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Mineral complex additive for concrete

Methods of accelerating the hardening of Portland cement without heat-moisture treatment, in the presence of complex chemical additives - hardening accelerators and finely dispersed crystallization centers, were considered. The study was divided into several stages, where the effect of each accelerator additive on the cement hardening process was investigated. Based on the results, both single and complex chemical additives were selected to accelerate the hardening of cement. The effect of finely dispersed crystallization centers on the cement hardening process was also investigated. Metakaolinite (palygorskite) was used as a dispersed additive, which was synthesized by the authors using kaolin clay and insoluble magnesium compounds and sulfate aqua complexes, iron-sodium alum. The effect of complex additives in various combinations on the properties of cement dough and hardened cement is presented. The optimal composition of additives is proposed, the use of which in combination with technological methods for manufacturing concrete products can provide an energy-saving effect in the production of building structures.

Keywords: additives-cement hardening accelerators, crystallization seeds, cement structuring, sulfate alums

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Introduction

The experience of using concrete and reinforced concrete products in all branches of construction confirms that the main condition for the efficiency of its work and reliability is not only the quality indicators of the material, but the features of work in structures and buildings. The main component of any concrete is a binder, the characteristics of which determine its properties, by the type of which concretes are distinguished: cement, silicate, gypsum, slag-alkali, polymer concrete, polymer-cement concrete. [1], [3].

Concrete is subject to a number of requirements, which are often impossible to fulfill without the use of so-called modifiers and plasticizers, i.e. chemical additives that allow adding properties to the concrete mixture that meet the requirements of both designers and contractors. It is obvious that the use of concrete modifiers is beneficial both technologically and economically. The use of additives allows you to reduce energy costs for laying concrete, reduce cement consumption, maintain the necessary mobility of the mixture, ensure the necessary durability of structures,

and achieve excellent indicators of early and final strength of concrete [2], [23].

In the practice of manufacturing concrete and reinforced concrete products, heat-moist treatment has proven itself well to accelerate their hardening. However, this method is associated with significant energy consumption for obtaining steam. The modern energy crisis requires a radical revision of the production technology of the specified products, the search for energy-saving ways of accelerating their hardening [9].

The following methods of increasing the rate of hardening and increasing the strength of concrete are known [2, 17, 30]:

– use of chemical additives-hardening accelerators;
– introduction of crystallization seeds and intensification of nucleation processes due to reduction of activation energy;
– regulation of the pH environment of the hardening system;
– repeated vibration after the first stage of structure formation;
– provision of internal energy reserves and heat storage of exothermic processes.

The use of additives is the most effective way to improve the quality of concrete and does not require large capital expenditures. The competent use of targeted complex additives allows you to solve problems associated with obtaining concrete with specified properties [8], [11], [25].

As a rule, additives are two- or multi-component. Thanks to certain combinations of additives, concrete plants are able to obtain high-strength concrete with the required properties. After all, the same additives, depending on their quantity and the mineralogical composition of the cement, act differently. The hardening process of cement can be increased by introducing a small amount of mineral additives - highly dispersed substances, the so-called crystallization centers. [4], [10].

Chlorides, nitrates, nitrites, soluble sulfates and carbonates are widely used, have a positive effect, but their effect depends on the concentration, mineral composition of the cement, and the hardening period of the cement. The most famous is calcium chloride. It helps increase strength at all stages of hardening, but chloride ions are aggressive towards reinforced concrete reinforcement. [5, 13]. Nitrates are inhibitors of reinforcement corrosion, but they reduce the rate of hydration of dicalcium silicate [6, 29].

To increase the rate of cement hardening, it is necessary to increase the solubility of clinker minerals in silica and lime, and a complex additive of sulfates and chlorides provides this solubility.

The introduction of a complex of salts of calcium chloride and sodium nitrite allows eliminating reinforcement corrosion and increasing the effect of increasing strength achieved by the introduction of calcium chloride. Therefore, it may be advisable to use a complex additive of calcium chloride, sodium sulfate and sodium nitrite [22].

Setting objectives. The listed methods are usually used separately from each other. In the technical literature there is little information on the effect of several methods of increasing the rate of hardening of cement and concrete at once, except for the use of complex additives, therefore the purpose of the work is to develop a complex method of influencing the hardening processes of Portland cement, which combines the effect of chemical additives, centers of crystallization and repeated vibration after the first stage of structure formation.

