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Kompozitsion Materiallar

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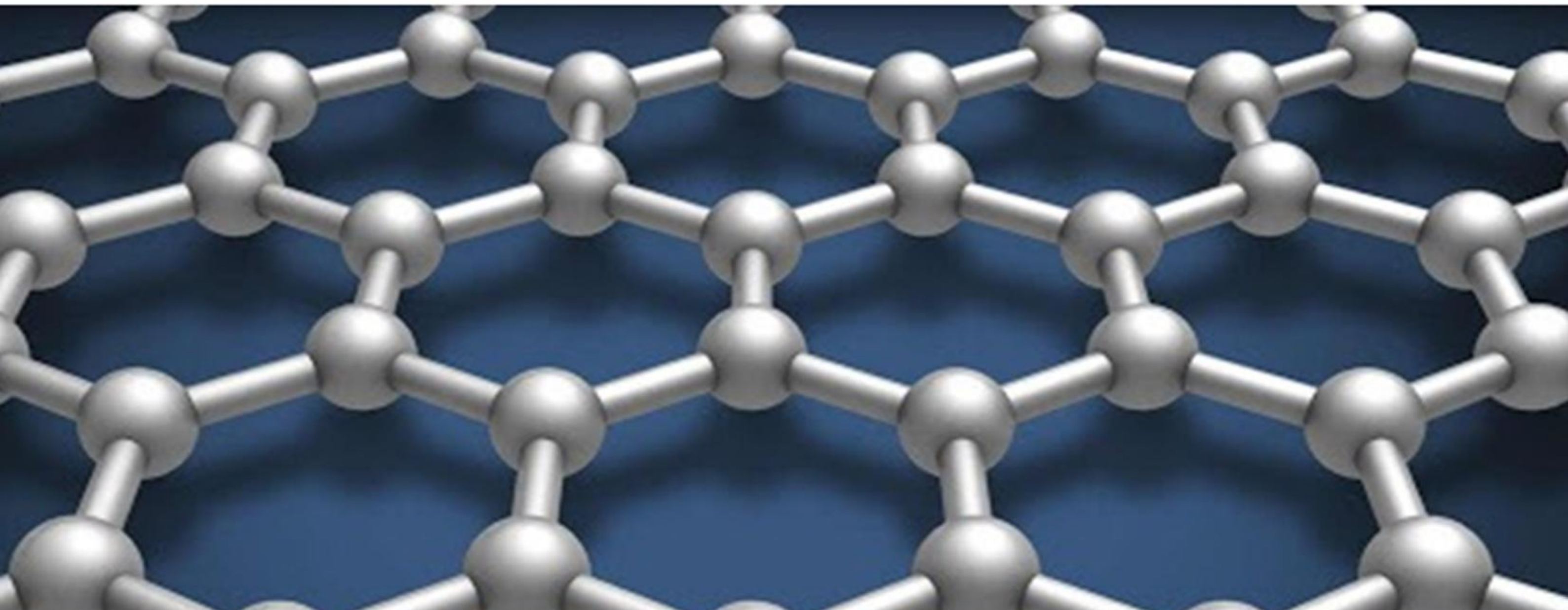

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Композиционные материалы

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EFFECTIVE FILLING MIXTURES BASED ON SYNTHETIC ANHYDRITE

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Introduction. The surplus of sulfuric acid at non-ferrous metallurgy enterprises increases the importance of searching for new (alternative) methods for its utilization, one of which is the treatment with calcium-containing neutralizing agents like limestone, chalk, lime and lime milk, which results in obtaining various modifications of calcium sulfate that can be used as gypsum binders [1-3].

The existing methods of neutralizing the sulfuric acid presume sophisticated technological processes, low manufacturability, engineering complexity and also heavy economic expenditures. At the moment, a new preparation method for the artificial anhydrite has been developed using strongly acid solutions, as a result of the interaction of sulfuric acid with limestone flour. The phase composition of calcium sulfate corresponds to insoluble anhydrite. Drawback of the obtained artificial anhydrite is its low reactivity. One of the most promising areas for the future use of artificial anhydrite is the use infilling mixtures, the production and use of which could solve the utilization problem of concentrated sulfuric acid and create an effective competitive analogue of the materials which are currently in use that also satisfies technical and technological requirements [4-8].

Experimental. The applicability of artificial anhydrite for filling mixtures was determined by physical and mechanical parameters including comparison with similar values for natural and thermal anhydrite (obtained by heat treatment of gypsum stone). Researches of the effect of hardening catalysts such as potassium sulfate, portland cement and plasticizing surfactants were carried out, also properties of natural, thermal and artificial anhydrite with these catalysts were analyzed.Compressive strength dependency on the concentration of lime and potassium sulfate was obtained, and the degree of hydration dependency on the concentration of lime and potassium sulfate were established, which corresponds with the data of leading scientists in the field of gypsum materials [9-12].

