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Research of nitric acid processing of phosphorite meal and man-made waste of phosphorite enrichment of Central Kyzylkum

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ABSTRACT: The results of studies of the fractional composition of the mineralized mass, the effect of an increased rate of 40% nitric acid on the composition of nitric acid pulps, and the filtration rate of acidic and ammoniated pulps are presented. The possibility of obtaining liquid nitrogen-calcium fertilizer and fertilizer precipitate is shown.

I. INTRODUCTION

Uzbekistan has a powerful industrial base for the extraction and processing of phosphate raw materials. The raw materials are phosphorites of the Central Kyzylkum (CK). The Kyzylkum Phosphorite Complex supplies 716 thousand tons of washed calcined phosphate concentrate per year. This is about 265 thousand tons of P_2O_5 , while the needs of the Republic exceed 800 thousand tons of P_2O_5 per year [1, 2]. The shortage of phosphate fertilizers is aggravated by the fact that a large amount of nutrients are removed from the soil with the harvest. Another important factor is the low coefficient of phosphorus use of mineral fertilizers by plants - 20-25% in the first year of phosphorus application of phosphate fertilizers and 40% in the next 2-3 years [1, 3]. The low use is explained by the fact that phosphate fertilizers are in the soil in a sedentary state and are not completely absorbed by the roots of plants. The lack of phosphate fertilizers leads to a decrease in crop yields, as well as to a decrease in the resistance of cotton to wilt. All this indicates that it is necessary to increase the production of phosphorus-containing fertilizers in the Republic.

II. LITERATURE SURVEY

On the other hand, when enriching 1875 thousand tons of phosphorite ore, 1158 thousand tons or 134770 tons of P_2O_5 , are not suitable for obtaining extractive phosphoric acid (EPA), which is 42% of the total amount of mined phosphorite ore [4]. The main part of the phosphorite enrichment waste of the Central Committee is the mineralized mass (MM), which is currently not used and stored. More than 14 million tons of waste have already accumulated, which contain from 1.56 to 1.82 thousand tons of P_2O_5 with an average content of 13.75% P_2O_5 . The main amount of enrichment waste is mineralized mass (MM). If we take into account that phosphate rock - unenriched phosphate raw materials (UPRM) is used only to obtain simple ammoniated superphosphate, then the involvement of MM and UPRM in industrial production will provide additional production of phosphate fertilizers. With the provision of the agro-industrial complex of the republic in phosphate fertilizers by 30-35%, this is a very large reserve.

Solving the problem of processing unenriched phosphate raw materials (UPRM) and mineralized mass (MM) is an urgent problem not only for the chemical industry, but also for environmental protection, the solution of which will allow involving an additional amount of phosphate raw materials into processing, increase the production of phosphorus-containing fertilizers, and provide agriculture with an additional amount of phosphate fertilizers. Therefore, our research was aimed at finding the conditions for processing MM and UPRM with the separation of the phosphate part.

**III. RESEARCH METHODS**

The chemical, mineralogical composition, physicochemical characteristics of phosphorites of the Central Committee are well studied and reflected in the scientific and technical literature [1, 2]. Information on the use of MM and its properties is very limited. To supplement these data, the literature data on the compositions of NFR and MM were analyzed in detail. Table 1 shows data on the compositions of NFRs and MMs used for scientific research by various authors [5–8].

The phosphate rock used by the authors contains 16.44-18.78% P_2O_5 , 45.76-48.62% CaO, 1.00-1.82% MgO, 0.78-2.47 Fe_2O_3 , 1.10-2, 85% Al_2O_3 , 3.02-17.55% CO_2 , 1.70-2.17% F and has a calcium modulus of 2.51-2.93. The mineralized mass contains (mass %): 11.73-14.71% P_2O_5 , 40.80-43.99% CaO, 0.53-1.44% MgO, 0.53-1.44% Fe_2O_3 , 1.04-1.37% Al_2O_3 , 12.84-19.20% CO_2 , 1.30-1.91 F and has a calcium modulus of 2.78-3.34.

For our studies, we used NFS composition (wt.%): P_2O_5 -16.97; CaO - 47.02; MgO - 1.48; Fe_2O_3 - 1.28; Al_2O_3 - 1.21; CO_2 - 15.44; SO_3 - 1.95; F- 1.92 N.O. - 6.99 and with calcium module 2.78, MM composition (wt.%): P_2O_5 -12.86; CaO - 42.80; MgO - 0.80; Fe_2O_3 - 1.37; Al_2O_3 - 1.17; CO_2 - 12.81; SO_3 - 2.00; F- 1.85 with a calcium module of 3.17. H.O. - 11.89, H_2O - 0.89 and with a calcium module 3.17.

