### INTENSIFICATION OF TECHNOLOGY FOR PRODUCTION OF EXTRACTION PHOSPHORIC ACID DECOMPOSITION OF APATITE AND PHOSPHORITE SOLUTIONS OF PHOSPHORIC AND SULFURIC ACIDS

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*Abstract.* The introduction discusses the relevance and justification of the tasks for solving the problem of intensifying the production of EPA.

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### **INTRODUCTION**

The introduction discusses the relevance and justification of the tasks for solving the problem of intensifying the production of EPA.

The introduction of the article examines the structure of natural reserves and mineralogical composition of Russian phosphate raw materials used in the production of EPA. It is noted that the specific structure of phosphorite ores and the presence of impurities in them require scientific and technical research to determine ways to economically process each type of phosphorite.

An analysis of the known patterns of decomposition of phosphate raw materials was carried out. The physicochemical basis for the production of extraction phosphoric acid is considered. It is shown that the decomposition stage includes two processes: dissolution of the initial phosphate raw material and gypsum crystallization. In modern standard technological schemes, these processes are combined in hardware and time, which leads to the deposition of falling gypsum crystals on dissolving particles, and as a consequence to a decrease in the degree of extraction of phosphate raw materials and a decrease in technology productivity.

When considering standard technologies for producing extraction phosphoric acid, four main disadvantages leading to a decrease in productivity were identified: uneven mixing of pulp with sulfuric acid; joint dosing of various types of phosphate raw materials; combination of dissolution and crystallization processes; low filter performance, limiting the process.

Analysis of hardware and technological solutions for EFC production. showed that the main trends in the development of the hardware design of reaction equipment for the production of EPA consist in organizing a more rational movement of pulp flows, which make the reaction pulp more homogeneous in chemical characteristics, and also simultaneously reduce and equalize the temperature gradient at various points of the reaction system.

An analysis of the physico-chemical foundations of absorption methods for capturing fluorine-containing gases and catching HF and HCl fog has been completed. An analysis of the apparatus and process of absorption of fluorine-containing gases produced by EPA showed that by absorption with water it is possible to reduce the concentration of fluorine in the exhaust gases to 20-30 mg/m3. It is noted that the absorption process of fluorine-containing gases in the production of EPA is accompanied by the formation of gel-like compounds that clog the structural elements of the devices and gas ducts, which requires additional experimental research into the

patterns of kinetics of absorption of HF and HCl in order to increase the efficiency of gas absorption processes and solve environmental problems in the production of EPA.

Tasks have been set for further intensification of EPA production technology at the stages of raw material preparation, decomposition of phosphate raw materials, crystallization of phosphogypsum and purification of waste gas emissions.

a methodology for experimental study of the laws of the decomposition process of phosphate raw materials is presented. An experimental setup for studying the kinetics of thermal decomposition of phosphorites, and an experimental setup for studying the patterns of processes for purifying fluorine and chlorine compounds from waste gases are described. Diagrams of the experimental setups are presented in Fig. 1.



Fig. 1. Diagram of the experimental setup for studying the kinetics of thermal decomposition of phosphorites. 1 - laboratory scales (electronic), 2 - stand, 3 - quartz tube, 4 - oven, 5 - thermocouple, 6 - glass tube, 7 - millivoltmeter, 8 - LATR, 9 - sample.

The study of the patterns of vapor absorption and mist capture of HF and HCl was carried out through the absorption of model gases produced by EPA in the presence of fiber filter packages at different HF ratios.



## Fig. 2. Diagram of the experimental setup for studying the laws of the process of vapor absorption and HF and HCl mist capture.

1 – reactor, 2 – splash guard, 3 – main element – absorber, 4 – air outlet for analysis, 5 – filter element, 6 – filter material, 7,8,9,10 – Drexel, 11 – rheometers, 12,13 – cartridges for AFA filters, 14,15,16,17,18 – taps, 19 – pump, 20 – gas meter, 21 – thermostat, 22,23 – thermometer.

A description of the results of an experimental study of the laws of the process of decomposition of phosphorytes and apatitis of phosphorus and sulfuric acid was performed.

The results of a study of the patterns of the formation of phosphogypsum crystals during the decomposition of phosphate raw materials are presented.

The results of an experimental study of the influence of the quality of preparation of raw materials on the efficiency of the decomposition process of phosphate raw materials are described.

The results of an experimental study of the laws of the process of purification of waste gases from EPA production from fluorine compounds are presented.

