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Issue Article

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STUDYING THE STRUCTURE AND PROPERTIES OF CEMENT STONE WITH A COMPLEX OF MODIFYING ADDITIVES

Abstract*: The article presents the results of studies on the influence of various additives on the water resistance of concrete, optimization was carried out, optimal values of additives were found, the microstructure of filled cement stone was also studied using X-ray phase analysis and electron microscopy. It follows from the X-ray phase study that the introduction of the superplasticizer additive reduces the content of portlandite in cement stone, which correlates with the results of thermal analysis, the results of the microstructure of cement stone modified by the introduction of polycarboxylate superplasticizer are represented by gel-like highly basic hydrosilicate phases, which is confirmed by the data of the X-ray microanalyzer.*

Key words*: cement, silica, polycarboxylate, superplasticizer, hydrophobizer, cement stone, modifying hydrophobizer, concrete, X-ray phase analysis, microstructure.*

Language*: English*

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Introduction

Waterproof concrete is used to build structures, most of which are exposed to moisture. This can be a screed in the bathroom, the facade of a building, a foundation or a hydraulic structure. In each case, different compositions are used, the main difference here is water resistance. Under adverse conditions of hardening and operation of the Republic of Karakalpakstan, the water resistance of the concrete mixture plays a key role in its further physical, mechanical and operational performance [1].

The purpose of the study is to study the structure and properties of cement stone with a complex of modifying additives.

This article presents the results of studies of the structure and properties of cement stone. Cement stone with microsilica and polycarboxylate additives.

Samples of cement stone, without aggregates, were made for the subsequent study of their structure using physicochemical methods of analysis. To conduct research on the influence of modifiers, a three-factor experimental plan was implemented, in which the following were taken as significant factors: X_1 - dosage of polycarboxylate superplasticizer, variable in quantity from 0 to 1,2%, and X_2 - dosage of micro silica, in an amount from 0 to 12%, based on the mass of cement. X_3 - modifying water repellent from 0,01-0,1.

During the experiment, the following characteristics of the cement paste were evaluated:

W/A (water/astringent) (the ratio of the mass of water to the mass of cement with micro silica) and setting time. Cement stone samples were tested for compressive strength and water absorption. Fragments were taken from these samples for physicochemical studies of the structure of hydrated phases. The results of the conducted studies are presented in table 1 [2].

Table 1. Variable factors and properties of cement paste and stone

№	X_1 Content SP, %	X_2 Content MS, %	X_3 Content MH, %	W/A	Setting time, min.		Compressive strength, R_{str}, MPa
					beg.	end	28-days
1	+1	+1	+1	0,25	50	120	38,5
2	-1	+1	+1		75	165	40,5
3	+1	-1	+1		85	190	40,0
4	-1	-1	+1		72	165	35,0
5	+1	+1	-1		120	161	34,0
6	-1	+1	-1		125	175	50,5
7	+1	-1	-1		75	205	45,0
8	-1	-1	-1		84	185	40,0
9	0	0	-1		72	190	51,5
10	0	0	+1		65	180	50,5
11	0	-1	0		55	135	49,5
12	0	+1	0		50	125	47,5
13	+1	0	0		60	130	45,5
14	-1	0	0		65	130	48,5
15	0	0	0		50	130	52,0

Parameters of the regression equation, adequacy and equal accuracy of the obtained mathematical model, at a significance level of 0.05.

Since micro silica (MS) has pozzolanic properties and interacts with secondary calcium hydroxide released during C_3S hydration, it was taken as a binder component and the water demand was estimated not by water-cement, but by water-binding ratio.

The initial water demand of a cement paste without additives does not change or decreases with the proportional introduction of modifier additives, which balances the multidirectional change in water demand. The optimal content of MS at a dosage of 8-10% by weight of cement increases the value of C/MS, which is compensated by the introduction of polycarboxylate in an amount of 0.5-0.75% by weight of cement, and with a higher content of superplasticizer (SP), water demand decreases with C/MS =0.25 to C/MS=0.23 [3].

