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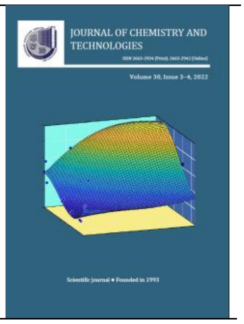

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TECHNOLOGY OF OBTAINING CONCENTRATE OF RARE-EARTH ELEMENTS FROM PHOSPHOGYPSUM AND ITS MATHEMATICAL DESCRIPTION

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Abstract

Aim. A mathematical model of the process of obtaining a concentrate of rare- earth elements from phosphogypsum using nitric acid was obtained. Methods. Extraction of rare-earth elements concentrate was carried out using the acid leaching method. To identify the relationship between the parameters of the studied process of rare-earth elements concentrate extraction, the correlation analysis had been performed, namely the calculation of Pearson's linear correlation coefficient, confidence intervals and the hypothesis of the significance of pairwise correlation coefficients had been tested. Results. On the basis of experimental studies and the description of the mathematical model of the obtained data, the optimal technological parameters were established, namely, the concentration of nitric acid in the range of 24–26 % and the processing temperature of 68–70 °C. Conclusions. On the basis of experimental studies, regression equations were obtained, that allow determining the dependence on temperature and acid concentration on the extraction of the concentrate of rare-earth elements from phosphogypsum. The technological scheme for the processing of phosphogypsum with the selection of a concentrate of rare-earth elements and the simultaneous production of calcium sulfate hemihydrate is proposed.

Keywords: phosphogypsum; rare-earth elements; temperature; concentration; mathematical description; multiple regression.

ТЕХНОЛОГІЯ ОДЕРЖАННЯ КОНЦЕНТРАТУ РІДКІСНОЗЕМЕЛЬНИХ ЕЛЕМЕНТІВ ІЗ ФОСФОГІПСУ ТА ЇЇ МАТЕМАТИЧНИЙ ОПИС

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Анотація

Мета роботи. Створення математичної моделі процесу одержання концентрату рідкісноземельних елементів із фосфогіпсу з використанням нітратної кислоти. Методи. Вилучення концентрату рідкісноземельних елементів проводилось з використанням методу кислотного вилуговування. Для виявлення взаємозв'язку параметрів досліджуваного процесу вилучення концентрату рідкісноземельних елементів виконано кореляційний аналіз, а саме: розрахунок лінійного коефіцієнта кореляції Пірсона, довірчих інтервалів та перевірено гіпотезу про значущість парних коефіцієнтів кореляції. Результати. На основі експериментальних досліджень та опису математичної моделі одержаних даних встановлено оптимальні технологічні параметри, а саме: концентрацію нітратної кислоти в межах 24–26 % та температуру обробки 68–70 °C. Висновки. На основі експериментальних досліджень одержано рівняння регресії, які дозволяють визначити вплив температури та концентрації кислоти на вилучення концентрату рідкісноземельних елементів із фосфогіпсу. Запропоновано технологічну схему переробки фосфогіпсу з виділенням концентрату рідкісноземельних елементів і одночасним одержанням напівгідрату кальцій сульфату.

Ключові слова: фосфогіпс; рідкісноземельні елементи; температура; концентрація; математичне описання; множинна регресія.

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Introduction

Currently, a significant part of phosphorus-containing mineral fertilizers is produced using extractive phosphoric acid.

The process of obtaining extractable phosphoric acid consists in the decomposition of natural phosphates with sulfuric acid, which is accompanied by the crystallization of calcium sulfate and the separation of the latter on vacuum filters.

While mixing the crushed phosphate with sulfuric acid, a thick slow-moving pulp is formed. To ensure thorough mixing of the reagents and to facilitate pulp pumping, a solution of acid and washing solution is introduced into the extractor. The ratio between the liquid and solid phases is usually maintained in the range from 1.7 : 1 tol 3.5 : 1 [1].