Characteristics of input materials

The following materials were used for the experiments:

1. Portland cement brand PC I-500-N, with a compressive strength of 58 MPa at the age of 28 days, hardening time beginning 2 h end 8 h, specific surface area, 2500 cm2⁄g
2. Tap water was used for the samples.
3. For the manufacture of chemical additives were used: calcium chloride CaCI2, sodium nitrate (pure for analysis) - NaNO2, sodium sulfate crystallization (sodium sulfate) - Na2SO4, concentrated perchloric acid HCl with a density of 1.19 g/cm3, ferric chloride FeCl3, potassium carbonate K_2CO_3 [20], [28].

Methods

The work is divided into several stages:

I - selection of the type and optimal amount of chemical additives-hardening accelerators;
II – development of a method of preparation of highly dispersed mineral substances - centers of crystallization;
III – development of technological modes of manufacturing and hardening of concrete products.

At the first stage, well-known chemical additives were used: calcium chloride ($CaCl_2$), iron chloride ($FeCl_3$), sodium nitrite ($NaNO_2$), potassium carbonate (K_2CO_3), sodium sulfate (Na_2SO_4), C-3 superplasticizer, specially prepared and processed hydrated cement (SRN).

Single, double and those containing three or four chemical substances were studied. All samples in the form of cubes measuring 3×3×3 cm were formed from pure cement dough with a normal water demand of 25.4%. Portland cement of the PC 42.5 brand of Eurocement-Ukraine was used.

About 120 experiments were performed, the results of which were processed using the computer system, [18], [19].

Result and discussions

Table 1 shows the terms of hardening and strength characteristics of cements containing the most promising combinations of additives, after processing by a computer program.

It is possible to describe the following observations of single additives, which were introduced for 1.5% of the cement mass.

Calcium chloride, according to the literature, increases the solubility of clinker minerals, so there is

an increase in strength at all times of hardening. At the same time, it is an accelerator of the setting process.

Iron chloride helps to obtain higher strength at 7-day age, but in the initial period, it does not give the effect of acceleration of hardening. It requires 9-11% more mixing water than cement without additives. Not compatible with sodium nitrite, so it was excluded from further studies [14], [16].

Sodium nitrite plays the role of an accelerator to some extent during the first and second days, without particularly shortening the period of setting, but further inhibition of the rate of hydration is found.

Potassium carbonate causes very rapid structure formation in the first period, hardening times are sharply reduced, but there is no effect on the acceleration of cement hardening.

Sodium sulfate, according to the literature, has no equal in terms of increasing the strength of cement immediately after steaming. Sulfates increase the solubility of Portland cement clinker minerals in silica, better conditions are created for the hydration of minerals, but under normal conditions, the accelerating effect of hardening is not detected.

Superplasticizer C-3 is widely used in concrete technology, especially in monolithic construction. Its use helps to increase the mobility of the concrete mixture without reducing the amount of water, or reducing the water-cement ratio, which helps to increase the strength of concrete.

It is interesting to note the role of the mineral additive - the center of crystallization (CPH). Without any other chemical additives, the strength of Portland cement increased during the first day by 1.3 times, during two days by 1.8 times, and after 7 days - by 1.09 times. The effect of CPH on the rate of cement hardening in the initial stages can be compared with the effect of $CaCl_2$ in the amount of 1.5%. As noted earlier, CPH is pre-hydrated Portland cement using a special technology. In it, hydro silicates are in a gel-like state, but more crystallized compared to a cement block of ordinary concrete. These particles of hydro silicates form a spatial structure in "compressed conditions" and can be centers of crystallization.

In the theory of structure formation and hardening of binder systems, the presence of an induction period is noted, when the rate of hydration decreases by 15 times. Some researchers associate the end of the induction period with the crystallization of $Ca(OH)_2$ [5]. Calcium hydroxide in crystallized form is definitely present in the highly dispersed mineral additive of hydrated cement, which obviously affects the shortening of the induction period.

Complex additives for concrete are distinguished by the multifunctionality of their action. They affect simultaneously several characteristics of concrete. The use of complex additives enhances the effect achieved when a single-component additive is introduced (increase in strength), or eliminates a negative side effect (corrosion of fittings, shrinkage, etc.) [15].

In our studies, the concentration of chemicals in complex supplements was maintained at 1% each.

Hydrated Portland cement contained 31.2% $Ca(OH)_2$, which was determined by titration with hydrochloric acid. Such a large amount of calcium hydroxide is artificially introduced into the experimental cement before the start of hydration, so it will definitely affect the kinetics of the structure formation process. To reduce the amount of introduced $Ca(OH)_2$, a hydrochloric acid solution was added to the aqueous seed suspension at the rate of neutralization of 30, 60 and 90% calcium hydroxide, thereby increasing the content of CaCl2 in the system, which was formed during neutralization [27].

