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Article

Testing of cement stone modified with an additive based on industrial waste

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Abstract. Nowadays under the conditions of intensively developing construction production, hardening time and strength of Portland cement are becoming more and more relevant research issues of this type of binder. The basic requirements to the cement stated in the normative industry documents are considered. The strength limits of Portland cement of normal and fast-hardening are given. Raw materials for the production of fast-hardening and high-strength cement must be homogeneous and provide a high degree of fineness of the binder to increase its surface area. The production of cement with the addition of microsilica will make it possible to obtain concretes with the given characteristics without additional costs. To describe the methodology of testing cement recommendations of the main regulatory documents operating in Kazakhstan are used. Necessary means and equipment used in the testing laboratory and satisfying the existing standards are listed in a sequential manner. The procedure of preparing standard samples of cement dough is described step by step. The prepared specimens were kept in water at the required temperature regime. Determination of bending and compressive strength was carried out on accredited equipment. Processing of test results of cement samples with and without additives is given. Setting times of cement samples of two compositions were carried out according to the requirements. The dependence of the activity of the cement setting process on its temperature has been determined. **Keywords:** fast-hardening cement, high-strength cement, active mineral additive, test of strength, cement setting time.

1. Introduction

For a number of construction needs, in particular, for the factory production of reinforced concrete building structures and parts, as well as for high-speed construction, fast-hardening cement is required, which is more intense than conventional Portland cement, characterized by increased strength at the initial stage of hardening. Over a long period of time, the growth of strength in it slows down and after a long period of time can reach the strength of ordinary Portland cement [1, 2].

After 3 days, fast-hardening Portland cement M400 and M500 in accordance with Interstate Standard GOST 31108-2020 [3], the tensile strength must be at least 25 and 28 MPa, respectively, and after 28 days – 40 and 50 MPa. For these brands, the bending strength limit should not be less than 4 and 4.5 MPa after 3 days. According to Interstate Standard GOST 31108-2020 [3], the state Quality Mark was issued for fast-hardening cement, the bending strength limit after 3 days should be at least 4.5 MPa, the tensile strength after 3 days – at least 28, and after 28 days – at least 40 MPa. Tensile strength when compressing samples from this cement after 1 Day 3+2+2 must be at least 26 MPa.

The use of fast-hardening cement in factories for the production of reinforced concrete structures and parts significantly speeds up the production process [4]. The use of fast-hardening portland cement for monolithic concrete reduces the cutting time and reduces the mass of the structure, since the high strength of the resulting concrete allows you to reduce the cross-section of the structure [4]. To obtain high-grade prestressed concrete, high-strength Portland cement is

required. According to Interstate Standard GOST 31108-2020 [3], when testing on solutions of plastic consistency, the tensile strength of this cement at the age of 28 days should be at least 60 MPa and bending strength limit – at least 6.5 MPa. This cement should not have signs of "false installation".

Raw materials to produce fast-hardening and high-strength portland cement should be as homogeneous as possible in chemical composition and contain the minimum number of undesirable impurities [5]. The atmospheric or small crystal structure of the material is preferred they enter chemical interactions more easily than materials of a large crystal structure. It is desirable that the raw material mixture does not contain thermally resistant aluminosilicate of magnesian and siliceous and calcareous feldspar of limestone. Fast-hardening cement and high-strength Portland cement as raw materials, marls and montmorillonite clays are easier to learn when they are easily blurred and have a high surface [6].

To fulfill the tasks and goals, we used materials that meet the requirements of state standards. The raw material, in turn, was adopted in accordance with the quality of the material provided for reinforced concrete structures or hydraulic structures [3, 7–9].

The main characteristics that we have taken for research are presented in Tables 1, 2 which are shown below.

Table 1 – Chemical composition of Portland cement								
Cement	Cement	Chemical composition, %						
name	mark	SiO ₂	Al ₂ O	Fe ₂ O ₃	CaO	MgO	SO ₃	
CEM II/A-	500	21.5	5.8	3.95	63.8	2.05	2.9	
S42,5 N								

Table 2 – Mineral composition of cement								
Cement	Mineral composition, %				Alkali co	ontent, %	Gypsum	Active mineral
designation	C_3S	C_2S	C_3A	C_4AF	Na ₂ O K ₂ O		content by	supplements,
							SO3, %	%
1	2	3	4	5	6	7	8	9
Ι	47.91	19.09	8.64	13.56	-	-	2.10	-

Polyfractional sand was selected for testing of cement in accordance with the requirements of Interstate Standard GOST 6139-2020 [10].