Thus, the phosphate is decomposed by a mixture of sulfuric and phosphoric acids [2]:

$$Ca_5(PO_4)_3F + 5H_2SO_4 + n\,H_3PO_4 = (n+3)\,H_3PO_4 + 5CaSO_4 \cdot mH_2O + HF. \qquad (1.1)$$

Depending on the temperature of the decomposition process and the concentration of phosphoric acid in the reaction mixture calcium sulfate can precipitate in the form of dihydrate (m = 2), hemihydrate (m = 5) and anhydride (m = 0). Accordingly, there are the following methods of obtaining phosphoric acid: dihydrate, semihydrate and anhydride ones.

Dihydrate and semihydrate methods have been mastered in industry. The stable calcium sulfate dihydrate precipitates at the temperature of 70–80°C and concentration P_2O_5 in the reaction mixture of 20–32 %. The hemihydrate precipitates at higher temperatures and concentrations of phosphoric acid: 90–100 °C i 35–42 % P_2O_5, respectively [3; 4].

These technologies made possible to sharply (significantly) increase the volume of the final product – the phosphoric acid – obtaining, while also increasing the amount of phosphogypsum waste. Today, the annual accumulation of phosphogypsum in the world accounts for 120–130 million tons, while the mass share of its utilization does not exceed 10–15 % [5]. About 90 million tons of phosphogypsum have been accumulated on the territory of Ukraine, and these dumps are the main source of environmental pollution in areas where mineral fertilizer production is located, in the cities of Sumy, Rivne, Vinnytsia and Kamianske.

Phosphogypsum is a large-tonnage waste of the chemical industry, which is formed in the amount of 4.5 tons per 1 ton of P_2O_5 in the production of extractable phosphoric acid [6]. Phosphogypsum occupies large areas of land and poses a serious threat of environmental pollution, therefore, the processing of these dumps is a very urgent task, and a number of technologies have already been developed for obtaining various products based on phosphogypsum [6, 7]. Phosphogypsum is a polydisperse material of gray-white color, represented by aggregates of particles, lumps with gaps between aggregates. Phosphogypsum becomes more finely dispersed after drying.

The environmental problems of processing phosphorite raw materials, in particular phosphogypsum waste, are considered in the work [8]. A multicomponent mineral complex fertilizer was obtained by the exchange reaction of phosphogypsum with ammonium nitrate interaction, the optimal technological parameters were determined and its fertilizing properties for radish cultivation were checked.

In agriculture, phosphogypsum is used without special preliminary treatment for plastering saline soils and as fertilizers or mixtures with other additives [9].

The chemical composition of phosphogypsum is usually determined by the quality of the used phosphate raw material and the method of its production.

One of the promising ways to utilize phosphogypsum is using it as a raw material for obtaining rare earth-elements [10].

Rare-earth elements (REE) have unique magnetic, optical, and electrical properties that make them indispensable elements in many high-tech processes [11; 12].

Rare-earth elements are the group of metals with similar properties, including scandium 21 Sc, yttrium 39 Y and fifteen lanthanides [13]. They are conventionally divided into light (from La to Sm) and heavy ones (from Eu to Lu and Y) [14]. Due to their important use in many industries, the need for these elements increases by almost 8 % per year [14].

The observed high demand for rare-earth elements is explained by their numerous applications in the basis of a wide range of new technologies, which can be grouped into three main groups: clean energy, defense and high-tech and everyday devices [15; 16].

Due to the great need for rare-earth elements in many technologies, different methods of extracting them from various rocks, including phosphogypsum, are emerging [17].

Depending on the technological scheme of EFC production, from 50 to 70% of REE passes into the phosphogypsum phase in the case of using the dihydrate scheme or up to 90% in the case of the semi-hydrate scheme. The main method of cleaning phosphogypsum from REE impurities is their leaching with mineral acids [9; 18].

The most widespread method is acid leaching of rare-earth elements from phosphogypsum using sulfuric, nitric and chloride acids [19].

