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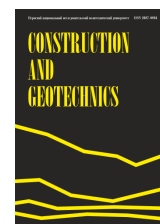


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THE UTILIZATION OF FLUOROANHYDRITE FOR DEVELOPMENT OF COMPOSITE GYPSUM BINDERS AND DRY CONSTRUCTION MIXTURES FOR SELF-LEVELING FLOORS

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ABSTRACT

The significant inventories of synthetic gypsum – the wastes of different productions are the promising raw material for building materials industry. In this work the waste of etching acid industry – fluoroanhydrite has been investigated. The microstructure of fluoroanhydrite binder in comparison of gypsum binder has been analyzed by scanning electron microscopy method. On the base of high-grade gypsum G-16 the composition of composite nonshrink gypsum-fluoroanhydrite binder contained 15 % of fluoroanhydrite has been selected. The mechanical properties and microstructure of gypsum-fluoroanhydrite binder that had been mechanochemically activated using such activators as fly ash, Portland cement and manganese sulfate have been explored. The activation of binder had allowed to increase its compressive strength by 46 % and to provide the value of softening coefficient no less than 0.75. On the base of activated gypsum-fluoroanhydrite binder the composition of dry mixture for self-levelling floors was selected using methods of mathematical experimental design. The optimal dosage of admixtures of hyperplasticizer, defoaming agent and setting time retarder had been found. The developed composition is nonsink and has high strength and adhesion to concrete basement and also it is not inferior in quality to dry construction mixtures for self-levelling floors presented at the modern construction market.

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ПРИМЕНЕНИЕ ФТОРАНГИДРИТА ДЛЯ РАЗРАБОТКИ КОМПОЗИЦИОННЫХ ГИПСОВЫХ ВЯЖУЩИХ И СУХИХ СТРОИТЕЛЬНЫХ СМЕСЕЙ ДЛЯ САМОНИВЕЛИРУЮЩИХСЯ ПОЛОВ

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АННОТАЦИЯ

Значительные запасы синтетического гипса – отходов различных производств – являются перспективным сырьем для промышленности строительных материалов. В данной работе исследовался фторангидрит – отход производства плавиковой кислоты. С помощью метода электронной микроскопии была проанализирована микроструктура фторангидритового вяжущего в сравнении со строительным гипсом. На основе высокопрочного гипса марки Г-16 был подобран состав композиционного безусадочного гипсофторангидритового вяжущего, содержащего 15 % фторангидрита. Были также исследованы механические свойства и микроструктура гипсофторангидритового вяжущего, механически и химически активированного с использованием таких активаторов, как зола-унос, портландцемент и сульфат марганца. Активация вяжущего позволила увеличить прочность на сжатие на 46 % и обеспечить значение коэффициента размягчения не менее 0,75. На основе активированного гипсофторангидритового вяжущего был подобран состав сухой строительной смеси для самонивелирующихся полов с помощью метода математического планирования эксперимента. Были определены оптимальные дозировки добавок гиперпластификатора, пеногасителя и замедлителя схватывания. Разработанный состав является безусадочным и обладает высокой прочностью и адгезией к бетонному основанию, а также не уступает по качеству сухим строительным смесям для самонивелирующихся полов, представленным на современном строительном рынке.

Introduction

Building materials produced on the base of gypsum binder flood construction market more and more. The reason of this is high efficiency of their application for repair and finishing works, combination of cost and environmental performance, possibility of cooperative use with different ingredients for obtaining of composite materials and products.

The main volume of products produced from gypsum binder is dry construction mixture (DCM), plasterboard and gypsum blocks. Due to the good technical and technological equipment of modern factory specialized in production of this kind of product the main problems, which are had to solve, are concerns related to resource conservation and cost reduction of products. The most popular and justified variant including from the point of view of technology is one of solve of this concern through application of man-made waste of different trade in formulation of composite building material.

This technological decision particularly in recent decade is been the most widespread in trade of building materials and products that is verified by the results of numerous investigations [1–6].

Application of man-made origin components as part of gypsum building materials allows not only to decrease prime cost but to adjust initial properties of raw material, to decrease its consumption, to impart required service properties for product and also to recover industrial waste rationally. The one of the kinds of man-made raw material is a synthetic gypsum.

According to literature data [7] more than 150 million tons of synthetic gypsum are annually generated in the world, 1/10 part of which is fluorogypsum, titanogypsum and other, but the most part is fluoroanhydrite and phosphogypsum. The last trends demonstrate annual growth of this type of industrial wastes by 7 % and more.

Fluoroanhydrite (FA) – the waste of hydrofluoric acid industry, which consists from anhydrous calcium sulfate, has huge potential for application in construction. FA is formed as the result of reaction of fluorite with 98 % sulfuric acid: $\text{CaF}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{CaSO}_4 + 2\text{HF}$. In this case impurity composition of waste looks like this: CaSO_4 – 82–98.4 %, CaF_2 – 0.6–3.0 %, H_2SO_4 – 1–15 %, there is no hydrofluoric acid (HF). There are significant reserves of this material. For instance, only in Perm krai of Russia more than 300k tons of FA is annually formed in JSC “HaloPolymer” [8].

