Features of sulfate and carbonate activation of plasticized fly-ash cement binder systems

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Abstract. The article is devoted to the study of the features of sulfate and carbonate activation of plasticized fly ash-cement binder systems

In the course of research, the principles of composite build of plasticized ash-filled binding compositions containing 20-40% of OPC and coal by-product by increased operational characteristics have been proven. It is shown that the use of complex organo-mineral additives in fly ash-cement compositions, which contain a mixture of carbonate and sulfate compounds as a mineral component, and a plasticizing additive of the polycarboxylate type as an organic component, makes it possible to obtain an artificial stone that is not inferior in its properties to a stone obtained on the basis of additive-free cement type I.

The peculiarities of the synthesis of the strength of artificial stone based on fly ash-cement compositions modified with an organo-mineral additive containing carbonate and sulfate mineral components were studied. It is shown that when only carbonate additives are used for modification, the formation of low-base calcium hydrosilicates takes place on its surface, and when sulfate and carbonate additives are simultaneously introduced, solid solutions based on ettringite and its carbonate analogue prevail among neoplasms.

It has been confirmed that during the hydration of fly ash-cement binding systems activated by the addition of burnt gypsum stone, the greatest effect associated with the increase in strength at all stages of hardening is achieved when the additive is used in the amount of 10%. Modification of sand-cement compositions with a sulfate additive causes an increase in the strength of artificial stone at the age of: 2 days by 14.24; 25.0 and 35.47%; 7 days at 83.18; 43.6 and 40.6%; 28 days for 75.5; 41.6 and 31.5%, respectively, when used in the binder composition of cement 20; 30 and 40%.

Modification of fly ash-cement compositions with a carbonate component causes an increase in the strength of artificial stone at the age of: 2 days by 18.6-20%; 7 days by 105.12-45.3%; 28 days by 86.6-30.0%, respectively. With a consumption of 40% OPC, the optimal amount of carbonate additive is 9% and causes an increase in the strength of artificial stone at the age of: 2 days by 33.3%; 7 days by 56.63%; 28 days for 43.5%.

Keywords: fly-ash, sulfate activation, carbonate activation, modification, binders.

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INTRODUCTION

The construction industry needs the use of new highly effective materials, among which high-quality cement and products based on it occupy an important place. At the same time, the cement industry is one of the largest consumers of natural raw materials, fuel and electricity, and the production of cement is accompanied by known environmental problems associated with high energy costs and emissions of toxic substances into the atmosphere [1,2].

One of the important problems in the production of concrete and reinforced concrete products is the saving of cement when obtaining high-quality materials. There are many methods that allow in specific conditions to reduce the specific consumption of cement without deteriorating the technical properties of concrete. With the goal of saving fuel and energy resources in developed countries, the share of multicomponent cements is constantly increasing, the use of which allows not only to save fuel and electricity, but also to obtain concrete with improved operational characteristics, due to the introduction of active minerals additives and fillers [3-5].

The high degree of dispersity and appropriate chemical composition of fly ash make it one of the most effective mineral additives to OPC. From an economic point of view, the advantage is its prevalence, due to the presence of thermal power plants in all regions of the country, and therefore its transportation does not require large costs.

Successful attempts at the rational use of ash-carrying out TPP of dry selection in concrete technology to replace an equivalent part of cement have been known since the beginning of the century. The practice of concrete works confirms that such a replacement, in addition to saving cement, also improves the properties of the concrete mixture and concrete [6-10].

The requirements for ash as an active mineral additive are determined by the physical-chemical mechanism of their influence on the processes of hardening and structuring of concrete. In the concrete mixture, ash performs the role of not only a micro-filler, while increasing the total amount of binder and improving the granulometric composition of sand, but also an active mineral additive that positively affects the processes of cement stone structure [11-13].

Physical-chemical features of structure formation in artificial stone based on OPC are significantly related to its interaction with the external environment. It depends on the stability of the primary new formations, which are mainly represented by hydrosilicates and hydroaluminates (hydrosulfoaluminates), as well as calcium hydroxide. The change in the stability of primary hydrates and the formation due to recrystallization of new products modified by the components of the external environment initiates the adaptive capabilities of artificial stone and creates prerequisites for the structural and functional adaptation of concrete structures made on its basis.

An effective way to obtain materials capable of adaptation in the early stages of hardening is the use of composite cements and the introduction of additives-modifiers, which not only change the quantitative and qualitative composition of the products of structure formation, but also cause directed recrystallization of new formations cement stone, contributing to the formation of "modified hydrates" in its structure, which are more resistant to environmental influences compared to traditional hydration products [14-17].

Generalization of the considered information allows us to conclude about the expediency of using sulfate and carbonate additives as modifiers in order to increase the physical and mechanical properties of binder systems and concretes based on plasticized fly ash-cement binders. However, their use is limited by different views on the type of these additives and the instability of hydrosulfoaluminate phases during operation [18-20].

