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Activation of fluoranhydrite with various chemical additives for the production of gypsum fiberboards

Preliminary studies of fluoranhydrite as a binder showed that on its own it almost does not harden, intervention in the technological process of basic production is almost impossible, so in order to obtain materials it is necessary to develop ways of modifying it to initiate the binder properties or use "acidic" fluoranhydrite before the neutralization stage. In this work the influence of various additives (sodium sulfate and sodium sulfite, potassium sulfate, copper sulfate, iron sulfate crystallohydrate, alumina aluminate, sodium carbonate) on the properties of fluorine hydrite binders produced by neutralization of sour waste from hydrofluoric acid production with an excess of limestone was studied. In this work to obtain dependences of anhydrite binder technological properties on the number of introduced additives and determine the optimal composition of the binder, as well as create mathematical models of the processes under study and their statistical analysis used mathematical planning of the experiment. As the conducted studies showed the speed of setting of products based on anhydrite binder and their strength mainly depend on the temperature of water and its amount for mixing at the optimum dispersity of the binder. The strength of the samples made from the neutralized waste was found to be in the range of 0.5–1.2 MPa, and the strength of the samples based on the activated anhydrite binder — in the range of 5.3–10.7 MPa that corresponds to the parameters of the material suitable for the production of boards.

Keywords: fluorine gypsum, anhydrite, hydrofluoric acid, anhydrite binder, activator.

Introduction

Increase of construction rates is impossible without orientation of building complex to the most effective low-cost and non-deficit materials, allowing to improve the comfort of housing. One of the ways to solve this problem is the expansion of production and introduction of new highly effective products and structures on the basis of gypsum binder which has a number of advantages in comparison with other building materials — low cost with high consumer properties (hygiene, fire resistance, bio resistance, providing a favorable climate indoors). In addition, the rapid hardening of gypsum eliminates the need for significant energy consumption for heat treatment to obtain products for different purposes, which helps to reduce energy costs for the manufacture of products. To obtain a fluorine hydrite-based binder suitable for the production of partition walls, it is necessary to solve the problem of accelerating the time of setting and hardening of the material. It is known that insoluble anhydrite (AII), a component of fluoranhydrite, does not hydrate and does not harden under normal conditions. For its activation it is necessary to change the coordination of

Ca^{2+} ions in the crystal lattice of calcium sulfate, which is achieved by the introduction of hardening activators [1, 2]. As a rule, substances that increase the solubility of AII and are centers of crystallization are used as hardening activators [3].

The primary challenges in producing various construction materials based on fluoroaranhydrite stem from the sulfuric acid present within it, residing in the grains' pores and adsorbed on the surface. Neutralizing this acid with alkaline components (such as lime, lime flour, carbide silt, Portland cement, nepheline slime, etc.) is necessary [4, 5]. To enhance fluoroaranhydrite's activity and its suitability for construction product manufacturing, it is suggested to employ complex additives and techniques. However, this approach results in increased complexity and cost in the overall production processes and final products [6].

The purpose of this article is to select the optimal chemical and quantitative composition of activating additives and to study the effect of composition modification on the strength of anhydrite binder.

Materials and methods of research

The raw material for the production of anhydrite binder is gypsum-containing waste products of hydrofluoric acid. An anhydrite binder, also known as anhydrite cement, is a substance produced through the grinding of naturally occurring or artificially prepared anhydrite (achieved by heating at 600–700 °C) with activators. The primary source materials for anhydrite binder include natural two-water gypsum and anhydrite, with their quality specifications outlined in GOST 4013-82.

Immediately out of the furnace waste is granulated material of gray color, granule size of which is 0.3 mm to 60-70 mm. Physico-chemical characteristics of this waste and the possibility of obtaining binder material from it have been previously studied, the results of research are described in [2, 7] and [8]. According to the above-mentioned sources, the properties of the waste obtained at different times are close to each other, which indicate a fairly homogeneous composition of the waste output and the stability of their production regime [9]. The chemical composition of the waste is as follows (wt.%): CaO — 28–39; SO_3 — 38–56; SiO_2 — 0.2; Al_2O_3 — 0.5; Fe $_2O_3$ — 0.3; Cr_2O_3 — 0.01; TiO_2 — 0.012; Na — 0.015; K — 0.01; H_2SO_4 — 10–15; CaF_2 — 3, HF — 0.3.

