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The object of this study is the mechanism for adjusting a strength gain by modified systems based on white cements with different C_3A content.

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Since white cements have an increased content of the C_3A mineral, they are characterized by a drop in strength of up to 10 % in the late stages of hardening. To stabilize the properties of such cements, it is advisable to use modifying additives.

It has been established that when a plasticizing additive with a high C_3A content is introduced into cement in a small amount (0.5...1%), as a result of chemical interaction with the mineral C_3A , its residue in the liquid phase is not enough to disperse silicate phases. This reduces viscosity of the system. That subsequently leads to a decline in the strength of cement stone, up to 15%. At the same time, the addition of a plasticizing additive to a system with a low content of C_3A turns out to be more effective even at a lower dosage.

Modification of cement systems with nano-CaCO₃ additives helps stabilize the phase composition of new formations and guarantees the durability of the resulting cement stone. Nano-CaCO₃ changes the composition of new formations toward more thermodynamically stable compounds. The introduction of a nano-CaCO₃ additive leads to a significant increase in the rate of hydration and creates conditions for the formation of carbonate ettringite. The latter contributes to the directed synthesis of low-base fibrous hydrosilicate phases, including tobermorite, and prevents the conversion of hydroaluminate phases, which eliminates the decline in strength in white cements with an increased content of C_3A .

Applying modified systems will make it possible to stabilize the strength characteristics of not only white but also colored cements, the introduction of pigments to which leads to a decrease in their strength. This approach will make it possible to effectively use such systems as a basis for decorative concrete and mortars

Keywords: nanomodified systems, white cements, C₃A mineral, nano-CaCO₃ additives, structure formation processes

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1. Introduction

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Various design solutions for creating interiors and exteriors with an attractive colorful appearance are gaining more and more popularity in modern construction. An improved appearance of building structures can be obtained by using white Portland cement as a base for concrete or mortar instead of ordinary Portland cement. White Portland cement makes it possible to expand the scope of application of cement systems for finishing building structures. However, due to the fact that white cements usually have an increased content of the C₃A mineral, such cements are characterized by reduced frost resistance and a possible decrease in strength in the late stages of hardening. Usually, carbonate additives are used to solve such problems, but the use of nanocarbonate additives is more effective [1, 2]. The high specific surface of the latter will make it possible to reduce the volume of the pore space and compact the system through the appearance of additional nucleation zones and the development of calcium hydrosilicates, that is, the manifestation of the nucleation effect [3].

Therefore, it is a relevant task to carry out studies on mechanisms that regulate the strength gain by nanomodified systems based on white cements with different content of the mineral C_3A . UDC 691.5

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DEVISING APPROACHES TO ADJUSTING A STRENGTH GAIN BY MODIFIED WHITE CEMENTS WITH DIFFERENT C₃A CONTENT

Kateryna Pushkarova Doctor of Technical Sciences, Professor, Head of Department*

Liliia Kushnierova

Corresponding author PhD, Associate Professor* E-mail: kushnierova.lo@knuba.edu.ua **Larysa Tereshchenko** PhD Student* *Department of Building Materials Kyiv National University of Construction and Architecture

Povitrianykh Syl ave., 31, Kyiv, Ukraine, 03037

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2. The aim and objectives of the study

It is well known that the use of polycarboxylate plasticizing additives in cement systems make it possible to reduce water consumption of the mixture and increase the strength of the artificial stone at all stages of hardening. The effectiveness of superplasticizers depends on the fineness of cement grinding and the content of individual minerals in it. Work [4] shows that the most reactive mineral in cement is C_3A , which exhibits the strongest interaction with plasticizing additives, and its content affects the rheological properties of cement slurry. Studies [5] confirmed that with an increase in the content of the mineral C_3A in cement, the need for plasticizing additives also increases. But in these works, the influence of the mineral C_3A on the properties of cement systems based on ordinary Portland cement was investigated. The specificity of the effect of C₃A content on the effectiveness of plasticizing additives in white cements have remained unexplained and require additional research.

