



# Communication **Production of Anhydrite Binder from Waste Fluorangydrite**

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**Abstract:** The technology for obtaining hydrofluoric acid and the peculiarities of its production were studied, and the physical and chemical properties of the waste were examined. Activators that accelerated the hardening of the anhydrite binder were selected. The process of recycling fluorine hydrite waste from the production of hydrofluoric acid at Ulba Metallurgical Plant JSC was studied, and anhydrite unburnt binder with a setting time of 30 min was obtained. On the basis of the obtained data, a technological scheme of anhydrite binder production was developed. The effectiveness of the technological scheme was confirmed experimentally. This work aimed to study the possibility of the integrated use of secondary and anthropogenic raw materials from Ulba Metallurgical Plant, which represents an important means of not only increasing production efficiency and economic benefits and reducing the irrational alienation of land resources, but also protecting against the pollution of water and air basins, as the environmental policy of UMP JSC is nowadays of great importance.

Keywords: fluorine gypsum; anhydrite; hydrofluoric acid; anhydrite binder; activation; activator



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). 1. Introduction

As is known, along with quality, one of the most important factors in determining the competitiveness of building materials is their low cost, which is achieved by energy- and resource-saving technological solutions. With the intensive development of the construction industry, there is an urgent need to produce high-quality and cost-competitive gypsum binders and related products [1,2]. These materials are characterized by lightness; ease of manufacture; good heat and sound insulation properties; biological stability; fire resistance; chemical neutrality; and favorable architectural, decorative, and hygienic qualities [3].

At present, mainly natural gypsum raw materials are used for the production of gypsum binders. However, in regions where no reserves of natural gypsum stone have been explored, the potential raw materials for the production of gypsum binders include gypsumcontaining wastes of anthropogenic origin. Moreover, given the current environmental situation, the involvement of man-made waste in the production of these or other necessary material resources for other industries is one of the best solutions to the problems of environmental protection. The removal of gypsum-containing wastes to dumps and their maintenance require significant capital and operating costs. In addition, their storage requires a large area and has a negative effect on the environment in which the production is located.

The main task of engineering as a science is to identify physical, chemical, and other regularities in order to select the most efficient and cost-effective technological processes. The production and application of binders involve a set of complex processes that includes elements of mechanical and chemical technologies: the processing of materials not only

changes their type and form, but also transforms the composition of the initial substances. The processes of the production and use of binders are based on the physical and chemical transformation of materials. Knowledge of these processes and the ability to manage them are prerequisites for the production of high-quality binders and the effective use of the unique properties of these important building materials.

To date, more than 50 types of gypsum- and anhydrite-containing industrial wastes, such as phosphogypsum, calcium sulfate phosphohydrate, fluorangydrite, borohypsum, titanium gypsum, silicon gypsum, hydrolysis gypsum, cytrogypsum, sulfogypsum (waste products of flue gas desulfurization in thermal power stations and neutralization products of sulfuric acid or calcium waste from various industries), have been studied as raw materials. Some works have suggested the use of synthetic calcium sulfate dihydrate produced from chalk suspensions and sulfuric acid [4,5].

Due to the fact that the East Kazakhstan region does not posses natural gypsum raw materials, there is great interest in fluoranhydrite (acidic anhydrous calcium sulfate), a by-product of the production of hydrofluoric acid at Ulba Metallurgical Plant JSC, obtained by the thermal decomposition of fluorite concentrate with sulfuric acid. After the production of 1 ton of hydrofluoric acid, 1.8 tons of waste products are formed. The waste products generated in the next few years will amount to 30–35 thousand tons annually, and if we consider the possibility of creating new production facilities, it may reach more than 100 thousand tons annually.

As with other types of gypsum-containing waste, this product is pre-neutralized with crushed limestone and stored in the dumps of the plant as a large-tonnage man-made waste, which negatively affects the ecological environment of the city. Therefore, using this waste to obtain a high-quality and cheap material for the construction industry in the region is of high relevance.

However, acidic fluorine hydride, which comes directly from the furnace, and the neutralized waste cannot be immediately used as an anhydrite binder without additional technological intervention, as they have an inconstant composition, rather long setting time, and low strength, which makes them inapplicable for gasket slab production.

In view of the above, the purpose of this work was to determine a formulation of activating additives for obtaining an anhydrite binder from fluorine gypsum that will be used for the production of gasket slabs.

