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Effect of a complex modified additive on the setting time of the cement mixture

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ABSTRACT
The article presents studies of the effect of a complex modified additive on the setting time of cement paste. The work describes the method of determining the setting time of the cement paste of standard consistency, the selection of the optimal composition of the additive at different percentages of its components, allowing accelerating the setting time. In this work, the authors used a complex modified additive including alkali (caustic soda NaOH), post-alcohol bard (alcohol production waste), and hardening accelerator (gypsum) in different percentages. Performed a comparative study of the effect of additives on changes in setting time. The analysis suggests that the additive in the optimal amount leads to changes in the setting time compared with the reference sample, but within the standards. It is shown that the combined use in the composition of a complex modified additive, having well-compatible mechanisms of their influence on the processes of hydration, setting, and hardening of the cement paste, mutually complements and enhances the effect of each ingredient of the additive. Increasing the concentration of the complex modified additive in the cement paste not only affects the liquefaction process but also reduces the setting time and hardening of the cement paste.
Keywords: cement paste, hardening accelerator, post-alcohol bard, complex modified additive, setting time.

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Introduction

In modern conditions of development of construction in Kazakhstan on the background of rapidly developing technologies, the question is raised about the introduction of new quickly recouped technologies, which are based on the extensive use of local raw materials and new technical methods in order to obtain highly efficient materials.

The basis of modern concrete technology is the creation of high-quality artificial stone, characterized by high dispersion, a small imperfection, and structural stability. Improvement in the quality of concrete compositions can be achieved both by the use of chemical additives, and when using local components to create a new generation of concrete, which is a highly relevant objective of concrete technology. A new generation of concrete is high-tech, high-quality, multiconcrete mixtures and compositions with additives that preserve the required properties at a service in all operating conditions. Growing multicomponent concretes are due to significant systemic effects, which enables to manage of the structure formation at all stages of the technology, ensuring receipt of composites of "directed" quality, composition, structure, and properties [[1], [2], [3], [4], [5], [6], [7]].

Modern cement concrete is a composite building material, which can be produced with the specified characteristics for certain service conditions by modifying its structure and properties with various admixtures [[8], [9], [10], [11]]. This provides the material with durability, performance reliability, ecological safety, and applicability in any service conditions [12].

The present economic constraints require acceleration in the speed of work in the construction industry. The need for concrete with sufficient strength at a very early age is in many situations very important. Such concrete is obtained through the use of some admixtures such as water-reducing superplasticizers, set accelerators, and hardening accelerators. Standards about admixtures for concrete and mortar differentiate between set and hardening accelerators namely:

– Set accelerator is defined as an admixture that decreases the initial setting for the transition of the mix from the plastic to the rigid state.

– Hardening accelerator is defined as an admixture that increases the rate of development of early strength in the concrete with or without affecting the setting time [13].

A wide range of domestic and imported chemical additives makes it difficult to make a choice. Concrete manufacturers seek to improve its properties by modification while reducing the consumption of cement, reducing energy costs in the production of reinforced concrete, and minimizing the cost of additives under stable terms of their quality. It is quite a challenging task that can be solved using a variety of waste and coproducts of many industries as mineral and chemical modifiers of concrete [[14], [15], [16], [17], [18]]. Most often, the properties of concrete are modified by chemical additives. At the same time, optimal characteristics, such as strength, workability, and so on are achieved at a certain critical dose of the chemical modifier, after which the effect falls off [[19], [20], [21], [22], [23]].

The setting of concrete is identified as the transition of fresh concrete from the liquid phase to the solid phase. It is important to identify this phase change to plan to transport and place concrete [24].

It is known that the main purpose of plasticizing additives is to increase workability, which provides a reduction in energy and labor costs for laying. On the other hand, the use of such additives allows, by reducing the water-cement ratio, while maintaining the given mobility of the mixture, to increase significantly the strength and durability of products. In addition, the introduction of plasticizers can affect the setting time and hardening kinetics of cement, increase strength, frost resistance, and water resistance of concrete due to water reduction, as well as reduce cement consumption and energy consumption for the production of concrete, mortars, etc. Therefore, the development of compositions of modified heavy concretes is relevant for general construction purposes with improved technological parameters by using effective modifying additives.

