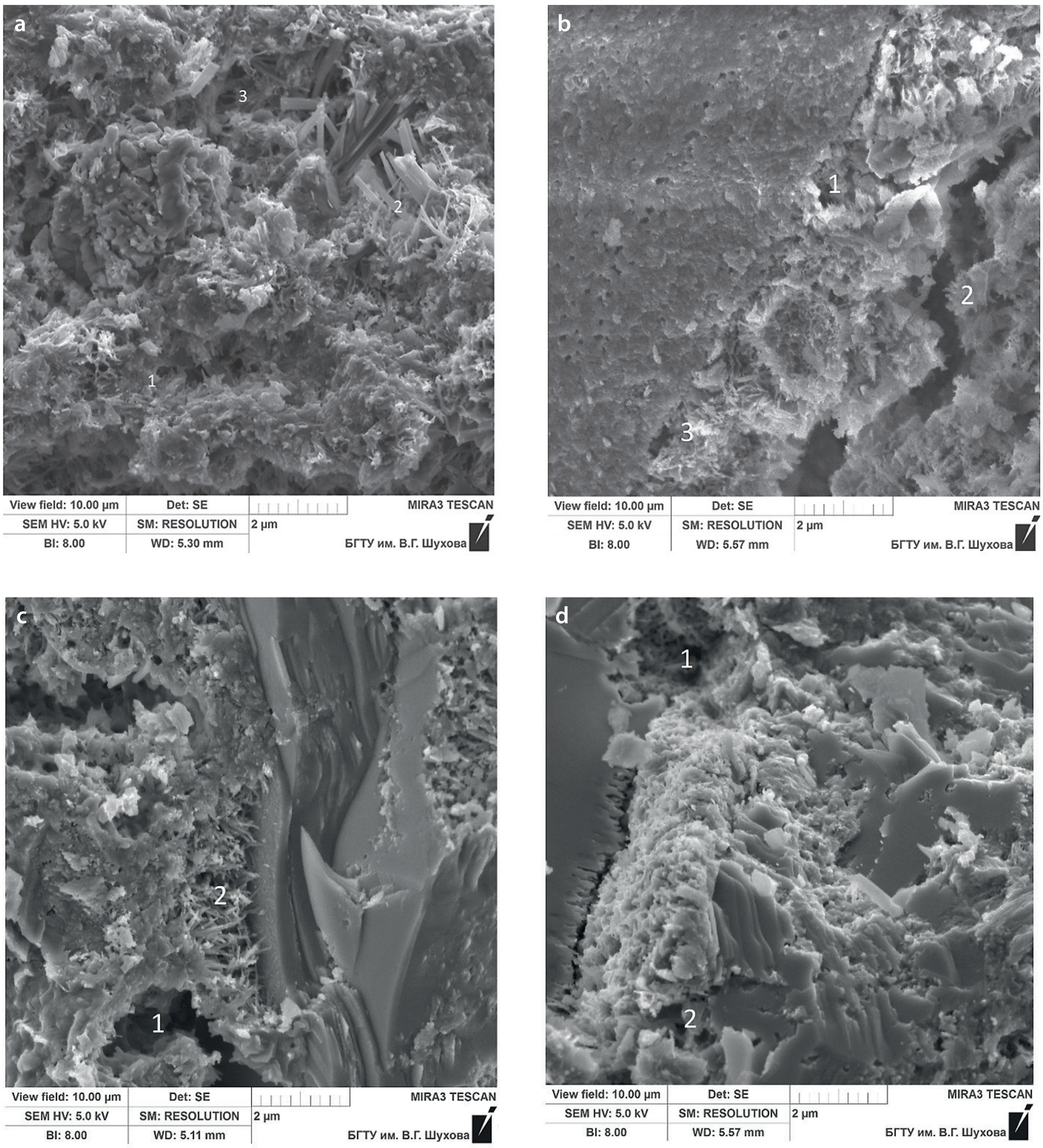

Fig. 4. Microstructure of the cement stone, modified with nano-dispersed silica and its compositions with surfactants: a – control sample; b – sample with NS; c – sample with NS + PC type S; d – sample with HS + PFM

ISSN 2075-8545 (online)

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The control sample is characterized by the formation of a developed spatial framework of needle-shaped ettringite crystals and large lamellar portlandite formations. The introduction of NC leads to a transformation of the morphology, with the crystalline products being represented by smaller mixed phases, intensive pore filling, and the formation of a denser gel-like matrix. The use of complex additives with surfactants enhances the positive effect in the system with type S PC: the gel phase is characterized by increased dispersion, and the PFM additive promotes active overgrowth of porous zones.