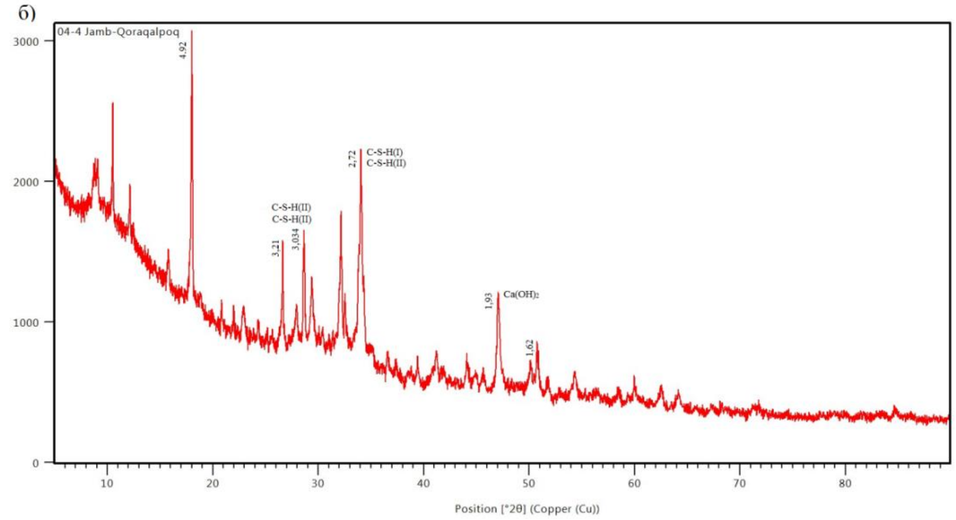
The polycarboxylate superplasticizer, having surface-active properties, slows down the onset of setting of the cement paste, while microsilica does not affect this characteristic. The joint introduction of additives in optimal amounts causes a more intense delay in the onset of setting, compared with individual modifiers. The end of setting of the cement paste, with the joint introduction of the optimal amount of additives, slows down by 25–30% of the duration of the end of setting of the composition without additives. This indicator should be taken into account in the heat and moisture treatment of modified concrete, since with an increase in setting time, it is necessary to lengthen the preliminary holding of reinforced concrete products.

Slowing down the start of setting of the cement paste for the concrete mixture is a positive thing - this increases the persistence of its workability and increases the range of transportation.

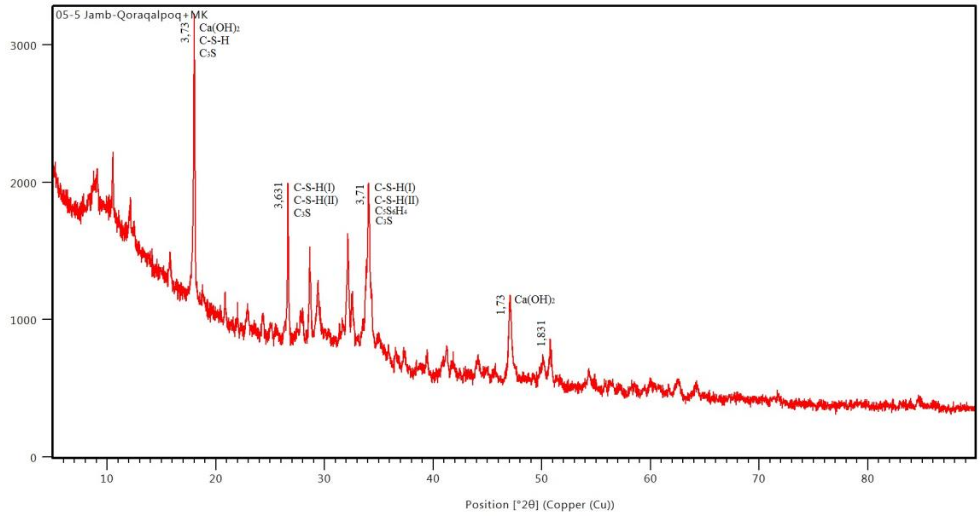
The maximum value of compressive strength after a day of normal hardening of samples of cubes with an edge of 20 mm was established at a content of SP within 0.6%, MS 8%, modified water repellent (MH) - 0.05%. The increased content of modifier additives slows down the set of daily strength, despite water reduction, that is, at the daily age, the set of strength is inhibited mainly due to the presence of a surface-active additive in the composition of the samples.