The composition of the complex modified additive includes gypsum (hardening accelerator), alcohol production waste (post-alcohol bard), and alkali (caustic soda, NaOH) to neutralize the acidity of post-alcohol bard, this is due to the fact that the purification is not done well and post-alcohol bard retains acidity. Thus, the combined use of gypsum, alkali (caustic soda NaOH), and plasticizer (post-alcohol bard) improve the physical and mechanical properties. The post-alcohol bard or sulfite-yeast bard is a waste product of alcohol production. After distillery bard is a valuable product, which can be used in solving the problems of environmental pollution and obtaining cheap raw materials.

The aim of the study is to develop a complex modifying additive (CMA) and study its effect on the physical and mechanical properties of cement systems.

As part of this study, a set of laboratory tests to assess the physical and mechanical properties of experimental samples, followed by a comparative analysis of changes in the qualitative characteristics of the cement and the effect of a complex modifier on it. However, within the framework of this article the results of the first stage of the study will be presented, exactly the effect of the variable composition of CMA on the setting time of the dough of standard consistency, as well as its effect on the flow of the mixture.

In order to achieve the goal, the following tasks were solved:

1. Selection of the optimal composition of the additive at different percentages of its components

2. Preparation of samples of variable composition of additive components in laboratory conditions

3. Laboratory research of physical and mechanical properties of experimental samples.

Experimental technique

To conduct research and fulfill the set goal and objectives we used materials that meet the requirements and standards.

Cement. Raw materials were taken according to the geographical location of the factories of manufacturers, as well as the qualitative indicators of the material. Portland cement of M400 grade was used as a binder, due to the availability of this binder.

Modifying additive. The main component of the modifying additive is post-alcohol bard - the residue after the distillation of alcohol from brewers, ethanol production waste, which meets the requirements of TU 1110 RK 00393896 OJSC -01-2003, in amounts of 2.5%, 5.0%, 7.5%, 10%, a multiple of 2.5%. It is supplied in liquid form by the manufacturer JSC "Aydabul distillery".

Hardening accelerator - gypsum which accelerates the hardening process in amounts of 1%, 1.5%, 2.0%, 2.5% of cement mass, a multiple of 0.5%.

At the first stage, the joint effect of the optimal composition of CMA on the setting time of the paste of standard consistency (beginning and end of setting) was studied, and determined in accordance with the requirements of GOST 310.3-76 types of cement. Methods for determining the normal thickness, timing of setting and volume change uniformity". The consistency, initial and final setting of the cement paste were investigated using a Vica device, as shown in Figure 1. Setting time of cement paste is one of the most important parameters of the concrete mixture, as they determine the further performance properties of the material.

The setting time test was carried out using six mixing proportions (17 samples) consisting of Portland cement, additive, and water. The sample (Type 1) containing only cement and water was called the reference sample, while the proportion of additives was varied as shown in Table 1. The reference sample (100% Portland cement, which is equivalent to 350 g and water weight of about 105 g). Consumption of raw materials of mortar samples (required for measuring the setting time) is presented in Table 1.

Table 1 – Composition of compared samples

Type of sample	Cement, g	Gypsum, g	Post-alcohol bard, g	Caustic soda, g	Water, g
Type 1 Reference sample	350	-	-	-	105
Type 2-1	346.5	3.5	8.75	0.4375	95.8125
Type 2-2	346.5	3.5	17.5	0.875	86.625
Type 2-3	346.5	3.5	26.25	1.3125	77.4375
Type 2-4	346.5	3.5	35	1.75	68.25
Type 3-1	344.75	5.25	8.75	0.4375	95.8125
Type 3-2	344.75	5.25	17.5	0.875	86.625
Type 3-3	344.75	5.25	26.25	1.3125	77.4375
Type 3-4	344.75	5.25	35	1.75	68.25
Type 4-1	343	7	8.75	0.4375	95.8125
Type 4-2	343	7	17.5	0.875	86.625
Type 4-3	343	7	26.25	1.3125	77.4375
Type 4-4	343	7	35	1.75	68.25
Type 5-1	341.25	8.75	8.75	0.4375	95.8125
Type 5-2	341.25	8.75	17.5	0.875	86.625
Type 5-3	341.25	8.75	26.25	1.3125	77.4375
Type 5-4	341.25	8.75	35	1.75	68.25