When combining single additives into complex ones, the following is sharply distinguished: CaCl2 + $NaNO_2$; $CaCl_2$ + CPH; $NaNO_2$ + Na_2SO_4, which give a significant increase in strength during all test periods; $NaNO_2$ + CPH; Na_2SO_4 + CPH without any special effect in the first day increase the strength of

At 7 days of age, and this, of course, is due to the presence of CPH [15].

Potassium carbonate in any combination, and even together with C-3 superplasticizer, dramatically shortens hardening times (up to 15-25 min), which makes its practical use impossible [21], [26].

Among the triple complex supplements, the following deserve attention:

$CaCl_2$ + $NaNO_2$ + C-3; $CaCl_2$ + $NaNO_2$ + CPH; $CaCl_2$ + Na_2SO_4 + CPH; $CaCl_2$ + $NaNO_2$ + C-3 + CPH. These additives already in the first day increase the strength of cement stone by 1.5 times while maintaining a higher rate of hardening during other terms. The research results show that in almost all cases with the best strength indicators, complex additives contain calcium chloride, which is recognized worldwide as an effective accelerator of cement and concrete hardening.

Unfortunately, chloride ions cause intense cor-rosion of reinforcement, especially when their con-tent is more than 1%.

There is information in the literature that the negative effect of calcium chloride on reinforced concrete reinforcement is almost completely elimi-nated by sodium nitrite. Moreover, NaNO¬2 at 20˚C in the amount of 2% shortens the induction period of hydration of tricalcium silicate from 7 hours to 20 minutes. The effect of NaNO2 on the kinetics of hydration of 3CaO·SiO2 in the initial stages is simi-lar to the effect of heat-moist treatment, which accelerates the hydration of mineral alit only after the formation of hydro silicate nuclei. The results of our experiments confirm these judgments in the case of using NaNO2 in combination with a mineral additive as a crystallization center.

In order to reduce the negative effect of CaCl2 on reinforcement, but to use its positive effect on the rate of cement hydration, a complex composi-tion additive is proposed: CaCl2 - 0.5%; NaNO2 – 0.5%; Na_2SO_4 – 0.5%; CPH - 1.5%. In it, each of the chemical substances enhances the effect of the other, as a result, Portland cement in the presence of such an additive shows a stable increase in strength during all periods of hardening, and in the first day, the increase in strength is 61% of cement without additives.

Table 1. - The effect of chemical additives on the hardening time and strength of hardened cement

№	Type and combination of additives	Hardening terms h/min		Compressive strength, MPa, per day		
		Beginning	End	1	2	7
1	Cement without additives	3^{15}	4^{30}	33,7	57,8	76,6
2	$CaCl_2$	1^{30}	2^{15}	45,7	69,1	86,6
3	$FeCl_3$	1^{25}	2^{40}	30,5	50,6	100,6
4	$NaNO_2$	2^{30}	4^{20}	42,4	66,4	62,9
5	K_2CO_3	0^{50}	1^{25}	19,7	41,3	60,9
6	Na_2SO_4	1^{30}	1^{55}	33,7	56,3	74,5
7	C-3	5^{30}	8^{15}	34,1	59,6	72,1
8	CPH	2^{00}	2^{50}	43,7	68,1	83,0
9	CPH+C-3	4^{35}	4^{40}	43,5	53,4	88,7
10	$CaCl_2$+$NaNO_2$	2^{00}	2^{45}	51,7	69,0	82,4
11	$CaCl_2$+ K_2CO_3	2^{05}	4^{35}	42,5	72,9	99,3
12	$CaCl_2$+ Na_2SO_4	2^{10}	4^{25}	42,2	58,4	78,4
13	$CaCl_2$+ C-3	3^{05}	5^{50}	44,1	61,2	97,1
14	$CaCl_2$+ CPH	1^{20}	2^{00}	44,6	71,7	119,9
15	$NaNO_2$+ K_2CO_3	0^{15}	0^{25}	19,1	38,5	46,1
16	$NaNO_2$+ Na_2SO_4	3^{25}	4^{25}	44,5	74,9	100,9
17	$NaNO_2$+ C-3	4^{00}	5^{00}	28,9	59,1	63,3
18	$NaNO_2$+ CPH	2^{35}	3^{45}	36,6	73,4	95,2
19	K_2CO_3+ Na_2SO_4	0^{05}	0^{10}	25,4	31,1	56,3
20	K_2CO_3+ C-3	1^{30}	2^{00}	29,0	39,0	69,7
21	K_2CO_3+ CPH	0^{05}	0^{15}	26,5	39,6	62,3
22	Na_2SO_4+ C-3	4^{30}	7^{25}	37,2	44,3	57,1
23	Na_2SO_4+CPH	3^{00}	4^{05}	36,2	42,1	91,0
24	$CaCl_2$+ CPH+ C-3	3^{00}	3^{30}	40,0	53,9	80,2
25	$CaCl_2$+ Na_2SO_4+ CPH	1^{40}	2^{15}	43,0	65,4	77,7
26	$NaNO_2$+ K_2CO_3+ C-3	1^{25}	1^{35}	31,5	59,5	89,4
27	$NaNO_2$+ Na_2SO_4+ C-3	0^{40}	4^{55}	26,5	64,3	75,9
28	$NaNO_2$+ K_2CO_3+ CPH	0^{05}	0^{15}	34,9	49,3	51,4
29	$NaNO_2$+ Na_2SO_4+ CPH	4^{15}	5^{15}	39,0	39,8	79,5
30	K_2CO_3+ C-3+ CPH	1^{10}	1^{20}	23,3	48,5	74,3
31	Na_2SO_4+ C-3+ CPH	5^{55}	6^{10}	39,8	64,2	75,0
32	$CaCl_2$+ $NaNO_2$+ C-3+ CPH	1^{55}	2^{45}	51,8	61,3	96,3
33	$CaCl_2$+ K_2CO_3+ C-3+ CPH	0^{20}	0^{40}	38,2	56,6	70,0
34	$CaCl_2$+ Na_2SO_4+ C-3+ CPH	2^{35}	4^{20}	43,7	62,7	79,0
35	$NaNO_2$+Na_2SO_4+C-3+CPH	2^{20}	4^{40}	38,1	49,9	82,9
36	$CaCl_2$+ $NaNO_2$+ Na_2SO_4	2^{40}	3^{50}	38,5	62,3	65,7
37	$CaCl_2$(0,5)+Na_2SO_4(0,5)+ $NaNO_2$(0,5)+ CPH	2^{20}	3^{35}	54,4	68,0	79,3