# 2. Materials and Methods

In order to achieve this goal, it was necessary to:

- Conduct a set of studies on the neutralization of acidic fluoranhydrite waste;
- Choose activators that accelerated the hardening of the anhydrite binder;

The raw material used to obtain the anhydrite binder according to the purpose of this work was a gypsum-containing waste product of hydrofluoric acid. An anhydrite binder (anhydrite cement) is a material obtained by grinding together natural or artificially prepared anhydrite (firing at 600–700 °C) with hardening activators. The main raw materials for anhydrite binders are natural two-water gypsum and anhydrite. The requirements for their quality are described in GOST 4013-82.

The waste directly extracted from the furnace is a granular material, gray in color, with granule sizes ranging from 0.3 mm to 60 mm. The physical and chemical characteristics of this waste and the possibility of obtaining a binder material from it have been previously studied, and the results of this research are described in [6,7]. According to the abovementioned sources, the properties of the waste obtained at different times were similar, which indicates the fairly homogeneous composition of the waste output and the stability of the mode of their production. The chemical composition of the waste was as follows (wt.%): CaO-28-39; SO<sub>3</sub>-38-56; SiO<sub>2</sub>-0.2; Al<sub>2</sub>O<sub>3</sub>-0.5; Fe <sub>2</sub>O<sub>3</sub>-0.3; Cr<sub>2</sub>O<sub>3</sub>-0.01; TiO<sub>2</sub>-0.012; Na-0.015; K-0.01; H<sub>2</sub>SO<sub>4</sub>-10-15; CaF<sub>2</sub>-3; HF-0.3.

The crystal structure consisted of finely dispersed translucent idiomorphic anhydrite crystals 5–10  $\mu$ m in size. The average refractive index was N = 1.57. HF, fluorite CaF<sub>2</sub>, and

iron hydroxides were observed as impurities. The total anhydrite content was about 95%. The phase composition of the gypsiferous waste was determined by XRD. The acidic waste was identified as  $CaSO_4$ -calcium sulfate. The neutralized waste was an anhydride of an orthorhombic structure.

The water content of the anhydrite binder was determined using a Vick's pestle instrument.

The neutralization of the waste was carried out using dry limestone until a pH of ~9 was reached. Limestone was introduced in powdered form. The specific surface area of the limestone was determined, because it is the most complete characteristic of the dispersion of powders.

# 3. Results and Discussion

#### 3.1. Obtaining Anhydrite Binders

In order to convert an acidic waste (fluorine hydride) into an anhydrite binder, it is necessary to carry out its neutralization. It is known that the neutralization of acids and other harmful impurities contained in man-made waste is carried out by dry or wet methods with a reagent containing lime (lime flour, lime milk, limestone, dolomite, etc.) [8].

In carrying out this work, limestone from the Sazhaevskoye field (a cheap, almost unlimited reserve from the East Kazakhstan region) was used as a neutralizing reagent (Table 1).

Table 1. The standard chemical composition of limestone.

Description of Parameter	Range
Mass fraction of calcium and magnesium oxides (CaO + MgO), $\%$	52–55
Mass fraction of magnesium oxide (MgO), $\%$	0.5–7
Mass fraction of silicon dioxide (SiO <sub>2</sub> ), $\%$	0.5–2.5

The anhydrite binder was obtained according to the following scheme:

- 1. The neutralization of phthoranhydrite waste by the dry method;
- 2. The additional neutralization of the obtained mixture by mechanical activation.

## 3.2. Neutralization of Fluorohydrate Waste

The technological scheme of waste neutralization is shown in Figure 1.



Figure 1. Technological scheme of fluorogypsum neutralization.

The initial raw material (fluorine gypsum) was delivered from the shop for the production of hydrofluoric acid by a special truck. Large amounts of feedstock should not be accumulated prior to processing, owing to the release of toxic sulfuric acid fumes. Under the proposed scheme, raw materials were unloaded into the hopper of feedstock, from which they were transported by an elevator to the receiving hopper of the chain of technological equipment for the preparation and neutralization of fthrogyps, conducted according to the cascade scheme. The raw materials were subsequently disintegrated and ground to a particle size of less than 10 mm and then moved by a screw to the fine-grinding machinery to reach a particle size of less than 0.2 mm and pelletized in a vibration apparatus. As a result of neutralization, a granular product of fluorine gypsum and limestone mixture emerged from the vibrating milling–mixing–pelletizing unit. The obtained granules, 5 to 25 mm in size, were fed by the conveyor into the intermediate storage tanks (silos) in the amount required daily. The bulk of the free sulfuric acid (>95%) was bound in the limestone during mixing and granulation. The remaining, unreacted acid was neutralized inside the pellets within 3 to 5 h.

