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ELEMENTAL COMPOSITION OF INDIVIDUAL PHOSPHOGYPSUM FRACTIONS AFTER DRYING IN A FLASH DRYER

O. G. Gorovyk[1], A. V. Volosach[2]

[1] Candidate of Technical Science, Associate Professor, Head of Laboratory, Belspetskomplekt LLC, Minsk City Technopark, Minsk, Belarus, e-mail: olgreda@tut.by

[2] Researcher, Senior Lecturer, Department of Advanced Training, Branch of the Institute for Retraining and Advanced Training, University of Civil Defense, Ministry of Emergency Situations of the Republic of Belarus, Svetlaya Roshcha village, Borisov District, Minsk Region, Belarus, e-mail: kpkippk@ucp.by

Abstract

This paper demonstrates for the first time that individual fractions of phosphogypsum dried in a flash-drying system to a residual sorption moisture content of 0.01–0.03% have different contents of individual elements. Drying waste phosphogypsum in the described drying system produces highly dispersed products, eliminating the costly milling of the phosphogypsum after drying. The particle size of the dried product corresponds to the size of the phosphogypsum crystals formed in the baths during phosphoric acid extraction. Fractions with the required impurity content can be separated from the powder obtained after drying the waste phosphogypsum using a mechanical sieve analyzer. Thus, fractions of 0.050–0.045 mm (0.045 sieve residue), 0.140–0.090 mm (0.100 and 0.090 sieve residue), as well as dried material from the cyclone, bag filters, and the original waste phosphogypsum, were analyzed for the content of individual elements using X-ray fluorescence analysis. It was found that the content of certain rare earth elements and strontium in the finely dispersed product (obtained from the filters) increased by more than twofold compared to the original waste phosphogypsum, while the content of elements such as cerium, lanthanum, and lutetium increased by up to fourfold. The results of the studies showed that the 0.140–0.090 mm fraction (residue on 0.100+0.090 sieves) has significantly reduced amounts of individual elements compared to both the original waste phosphogypsum and the dried product collected from the cyclone. Specifically, the 0.140–0.090 mm fraction has reduced the content of the following elements (in terms of their oxides): barium (more than 17 times), iron (up to 10 times), titanium (up to 7 times), etc. The 0.140–0.090 mm fraction also lacks the following elements: potassium, nickel, silicon, germanium, selenium, palladium, indium, erbium, lutetium, hafnium, tantalum, and mercury. This suggests that the 0.140–0.090 mm fraction does not contain elemental compounds that limit its use in building materials and can be used as a substitute for natural gypsum. Furthermore, the increased REE content allows the filter product to be considered a REE concentrate, with the potential to utilize numerous developed "wet" technologies for their extraction.

Keywords: phosphogypsum, phosphogypsum fractions, chemical composition, elemental oxides, X-ray fluorescence analysis, microscopy.