Chemical analysis of initial, intermediate, and final products was carried out by known methods of analysis [9–11].

IV. EXPERIMENTAL RESULTS

For MM, the fractional chemical composition was studied (Table 2). The table shows that the main fractions are -7 mm and -2 mm, which in total amount to 74.72%.

The content of P_2O_5 decreases from 14.33% for the +7 mm fraction to 11.55% for the -2 - +1 mm fraction and increases in the -1 mm fractions. The maximum amount of P_2O_5 is contained in fractions +7 mm and -1 mm. The content of CaO averages 40-43%. The exceptions are fractions -7 - +5 mm, -1 - +0.5 mm, where the minimum content is 36.79% and 36.90%, respectively. The maximum content of CaO is observed in the -0.5 mm fraction, which is 48.15%. The content of CO_2 in concentration from 2.35% for the number +7 mm and is 0.46-1.69% for the number from -7 mm to +0.5 mm. The maximum content of CO_2 occurs in fractions -0.5 mm -4.31%.

Table 1

The chemical composition of unenriched phosphate raw materials and mineralized mass used for research by other authors.

| № | Chemical composition,, mass. % | | | | | | | CaO:P ₂ O ₅ |
|------|------------------------------------|-------|------|--------------------------------|--------------------------------|-----------------|------|-----------------------------------|
| | P ₂ O ₅ общ. | CaO | MgO | Fe ₂ O ₃ | Al ₂ O ₃ | CO ₂ | F | |
| UPRM | | | | | | | | |
| 1 | 16,44 | 45,76 | 1,00 | 0,80 | 1,10 | 17,55 | 1,70 | 2,78 |
| 2 | 16,60 | 48,62 | 1,42 | 0,78 | | 13,02 | 2,17 | 2,93 |
| 3 | 17,20 | 46,22 | 1,75 | 1,05 | 1,24 | 16,00 | 2,00 | 2,69 |
| 4 | 17,65 | 47,48 | 1,75 | 2,47 | 1,22 | 15,20 | 1,81 | 2,69 |
| 5 | 18,45 | 46,47 | 1,50 | 2,09 | 2,85 | 15,20 | 1,81 | 2,52 |
| 6 | 18,78 | 47,09 | 1,82 | 1,05 | 1,24 | 15,65 | 2,00 | 2,51 |
| MM | | | | | | | | |
| 7 | 12,86 | 42,80 | 0,80 | 1,37 | 1,17 | 12,81 | 1,85 | 3,17 |
| 8 | 13,75 | 43,99 | 0,70 | 1,44 | 1,31 | 19,20 | 1,91 | 3,20 |
| 9 | 14,60 | 43,99 | 1,01 | 0,89 | 1,04 | 14,11 | 1,30 | 3,01 |
| 10 | 14,68 | 40,80 | 0,53 | 1,37 | 1,17 | 12,84 | 1,86 | 2,78 |
| 11 | 14,71 | 41,52 | 0,53 | 1,37 | 1,17 | 15,61 | 1,85 | 2,82 |
| 12 | 11,73 | 41,52 | 1,17 | 0,53 | 1,37 | 15,61 | 1,85 | 3,34 |

The chemical composition of unenriched phosphate raw materials and mineralized mass, used for research by other authors.

Nitric acid processing of phosphate raw materials allows the use of poor phosphorites, such as NFS and MM. However, the presence of organics in their composition forms a non-separable consistency, which is practically not separated. The existing methods for obtaining phosphorus-containing fertilizers are based on the further processing of nitrogen-calcium extract (NCE).

Our research was aimed at finding the possibility of separating the pulp of nitric acid decomposition. An increase in the S:L ratio during the acid decomposition of phosphate raw materials intensifies the processes of separation of the liquid and solid phases [8]. Therefore, studies of the decomposition process of UPRM and MM were carried out with 40% nitric acid at its rate of 100, 105, 110, 125 and 150% at room temperature, constant stirring speed and process duration of 30 minutes. For this, a theoretical calculation of the norm of 40% nitric acid was carried out, taking into account all the components of the raw material. The acid concentration is chosen for the purpose of obtaining a pulp with a density of at least 1.55-1.65 g/cm³. A suspension with such a density is considered "heavy", where carbon dioxide bubbles rising from its volume, when detached from the phase interface, are not able to entrain heavy liquid phase spheres in the composition of a thin layer of spheres. Annihilation of the bubbles occurs, with the release of their contents in the form of carbon dioxide and liquid. This helps to reduce foaming during the decomposition of (UPRM) and MM. The data obtained are shown in Table 2.

