

Discussion paper

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Modeling the Kinetics of Cement Composite Processes Modified with Calcium-Containing Additives

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ABSTRACT: Introduction. Rock wastes from dust collection systems can be used as mineral additives for making artificial conglomerates based on mineral binders. At the same time, the size of waste particles allows them to be used without additional grinding. Such materials may include fine powders of calcium-containing rocks such as limestone, wollastonite, diopside. Waste rock from dust collection systems can be used as mineral additives for manufacturing artificial conglomerates based on mineral binders. The particle size of the waste allows their utilization without additional grinding. Such materials can include finely dispersed powders of calcium-containing rocks, such as limestone, wollastonite, and diopside. **Methods and materials.** We used Portland cement CEM II/A-W 32.5F was used as a binder in the study. The selection of additives was determined through a comparative analysis of their thermodynamic properties with those analogous to clinker minerals. Standard research methods were used to study cement composites, as well as mercury porometry, X-ray phase analysis and electron microscopy. **Results.** The addition of calcium-containing additives makes it possible to strengthen the structure of cement materials. The greatest increase in strength during the initial hardening period can be achieved by adding 2% of limestone. In the late period of strength gain (in 14 days), the greatest hardening of the stone was obtained with the addition of 9% wollastonite or 7% diopside. **Discussion.** The results of cement stone diffractogram of the control composition and with the use of additives are given. With the addition of additives, there is a decrease in the intensity of calcium hydroxide reflexes and an increase in the intensity of calcium hydrosilicates reflexes. The study of the macrostructure revealed a significant difference in the structure of the stone. The porosity analysis showed that when calcium-containing additives are added into the system, the total pore volume decreases, the number of small pores increases (size 0.003–1.2 μm). **Conclusion.** The increase in strength up to 48% is due to the close chemical composition and thermodynamic characteristics of the binder and calcium-containing additives. Wollastonite, due to its fibrous structure, creates micro-reinforcement of the system, and diopside, in its turn, having the highest hardness and elastic modulus of the presented additives, leads to the greatest hardening of the stone.

KEYWORDS: cement composites, calcium-containing additives, strength characteristics, porosity.

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INTRODUCTION

Obtaining fast-hardening high-strength cement composites is the most important task in the development of materials science. In case fillers from dense and coherent rocks are used in construction composites based on mineral binders', the strength of which is several times higher than their strength, then the

matrix of the binder is responsible for the strength of the composite. At the same time, the phase composition and structure of crystalline hydrates in the hydrated binder and artificial composite do not differ significantly [1–5]. The use of fine mineral wastes from mining and mining processing facilities as additives can increase the strength of artificial composites by strengthening the matrix of the hydrated mineral binder. At the same time, the addition

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of these wastes can reduce the consumption of expensive mineral binder [6–12].

Dogmatic documents require finding rational ways to use waste from mining and mining processing facilities in construction in the Russian Federation. The statements of the Innovative Development Strategy of the Russian Federation for the period up to 2030 (approved by Order of the Government of the Russian Federation dated 08.12.2011 No. 2227-p (amended by 18.10.2018)), the Strategy for the Development of Building Materials Industry for the Period up to 2020 and the Future further until 2030 (approved by Order of the Government of the Russian Federation dated 10.05.2016 N 868-p) are aimed at expanding the use of waste in materials science. Thus, the expediency of fine man-made or natural raw materials utilization in the manufacture of artificial composites based on mineral binders is due to the task of improving their quality and to the utilization requirement of multi-tonnage waste from mining processing facilities [13]. In the Russian Federation, over 3500 thousand tons of such wastes are generated every year. At the same time, the building materials industry currently processes only 4% of the annual volume of these wastes. [14–17].

Fine rock wastes from dust collection systems can be used as mineral additives to make artificial stone materials based on mineral binders. At the same time, the size of the powders of these wastes allows them to be used without additional grinding. Such powders can include finely dispersed powders of calcium-containing rocks such as limestone, wollastonite, diopside [4, 18–20]. Fine mineral powders of rocks can participate in the process of crystalline hydrates formation and the structure of artificial stones on a cement basis. The interaction of mineral powder particles with hydrated matrix of cement occurs mainly along the plane of their contact. When selecting mineral wastes of rocks, it is necessary to pay attention to the type of their chemical bond and the proximity of their energy indicators (entropy and enthalpy of formation) with clinker minerals [8, 9, 18, 21, 22]. Russian scientists believe [8, 9, 21, 22] that calcium-containing wastes can be the most effective of those fine rock wastes that can be used to improve the properties of cement systems. At the same time there is necessary energy compatibility between finely dispersed powders of rocks (mineral additives) and hydrate innovations of cement.