Figure 1 – Determination of the setting time of the cement paste on the Vica device

Results and Discussion

On the diagram (figure 2) of the setting times, the first peak corresponds to the beginning of setting, the second to the end. The arrangement of the types of compositions compared in ascending order from bottom to top, where red corresponds to type 1 - the control sample (reference sample) without additives, with respect to which comparisons are made. Setting the time of cement paste is one of the indicators that characterize the manufacturability of the preparation of cement concrete mixtures. The introduction of additives can reduce the time between the beginning setting and the end of the setting. This is of practical interest, as the introduction of additives - in small quantities will accelerate the process of making products.

The choice of additive was determined based on the main mechanism of their action. Post-alcohol bard has hydrophilic and hydrophobic properties. Post-alcohol bard has a plasticizing effect, which leads to obvious consequences, i.e. reducing the w/c ratio and maintaining mobility of the concrete mixture, increasing workability, and giving strength and durability to structures. This additive wets the surface of cement particles, thus providing a decrease in the w/c ratio. The combined use of a complex additive, which has well-compatible mechanisms of their influence on the processes of hydration, setting, and hardening of the cement mass, mutually complements and enhances the effect of each ingredient of the additive.

It may be noted that in the case of the effect of CMA on the time of setting of cement paste to a greater extent associated with the dosage. The effect of the studied compositions may be due to the presence in their composition gypsum, which traditionally are hardening of cement setting and additives that form shells on the cement particles, preventing the penetration of water for hydration.

According to Table 1, it is the sodium compounds indicator that changes with changes in the quantitative indicator of the post-alcohol bard. Sodium compounds were used in small quantities (previous studies have revealed the optimal concentration), exactly in the amount to get a neutral medium, i.e. to stabilize the hydrogen index (pH) of the additive. Consequently, the sodium compound values do not become uneconomical for cement systems production. The hydrogen index needs neutral because increased acidity slows down the setting time of the concrete.

It can be seen from the test results in Figure 1 that not only the setting time is reduced, but also its end. The complex additive has a coarse effect on the system, that is, when increasing the dosage sharply reduces the process of cement setting compared with samples without additives (Type 1), and intensifies the hardening of cement in the early hydration time at the age of 3 and 7 days.