Table 2.
Influence of the norm of 40% nitric acid on the chemical composition of the pulp from UPRM and MM.

| № | HNO ₃ % | Chemical composition,, mass. % | | |
|------|-----------------------|--------------------------------|-------|-------------------------------|
| | | P ₂ O ₅ | CaO | R ₂ O ₃ |
| UPRM | | | | |
| 1 | 100 | 6,95 | 20,58 | 1,01 |
| 2 | 105 | 6,64 | 19,87 | 0,97 |
| 3 | 110 | 6,34 | 19,20 | 0,92 |
| 4 | 125 | 5,50 | 17,35 | 0,81 |
| 5 | 150 | 4,57 | 14,46 | 0,68 |
| MM | | | | |
| 6 | 100 | 4,76 | 15,63 | 0,95 |
| 7 | 105 | 4,66 | 15,27 | 0,93 |
| 8 | 110 | 4,56 | 14,92 | 0,91 |
| 9 | 125 | 4,27 | 13,89 | 0,85 |
| 10 | 150 | 3,56 | 11,57 | 0,71 |

The table shows that with an increase in the rate of nitric acid, the content of all components decreases. Thus, the content of P₂O₅ at an acid number of 100% is 6.95% for the decomposition of UPRM and 4.76% for the decomposition of MM. Increasing the acid rate to 150% leads to a decrease in these indicators to 4.57% and 3.56%, respectively. CaO values decrease from 20.58% and 15.63% to 14.46% and 11.57%, respectively, for UPRM and MM. Similarly, the decrease in the content of sesquioxides.

The results obtained showed that, regardless of the norm of nitric acid, the resulting pulps practically do not delaminate and are practically not filtered.

N. Abdullaeva found that the filtration rate of nitric acid pulp increases 7-10 times in the presence of ammonium nitrate [12]. Therefore, the influence of the ammonization process on the filterability of nitric acid pulp was studied and it was found that when pH reaches 5-7, the pulp filtration rate reaches 1652-2380 kg/m².h. (Fig. 1).

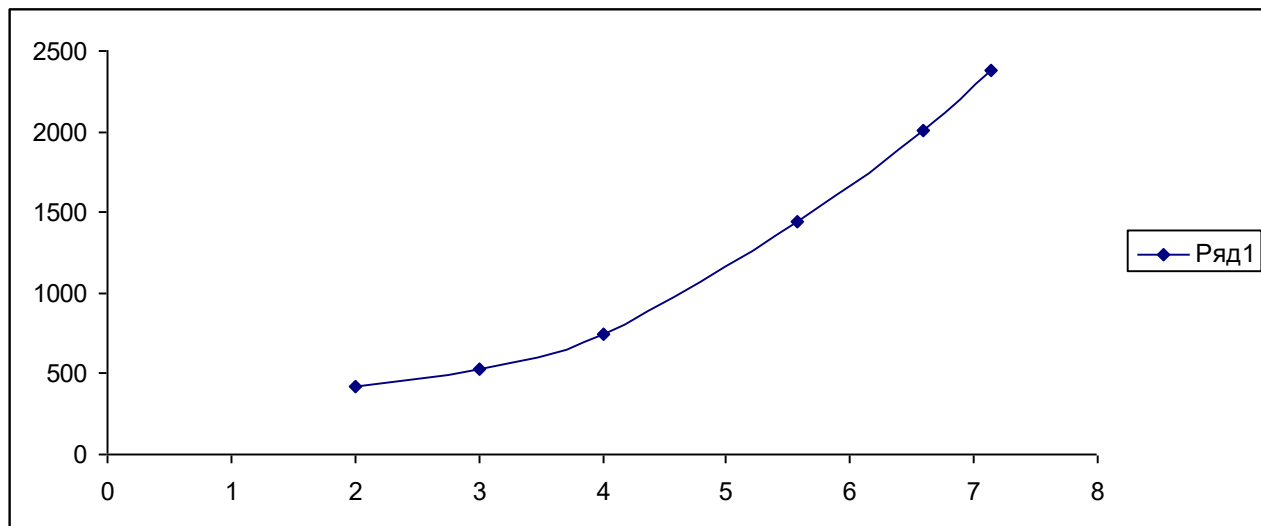


Figure. 1. Influence of pH on the filtration rate of nitric acid pulp during ammonization

As a result of the separation of the ammoniated pulp, obtained at a rate of nitric acid of 105% and ammoniated to pH 7, a filtrate was obtained, which is a solution of calcium nitrate and a precipitate in the form of a fertilizer precipitate in one technological cycle.

IV. CONCLUSION

As a result of the research, a technical solution was found that makes it possible to separate the nitric acid pulp obtained on the basis of phosphorite flour (UPRM) and man-made enrichment wastes (MM) of phosphorites of the Central Kyzylkum in one technological cycle and obtain two types of fertilizers.

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