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# Fluoroanhydrite based composites with the thermoplastic additive

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**Abstract.** The article represents the results of using industrial sulfur as an additive to a fluoroanhydrite based binder and the estimation of its influence on mechanical and physicochemical properties. Waste generated by human activities on an industrial scale, such as industrial sulfur and fluoroanhydrite, pose a serious environmental problem in terms of storage and disposal. Moreover, industrial sulfur and fluoroanhydrite have particular properties to form composite material with required properties. A number of studies have been carried out on using industrial waste as components of building materials, performance properties of the products obtained being improved and the functional use of the products being expanded. In order to study changes in the mechanical properties and physicochemical composition of material based on synthetic fluoroanhydrite, conventional testing methods accompanied by modern methods including scanning electron microscopy and X-ray analysis and infrared spectroscopy, were used. According to the obtained results compressive strength of composition modified with 10 % of thermoplastic additives was 35.77 MPa, water resistance was 0.68. This increase in mechanical properties is due to an interaction between chemically reactive polymorphic types of sulfur which are formed by transformation of α- type to β- and fluoroanhydrite binder. The results of the presented study prove the possibility of creating a building material, the composition of which is fully represented by industrial waste and the characteristics of which are not inferior to its analogues in terms of technical and economic properties.

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## *1. Introduction*

The world oil and gas processing complex annually produces more than 70 million tons of industrial sulfur in the form of solid and liquid waste. In addition, this waste is generated by the activities of various sectors of the mining and chemical industries. Currently, conducting scientific research in the field of finding new methods of disposal and reducing the level of environmental impact from by-products is of great importance [1, 2].

Sulfur compositions are used in various sectors of the construction industry, in industrial and transportation engineering. Sulfur-based composite materials are used mainly in those conditions where traditional materials based on Portland cement quickly lose the required properties, for example, when operating in acidic and alkaline environments [3–5]. Varying the content of industrial sulfur in the composition of traditional compositions made it possible to achieve an increase in a number of physicotechnical and physicochemical properties of products (increased impact strength, heat, electrical, and sound insulation, shielding from natural radiation and anthropogenic electromagnetic background) [6].

In addition, it is necessary to consider that, according to expert estimates, more than 300 million tons of synthetic gypsum are produced annually in the world, 20 million tons of which are fluorogypsum, titanium gypsum, etc., and the rest is phosphogypsum. In recent years, the annual increase in the volume of gypsum-containing by-products of the chemical industry is on average 7–8 %. Currently, synthetic gypsum is used in small quantities in agriculture and insignificantly in construction; the main part of gypsum-containing waste is usually stored in open dumps or dumped into water bodies [7, 8].

The limited use of fluoroanhydrite is due to the low quality of products made directly from raw synthetic materials [9, 10]. Most of the research in the field of using fluoroanhydrite has been focused on varying the mechanical treatment and mechanical activation of the binder as well as on selecting effective curing catalysts and various mineral modifying additives [11–13].

As practice shows [14], the use of additives containing sulfate ions can accelerate the setting time of a synthetic binder by increasing the solubility of calcium sulfate. Sulfates do not affect the water demand of the fluoroanhydrite binder, the crystallization of calcium sulfate from the solution accelerating, which has a significant effect on the rate of formation of the nuclei of the hydrate phase [15].

Many Russian and foreign researchers [15–17] have found that sodium sulfate has a significant effect on the setting time of sulfate-containing binders ($Na_2SO_4$). Research has shown [18] that with the amount of sodium sulfate exceeding 4 %, the setting time of the binder is shortened due to an increase in the solubility of anhydrite. Some research results [19] show that for the production of dry mortars based on fluoroanhydrite binder a dosage of 2–3 % is recommended.

Products based on fluoroanhydrite are known for their fire resistance and heat and sound insulation properties. At the same time, due to its low water resistance and mechanical strength, the use of fluoroanhydrite is limited in the production of materials and structures for humid conditions [20].

A review of literary sources has demonstrated insufficient information on the issue of the integrated use of industrial sulfur, sulfate activator, and synthetic anhydrite in the production of composite building materials.

The scientific research of rational ways of recycling industrial wastes such as industrial sulfur and fluoroanhydrite is a paramount importance in case of preserving sustainable environment [2, 3].

Currently, the main direction of research in the field of the use of synthetic gypsum is the development of methods for increasing the hydrophobic properties of products [21, 22]. The need for this research is also driven by the demand in developing countries' markets for quality and cost-effective building materials.

The main aim of the research is to design proper composite material based on fluoroanhydrite activated by water solvable additive and thermally activated industrial sulfur.