PURPOSE AND METHODS

The purpose of the work is to increase the effectiveness of the modification of plasticized fly ash-cement compositions with complex mineral additives containing carbonate and sulfate compounds,

To achieve the set goal, it is necessary to solve the following problems:

- determine the influence of sulfate and carbonate additives on the hardening processes of plasticized fly ash-cement compositions;
- to investigate the rate of synthesis of new formations in the early stages of hydration, depending on the type and amount of the additive used;

- to study the possibilities of complex modification of plasticized fly ash-cement compositions with sulfate-carbonate additives;
- to investigate the influence of the phase composition of neoplasms on the stability of the physical and mechanical characteristics of artificial stone.

The activity of binders was studied on beam samples with dimensions of 4x4x16 cm, made from a cement-sand solution based on the developed compositions of binders and sand in a ratio of 1:3, mixed with a mixing liquid represented by water and C-3 superplasticizer (TU 6-14-625) in the amount of 1.0% by mass of cement, to obtain a mixture of the necessary consistency (at V/C=0.39, pour a standard cone of at least 135 mm DSTU B V.2.7–46:2010).

In order to identify the synthesized new formations and establish the features of structure formation in ash-based compositions, the study of the phase composition of low-clinker ash-cement binders modified with sulfate and carbonate additives was performed using X-ray phase, electron-microscopic, and differential-thermal methods analysis

RESULTS AND EXPLANATIONS

For the development of conception of low energy consumption low-clinker binding systems with the high strength gain, as a basic system were used fly ash-cement systems, containing 20…40 % by mass of OPC and 60…80 % by mass of fly ash. Strength gain was determined for cement paste for compositions, containing 60…80 % by mass of fly ash (Fig. 1).

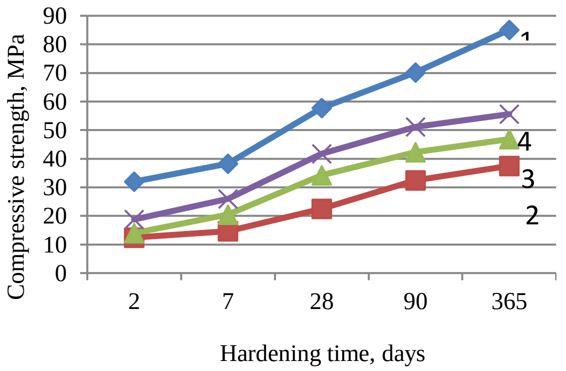


Figure 1. Strength gain of plasticized fly ash-cement compositions, containing 20% (2), 30% (3) and 40% (4) of OPC, and corresponding composition – additive free OPC (1)

The strength of such compositions is ensured due to the hydration of cement clinker minerals in the early stages of hardening and due to the synthesis in the hydration products of an additional amount of low-base calcium hydrosilicates in the late stages of hardening. This is confirmed by the data and indicates the need for additional chemical activation of ash.

The speed of increasing the strength of fly ash-cement compositions can be regulated by mechanical, chemical and thermal technological methods. However, the use of the technological techniques given separately does not allow to obtain compositions that would be characterized by the kinetics of strength growth close to the strength growth of type I OPCs in the early stages of hydration.

Taking into account the chemical and mineralogical composition of the ash, it can be assumed that its chemical activation is most appropriate with sulfate and carbonate compounds, and it is desirable to introduce mineral active components together with organic plasticizing additives, the combined action of which provides the best conditions for the hydration process and creates the necessary conditions for synthesis strength of artificial stone.

As is known, the rate of increase in strength of fly ash-cement compositions can be adjusted by choosing the amount of sulfate additive. The use of plasticized fly ash-cement systems, modified with anhydrite, obtained during the firing of gypsum stone, allows the synthesis of binding compositions, which in their physical and mechanical properties differ little from the properties of OPC stone.

Calcium sulfate was added to the plasticized fly ash-cement binding compositions in the amount of 6, 8, and 10% by mass to replace ash. The research was carried out on cube samples with a rib size of 2x2x2 cm, made on the basis of cement dough of normal density. Binder compositions were prepared by simultaneous grinding of the starting materials to a specific surface area of S=450...500 m^2/kg. In order to reduce water consumption, improve ease of installation, and increase strength, C-3 superplasticizer was introduced into the system in an amount of 0.75% by mass from the mass in caustic substance. The choice of plasticizer is due to the fact that in systems containing a sulfate component, plasticizers containing a sulfate group have a more pronounced positive effect.

The kinetics of increasing the strength of the artificial stone obtained on the basis of plasticized fly ash-cement compositions modified with sulfate additive in different quantities with different content of OPC was studied (Fig. 2) and the phase composition of neoplasms was investigated using X-ray phase analysis and SEM (Fig. 3; Fig. 4).

