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Production of Portland cement using fluorine gypsum – hydrofluoric acid waste

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Abstract. Introduction. The purpose of the research was to explore the possibility of making PC400–D20 Portland cement, as the gypsum stone was replaced with fluorogypsum during the milling process. The study deals with physical and mechanical properties of Portland cement obtained by mixing milled Portland cement clinker, fly ash (consisting of nanosized particles of SiO_2 and Al_2O_3) and sulphate additives (gypsum stone or fluorogypsum). Methods. In accordance with the quality indicators set by Russian State Standard GOST 4913–82 (EN 13279–2), Angarsk Electrolysis Chemical Plant (JSC AECP) fluorogypsum is significantly superior to the gypsum stone mined in the Nukutsky Gypsum Quarry (Irkutsk Region). The use of high-quality fluorite concentrate in the production of the hydrofluoric acid can be accounted for exceptional characteristics of fluorogypsum found at the sludge fields of JSC AECP. Results and Discussion. The composition of sludge fields of Angarsk Electrolysis Chemical Plant has been investigated. The quality of its main component, fluorogypsum, has been determined with accredited classification. The studies of chemical composition and physical properties of fluorogypsum deposited at the sludge fields of JSC AECP proved that it can serve as a first-grade raw material in accordance with Russian State Standard GOST4913–82 for the production of high-grade gypsum. Laboratory tests confirmed the practicability of using fluorogypsum as a substitute for natural gypsum stone in cement production process (when grinding cement clinker). A technology and parameters for the production of high-grade gypsum production from the fluorogypsum have been developed. Conclusions. Laboratory tests confirmed the possibility of using fluorogypsum as a setting regulator in cement production when grinding cement clinker. The exact quantity of fluorogypsum when grinding cement clinker was determined, which ensures the production of high-quality cement in compliance with Russian State Standard GOST 10187–85. The prospects of using fluorogypsum as a raw material for the production of high-quality gypsum as well as a wide range of building mixtures based on it has been identified.

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1. Introduction

Millions of tons of solid by-product materials are produced every year by chemical industries all over the world. The accumulation of these materials causes substantial societal costs for containment and disposal, including environmental pollution and related economic losses [1]. Therefore, finding new beneficial applications for these large reserves of unused and/or underutilized materials is of great interest and provides important opportunities for sustainable economic development. At the same time, the construction industry is always searching for alternative supplies of usable materials in order to curb its carbon footprint, reduce the cost of new projects, and ensure long term sustainability of the industry itself.

Gypsum binders have increasingly been used in the construction industry due to their relatively low production energy intensity as well as such important properties as short curing time, which, in turn, reduces the duration of technological operations and ensures good formability and workability of the material, volume stability, and no necessity for compaction. Gypsum chemical composition is non-toxic, so its processing does not cause carbon dioxide emissions into the environment [1–3].

Gypsum raw materials are extracted predominantly from open pit mines. Gypsum mining enterprises are mainly large, highly mechanized industries whose production volume reaches 2 million tons of stone per year. The stone is usually shipped to the consumer after primary crushing and grading into 0–300, 0–60, and 60–300 mm fractions. Unfortunately, gypsum deposits in quarries and mines are not subjected to enrichment.

Gypsum-containing waste, including phosphogypsum, borogypsum, fluorogypsum, as well as wastes resulted from citric acid production in chemical, food and other manufacturing, represents an important part of the raw material base in the gypsum industry of the country.

Hydrofluoric acid was a key product of Angarsk Electrolysis Chemical Plant (JSC AECP). The production process was commenced in the fifties of the last century and had successfully been conducted up until 2015. However, due to the logistical concerns, the line for hydrofluoric acid production was closed by the top-management of Rosatom State Atomic Energy Corporation.

Hydrofluoric acid was produced by treating fluorite CaF_2 with sulfuric acid in an externally heated rotary kiln at temperature of 450–500 °C ($CaF_2 + H_2SO_4 \rightarrow CaSO_4 + 2HF$). This way, fluorogypsum or industrial gypsum ($CaSO_4$) was formed as a by-product. Upon undergoing the lime neutralization, fluorogypsum was stored in the form of calcium sulphate dihydrate $CaSO_4{\cdot}2H_2O$ in the sludge dumps on the territory of JSC "AIECP". The diffractogram of the fluorogypsum sample taken from the sludge dumps shows peaks indicative of calcium sulphate (Fig. 1). The peaks pointing at fluorite are represented in fewer numbers (2θ = 27.3°; 55.2; 61.3; 68.8°) and are characterized by low intensity.