Following tasks should be achieved during this research:

A. To estimate the proper use of man-made waste materials and find out the way of adding sulfur contained waste for composite formation.

B. To research characteristics of sulfur behavior under temperature treatment and following cooling.

C. To estimate the influence of thermoplastic additive on mechanical and performance characteristics.

# 2. Methods

## 2.1. Raw materials and experiment method

To study the effect of industrial sulfur on the physicochemical and physicomechanical properties of products based on synthetic anhydrite (fluoroanhydrite), a series of experiments was carried out to modify the binder with a pretreated dispersed sulfur additive.

Thermoplastic sulfur additive was obtained as a result of a two-stage process described in the studies [23]. The processed industrial sulfur was introduced at the stage of mixing the main components, and the hardening activator was introduced together with the mixing water. The resulting mixture was put into metal molds and removed after 2 hours, followed by heat treatment of the samples.

**Fluoroanhydrite.** Fluoroanhydrite (from Galogen LLC, Perm) was used in the study. This is a homogeneous low-density material with a constant chemical composition, whose particle sizes range from 1 to 20 mm. The raw material is laboratory-milled and sifted through a 0.4 mm sieve until less than 13 wt% residue is left, producing a white powder. In the process of mixing with water until standard consistency is reached (spreading on the Suttard viscometer 180 mm), it forms a dough of normal density at W/G = 0.33. Setting times are: start 0 h 21 min. end 0 h 41 min. The component composition is presented in Table 1.

***Table 1. Component composition of fluoroanhydrite, %.***

| $CaSO_4$ | | $CaF_2$ | $H_2SO_4$ | HF |
|---|---|---|---|---|
| $\gamma$ – $CaSO_4$ | $\beta$ – $CaSO_4$ | | | |
| 20 | 78 | 1 – 1.8 | 1 – 1.2 | – |

Sodium sulfate. Based on the research results [18], sodium sulfate corresponding to Russian State Standard GOST 21458-75, crystallization sodium sulfate, was taken as a hardening activator. The optimal introduction rate was determined based on researches [18].

Industrial sulfur. Industrial sulfur of grade 9998 corresponding to Russian State Standard GOST 127.1-93 was used as the main component of the thermoplastic sulfur additive. The content of the thermoplastic additive varied in the range from 0 to 10 % by weight of the binder, in increments of 2 %. The concentration range of sulfur additive was selected in accordance with the hypothesis discussed earlier in [24]. Physicomechanical and physicochemical properties are presented in Table 2.

***Table 2. Specifications of sulfur, grade 9998 [24].***

| Name | Particle shape | Bulk density | Mass fraction of sulfur | Mass fraction of ash | Mass fraction of organic matter | Mass fraction of water |
|---|---|---|---|---|---|---|
| Unit | – | $g/cm^3$ | % | % | % | % |
| Value | Hemispherical | 1.3 | 99.99 | 0.005 | 0.005 | 0.01 |

### *2.2. Experiment method*

In order to study changes in the physicochemical composition of artificial stone based on synthetic fluoroanhydrite, both scanning electron microscopy and additional informative research methods, including X-ray analysis and infrared spectroscopy, were used. Scanning electron images of the microstructure were obtained using a Thermo Fisher Scientific Quattro S scanning electron microscope at the “Surface and New Materials” shared knowledge center at the Udmurt Federal Research Center of the Ural Branch of the Russian Academy of Sciences. Infrared spectra were obtained using an IRAffinity-1 spectrometer. Images and spectra are presented without additional processing (except for adjusting the brightness and contrast of images). The compressive and flexural strength was measured using a PGM-100MG4-A hydraulic press.

To study the complex changes in the physicochemical composition, the method of differential thermal analysis of samples of the control and modified composite material modified with a thermoplastic additive was used, including thermogravimetric analysis (TGA). In addition, differential thermogravimetry (DTG) and differential scanning calorimetry (DSC) methods were used. Laboratory studies were carried out on a TGA/DSC1 thermal analyzer produced by Mettler-Toledo Vostok. Shooting conditions: measurement interval 50–1100°C, heating rate 10 deg/min, platinum crucibles, working medium – air.

## *3. Results and Discussion*

Comparative evaluation of the samples was carried out according to changes in the compressive and flexural strength of the modified and control compositions. The composition containing fluoroanhydrite and 2 % of the catalyst by weight of the binder was taken as a control sample.