The analysis of graphic dependences of changes in the strength of artificial stone based on plasticized fly ash-cement compositions modified with a sulfate additive shows the positive effect of calcium sulfate on the increase in strength both at the early and late stages of hydration.

According to the research results, increasing the amount of the modifying additive in the plasticized fly ash-cement composition, regardless of the content of OPC, helps to increase the strength characteristics of the obtained samples at all stages of hydration, obviously due to the synthesis of hydrosulfoaluminate phases.

At the same time, on the 2nd day of hydration, the increase in the compressive strength of the artificial stone ranges from 4-14.2%, 7.14-25.0% and 28.0-35.0% with the introduction of: 20%, 30% and 40% OPC, compared to the additive-free system (Fig. 2). At the design age (28 days), the increase in strength ranges from 66.2-75.5%, 32.8-41.6% and 17.7-31.57% for compositions containing 20%, 30% and 40% OPC.

A comparison of the obtained data allows us to note that the strength of the compositions modified with calcium sulfate in the amount of 10% after 28 days of hardening is 39.8, 48.5 and 55 MPa, respectively, when using 20, 30 and 40% OPC, which is close to the strength of pure OPC – 57.75 MPa. The synthesis of artificial stone strength, according to X-ray phase analysis data, is ensured by the formation of ettringite hydration products (d=0.992; 0.498; 0.277; 0.193 nm) and low-base calcium hydrosilicates (d=0.307; 0.21; 0.183; 0.162; 0.156 nm)) (Fig. 3).

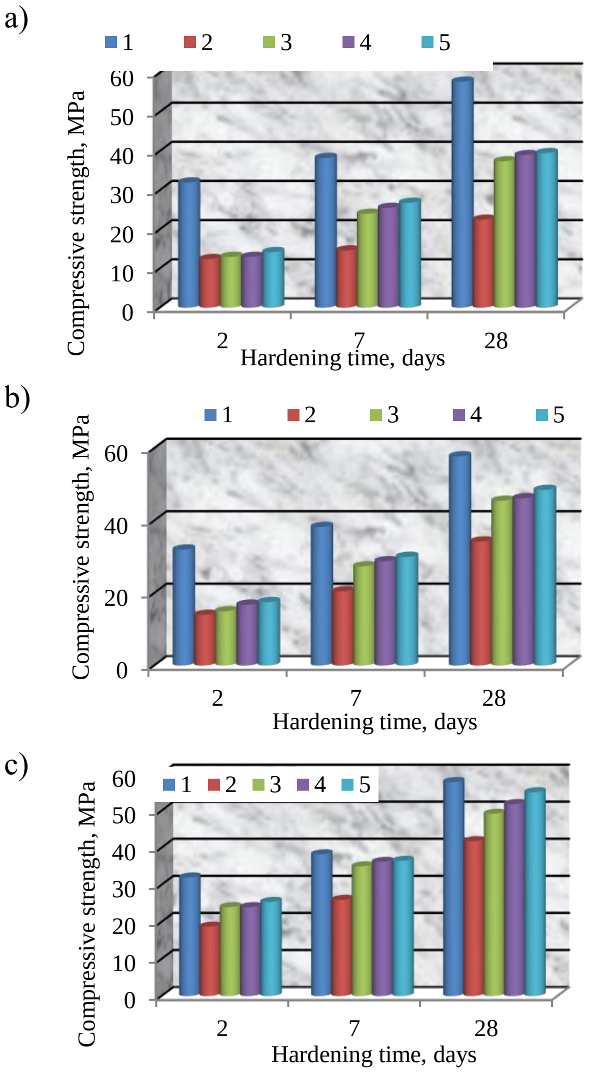


Figure 2. Strength gain of the pure OPC specimens (1) and compositions, containing OPC and fly ash without admixtures (2) and with CaSO4 admixture in quantity: 6% (3), 8% (4), 10% (5), containing 20% (a), 30% (b), 40% (c) of OPC.

The formation of ettringite crystals according to SEM is clearly visible in photographs of artificial stone modified with calcium sulfate at the early stages of hydration (Fig. 3.c).