The presence on the construction market of white cements with different contents of C_3A determines the expediency of conducting research on the influence of the amount of this mineral on the strength characteristics of cement compositions, especially considering the wide use of various plasticizing additives. A number of scientific studies reported stabilizing the properties of cement systems over time by introducing calcium carbonates into them. The research results [6] show that the addition of $CaCO_3$ to cement systems increases the density of the mixture and artificial stone. Many studies have confirmed that when calcium carbonates are added to 10 % of the cement mass, changes in the mineralogical composition of new formations occur. In [7], it was established that calcium carbonate additives affect the amount of free calcium hydroxide, as well as the balance between AFm and Aft phases.

During the reaction of calcium carbonate with C_3A , hydrocarboaluminates are formed. With an increased level of the aluminate phase in the cement, the effect of calcium carbonate on the properties of the matrix increases, including an increase in early strength. In addition, CaCO₃ contributes to the formation of monocarbonate ($C_3A \cdot CaCO_3 \cdot 12H_2O$) and stable hydrate (C_3AH_6), which increases the strength of the system. Acceleration of reactions with the introduction of calcium carbonate and crystallization of monocarbonate instead of monosulfate was also found in study [8]. This contributes to an increase in the amount of ettringite and, accordingly, to an increase in the total volume of the hydrated phase and a decrease in porosity.

Work [9] reports the results of large-scale studies on the influence of both finely dispersed lime powder and ordinary lime filler on the operational characteristics of concrete. It has been proven that it is advisable to use calcium carbonates to improve the operational characteristics of concrete structures. Although, as a result of these studies, it was established that the density and strength of the obtained artificial stone was increased due to the change in the mineralogical composition of the new formations, but these studies refer to several types of cements, among which there were no white Portland cements.

The efficiency of introducing calcium carbonate into cement systems can be increased by reducing the size of its particles to the nano-level, which is confirmed by paper [10]. In [11], it was established that the fineness of grinding limestone affects its effectiveness in the hydration process and contributes to shortening the hardening time.

At the same time, the hypothesis put forward in this study regarding the reduction of the negative impact of C_3A due to the introduction of nanocarbonate additives for the purpose of stabilizing strength may work in the future for pigmented systems. For example, the effect of nanomaterials on pigments was investigated in [12], as a result of which it was established that calcium nanocarbonate works as a filler in the structure of red and black pigments.

All this gives reason to assert that it is appropriate to study the combined work of nanocarbonate additives with white cements with different C_3A content in the presence of plasticizing additives. This could serve as a basis for obtaining finishing materials and, accordingly, requires additional research.

3. The aim and objectives of the study

The purpose of our research is to determine possible mechanisms for regulating the strength of nanomodified systems based on white cements with different C_3A content. This will make it possible to effectively use modifying addi-

tives to stabilize the properties of such cement systems. It will also make it possible to determine the appropriate areas of using nanomodified white cements with increased C_3A content as a basis for obtaining colored cements with stable physical and mechanical properties.

To achieve the goal, the following tasks were set:

- to investigate the influence of the content of C₃A on the strength characteristics of cement compositions in the presence of plasticizing additives;

– to establish patterns in the processes of structure formation and the formation of the phase composition of cement systems based on white cement with an increased content of C_3A , modified with plasticizer and nanocarbonate additives.

4. The study materials and methods

The object of our research is the mechanism for regulating the acquisition of strength by modified systems based on white cements with different C_3A content. The scientific hypothesis assumes the possibility of stabilizing new formations and physical-mechanical characteristics of artificial stone due to the characteristics of interaction between a complex modifying additive consisting of a plasticizer and nanocarbonate additive with the mineral C_3A .

White Portland cements CEM I 52.5 produced by Adana and Cimsa (Turkey) were used in the research, characterized by different contents of C_3A . These white Portland cements have a stable and homogeneous mineralogical composition given in Table 1.

Table 1

Mineralogical composition of white Portland cements with different C₃A content

Producer	Content of essential minerals						
	C ₃ S, %	C ₂ S, %	C ₃ A, %	C ₄ AF, %			
Cimsa (Turkey)	55.8	25.0	11.5	0.6			
Adana (Turkey)	73.0	7.0	3.0	1.0			

For the modification of cement systems based on white Portland cement, a polycarboxylate superplasticizer by the German producer BASF Constraction Polymers (Trostberg, Germany), Melflux 1641 F, was used. In addition, we used a nanocarbonate additive in the form of an "Enrich C 50" dispersion with a dry matter content of 50 %, an average density of 1.45 g/cm^3 and with a pH level of 7–9, produced by Nordcalk (Norway). It is believed that the plasticizer and nanocarbonate additive are evenly distributed in the system and do not form local areas of concentration.