To confirm the phase composition, a comprehensive analysis was performed using X-ray diffraction (Fig. 5) and differential thermal analysis (Fig. 6).

The NS sample exhibits an increased proportion of the amorphous component (diffuse halo) compared to the control, which is due to the pozzolanic reaction and the additional formation of low-basic C-S-H phases. A tendency toward a decrease in the intensity of portlandite reflections is observed in the modified samples, indicating its binding through interaction with active silica.

The sample with NS exhibits the highest weight loss (17%) in the temperature range of 445–454 °C, corresponding to the decomposition of portlandite, which testifies to a higher content of hydrated phases. The introduction of surfactants leads to a reduction in mass loss to 10% (PC type S) and 9.1% (PFM), indicating the formation of a denser and more thermally stable matrix. Similar results were obtained in study [22], where it was shown that colloidal nanosilica promotes the formation of high-density C–S–H phases even in systems with recycled aggregate. The lowest mass loss of the sample with PFM surfactant indicates the most effective modification of the cement stone structure.

The lowest mass loss for the sample with PFM surfactant indicates the most effective modification of the cement stone structure.

DISCUSSION

The obtained data show that the surface of sol-gel synthesized nanodispersed silica (NS) is characterized by a polyfunctional acid-base spectrum. Long-term storage does not lead to monotonic degradation of activity. On the contrary, a rearrangement of the acid-base spectrum occurs with the formation of bifunctional centers. This is confirmed by calorimetry data: NS stored for 1.5 years not only retains but also intensifies heat release.

The bifunctional surface provides a synergistic effect: basic centers adsorb calcium ions, initiating nucleation;