For further application of FA in building materials industry it is made neutralization of harmful substances of waste with alkaline agent (limestone) followed by grinding the product to granules of no more than 1.25 mm in size. Temperature interval for the neutralization stage is from 150 to 220 °C [9].

It is established by Russian and foreign researchers that FA can be effectively applied for production of composite binders, pigment, inhibitor of Portland cement setting times and also in technology of Portland cement clinker, gypsum blocks, plasterboards and in the composition of DCM [10–12]. One of the most demanded and material-intensive production spheres from the point of view consumption of raw materials in particular the man-made waste is sphere of DCM.

Particularly, the promising direction of application of FA with as maximum execution of its functional possibilities as possible is production of nonshrink composite binders and products on the base of it [13], for instance, DCM for self-levelling floors.

A lot of scientific works are dedicated to research of application of different types of synthetic gypsum in the composition of self-levelling floor dry mixes. For example, the authors of article [14] obtained the composition of self-levelling mortar on the base of Portland cement and sulfoaluminate cement with 45 % of titanium gypsum. Also, researchers [15] used phosphorus gypsum in the amount of 40 to 50 wt% along with sulfoaluminate cement and Portland cement to applicate self-levelling mixes. Meanwhile, it was established that phosphorus gypsum acted not only as a filler but also participated in the Portland cement hydration reactions. The authors of research [16] developed the composition for self-levelling floors on the base of phosphorus gypsum. As compositions of mixes on the base of phosphorus gypsum have low early strength and lose required workability rapidly, in this work sulfoaluminate cement, finely ground granulated blast furnace slag and Portland cement as alkali activator of slag are added in the composition of mixture for solution of this concern. As a result, the mixture composition with 72 % of phosphorus gypsum met the requirement of JC/T1023-2007 was obtained. In the research [17] anhydrite-II obtained as the result of phosphorus gypsum calcination at 750 °C was used as the base of binder for self-levelling mortar in the amount of 43 %. In this case the complex activator of anhydrite hydration consisted of steel slag prepared with sulphuric acid, gypsum β -hemihydrate and sulfoaluminate cement was used. In the work [18] the researchers optimized the composition of self-levelling mortar on the base of FA (40 % of mortar mixture mass) where potassium sulfate was applied as hydration accelerator.

As we can see, in the existed works on the use of synthetic gypsum in the composition of self-levelling floor mixtures the high price activator – sulfoaluminate cement is generally used for binder activation, that does not allow to get the quite available product. However, as for FA activation as shown by the results of researches of Russian and foreign scientists [19] others effective technics of anhydrite activation and increase of its physical and mechanical properties exist. The mechanical and chemical activation methods are applied to increase the hydraulic activity of insoluble anhydrite [9].

The mechanical activation means the increase of activity of FA by increasing of its specific surface but the chemical one – by addition of chemical hardening accelerator (activator) in the composition of FA binder. The last method is more widespread, perspective and economically

profitable. However, the more effective method is combined approach included both mechanical and chemical technic of FA activation [20].

Depending on chemical composition of hardening accelerator there are following methods of chemical activation of FA [21–23]:

- sulphate activation (sulfates of alkali, heavy and acidic metals: Na_2SO_4 , K_2SO_4 , FeSO_4 and others);
- alkali activation ($\text{Ca}(\text{OH})_2$, NaOH);
- alkali activation including application of active mineral admixtures (Portland cement clinker, blast furnace slag, slag Portland cement and others);
- combined activation (the combination of hardening accelerators mentioned above).

Moreover, to increase effect of reaction between FA binder and Portland cement and to provide formation of dense and water-resistant structure the application of effective superplasticizers in the composition of raw mixture can be applied.

Thus, the aim of present work consists in research of processes of composite gypsum-fluoroanhydrite (GFA) binder hardening and structurization of composite binders produced on the base of it with complex activation of FA and also in development of composition of DCM for self-levelling floors and methods of formation of predetermined properties of self-levelling floorings.

Materials and methods

The following binder materials were used in this research to get composite GFA binder and DCM for self-levelling floors. Grounded FA – the waste of etching acid production from JSC “HaloPolymer” met requirements of TU 5744-132-05807960 is light grey powder consisted of 1-100 μm particles with specific surface area 2000 cm^2/g and with bulk density in dry condition – 850 kg/m^3 . FA consists of very fine secondary agglomerated primary crystals of calcium sulfate. In the production the FA was neutralized to $\text{pH} = 8$ by dry neutralization method by cooperative grinding of FA granules with carbonate powder in the ball mill.