The tests were carried out on beams with dimensions of 40×40×160 mm. The samples were kept under normal conditions for 2 hours after stripping, then placed in a drying cabinet 80-01 SPU for heat treatment at 180 °C for 60 minutes [25]. At the same time, to prevent dehydration of water in the mineral matrix, the samples were sealed during the whole time of heat treatment. After the heat treatment, the samples were cooled to room temperature in a drying oven. Afterwards, the samples were kept under normal conditions for 7, 14, and 28 days; the required characteristics were measured on the control dates. The experimental compositions are presented in Table 3.

***Table 3. Component composition of produced samples.***

| Composition | Fluoroanhydrite, g | Dosage of catalyst, % | Industrial sulfur, % | Treatment temperature(°C) |
|---|---|---|---|---|
| Control | | | 0 | |
| C-1 | | | 2 % | |
| C-2 | 1200 | 2 | 5 % | 180 |
| C-3 | | | 7 % | |
| C-4 | | | 10 % | |

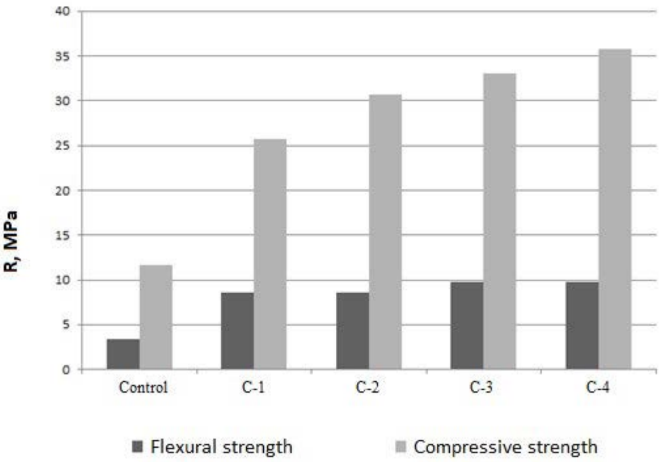


**Figure 1. Strength of modified fluoroanhydrite-based products at 28 days (Note to Fig. 2. The value of the coefficient of variation in determining the strength of the hardened binder samples was V = 4.5 %).**

The index of compressive strength with the introduction of 10 % of thermoplastic additive on the 28th day of hardening is on average 35.77 MPa (Fig. 1), which significantly exceeds the strength indicators of the control composition. Water resistance determined by the value of the softening coefficient of the fluoroanhydrite composition on the 28th day of hardening was 0.68 % for the composition C-4. Results are also higher than the indicators reported in studies [8, 12, 13].

As the study of physical and mechanical properties show, the optimal dosage of modified industrial sulfur is 10 %. At the same time, the dynamic of the growth of mechanical characteristics makes it possible to increase the concentration of sulfur in the composition, counting on a further increase in performance indicators; however, for the purposes of this study, the optimal content of the additive is 10 %.

At the same time, the increase in strength is due to the conditions of chemical interaction between various reactive polymorphic modifications of sulfur formed as a result of the transition of α sulfur to β sulfur with components of the fluoroanhydrite structure during its thermal activation in the matrix structure. It should also be noted that the growth of the physical and mechanical parameters of the material is due to the formation of polymer sulfur during polymerization in the temperature range from 70 °C to 206 °C. The extremum is observed at 189 °C. The transition energy corresponds to $\Delta E_p$ polymerization – 19.35 J/g. Previous research [1, 5, 6, 25] confirms this.

When comparing the microstructure of the control and the modified samples, dense, homogeneous formations were found in the structure of the modified artificial stone with an increased adhesion of the anhydrite matrix to the polymer component observed.

Structural changes are shown in Fig. 2, and the analysis of the modified sample revealed the formation of an amorphous and dense structure, in which the content of the crystalline typical phase of hydrated fluoroanhydrite is practically absent. In addition, the energy dispersive X-ray spectroscopy of the control and modified sample of the composition C-4 with reference to the analyzed area presented in Fig. 2 and 3 demonstrates the comparative changes in the component composition presented in Table 4.