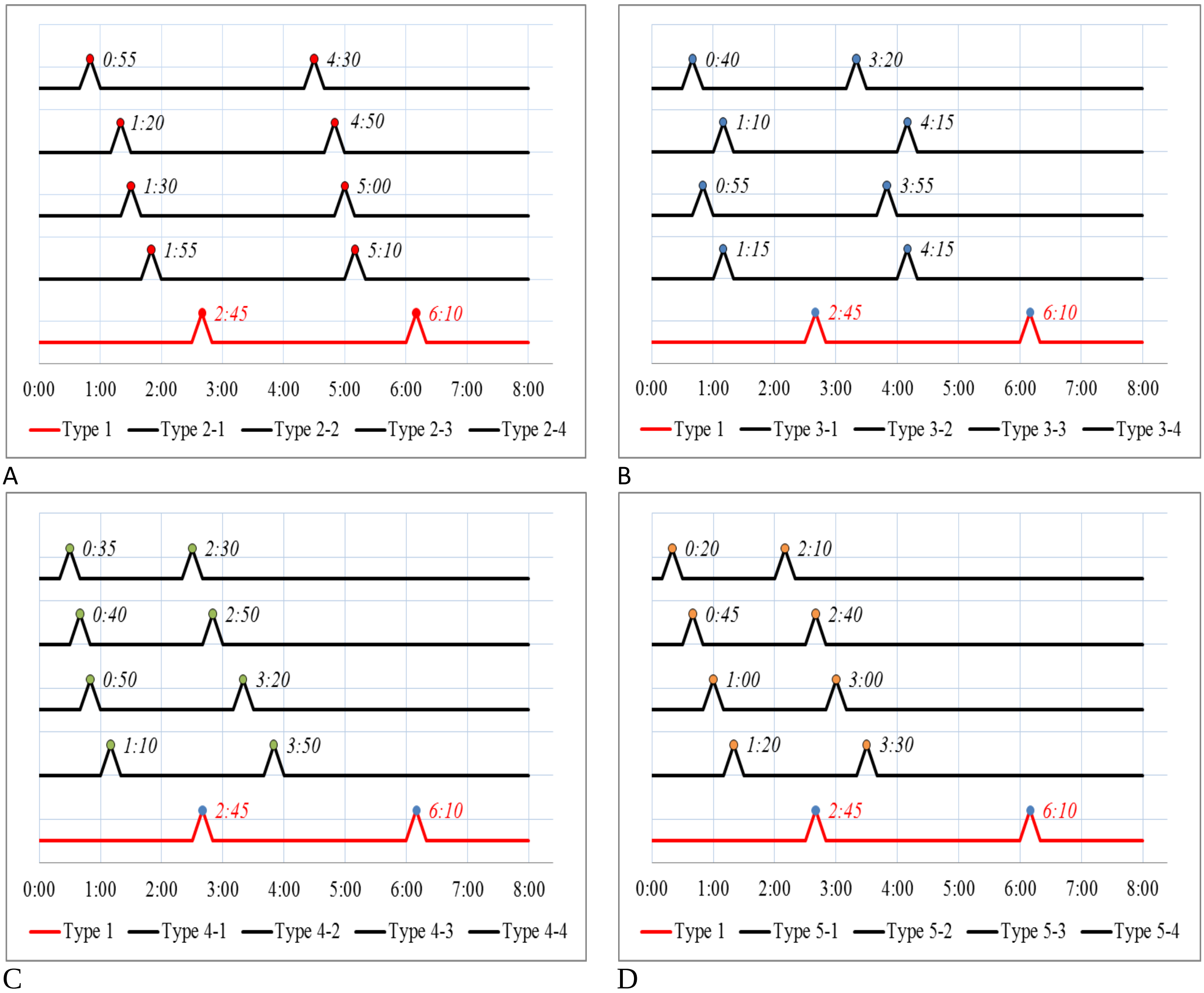

Figure 2 – Results of testing the setting time of the cement mixture

According to the results (figure 2) of the study determined the setting time of the compositions for:

Type 1. The beginning of setting of the cement mass without additives is 2 h 45 min, and the end of setting 6 h 10 min.

Type 2-1. When adding the composition with 1.0 % gypsum (by weight of cement of the standard) and 2.5% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 55 min, and the end of setting time is 5 h 10 min, respectively.

Type 2-2. When adding the composition with 1.0 % gypsum (by weight of cement of the standard) and 5.0% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 30 min and the end of setting time is 5 h 00 min, respectively.

Type 2-3. When adding the composition with 1.0 % gypsum (by weight of cement of the standard) and 7.5% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 20 min and the end of setting time is 4 h 50 min, respectively.

Type 2-4. When adding the composition with 1.0 % gypsum (by weight of cement of the standard) and 10 % additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting is 55 min and the end of setting time is 4 h 30 min, respectively.

Type 3-1. When adding the composition with 1.5 % gypsum (by weight of cement of the standard) and 2.5 % additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 15 min and the end of setting time 4 h 15 min, respectively.

Type 3-2. The addition of 1.5 % gypsum (by weight of standard cement) and 5.0% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 55 min and the end of setting time is 3 h 55 min, respectively.

Type 3-3. When adding the composition with 1.5 % gypsum (by weight of cement of the standard) and 7.5 % additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 10 min and the end of setting time is 4 h 15 min, respectively.

Type 3-4. When adding the composition with 1.5 % gypsum (by weight of cement of the standard) and 10 % additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 40 min and the end of setting time is 3 h 20 min, respectively.

Type 4-1. When adding the composition with 2.0 % gypsum (by weight of cement of the standard) and 2.5 % of additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 10 min and the end of setting time is 3 h 50 min, respectively.

Type 4-2. When adding the composition with 2.0% gypsum (by weight of cement of the standard) and 5.0% of additive (post-alcoholic bard) is introduced into the cement paste, the beginning setting time is 50 min and 3 h 20 min for the beginning and the end of setting time is 3 h 20 min, respectively.