We carried out the systematic studies of the process of obtaining rare-earth elements from phosphogypsum by means of sulfuric acid treatment. The concentration of acid and rare-earth elements in the filtrates after the separation of phosphogypsum was determined depending on the ratio "phosphogypsum acid" of 1 : 1.5, 1 : 2 and 1 : 2.5. The purified calcium sulfate was tested as a reagent for extracting phosphates from the sludge of urban sewage treatment plants, and an organic-mineral fertilizer was obtained from purified phosphogypsum and activated sludge of sewage treatment plants [20].

In the present work acid leaching of rare-earth elements with nitric acid was used.

The purpose of the work is to create a mathematical model of the process of obtaining a concentrate of rare-earth elements from phosphogypsum using nitric acid, to identify the optimal parameters of the process under which the highest degree of extraction of rare-earth elements is achieved.

Experimental

As the research object, phosphogypsum from the enterprise "Dniprovsky Mineral Fertilizer Plant" and from the dumps of the enterprise "Prydniprovsky Chemical Plant" was chosen. The filtration method was used in the research on obtaining a concentrate of rare-earth elements.

The chemical composition of phosphogypsum used for testing is within the following standards:

- mass fraction of calcium sulfate ($CaSO_4 \cdot 2H_2O$) in terms of dry dihydrate 90.0–96.0 %;
- mass fraction of hygroscopic water 15.0–16.3 %;
- mass fraction of water-soluble fluorine compounds (Na_2SiF_6, K_2SiF_6, HF) when converted to F 0.2–0.3 %;
- mass fraction of total phosphates (P_2O_5 total) 0.8–1.0 %;
- mass fraction of rare-earth elements 0.6–1%.

In order to obtain a concentrate of rare-earth elements from phosphogypsum and develop a mathematical model of the process, the laboratory setup was created [20], on which a study of nitrate-acid treatment of calcium sulfate waste was carried out, Fig. 1.

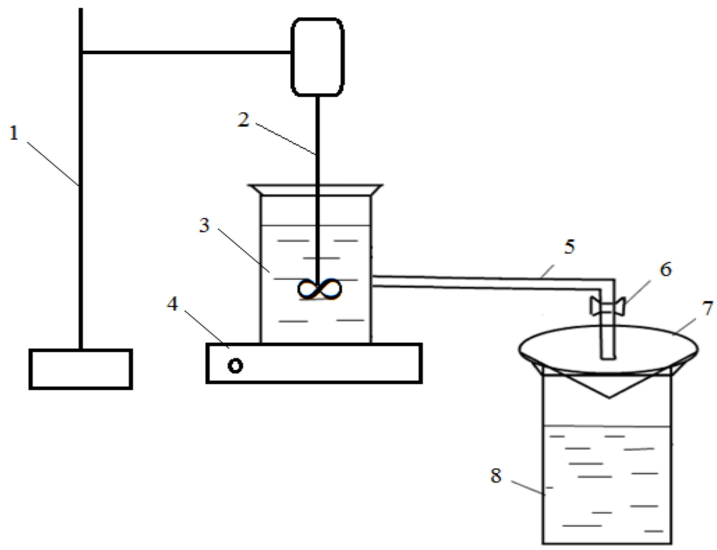

Fig.1. Diagram of the laboratory setup for extracting a concentrate of rare-earth elements from phosphogypsum: 1 – tripod; 2 – vertical mixer; 3 – container with pulp; 4 – furnace; 5 – connecting tube; 6 – faucet; 7 – filter; 8 – container with filtrate

The research was conducted in following way. 250 g of phosphogypsum obtained at the Dniprovsky Mineral Fertilizer Plant and from the dumps of the Pridniprovsky Chemical Plant were added in container 3 and filled with 500 ml of nitric acid solution with a concentration of 25%, 30%, and 35%, respectively. After that, the container with the pulp was placed on the stove and heated for 30 minutes at different temperatures, namely 70, 75, and 80°C with stirring and air bubbling [21].

After 20 minutes, the faucet was opened and the sediment was filtered through the tube through the filter and washed with 50 ml of water. The filtrate was sent to separate the sediment from it - a concentrate of rare-earth elements. The washed sediments were removed as a final product – the purified phosphogypsum, which was later used in the technology of obtaining complex mineral fertilizer [21].