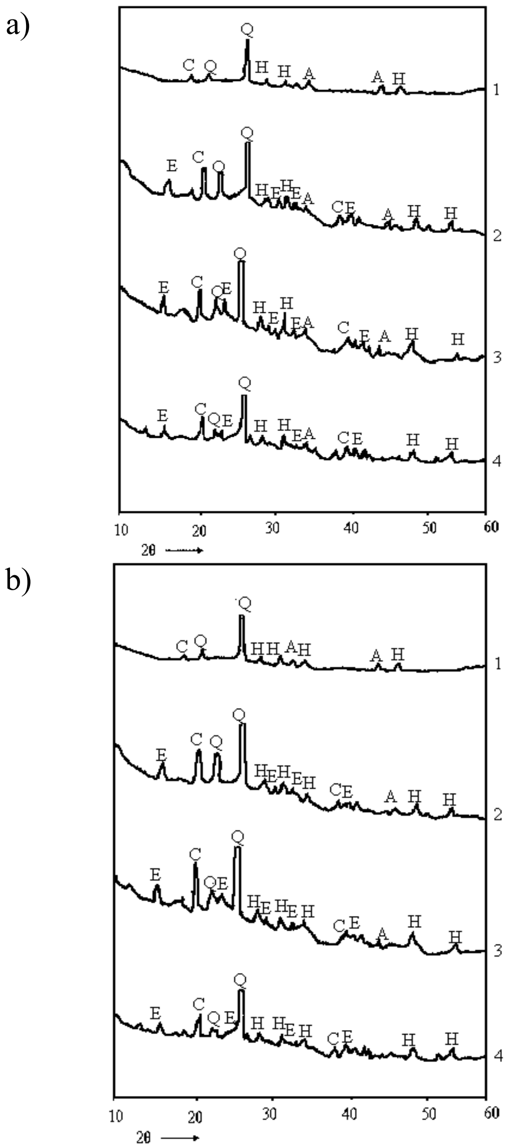


Figure 3. X-ray patterns of artificial stone of plasticized fly ash-cement compositions, containing 30 % of OPC and 70% of fly ash (1) and plasticized fly ash-cement compositions, modified by calcium sulfate admixture, burned at 450ºC in the quantity 6% (2), 8% (3) and 10% (4) after hardening during: 7 (a) and 28 (b) days. Marking: C – portlandite, A – calcium hydro aluminate, H – calcium hydro silicate, Q – b-quartz, E – ettringite

Analysis of the distribution of elements on the surface of a chip of cement stone modified with calcium sulfate (Fig. 4 d) shows that, along with ettringite, calcium hydrosulfoferite, portlandite, and calcium hydrosilicates are also present in the composition of hardening products. According to the chemical composition and mass content of the elements, the observed neoplasms correspond to ettringite, the appearance of a certain amount of iron may indicate the formation of solid solutions of ettringite with calcium sulfoferite.

Thus, sulfate activation of plasticized flty ash-cement binding systems allows to increase the strength of artificial stone by 4.5-35% in the early stages of hardening, compared to compositions without additives, due to the synthesis of ettringite (3-7 days) and by 17.7-75 % after 28 days of hydration (due to the additional formation of calcium hydrosilicates), but the issue of maintaining the effectiveness of the additive at late stages of hardening of artificial stone. According to the data, there is a danger of recrystallization of ettringite into the monosulfate form or the formation of secondary ettringite in such systems, which in turn may be the cause of the development of destructive processes and loss of strength of the artificial stone.

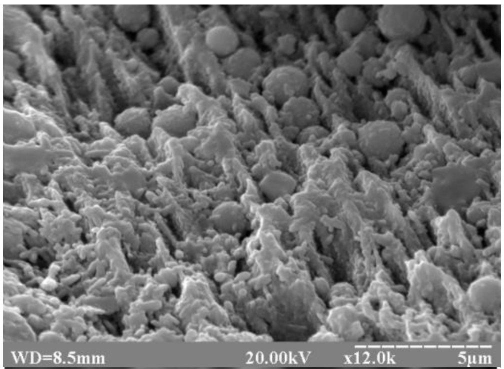
Therefore, the introduction of sulfate additives into the composition of plasticized fly ash-cement binders contributes to the synthesis of an additional amount of low-basic hydrosilicates on the surface of ettringite crystals.

It is known that finely dispersed carbonate additives play an important structure-forming role in the formation of cement stone. This is due to the chemical interaction of calcium and magnesium carbonates with the hydration products of aluminum-containing phases of clinker, as a result of which crystal hydrates are formed, which have a positive effect on the construction and technological properties of cement stone.

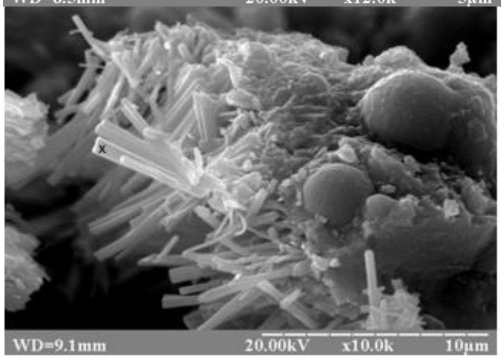
When $CaCO_3$ interacts with the aluminate component of OPC clinker, the formation of monohydrocarbo-aluminate and hexagonal calcium hydro-aluminates takes place.