The building gypsum G-5 and G-16 grades produced by JSC “Prikamskaya gypsum company” according to GOST 125-2018 was used in this research.

Performance of applied binders is presented in Table 1.

The following components were used as hardening activators of FA:

- manganese sulfate (this type of salt was determined during previous research which is aimed to assessment of effectiveness of salt admixtures);
- acidic fly ash of Reftinskaya CHP;
- Portland cement CEM I 42.5 R from Gornozavodsk.

Also following materials were used as components of developed DCM:

- hyperplasticizer on the base of polycarboxylates;
- defoaming agent on the base of mixture of organosilicone polymers and hydrophobic ether;
- cellulose ether;
- setting time retarder on the base of citric acid.

Physical and mechanical properties of gypsum and anhydrite binder were determined according to GOST 23789 (see table 1). Physical and mechanical properties of DCM, fresh mortar and mortar were determined according to methods presented in current Russian standards: GOST 8735, GOST 10181, GOST 31356, GOST 5802, GOST 23789, GOST 33699, GOST R 56387 and GOST 24544.

Table 1

Performances and binder properties of neutralized FA,
 building (G-5) and high-grade (G-16) gypsum

Таблица 1

Характеристики и вяжущие свойства нейтрализованного фторангидрита,
 строительного (Г-5) и высокопрочного (Г-16) гипса

Performances	FA	G -16	G -5	Test method
Water requirement (W/G)	0.35	0.4	0.6	GOST 23789-2018, item 6
Setting times:				GOST 23789-2018, item 6
– initial	2.5 h	4.5 min	6 min	
– final	12 h	20 min	30 min	
Strength:				GOST 23789-2018, item 7
– compressive, MPa	7.1 (9)*	16	5	
– flexural, MPa	3.2 (4)*	6	2.5	
Fineness, sieve residue N02, %	0	III, ≤ 2	II, ≤ 14	GOST 23789-2018, item 5
Dilatation, %, no more	–	0.15	0.2	GOST 23789-2018, item 9

Remark: *Strength of FA specimens was assessed at 28 days. Strength of specimens cured in water bath is presented in brackets.

Microstructure of hardened binders was investigated by electron microscopy method with scanning electron microscope S-3400N.

Results and discussion

Comparison of binder capacity of FA and hemihydrate plaster

As results presented the FA is characterized by quite low activity, long-lasting setting times and hardening like others anhydrite binders. The process of FA hardening proceeds due to formation of intermediate fine-crystalline compound – $\text{CaSO}_4 \cdot 0.62\text{H}_2\text{O}$ from soluble γ -anhydrite and part of insoluble β -anhydrite [24]. The compound formed is partly recrystallized eventually. However, the most part of insoluble anhydrite remains unreacted even after 28 days of hardening. Dip of specimens in water allows to accelerate the hardening process insignificantly.

The analysis of microstructure of high-grade hardened gypsum and FA (fig. 1) presents that the main phase of high-grade hardened gypsum is crystals of gypsum dehydrate having an elongated prismatic hexagonal structure (form of “dovetail”). The structure of anhydrite stone is less homogeneous and it is presented by small amount of gypsum dehydrate crystals randomly located between plate hexagonal crystals $\text{CaSO}_4 \cdot 0.62\text{H}_2\text{O}$ and also by fine-crystalline unreacted anhydrite. Moreover, it is necessary to pay attention that process of FA hardening is accompanied by shrinkage deformations (up to 6 mm/m). These deformations often lead to decreasing of strength of specimens and crack formation. The hydration hardening of FA with slow formation of calcium sulfate dihydrate increasing in volume does not allow to solve this concern completely although the structure of the stone becomes denser and more uniform. However, this approach to provision of curing condition of FA binders does not allow to apply it in the production of many building materials including DCM.

Thus, the following conclusion may be formulated. To regulate structurization of FA is possible through intensification of mineral formation increasing the proportion of soluble compounds.