The mineral C_3A was synthesized to reveal the mechanism of interaction of white cements with nanocarbonate and plasticizing additives. The synthesis was carried out by step-by-step firing of pressed samples from the reaction mixture of calcium and aluminum oxides at a temperature of 800 °C for one hour, 1000 °C for two hours, and 1350 °C for 19 hours. The chemical composition of the initial components for the synthesis of C_3A was as follows: CaO – 62.2 %; $Al_2O_3 - 37.8$ %.

X-ray patterns of the synthesized mineral C_3A and nanocarbonate additive are shown in Fig. 1.

The identification of the diffraction patterns of the starting materials fully corresponds to the diffraction patterns of the mineral C₃A (d=0.408; 0.278; 0.270; 0.243; 0.22; 0.19; 0.155; 0.135 nm - Fig. 1, a) and CaCO₃ calcite (d=0.386; 0.303; 0.249; 0.228; 0.209; 0.191; 0.187; 0.161; 0.152 nm - Fig. 1, b).

The research was carried out on cement systems with a water content corresponding to the consistency of slurry of normal thickness. To prepare such cement systems, dry components were first mixed to which water was added.

Physical-mechanical characteristics of cement systems were determined on $2 \times 2 \times 2$ cm samples, which hardened under normal conditions for 3, 7, and 28 days. The research was conducted under laboratory conditions, where temperature, humidity, and other environmental parameters had little effect on results.

X-ray phase analysis and scanning electron microscopy were used to identify new formations in hydrated binding systems.



Fig. 1. X-ray patterns: $a - mineral C_3A$; b - calcium nanocarbonate

Features of hydration of the mineral C_3A in the presence of polycarboxylate plasticizers (1 %) and nanocarbonate additives (3.5 %) were studied by investigating the processes in a dispersed medium at the early stages of hardening. The research was carried out on a glass slide under an ordinary optical microscope (magnification 400 times) every 10 minutes during the first hour and every 30 minutes during the 3 hours from the start of mixing the mixture. Changes in the viscosity of the suspension during hydration were not taken into account. Also, the phase composition of new formations was determined by analyzing the hardening products of slurry of normal thickness after 7 days using conventional physicochemical methods of analysis, including X-ray diffraction and scanning electron microscopy.

Compositions based on C_3A were prepared in the form of suspensions. The study of changes in the composition of such compositions was carried out using a MIN-4 optical microscope and was limited to the early stages of hydration, namely after 10 minutes and 3 hours. The results are considered fully sufficient for evaluating changes in composition, without additional methods of analysis. The water-solid ratio when obtaining the suspension was 1.5 (taking into account the short hardening time of the mineral C_3A).

5. Results of investigating the acquisition of strength by modified systems based on white cements with different C₃A content

5. 1. Influence of the content of C_3A on the strength characteristics of cement compositions with plasticizing additives

At the first stage of the research, a comparison of changes in the strength of cement samples based on white cement of different manufacturers, obtained in the conventional way without the introduction of additives, was carried out (Fig. 2).

Analysis of our results makes it possible to argue about a stable acquisition of strength over time; and cement with a low content of C_3A has 7...8 % higher indicators than cement with a high content of C_3A .

At the same time, the construction market is dominated by cheaper cements with a high content of C_3A since obtaining white cement with a reduced content of C_3A requires additional technological methods, which significantly increases the costs of its production. Taking into account the peculiarity of construction market, including in Ukraine, cements with both the minimum amount of C_3A and the maximum C_3A amount were selected for research. The results of physical and mechanical tests of such samples are given in Table 2 and Fig. 3.

When a plasticizing additive is introduced, white cements behave differently during their hardening over time, depending on the content of C_3A in them.

In cement systems based on white cement with a C_3A content of 11.5 %, with the introduction of a plasticizing additive in the amount of 0.5 % and 1 %, we observe a decrease in strength to 15 % at the age of 28 days.

With the introduction of a larger amount of plasticizing additive (1.5 % of the weight of cement), the decrease in strength is almost absent.