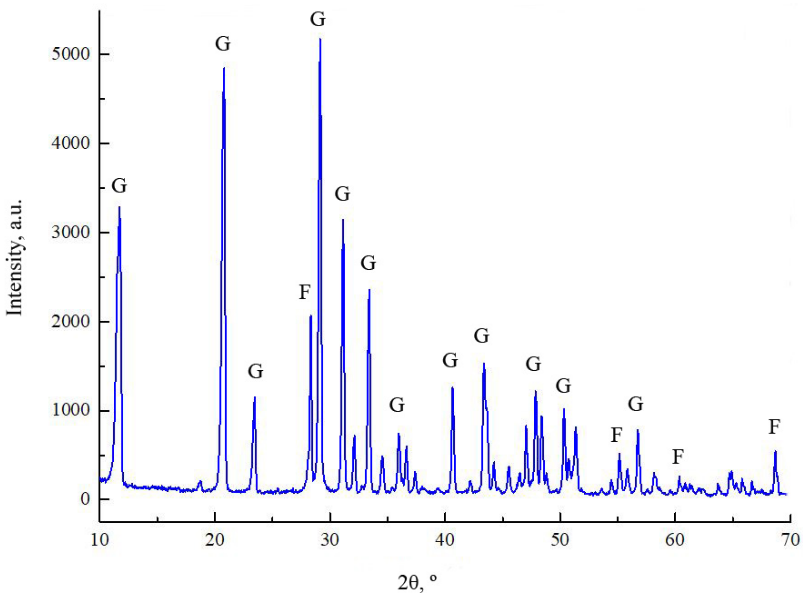

Figure 1. The diffractogram of the fluorogypsum sample taken from the sludge dumps of JSC AECP (G – gypsum $CaSO_4{\cdot}2H_2O$; F – fluorite CaF_2).

During the production process, up to a total millions of cubic meters of the material was accumulated. It remained discarded of a long time (Fig. 2) owing to a misconception that fluorogypsum might be radioactive (due to the activity of the enterprise). Therefore, the first stage of the studies was the assessment of radioactivity of the sludge fields. During the course of the research, several samples were collected from the different points of the mass.

Figure 2. Sludge Dumps of JSC AECP.

According to the results of the radiation safety monitoring, administered in the certified laboratory pursuant to the existing standards and specifications, including Russian State Standard GOST 30108–94, fluorogypsum was recognized as a first class radiation-proof material (SA = 88 < Bq/kg) suitable for all types of construction (certificate 31/10; dd. October 14, 2010).

In respect of the quality indicators set by Russian State Standard GOST 4913–82 (EN 13279–2), JSC AECP fluorogypsum is significantly superior to the gypsum stone mined in the Nukutsky Gypsum Quarry (Irkutsk Region). The gypsum stone, however, enjoys wide application due to the perceived lack of alternatives (as there exist only one gypsum stone quarry in the Eastern Siberia). The use of high-quality fluorite concentrate in the production of the hydrofluoric acid can be accounted for exceptional characteristics of fluorogypsum found at the slurry fields of JSC AECP.

Fluorogypsum has already been considered a raw material for the production of mineral binders and, when fused with cement, a component of dry building mixes [16]. In addition, fluorogypsum treated with sulfuric acid was viewed as an initial product for manufacturing mineral fertilizers [7].

Along with fluorogypsum, there exist the materials exhibiting similar chemical properties and physical characteristics [8–25]:

- phosphogypsum, a by-product of phosphoric acid production;
- borogypsum, a by-product of boric acid production.

Some attempts were made to produce gypsum-cement-pozzolana binders with phosphogypsum [3, 4, 7]. Investigations revealed that a gypsum binder made of a phosphogypsum anhydrite and blast furnace slag mixture 70–24 % with appropriate activators achieved 23 MPa strength after 28 d curing at 27 °C under high humidity and a mixture composed of 75 % gypsum, 20 % OPC and 5 % microsilica as cementitious binder developed a strength of up to 17 MPa after 200 days under water [17].