Microsilica is formed in the melting process of ferrosilicon and silicon. After oxidation and condensation, the percentage of silicon monoxide is very small. The use of microsilica gives us a great opportunity to produce concretes with special characteristics for structures and products from simple materials. Concrete with microsilica has an excellent compressive strength that exceeds the strength of conventional concrete [11, 12].

In carrying out tests of cement were used standard methods of examination, which meet the requirements of the Standards of the Republic of Kazakhstan, Interstate standards.

2. Methods

In carrying out tests of cement were used standard methods of examination, which meet the requirements of the Standards of the Republic of Kazakhstan, Interstate standards.

To determine the high-strength cement, special controls and additional equipment were used, as well as a mixer equipped with a dosing device for sand.

Triple $40 \times 40 \times 160$ mm mold for making samples. The design of their mold must provide the ability to remove finished samples from them without defects or damage. This is necessary for ease of use when forms are attached to each other and to the baseplate to prevent water leakage. The baseplate must be firm to prevent subsequent vibrations. Forms for the plates are 210×185 mm in size and 6 mm thick, they must be made of glass with smooth edges.

Trowel and ruler – for laying the cement mortar.

A vibrating table was used to compact the mortar in the formwork.

A device for determining the bending strength of finished beam samples of the structure, providing the application of additional load according to the required scheme. Limit load up to 10 kN and additional average rate of load growth should be 50 ± 10 N/s.

Device for testing half specimens of any design, providing sample loading in pure compression mode only. Maximum load limit up to 500 kN. An error of $\pm 1\%$ is allowed only in the upper ranges.

Finished specimen shall have movable balls to compensate for spatial disturbance of bearing surfaces:

- moisture storage chamber.
- water storage tub.
- scales (error not exceeding 2 g);
- measuring cylinder.

Preparation of the standard sample:

The sample was prepared from cement as well as from polyfractional sand in a W/C ratio of 1:3 by weight. Cement mortar is used to prepare one mix, which is further used in the sample beams. Initially, the sand was poured into the mixer measuring device. The container of the mixer was wiped with a damp rag, poured with water, added cement, after which the mixer was turned on low speed (Figure 1).



Figure 1 – The steps of preparing the cement dough

Before making the samples, the inside of the mold walls should be lubricated with a small layer of machine oil. The joints of the outer walls of the formwork with the plate are oiled.

The nozzle is placed in the already prepared mold, placed on the platform of the shaking table and secured with clamps. The ready CR was removed one at a time directly from the mixer bowl using a spatula, 3 portions of CR each weighing about 300 g. We used a trowel to flatten the surface and a shaking table to compact each layer of mortar, 60 times per cycle.

After compaction was completed, we grinded the surface of the specimens with a metal ruler. Three specimens were prepared for each test.

The mold with the samples was covered with a plate and placed in the humidity chamber.

Twenty-four hours after making, the sample molds were removed from the chamber and very carefully removed. Next, the samples are measured to check the quality of the work done.

Twenty minutes before the test, the specimens were removed and placed in a water bath in a supine position. The water temperature was (20 ± 1) °C. Every 2 weeks we changed half of the water in the bath with clean water.

2.1 Determination of bending strength

The finished test specimen was mounted on the supports of the instrument so that its horizontal surfaces were in a vertical position and the facets with the numbered symbols were facing us. The specimen was tested according to the instructions for the device.

The load on the sample, that is, the average rate of loading, was 50 ± 10 N/s (Figure 2).



Figure 2 – Bend strength test of the specimen

The halves that passed the bend test were immediately subjected to a compression test. The specimen halves were placed between the plates so that its horizontal surfaces were in a vertical position and the marked surface was facing us.

The ends of the patterns protruded from the clamping plates by about 10 mm. The samples were tested in the same way as described in the instructions for the instrument. The load on the sample, that is, the average loading rate was 2400 ± 200 N/s.