Since the greatest hardening of the cement matrix can be achieved with the smallest porousness between the particles of the additive and the binder, a number of scientists indicate the need to control the dimension of their grains [8, 23, 24].

Thus, the objective of the work is to study the degree of influence of calcium-containing mineral additives on the kinetics of hardening and increasing the strength of the cement matrix, and to establish the factors affecting this process.

METHODS AND MATERIALS

Portland cement CEM II/A-SH 32,5F Iskitimcement JSC (Iskitim, Novosibirsk Region), manufactured in accordance with GOST 31108-2020, was used as a binder in the work. The main characteristics of the binder are shown in Table 1.

The additives used in the work were selected based on the comparison of their thermodynamic characteristics with similar characteristics of clinker minerals (Table 2).

The comparison of thermodynamic characteristics (enthalpy and entropy of formations) showed their close values (differences of no more than 9%).

Calcium-containing additives were used in the work that is the powders of natural stone materials, which are man-made products: diopside, wollastonite and limestone. The chemical composition of these additives is shown in Table 3.

Table 4 shows the hardness of the test additives on the Mohs scale.

Clinker hardness is quite high and is 6–7 on the Mohs scale. However, the hardness of hydrate neoplasms is much lower and, as a rule, does not exceed 3.5–4.0.

Thus, the choice of calcium-containing additives (wollastonite, diopside, limestone) adopted in the study is determined by the proximity of their thermodynamic characteristics and chemical composition with clinker minerals, and, therefore, the possibility of these additives to act as supports for crystallization of hydrated new-growths.

In addition, the choice of mineral additives is due to their hardness, and therefore, the modulus of elasticity. If the modulus of elasticity of mineral additives and hydrated cement differs, then, when external forces act on the system, the stresses will redistribute towards the component with the largest modulus of elasticity.

In this case, the additives that have a fibrous structure will micro-strengthen the cement matrix of artificial composites.

Calcium-containing additives were added to portland cement in an amount of 2 to 11% weight. The mixture was stirred dry. Standard density cement dough was prepared from the resulting modified binder and the samples were then formed. Water under the requirements of GOST 23732 was used to cure the modified binder during experimental studies.

The strength characteristics of the cement system were determined on cubes with a 20 mm rib. Five batches of samples were manufactured, which gained strength under steam curing conditions and under standard conditions. Curing mode: 4 hours – preliminary curing at a temperature of $20 \pm 2^\circ\text{C}$; 3 hours – temperature rise to 80°C , 8 hours – curing at this temperature; 3 hours – temperature decrease. Under standard conditions (temperature $20 \pm 2^\circ\text{C}$, humidity – not less than

Table 1

Portland Cement Specification

No.	Indicator	Required value	Actual value
Chemical composition			
1	Content SiO ₂ , %	not rated	22.64
2	Content Al ₂ O ₃ , %	not rated	6.24
3	Content Fe ₂ O ₃ , %	not rated	3.48
4	Content CaO, %	not rated	59.50
5	Content MgO, %	not rated	2.89
6	Content of potassium and sodium oxides in terms of Na ₂ O (Na ₂ O+0.658K ₂ O)	not rated	0.84
7	Insoluble residue content, %	not rated	0.47
8	Content SO ₃ , %	not more 3.5	2.59
9	Content Cl ⁻ , %	not more 0.10	0.02
Estimated mineralogical composition of clinker			
10	Content C ₃ S, %	not rated	67.0
11	Content C ₂ S, %	not rated	11.0
12	Total content C ₃ S и C ₂ S, %	not less 67.0	78.0
13	Content C ₃ A, %	not rated	6.4
14	Content C ₄ AF, %	not rated	12.0
15	Content MgO, %	not more 5.0	1.57
Physical characteristics			
16	Fineness, %	not rated	8.1
17	Specific surface area, m ² /kg	not rated	304
18	Initial set, min Final set, min	not early 75 not rated	192 235
19	Soundness	not more 10	0.4
20	Water-need, %	not rated	25
Strength characteristics			
21	Bending strength, MPa, age: – at 2 days – at 28 days	not rated not rated	3.8 7.5
22	Compressive strength, MPa, when hardening: – under curing – under normal conditions 2 days – under normal conditions 28 days	more 30.0 not less 10.0 from 42.5 to 62.5	31.4 17.2 43.6