For all operations, crushed limestone was fed into the feedstock to neutralize the excess sulfuric acid. The reaction produced calcium sulfate, water, and carbon dioxide. The resulting water was in a semi-hydrous, two-water crystalline hydrate form. The entire reaction can be expressed as:

$$H_2SO_4 + CaCO_3 \Rightarrow CaSO_4 \cdot 0.5H_2O + CaSO_4 \cdot 2H_2O + CO_2 + Q_{dissipation}$$
(1)

When carrying out the neutralization process in industrial volumes, the thermal effect of the reaction leads to the heating of the neutralized mixture to a temperature of 60–70  $^{\circ}$ C, which must be considered at the design stage.

To carry out the neutralization process effectively, the lumpy limestone was first crushed to a particle size of less than 0.2 mm using a disintegrator.

The limestone was fed into the system, with a small degree of excess, for neutralization at a rate of 1.5 wt.% of limestone per 1 wt.% of sulfuric acid.

Protective equipment was required, because the 10-15% content of sulfuric acid created a strongly acidic environment (pH = 0.43), and the "traces" of hydrofluoric acid produced a stifling smell.

Specific surface area determination by air permeability is the most common method for the dispersion analysis of powders. It is widely used in scientific research and especially for the control of production processes.

Studies have established the influence of limestone dispersibility on the completeness of neutralization reactions [9,10]. Thus, increasing the specific surface area of limestone from  $4000 \text{ cm}^2/\text{g}$  to  $9000 \text{ cm}^2/\text{g}$  led to an increase in pH (Figure 2).

The figure shows that the optimum dispersity was around  $6000 \text{ cm}^2/\text{g}$ , as a further increase in the specific surface area of limestone only slightly increased the pH and led to additional energy costs, which is not rational in economic terms.

Experiments were carried out with different amounts of limestone in order to establish the necessary pH level. The optimal amount of limestone was determined to be 20% (Figure 2). The use of limestone in amounts less than 20% led to a further increase in the consumption of lime, which was used in the next step to adjust the setting time.

When acidic waste was mixed with finely ground limestone, a chemical reaction (exothermic) occurred between the sulfuric acid and the limestone, which is described by the following formula:

$$H_2SO_4 + CaCO_3 = CaSO_4 + CO_2\uparrow + H_2O + Q_{dissipation}$$
(2)

During the reaction, carbon dioxide was released, and the temperature of the mixture rose to 70–80 °C. The end of the neutralization reaction was fixed by reducing the temperature of the mixture to room temperature. At the same time, it was necessary to ensure the removal of carbon dioxide.



Figure 2. The dependence of the pH of the mixture on the amount of limestone.

Water formed as a result of the reaction led to the wetting of the resulting mixture, so the mixture had to be dried to a humidity of no more than 1%.

The operation of drying the mixture to a moisture content of 1% was a necessary step in preparation for its further use.

#### 3.3. Fine Neutralization of a Mixture of Fluorohydrite Waste with Limestone

The resulting mixture was not fully neutralized, because the reaction was inhibited by the granular structure of the waste (the neutralization process occurred on the surface of the granules) and the fact that these granules were covered with a film of calcium fluoride. Therefore, additional neutralization was carried out by mechanical activation, i.e., the fine grinding of the dried mixture. The dispersity of the mixture was controlled by a set of indicators: the achievement of minimum values of water consumption and setting time.

During grinding, the water vapor had an effect on the grinding of the mixture (the release of water vapor was associated with the continuation of the neutralization).

If the wet mixture was not dried after the preliminary neutralization step, some of the powder adhered to the grinding bodies during mechanical activation. This was due to the increased water content, which bound the highly dispersed particles together in thin layers and hence significantly reduced the efficiency of further grinding.

Thus, the necessity of drying the mixture to a moisture content of not more than 1% after the preliminary neutralization step was confirmed.