Research on the possibility of activation of hydration processes and strength synthesis of plasticized fly ash-cement compositions with carbonate additives, performed using finely ground chalk in different quantities. The quality of cements obtained with the addition of finely ground carbonate components mainly depends on the quantitative ratio of clinker and additives, their specific surface, the degree of particle dispersion and their uniform distribution in the mixture.

a)



c)



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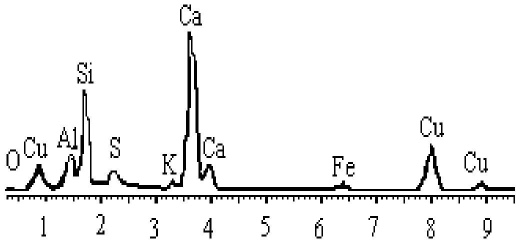

d)

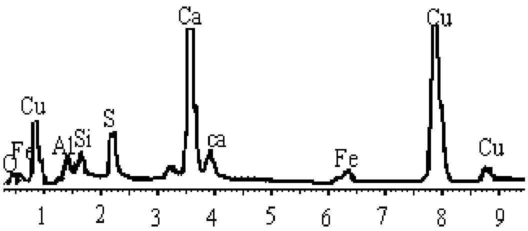

Figure 4. Micro photos of surface of artificial stone crack (a, c) and results of micro analysis of exact phases (b, d) on the basis of fly ash-cement composition, containing 30% of OPC without sulfate component (a), and modified by calcium sulfate, burned at 450°C, in the quantity 10% (c), after 7 days of hardening in normal conditions.

The carbonate additive was used in amounts of 3, 6, and 9% to replace fly ash at different cement consumption rates of 20, 30, and 40%. The effectiveness of the application of modifying additives was studied according to the criteria of compressive strength of cube samples measuring 2x2x2 cm. Binding compositions were prepared by simultaneous grinding of the starting materials to a specific surface area of S=450-500 m^2/kg. To reduce water consumption, improve ease of installation and increase strength, C-3 superplasticizer was introduced into the system in the amount of 0.75% of the mass of the binder.

The analysis of graphical dependencies (Fig. 5) of changes in the strength of the developed binders shows that the use of $CaCO_3$ for the modification of plasticized fly ash-cement compositions containing 20-40% OPC helps to increase the strength of the modified compositions at all stages of hardening. Therefore, the introduction of a carbonate additive at the early stages of hardening allows to increase the strength of modified binders on the 2nd day by 10.6-18.6%, 20-32.1% and 26.6-33.3% and - on the 7th day by 87.9-105%, 24.8-45.4% and 40.9...56.4, respectively, with a cement consumption of 20, 30 and 40%.

On the 28th day of hydration, with an increase in the amount of carbonate additive, the strength of the artificial stone increases by 52-88.8%, 24-32.8% and 20-43.5% with the corresponding cement consumption - 20, 30 and 40%. Moreover, it should be noted that with a consumption of 20% of OPC, compositions containing 6% of the carbonate component have the highest strength (R_{2d} – 14.75 MPa, R_{7d} – 29.8 MPa, R_{28d} – 41.75 MPa); when using 30-40% OPC, the greatest strength is achieved when using 9% $CaCO_3$ (R_{2d} – 18.5 MPa, R_{28d} – 45.5) and (R_{2d} – 25.0 MPa, R_{7d} – 40.6 MPa, R_{28d} – 60.0MPa) in accordance.

The analysis of the obtained compressive strength results of cement dough samples based on plasticized fly ash-cement compositions modified with a carbonate additive allows us to note the positive effect of $CaCO_3$ on the increase in strength at all stages of hardening compared to the additive-free ash-filled system. However, in the early stages of hardening (2-7 days), the carbonate additive more effectively increases the strength characteristics of artificial stone when it is used in the amount of 3-6%, while after 28 days of hardening, compositions modified with the maximum amount of $CaCO_3$ - 9% are characterized by higher strength indicators.