Table 2

Specific thermodynamic characteristics of compounds

Compound description	Enthalpy of formation (ΔH_{298}°), kJ/g	Entropy of formation (S_{298}°), J/(g · K)
Tricalcium silicate (3CaO · SiO ₂)	–12.83	0.74
Dicalcium silicate (β -2CaO · SiO ₂)	–13.40	0.74
Tricalcium aluminate (3CaO · Al ₂ O ₃)	–13.29	0.76
Diopside (CaO · MgO · 2SiO ₂)	–14.80	0.66
Wollastonite (CaO · SiO ₂)	–14.10	0.71
Limestone (CaCO ₃)	–12.06	0.88

Table 3

Chemical composition of calcium-containing additives (according to the manufacturer)

Additives	Chemical composition, % mac.							
	CaO	SiO ₂	MgO	Al ₂ O ₃	Fe ₂ O ₃	R ₂ O	TiO ₂	Loss on ignition
Diopside	26.23	53.44	17.91	0.19	0.08	0.12	0.11	1.90
Wollastonite	34.72	53.43	0.30	3.06	2.34	–	–	6.24
Limestone	54.7	0.5	0.5	0.2	0.1	–	–	40.4

Table 4

Hardness of compounds

Compound	Mohs' hardness
CaO • MgO • 2SiO ₂ (diopside)	6.5
CaO • SiO ₂ (wollastonite)	5.0
CaCO ₃ (limestone)	3.0

95%), the samples were tested at the ages of 1, 3, 7 and 28 days, respectively.

X-ray diffraction pattern of hydrated portland cement of the control composition and with calcium-containing additives were taken on a D8 Advance powder diffractometer (Bruker AXS, Germany).

The microstructure of the cement stone was studied with the use of Hitachi TM 1000 Hitachi Science Systems Ltd scanning electron microscope. The degree of increase of the microscope is from 20 to 10 000.

The structural characteristics of the hydrated stone were examined with the use of automated mercury porosimeter (porosimeter) AutoPore IV 9520 from Micromeritics (USA).

RESULTS

The results of the calcium-containing additives impact on the strength characteristics of the modified cement stone are shown in Figures 1, 2.

DISCUSSION

The analysis of the obtained experimental data shows that the addition of even a small amount (2%) of the studied calcium-containing additives (wollastonite, diopside, limestone) leads to a significant hardening of the modified cement.

The obtained experimental dependencies on the effect of the type and amount of calcium-containing additives on the kinetics processes of cement composites show that during the initial period of strength gain the greatest strengthening of the material can be achieved by adding limestone. The addition of 2% crushed limestone

strengthens the cement stone hardened for 1–3 days up to 20%. In the late periods of strength gain (7–28 days), the greatest hardening of the stone was obtained when wollastonite and diopside were added. The optimal amount of wollastonite from the point of view of strengthening effect was 9%, diopside – 7%. Modification of Portland cement with 9% wollastonite increases the strength to 27.5%. The addition of 7% diopside results in more hardening (up to 48%). The addition of calcium-containing additives in excess of the optimum amount results in the strength decrease of the modified cement stone.

The strengthening effect when wollastonite and diopside are added is associated with the redistribution of stresses in the cement stone under the external forces, which is due to their greater hardness and a greater modulus of elasticity than the hydrated stone.

The hardness of limestone is lower than that of hydrated cement. That is, the effect of the additive is due to other reasons. The addition of limestone is revealed in the process of hydration of the binder by forming a contact zone between the additive particles and the hydrated cement. To do this, X-ray phase analysis of cement stone of a non-filler composition and composition with the addition of limestone was carried out, and their microstructure was also studied.