ЭЛЕМЕНТНЫЙ СОСТАВ ОТДЕЛЬНЫХ ФРАКЦИЙ ФОСФОГИПСА ПОСЛЕ СУШКИ НА ФЛЭШ-СУШИЛКЕ

О. Г. Горовых, А. В. Волосач

Реферат

Впервые показано, что отдельные фракции фосфогипса, высушенного в сушильном комплексе с флэш-сушилкой до остаточной сорбционной влажности 0,01–0,03 %, имеют различное содержание отдельных элементов. Сушка отвального фосфогипса на описываемом сушильном комплексе обеспечивает получение высокодисперсных продуктов, использование которых позволяет исключить дорогостоящую операцию помола фосфогипса после сушки. Величина частиц в высушенном продукте соответствует величине кристаллов фосфогипса, формирующихся в ваннах при экстракции фосфорной кислоты. Из порошка, полученного после сушки отвального фосфогипса, можно выделить с применением ситового механического анализатора фракции с требуемым содержанием примесей. Так фракции 0,050–0,045 мм (остаток на сите 0,045), 0,140–0,090 мм (остаток на ситах 0,100 и 0,090), а также высушенный материал из циклона, рукавных фильтров и исходный отвальный фосфогипс проанализировали методом ренгенофлоуресцентного анализа на содержание отдельных элементов. Было обнаружено, что содержание некоторых редкоземельных элементов и стронция в мелкодисперсном продукте (полученном из фильтров) повысилось, по сравнению с исходным отвальным фосфогипсом более чем в два раза, а содержание таких элементов, как церий, лантан и лютеций до четырех раз. Результаты исследований показали, что во фракции 0,140–0,090 мм (остаток на ситах 0,100+0,090) значительно снижено количество отдельных элементов по сравнению как с исходными отвальным фосфогипсом, так и высушенным продуктом, отбираемым из циклона. А именно, во фракции 0,140–0,090 мм уменьшилось содержание таких элементов (в пересчете на их оксиды), как барий более 17 раз, железа – до 10 раз, титана – до 7 раз и т. д. Также во фракции 0,140–0,090 мм отсутствуют такие элементы, как калий, никель, кремний, германий, селен, палладий, индий, эрбий, лютеций, гафний, тантал и ртуть. Это даёт основание говорить о том, что фракция 0,140–0,090 мм не содержит соединений элементов, ограничивающих её использование в строительных материалах, и может применяться как заменитель природного гипсового камня. Кроме того, повышение содержания РЗЭ уже позволяет считать продукт из фильтров концентратом РЗЭ с возможностью применения многочисленных разработанных «мокрых» технологий для их извлечения.

Ключевые слова: фосфогипс, фракции фосфогипса, химический состав, оксиды элементов, ренгенофлоуресцентный анализ, микроскопия.

Introduction

One of the largest waste streams, the third largest in Belarus, is waste from the production of wet-process phosphoric acid from apatite concentrate at the Khibiny deposit. Over the 50 years of operation of the Gomel Chemical Plant, over 18 million tons of phosphogypsum waste have accumulated, contaminating soils, surface water, and groundwater, covering an area of over 70 hectares. Numerous studies have been devoted to the use of phosphogypsum (PG) in various industrial fields, most of which concerned the production of artificial gypsum [1–6] using autoclave or thermal treatment methods (firing PG at 900–1000 °C), sulfate-slag binders, as an additive to agricultural soils, as raw material for the

production of gypsum bricks, stone walls, binders for road bases, etc. However, all the considered areas of PG use have not found widespread application due to the high content of various impurities [7] present in waste PG (WPG). Impurities negatively affect the quality of the resulting building materials [8–14], and operations to reduce the amount of impurities in PG (neutralization, enrichment, drying, thermal treatment) increase the cost of gypsum binders and reduce its competitiveness in comparison with natural gypsum rock. A reserve for increasing the efficiency of FG processing is the elimination of heat treatment, grinding, and, in the future, filtration of the product, since these stages consume about 45 % of capital, about 50 % of current, and more than 60 % of heat and energy costs.

The chemical composition of the FG of the Gomel Chemical Plant OJSC was carried out by researchers many times, as a result of which it was established that the FG of the Gomel plant contains from 95 % to 97 % of dihydrate and hemihydrate of calcium sulfate, and impurities [15], including compounds of the elements Cd, Zn, Pb, Hg, Zr, Cu, Ba, REE, Y, Th, U. However, the determination of REE and other elements was carried out in the total mass of the FG, even if it was subjected to heat treatment, then after grinding the sintered agglomerates, averaged samples selected from the total mass of the dried material were again examined.

As a result of the presence of difficult-to-remove impurities (with the exception of a small volume of secondary processing from 4 to 10 %), the predominant storage of FG occurs in the form of waste heaps, the formation and maintenance of which requires significant costs (the rate of environmental tax for the storage of 1 ton of FG in 2024 was 1.04 Belarusian rubles [16]).

Study of individual fractions of phosphogypsum dried in a flash dryer

The purpose of the study: to establish the elemental composition of individual fractions of PhG, with the determination of the possibility of using individual fractions in the production of building materials or other areas.