At the age of three days, the set of strength is determined by water reduction - the greater the dosage of the joint venture, the greater the compressive strength. The addition of microsilica increases the water demand of the mixture during the manufacture of samples, which causes a decrease in strength with the introduction of a complex additive.

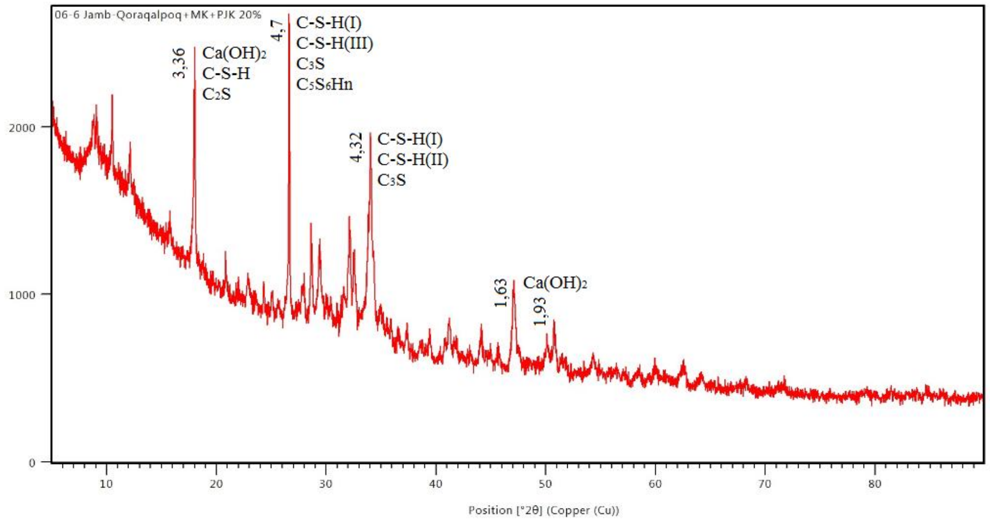
At 28 days of normal hardening, the pozzolanic microsilica additive contributes more actively to the increase in strength, both due to the greater degree of alite hydration and due to the formation of gel-like hydrosilicates, of the C–S–H(I) type. Such an effect of microsilica is especially characteristic at the optimal content of modifier additives, in which the introduction of MS to a lesser extent affects the strength of the cement stone.



Data of X-ray phase analysis of cement stone without additives.