Table 2

Results of physical and mechanical tests of the studied compositions based on white Portland cements with different C_3A content

		Compressive strength, MPa, age, days						
Composition of the binder	C ₃ A 11.5 %			C ₃ A 3 %				
		7	28	3	7	28		
WPC	64	77	82	67	73	84		
WPC+SP 0.5%	78	99	70	78	84	89		
WPC+SP 1%	80	104	80	98	102	118		
WPC+SP 1.5 %	87	110	116	89	110	126		
WPC+SP 1%+nano 2.5%	86	96	112	96	106	120		
WPC+SP 1%+nano 3.5%	94	108	116	98	114	123		
WPC+SP 1%+nano 4.5%	94	110	124	100	116	128		





Fig. 3. Change in the strength of cement samples based on white cement: a - with the C₃A content of 11.5 %; b - with the C₃A content of 3 %

Fig. 2. Change in strength of cement samples obtained using white

5. 2. Features in the structure formation of plasticized cement systems with an increased content of C_3A with a nanocarbonate additive

The introduction of a nanocarbonate additive is expedient for the modification of white cements because according to the manufacturer and previously conducted studies [2, 13–16], this additive contributes to the stabilization of the phase composition of new formations and guarantees the durability of the resulting cement stone. At the same time, the phase composition of new formations changes toward more thermodynamically stable compounds.

In order to substantiate the effect of C_3A on the strength characteristics of cement compositions, studies were conducted using the synthesized mineral C_3A ; the features of structure formation processes in the presence of polycarboxylate and nanocarbonate additives were investigated (Fig. 4).

Immediately after mixing the composition based on the mineral C_3A , plasticizer, and nanocarbonate additive, the mixture looks quite homogeneous. After 10 minutes of hydration, the process of structure formation begins, and even at a magnification of 400 times, the formation of aggregated complexes that connect into certain chain structures is visible.



Fig. 4. Photographs of dispersed systems in the form of a suspension after the start of hydration of the investigated compositions mineral C₃A + 1 % plasticizer+water+3.5 % nanocarbonate additive (400-fold increase): a - immediately after mixing; b - after 10 minutes;

c-30 minutes; d-after 1; e-2; f-3 hours

After 2 hours from the start of mixing, the number of centers of crystallization of new formations increases when

the nanoadditive is introduced. The same trend persists 3 hours after the start of hydration.

According to the data from electron raster microscopy (Fig. 5), after 7 days of hardening, the phase composition of the hydration products of the mineral C_3A in the presence of nanocarbonate is mainly represented by an amorphized globular mass consisting mainly of C_3AH_6 , even when magnified up to 2500...5000 times.



Fig. 5. Microstructure of cement stone obtained after 7 days of hardening of compositions based on the mineral C₃A in the presence of 1 % plasticizer and 3.5 % nanocarbonate additive: a – magnification x1000; b – magnification x2500; c – magnification x5000; d – magnification x10000

Some new formations can be identified only when magnified up to 10,000 times as hexagonal plates of calcium hydroaluminates and hydrocarboaluminates.

The X-ray phase analysis data after 7 days of hardening of the C_3A hydration product in the presence of a plasticizer and a nanocarbonate additive are shown in Fig. 6.

Our results confirm the formation of C_3AH_6 (*d*=0.445; 0.337; 0.315; 0.182; 0.175 nm). The formation of carbonate ettringite C_3A ·3CaCO₃·32 H_2O (0.38; 0.2773; 0.269; 0.203; 0.175 nm) is also possible [17].

X-ray phase analysis data after 28 days of hardening of plasticized white cement (which is characterized by an increased content of calcium aluminate) with the addition of a nanocarbonate additive are shown in Fig. 7.

When adding a nanocarbonate additive to plasticized white cement, it interacts with the mineral C₃A. This leads to the possibility of formation of carbonate ettringite C₃A·3CaCO₃·32H₂O (d=0.38; 0.2773; 0.269; 0.243; 0.203; 0.194; 0.175 nm), which serves as a substrate for the directed synthesis of tobermorite-like calcium hydrosilicates (d=0.307; 0.228; 0.207; 0.161 nm). In addition, there is a possibility of formation of scoutite Ca₇Si₆O₁₈CO₃2H₂O (d=0.303; 0.227; 0.207; 0.193; 0.176; 0.163; 0.148 nm).

The crystal structure of the unit cell of calcite, carbonate ettringite, and the crystallographic plane of the interlayer of the Ca–O chemical bond in calcite and carbonate ettringite are shown in Fig. 8. The structure of the unit cell of tobermorite and the crystallographic plane of the interlayer of the Ca–O chemical bond in tobermorite are displayed in Fig. 9.