The crystal structure consists of finely dispersed translucent idiomorphic anhydrite crystals 5–10 μm in size. The average refractive index N = 1.57. HF, fluorite CaF_2 and iron hydroxides were observed as impurities. The total anhydrite content is about 95%. The phase composition of the gypsiferous waste was determined by the X-ray phase method. Acidic waste is identified as $CaSO_4$ ― calcium sulfate. The neutralized waste is an anhydride of orthorhombic structure [2, 10–12].

Interference in the main production process to regulate the properties of the by-product is undesirable, so we studied the properties of the initial fluorine hydride binder obtained by: 1) the method of co-milling with a neutralizing component in the form of limestone in the enterprise (JSC “Ulba Metallurgical Plant”); 2) in the laboratory of the “Institute of Composite Materials” [13].

When performing this work as activating additives were used: (K_2SO_4), copper sulfate ($CuSO_4$), crystalline hydrate of iron sulfate ($FeSO_4 \cdot 7H_2O$), alumina aluminate, Na_2CO_3, NaCl. By analysis of literature data the content of additives was accepted (0.5–3 %) from weight of dry matter with a step of 0.5%. All additives were introduced with mixing water both separately and in combination of the two components. The additives used, in addition to activating hardening, also affect the other physical and mechanical properties of the binder. Mathematical planning of the experiment was used to accelerate the selection of hardening activators.

A fractional three-factor experiment was used to select the modifier additives. Calculations were carried out on a computer program “STATISTICA”.

Results and discussion

According to the ideas of most modern researchers, the process of hardening of anhydrite binder proceeds mainly as a result of hydration when dissolving anhydrite in water and subsequent crystallization of the resulting gypsum. Acceleration of hydration and hardening of anhydrite to technically acceptable terms is achieved by the introduction of additives ― curing activators, formula 1:

$$m\mathrm{CaSO_4} + (\text{salt})\ n\mathrm{H_2O} = (\text{salt})\ m\mathrm{CaSO_4} \cdot n\mathrm{H_2O} \quad (1)$$

which then disintegrates to form two-water gypsum equation 2:

$$(\text{salt})\ m\mathrm{CaSO_4} \cdot n\mathrm{H_2O} \rightarrow (\text{salt})\ \mathrm{H_2O} + \mathrm{CaSO_4 \cdot 2H_2O} \quad (2)$$

Two-water gypsum is released first in a colloidal state and then crystallizes. The period when a significant amount of colloidal $CaSO_4 \cdot 2H_2O$ is formed is accompanied by heat release. During this period, the setting process takes place, and recrystallization processes contribute to the solidification. In the initial hours, the strength of the anhydrite binder rises, followed by a subsequent decrease in strength. This decline can be attributed to the disintegration of the unstable complex hydrate during this phase. Subsequently, the strength undergoes a prolonged increase. As the hardening progresses, there is a gradual augmentation in the quantity of bound water within the products [14].

According to research conducted by other scholars, there is no formation of intermediate compounds between sulfates and anhydrite. The accelerating impact of additives is attributed to factors other than the formation of such compounds. Some researchers posit that the activating influence of lime, caustic dolomite, and other low-soluble compounds, which share crystallochemical similarities with gypsum, lies in their ability to act as centers of crystallization. In this view, the particles of additives serve as nuclei for crystallization, facilitating the rapid removal of a supersaturated dihydrate solution formed during the hydration of anhydrite. This process leads to the equilibrium state with the separation of $CaSO_4 \cdot 2H_2O$ in the sediment, thereby reducing supersaturation and creating conditions for the dissolution of new portions of anhydrite [15].

Additionally, some researchers propose that activators, such as acidic anhydrite hardening agents, enhance its solubility and chemical potential in the early stages of hardening [16]. The types of activation utilized are determined based on the chemical composition of the introduced additives.

Despite a significant amount of research on the activation of anhydrite binders (including those from man-made waste) and the selection of optimal chemical and quantitative composition of activating additives, this process also requires an individual approach.

Using the statistical package, we analyzed the experimental data for the mixture in terms of strength, the beginning and the end of setting.

The results of the processing of the experimental data on the strength index are presented below.

In order to identify statistically significant effects a variance analysis of these models was performed, the results of which are presented in Table 1.