In order to usethe artificial anhydrite as a binder in the manufacture of filling mixtures, their properties, which are given in Tables 1 and 2, should be ensured. Provision of technological, physic and mechanical properties of such filling mixtures can be achieved by directional modification of artificial anhydrite with various activators. Table 3 shows the research results of the properties of artificial anhydrite with various types and contents of additives. The analysis of the obtained results revealed that the optimum modifying additive is a mixture of an alkaline activator (portland cement) and a sulfate one (potassium sulfate), which provides the required time for initial hardening of the activated artificial anhydrite.

Table 1. – Technological properties of filling mixtures

Standardized values of filling mixtures (without a coarse aggregate or with a fine aggregate content)	Value
Slump of the standard cone (complete immersion)	9.0-14.0 cm
Spreadability of mixture from Southard viscosimeter	13-20 cm
Delaminatability index	no more than 1.3
Mixture setting	no less than 2 h
Water segregation from filling mass	no more than 2%

Table 2. –Filling mixture strength depending on size of the excavation

Excavation height, m	Characteristic strength, MPa	Excavation span width, m	Characteristic strength, MPa	
			(assurance factor 3)	(assurance factor2)
< 10	1.0	6	4	3
< 20	1.5	7	5	4
< 30	2.0	8	6	5
< 40	2.5	9	7	6
< 50	3.0	10	8	7

Table 3. – Properties of artificial anhydrite with hardening activators

№	Accelerating agent		W/A, %	Setting time, h-min	
	Type	Amount,%		Start	End
1	No agent	--	28	Doesn't set	
2	CEM I 42,5 K_2SO_4	5 2	48	1-10	2-30
3	CEM I 42,5 K_2SO_4	2.5 2	41	1-10	2-50
4	CEM I 42,5 K_2SO_4	2.5 1	40	1-50	3-00
5	$Ca(OH)_2$ K_2SO_4	0.3 1	39	0-50	1-50

Table 4. – Properties of artificial anhydrite with hardening activators

Factors	Notation	Variability levels		
		-1	0	+1
Content of portland cement CEM I 42,5,% by weight of binder	X_1	2.0	3.5	5.0
Content of K_2SO_4, % by weight of binder	X_2	0.5	1.25	2,0

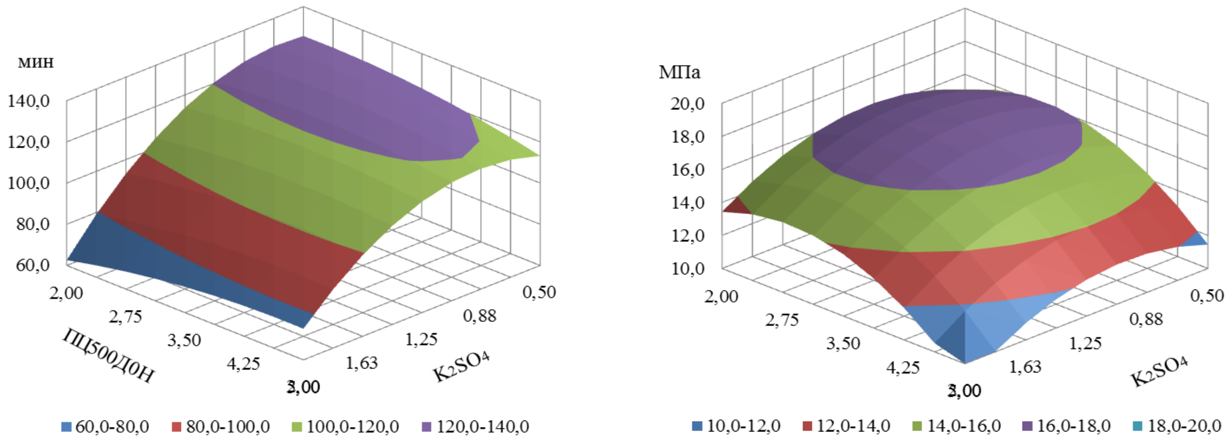
As a result of the conducted experiment, mathematical models (regression equations) were obtained according to the number of determinable characteristics (setting start, compressive strength at the age of 1 and 7 days) with significant factors for the adopted variables:

$$S.S. = 117.52 – 25.3 \cdot X2 + 6.25 \cdot X1 \cdot X2 – 3.7 \cdot X12 –19.7 \cdot X22,$$

$$R1 = 3.89 – 0.45 \cdot X1 + 0.47 \cdot X2 – 0.58 \cdot X1 \cdot X2 – 0.68 \cdot X12 – 0.23 \cdot X22,$$

$$R7 = 16.9 – 2.07 \cdot X1 –1.08 \cdot X2 – 0.25 \cdot X1 \cdot X2 – 2.63 \cdot X12 – 2.08 \cdot X22$$