II method of preparation of mineral seed - center of crystallization during hardening of Portland cement.

The previous part shows the positive role of a highly dispersed mineral substance - the center of crystallization in combination with the proposed complex chemical additive composition: $CaCl_2$ - 0.5%; $NaNO_2$ – 0.25%; Na_2SO_4 - 0.5%. Although this composition of the additive has a positive effect on aluminate cement PC 42,5, and no clear effect was found for composite cement PC II B-Sh-32,5, obviously the hydrated cement primer is suitable for cements with a sufficiently high content of calcium aluminates.

Literary sources testify to the effectiveness of aluminosilicate crystallization centers, especially palygorskite [1], or natural alum stone - alunite [6]. Such breeds are very rare in nature.

Kaolin from the deposit of the Kirovohrad region was used to artificially obtain the aluminosilicate center of crystallization. In order to bring its properties closer to palygorskite or alunite, modifiers (insoluble magnesium salts and sulfate aqua complexes, iron-sodium alum) were added to kaolin. The content of magnesium cations Mg^{2+} corresponded to their content in palygorskite, and SO_4^{2-} anions corresponded to their content in alunite. The specified components were mixed with kaolin; the resulting mass was subjected to heat treatment at 800 ^{0}C.

After cooling, the product was ground in a laboratory ball mill to a specific surface area of 1500 cm^2/g.

Portland cement PC SH – 32,5 was used to test the efficiency of the new MKM crystallization center (metakaolin modified).

Dosing of additives was carried out in such a way that the cement contained 3% metakaolin and 1.3% SO_3. Based on the data in Table 1, in some experiments, potassium carbonate (potash K_2CO_3) in the amount of 0.25% or C-3 superplasticizer (0.15%) was additionally introduced into the cement. The strength characteristics of the obtained samples are shown in Table 2, they show that the use of complex additives plays a significant role in the processes of cement hydration and hardening. Cement without additives shows sufficiently low strength (13.98 MPa) per day. The addition of only metakaolin increases the strength, but very slightly (19.3 MPa). At the same time, modified metakaolin increased the strength in the first day to 24.9 MPa, i.e. 43.8% more than without additives.

It is very effective to introduce 0.25% potash along with modified metakaolin. In this case, the increase in strength was 70% on the first day, and 74% after 2 days. Compared with the data in Table 1, the overall strength index corresponds to the data in Table 2, although in the first case, high-quality PC 42,5 cement was used, and in the second - PC Sh-32,5, i.e. cements of different quality [31].