Addition of wastes containing boron in cement based materials causes mostly decrease in strength parameters and increase in setting times. Researchers agree that impurities and B_2O_3 in the wastes are the most influencing parameters effecting strength reduction and retardation of setting time [18].

Fluorogypsum can be used as:

1. a set-controlling admixture for the clinker milling [2–5];
2. a raw material in production of high-quality gypsum [11, 16].

The purpose of the research is to explore the chemical composition and physical properties of fluorogypsum (the waste of hydrofluoric acid production) as well as the possibility of making PC400–D20 Portland cement (according to Russian State Standard GOST10178–85), as the gypsum stone was replaced with fluorogypsum during the milling process.

2. Material and Methods

Fluorogypsum is a light gray paste where the particle size does not exceed 0.08 mm, and the true density equals 2.35 g/cm^3. The range of water content of the average sample taken from the settled sludge is 15–20 %. The dried sample is a powder with a specific surface area of 480 m^2/kg. The chemical composition of the samples was determined by the flame atomic absorption spectrophotometry: the method was finalized by the bulletins № 61 and № 172–S issued by the Scientific Committee on Analytical Research Methods in 2016 and October 27, 2015 respectively, approved by The Federal Methodical Center for Laboratory Research and Certification of Mineral Raw Materials of the Ministry of Natural Resources and Environment of the Russian Federation, and revised by the Federal State Unitary Enterprise "All-Russian Research Institute of the Mineral Raw Materials named after N.M. Fedorovsky".

A flame photometer based on a DFS–12 spectrometer and the PerkinElmer 403 atomic absorption spectrophotometer were used for the chemical analysis of the fluorogypsum which is presented in Table 1.

Table 1. Chemical analysis of fluorogypsum.

Content, %	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	Other vapor impurities
Max.	2.07	0.64	0.429	31.88	0.768	0.073	44.25	21.29
Min.	1.93	0.58	0.422	31.56	0.731	0.071	44.13	22.25
Average (based on 3 samples)	1.97	0.61	0.426	36.63	0.745	0.072	44.19	21.76

Note: Chemical analysis of the fluorogypsum was conducted in the analytical department of Vinogradov Institute of Geochemistry SB RAS.

Based on the acquired data, the fluorogypsum can be classified as the premium grade material as set by Russian State Standard GOST 4013–82. In the course of establishing the quantitative composition of the trace elements, the absence of harmful and dangerous impurities was determined as well.

This makes it possible to organize a production of high-quality gypsum, as the deposits of the slurry fields of JSC AECP can be used to create gypsum of grades G 10 and higher according to Russian State Standard GOST 12579. Likewise, the material can be referred to as the first category in terms of whiteness – a factor proven to be especially valuable for the producers of dry construction mixtures.

Currently there is no production of high-quality gypsum in Irkutsk region. The established fields of high-quality raw materials used to manufacture gypsum are mainly located in European part of Russia, but the reserves are limited and do not satisfy the needs of the entire country. This shortage is fulfilled by means of external supply. Angarsk Gypsum Plant produces gypsum that does not exceed G5, as the plant's production lines are designed to process the raw materials of the Nukutsky Gypsum Quarry.

Processing fluorogypsum of the slurry fields of JSC AECP relies on the removal of hygroscopic moisture from the material before converting calcium sulphate dihydrate into its end product – hemihydrate. At the temperatures of 100–140 °C, the process of transformation of calcium sulphate dihydrate into hemihydrate takes places according to the following reaction:

$$CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 \cdot 0.5H_2O + 1.5H_2O.$$

Despite its simplicity, the production of high-quality gypsum requires special conditions. There are two structurally different modifications of calcium sulphate hemihydrate, namely, α- and β-modifications. Alpha-hemihydrate, which is formed at a temperature slightly above the initial boiling point under the increased pressure of water vapor, is considered the most valuable. The production process is carried out in the autoclave under certain pressure, so that the removal of internal water from the gypsum does not cause loosening and destruction of the grains (particles). Dense hemihydrate smooth surfaced crystals are formed. In this case, the compliance with such technological parameters as vapor pressure, temperature, and processing time serves as a prerequisite.