Calcite (Fig. 8, a), which is part of the nanocarbonate additive, is a substrate for the crystallization of carbonate ettringite (Fig. 8, b) due to the similarity of crystal lattices in which there is an interlayer consisting of CaO atoms. The length of the Ca–O bonds is 2.35 A for calcite and 2.43–2.61 A for the carbonate ettringite.

Directional crystallization is possible along crystallographic planes passing through the CaO layer and corresponding to crystallographic planes (001) for calcite (Fig. 8, c) and (001) for carbonate ettringite (Fig. 8, d).



Fig. 6. X-ray pattern of the phase composition of new formations after 7 days of hydration of compositions based on the mineral C₃A, 1 wt.% polycarboxylate plasticizer and 3.5 wt.% nanocarbonate dispersion



Fig. 7. X-ray pattern of hydration products of a system based on white Portland cement containing 1 wt.% polycarboxylate plasticizer and 3.5 wt.% nanocarbonate dispersion after 28 days of hardening



Fig. 8. Crystal structure: a – elementary cell of calcite; b – elementary cell of carbonate ettringite; c – crystallographic plane of the Ca–O chemical bond interlayer in calcite; d – crystallographic plane of the Ca–O chemical bond interlayer in carbonate ettringite



Fig. 9. Crystal structure: *a* – unit cell of tobermorite; *b* – interlayer of the Ca–O chemical bond

In turn, carbonate ettringite is a substrate for the crystallization of tobermorite-like hydrosilicates (Fig. 9, *a*), due to the presence of a layer of Ca–O bonds (length 2.4–2.52 A for tobermorite), which ensures the formation of a contact in the zone, which is located along crystallographic planes (001) (Fig. 9, *b*).

6. Discussion of results of investigating the influence of the mineral C₃A content in modified white cements on the processes of strength gain

The mineralogical composition of cement produced by Adana is characterized by an almost optimal ratio between the main minerals (Table 1). The maximum content of C_3S guarantees the strength of cement stone in the early stages of hardening (Fig. 2), and the minimum content of C C_3A contributes to the stability of properties over time. At the same time, in such cement there is a certain disproportion between C_3S and C_2S silicate minerals. A low C_2S content (7 %) indicates an insufficient clinker fund, which leads to a limited ability to increase the strength of the formed artificial stone in the late period of hardening (after 1 year or more).

The decline in strength in cement systems based on white cement with a C_3A content of 11.5 % when a plasticizing additive is introduced in a small amount (Table 2, Fig. 3) is also confirmed by research reported by Indian scientists [18]. They explain this result by the features of the chemical structure of the plasticizing additive and its interaction with C_3A and its hydration products. That is, there is surface chemisorption or chemical interaction between C_3A or a mixture of C_3A and gypsum and a plasticizing additive [19].

When a plasticizing additive is added to cement with a high C_3A content, it is absorbed on its surface and its residue is not enough to disperse silicate phases and reduce viscosity in the system. Subsequently, this leads to a decline in strength.

When adding a larger amount of plasticizing additive (in the amount of 1.5 % of the mass of cement), there is no phenomenon of strength decline since its amount increases

in the liquid phase after adsorption on the surface of the mineral C_3A .

Based on this, the introduction of a plasticizing additive to the cement system with a low content of C_3A is more effective and at a lower dosage and provides a more stable change in strength indicators over time.

During C_3A hydration in the presence of plasticizer and nanocarbonate additive, the process of adsorption of Ca^{2+} ions on active nanocarbonate centers takes place. This leads to a significant increase in the rate of hydration and an increase in the pH of the pore solution and creates conditions for the stable existence of carbonate ettringite and C_3AH_6 (Fig. 4).

Such hydration products of the mineral C_3A in the presence of a nanocarbonate additive as an amorphized globular mass consisting of C_3AH_6 , carbonate ettringite, hexagonal plates of calcium hydroaluminates and hydrocarboaluminates are formed as a result of recrystallization of the above phases due to the gradual binding of free CaO into hydrated compounds and a corresponding decrease in pH pore solution (Fig. 5).