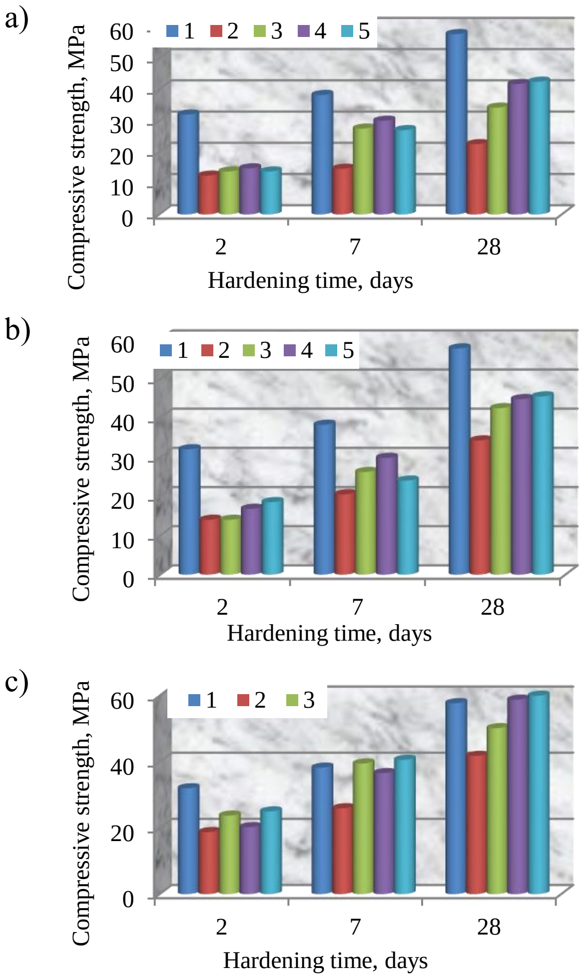

Figure 5. Strength gain of the specimens on the pure OPC basis (1) and plasticized fly ash cement compositions without admixtures (2) and with $CaCO_3$ admixture in the quantity: 3% (3), 6 % (4), 9% (5) containing 20% (a), 30% (b) and 40% (c) of OPC.

Finely dispersed carbonate additives play an important structure-forming role in the formation of cement stone, which is related to the chemical interaction of calcium carbonates with the hydration products of aluminum-containing phases of clinker, and also have a positive effect on the construction and technical properties of the obtained artificial stone due to the stability of the formed crystal hydrate phases.

The synthesis of the strength of the artificial tone obtained on the basis of modified fly ash-cement compositions containing 20-40% of OPC in the early stages of hardening (2-7 days) is ensured due to the hydration of OPC clinker minerals and the formation of portlandite in the composition of hardening products (0.263; 0.193; 0.179; 0.169 nm), ettringite (d=0.977; 0.561; 0.495; 0.3883; 0.2779; 0.1929 nm) and low basic calcium hydrosilicates (d=0.307; 0.2094; 0.1825; 0.162; nm), intensively crystallized on calcite grains (d=0.3042; 0.228; 0.191; 0.1605 nm) (Fig. 6a). Moreover, in the compositions modified with calcium carbonate in the amount of 6-9 wt.%, the intensity of the characteristic peak's characteristic of ettringite increases (Fig. 6a) (3), (4)). This phenomenon can be explained by the formation of carbonate-containing ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaCO_3{\cdot}32H_2O$ (d = 0.2702; 0.2501 nm).

At the late stages of hardening (28 days), the increase in strength indicators occurs mainly due to the additional formation of calcium hydrosilicates, which are synthesized as a result of the interaction of fly ash and portlandite as a result of the pozzolanic reaction, which is evidenced by a decrease in the intensity of the $Ca(OH)_2$ peaks (0.263; 0.193 ; 0.179 nm) (Fig. 6 b) and an increase in the intensity of the peaks characteristic of low basic calcium hydrosilicates of CSH(I) type (d=0.307; 0.2094; 0.1825; 0.162; nm). Also, the increase in strength can be explained due to the interaction of calcium carbonate with ettringite and the subsequent formation of solid solutions in the system $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$ - $3CaO{\cdot}Al_2O_3{\cdot}$and $3CaCO_3{\cdot}32H_2O$.

The interaction of calcium carbonate with hydration products of modified fly ash-cement binders can be evidenced by a decrease in the intensity of the peak's characteristic of $CaCO_3$ (0.30429; 0.2289; 0.1912; 0.1875; 0.1605 nm) after 28 days of hydration.

Synthesis of calcium hydro silicates on the surface of calcium carbonate after 7 days of hardening is confirmed by the SEM results (Fig.7 a, c) and microanalysis (Fig.7 b, d).