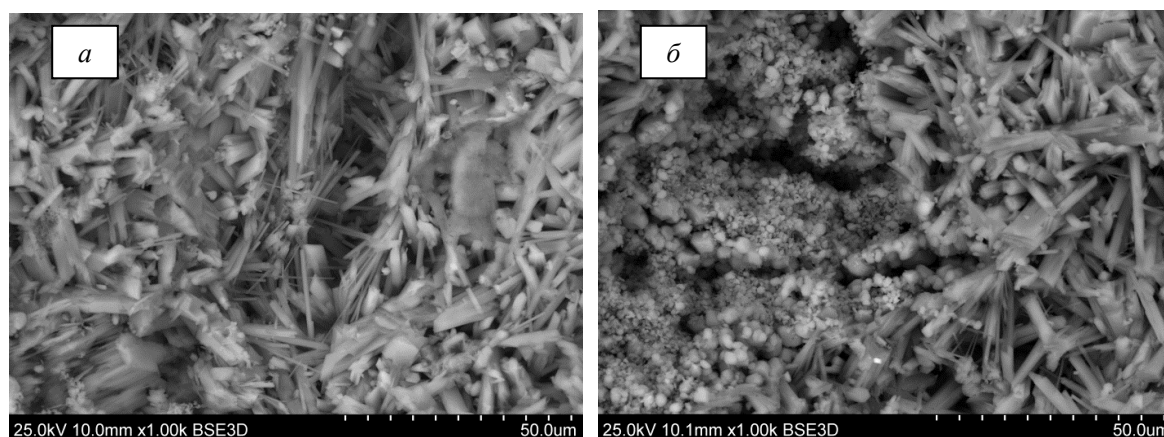


Fig. 1. The SEM images of structure of gypsum (a) and anhydrite (б) stone

Рис. 1. Структура гипсового (a) и ангидритового (б) камня

Investigation of composite GFA binder

At the next stage the initial assessment of possibility of FA application in composition of composite GFA binder on the base of high-grade gypsum G-16 was conducted. FA content in the composition varied from 0 to 100 %. The strength of specimens was assessment after 28 days. Results of tests are shown in table 2.

Table 2

Properties of composite GFA binder

Таблица 2

Свойства композиционного гипсофторангидритового вяжущего

No.	FA dosage, %	Normal consistency, %	Setting times, min		R_{comp} , MPa	R_{flex} , MPa	Deformations, mm/m
			initial	final			
1	0	0.4	4.5	20.0	16.1	6.2	+1.5
2	5	0.46	4.5	18.5	14.6	6.9	+1.4
3	10	0.46	5.5	16.0	13.9	6.8	+0.5
4	15	0.46	5.5	14.5	16.8	7.7	+0.3
5	20	0.43	6.5	13.0	11.4	5.2	+0.1
6	25	0.40	8.5	20.0	9.2	4.9	-1.0
7	50	0.37	10.0	15.0	8.7	4.5	-1.2
8	75	0.36	20.0	32.0	8.0	3.8	-1.3
9	100	0.34	150.0	1080.0	7.1	3.2	-6.0

Analyzing the test results, the following conclusion can be stated. The FA in a certain amount impacts on dynamic of setting and strength gain of composite binder, but its introduction into the mixture does not allow to correct significantly physical and mechanical properties of binder because of low activity.

The optimal dosage of FA for introduction to gypsum binder is 15 %. When using such an anhydrite dosage the strength increasing of test specimens is obtained. Besides, the shrinkage phenomena related to specificities of hardening of FA and Portland cement, which can also be in the composite binder formulation, can be compensated by the expansion deformations of this specimens. Thus, it is possible to control the properties of the resulting composite materials by varying the content of various modifications of calcium sulfate in the blended binder.

The analysis of strength gains dynamic variation and the results of electron microscopy of specimens prepared with blended GFA binder show that the increasing of specimen's strength occurs over

28 days. Meanwhile, the insoluble anhydrite dominates in the mineralogical composition of stone at the age of 3 days, the main strength is formed by hydration processes of plaster. The anhydrite reacts with water and transforms into the anhydrous calcium sulfate between 7 and 14 days of hardening. After 28 days the specimens are characterized by dense and less fragmented plate structure (fig. 2), while plaster stone has the fragmented needle and multidirectional crystalline structure. This fact explains the higher strength of GFA stone comparison with the reference specimens.

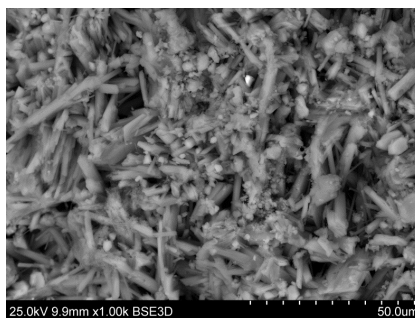


Fig. 2. The SEM image of structure of GFA binder stone

Рис. 2. Структура гипсофторангидритового камня

Efficiency assessment of comprehensive activation of composite GFA binder

Then, the efficiency assessment of complex activation of composite GFA binder containing 15 % of FA was made. To grind and mix FA, high-grade gypsum and also activation admixtures together with specific surface $S \geq 3000 \text{ cm}^2/\text{g}$ the laboratory ball mill was used.

According to conducted researches it was established the impact of following activation admixtures: manganese sulfate, fly ash and Portland cement on the formation of structure and properties of GFA binder, and also it was determined optimal dosage of this admixtures in terms of setting times of binder.

Thus, the addition of manganese sulfate in the composition of mixture insignificantly accelerates setting times both pure FA and composite gypsum binder on the base of high-grade gypsum and FA (the optimal dosage is 0.5 %) by increasing of solubility of calcium sulfates. Unstable intermediate compounds facilitating the hydration process are formed by reaction between manganese sulfate and calcium sulfate. The addition of this admixture provides more rapid crystallization of calcium sulfate from solution [25].