The second form, β-hemihydrate, is produced under the atmospheric pressure: during the dehydration process, the water comes out in the form of the steam which results in strong mechanical dispersion of grains and the formation of a rough surface, "pitted" with a large number of cracks and capillaries. The higher the temperature and the lower the water vapor pressure, the finer the surface of the crystals are.

Structural differences determine the properties of α- and β-modifications of calcium sulphate hemihydrate. β-hemihydrate is distinguished by its increased solubility and higher hydration rate; however, to obtain a so-called rolling gypsum paste, it requires more water (50–70 wt. % of gypsum compared to 30–45 % for the α-hemihydrate). A result, it possesses less strength.

The production of high-quality gypsum opens up the prospects for the manufacturing a wide range of building mixtures. Currently, the construction market is 80 % filled with materials of the foreign origin represented by a wide range of products for all types of work and of various complexity. Upon conducting an in-depth study, it can be concluded that the production technology of the imported building mixtures sold on the Russian market is quite simple and based upon:

- strict selection of inert aggregate grain-size distribution
- high-quality binder;
- optimum input of special additives.

Special additives are classified into plasticizing, hydrophobic, air-retraining, as well as organic and non-organic substances.

All things considered, given the availability of high-quality gypsum of our own production, it becomes possible to manufacture building mixtures of the enhanced properties. This will secure the share on the market of construction materials.

3. Results and Discussion

3.1. The use of fluorogypsum as a set-controlling admixture for the clinker milling

The process applied in large-scale cement production based on the traditional technology of burning cement clinker in tubular rotary kilns at the grinding stage. In this case, calcium sulphate dihydrate serves a regulator of the setting time and is added in the amount of 3–5 % of the clinker weight during the grinding process (in accordance with the adopted technology).

During the study, the raw materials used by Angarsk Cement Plant (i.e. the gypsum stone mined at the Nukutsky Gypsum Quarry) were replaced with the fluorogypsum deposited in the sludge dumps of JSC AECP. The needs of the cement plant for this type of the raw materials amounted to 3000 tons per month.

The purpose of the research was to explore the possibility of making PC400–D20 Portland cement (according to Russian State Standard GOST 10178–85 (EN 197–1), as the gypsum stone was replaced with fluorogypsum during the milling process. The object of the study dealt with physical and mechanical properties of Portland cement obtained by mixing milled Portland cement clinker, active mineral additives (fly ash) and sulphate additives (gypsum stone or fluorogypsum).

<u>Properties of the test materials:</u>

a) *Portland cement clinker (PCC)* of the specified mineral composition manufactured by the Cement Factory “Angarsk Cement” in the form of the dark gray particles of 3 to 40 mm each;

b) *fly ash (FA)* of Irkutsk Thermal Power Plant – 1 in the form of light gray powder (density of 2.3–2.4 g/cm^3, bulk density of 780–820 kg/m^3 CaO absorption activity 40–50 mg);

c) *gypsum stone (GS)* in the form of white crushed gypsum stone of particles of 20 mm each, used as a sulphate-containing additive in the production of the Portland cement (according to Russian State Standard GOST 4013);

d) *fluorogypsum (FG)*, waste deposits in the form of suspension (mass humidity 12–15 %) containing solid particles not exceeding 0.08 mm and possessing true density of 2.35 g/cm^3;

e) *standard quartz uniformly graded sand* used to prepare cement mortar according to Russian State Standard GOST 310–4 (ENG 196–1).

All the materials with the exception of the clinker were dried in order to obtain constant weight [18, 21]. The drying of fly ash and sand was carried out at the temperature of 105±5 °C. Gypsum stone and fluorogypsum were dried at the temperature of 68±2 °C to avoid dehydration.

Upon the drying, all the materials with the exception of fluorogypsum were ground in the ball mill to the required particle size (residue on sieve No. 008 not exceeding 15 wt. %) and then subjected to drying for one more hour before being placed in hermetically sealed containers. The containers then were stored in the chamber with low relative air humidity as ensured by calcium chloride placed on the tin. The materials were stored that way up until being used in the experiment.