The chemical parameters of nitric acid used in the research are presented in the Table 1 [22].

Table 1

Quality of nitric acid used in research

Characteristic	Norm
Mass fraction of nitric acid (HNO_3), %, no less:	56
The mass fraction of the residue after piercing, in the form of sulfates, %, no more	0.00.3
Mass fraction of sulfates (SO_4), %, no more	0.00.05
Mass fraction of phosphates (PO_4), %, no more	0.00.005
Mass fraction of chlorides (Cl), %, no more	0.00.010
Mass fraction of ferrum (Fe), %, no more	0.00.010
Mass fraction of arsenic (As), %, no more	0.00.0001
Mass fraction of heavy metals (Pb), %, no more	0.00.002

The mathematical model of the process of obtaining a concentrate of rare-earth elements from phosphogypsum using nitric acid was developed on the basis of the obtained experimental data for more efficient conducting of experiments and reduction of costs for their organization.

The following factors were chosen as the factors on which the degree of extraction of the concentrate of rare-earth elements depends: X_1 – concentration of nitric acid, %; X_2 – temperature, °C; Y_1 – the mass of the extracted REE concentrate from the phosphogypsum dumps of the enterprise "Prydniprovsky Chemical Plant", g; Y_2 – the mass of extracted REE concentrate from phosphogypsum obtained at the enterprise "Dniprovsky Mineral Fertilizer Plant".

The results of a series of laboratory experiments for phosphogypsum from the dumps of the enterprise "Prydniprovsky Chemical Plant" and obtained at the enterprise "Dniprovsky Mineral Fertilizer Plant" are summarized in the Table. 2, which shows the values of the initial parameters – X_1, X_2 and extraction of REE – Y_1, Y_2.

Table 2

Values of input parameters and results of experiments for phosphogypsum from the dumps of the enterprise "Prydniprovsky Chemical Plant" and obtained at the enterprise "Dniprovsky Mineral Fertilizer Plant"

№	X_1	X_2	Y_1	Y_2
1	25	70	26.0	16.7
2	30	70	16.0	13.3
3	35	70	11.0	11.1
4	25	75	15.8	12.0
5	30	75	13.1	10.1
6	35	75	9.8	8.6
7	25	80	14.0	9.0
8	30	80	10.6	7.8
9	35	80	8.7	6.3

In order to identify the interrelationship of the parameters of the studied process of REE concentrate extraction, a correlation analysis was performed, namely, the calculation of the Pearson linear correlation coefficient, confidence intervals, and the hypothesis about the significance of paired correlation coefficients was tested; the results of which are presented in the Table. 3 and 4.

Table 3

Matrix of paired correlation coefficients of extraction of REE(Y_1) concentrate from initial parameters

	Temperature, °C	Concentration, %	Extraction of REE, g
Temperature, °C	1	0	–0.54.4
Concentration, %	0	1	**–0.72.7**
Extraction of REE, g	–0.54.4	**–0.72.7**	1

The highlighted correlation coefficients are significant at the $p < 0.05.000$ level, which determine the significant influence of the nitrate concentration parameter on the efficiency of REE extraction.

Table 4

Matrix of paired correlation coefficients of extraction of REE(Y_2) concentrate from initial parameters

	Temperature, ^{0}C	Concentration, %	Extraction of REE, g
Temperature, ^{0}C	1	0	**–0.82.2**
Concentration, %	0	1	–0.53.5
Extraction of REE, g	**–0.82.2**	-0.53.5	1

The selected correlation coefficients are significant at the $p < 0.050.00$ level, which determine the significant influence of the temperature parameter on the efficiency of REE extraction.

Analyzing the tables, we can see that in the first case the concentration parameter has a significant effect on the efficiency of REE extraction, and in the second case, that is the temperature parameter.