1 Research methods

To study the elemental composition of the Gomel plant's fluorinated gas, representative samples from the Gomel chemical plant's waste heaps with five- and ten-year storage periods were analyzed.

The elemental composition of the samples was determined by X-ray fluorescence analysis using an Epsilon 1 PANalytical energy-dispersive spectrometer equipped with an X-ray tube with a silver anode, six filters, and a high-resolution silicon detector. Automatic data processing was performed using Epsilon 3 software. Results were also automatically corrected for ambient temperature and pressure.

The sorption moisture content of the samples was monitored using an Elvis-2S moisture analyzer at 55 °C (exceeding this temperature resulted in the onset of loss of crystallization water).

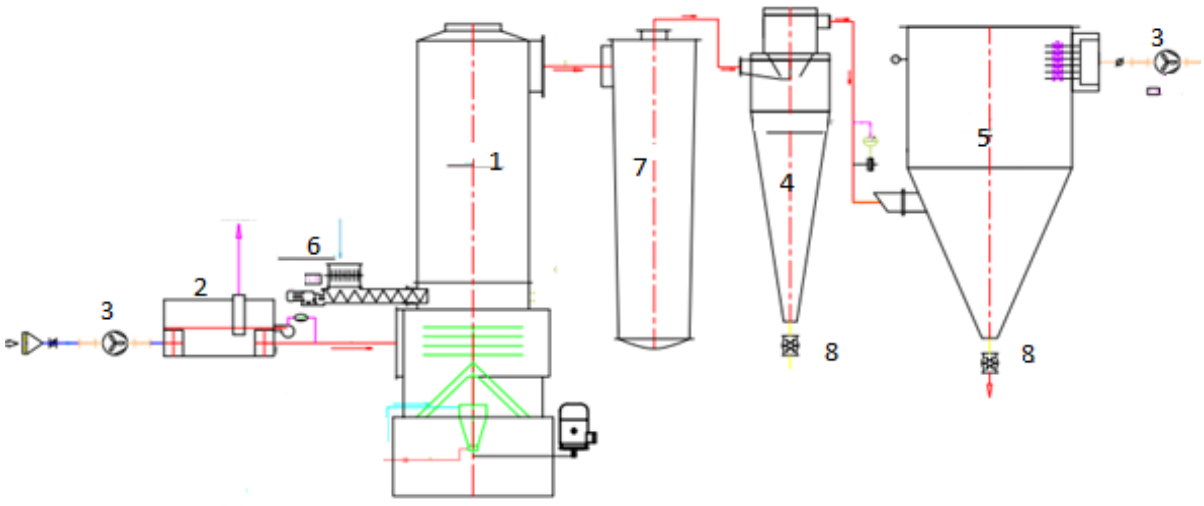
Micrographs were taken using a Phenom Pro scanning microscope (Thermo Fisher Scientific, USA). Separation into fractions was carried out on a sieve mechanical analyzer "Vibroprivod VP 30 T" No. 4938, on metal sieves with mesh cell sizes, mm: 1.000; 0.140; 0.100; 0.090; 0.080; 0.071; 0.050; 0.045; 0.040.

2 Obtaining phosphogypsum fractions

The phosphoric acid produced at any plant worldwide during the production of wet-process phosphoric acid has an adsorption moisture content of 30–40 %. This high adsorption moisture content causes it to easily clump, forming lumps of varying sizes (Figure 1).

Figure 1 – Type of phosphogypsum collected from waste heaps

To remove adsorbed moisture from FG, dryers such as drum, belt, shaft, and IR dryers are used. Drying this material in these dryers results in the formation of solid lumps of varying sizes that do not disintegrate spontaneously and require subsequent grinding of the dried product. To eliminate the need for grinding, FG was dried in an industrial complex (Figure 2) comprising a flash dryer, a fuel furnace, a cyclone, bag filters, a feed auger, a final drying unit, and unloading devices. The flash dryer is a combination of a suspended bed dryer and a cyclone vortex dryer with a cocurrent flow of incoming drying gas.