The diffractograms of cement stone with the most characteristic interplane distances of hydrate phases are shown in Figures 3–4.

The diffractograms of the hydrated stone of the control and limestone modified composition confirmed its impact on the process of crystalline hydrates formation. When modifying cement stone with limestone, the intensity of calcium hydroxide reflexes decreases ($d = 0.493; 0.262; 0.192; 0.179; 0.169; 0.148$ nm), and the intensity of calcium hydrosilicates reflexes increases ($d = 0.310; 0.302; 0.288; 0.277; 0.260$ nm). In addition, calcium hydrocarboaluminate reflexes were found in the diffractograms of the modified stone ($d = 0.302; 0.288; 0.277; 0.227$ nm) and calcium hydrocarbosilicate ($d = 0.349; 0.311; 0.288; 0.262; 0.228$ nm).

To identify further the strengthening factors of hydrated stone, both the macro and microstructure of cement stone, both non-additive and additive were studied during

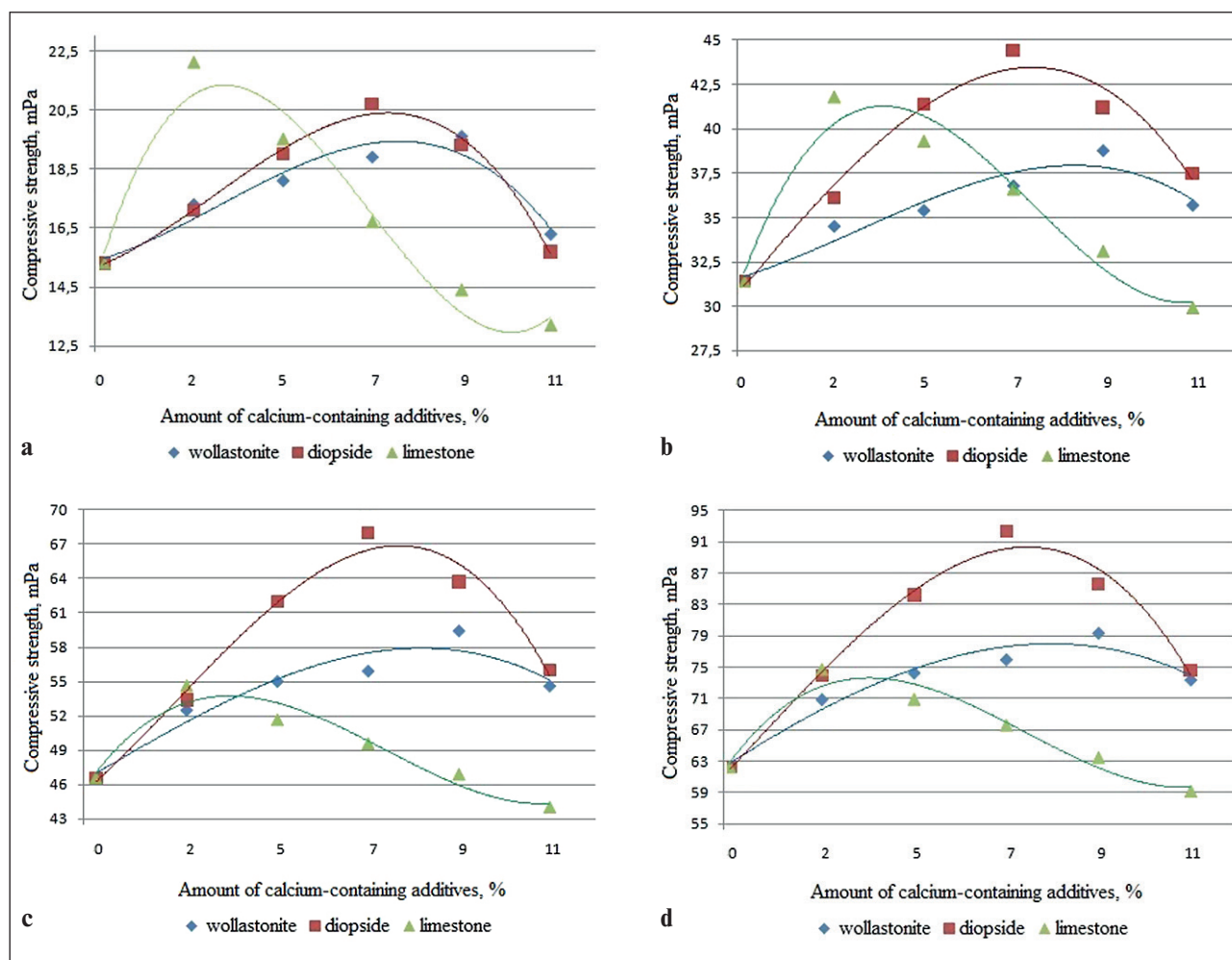


Fig. 1. Change of cement stone strength at addition of calcium-containing additives. Hardening under regular conditions within the duration of: a) 1 day; b) 3 days; c) 7 days; d) 28 days