Results and discussion

As a result of the processing of experimental data by methods of mathematical statistics, correlation coefficients (Tables 3, 4) and regression equations for the entire array of studied variables were determined; graphs (diagrams) of the dependence of the initial parameters are given(Y_1,Y_2) from incoming (X_1,X_2) in the form of graphsY_1(X_1) and Y_2(X_2) and multiple regression equations Y_1 (X_1,X_2), Y_2 (X_1,X_2), which are represented by 3D graphs of response surfaces and 2D graphs of the level line maps (X_1, X_2,Y_1 and X_1, X_2,Y_2).

According to the data of the experiments presented in the Table.2, the multiple regression equation of the dependence of REE extraction on concentration and temperature was also obtained in the following form:

$$Y_1=675.1944 - 1051.1667\cdot X_1 - 12.4667\cdot X_2+393.3333\cdot X_1^2+9.7\cdot X_1\cdot X_2+0.0593\cdot X_2^2. \quad (1)$$

The average relative error of the equation accounts for 6.27% with a standard deviation of 4.86.

The multiple regression equation Y_1 (X_1, X_2) (1) is represented by the graph of 3D response surfaces in Fig. 2 and the graph of 2D line maps of REE extraction levels (Y_1) depending on concentration (X_1) and temperature (X_2), which are presented in Fig. 3.

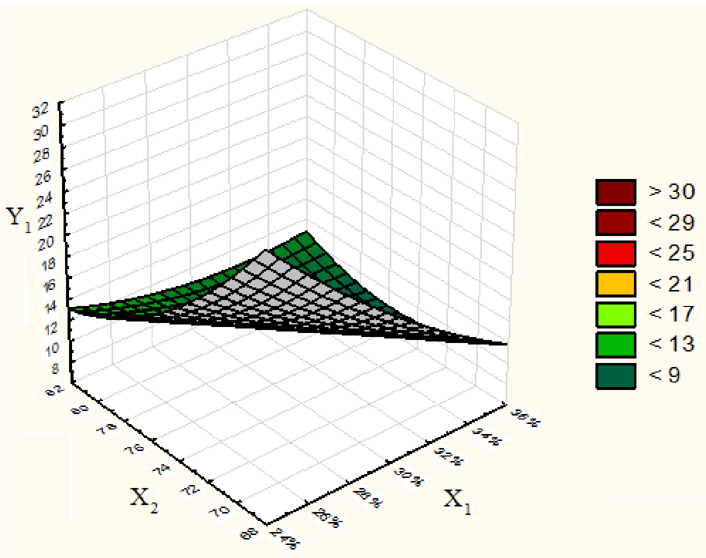

Fig.2. The response surface for the multiple regression equation Y_1 (X_1, X_2), equation (1)

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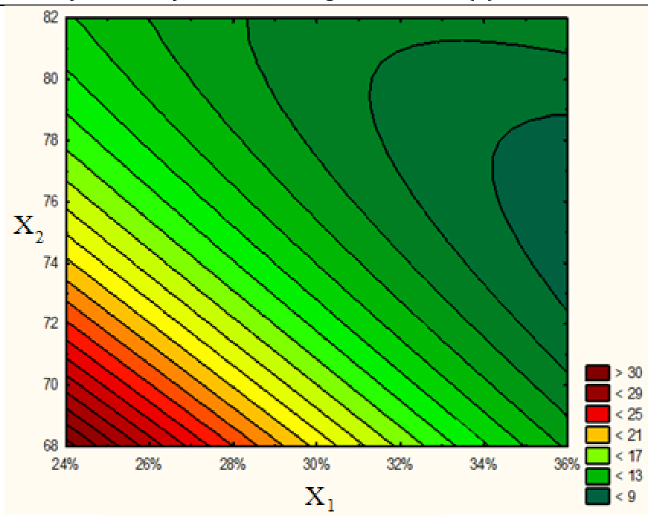

Fig.3. The graph of the dependence of extraction of REE concentrate Y_1 on the concentration of nitric acid X_1 and treatment temperature X_2, equation (1)

Analyzing the graph, it can be seen that the maximum extraction of rare-earth elements from phosphogypsum can be obtained at the concentration of nitric acid X_1 (24–26 %) and the values of the treatment temperature X_2 (68–72°C).