1 – flash dryer, 2 – indirect fuel furnace, 3 – air blower, 4 – cyclone, 5 – bag filters, 6 – feed auger, 7 – final drying device, 8 – unloading devices

Figure 2 – Schematic diagram of the complex for drying waste phosphogypsum

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The FG was dried to a residual sorption moisture content of no more than 0.03 %. This drying method allowed us to obtain products with a dispersion similar to that of the FG precipitate in baths used for phosphoric acid extraction.

The drying temperature regime for obtaining a product (dried FG) with a sorption moisture content of no more than 0.03 % was set as follows: the coolant temperature at the dryer inlet was 180–200 °C, and at the dryer outlet, 85–95 °C.

Increasing the temperature above 200 °C at the dryer inlet resulted in the formation of calcium sulfate hemihydrate. Being a strong desiccant, calcium sulfate begins to adsorb water from the surrounding air, leading to a subsequent change in the overall moisture content of the product within 1 hour (an unstable product). When the dryer inlet temperature exceeded 205 °C, reactions began to occur, leading to the release of chemically bound (crystallization) water from the crystal lattice. Depending on the conditions under which these reactions occur, the dehydration products of calcium sulfate dihydrate can be represented in various ways:

$CaSO_4 \cdot 2H_2O$ = a $CaSO_4 \cdot 0{,}5H_2O + 1{,}5H_2O$ (п) – 83,17 kJ/mol;
$CaSO_4 \cdot 2H_2O$ = a $CaSO_4 \cdot 0{,}5H_2O + 1{,}5H_2O$ (ж) – 17,9 kJ/mol;
$CaSO_4 \cdot 2H_2O$ = b $CaSO_4 \cdot 0{,}5H_2O + 1{,}5H_2O$ (п) – 85,3 kJ/mol;
$CaSO_4 \cdot 2H_2O$ = b $CaSO_4 \cdot 0{,}5H_2O + 1{,}5H_2O$ (ж) – 19,3 kJ/mol;
$CaSO_4 \cdot 2H_2O$ = $CaSO_4 + 2H_2O$ (ж) – 16,9 kJ/mol;
$CaSO_4 \cdot 2H_2O$ = $CaSO_4 + 2H_2O$ (п) – 95 kJ/mol.

The dried product obtained using the flash dryer was essentially only the dihydrate form of calcium sulfate, as confirmed by the X-ray diffraction pattern shown in Figure 3.

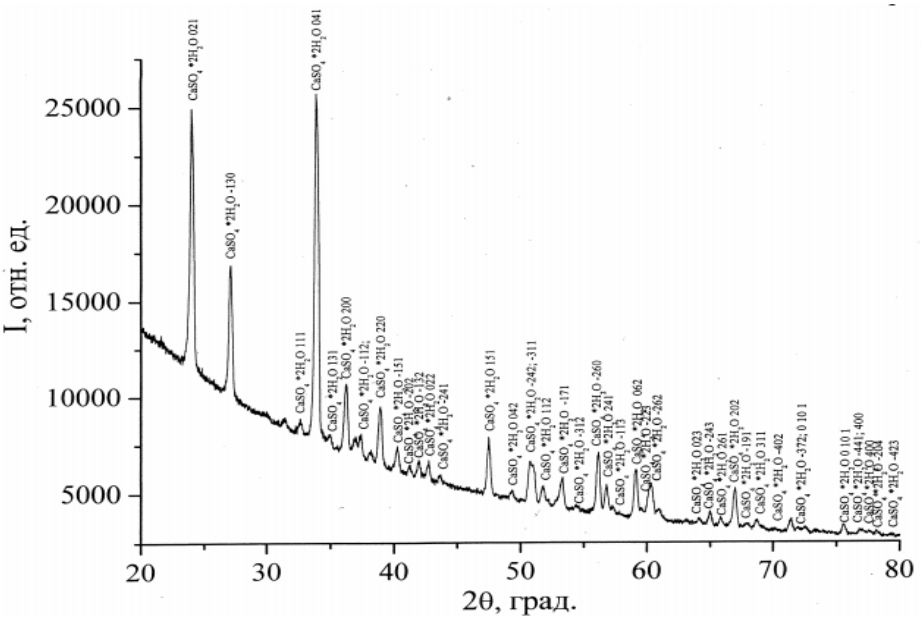

Figure 3 – Radiograph of dried OFG

Heating to 75–80 °C is sufficient for the slow release of sorption moisture from the FG, but lowering the dryer inlet temperature to 75–80 °C resulted in increased sorption moisture content at the dryer outlet.

