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Research of the Process of Processing of Nitrogen Sulfate Solutions for Enhancing the Phosconcentrate of Central Kyzylkum to Liquid Fertilizers

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ABSTRACT: The results of processing the solutions of enrichment of the washed calcined phosconcentrate of the Central Kyzylkum with weak solutions of nitric acid are given. It has been established that the enrichment solutions even after the third cycle of return to the enrichment stage contain no more than 6.5% of nitrogen and, in order to obtain liquid nitrogen-calcium fertilizers, it is necessary to introduce additionally into nitric acid enrichment solutions and ammoniate them with gaseous ammonia to pH of 7-7.5.

KEYWORDS: washed burnt phosconcentrate, nitric acid, enrichment solution, gaseous ammonia, liquid nitrogen-calcium fertilizers.

I.INTRODUCTION

The identified world reserves of phosphate ores are estimated at 70.6 billion tons of P_2O_5, of which 65.3 billion tons are phosphorite and 5.3 billion tons are apatite [1]. The largest and richest deposits are concentrated in 10 countries and make up 87% of the world's total reserves. In recent years, there has been decline in the quality of phosphate raw materials. So for the last 40 years the average content of phosphorus anhydride has decreased from 34% P_2O_5 to 30-32%. The reserves of the rich in P_2O_5 ores are steadily becoming depleted and the poorer phosphorite and apatite ores are being involved in industrial processing.

For enterprises of Uzbekistan producing phosphorus fertilizers, the only raw materials are phosphorites of the Central Kyzylkum, which along with high carbonate content are characterized by thin germination of the phosphate mineral with calcite and the presence of chlorine. The existing ways of preparing the ore did not yield positive results. Therefore, the ore is washed and subjected to calcination. As a result, the composition of the washed burnt phosconcentrate contains up to 17% of free calcium oxide, which is the reason for the temperature rise in the extractor, the formation of calcium sulphate hemihydrate, and the increased corrosion activity of phosphoric acid [2]. Therefore, it is very important to search for new and effective methods for enriching the phosphorites of the Central Kyzylkum. An important problem is the processing of enrichment solutions.

Known methods of chemical enrichment of phosphorites due to the formation of large volumes of solutions of enrichment and consumption of acid reagents have not found industrial application [3-7]. In this regard, our research was aimed at finding ways to enrich the washed burnt phosconcentrate (WBPhC) of the Central kyzylkum and processing enrichment solutions.

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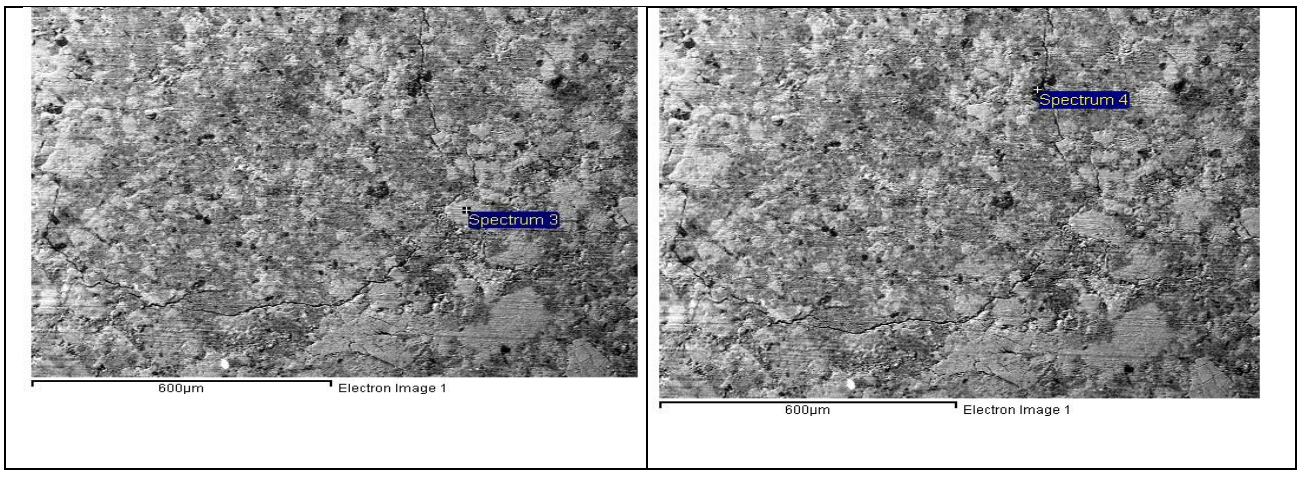
II. SIGNIFICANCE OF THE SYSTEM