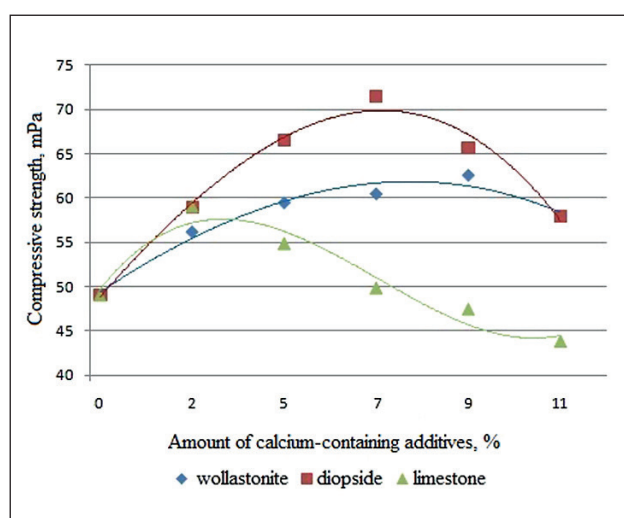


Fig. 2. Change of cement stone strength at addition of calcium-containing additives. Hardening under steam curing

the addition of dispersed additives. The macrostructure was evaluated by the nature and pore size of the formed samples. The results are shown in Tables 5 and 6.

The structure of cement stone without additives is porous, it contains a large number of large pores (up to 50 microns in size). Crystalline hydrates are mainly highly basic hydrosilicates with a pore size of 10–15 μm and large ettringite crystals.

The structure of the stone with the additive is denser, homogeneous and finely crystalline. The pore sizes of the modified stone are significantly smaller (0.15 μm or less).

The analysis of the structure porosity showed that when calcium-containing additives are added into the system, the total pore volume decreases, the pore content increases by a size of 0.003–1.2 μm . In this case, the structure of the pores themselves changes, the characteristic length of the pores increases markedly and the tortuosity of the pores significantly decreases. The main part of the pore volume (more than 95%)