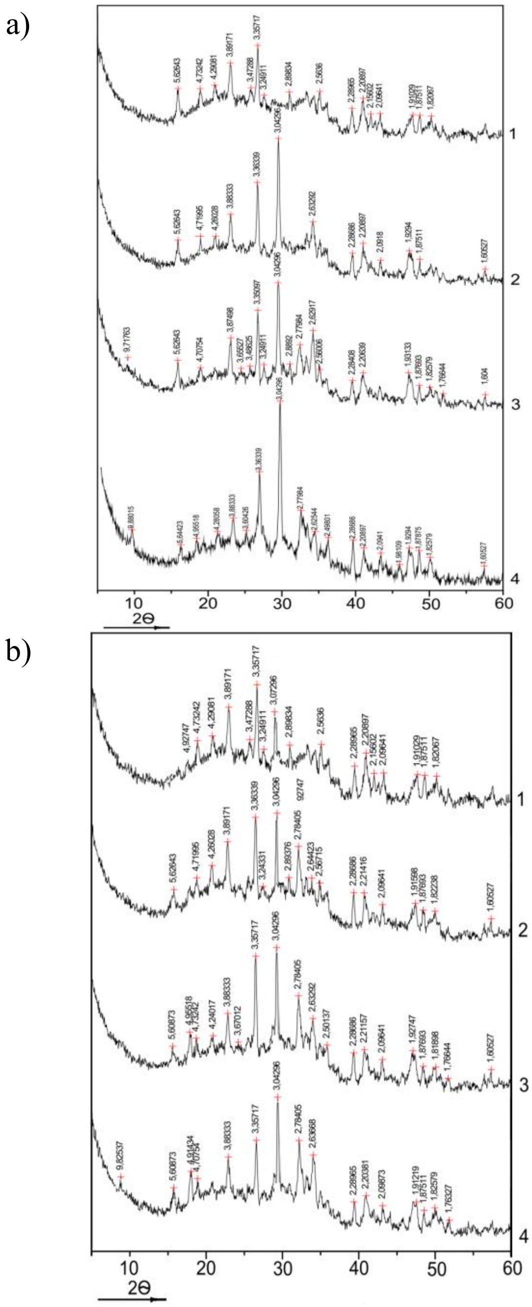

Figure 6. X-rays patterns of artificial stone of plasticized fly ash-cement binders without admixtures, containing 30% of OPC and 70% of fly ash (1) and plasticized fly ash-cement compositions, modified by calcium carbonate admixture in quantity 3% (2), 6% (3) and 9% (4) after hardening during: 7 (a) and 28 (b) days.

Chemical analysis of the matter, filling the space between fly ash spheres (Fig.7 a, b), confirms data obtained by X-ray analysis and witnesses about formation in the scope of hydration products in fly ash-cement composition without carbonate component – calcium hydro silicates. Analysis of elements distribution on the crack surface of artificial stone of the specimen, based on composition, modified by calcium carbonate (Fig.7 d) makes it possible to admit that in hardening products composition are present calcium hydro silicates, synthesized on the surface of carbonate admixture (Fig.7 c).

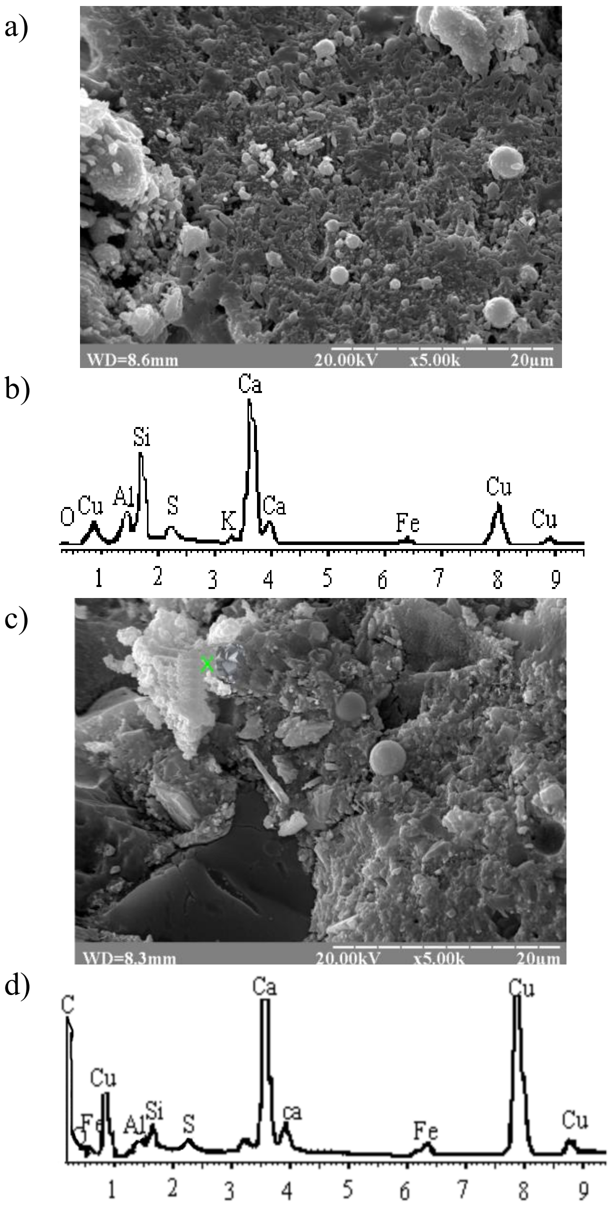

Figure 7. Micro photos of surface of artificial stone crack (a, c) and results of micro analysis of separate phases (b, d) on the basis of plasticized fly ash-cement composition containing 30% of OPC and 70% of fly ash without carbonate component (a, b) and composition, modified by calcium carbonate (c, d), in quantity 6%, after 7 days of hardening in normal conditions.