According to the graphical construction of the received mathematical models shown in Figure 1, for the setting startof the binding agent and for its strength at the age of 7 days it was determined that the optimum composition of the anhydrite binder modifier, which ensures the correspondence with the demands raised on the binder for filling mixtures, contains CEM I 42,5at a rate of 2.5% of binder mass and 1% of K_2SO_4. The binder with this modifier was adopted as the default one for further development of filling mixtures.



a) b)

Fig. 1. Correlation between the binder setting start (a), the compressive strength at the age of 7 days (b) and the content of Portland cement and potassium sulfate

Additional modification of the binder was conducted with plasticizing additives, the most efficient additive was Melment F10, injected in an amount of 0.4 to 1% of the binder mass. For the further assessment of the structure of the obtained binder with different contents of the Melment F10 plasticizer, additional structural studies were conductedusing the focused beam microscope, alsostudies of mineral composition using the powder diffractometry were conducted. Table 5 shows the study results of the mineral composition of solidified samples based on synthetic anhydrite.

Table 5. Mineral composition of samples

№	Composition	Mineralcontent, %		
		$CaSO_4$	$CaSO_4 \cdot 2H_2O$	Silex
1	CEM I 42,52.5%, $K_2SO_4$1%	54.2	45.0	0.8
2	CEM I 42,52.5%, K_2SO_4 1%, Melment F10 - 0,7 %	40.7	58.6	0.6
4	CEM I 42,52.5%, K_2SO_4 1%, Melment F10 - 0,4 %	40.8	58.5	0.6
3	CEM I 42,52.5%, $K_2SO_4$1%, Melment F10 - 1 %	*45.6*	*53.8*	*0.6*

For the further assessment of the structure of received binder with various Melment F10 plasticizer contents, additional structural studies were carried out using focused beam microscope.

A reference sample without a plasticizing additive is characterized by the lowest reactivity, the content of the calcium sulfate dehydrate is 45%, and the content of unreacted anhydrite is 54%, which determines its minimal physical mechanical characteristics. Decreasing the water-binder ratio by the means of reduction of the plasticizer content to 0.4 and 0.7% leads to theincrease of the binder reactivity, to the decrease of anhydrite content to 41%, and also to the increase of calcium sulfate dehydratecontent to 59%. Such ratio of minerals leads to the development of crystals of comparable sizes, and as a result, to the increase of strength and water resistance. The most desirable characteristics of the modified anhydrite binder are provided when the plasticizing additive Melment F10 is added in an amount of 1% due to the optimum content of anhydrite and calcium sulfate dehydrate in an amount of 46 and 54% respectively. The structure with the maximum strength and with the crystals of various shapes is developed at the given ratio of anhydrite and calcium sulfate dehydrate, in which anhydrite serves as a reinforcementcage, and a calcium sulfate dehydrate serves as a binder matrix.

As a result of the conducted research, an effective binder based on an artificial anhydrite was obtained, which can be used for the development of various types of filling mixes [13-16].

Selecting the composition of the filling mixtures with the retrained binder based on the synthetic anhydrite was carried out by the means of selecting the optimum amount of sand, while ensuring the required mobility and other properties. As a result of the conducted research, two mixture compositions are determined, whichcorrespondto the design technological, physical and mechanical properties required for filling mixtures. Technological properties, composition and strength parameters of the developed filling mixtures are presented in Table 6.

Table 6.Composition and properties of filling mixtures

№	Mixture composition, kg/m^3	Composition 1	Composition 2
1	Cement	0	22
2	Modified anhydrite binder	740	840
3	Sand	1110	840
4	Water	350	370
Свойства закладочных смесей			
5	Density, kg/m^3	2150	2040
6	Cone slump, cm	14	14
7	Southard Spreadability, cm	12	12
8	Spread on the jolting table (15 jolts), cm	20	21
9	Water gain, %	0.5	0.1
10	Compression strength 1 dayMPa	3.0…5.3	6.0...11.1
11	Compression strength (7 day, dry), MPa	10.0…10.7	15.1...24.1
12	Compression strength (7 day, water saturated), MPa	5.2…6.4	8.3…14.5
13	Softening facor	0.52…0.6	0.55...0.6

The obtained research results have shown an opportunity and a prospectivity of application of the modified synthetic calcium sulphate in the composition of filling mixtures. The use of various local aggregates (coarse and fine) in the development of filling mixture compositions with various grades based on modified synthetic anhydrite gives the opportunity to achieve the results received in this study, and in some cases to achieve even better results.

5. Методы исследовании, приборов и оборудований композиционных материалов