According to the data of the experiments presented in the Table.2, the multiple regression equation of the dependence of REE extraction on concentration and temperature was also obtained in the following form:

$$Y_2 = 244.8389-308.5 \cdot X_1-4.27 \cdot X_2+86.6667 \cdot X_1^2+2.9 \cdot X_1 \cdot X_2+0.0187 \cdot X_2^2. \quad (2)$$

The average relative error of the equation accounts for 4.67 % with a standard deviation of 3.19.

The multiple regression equation Y_2 (X_1,X_2) (2) is represented by the graph of 3D response surfaces in Fig. 4 and the graph of 2D line maps of REE extraction levels (Y_2) depending on concentration (X_1) and temperature (X_2), which are presented in Fig. 5.

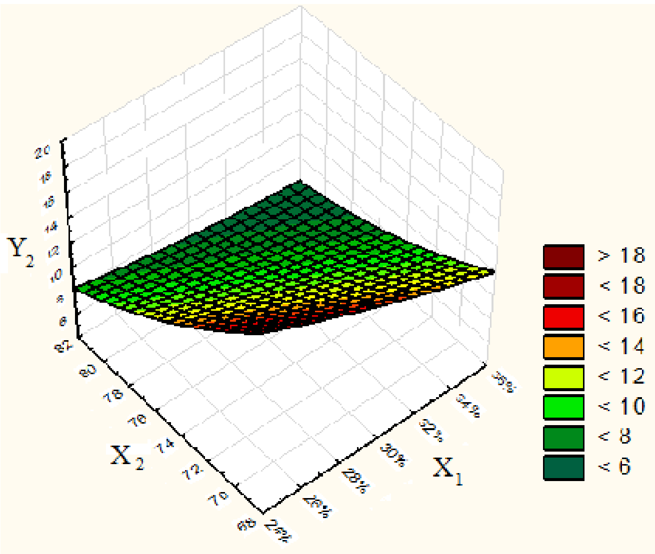

Fig.4. The response surface for the multiple regression equation Y_2 (X_1, X_2), equation (2)

After analyzing the graph, it was established that the extraction of rare-earth elements from phosphogypsum increases at the temperature (68–70°C) and acid concentration (24–26 %).

As the result of the conducted research and its mathematical processing, it was established that for the extraction of rare-earth elements from phosphogypsum in both cases, the most optimal parameters are the processing temperature of 70 °C and the concentration of nitric acid of 25 %.

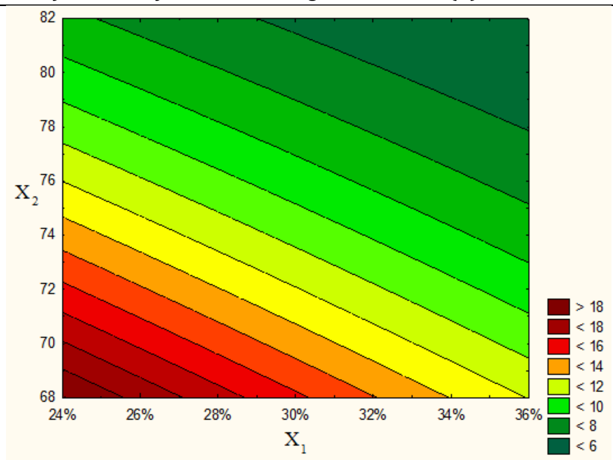

Fig.5. The graph of the dependence of extraction of REE concentrate Y_2 on the concentration of nitric acid X_1 and treatment temperature X_2, equation (2)

After analyzing the obtained data, the technological scheme for the processing of phosphogypsum was created with the selection of the concentrate of rare-earth elements and the simultaneous production of calcium sulfate hemihydrate, which is presented in Fig. 6.