The studies were carried out on laboratory unit consisting of reaction flask, an electromechanical stirrer, reflux condenser, thermometer and water thermostat. To this end, calculated amount of nitric acid was poured into the reaction vessel, kept in thermostat with working stirrer for 15 minutes at temperature of 25 °C, and then portion of the WBPhC was added and, with stirring, held for predetermined time. The contents of the reaction flask were then filtered, washed and dried at temperature of 105 °C. until constant weight. The liquid and solid phases were analyzed for P_2O_5, CaO content by known methods [8-10]. As the phosphate raw materials of the Central Kyzylkum, the composition of the composition was used (mass. %): P_2O_5 - 26.20; CaO - 57.70; CaO: P_2O_5 - 2.202; MgO = 0.60; Fe_2O_3 - 0.43; Al_2O_3 - 0.60; SO_3 - 3.78.

III. EXPERIMENTAL RESULTS

It is implemented on a training dataset consisting of five legitimate messages and five spam message. The true rate and For the convincing data that during the enrichment, washing and calcination of phosphorylates of the Central Kyzylkum, calcium compounds are retained in the enriched phosconcentrate, microphotographs of the raw unrefined phosphate raw materials were taken with an accurate analysis of the chemical elements present on the scanning electron microscope (Fig.). For comparison, photos of phosphorite were taken after enrichment, washing and calcination. In the photographs A and B, the dark inclusions refer to silicate compounds and contain silicon, iron, calcium aluminum, phosphorus. A light background is represented by compounds of calcium, phosphorus, iron. Enriched phosphorite has dark background and is due to an increase in the proportion of silicate compounds (Figure B). Light spots on microphotographs are represented by compounds of calcium, phosphorus, iron (Fig. D). The data confirm the information about decrease in the concentration of calcium carbonate during enrichment, washing and calcination and preservation of calcium in the form of an oxide, which is responsible for the high calcium modulus of phosconcentrate.