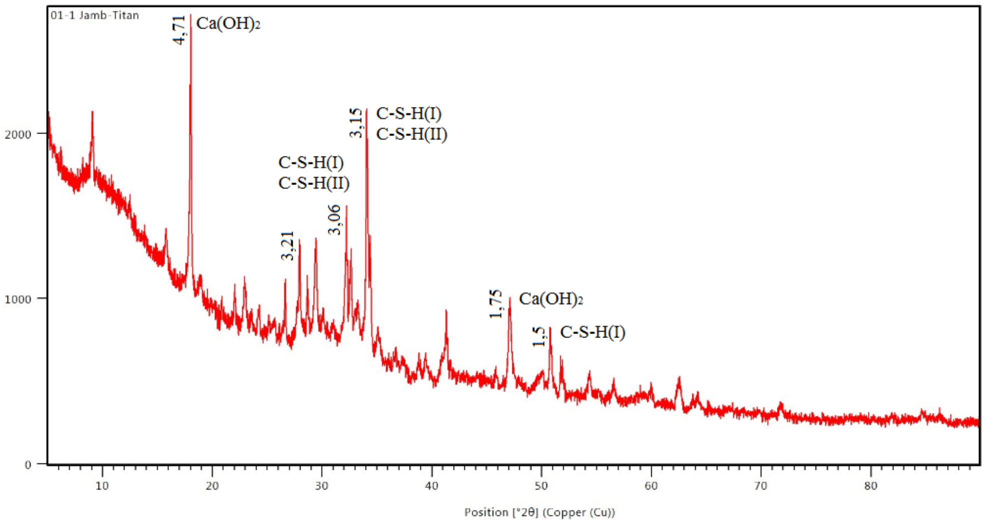


Data of X-ray phase analysis of cement stone +MS

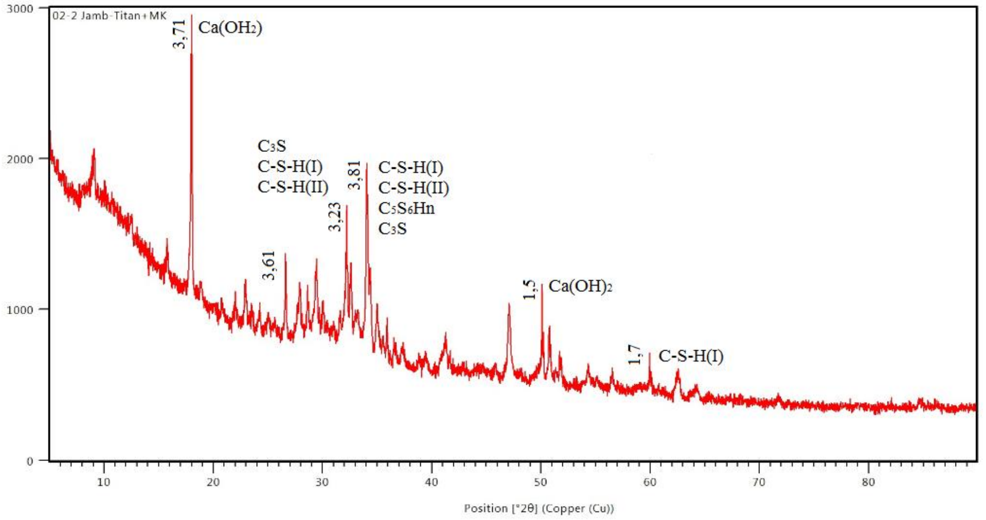


Data of X-ray phase analysis of cement stone SP+ MS + MH

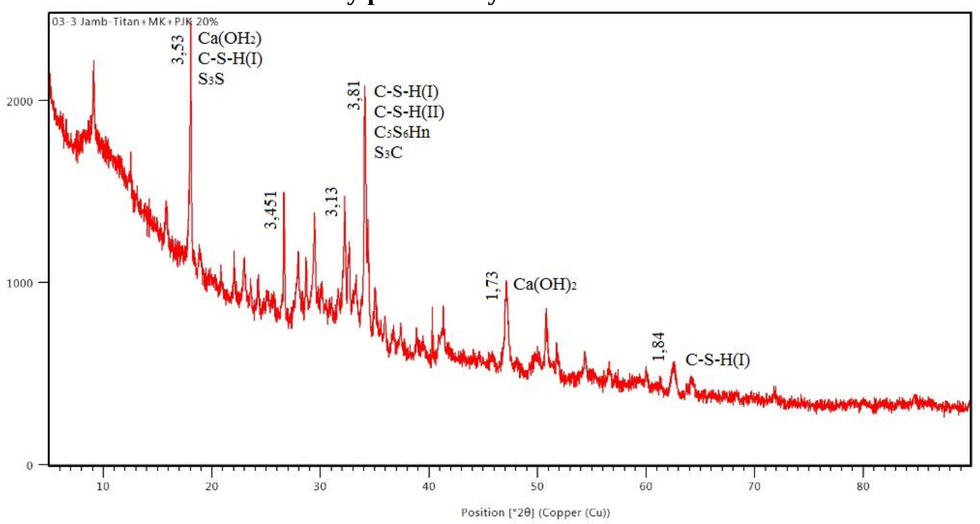
Fig.1. Data of X-ray phase analysis of Portland cement produced at the Karakalpak plant.



Data of X-ray phase analysis of cement stone without additives.



Data of X-ray phase analysis of cement stone +MS



Data of X-ray phase analysis of cement stone SP + MS + MH

Fig.2. Data of X-ray phase analysis of Portland cement produced at the plant "Titan".