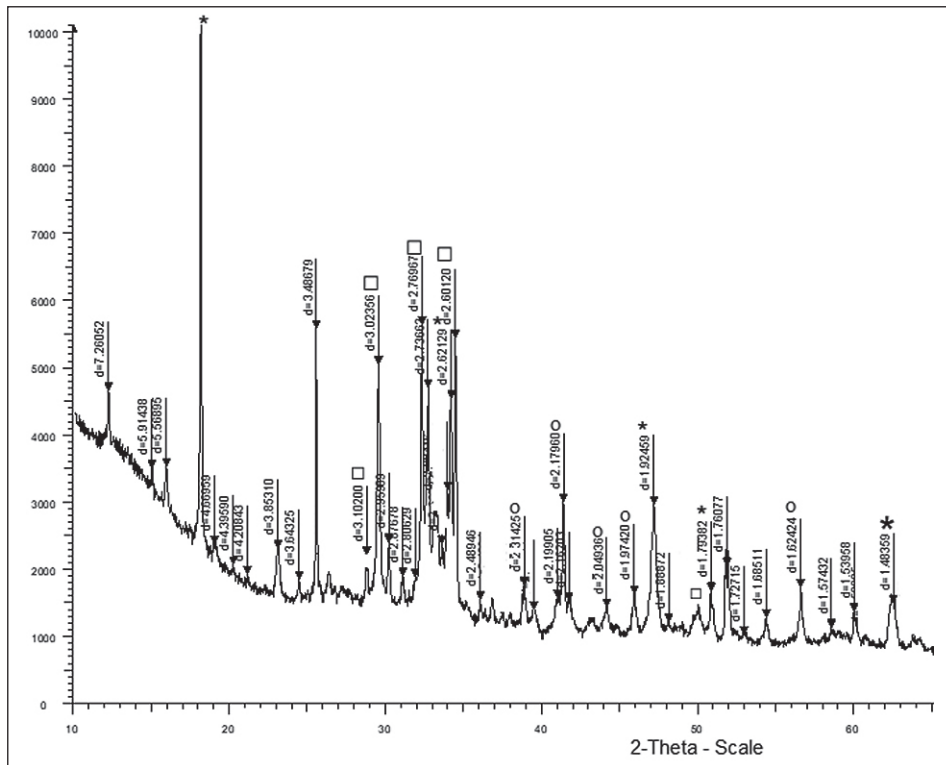


Fig. 3. Diffractogram of cement stone of plain composition:

* – $\text{Ca}(\text{OH})_2$; □ – C–S–H; ○ – C–A–H; ● – $\text{C}_3\text{A} \cdot \text{CaCO}_3 \cdot 12\text{H}_2\text{O}$

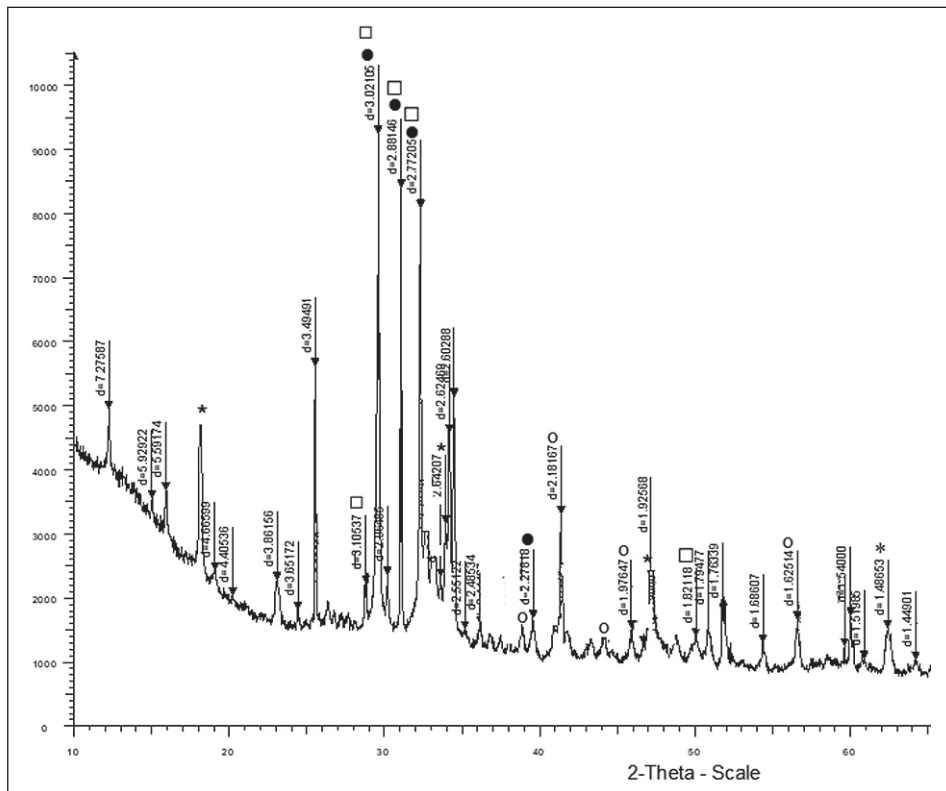


Fig. 4. Diffractogram of cement stone containing limestone:

* – $\text{Ca}(\text{OH})_2$; □ – C–S–H; ○ – C–A–H; ● – $\text{C}_3\text{A} \cdot \text{CaCO}_3 \cdot 12\text{H}_2\text{O}$