The materials were used to determine the quality indicators of two compositions of cement, where either gypsum stone or the fluorogypsum of JSC AECP slurry fields were used as set regulators. The qualitative composition of such types of the cement conforms to the standards accepted at “Angarsk Cement” plant, namely:

– Portland cement clinker (PCC);
– active mineral additive fly ash: 18 % of the total clinker mass;
– gypsum additive (GS or FG): 5.2 % of the total clinker mass.

The components of each type of the cement in the quantities required for the experiments in accordance with Russian State Standard GOST 310.2–76 and Russian State Standard GOST 25328 (EN 2061) were mixed in a laboratory mill for two hours as the metal balls were replaced with rubber and plastic “grinding elements”. The property values of two composition of the cement (*PCC = 1.0 + FA = 0.18 + GS = = 0.052* and *PCC =1 + FA =0.10 + FG = 0.052)* are summarized in the Table 2.

Table 2. Property values of cement produced with addition of either the gypsum stone or the fluorogypsum.

No.	Properties	Property values		
		Cement: PCC + FA+ GS	Cement: PCC+FA+ FG	According to Russian State Standard GOST 10178–85
1	Density, g/cm^3	3.05	3.02	2.9–3.1
2	Fineness: residue on sieve № 008, wt. %	13.5	14.2	≤ 15.0
3	Fineness: surface area, m^2/kg	371	356	≥ 250
4	Consistency, wt. %	26	27	25–30
5	Initial Setting	3 hours 05min	3 hours10 min	≥ 45 min
6	Final Setting	7 hours 25 min	7 hours 50 min	≤ 10 hours
7	Strength of the 1-day steam-cured samples, MPa: a) bending strength; b) compression strength	 3.72 20.20	 3.97 23.13	1st group > 27.0 2nd group: 24–27 3d group ≤ 24
8	Strength of the 7-day water-cured samples, MPa: а) bending strength; б) compression strength	 3.83 23.19	 4.02 24.18	not standardized
9	Strength of the 28-day water-cured samples, MPa: а) bending strength; б) compression strength	 6.15 41.20	 6.31 42.2	 > 5.9 > 39.2
10	Bulk density, kg/m^3	1100	1080	
11	Bleeding, vol. %	32	30	According to Russian State Standard GOST 25328–82 ≤ 30

The results of the study demonstrate that physical and mechanical properties of the produced cement samples satisfy the technical requirements set by Russian State Standard GOST 10187–85 (EN197–1). Thus, the samples can be classified as PC400–D20 cement brand.

Substitution of a gypsum additive from natural gypsum stone to the one of fluorogypsum did not present to have any significant effect on the quality of the cement. However, adding fluorogypsum to Portland cement resulted in a slight increase in the water requirement for the material. This is reflected both in the consistency and in the reduction of water release (a positive effect from the increased dispersity of fluorogypsum).

Increase in the setting time of the cement paste is one of the potential positive impacts of using the fluorogypsum. This, in turn, makes the cement more effective in concrete to be subjected to electrical heating in winter. As a result, this phenomenon allows for reclassifying Portland cement from group 3 to group 2 (with regard to the curing).

However, replacing the Nukut gypsum [18, 19] with fluorogypsum may prove complicated due to the increased hygroscopic moisture in the latter (15–25 %). The moisture removal procedure conducted in the usual manner is not cost-effective. According to the conservative estimate, the energy cost of the water removal is 156.4 kWh per ton of fluorogypsum, and with the usual 50 % efficiency of using thermal units, these costs would double.

In this regard, an original and economical method of water removal was designed and then tested under the laboratory conditions. The method is a combination of chemical absorption of hygroscopic moisture by the hemihydrate, with the subsequent transformation of the latter into the dihydrate of normative humidity. This technology allows to reduce the cost of bringing fluorogypsum to the normalized humidity by 25 %.

Another requirement to be met for the use of fluorogypsum in the cement industry deals with the loading and transportation of the material as it should not create dust areas. Hence, the process of moisture removal must be combined with the refining the fluorogypsum through the rotary drum granulator or the roller press. The particles (granules or pellets) formed this way will possess the necessary strength to retain their shape during loading and transportation.

The nature of incorporating fluorogypsum additives in the structure of the cement stone is revealed by crystal-optical examination. Filamentous contact points between the fluorogypsum and the cementing mass are clearly defined (Fig. 3).