2.2 Processing of results

The bending strength of the sample of a single beam R_b, MPa, is calculated by the formula:

$$R_b = \frac{1.5Fl}{b^3},\tag{1}$$

F – load on the sample, N;

b – value of the side of the square section of the beam, mm;

l – distance between the axes of supports, mm.

For the bending strength, the arithmetic mean of the test results of three specimens is taken. The calculation result is rounded to 0.1 MPa.

The compression strength of the individual half of the beam specimen R_c , MPa is calculated by the formula:

$$R_c = \frac{F}{s},\tag{2}$$

F – breaking load, N;

b – working surface area of the pressure plate, mm^2 .

For compressive strength, the arithmetic mean of the test results of the six halves of the beam specimens is calculated. The calculation result is rounded to 0.1 MPa.

If one of the six results differs from the arithmetic mean by more than 10%, this result must be excluded and the arithmetic mean of the remaining five results must be calculated.

If another result differs from the arithmetic mean of the other five results by more than 10%, the tests are considered unsatisfactory, in which case all results are invalid.

3. Results and Discussion

The setting time is the shortest curing period of the concrete mixture and occurs first.

The setting time depends on the composition of the concrete mixture and the ambient temperature. The higher the temperature, the more active the processes.

A sample without additives and a sample with an active mineral additive were prepared to compare the results (Tables 3 and 4).

Table 3 – Determination of cement setting time without additives

Parameter	_				Test resu	ults			
	1	2	3	4	5	6	7	8	9
	60	70	80	90	100	110	120	130	160
	min	min	min	min	min	min	min	min	min
Indicator of the device	0 mm	0 mm	2 mm	5 mm	1 mm				

Starting time of setting -130 min, end of setting -160 min.

1 au	H = DC	ummati		ment sett	mg time	with addit	110	
Parameter	Test results							
	1	2	3	4	5	6	7	8
	60	70	80	90	100	110	120	140
	min	min	min	min	min	min	min	min
Indicator of the device	0 mm	0 mm	0 mm	0 mm	0 mm	3 mm	5 mm	1 mm

Table 4 – Determination of cement setting time with additive

Starting time of setting -100 min, end of setting -140 min.

Thus, in Table 3, you can observe the studied sample without adding the active mineral additive. The beginning of formation of normal density of the sample occurred at the 120^{th} minute, and the completion at the 140^{th} minute.

If we compare the sample with the additive, we can see that the setting process will start 10 minutes faster and at the same time will finish 10 minutes earlier.

3.1 Determining the uniformity of volume change

The hardening process of cements is accompanied by a change in volume of the material. Portland cement shows a slight decrease in volume during hardening. If there is free calcium oxide CaO (more than 1%) in the cement clinker, and if there is excess MgO magnesium oxide (more than 5%), the quenching process of CaO and MgO, accompanied by a local increase in volume, represents an irregular volume change of cement during hardening, which leads to deformation and cracking of cement stone. Therefore, cement paste specimens during hardening by boiling in water are tested for the uniformity of cement volume change, which increases the damping of CaO and MgO and accelerates the testing.

Table 5 – Determination of cement setting time with additive					
Results of visual inspection after	Conclusion on the conformity of the cement mixture to				
boiling samples	the requirements for homogeneity of volume change of				
	cement during hardening				
No radial cracks reaching the edge,	When working with the mineral additive, an increase in				
a network of visible small cracks,	workability was observed after it was mixed and placed in				
or any bending or increase in	a Le Chatelier ring.				
sample size were observed on the	When the tiny particles of the mineral additive are				
front of the samples. The change in	dispersed throughout the volume, the mixture stabilizes				
volume was 0 mm.	and compacts, which significantly reduces splitting and				
	water outflow.				

4. Conclusions

This study was conducted as part of a project to develop a mixture to improve the strength of cement binder based on waste from a ferroalloy plant. The set tasks have been accomplished:

1. The composition and conditions of the samples were chosen.

2. The effect of dispersion of active mineral additive on cement binder has been proved. High strength characteristics as well as durability at work are provided.

3. Experience in demonstrating high grades of cement binders from M500 to M900 and peculiarities of their application.

4. Considered the possibility of using raw materials of the Republic of Kazakhstan, intended for the production of cement binder.

5. The optimal modes of production mixture and studied its physical and mechanical, construction and technical properties.

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