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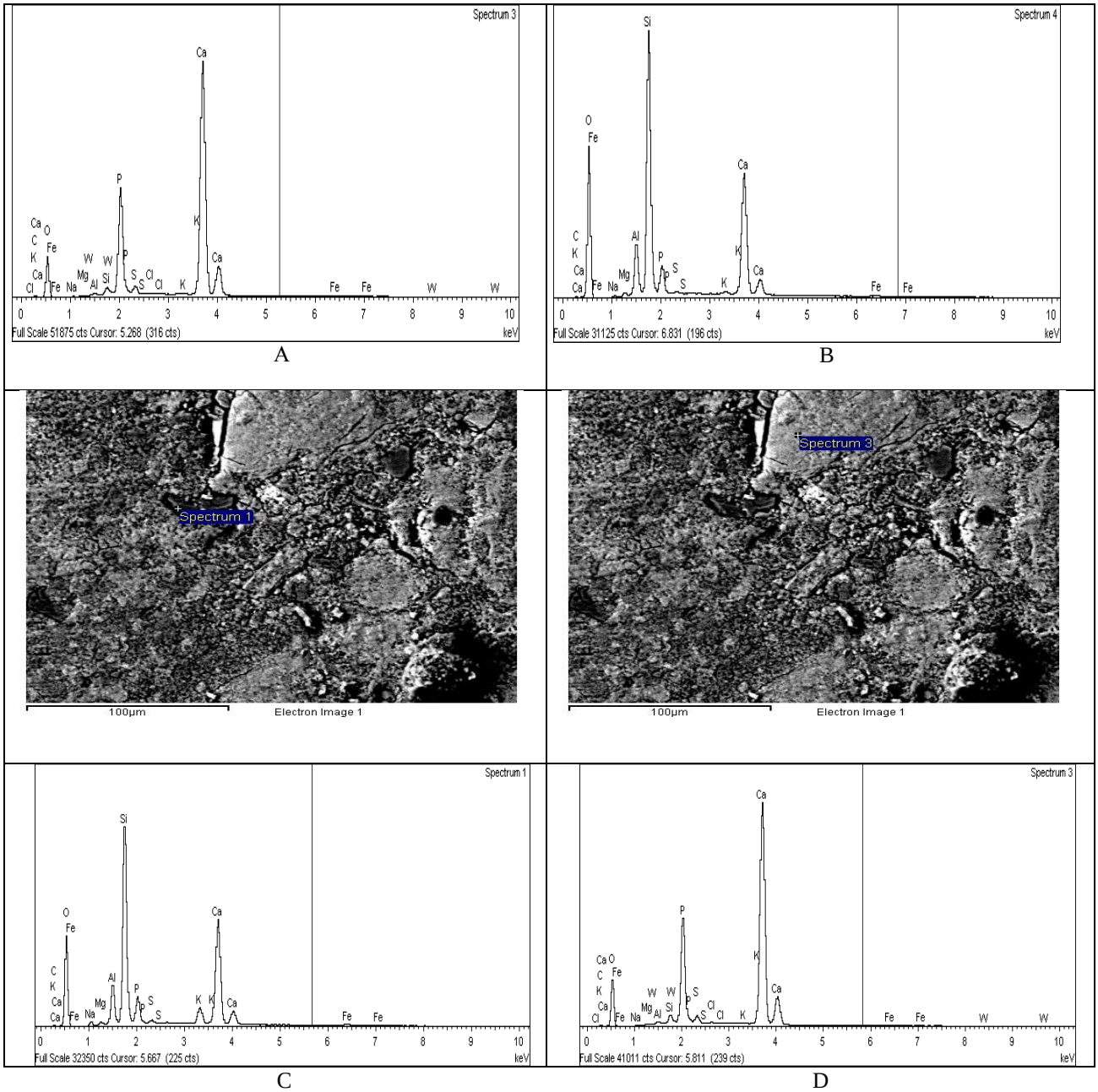

Fig. Microphotographs of the phosphate of the Central Kyzylkum, not enriched and enriched at 950 ° C, 6 a, b - unenriched, c, d - enriched.

To reduce the calcium module of the phosconcentrate, studies were carried out to enrich 15-20% with solutions of nitric acid at rate of 25-28% for CaO_2. Table 1 shows the effect of the concentration and the rate of nitric acid on the composition of the liquid and solid phases during the enrichment of the washed burnt phosconcentrate. These tables indicate the possibility of reducing the calcium module without loss of P_2O_5 in the enrichment solutions. At the same time, the calcium modulus decreases from 2.20 to 1.68, the P_2O_5 content rises from 26.20 to 31.97, and enrichment solutions containing 8.82-9.36% CaO or 25-28% calcium nitrate pH 2,89-4,17, which can be used to produce liquid and granular nitrogen-calcium fertilizers [11-12].

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Table 1

Effect of the concentration and the rate of nitric acid on the contents of the liquid and solid phases of enrichment of the washed burnt phosconcentrate

№	HNO_3, %	Norm HNO_3, %	The contents of the solid phase, mass. %		C.M.	Contents of the liquid phase, mass. %		
			CaO	P_2O_5		CaO	P_2O_5	pH
1	15	35	47,79	32,26	1,4814	8,82	1,02	2,89
2	15	30	47,61	31,97	1,4892	9,36	0,28	4,17
3	15	25	53,22	31,61	1,6836	6,62	0,00	7,34
4	20	25	53,68	31,41	1,7090	8,57	0,00	7,39

In view of the fact that in enrichment solutions the nitrogen content is not more than 4%, studies were conducted on the secondary use of enrichment solutions in order to increase the nitrogen content. The results are shown in Table 2.
When the enrichment solution was reused, 56% nitric acid was added to it, in an amount providing nitric acid rate of no more than 28% and water to maintain the initial H:L.