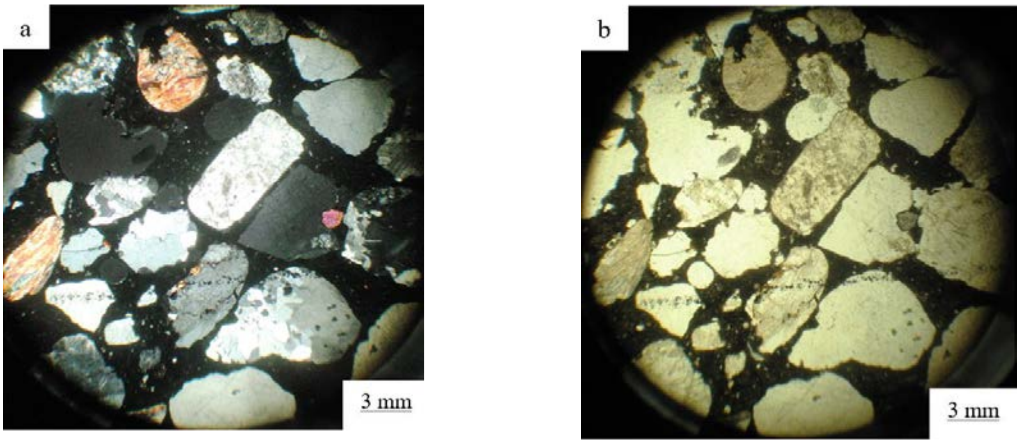

Figure 3. Cement with fluorogypsum as an additive: general layout (a – transmitted light, b – polarized light).

Fig. 4 demonstrates the processes of ettringite particles thin shells formation. Contact points between the grain and the cementing material are clearly defined.

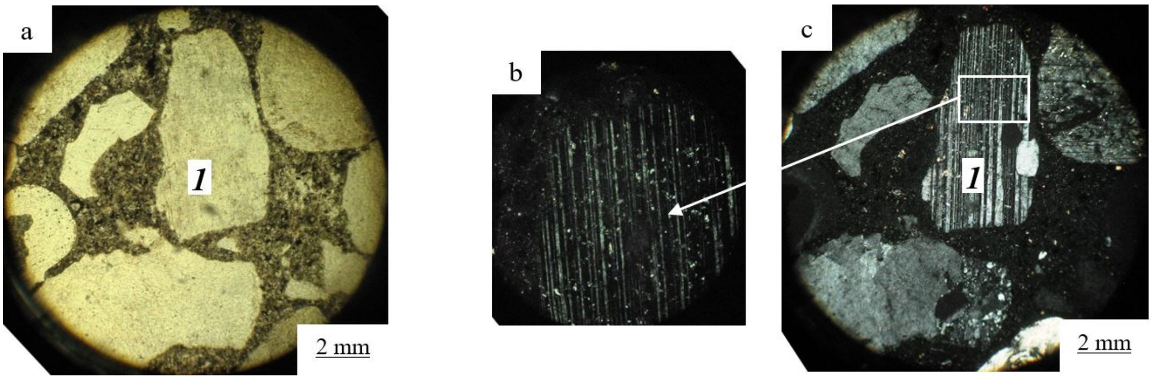

Figure 4. The processes within an ettringite grain (1) (a – transmitted light, b and c – polarized light).

Fig. 5 shows a crystallographic image of a fluorogypsum grain, as well as the processes of ettringite shells formation between the clinker grains. It should be noted that replacements are observed only within a given grain. Fig. 6 shows a cementing mass, in individual grains of which recrystallization of the shell is observed (1), and needle-shaped formations bind cement particles into a monolith.