Two thermocouples were used to monitor the drying process, with information being output to a secondary device mounted on the control panel. Some of the results of the studies on selecting the operating temperature parameters for the flash dryer system are presented in Table 1.

Table 1 – Flash dryer operating modes and residual sorption moisture content in the final product

Feed rate, kg/hour	Temperature in the dryer, ºC		Humidity of the obtained product, %
	at the entrance	at the exit	
1000	200	65	10
1000	250	70	4
500	180	70	3
500	200	85	1
350	180	95	0,04
350	200	85	0,01
200	180	75	0,01*

* strongly absorbs water from the air.

The moisture content of the OFG fed to the drying chamber depends on the season and transport conditions and varies by up to 10 %. This affects the final moisture content of the dried product and requires adjustment of the dryer temperature.

The finished dried material consists of two products: a coarse product (from the cyclone) and a fine product (from the bag filters). The particle size distribution of the products obtained after drying the OFG is presented in Table 2. Figure 4 shows a micrograph of the dried FG.

Table 2 – Granulometric composition of fractions obtained at the drying complex

Fraction	Sieve size, μm [GOST 6613-86]					
	<45	45	50	80	100	1000
	Content, %					
Finely dispersed (from bag filters)	64,5–71,5	25–30	3–5	0,5–0,6	–	–
Coarse (from cyclone)	15–18	5–10	45–50	10–12	3–10	–

Figure 4 – Micrograph of the coarse fraction (from the cyclone) of dried PhG. Magnification 1000x

3 Elemental composition of dried phosphogypsum fractions

The fine and coarse products obtained from drying the OFG in the drying complex were analyzed using X-ray fluorescence analysis. The content of individual elements, including rare earth elements, in these products is listed in Table 3.

The data in Table 3 show that the strontium content in the filter product increased more than twofold compared to the original FGZ, the total REE increased threefold, while the content of almost all elements, including the total REE, decreased in the coarsely dispersed product. This can only be explained by the relationship between the crystal size and the amount of elements sorbed by the surface or coprecipitated during crystallization. However, since the area-to-volume ratio of large crystals is smaller than that of small ones, their sorption surface is also smaller, which is evident from the results presented. The process of impurity element coprecipitation is also not the same for particles of different sizes, since this change in the REE content cannot be explained by the area-to-volume ratio of a particle alone. It is known from [17] that the distribution of rare earth elements (REE) among apatite processing products is as follows: up to 80 % of the REE present in the ore is transferred to the FGZ, and 20 % is transferred to the extraction phosphoric acid. In [18] it is stated that coprecipitation of REE with calcium and sodium sulfate occurs at temperatures above 85 ºC, presumably with calcium sulfate hemihydrate. In [19] it is shown that REE – La, Ce, Nd are present in PG in the form of solid solutions of the compounds NaLn(SO4)2·H2O, KLn(SO4)2·H2O and the hemihydrate CaSO4·0.5 H2O, PG also contains REE in the form of the compounds NaLn(SO4)2·H2O, KLn(SO4)2·H2O, which are not included in the crystal structure of the monoclinic cell of CaSO4·2H2O and do not form solid solutions with CaSO4·2H2O. In [20] it is stated that the presence of REE leads to a decrease in the size of calcium sulfate crystals.

Based on these data, it can be assumed that large crystals of calcium sulfate dihydrate have a small amount of adsorbed or co-precipitated REE sulfates, while small particles will contain a larger amount of impurity elements, including REE.