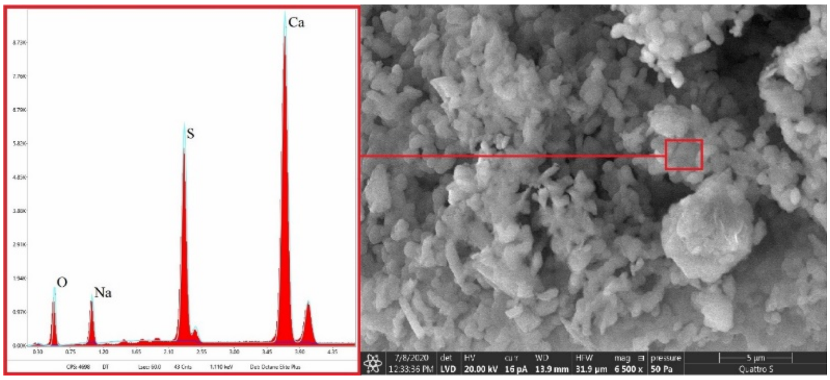


**Figure 2. Microanalysis and microstructure of the control sample.**

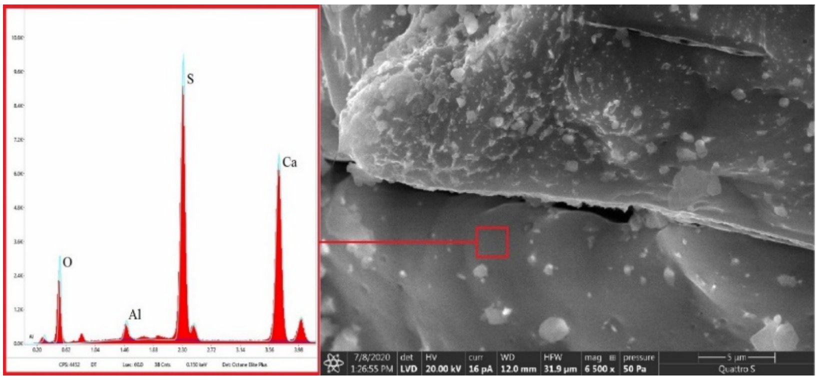


**Figure 3. Microanalysis and microstructure of the modified sample (sample C-4).**

The elemental composition shows an increased intensity reflecting the content of sulfur, which may presumably indicate its structuring role in the component composition.

***Table 4. Component composition of the control and modified samples according to the results of X-ray spectroscopy.***

| Element | C-4 | | | | Control | | | |
|---|---|---|---|---|---|---|---|---|
| | Weight % | Atomic % | Error % | Net Int. | Weight % | Atomic % | Error % | Net Int. |
| O | 54.5 | 72.5 | 11.4 | 227.97 | 0.0 | 0.0 | 0.0 | 124.1 |
| Al | 1.9 | 1.5 | 10.3 | 75.8 | – | – | – | – |
| Na | – | – | – | – | 15.2 | 22.6 | 10.9 | 126.6 |
| S | 20.96 | 13.9 | 4.8 | 1389.8 | 24.2 | 25.8 | 5.4 | 863.2 |
| Ca | 22.7 | 12.1 | 3.6 | 1141.5 | 60.6 | 51.6 | 3.7 | 1642.1 |

Analysis of the IR spectra of the control and modified compositions (Fig. 4) showed that with the addition of the modified industrial sulfur, the nature of the peaks of the main functional groups of the material changes and new wavenumbers appear (Table 5). This may be due to the formation of new polymer forms of sulfur and the change in the anhydrite crystallization conditions.

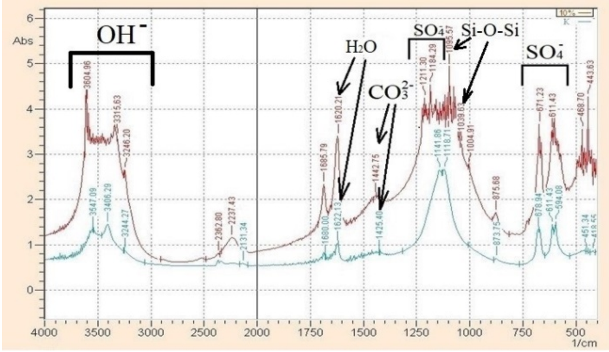


**Figure 4. Comparison of IR spectra of the control (red) and modified (turquoise) samples with the introduction of 10 % of industrial sulfur.**

***Table 5. Change in wavenumbers when comparing infrared spectra.***

| Ion | Wavenumbers cm$^{-1}$, control sample | Wavenumbers cm$^{-1}$, modified sample |
|---|---|---|
| $SO_4^{2-}$ | 594.08; 611.43; 679.94;1141.86; 1118.71 | 611.43;671.23;1184.29;1211.30 |
| $CO_3^{2-}$ | 1442.75; 873.5 | 1425.40; 875.68 |
| Si-O-Si | – | 1095.57; 1039.63 |
| $OH^-$ | 3547.09; 3406.29; 3244.27 | 3604.96; 3315.63; 3246.20 |
| $H_2O$ | 1620.21 | 1622.13 |