The analysis of microphotographs of the structure of the studied specimens at the different ages (1, 3, 7 and 28 days) was revealed that the amount of insoluble FA decreases, the crystalline $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is formed, the amount of $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ and $\text{CaSO}_4 \cdot 0.62\text{H}_2\text{O}$ minerals decreases and the mineral's structure becomes denser, more shaped and stable (fig. 3).

It is established that the structure of gypsum stone after 28 days without admixture contains mainly of needleshaped and fused into druses column crystals and also numerous of voids and nonhomogenities is obtained. The structure of composite obtained by mechanical activation and manganese sulfate addition is denser that is typically for strength structure of high-grade gypsum [26].

The investigations of hardening processes of complex GFA binder activated by fly ash revealed that the admixture has no significantly effect on setting times (the optimal dosage is 10 %)

and setting times of pure FA with the highest ash dosage accelerate by about 3 hours. In the composition of binder, the fly ash plays the role of nucleus, compacts the system and also rearranges mechanical stresses between the main frame (matrix) and particles of fly ash, which is confirmed by pictures of microstructure of modified specimens (fig. 4).

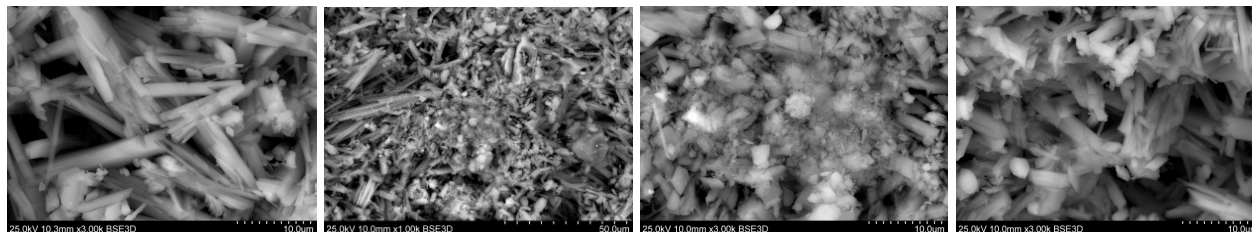


Fig. 3. The SEM images of microstructure of GFA binder specimens with manganese sulfate admixture at the different ages (from left to right: 1, 3, 7 and 28 days)

Рис. 3. Микроструктура гипсофторангидритового вяжущего с добавкой сульфата марганца в различном возрасте (слева направо: 1, 3, 7 и 28 суток)

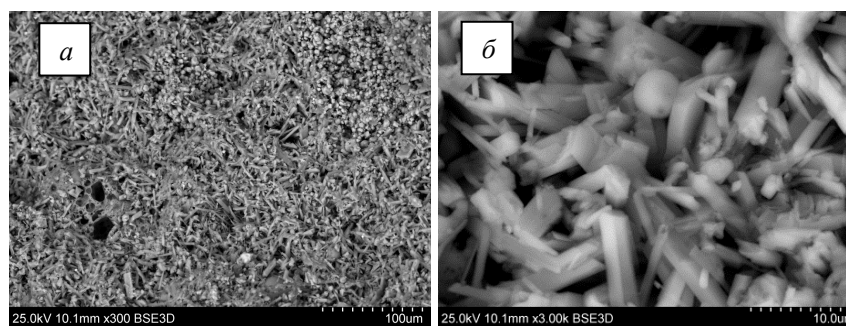


Fig. 4. The SEM images of microstructure of specimens of FA contained binder modified by fly ash: *a* – magnification $\times 300$; *b* – magnification $\times 3000$

Рис. 4. Микроструктура фторангидритового вяжущего, модифицированного золой-уноса: *a* – увеличение $\times 300$; *b* – увеличение $\times 3000$

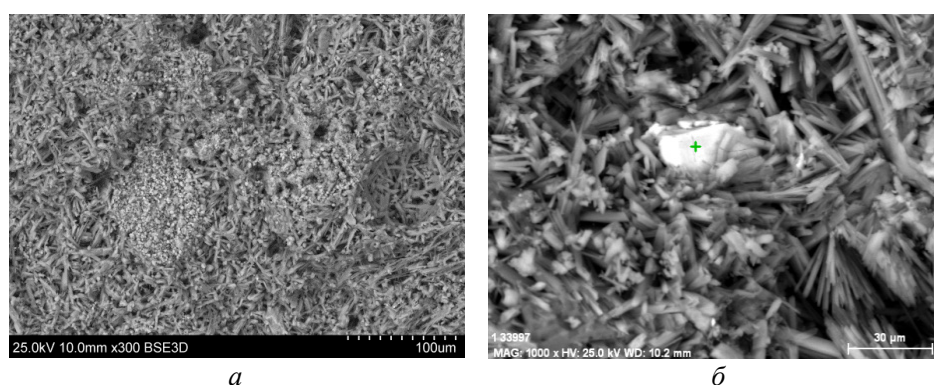


Fig. 5. The SEM images of microstructure of specimens of FA contained binder modified by Portland cement: *a* – magnification $\times 300$; *b* – magnification $\times 3000$