Type 4-3. When adding the composition with 2.0% gypsum (by weight of cement of the standard) and 7.5% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 40 min and the end of setting time is 2 h 50 min, respectively.

Type 4-4. When adding the composition with 2.0% gypsum (by weight of cement of the standard) and 10% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 35 min and the end of setting time is 2 h and 30 min, respectively.

Type 5-1. When adding the composition with 2.5% gypsum (by weight of cement of the standard) and 2.5% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 20 min and the end of setting time is 3 h 30 min, respectively.

Type 5-2. When adding the composition with 2.5% gypsum (by weight of cement of the standard) and 5.0% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 1 h 00 min and the end of setting time is 3 h 00 min, respectively.

Type 5-3. When adding the composition with 2.5% gypsum (by weight of standard cement) and 7.5% masterbatch (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 45 min and the end of setting time is 2 h and 40 min, respectively.

Type 5-4. When adding the composition with 2.5% gypsum (by weight of cement of the standard) and 10% additive (post-alcohol bard) is introduced into the cement paste, the beginning of setting time is 20 min and the end of setting time is 2 h 10 min, respectively.

As can be seen from the results, the maximum plasticizing effect of the additive in mortar cement mixture is achieved at a concentration of 5-10% (post-alcohol bard) and 1.5-2.5% (gypsum) in relation to the mass of cement at w/c ratio = 0.3. The introduction of the additive into the mortar mixture has a plasticizing effect, which allows reducing the water-cement ratio by 10% as compared to the control composition. The effect of plasticizing cement paste is explained by the adsorption of hydrophobic molecules on the surface of dispersed cement particles. The setting time of mortar mixtures significantly depends on the concentration of the additive in them. Increasing the additive concentration in the cement mixture up to 10% (post-alcohol bard) and up to 2.5 % (gypsum) of the cement mass not only affects the process of liquefaction of the cement mortar, but reducing the time of setting and hardening of the cement paste, but also increases the compressive strength of the cement stone.

Analyzing the graph in Figure 2, the authors concluded that the additives have a different effect: with the addition of additives in cement, there is a beginning of setting at about 20 min - 1 h 55 min, while in the without additives (Type 1) cement begins to set after 2 h 45 min. Taking into account it can be argued that these additives have a higher efficiency than the without additives (Type 1) composition. Analyzing the graph in Figure 2, it can be noted that the additives have the same effect, but with different efficiencies. With the introduction of a complex additive setting time is reduced by up to 30% compared with pure cement. In this case, obviously, the duration of the cement paste remains unchanged, as the setting time is reduced by reducing the time of the end of the setting. At the same time, the interval between the beginning and the end of the setting is reduced by

40 %. This index is important for dry construction mixtures, as it enables cement-containing compositions after hardening with water to keep plasticized state during adjustable time intervals depending on additive content and to set quickly after application, which results in accelerated speed of construction works.

The question of fast setting is highly topical because the tendency of reduction of terms of construction goes at the expense of fast stripping. Influences of alcohol production waste consumption up to ≈ 10% showed the rationality of this range, i.e. with an increasing quantitative indicator of post-alcohol bard we do not get the specified effect. The maximum optimal range was determined by optimizing the composition. The additive range was determined by a complex of studies such as strength, water absorption, and other indicators that affect the workability of concrete, so within this article only the indicators of the setting time are shown.

The amount of the additive introduced was set from the condition of the greatest effect of accelerating hardening, as well as obtaining the maximum increase in the strength of the samples compared to their counterparts without additives. The results obtained show that CMA acts as a good hardening accelerator. Consequently, this additive can be used as a setting time regulator.

Conclusions

Based on the experimental studies, the following conclusions can be made that the complex modifying additive (CMA) has a better water - reducing effect than the without additive composition (Type 1). The addition of the additive (CMA) tended to accelerate both initial and final setting times. The addition of the additive (CMA) gave the best results because it delayed the initial setting time from (1 h 55 min) to (20 min) and the final setting time from (5 h 10 min) to (2 h 10 min). According to the results of studies, the authors concluded that the introduction of additives - plasticizers (post-alcohol bard) reduces the amount of water by 35%. Thus, the experiments showed that when mixing cement paste with CMA there is a plasticizing effect on cement paste and a significant reduction of the time at the beginning and end of the setting time of cement. This is of practical interest, as the introduction of additives will accelerate the process of making products.