In this regard, further studies on the effect of various surfactants on the dispersion (grinding) of limestone, lime, and neutralized waste in order to increase the productivity of grinding equipment are of interest.

The experiments showed the importance of the gradual addition of limestone in all operations. With the right choice of regime, the output was a granulated neutral gypsum with acceptable free-flowing properties.

The properties of the obtained anhydrite binder were as follows:

- Specific surface area—9000 cm<sup>2</sup>/g;
- Setting time:
  - 1. The beginning of setting—19 h 55 min;
  - 2. The end of setting—26 h 46 min.

According to [11], the greatest activation of anhydrite binders occurs at pH = 7-9. Our studies established that the greatest activation for fluorine hydrite waste occurred when the pH of the mixture was increased to 12. In an alkaline environment, the formation of

basic salts and the increased hydrolysis of calcium sulfate are the determining factors in the process of hydration.

To this end, we proposed the use of an alkaline activator for hardening the quicklime.

3.4. Activation with Burnt Lime

The activation of the anhydrite binder was carried out as follows:

- 1. The anhydrite binder was mixed with quicklime for 1 min (without kneading).
- 2. The introduction of burnt lime shortened the setting time (Table 2) and also gave the samples a white color.
- 3. The water consumption of the mixture was 35–36 %.

**Table 2.** Effect of quicklime on setting time.

Limestone Content, %	Content of Burnt Lime, %	pH of the Mixture –	Setting Time, h:min	
			Start	End
20	-	9.0	19:55	26:46
20	2	9.2	2:30	4:30
20	10	10.1	0:27	3:45
20	20	12.0	0:16	0:48

As can be seen from the table, the setting time was markedly reduced when the pH level was increased.

Considering the effect of the amount of quicklime on anhydrite binder activation, the studies determined that the minimum setting times were achieved when adding lime in an amount of 20% (Table 1) at a pH of 12. Increasing the amount of lime led the pH to exceed 12, which also reduced the solubility of Ca(OH)<sub>2</sub>.

It should be noted that the use of prequench lime, as shown by further studies, led to an increase in the setting time of the anhydrite binder (Table 3).

Table 3. Influence of hydrated lime on setting time.

Limestone Content, %	Content of Burnt Lime, %	Water Consumption of the Mixture -	Setting Time, h:min	
			Start	End
20	-	9	1:10	4:00
20	20	33	0:49	1:35
20	20	35	0:58	1:51
20	20	36	1:00	2:10

In the course of research, the dependence of the setting time of the anhydrite binder on the mixing water temperature, water consumption, and the specific surface area of the binder was established.

#### 3.5. Study of the Effect of Mixing Water Temperature

In the production process of anhydrite binders, we proposed the use of burnt lime. The tests revealed the effect of the mixing water temperature. The results of the tests are shown in Figure 3.

As can be seen from the figure, the minimum setting time was fixed when using a water temperature of 5–8 °C. The use of water with a temperature of 12 to 40 °C increased the setting time by approximately twofold. When the water temperature was further increased, the setting time increased dramatically. This was associated with a decrease in the solubility of Ca(OH)<sub>2</sub>. Therefore, we suggest that, in binder production, a water temperature no higher than 8 °C should be used.



Figure 3. The dependence of the end of anhydrite binder setting on the temperature of the mixing water.

# 3.6. Determination of Water Consumption of Anhydrite Binder

It should be noted that such a short anhydrite binder setting time would not allow the use of casting technology for product manufacturing. The determination of the plasticity of the dough was performed by applying a Vick's device with a pestle (normal dough density indicated by "0"); the results are shown in Figure 4.



Figure 4. Dependence of setting time on the plasticity of the dough.

The figure shows that the shallower the depth of the immersion of the pestle in the dough, the faster the setting of the binder. However, reducing the amount of water affected the workability of the dough in the molds, so the optimal amount of water was 35% when using vibration compaction.

# 3.7. Study of the Effect of Anhydrite Binder Dispersion on the Setting Time

The experiments showed that in order to ensure the required setting time, it was necessary to ensure the fine grinding of the anhydrite binder and lime until the specific surface area of the mixture was  $9000 \text{ cm}^2/\text{g}$  (Figure 5).



Figure 5. Dependence of the setting time on the dispersity of the anhydrite binder and lime mixture.