Micro silica additive interacts with portlandite in the cement stone structure, promoting the formation of the C–S–H(I) phase; this reaction proceeds most intensively without the addition of SP. The polycarboxylate additive also reduces the content of portlandite in the cement stone due to complexation with calcium ions, but at the same time reduces the W/A and slows down the degree of cement hydration [4-6]. The maximum decrease in portlandite in cement stone, with the joint introduction of the optimal amount of SP and MS, MH is due to the fact that such a decrease in the amount of $Ca(OH)_2$ does not lead to a deterioration in the protective properties of concrete in relation to steel reinforcement. It is known from literary sources that until all the portlandite in the cement stone has reacted, concrete does not lose its protective properties in relation to steel reinforcement [5-6]. The obtained data on the amount of $Ca(OH)_2$ in the cement stone are confirmed by H. Taylor, who studied the structure of the cement stone modified with micro silica at low C/MS values at different ages [7].

According to the authors, micro silica, interacting with portlandite, contributes to the hydration of alite, which leads to an increase in the degree of hydration from 67% to 92%. With the introduction of a superplasticizer additive, the hydration of alit slows down to 63%, and the joint introduction of SP with active pozzolana levels this effect and increases the degree of hydration to 84%. That is, this complex of additives exhibits the properties of a cement hardening accelerator.

The kinetics of cement stone strength increase and strength at a certain age are a function of the phase composition of cement hydrate neoplasms.

Radiographs of cement stone modified with various additives at the age of 28 days are shown in Figures 1-2.

It follows from the X-ray phase study that the introduction of the SP additive reduces the content of portlandite in the cement stone, which correlates with the results of thermal analysis. The predominant hydrosilicate phase of cement stone with SP is a highly basic weakly crystallized phase of the C–S–H(II) type. The remaining hydrate phases are amorphous.

Modified with the addition of silica, cement stone is formed mainly from low-base hydrosilicates.

Joint introduction of plasticizing and pozzolan additives –0.8– 1% SP and 8-12% MK, and MG-0.05% contribute to the formation of a small amount of $Ca(OH)_2$ in cement stone, and hydrosilicates are mainly represented by the C–S–H(I) phase, reflections of calcium hydroaluminates, calcium hydrosulfoaluminates and non-hydrated residues of clinker minerals are also recorded. Complex additive SP+MS+MH contributes to the formation of gel-like hydrosilicate phases, of varying degrees of basicity, and the degree of their amorphization is higher than with the separate use of additives.

To clarify the available data, using a scanning electron microscope with an X-ray microanalyzer, studies of the structure of cement stone with various additives were carried out.

A photo of the microstructure of cement stone is shown in Figure 3-4.

Well–formed $Ca(OH)_2$ crystals, weakly crystallized masses of calcium hydrosilicates, type C–S–H(II), which, according to the X-ray microanalyzer, have increased basicity - $C/S \geq 1.5$, and areas of gel-like fine crystalline mass are visible on the chip surface.

Cement stone modified with the addition of MS (see Figure 1-2) is characterized by a dense surface with a cancerous-splintery fracture, a free calcium hydroxide content of 6.5% and consists mainly of low–base calcium hydrosilicates – C–S-H(I) phase.

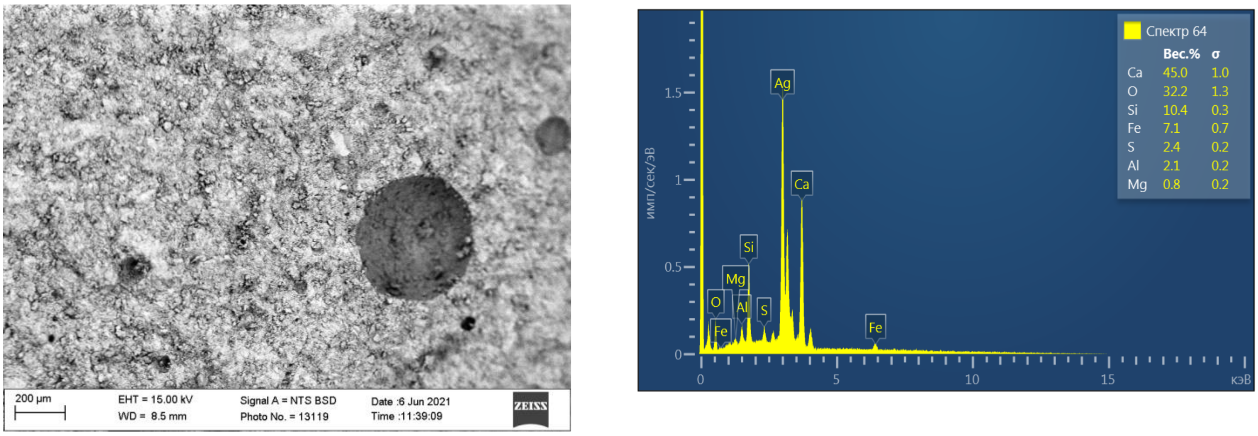
The microstructure of the cement stone modified by the introduction of a polycarboxylate superplasticizer is represented by gel–like highly basic hydrosilicate phases of type C–S-H(II) with $C/S \geq 1.5$, which is confirmed by the data of an X-ray microanalyzer.