Table 1

Results of the analysis of variance on the strength index

	SS	df	MS	SS	df	MS	F	p	R-Sqr	R-Sqr
Linear	235.6788	3	78.55961	236.8854	15	15.79236	4.974533	0.013618	0.498723	0.398468
Quadratic	127.6587	6	21.27646	109.2266	9	12.13629	1.753126	0.215443	0.768864	0.537728
Special Cubic	9.2003	4	2.30008	100.0263	5	20.00526	0.114974	0.971532	0.788333	0.237998
Total Adjusted	472.5642	18	26.25357							

Table 1 shows that statistically significant effects are observed in the linear model (p-value criterion less than 0.05). For other models this criterion has a higher value. Therefore, we will further consider the linear model.

Regression equation will have the following form

$$Y_{пр} = 0.67 \cdot A + 12.35 \cdot B - 1.99 \cdot C + 7.39 \cdot D. \quad (3)$$

For the selected model statistics were calculated, the values of which are presented in Table 2.

Table 2

Value of statistics for the selected model

	Coeff.	Std.Err.	t(15)	p	–95, %	+95, %
(A)Var1	0.66734	2.682398	0.248784	0.806902	-5.05006	6.38473
(B)Var2	12.34584	2.682398	4.602539	0.000345	6.62845	18.06324
(C)Var3	-1.98789	2.682398	-0.741086	0.470085	-7.70528	3.72951
(D)Var4	7.38734	2.682398	2.754004	0.014767	1.66994	13.10473

However, only B and D are significant factors in this model.

The table of variance analysis shows good results for the fitted linear model, which are presented in Table 3.

Table 3

Analysis of variance in the linear model

	SS	df	MS	F	p
Model	235.6788	3	78.55961	4.974533	0.013618
Total Error	236.8854	15	15.79236		
Total Adjusted	472.5642	18	26.25357		

Below are the results of processing the experimental data on the beginning of the setting of the mixture.

As a mathematical model the followings were tried: linear, quadratic and special cubic.

In order to identify statistically significant effects, a variance analysis of these models was performed, the results of which are presented in Table 4.

Table 4

Results of the analysis of variance on the beginning of setting

	SS	Df	MS	SS	df	MS	F	p	R-Sqr	R-Sqr
Linear	671.938	3	223.9792	376.6788	15	25.11192	8.919238	0.001239	0.640785	0.568942
Quadratic	223.116	6	37.1860	153.5625	9	17.06250	2.179401	0.141220	0.853557	0.707114
Special Cubic	116.034	4	29.0084	37.5290	5	7.50579	3.864800	0.085301	0.964211	0.871159
Total Adjusted	1048.616	18	58.2565							

Table 4 shows that statistically significant effects are observed in the linear model (p-value criterion less than 0.05). For other models this criterion has a higher value. Therefore, we will further consider the linear model.

The regression equation will have the following form

$$Y_n = 23.52 \cdot A + 6.06 \cdot B + 26.73 \cdot C + 6.46 \cdot D. \quad (4)$$

For the selected model statistics were calculated, the values of which are presented in Table 5.

Table 5

Value of statistics for the selected model

	Coeff.	Std.Err.	t(15)	p	–95, %	+95, %
(A)Var1	23.52158	3.382515	6.953874	0.000005	16.31192	30.73124
(B)Var2	6.06337	3.382515	1.792564	0.093230	-1.14629	13.27303
(C)Var3	26.72606	3.382515	7.901239	0.000001	19.51640	33.93572
(D)Var4	6.45740	3.382515	1.909054	0.075575	-0.75226	13.66706

As follows from Table 5, factors *A* and *C* are significant for the selected model (p-value is much less than 0.05).

The table of variance analysis shows good results for the selected linear model, which are presented in Table 6.

Table 6

Analysis of variance in the linear model

	SS	df	MS	F	p
Model	671.938	3	223.9792	8.919238	0.001239
Total Error	376.679	15	25.1119		
Total Adjusted	1048.616	18	58.2565		

Processing of the results on the indicator of the end of setting.

Below there are the results of dispersion analysis of experimental data at the end of setting of the mixture.

As a mathematical model the following models were tried: linear, quadratic and special cubic. The results of the analysis of variance on these models are shown in Table 7.