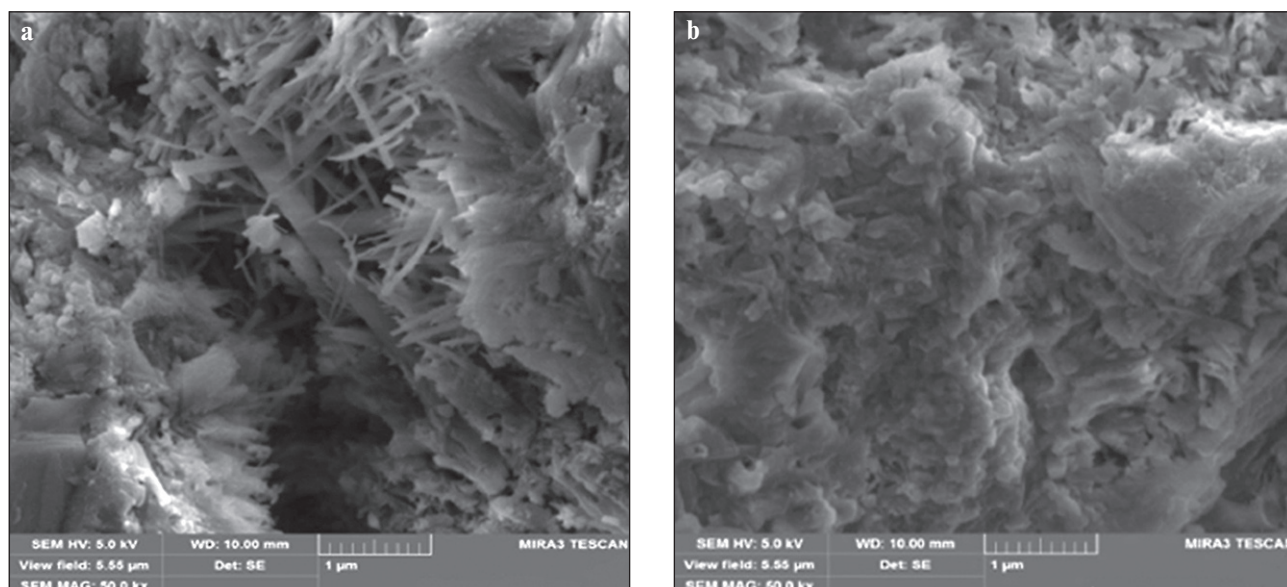


Fig. 5. The structure of cement stone hardened for 7 days with an increase of 10,000 times: a – cement stone of plain composition; b – cement stone with limestone addition