Calcium hydroxide is distributed in cement stone in the form of small areas, in most cases, located between the hydrosilicate components.

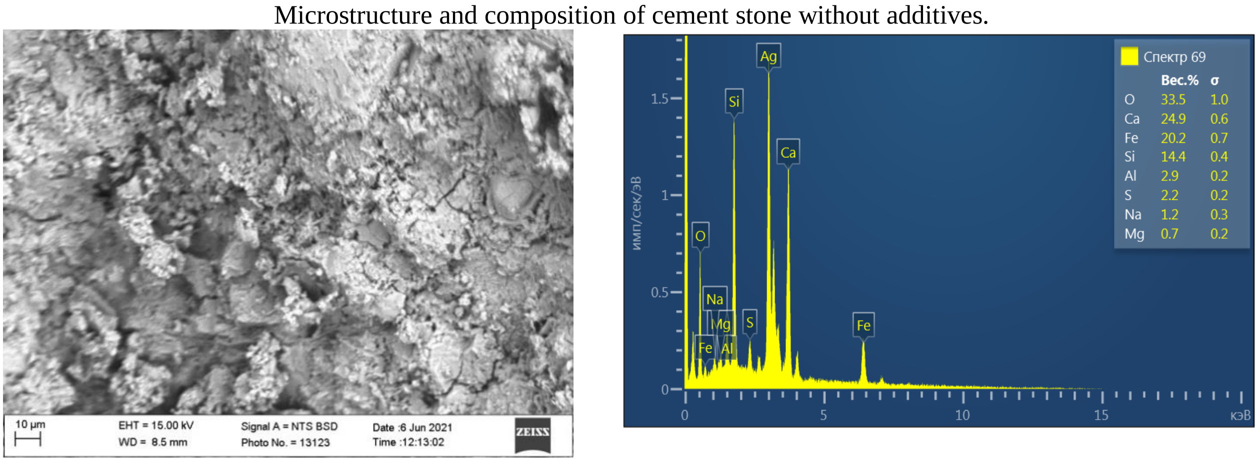
Areas of weakly crystallized gel–like C–S–H(I) phase with a ratio of C/S = 1.1- 1.3 are visible on the surface of the cement stone chip (see Figure 14) modified with additives of SP, MS, MH, and individual inclusions of calcium hydroxide are observed, with a total content of no more than 5%.

With the introduction of additives SP + MS +MH the structure of cement stone is formed mainly from weakly crystallized hydrate phases of lamellar form, type C–S–H(I), which leads to an increase in its density and strength.