Рис. 5. Микроструктура фторангидритового вяжущего, модифицированного портландцементом: *a* – увеличение $\times 300$; *b* – увеличение $\times 3000$

The usage of Portland cement in the coposition of composite gypsum binder was contributed to the increasing of softening coefficient from 0.3 for pure GFA binder to 0.6 for composition

with Portland cement and also to densification of finely porous structure of stone by formation and filling of defect with tangled needleshaped minerals of ettringite formed as a result of Portland cement hydration process at the initial stage of hardening that is confirmed by microphotographs (fig. 5). These processes provide the formation of the new type of structure that promotes the increasing of strength properties and durability of the material. The optimal dosage of Portland cement for GFA binder in terms of setting times is 6 %.

The following results reflecting the strength properties of binders (fig. 6) were obtained depending on the dosage of FA and activation admixture.

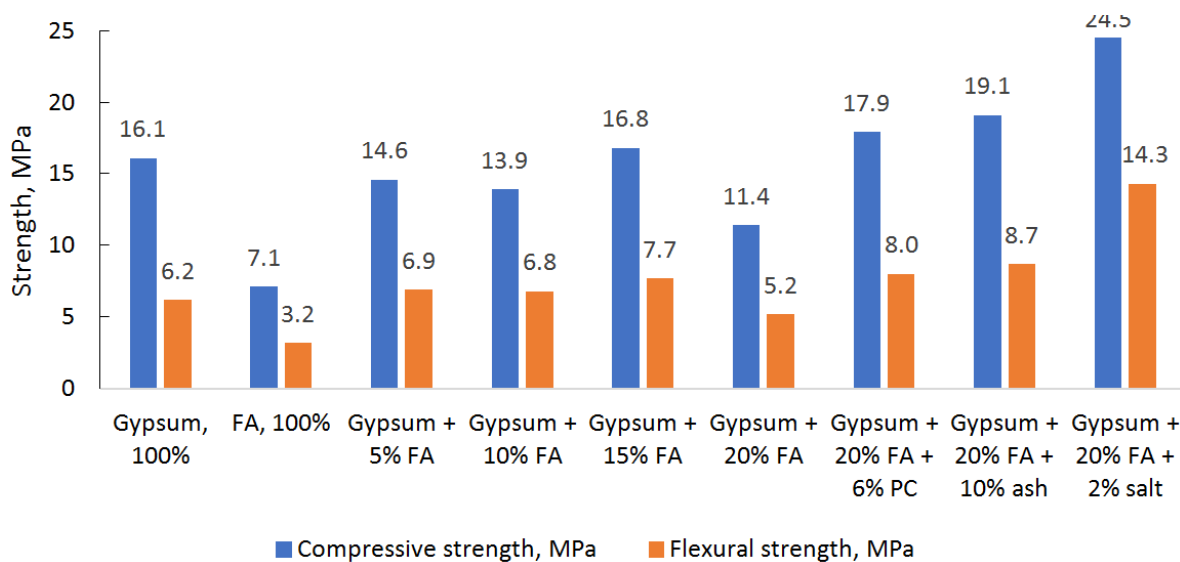


Fig. 6. The strength properties of modified GFA binder

Рис. 6. Прочностные свойства модифицированного гипсофторангидритового вяжущего

Based on the results of investigation of GFA binder activation it can be concluded that required properties of DCM can be regulated by variations of combination of activation method. Moreover, the addition of complex polyfunctional admixtures increasing solubility, hydration rate, dense of the system, forming hardly soluble compounds, regulating linear strain in the composition of DCM can be simultaneously used with investigated activators.

Development of DCM composition for self-levelling floor based on the composite GFA binder

The potential of composite binders on the base of FA can be effectively used in the technology of production of DCM for self-levelling floors. The main advantages of these floors are rapid strength gain, absence of deformations and volume change during hardening, water resistance, satisfactory adhesion to mineral basement and low cost in comparison with analog materials.

Based on the results of carried out investigations and according to literature prescription data the basic composition of DCM for self-levelling floors was selected. Dosage and function of each component are presented in the table 3.

The process of the formation of structural strength and main physical and mechanical properties of floor mortar created with development composition is mainly due to composite GFA binder, the hardening process of which was considered in details early.