Table 7

Results of analysis of variance

	SS	df	MS	SS	df	MS	F	p	R-Sqr	R-Sqr adjusted
Linear	328.107	2	164.0533	820.2934	7	117.1848	1.399954	0.308011	0.285708	0.081624
Quadratic	370.594	3	123.5314	449.6994	4	112.4248	1.098791	0.446624	0.608412	0.118928
Special Cubic	208.983	1	208.9826	240.7168	3	80.2389	2.604504	0.204965	0.790389	0.371168
Total Adjusted	1148.400	9	127.6000							

Table 7 shows that statistically significant effects are not observed for any model (p-values for all models are greater than 0.05). However, this index is the smallest for the special cubic model, which has the smallest mean square error (MS = 80.2) and the largest Fisher index (F = 2.6).

The quality of this model is estimated by such indicator as the coefficient of determination Rsqr, which is 0.79 for this model. This indicates a high quality of the model. We can say that 80 % of the observed effect is explained by the selected factors.

The regression equation will be as follows:

$$Y_{regressio} = +59.267319210644 \cdot A + 26.812866336268 \cdot B + 33.44914754712 \cdot C - 51.840010145936 \cdot A \cdot B - 78.566741356355 \cdot A \cdot C + 6.524344955884 \cdot B \cdot C + 463.76702491378 \cdot A \cdot B \cdot C. \quad (5)$$

Next, the statistics for the selected model were calculated. These values are presented in Table 8.

Table 8

Value of statistics for the selected model

	Coeff.	Std.Err.	t(3)	p	–95, %	+95, %
(A)Var1	59.2673	8.6593	6.84438	0.006384	31.710	86.825
(B)Var2	26.8129	8.6592	3.09644	0.053444	–0.745	54.370
(C)Var3	33.4491	8.6592	3.86283	0.030675	5.892	61.007
AB	–51.8400	43.5889	–1.18929	0.319876	–190.559	86.879
AC	–78.5667	43.5889	–1.80245	0.169264	–217.286	60.153
BC	36.5243	43.5888	0.83793	0.463575	–102.195	175.243
ABC	463.7670	287.3673	1.61385	0.204965	–450.764	1378.298

As it follows from Table 8, factors A and C are the most significant for the chosen model (p-value is much less than 0.05).

Using the obtained regression equation we calculated parameter values for different points of the plan, which are shown in Table 9.

Table 9

Experimental and theoretical parameter values

	Observed	Predictd	Resids
1	57.00000	59.26732	–2.26732
2	25.00000	26.81287	–1.81287
3	35.00000	33.44915	1.55085
4	26.00000	30.08009	–4.08009
5	26.00000	26.71655	–0.71655
6	39.00000	39.26209	–0.26209
7	39.00000	46.58826	–7.58826
8	54.00000	44.66838	9.33162
9	46.00000	38.03215	7.96785
10	37.00000	39.12314	–2.12314

The calculated theoretical values were compared with the experimental values and a divergence histogram was plotted in Figure 1.

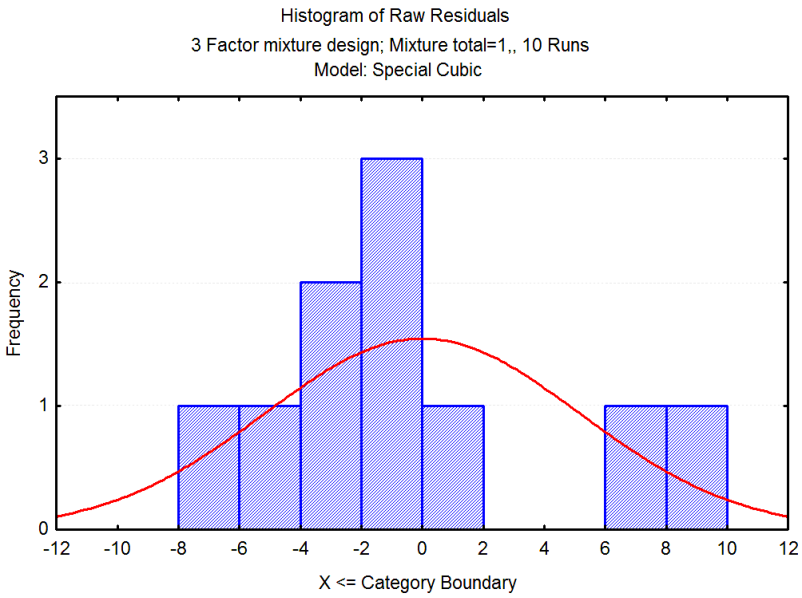

Figure 1. Histogram of the difference between the theoretical and experimental values

This histogram obeys the normal law of distribution, which is a prerequisite for the construction of regression models.