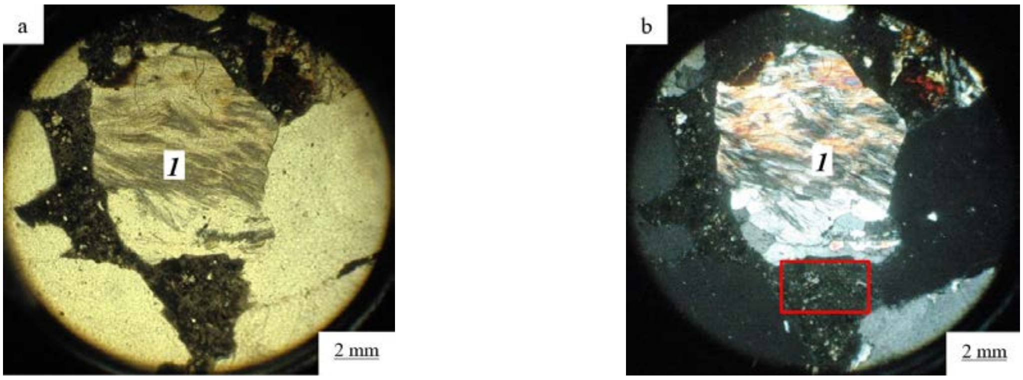

Figure 5. Fluorogypsum grain (1)
a – transmitted light, b – polarized light.

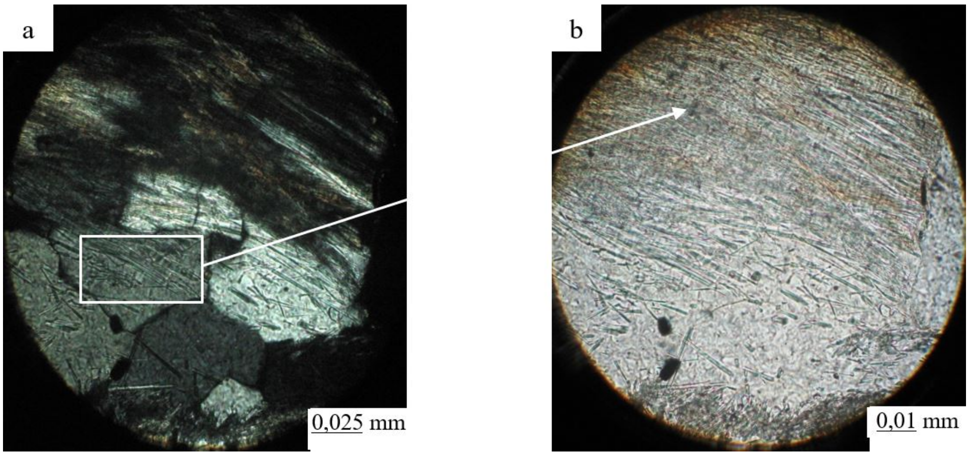

Figure 6. Element from Fig. 3. Cementing mass: recrystallization of the shell is observed in individual grains (1).

This study corroborates a classical scheme of cement stone formation as fluorogypsum is used to prevent false setting [3–4].

When mixing cement containing fluorogypsum and water, film-like ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$ is formed on the surface of the clinker grains. The ettringite inhibits the interaction of cement and water which leads to a so-called induction period, or a dormant period. Subsequently, the shielding shells are gradually destroyed and the dormant period ends after 3–6 hours. Cement starts interacting with water, and the structural strength of the system increases.

Within a few hours after mixing cement containing fluorogypsum and water, ettringite particles recrystallize on the clinker grains of the shell to form elongated crystals that gradually bind the cement particles to each other. This creates a setting effect. Due to recrystallization, the shells become more permeable to water, which leads to its increased interaction with cement and further strengthening of the structure.

4. Conclusions

1. The in-depth studies of chemical composition and physical properties of fluorogypsum deposited at the slurry fields of JSC AECP proved that it can serve as a first-grade raw material in accordance with Russian State Standard GOST 4913–82 for the production of high-grade gypsum.

2. Laboratory tests confirmed the possibility of using fluorogypsum as a setting regulator in cement production when grinding cement clinker. Crystal-optical studies have proved the classical scheme of the behavior of gypsum during the formation of cement stone as well as its role in the setting of cement paste. The exact quantity of fluorogypsum when grinding cement clinker was determined, which ensures the production of high-quality cement the corresponding to the requirements set by Russian State Standard GOST 1018785 (EN 197–1).

3. The prospects of using fluorogypsum as a raw material for the production of high-quality gypsum as well as a wide range of building mixtures based on it has been identified. That, in turn, makes it possible to firmly gain a foothold in the market of high-quality dry building mixtures.

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