Table 3

The basic composition of DCM for self-levelling floors
on the base of the composite GFA binder

Таблица 3

Базовый состав ССС для самонивелирующихся полов
на основе композиционного гипсофторангидритового вяжущего

Component	Function	Dosage, % by mass
Hight-grade gypsum	A main component of the binder	42.3
FA	A component in the composition of composite binder	9.8
Sand	An aggregate created the spatial framework of the composite, the largest size ≤ 0.4 mm	37.5
Fly ash	A component provided pozzolanic reaction and it is the nucleus for soluble anhydrite and filler and it creates dense packing of particles and decreases the risk of cracking	5.2
Portland cement CEM I 42.5 R	An activator of FA hardening, it participates in the formation of dense and strength structure of composite GFA binder	4.7
Hyperplasticizer	It provides increased flowability of fresh mortar with decreasing of water to solid ratio	0.1-0.4
Defoaming agent	It decreases air entrainment in the process of adding water and prevents the appearance of craters on the surface of the coating	0.1-0.2
Cellulose ether	It provides water-retaining effect, increases cohesiveness of mix and densifies the structure of the composite	0.02-0.2
Setting retarder	It provides required lifetime of the mortar	0-0.3

At the same time assurance of technological properties of fresh mortar (flowability, homogeneity, cohesion, viability, water-retaining capacity and others), which is achieved by addition of different modifying additives, is important.

It should be noted, that in Russian practice there are no specifications of production of gypsum DCM for floor. That's why some technical characteristics and test methods were taken from foreign standards or Russian normative documents concerning materials with similar functionality.

The requirements for DCM for self-levelling floors is presented in the table 4.

The important thing for selection of mix composition is determination of optimal dosages of admixtures provided the technological properties of mortar.

The optimal content of setting retarder was determined according to requirements of viability of mortar and retention of its properties over time. The amount of defoaming agent was determined visually by the presence or absence of air bubbles on the surface and into the structure of hardened specimens. The modifiers mentioned above have no mutual impact and functional influence on others components of a mixture, that's why its content was determined by sequential introduction into the mixture each of them.

But in the case of plasticizer and cellulose esters this approach is not appropriate because these admixtures have mutual impact on each other and not only technological properties of fresh mortar but also formation of structure and quality of mortar is modified by its co-administration.

Based on this, the methods of mathematical experimental design with statistical processing of experimental data were used for finding the optimal dosages of plasticizer and cellulose ester.

The dosages of plasticizer and cellulose ester were variable parameters in the experiment. The content of admixtures varied from 0.05 to 0.15 % with the range of variation 0.05 %.

Table 4

The requirements imposed on DCMs and mortars for self-levelling floors

Таблица 4

Требования, предъявляемые к ССС и растворам для самонивелирующихся полов

Parameter	Normalized values (normative document)	Test method
Properties of DCM (powder)		
The largest size of the aggregate, mm	0.4	
Humidity, no more, %	0.1 (GOST 28013)	GOST 8735
Color	light grey	
Bulk density, kg/m ³	1100–1500	GOST 8735
Properties of fresh mortar		
Flowability, mm	160	DIN 1048 (cylinder $d = 50$ mm, $h = 22$ mm)
	230 ±10	Split ring of Vicat apparatus (GOST 310.3)
	260 ±10	Souttard viscometer
Viability, min	≥ 30	GOST 10181, GOST 31356 Visually by merging the mixture after cutting with a knife
Water-retaining capacity, %	≥ 95 (GOST 31357)	GOST 5802
Segregation, %	≤ 10 %	GOST 5802
Mortar density, kg/m ³	1200–2400	GOST 5802
Properties of hardened mortar		
Tensile strength in bending (1, 7, 28 days), MPa	3.0–7.0	GOST 23789, GOST 310.4
Compressive strength (1, 7, 28 days), MPa	12.0–40.0	GOST 23789, GOST 310.4
Crack growth resistance	Absence of cracks in the design layer (1–10 mm)	The wedge method (DIN 18156), GOST 33699
Shrinkage strains	Absence of shrinkage strains	GOST 24544 Dial indicators
Time to achieve compressive strength of 1,5 MPa (when walk is possible), h	≤ 12	GOST 23789
Adhesion strength to the base, MPa	> 0.5	GOST 31356 GOST R 56387
Density, kg/m ³	400–2400	GOST 5802
Water resistance (K_{soft})	≥ 0.75	Assessed by softening coefficient

The following properties were chosen as optimization parameters:

- the flow of mixture (by Souttard), mm;
- value of adhesion strength to the concrete base, MPa;
- compressive strength, MPa.

The orthogonal two-factor experiment with three levels of variable parameter values was conducted for precise determination of optimal dosages of admixtures. According to the results of statistical processing of the experimental data, mathematical models for optimization parameters depended on the dosages of investigating admixtures were created. The graphical interpretations of the obtained mathematical models are presented on the fig. 7.