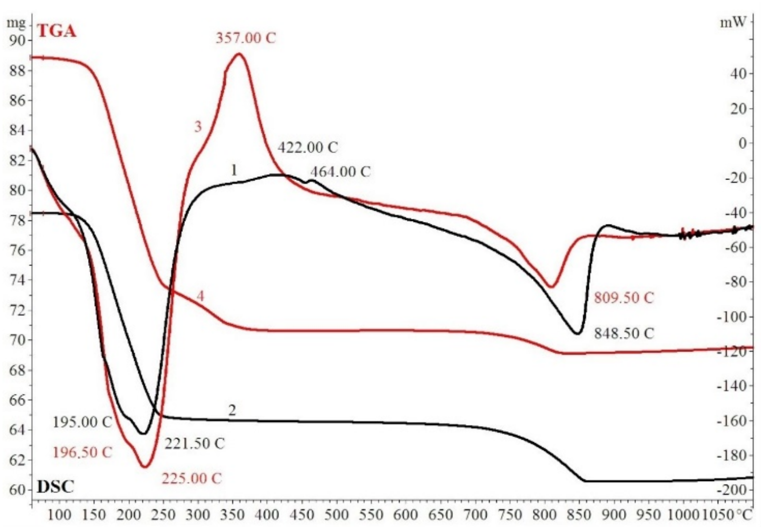


**Figure 5. Differential scanning calorimetry spectra: control sample (1, 2), C-4 sample of modified industrial sulfur (3, 4).**

The derivatogram of the control sample (Fig. 5, isotherms 1 and 2) of fluoroanhydrite with a hardening activator shows a double endothermic effect of 195 and 221.5°C typical for the removal of crystallization water as well as an endothermic effect of 848.5, the area typical for calcite dissociation into calcium oxide and carbon dioxide and partial dissociation of calcium sulfate and low-basic calcium hydrosilicates in an insignificant volume. The effects in the range of 420–470°C are associated with the rearrangement of the crystal lattice with the formation of insoluble anhydrite. Fluoroanhydrite, in its turn, is represented by a combination of anhydrite, calcium carbonate, and gypsum.

When modifying fluoroanhydrite with a hardening activator and 10 % (Fig. 5, isotherms 3 and 4) of processed industrial sulfur, a slight endothermic effect was noted in the region of 90–150°C associated with a change in the phase state of sulfur, namely, the transition from the alpha form to the beta form. A shift in temperatures corresponding to the removal of crystallization water up to 196.5 and 225.0 degrees, respectively, was noted, which indirectly confirms the change in the hydration conditions of fluoroanhydrite in the direction of acceleration and completeness of the reaction. A strong exothermic effect was noted in the temperature range of 340–370°C, which is associated with the burnout of sulfur in air with the formation of $SO_2$ and $SO_3$, while the effect manifests itself in a significant volume smoothing out the concomitant effects of the rearrangement of the crystal lattice of the binder, preventing them from being identified. An endothermic effect in the region of 800°C associated with the dissociation of calcium sulfate and low-basic calcium hydrosilicates was also noted.

Thus, the spectral data are consistent with the results of the microstructural analysis, indicating the formation of a matrix of increased density including amorphous structures based on the sulfur component. The change in the size and intensity of the peaks of the main wave numbers and/or typical reactions confirms the hypothesis about the influence of a man-made additive on the conditions for the structure formation of fluoroanhydrite.

# *4. Conclusions*

The influence of industrial sulfur on the structure formation of the mineral binder has been analyzed. The results of the research are as follows:

1. As a result of the research it was found that the composite material based on industrial sulfur (Taneco, PJSC) and fluoroanhydrite binder (Galogen, Ltd) has the increased physical and technical indicators. With 10 % of modified industrial sulfur introduced, the flexural strength increases by 55.6 %, the compressive strength by 2 times, and the softening coefficient is 0.68 %.

2. The introduction of a thermoplastic additive based on industrial sulfur has a significant effect on the physicochemical properties of artificial stone-based man-made anhydrite. A comparative analysis of the results of energy dispersive X-ray spectroscopy showed that in the process of modifying the anhydrite matrix, a high-density amorphous structure is formed possibly due to changes in hydration conditions confirmed by the results of IR spectral analysis and the appearance of oscillations in the region of 1184.29 and 1211.30 wavenumbers corresponding to $SO_4^{2-}$.

3. The results obtained confirm the possibility of rational use of man-made waste and the prospects for thermal activation of sulfur in the structure of the mineral matrix in order to improve the performance characteristics and expand the area of application of products based on man-made anhydrite.

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