The addition of K_2CO_3 thickens the cement dough; therefore, superplasticizer C–3 (0.15%) was additionally added. The mobility of the cement paste increases sharply, but the rate of hardening slows down. The increase in strength in the first day is only 59%, although in 2 days the strength is equalized. The effectiveness of MKM action was tested on concrete samples. The samples measuring 10×10×10 cm were made from a concrete mixture of the following composition: PC Sh-32,5 – 350 kg/m^3; crushed stone fraction 5-10 – 1200 kg/m^3; quartz sand (Size module = 1.17) – 800 kg/m^3; MKM - 3%; W/C = 0.55. The test results are shown in Table 3.

Even concrete samples show the high efficiency of the proposed complex additive, especially in combination with potash K_2CO_3 and C-3 superplasticizer. The increase in strength is 65%, with the total strength in 2 days of hardening, giving reason to judge the possibility of early use of products, as well as a reduction in cement consumption per $1m^3$, since the additive contributes to the full use of the potential of cement.

Table 2. - The effect of chemical additives and mineral aluminosilicate seed on the rate of cement hydration

№	Consumption of materials					Compressive strength, МПа	
	Cement, g	МК, %	МКМ, %	K_2CO_3, %	W/C	1 day	2 days
1	350				0,32	13,98	14,74
2	350	3			0,32	19,3	23,8
3	350		3		0,32	24,9 + 43,2%	30,23 + 51%
4	350		3	0,25	0,32	47,8 + 70%	57,9 + 74%
5*	350		3	0,25	0,32	34 + 59%	53,4 + 72%

МК – metakaolin; МКМ - modified metakaolin

* – C-3 superplasticizer is additionally added to the mixing water.

Table 3. - The effect of a complex additive on the strength of concrete

№ з/п	The presence of an additive	Compressive strength, MPa	
		1 day	2 days
1	No additives	5,59	8,09
2	МКМ 3% + K_2CO_3(0,25%)+С3 (0,15%)	12,36	23,1

Conclusions.

Three experiments were carried out during the research work. In order to study the effect of complex chemical additives on the hardening rate of Portland cement, more than 120 experiments were carried out. It has been established that the combination of single additives into double or triple is not always justified, but there are combinations when one additive enhances the effect of another with a positive effect on accelerating the hardening of cement.The expediency of using mineral seeds - cement crystallization centers - is shown. Pre-hydrated Portland cement or aluminosilicates prepared in a certain way can act as a primer.

The combination of complex chemical additives with a mineral seed increases the strength of the cement stone compared to the one without additives already in the first day by 1.6-1.8 times.

Recommended composition of additives for aluminate cement PC – 42,5: $CaCl_2$ - 0.5%, $NaNO_2$ - 0.25 - 0.5%; Na_2SO_4 - 0.5 - 1.0%; mineral seed (hydrated cement) - 1.5 - 3.0%. For composite cement type PC-Sh -32,5 the center of crystallization is more suitable.

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Мінеральна комплексна добавка для бетону

Розглянуто способи прискорення тверднення портландцементу без застосування тепловологісної обробки, з використанням комплексних хімічних добавок–прискорювачів тверднення та тонкодисперсних центрів кристалізації. Встановлено доцільність поєднання кількох технологічних прийомів, зокрема хімічного впливу, введення попередньо гідратованого цементу як активного мінерального наповнювача, а також використання повторного вібрування після першої стадії структурування. Дослідження поділено на кілька стадій, у межах яких проведено понад 120 експериментів з різними варіантами добавок та їх поєднань. Вивчено дію одно- та багатокомпонентних комплексних добавок, до складу яких входили хлориди, нітрити, сульфати, залізо-, калій- та натрійвмісні сполуки, а також надпластифікатор С-3. Виявлено оптимальні комбінації компонентів, які забезпечують прискорене тверднення цементного тіста без погіршення фізико-механічних характеристик. Окремо досліджено вплив тонкодисперсних центрів кристалізації, зокрема попередньо гідратованого портландцементу, який виконує функцію структурного ініціатора в період індукційного сповільнення гідратації. Показано, що поєднання мінеральних центрів кристалізації з комплексними хімічними добавками забезпечує приріст міцності цементного каменю вже на першу добу в 1,6–1,8 рази порівняно з контрольними зразками без добавок. Запропоновано рекомендовані склади добавок для цементів різних типів (ПЦ 42,5; ПЦ-Ш 32,5), які дозволяють скоротити тривалість технологічного циклу тверднення та досягти енергозберігаючого ефекту у виробництві бетонних та залізобетонних конструкцій.

Ключові слова: добавки–прискорювачі твердіння цементу, затравки кристалізації, структуроутворення цементу, квасці сульфатні

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