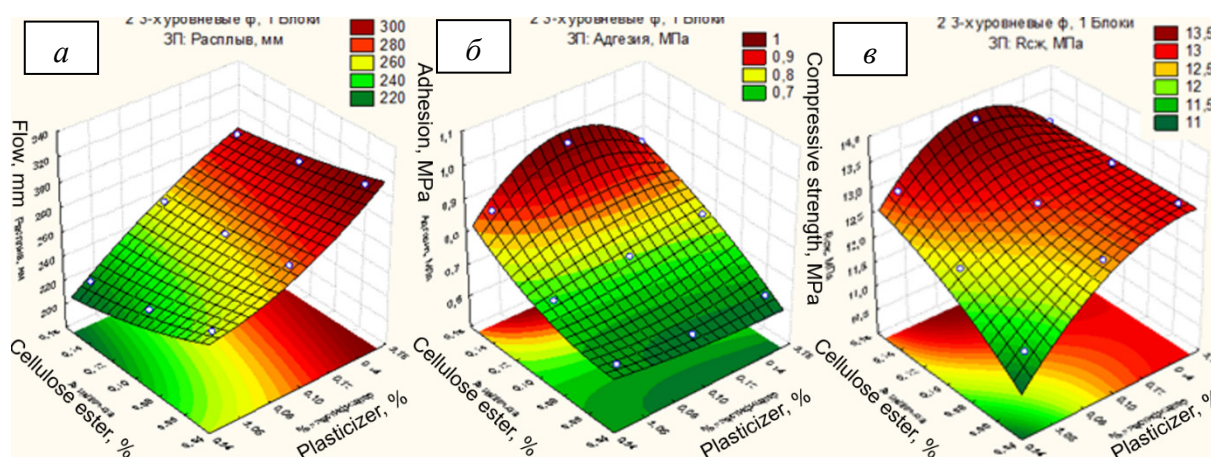


Fig. 7. The response surfaces of: *a* – flow; *b* – adhesion; *c* – compressive strength

Рис. 7. Поверхности отклика для: *a* – распыля; *b* – адгезии; *c* – прочности на сжатие

Then, as the result of solution of optimization problem the optimal dosage of admixtures was found: hyperplasticizer – 0.1 % by mass of DCM and cellulose ester – 0.15 %.

Thus, the content of modified admixtures determined the technological properties of mortar is as follows:

- hyperplasticizer – 0.1 %;
- defoaming agent – 0.15 %;
- cellulose ester – 0.15 %;
- setting retarder – 0.1 %.

According to the results of determination of quality parameters of DCM based on the developed composition (table 5) it was established that the technological properties of fresh and hardened mortar not only meet the all requirements of normative documents but also is not lower than properties of similar products offered in the modern market of DCM.

Table 5

The requirements imposed on DCMs and mortars for self-levelling floors

Таблица 5

Требования, предъявляемые к ССС и растворам для самонивелирующихся полов

The quality parameter	Value for developed DCM	Value for “Ecopol” DCM	Value for “Bergauf Easy Boden” DCM
Flowability, mm	260	260	260
Viability time, min	80	90	95
Water-retaining capacity, %	95.5	93.9	98.1
Crack formation	Absence of cracks	Absence of cracks	Absence of cracks
Adhesion strength to the concrete base, MPa	0.98	0.55	0.65
Tensile strength in bending, MPa	10.4	9.8	7.2
Compressive strength (28 days), MPa	22.7	18.5	15.1
Linear deformations, mm/m	+ 0.02	+ 0.04	+ 0.1
after 3 days			
after 7, 14 and 28 days	–	–	+ 0.04
Density, kg/m ³	1783	1734	1750

At the same time the DCM on the base of developed GFA binder is nonshrink and it provides the absence of cracks as well in the stage of strength gain as in the operation process.

Conclusions

Thus, based on the results of investigation of FA and composite GFA binder and DCM for self-levelling floors the following conclusions can be drawn:

1. The investigation of hardening process of FA indicated that mineral formation and formation of binder structure is very slow with formation a small amount of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ from soluble part of anhydrite and $\text{CaSO}_4 \cdot 0.62\text{H}_2\text{O}$ from insoluble part which eventually turns into gypsum dehydrate gradual and with sufficient amount of moisture.

2. The investigation results of influence of FA on the physical and chemical hardening processes of composite GFA binder indicated that application of mechanisms of complex chemical and mechanical activation of FA allows to affect directionally the hydration processes of GFA binder in particular the formation of dense and strength structure.

3. The regularities of properties and microstructure variations of complex binder on the base of high-grade gypsum and FA activated by different chemical and mineral admixtures were established. Also, the efficiency and mechanisms of activator influence linked with acceleration of hydration processes and formation of ordered structure with dense contact zone between the crystals that allowed to provide the increasing of strength of composite gypsum binder by 46 % (R_{comp}) and 86 % (R_{flex}) and also to provide the value of softening coefficient no less than 0.75.

4. On the base of composite GFA binder the composition of DCM for nonshrink self-levelling floors has been developed. Also, it was established that properties of obtained mixture not only meet the requirements of existing normative documentations but also are not inferior to quality parameters of analog products presented at the modern market of building materials.

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