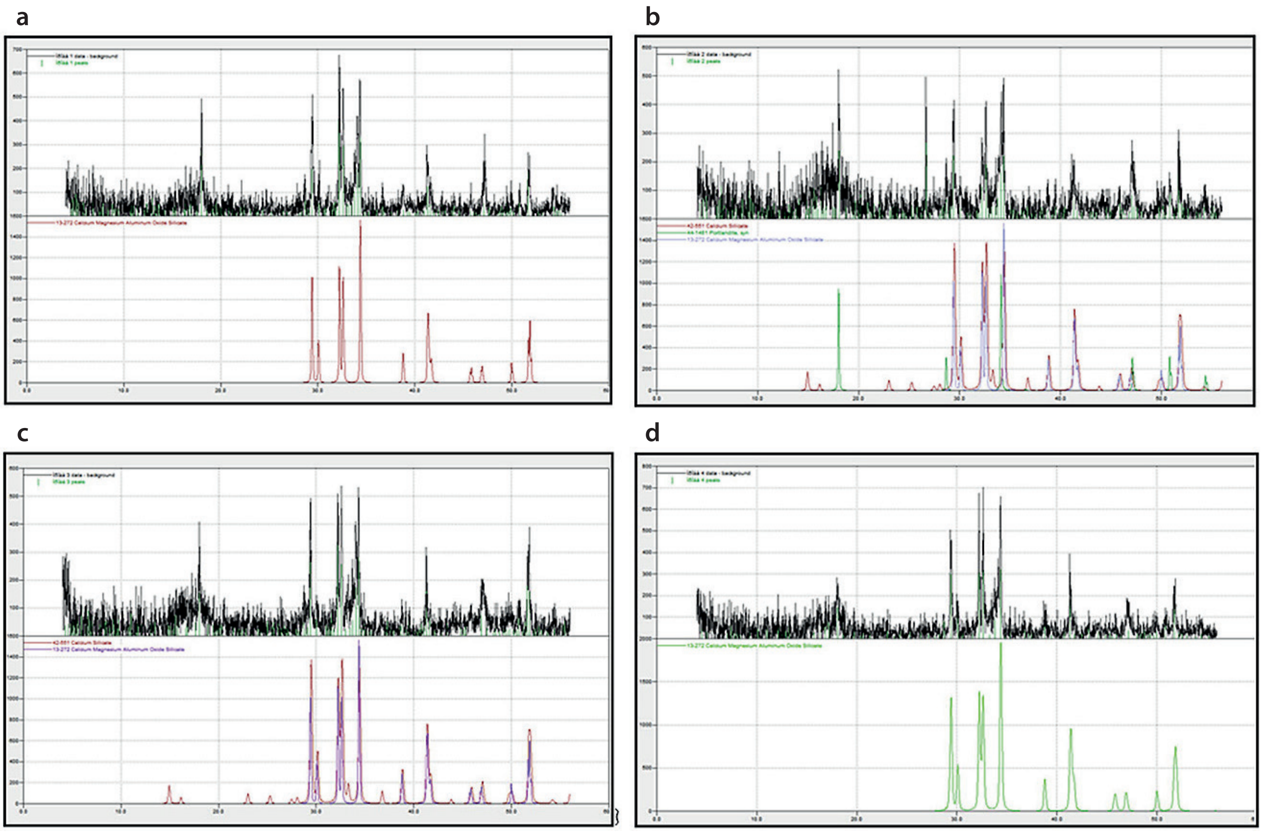

Fig. 5. Diffraction patterns of cement stone of control and modified compositions: a – control; b – NS; c – NS + PC type S; d – NS + PFM

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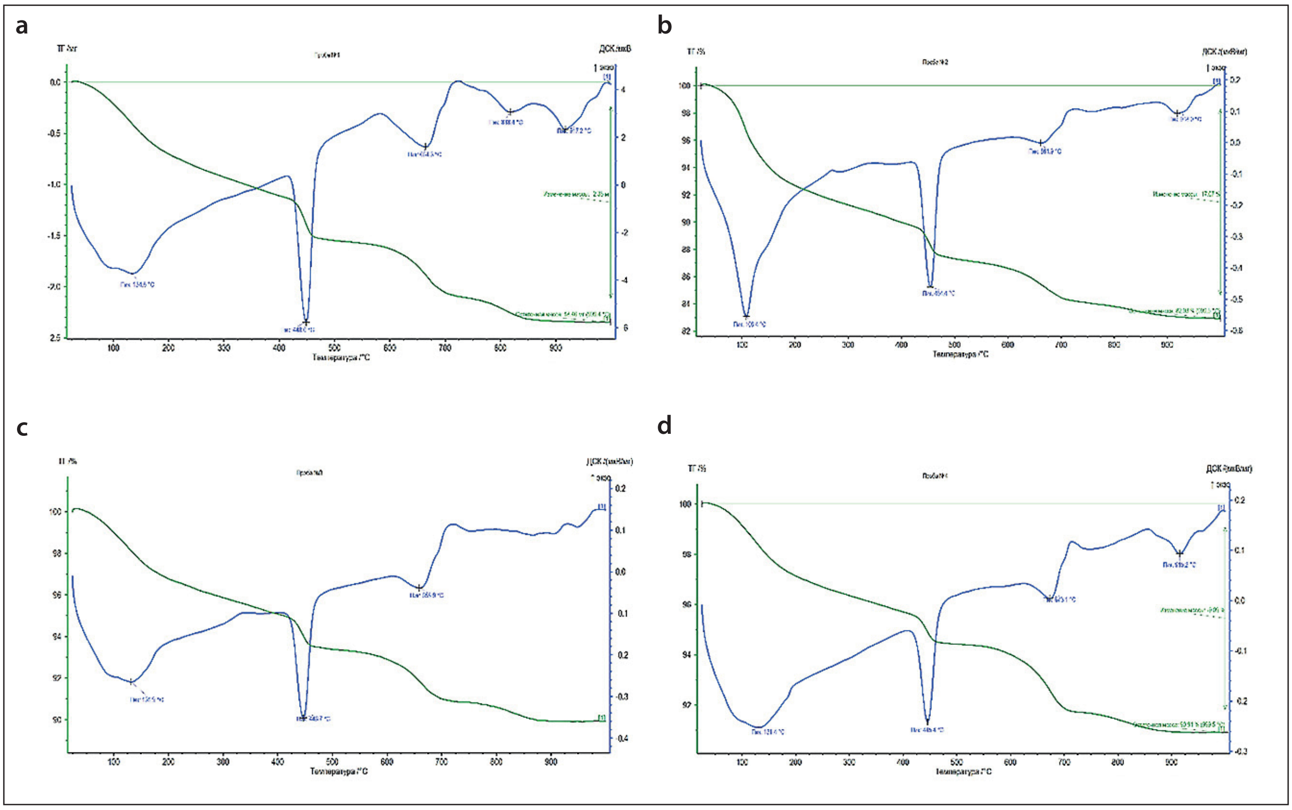