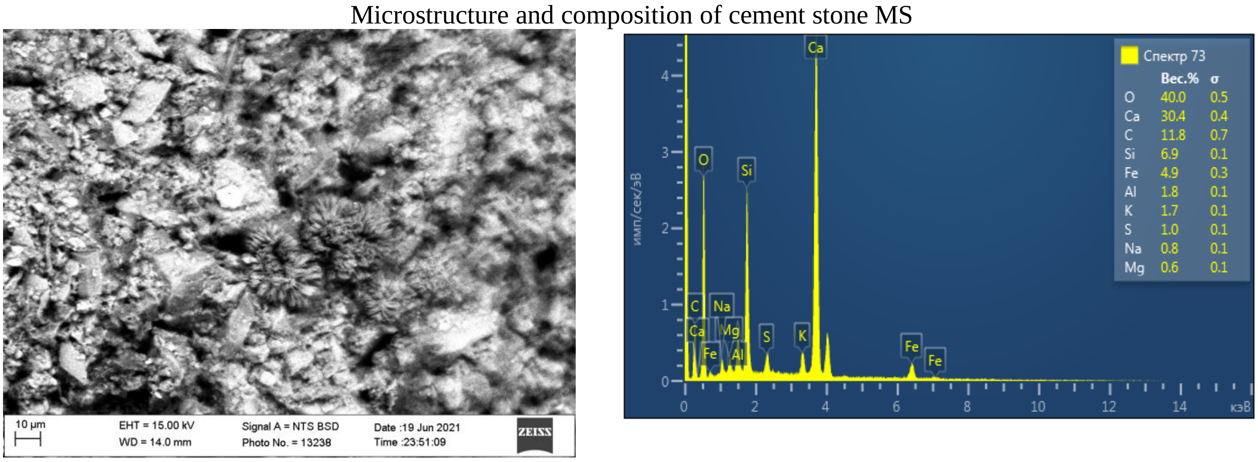
The specific surface area of cement stone samples (see Figure 3-4) was tested at the age of 28 days using the Brunauer-Emmett-Teller method. It is established that the additives SP + MS+MH contribute to an increase in the specific surface area of hydration products, respectively, a greater degree of amorphization of the structure.



Microstructure and composition of cement stone without additives.

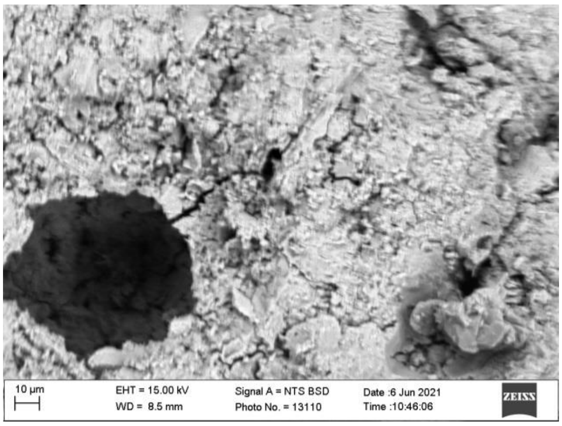


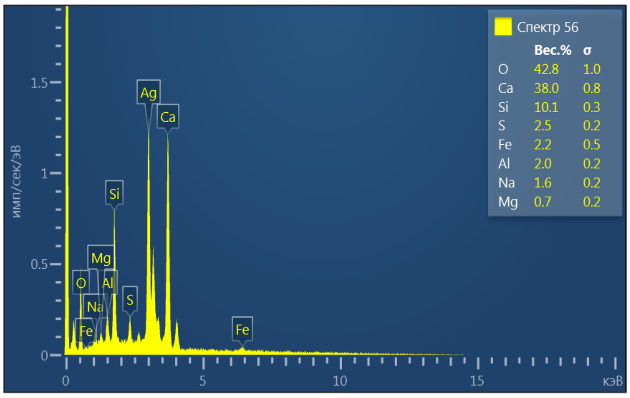
Microstructure and composition of cement stone MS



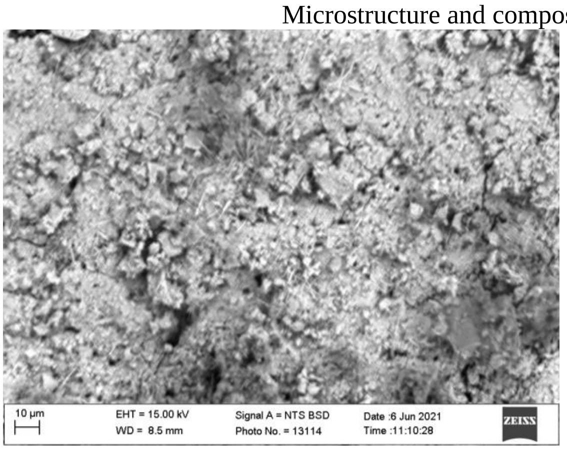
Microstructure and composition of cement stone SP+MS+MH