Table 3 – Content of some elements in fractions of dried FG

Element	Content, %		
	OFG (original)	Fine fraction (from filters)	Coarse fraction (from cyclone)
Cerium (Ce)	0,529±0,015	1,746±0,032	0,695±0,285
Lanthanum (La)	0,293±0,003	1,264±0,023	0,298±0,065
Neodymium (Nd)	0,121±0,001	0,431±0,023	0,109±0,020
Yttrium (Y)	0,029±0,004	0,048±0,001	0,017±0,005
Dysprosium (Dy)	0,022±0,002	0,025±0,002	0,005±0,001
Scandium (Sc)	0,014±0,001	0,019±0,002	0,003±0,001
Tellurium (Te)	0,022±0,001	0,020±0,005	0,015±0,004
Lutetium (Lu)	0,002±0,001	0,006±0,001	0,003±0,001
Total Rare Earth Elements	*1,031±0,021*	*3,555±0,086*	*1,145±0,382*
Strontium (Sr)	4,908±0,073	14,800±1,160	4,065±0,09
Titanium (Ti)	0,365±0,033	0,442±0,190	0,368±0,018
Mercury (Hg)	0,002±0,001	0,004±0,002	0,002±0,001
Iron (Fe)	0,233±0,052	0,635±0,119	0,200±0,007

In the next stage of the study, the product from the cyclone was separated into fractions using a mechanical analyzer. The fractions of 0.140–0.090 mm (a combined sample of residues from 0.100 and 0.09 sieves) and 0.050–0.045 mm (residue on a 0.045 sieve) were also analyzed using X-ray fluorescence analysis. The results of the analysis of both the product collected from the cyclone and the individual fractions of particle sizes 0.140–0.090 mm and 0.050–0.045 mm are presented in Table 4.

Table 4 – Content of element oxides in various fractions

Oxides	Fractions, mm		
	0,900–< 0,045	0,140–0,090	0,050–0,045
	Content, %		
Cerium oxide (CeO_2)	0,854±0,35	0,318±0,013	1,337±0,025
Lanthanum oxide (La_2O_3)	0,699±0,153	0,152±0,024	0,911±0,037
Neodymium oxide (Nd_2O_3)	0,254±0,047	0,077±0,011	0,274±0,04
Yttrium oxide (Y_2O_3)	0,043±0,013	0,014±0,006	0,025±0,009
Dysprosium oxide (Dy_2O_3)	0,012±0,003	0,001±0,001	0,014±0,005
Scandium oxide (Sc_2O_3)	0,009±0,003	0,062±0,016	0,047±0,011
Tellurium oxide (TeO_2)	0,019±0,005	0,005±0,001	0,006±0,002
Lutetium oxide (Lu_2O_3)	0,007±0,002	0,001±0,001	0,001±0,001
Total rare earth oxides	*1,896±0,575*	*0,629±0,072*	*2,616±0,131*
Silicon oxide (SiO_2)	1,881±0,069	0,001±0,001	2,358±0,101
Strontium oxide (SrO)	4,808±0,107	1,994±0,305	10,954±1,568
Titanium oxide (TiO_2)	0,613±0,03	0,088±0,017	0,521±0,075
Mercuric oxide (HgO)	0,002±0,001	0	0,003±0,001
Barium oxide (BaO)	0,291±0,103	0,016±0,003	0,113±0,012
Iron oxide (Fe_2O3)	0,629±0,021	0,065±0,006	0,510±0,094

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An analysis of the presented results shows that the fraction with a particle size of 0.140–0.090 mm, compared to the product coming from the cyclone, has a 17-fold decrease in the amount of barium, approximately 10-fold decrease in iron, and 7-fold decrease in titanium. It also contains less strontium, yttrium, cerium, and other elements. Furthermore, the 0.140–0.090 mm fraction is free of oxides of potassium, nickel, silicon, germanium, selenium, palladium, indium, erbium, lutetium, hafnium, tantalum, and mercury. The calcium sulfate content has increased due to a decrease in the total amount of other components. The fraction with a particle size of 0.050–0.045 mm has increased contents of strontium, silicon, and zirconium, which once again confirms the theory of a relationship between the particle size of the PG and the adsorbed and coprecipitated elements. It should be noted that chlorine and bromine were absent from both analyzed fractions, which can be explained by the volatilization of these elements and their compounds during passage through the drying system. This means that during the flash dryer drying of the OFG, not only the sorption moisture is lost, but also volatile components such as fluorine compounds and some phosphorus compounds.