Figure 2 shows the response surface of the setting time depending on the selected factors.

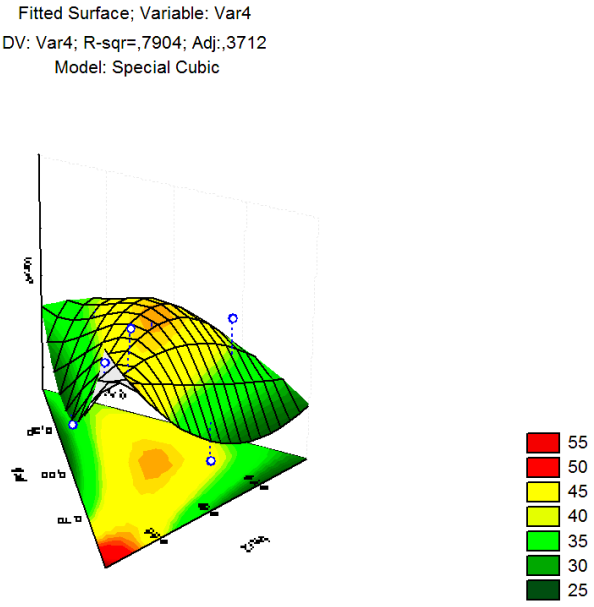

Figure 2. Response surface of the setting time

The contour plot of the studied dependence is shown in Figure 3.

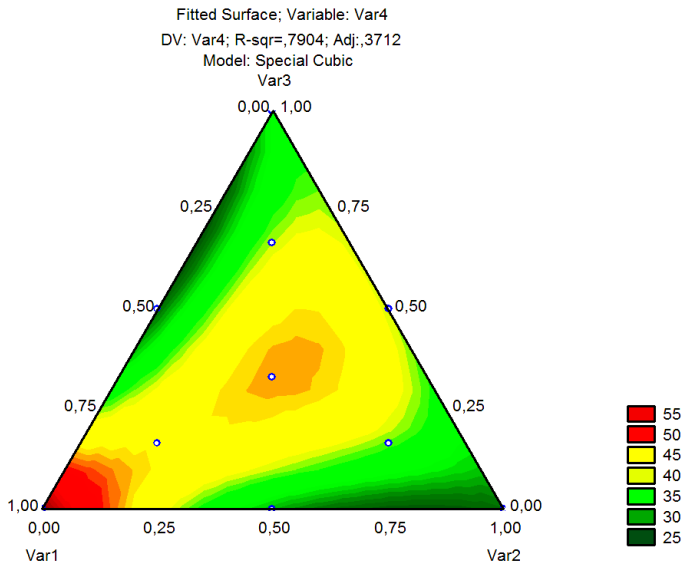

Figure 3. Contour plot of the studied dependence

According to the model obtained, using the contour plot of the studied dependence by varying the percentage of components anhydrite binder with the necessary setting time was obtained.

The greatest effect was obtained by using potassium sulfate in an amount of 1.5 %. The results of the experiment are shown in Figure 4.

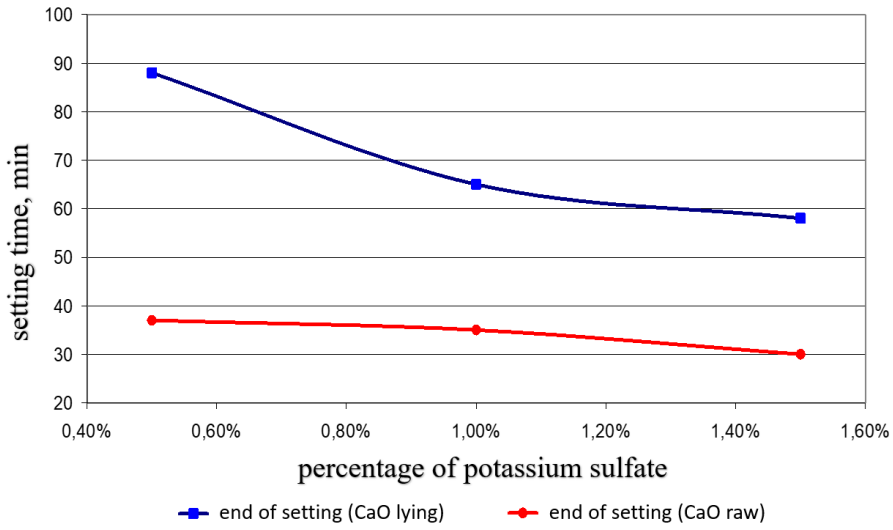

Figure 4. Dependence of setting time on the amount of potassium sulfate and lime quality.