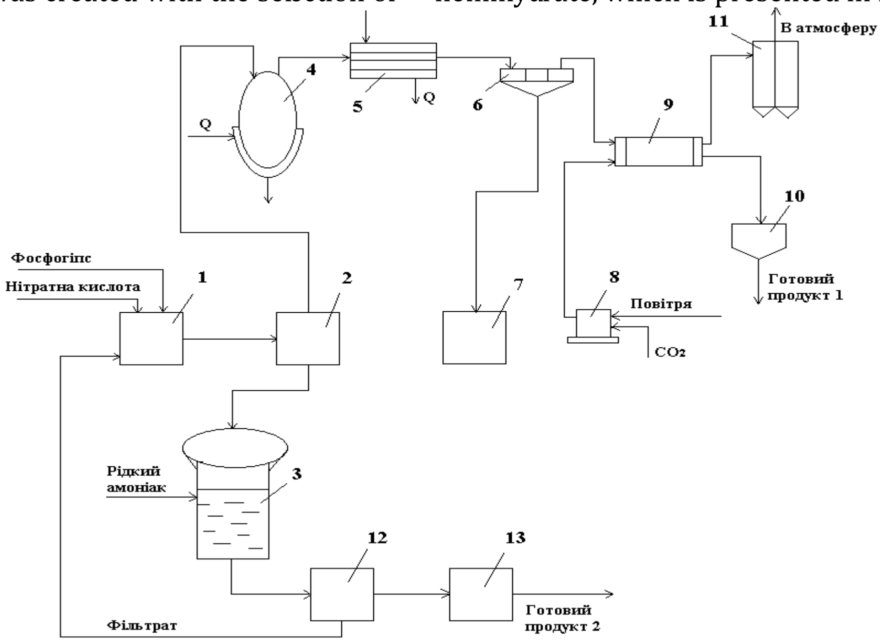

Fig.6. The technological scheme of phosphogypsum processing with the selection of the concentrate of rare-earth elements and simultaneous production of calcium sulfate hemihydrate: 1 – mixer; 2, 6, 12 – vacuum filters; 3, 7 – leachate collectors; 4 – autoclave; 5 – heat exchanger; 8 – furnace; 9 – drum dryer; 10 – hopper with final products; 11 – cyclone; 13 – steam dryer

The processing of phosphogypsum according to the given scheme takes place by means of its nitric acid treatment in the form of a pulp with a ratio of "phosphogypsum: acid" of 1:2.

Nitric acid with a concentration of 25 % and phosphogypsum are fed into the mixer 1, the components are mixed for 30 minutes at the temperature of 70 °C.

After mixing, the reacted pulp is filtered through the vacuum filter 2. The filtrate is sent to separate the concentrate of rare-earth elements from it. Liquid ammonia with the concentration of 25 % is added to the filtrate in the collector 3 to bring the pH value to 6–7, after which the filtrate, together with the separated REE sediment, goes to the vacuum filter 12 for filtration and washing. The filtered and washed sediment of rare-earth elements is fed into the steam dryer, where it is dried at the temperature of 100 °C and discharged as the final product, and the filtrate from the vacuum filter 12 is returned to the process by mixing with fresh nitric acid for feeding to the mixer 1.

Conclusions

Based on the analysis of literary sources, it was concluded that the effective use of phosphogypsum requires the development of the complex technology for its processing, which involves the extraction of REE and the use of washed phosphogypsum in the chemical industry. With the help of the conducted research, the mathematical model of the process of obtaining the concentrate of rare-earth elements from phosphogypsum using nitric acid was created.

On the basis of experimental studies, regression equations were obtained, that allow determining the dependence on temperature and acid concentration on the extraction of the concentrate of rare-earth elements from phosphogypsum.

With the help of constructed 3D models, it is shown that the greatest concentration of rare-earth elements is observed in the sample of phosphogypsum taken from the dumps of the enterprise "Prydniprovsky Chemical Plant" in the city of Kamianske.

As the result of the conducted research and description of the mathematical model of the obtained data, it was found that the most optimal parameters for both types of phosphogypsum are the concentration of nitric acid in the range of 24–26 % and the processing temperature of 68–70°C.

The technological scheme for the processing of phosphogypsum with the selection of the concentrate of rare-earth elements and the simultaneous production of calcium sulfate hemihydrate with optimal parameters is proposed: the processing temperature accounts for 70^{0}C and the concentration of nitric acid accounts for 25 %.

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