Figure 5 shows that a further increase in the specific surface area to more than  $10,000 \text{ cm}^2/\text{g}$  led to an increase in water demand up to 50% and, correspondingly, an increase in the setting time.

Because anhydrite binder activation with lime alone did not provide the required setting time (30 min to the end of setting), it was necessary to carry out further activation. The initial characteristics of the binder for further activation were as follows:

- Mixture composition: 60% acidic fluorangydrite, 20% limestone, 20% quicklime;
- pH = 12;
- Setting time: beginning—16 min, end—48 min.

# 3.8. Activation of Fluorohydrite by Various Chemical Additives

It is known that after the addition of small amounts of certain salts, insoluble anhydrite (natural or artificially produced) acquires the ability to hydrate. The hardening process of anhydrite binders consists in the hydration of insoluble anhydrite and its subsequent recrystallization [11]. In the presence of water and a catalyst on the surface of anhydrite particles, an unstable complex hydrate is formed according to Formula (3):

$$mCaSO_4 + (salt) nH_2O = (salt) mCaSO_4 nH_2O$$
(3)

This then disintegrates to form two-water gypsum according to Formula (4):

$$(salt) mCaSO_4 \cdot nH_2O \rightarrow (salt) H_2O + CaSO_4 \cdot 2H_2O$$
(4)

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. At this time, setting occurs. Recrystallization processes lead to hardening. In the first few hours, the strength of the anhydrite binder increases; then comes a certain decline in strength, which can be explained by the fact that the unstable complex hydrate decays at this point; next, the strength increases again for quite a long time. As a result of curing, the amount of bound water in the products gradually increases.

According to existing studies, intermediate compounds between sulfates and anhydrites are not formed, and the accelerating effect of additives is due to other reasons [8]. Some researchers believe [12] that the activating effect of lime, caustic dolomite, and other insoluble compounds that present crystallochemical similarity with gypsum is due to the additive particles serving as crystallization centers. Some researchers believe that acidic anhydrite hardening activators increase its solubility and chemical potential in the early stages of hardening [13]. According to the chemical composition of the additives introduced, the following types of activation are used:

- Sulfate activation (using alkali and heavy metal sulfates and acid sulfates);
- Alkaline activation (Ca(OH)<sub>2</sub>, NaOH);
- Mainly alkaline activation (Portland cement clinker, blast furnace slag, Portland slag cement, etc.);
- Combined activation (alkaline component and sulfate additive).

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

In carrying out this work further, the following were used as activating agents:  $((K_2SO_4), copper sulfate (CuSO_4), crystalline hydrate of iron sulfate (FeSO_4·7H_2O), alumina aluminate, Na<sub>2</sub>CO<sub>3</sub>, and NaCl. According to the analysis of literature data, the content of the additives was taken as 0.5–3% of the mass of dry matter in increments of 0.5%. All additives were introduced with mixing water both separately and in combination. The used additives, in addition to activation of hardening, also affected other physical and mechanical properties of the binder [9].$ 

#### 4. Conclusions

Analyzing the obtained data, the following conclusions were drawn:

- 1. By studying the characteristics of man-made wastes of hydrofluoric acid production, their potential suitability for the production of anhydrite binders and building materials was shown.
- 2. The influence of the limestone dispersity on the completeness of the reaction of neutralization was established. Thus, increasing the specific surface area of the limestone from  $4000 \text{ cm}^2/\text{g}$  to  $9000 \text{ cm}^2/\text{g}$  led to an increase in the pH.
- 3. It was determined that the greatest activation for fluorohydrite waste occurred when the pH of the mixture was increased to 12.
- 4. In the course of the research, the dependence of the anhydrite binder setting time on the mixing water temperature, water consumption, and the specific surface area of the binder was determined.
- 5. Experiences in the production of construction materials from anhydrite binders were analyzed using sources of technical information.

Our further research will be aimed at determining the strength characteristics of fluorangydrite, the development of industrial technology for obtaining products, the calculation of the material balance of the technological process of neutralization, and the process of the production of gasketed slabs.

**Author Contributions:** Z.S., B.R. and M.K. designed the experiments; A.K., M.A. and S.A. performed the experiments; B.R., Z.S. and M.K. analyzed the data; Z.S., L.B. and B.R. wrote, reviewed, and edited the paper. All authors have read and agreed to the published version of the manuscript.

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