The hydrogen ion activity (pH) of the 0.140–0.090 mm fraction solutions is 7.2; and the pH of the cyclone product is 6.79, which also indicates different contents of some compounds in these products. Comparing the values of individual indicators of the 0.140–0.090 fraction with the requirements of TU 400069905.047–2019 [21] for artificial gypsum stone, its full compliance is revealed (Table 5).

If we compare it with natural gypsum stone [22], all indicators correspond to grade 1 (Table 6).

Table 5 – Comparison of the 0.140–0.090 mm fraction with the requirements for artificial gypsum stone

Name of the indicator	Meaning, according to [21]	Fraction 0.140–0.090 mm
Content of the main substance - calcium sulfate dihydrate ($CaSO_4$ $2H_2O$), %,	Not less than 90	95
Total content of phosphates in terms of P_2O_5, %,	Not more than 0.8	0,77
including the content of water-soluble phosphates	Not allowed	Absent
Total content of fluorine, %	Not more than 0.40	Absent
including the content of water-soluble fluorine (fluorine ion), %	Not more than 0.005	Absent
Hydrogen ion activity (pH)	7.0–8.0	7,2

Table 6 – Content of main components in the 0.140–0.090 mm fraction and the requirements of GOST 4013–2019 [22]

Contents	Gypsum ($CaSO_4 \cdot 2H_2O$), %	Water of crystallization, %	Sulfur(VI) oxide (SO_3), %
Gypsum stone grade 1	Not less than 95	Not less than 19.9	Absent
Gypsum-anhydrite stone grade 1	Not less than 95	Absent	Not less than 44.2
Fraction of dried PhG (0.100+0.090) mm	95,8	Absent	49.7

Conclusion

1. Using a drying system with a flash dryer enables the production of phosphorus hydrate (FG) as a fine powder, primarily in the form of calcium sulfate dihydrate, with an adsorbed moisture content of no more than 0.03%, without loss of crystal water and, consequently, without crystal breakage or reduction in size. The particle size of the dried FG corresponds to the size of crystals formed in extraction baths during the production of phosphoric acid from apatite concentrate. Producing a fine powder eliminates the need for FG milling before further use, reducing its cost. Furthermore, the fine particle size of the dried FG allows it to be separated into individual fractions using mechanical classifiers.

2. The chemical composition of the fractions with particle sizes of 0.140–0.090 mm and 0.140–0.090 mm differs in impurity element content from their content in the OFG and from the product obtained from the cyclone. This can only be explained by the relationship between the crystal size and the amount of elements sorbed on the surface or coprecipitated during crystallization.

3. The fraction with particle sizes of 0.140–0.090 mm contains an amount of impurities that will not affect the quality of products manufactured from it compared to the use of artificial gypsum (calcium sulfate hemihydrate) and complies with the requirements of GOST 4013–2019 and TU BY 400069905.047–2019.

4. The presented data provide grounds for talking about the possibility of using a reagent-free, non-thermal method (firing FG at 900–1000 °C) to reduce impurities in FG and the prospects for using various fractions obtained from OFG dried in a flash dryer as raw material for the production of artificial gypsum applicable for building materials or other applications.

5. The data presented in the article indicate that further, finer separation of the dried FG into narrow fractions is possible, which will contain varying amounts of impurities, and this content will enable the targeted use of this fraction without the labor-intensive processes of precipitation of impurities with strong acids, separation of precipitate, etc. The product obtained from the filters can be used to obtain REE, since the concentration in this product is several times higher than in the original FG, and the yield of finely dispersed product is significantly less than the initial amount of OFG.

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