The decomposition of phosphates with sulfuric acid proceeds with the formation of phosphoric acid and calcium sulfate according to the reaction:

 $Ca_{5}(PO_{4})_{3}F + 5H_{2}SO_{4} + 5nH_{2}O \longrightarrow 5CaSO_{4} \cdot nH_{2}O + 3H_{3}PO_{4} + HF$ (1)

The results of the experimental study are presented in Fig. 3 show that the starting phosphate reacts first with phosphoric acid according to the reaction:

 $Ca_{5}F(PO_{4})_{3} + nH_{3}PO_{4} \rightarrow 5Ca(H_{2}PO_{4})_{2} + (n-7)H_{3}PO_{4} + HF$  (2)

Then, the resulting monocalcium phosphate, Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>, reacts with sulfuric acid in the presence of phosphoric acid according to the reaction:

 $Ca(H_2PO_4)_2 + H_2SO_4 + mH_3PO_4 \rightarrow CaSO_4 + (m+2)H_3PO_4$ (3)



# Fig. 3. Change in the mass of the solid phase during the decomposition of apatite by sulfuric acid in the system: Apatite: H3PO4:H2SO4, at different initial L:S ratios.

1 - F: T = 20:1; 2 - F: T = 40:1.

From the curves in Fig. 3 it is clear that at a ratio of L:T = 40:1 (line 2), the process of apatite decomposition occurs with almost complete dissolution of the original apatite (the initial section of curve 2 to a minimum) and the formation of a solution of monocalcium phosphate, and then with the release from the solution crystalline reaction product - phosphogypsum.

Studies of the process mechanism under conditions close to industrial ones in terms of the L:S ratio = 2.5 have shown that the formulated process mechanism works not only at large L:S ratios, but also when the process is carried out at a temperature of  $85^{\circ}$ C with a L:S ratio = 2.5.

When the F: T reaches less than 2.5, a crust of calcium sulfate forms on the particles of decomposing phosphate raw materials.

In Fig. Figure 4 shows the dependence of the change in the rate of the decomposition process on the mass fraction of phosphate relative to the initial one.



## Fig. 4. Dependence of the change in the rate of decomposition of phosphate raw materials in a batch apparatus on the mass fraction of the feedstock (temperature 85°C).

1 – apatite, 2 – phosphorite.

From Fig. 4 it can be seen that the process of decomposition of phosphorite proceeds faster than the process of decomposition of apatite. However, the mechanisms of the decomposition processes of various phosphate raw materials are the same from the beginning of decomposition and almost to the end (up to a degree of decomposition of 99.9%).

At the ratio of liquid and solid phases – L: T = 2.5 and temperature  $T = 85^{\circ}$ C, the kinetics of the process is described by the equation:

$$\frac{dm}{d\tau} = km^{1} \tag{4}$$

Issues related to the choice of conditions for the industrial implementation of the process of decomposition of phosphate raw materials during the production of EPA are considered. Recommendations for further intensification of production and initial data for designing the reconstruction of an industrial plant\* have been developed.

Data from pilot industrial tests of the developed technology were analyzed. The results of pilot industrial tests of the developed technology showed that the decomposition coefficient increased from 96.8-97.2% to 97.2-98.3%. In addition, the performance of the filtration process has been significantly improved. Detailed data on the results of a comparative analysis of the indicators of the existing and developed technology according to the plant data are presented in.

#### CONCLUSIONS

1. The scientific and technical foundations of the method for producing extraction phosphoric acid by the decomposition of phosphorites and apatite with a mixture of sulfuric and phosphoric acids were further developed.

2. It has been shown that the process of firing the initial phosphorus-containing raw materials significantly affects the patterns of formation of phosphogypsum crystals. A mathematical description of the kinetics of the firing process of phosphate rock has been carried out. It is shown that after effective firing with a decrease in the carbonate part in phosphate raw

materials from 11.7% to 3%, with subsequent sulfuric acid decomposition of phosphorites, the size of phosphogypsum crystals increases from 50 to 250 microns.

3. For the first time, it was shown that the temperature dependence of the rate of decomposition of both apatite and phosphorite is a generalized straight line without kinks (in Arrhenius coordinates). For both dihydrate and hemihydrate decomposition methods in the temperature range  $70 - 120^{\circ}$ C. Generalization and mathematical description of the laws of the kinetics of decomposition processes of phosphate rock, apatite and their mixture showed that the activation energy of the decomposition process for both apatite and phosphorite Eact = 5.4 kcal/mol. The limiting stage of the decomposition process is diffusion through the monocalcium phosphate gel layer.

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