Studies have shown that the use of gypsum in combination with sodium compounds and post-alcohol bard significantly increases the physical and mechanical properties, creating a synergistic effect.

The use of plasticizers in concrete technology allows to reduce porosity and increase the strength of samples. The use of plasticizers in the concrete mixture significantly increases the resistance and durability of heavy concrete. Consequently, the use of a complex modifying additive allows you to purposefully change the structure of concrete and thus significantly increase the complexity of physical and mechanical parameters and durability of modified concretes.

Conflict of interest

On behalf of all the authors, the correspondent author states that there is no conflict of interest.

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Кешенді модификацияланған қоспаның цемент қамырының ұстасу мерзіміне әсері

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ТҮЙІНДЕМЕ

Мақалада цемент қамырының ұстасу мерзіміне кешенді модификацияланған қоспа әсерінің нәтижелері көрсетілген. Жұмыста қарапайым консистенциядағы қамырдың ұстасу мерзімін анықтау әдістемесі, ұстасу уақытын үдетуге мүмкіндік беретін компоненттердің әртүрлі пайыздық қатынасуында оңтайлы қоспа құрамын таңдау сипатталған. Бұл жұмыста авторлар құрамында әртүрлі пайыздық қатынаста сілті (каустикалық сода NaOH), этил спирт өндірісінің қалдығы (спирттік кейінгі барда) және қатуды реттегіш (гипс) бар кешенді модифицирленген қоспаны қолданды. Ұстасу мерзімінің өзгеруіне қоспаның әсер ететіндігіне салыстырмалы зерттеу жасалды. Талдау негізінде оңтайлы мөлшердегі қоспа бақылаумен салыстырғанда белгілі бір шекте ұстасу мерзімінің өзгеруіне алып келеді деуге болады. Кешенді модификацияланған қоспа құрамында цемент қамырының қатаюы мен ұстасуы, гидратация үрдістеріне әсер ететін жақсы үйлесімді тетіктері бар, оларды толықтырады және ондағы әрбір ингредиенттің әрекетін күшейтеді. Цемент қамырында кешенді модифицикацияланған қоспа мөлшерін арттыру сұйылту үрдістеріне әсер етіп қана қоймай, сондай-ақ цемент массасының қатаюы мен ұстасу мерзімінің қысқаруына алып келеді.

Мақала келді: *22 ақпан 2022*
Сараптамадан өтті: *18 сәуір 2022*
Қабылданды: *22 тамыз 2022*

Түйін сөздер: цемент қоспасы, қатуды үдеткіш, спирттік кейінгі барда, кешенді модификацияланған қоспа, ұстасу мерзімі.

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Влияние комплексной модифицированной добавки на сроки схватывания цементной смеси

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АННОТАЦИЯ

В статье приведены результаты влияния комплексной модифицированной добавки на сроки схватывания цементного теста. В работе описана методика определения сроков схватывания теста стандартной консистенции, подбор оптимального состава добавки при разном процентном соотношении его компонентов, позволяющие ускорить время схватывания. В данной работе авторы использовали комплексную модифицированную добавку, включающий в своем составе щелочь (каустическая сода NaOH), послеспиртовую барду (отходы спиртового производства) и регультор твердения (гипс) в разных процентных соотношениях. Выполнено сравнительное исследование влияния добавки на изменение времени схватывания. Анализ дает основание утверждать, что добавка в оптимальном количестве приводит к изменениям времени схватывания по сравнению с эталонным образцом, но в пределах норм. Показано, что совместное использование в составе комплексной модифицированной добавки, обладающей хорошо совместимыми механизмами их влияния на процессы гидратации, схватывания и твердения цементной массы, взаимно дополняет и усиливает действие каждого ингредиента добавки. Увелечение концентрации комплексной модифицированной добавки в цементную смесь не только воздействует на процессы разжижения, а также на сокращения времени схватывания и твердения цементной массы.

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