Table 2

Contents of solutions of nitric acid enrichment of WBPhC after one, two and threefold reuse

№	Multiplicity of use	pH	Chemical contents, mass. %			
			CaO	N	P_2O_5	$Ca(NO_3)_2$
1	Once	4,91	6,78	3,59	0,06	21,03
2	Twice	4,31	10,07	5,08	0,03	29,49
3	Threefold	4,00	12,96	6,48	0,03	37,95

It can be seen from the table that when the enrichment solution is reused, the nitrogen content is increased to 5.08%, and for triplicate use up to 6.48%. Accordingly, the content of calcium oxide increases from 6.75% to 10.07% and to 12.96%, respectively. The pH of the solutions varies within the range 3.96-4.91. This indicates that with the increase in the multiplicity of the use of the enrichment solutions, the degree of extraction of calcium oxide decreases. This can be explained by an increase in the concentration of calcium nitrate in the solutions and, correspondingly, an increase in the viscosity of the enrichment solutions. The concentration of calcium nitrate increases from 21.03% to 29.49% and 37.95%, respectively, after one-, two- and three-fold use.
Table 3 gives data on the change in density and viscosity of enrichment solutions, depending on the frequency of use and temperature.
The data in table 3 confirm our assumptions. Thus, the density of enrichment solutions at 20°C increases from 1.15 g/cm^3 to 1.19 g/cm^3 and up to 1.36 g/cm^3, and viscosity, respectively, from 1.34 mPa·s to 1.52 mPa·s and up to 2.65 mPa·s. With an increase in temperature from 20 to 80 °C, the density and viscosity are reduced.

Table 3

Influence of frequency of use and temperature on changes in density and viscosity of enrichment solutions

№	Multiplicity of use	Density, g/cm^3				Viscosity, mPa·s			
		20°C	40°C	60°C	80°C	20°C	40°C	60°C	80°C
1	Once	1,15	1,14	1,13	1,12	1,34	1,03	0,86	0,72
2	Twice	1,19	1,18	1,17	1,16	1,52	1,16	0,92	0,80
3	Threefold	1,36	1,34	1,33	1,32	2,65	2,04	1,61	1,41

The obtained solutions of enrichment have low concentration of nutrient components. Therefore, their application directly as liquid fertilizers is inappropriate.
Nitrogen deficiency can be compensated by introducing substandard grades of ammonium nitrate or by introducing additional amounts of nitric acid into the enrichment solutions before ammoniation. To obtain liquid fertilizers, nitric acid with concentration of 56% after triplicate use was added to the solution of the enrichment and ammoniated the resulting solution with ammonia gas to pH of 7-7.5. The results are shown in Table 4.

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Table 4

Chemical and salt composition of liquid nitrogen-calcium fertilizers

№	HNO_3, g/100 g	Chemical and salt contents, mass. %					
		CaO	$N_{total.}$	$N_{nit.}$	N_{NH4}	$Ca(NO_3)_2$	NH_4NO_3
1	-	13,26	6,48	6,48	-	37,95	-
2	10	12,22	8,75	7,43	1,32	35,80	7,55
3	20	11,56	10,78	8,28	2,50	33,88	14,28
4	30	10,58	12,61	9,05	3,56	32,16	20,34
5	40	10,45	14,27	9,75	4,52	30,61	25,81
6	50	9,96	15,76	10,37	5,39	29,19	30,77

As can be seen from the table, when 30 grams of nitric acid is added to 100 g of the enrichment solution, the total content of calcium and ammonium nitrates is 52.5%, and when 50 g of acid is added, 60% is achieved without ammonia, which can saturate the enrichment solutions.

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IV. CONCLUSION AND FUTURE WORK

Thus, the possibility of enriching the washed calcined phosconcentrate of the Central Kyzylkum with solutions of nitric acid and reducing the calcium modulus from 2.02 to 1.70 with nitric acid of 15-20% concentration at an acid norm of 25-28% without loss of phosphorus anhydride, and processing solutions for liquid nitrogen-calcium fertilizers and further on complex, liquid fertilizers by introducing into their contents potassium chloride, microelements, growth stimulators and plant development. In this case, the content of useful components in the form of compounds will exceed 60%.

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