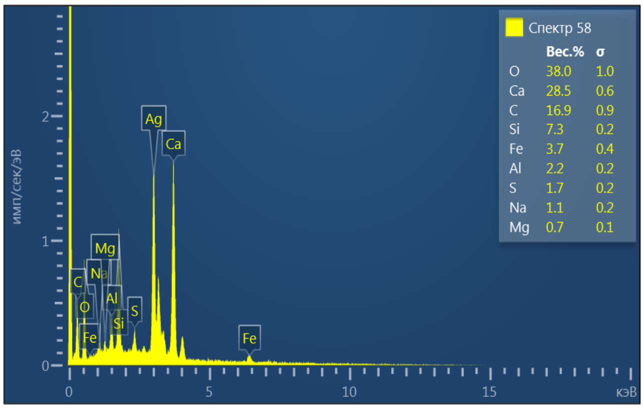
Fig.3. Data on the microstructure and composition of Portland cement produced at the Karakalpak plant.



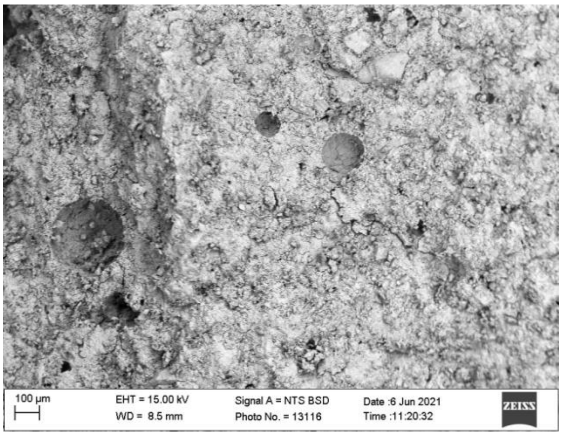


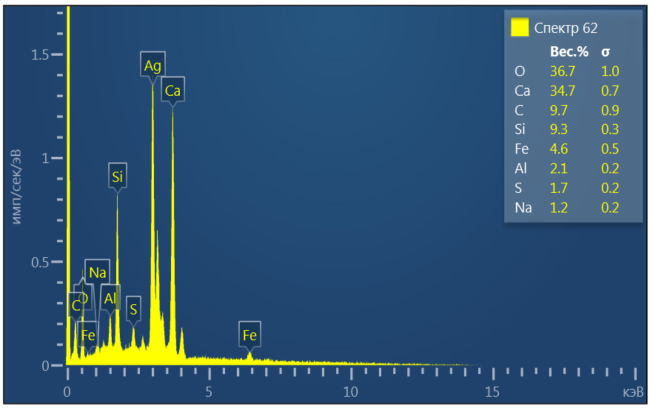
Microstructure and composition of cement stone without additives





Microstructure and composition of cement stone MS





Microstructure and composition of cement stone SP+MS+MH

Fig.4. Data on the microstructure and composition of Portland cement produced at the Titan plant.

Modifier additives, when co-injected into cement stone, reduce micro- and macroporosity to 16-17% with an increase in dosage, and the joint venture has a greater effect due to water reduction. Thus, according to the totality of all parameters, it was found that the modifying effect of each of the additives separately is not enough. The maximum effect, expressed in the directed formation of weakly crystallized low-base calcium hydrosilicates, with a minimum amount of portlandite of about 5%, is achieved with the combined use of additives at a dosage of 1% SP, 8-12% MS, 0.05% MH [6-8].

Conclusion

Modifier additives, when co-injected into cement stone, reduce micro- and macroporosity to 16-17% with an increase in dosage, and the joint venture has a greater effect due to water reduction. Thus, according to the totality of all parameters, it was found that the modifying effect of each of the additives separately is not enough. The maximum effect, expressed in the directed formation of weakly crystallized low-base calcium hydrosilicates, with a minimum amount of portlandite of about 5%, is achieved with the combined use of additives at a dosage of 1% SP, 8-12% MS, 0.05% MH [7-10].

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