Fig. 6. Thermal curves of cement stone with control and modified compositions: a – control; b – NS; c – NS + PC type S; d – NS + PFM

Lewis acid centers participate in the coordination of hydroxyl groups and water molecules, activating them.

Physical and mechanical tests have shown that by 28 days, all modified compositions exceed the control. The highest result (18% increase) was obtained for the NS + PC type S composition. The slight decrease in strength at early age (1 day) for the composition with individual NS is explained by water adsorption on the highly developed surface of nanoparticles.

SEM, XRD and DTA methods confirmed the binding of portlandite due to the pozzolanic reaction, an increase in the proportion of low-basic C–S–H, and the formation of a denser and more homogeneous microstructure. The lowest mass loss upon heating (9.1% for the NS + PFM composition) indicates the highest thermal stability of this composition.

Thus, the developed complex NS + surfactant compositions are recommended as repair compounds of class R3 (B35) and grouting mixtures for responsible structures.

CONCLUSION

1. Using an indicator method, it was established that the surface of the synthesized nanosilica is characterized by a multifunctional acid-base spectrum. A key result is the proof of an absence of degradation of its chemical activity during long-term storage (up to 1.5 years). The restructuring of surface centers observed is characterized by a relaxation transformation, resulting in the formation of a bifunctional surface able to maintain high reactivity, which is confirmed by non-isothermal calorimetry data demonstrating intensified heat formation for samples with long storage periods.

2. Experiments have proven that the introduction of nanodispersed silica into the cement system provides a significant increase in strength properties: by 28 days of solidification, compressive strength increase for the composition with individual NS was as much as 14.5%, while for complex additives with PC type S and PFM surfactants, it was 18% and 16.7%, respectively. The strengthening mechanism is due to the pozzolanic binding of portlandite to the active centers of nanoparticles.

3. SEM, X-ray diffraction, and differential thermal analysis (DTA) confirmed that NS nanoparticles promote the formation of more dispersed and dense low-basic calcium hydrosilicates, leading to a more compact microstructure and increased homogeneity. The combined introduction of nanosilica and surfactants provides a synergistic effect: surfactants promote uniform distribution of the nanoparticles and additional plasticization of the mixture, which intensifies the pozzolanic reaction and

the formation of a thermally stable, homogeneous microstructure (minimum mass loss upon heating is 9.09% for the composition containing PFM surfactant).

4. The compositions obtained are recommended for the creation of highly effective class R3 (B35) repair mortars and grouting mixtures for critical structures, including pluming manifold repair, equipment grouting, and anchor bolt installation. The practical significance of the work is also confirmed by the proven stability of the properties of nanodispersed silica during long-term storage, which is essential for the industrial production and logistics of repair materials.

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ADDITIONAL INFORMATION

The authors declare that generative artificial intelligence technologies and technologies based on artificial intelligence were not used in the preparation of the article.

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Alexandra I. Bukovtsova – research concept, methodology development, writing the original draft, final conclusions.

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Marina V. Antonenko – participation in data analysis, interpretation of SEM and XRD results, text revision.

Valeria V. Strokova – scientific supervision, general project management, final editing.

The authors declare no conflicts of interests.

The article was submitted 30.04.2026; approved after reviewing 08.06.2026; accepted for publication 12.06.2026.