The values of diffraction patterns after 7 days of hardening of the C_3A hydration product in the presence of a plasticizer and a nanocarbonate additive (Fig. 6) indicate the acceleration of crystallization of thermodynamically stable hydroaluminate phases. Subsequently, nanocarbonate additives will play the role of crystal chemical substrates and will contribute to the acceleration of the crystallization process of hydrated new formations similar to carbonate ettringite $C_3A\cdot3CaCO_3\cdot32$ H₂O.

The formation of carbonate ettringite $C_3A \cdot 3CaCO_3 \cdot 32H_2O$ during hydration of plasticized white cement with the addition of a nanocarbonate additive (Fig. 7) contributes to the directed synthesis of low-base fibrous hydrosilicate phases, including tobermorite and scoutite. This prevents the conversion of hydroaluminate phases, which eliminates the decline in strength in white cements with an increased content of C_3A (Fig. 3, *a*).

The possibility of such a process is also confirmed by studies [20-23] and the crystal-chemical similarity of these compounds and the possibility of their fusion, which is shown in Fig. 8, 9.

Thus, during the interaction of cement pastes with carbonate additives, two main stages can be distinguished. The first stage is associated with the accelerated formation of carbonate-aluminate hydrates, and the second stage is due to their interaction with C–S–H phases of the tobermorite type. This leads to the synthesis of thermodynamically stable hydrated phases, which is also confirmed by other studies [24, 25].

Therefore, it is the modification of plasticized white Portland cement with a high content of C_3A with nanocarbonate additives that will solve the issues related to the unstable acquisition of strength by such cement systems over time. Using the mechanism of nanomodification of plasticized cement systems will stabilize the strength characteristics of not only white but also colored cements, as it is known that the introduction of pigments leads to a decrease in the strength of cement systems. This is especially evident in systems modified with red pigment [26].

Our study relates only to determining the efficiency of modification with nanocarbonate additives of white cements of two specific manufacturers with a C_3A content in the range of 3...11.5 %, plasticized with polycarboxylate plasticizer Melflux 1641 F in the amount of 1 %.

The research makes it possible to determine the features of the strength gain processes in the selected modified cement systems and the role of the mineral C_3A content in them, but this is not enough to establish statistical regularities of these processes as a whole. To establish such laws related to the change in strength of cement systems based on modified white cements, it is necessary to conduct more experiments. Namely, cement systems based on white Portland cements of different manufacturers with different mineralogical composition require additional research.

It is appropriate to investigate the strength gain processes in such systems when they are modified with plasticizing additives of different composition and mechanism in the presence of nanocarbonate additives of different dispersion. As a result of conducting such research, it would be possible to obtain a reasonable statistical sample, which could provide grounds for generalizing the regularities of processes of strength gain in such cement systems depending on the content of C_3A in them. This approach will make it possible to design complex nano additives that will be effective for white and colored cements with a high content of the mineral C_3A , which today are most attractive to the consumer, taking into account current economic and environmental factors.

7. Conclusions

1. When introducing a plasticizing additive to cement with a high C_3A content (11.5 %) in a small amount (0.5...1 %) due to chemical interaction with the mineral C_3A , its residue in the liquid phase is not enough to disperse silicate phases and reduce viscosity in the system, which subsequently leads to a decrease in strength up to 15 %. To achieve the plasticizing effect of the cement system, it is necessary to increase the dosage of the plasticizing additive. When a plasticizing additive is introduced into the cement system with a low content of C_3A (3 %), the plasticizing effect is manifested with a smaller amount of the additive (0.5...1 %), and the strength gain over time is more stable.

2. When a nanocarbonate additive is added to plasticized white cement, it interacts with the mineral C_3A , resulting

in the possibility of the formation of $C_3A \cdot 3CaCO_3 \cdot 32 H_2O$ carbonate ettringite, which is a substrate for directional crystallization of low-base calcium hydrosilicates.

This approach makes it possible to effectively use white cements with an increased content of C_3A (11.5%) as a basis for obtaining finishing materials.

Adjusting the strength characteristics of white cements with an increased content of C_3A (11.5%) is especially important for colored cements since the introduction of dye is always accompanied by a certain decrease in strength, which reaches 5...7%. Solving this problem through nano-modification could reveal new possibilities for stabilizing the strength characteristics of not only white but also colored cements.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study, as well as the results reported in this paper.

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Data availability

All data are available, either in numerical or graphical form, in the main text of the manuscript.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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