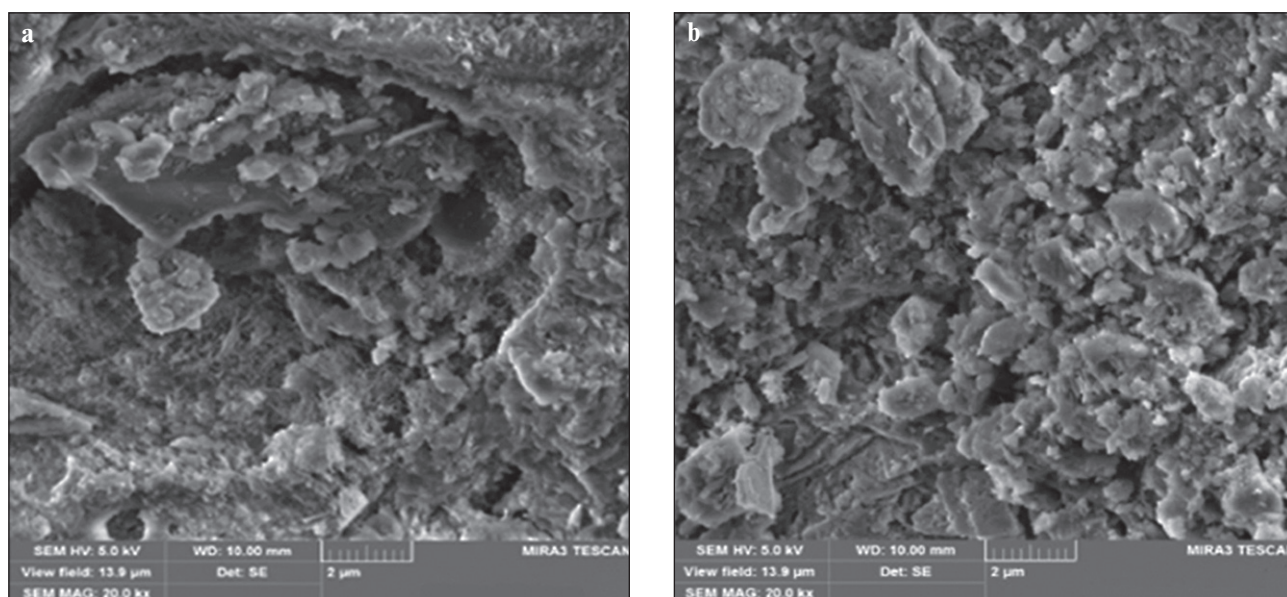


Fig. 6. The structure of the cement stone hardened for 28 days with an increase of 10,000 times: a – cement stone of plain composition; b – cement stone with limestone addition

falls on pores smaller than $0.15\ \mu\text{m}$. The water in such pores will be in a membranous state, which in turn can lead to an increase in frost resistance of modified cement composites.

Since the modified cement composite has a characteristic pore length that increases and decreases the pore tortuosity, more favorable conditions are created when part of the water is moved to the nearest air inclusions. During the freezing of the liquid, the pressure will not increase inside the concrete.

CONCLUSION

Thus, the following conclusions can be drawn:

1. The increase in stone strength by 20–48% with the addition of the studied calcium-containing additives (limestone, wollastonite, diopside) is due to the proximity of their thermodynamic characteristics and chemical composition with clinker minerals.
2. The greater strengthening effect of diopside addition (up to 48%) is due to the greater hardness and modulus

Table 5
Characteristics of porous structure of hydrated stone

Average pore diameter, μm	Characteristics of porous structure of hydrated stone depending on binder composition							
	Portland cement		Portland cement with 9% wollastonite weight		Portland cement with 7% diopside weight		Portland cement with 1% limestone weight	
	pore volume, ml/g	pore area, m^2/g	pore volume, ml/g	pore area, m^2/g	pore volume, ml/g	pore area, m^2/g	pore volume, ml/g	pore area, m^2/g
73.1–361.0	0.1275	0.003	0.0919	0.002	0.0903	0.002	0.1108	0.003
15.4–73.1	0.0146	0.003	0.0115	0.002	0.0131	0.002	0.0162	0.003
1.2–15.4	0.0164	0.033	0.016	0.029	0.0138	0.026	0.0165	0.031
0.05–1.2	0.036	0.741	0.041	0.944	0.043	0.958	0.039	0.872
0.05–0.15	0.031	4.281	0.035	4.806	0.037	4.824	0.033	4.232
0.003–0.05	0.088	22.456	0.101	24.939	0.105	25.012	0.090	21.865
Total	0.3128	27.517	0.2968	30.722	0.3026	30.824	0.3051	27.006

Table 6
Cement porosity characteristics (summarized data)

Characteristics	Binder composition			
	Portland cement	Portland cement with 1% limestone weight	Portland cement with 9% wollastonite weight	Portland cement with 7% diopside weight
Total pore volume, ml/g	0.3128	0.3051	0.2968	0.3026
Pore volume with diameter less than 0.15–1.2 μm , %	16.4	18.4	19.9	20.3
Pore volume with diameter less than 0.003–0.15 μm , %	32.9	34.6	39.9	41.0
Average pore diameter, μm	0.0571	0.0485	0.0450	0.0407
Characteristic pore length, μm	1.681	6.892	7.634	7.673
Pore tortuosity, rel. units	183.28	33.356	28.498	28.279

of diopside elasticity compared to wollastonite and limestone. The strength at the addition of which increases by 20–27%.

3. Wollastonite having a fibrous structure results in greater hardening of the stone compared to limestone by stone micro-reinforcing.

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Lilia V. Ilina – scientific guidance, concept development and development of the research methodology; processing and analysis of experimental data; systematization of experimental data; drawing up final conclusions; literature review.

Svetlana V. Samchenko – participation in the development of a scientific concept; processing and analysis of experimental data; finalization of the text.

Mikhail A. Rakov – conducting the experimental part; graphical and tabular representation of the study results; processing and analysis of experimental data using machine learning methods.

Dmitry A. Zorin – concept development and development of the research methodology.

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