As the studies have shown the rate of setting of products based on anhydrite binder and their strength mainly depend on the temperature of water and its amount for mixing at the optimum dispersity of the binder.

Experimental data showed that the strength of samples made from neutralized waste is in the range of 0.5–1.2 MPa, and the strength of samples based on activated anhydrite binder is in the range of 5.3–10.7 MPa, which corresponds to the parameters of the material suitable for the production of slabs.

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Ойықтақталарды өндіру үшін әртүрлі химиялық қоспалармен фторангидритті белсендіру

Фторангидриттің тұтқырлық сапасын алдын-ала зерттеуде оның өздігінен қатып қалмайтындығын, негізгі өндіріс процесіне араласу іс жүзінде мүмкін еместігін көрсетті, сондықтан материалдарды алу үшін оның тұтқырлық қасиеттерін қоздырып, түрлену тәсілдерін жасау қажет немесе бейтараптандыру сатысына дейін «қышқыл» фторангидритті қолдану керек. Бұл жұмыста әртүрлі қоспалардың (натрий сульфаты мен сульфиті, калий сульфаты, мыс сульфаты, темір сульфатының кристаллогидраты, калий-алюминий ашудастары, натрий карбонаты) фторангидрит тұтқырлығының заттардың қасиеттеріне әсері зерттелді, олар балқытқыш қышқылын өндіруде қышқыл қалдықтарын артық әктаспен бейтараптандырудан алынған. Сонымен қатар ангидрит тұтқырлығының технологиялық қасиеттеріне енгізілген қоспалар санына тәуелділігін алуда және тұтқыр заттың оңтайлы құрамын анықтауда, сондай-ақ зерттелетін процестердің математикалық модельдерін құру және олардың статистикалық талдауына эксперименттің математикалық жоспарлауы қолданылды. Зерттеулер көрсеткендей, ангидриттің тұтқырлығына негізделген өнімдердің қату жылдамдығы және олардың беріктігі негізінен судың температурасына және тұтқырдың оңтайлы дисперсияға айналу кезіндегі оның мөлшеріне байланысты. Бейтараптандырылған қалдықтардан жасалған үлгілердің беріктігі 0,5–1,2 МПа шегінде, ал белсендірілген ангидрит тұтқырлығы негізіндегі үлгілердің беріктігі 5,3–10,7 МПа шегінде екені анықталды, бұл плиталарды өндіруге жарамды материалдың параметрлеріне сәйкес келеді.

Кілт сөздер: фторгипс, ангидрит, балқытқыш қышқыл, ангидритті тұтқырлық, активатор.

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Активация фторангидрита различными химическими добавками для производства пазогребневых плит

Предварительные исследования фторангидрита в качестве вяжущего показали, что самостоятельно он практически не твердеет, вмешательство в технологический процесс основного производства практически невозможно, поэтому с целью получения материалов необходимо разрабатывать способы модифицирования его для инициирования вяжущих свойств или использовать «кислый» фторангидрит до стадии нейтрализации. В статье исследовано влияние различных добавок (сульфата и сульфита натрия, сульфата калия, сульфата меди, кристаллогидрата сульфата железа, алюмокалиевых квасцов, карбоната натрия) на свойства фторангидритовых вяжущих, полученных нейтрализацией «кислого» отхода производства плавиковой кислоты избытком известняка. Для получения зависимостей технологических свойств ангидритовых вяжущих от количества введенных добавок и определения оптимального состава вяжущего, а также создания математических моделей исследуемых процессов и их статистического анализа авторами применено математическое планирование эксперимента. Как показали проведенные исследования, скорость схватывания изделий на основе ангидритового вяжущего и их прочность в основном зависят от температуры воды и ее количества для затвердения при оптимальной дисперсности вяжущего. Выявлено, что прочность образцов, изготовленных из нейтрализованного отхода, находится в пределах 0,5–1,2 МПа, а прочность образцов на основе активированного ангидритового вяжущего — в пределах 5,3–10,7 МПа, что соответствует параметрам материала, пригодного для производства плит.

Ключевые слова: фторгипс, ангидрит, плавиковая кислота, ангидритовое вяжущее, активатор, оптимальная дисперсность.

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