Increased strength properties of modified fly ash-cement compositions in project age could be explained by formation of solid solutions between $CaCO_3$ and AFt – phases, leading to the synthesis of hydrocarboaluminates with different composition. $CaCO_3$ is not a hydraulic admixture, but it activates hydration processes and hardening OPC compositions. Moreover, as a background for crystallization of hydrate new formations, $CaCO_3$ intensifies hydration and provides good adhesion between cement stone components. Thus, it was proved positive influence of calcium carbonate represented by chalk on processes of structure formation and synthesis of strength of artificial stone based on fly ash-cement compositions, containing 20, 30 and 40% of OPC at all stages of hardening. It should be admitted that in case of increasing OPC content in the binder positive influence of carbonate admixture is also rising, that is probably connected with formation of $CaCO_3(Ca(OH)_2)$-like compositions, leading to the intensifying of crystallization processes of low-basic calcium hydro silicates.

CONCLUSIONS

In the course of the conducted research, the physical-chemical regularities of obtaining low-clinker plasticized fly ash-cement binders modified with complex mineral sulfate-carbonate additives containing 50-65% of fly ash, which in terms of their construction and technological properties are not inferior to OPC without additives (type PC - I and PC - II).

It is shown that when sulfate additive is added to plasticized fly ash-cement compositions, the strength of the artificial stone is ensured by the synthesis of new formations of ettringite and low-base calcium hydrosilicates on its surface. The introduction of a carbonate additive to the specified compositions accelerates the formation of low-base calcium hydrosilicates. Moreover, the processes of synthesis of the latter in the presence of a carbonate component are accelerated with an increase in the amount of OPC in the binder.

It was established that during the hydration of the fly ash-cement binder system containing 30% of OPC and 70% of fly ash, activated with a carbonate additive, the greatest effect related to the increase in strength is achieved when using chalk in the amount of 6%, in particular for a 2-day increase strength is 20%, and at 28 - 30.8%. When modifying the specified binding composition with a sulfate additive, its optimal amount is 10% and contributes to the increase in strength after hardening for 2 days by 25%, and after 28 days - by 41.6%.

Taking into account the mechanism of action of $CaCO_3$, as well as the influence of the sulfate component on the processes of structure formation of cement stone, the combined use of such mineral additives in multi-component complex systems represented by composite binders will be more effective.

With the correct selection and combination of mineral components in the composition of OPC, their mutual activation and increase in strength occurs due to the manifestation of a synergistic effect with the simultaneous introduction of sulfate and carbonate additives.

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Особливості сульфатної та карбонатної активації пластифікованих золоцементних в'яжучих систем

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Анотація. Стаття присвячена дослідженню особливостей сульфатної та карбонатної активації пластифікованих золоцементних в'яжучих систем

В ході досліджень доведено принципи композиційної побудови пластифікованих золонаповнених в'яжучих композицій, що містять 20-40% портландцементу, та відрізняються підвищеними експлуатаційними характеристиками. Показано, що в складі золоцементних композицій використання комплексних органо-мінеральних добавок, які містять як мінеральну складову суміш карбонатних та сульфатних сполук, а як органічну – пластифікуючу добавку полікарбоксилатного типу, дозволяє отримувати штучний камінь, що не поступається своїми властивостями каменю отриманому на основі бездобавочного цементу типу I.

Досліджено особливості синтезу міцності штучного каменю на основі золоцементних композицій, модифікованих органо-мінеральною добавкою, що містить карбонатну та сульфатну мінеральну складову. Показано, що при використанні для модифікації тільки карбонатної добавки має місце утворення на її поверхні низькоосновних гідросилікатів кальцію, а при одночасному введенні

сульфатної та карбонатної добавки серед новоутворень переважають тверді розчини на основі етрингіту та його карбонатного аналогу.

Підтверджено, що при гідратації золоцементних в'яжучих систем, активованих добавкою випаленого гіпсового каменю, найбільший ефект, пов'язаний зі зростанням міцності на всіх етапах твердіння, досягається при використанні добавки в кількості 10%. Модифікація золоцементних композицій сульфатною добавкою обумовлює зростання міцності штучного каменю у віці: 2 діб на 14,24; 25,0 та 35,47%; 7 діб на 83,18; 43,6 та 40,6%; 28 діб на 75,5; 41,6 та 31,5% відповідно при використанні у складі в'яжучої композиції цементу 20; 30 та 40%.

Модифікація золоцементних композицій карбонатною складовою обумовлює зростання міцності штучного каменю у віці: 2 діб на 18,6-20%; 7 діб на 105,12-45,3%; 28 діб на 86,6-30,0% відповідно. При витраті портландцементу 40%, оптимальна кількість карбонатної добавки становить 9% і обумовлює зростання міцності штучного каменю у віці: 2 діб на 33,3%; 7 діб на 56,63%; 28 діб на 43,5%.

Ключові слова: зола-виносу, сульфатна активація, карбонатна активація, модифікація, в'яжучі речовини.