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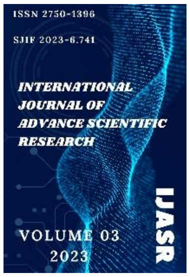

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Research Article

STUDY OF THE INFLUENCE OF NITRIC ACID ENRICHMENT OF PHOSPHORITES OF THE CENTRAL KYZYLKUM ON THE PROCESS OF OBTAINING PHOSPHORUS-CONTAINING FERTILIZERS BASED ON THEM

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ABSTRACT

An analysis of the literature shows that natural phosphates are suitable for de-composition with nitric acid, which does not contain significant amounts of cal-cium carbonate, magnesium carbonate and silicates, iron and aluminium com-pounds, which are easily decomposable acids, and as a result, complicate the processing of phosphates and degrade the quality of fertilizers. The best way to obtain phosphorus-containing fertilizers is the phosphoric acid decomposition of phosphate raw materials.

KEYWORDS

Phosphrite, acid, decomposition, phosphorus-containing fertilizer, chemical pro-cessing, roasting, combined flow chart, phosphorite enrichment.

INTRODUCTION

Uzbekistan is an agro-industrial country. It has more than 25 million 736 thousand hectares of agricultural land, including over 3.73 million hectares irrigated [1].

It is on irrigated lands that over 97% of all agricultural products of the republic are obtained. Uzbekistan ranks fifth in the world in cotton production. It has ensured its grain independence by collecting more than 6 million tons of cereal

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grain in recent years. The population of Uzbekistan today is over 36 million people. The annual growth is at the level of 2.5%. And irrigated arable land is not increasing due to the acute shortage of water resources. On a per capita basis, it is even falling.

Thus, in 1970, 0.22 hectares of irrigated land per person, and now this figure has decreased to 0.14 hectares [1].

Each ton of mineral fertilizers provides the annual need for food for 5-6 people. The cost of production and use of fertilizers is 2-3 times paid off by the cost of additional agricultural products. Thanks to the use of mineral fertilizers, an average of 40-50% of the increase in crop yields is provided. The effectiveness of the use of mineral fertilizers and chemical plant protection products is expressed not only in increasing yields and preserving products but also in a significant increase in labour productivity in agriculture. This can be seen, for example, from the following data. On the planet, in the period from 1900 to 1940, labour productivity in agriculture increased by 60%, and over the next 40 years by 1980, with the widespread use of chemicals, it increased 11 times [2].

A large branch of the chemical industry has been created in Uzbekistan, working in agriculture. Three open joint-stock companies: Maksam-Chirchik, NavoiAzot and FerganaAzot produce nitrogen fertilizers, the range of which consists of ammonium nitrate, urea and ammonium sulfate. Three JSCs: AmmophosMaxam, Samarkand chemical and Kokand superphosphate plants produce phosphorus-containing fertilizers, the range of which consists of amorphous, suprefos, ammonium sulfate phosphate, simple ammoniated superphosphate and nitrocalcium phosphate fertilizer. The Kyzylkum Phosphorite Plant provides phosphate raw materials to our factories that produce phosphorus-containing fertilizers. The Navoi Joint-Stock Association "Electrokhimzavod" produces various types of chemical plant protection products.

As a result of large-scale research and technological work, significant results have been achieved in the production of ammophos, suprephos-NS, ammonium sulfate phosphate, PS-Agro, simple and enriched superphosphates, nitrocalcium phosphate and phosphatized nitrate based on the processing of both enriched and unenriched phosphorites of the Central Kyzylkum.

It is known that one ton of raw cotton removes from the soil annually 45 kg of nitrogen, 15 kg of P_2O_5 and 45 kg of K_2O. One ton of wheat removes from the soil annually 35 kg of nitrogen, 10 kg of P_2O_5 and 24 kg of K_2O. With a gross harvest of 3 million tons of raw cotton and 6.1 million tons of wheat, only these two crops are annually carried away from the soil with a yield of 348.5 thousand tons of nitrogen, 106 thousand tons of phosphorus and 281.4 thousand tons of potassium. But other crops also take out a large amount of nutrients from the soil. They need to be replenished into the soil. We should not forget about the low utilization rate of nutrients from mineral fertilizers by plants. From nitrogen and potash fertilizers, on average, 60-70% of nitrogen

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and potassium are used, and from phosphorus fertilizers in the year of application - only about 20-25% and for 2-3 subsequent years about 40% of phosphorus. The weak use of phosphorus fertilizers in the year of application is caused by the fact that phosphorus compounds are in the soil in an immobile or extremely inactive state and therefore cannot be completely absorbed by the roots of plants. Therefore, phosphorus fertilizers must be applied in an amount that is 4-5 times higher than the phosphorus removal by the increase in yield (of a particular crop) that they want to receive [4].

All this suggests that in Uzbekistan it is necessary to increase the production of mineral fertilizers, especially phosphorus-containing ones. Questions arise about how this should be done and what brand of phosphorus-containing fertilizer is necessary for agriculture in the first place.

Naturally, to increase the production of phosphorus-containing fertilizers, it is necessary to increase the capacity of the Kyzylkum phosphorite plant. And at the same time, it is necessary to develop an acceptable, with good technical and economic indicators, technology for processing phosphorites of the Central Kyzylkum.

Objects and methods of research

The normal and stable operation of phosphate fertilizer plants depends primarily on the quality of phosphate raw materials. The world's best phosphate raw material is apatite concentrate from the Kola Peninsula in Russia. Its composition (wt%): 39.4 P_2O_5; 52.0 CaO; CaO: P_2O_5= 1.32; 3.0 (Fe_2O_3+Al_2O_3); 0.3 MgO; 0.9 (K_2O+Na_2O); 3.0F; no CO_2; 1.5 SiO_2; 2.0 insoluble residue.

Currently, only for it, there are technologies for processing almost any phosphorus-containing fertilizers with good technical and economic indicators. The transition to the processing of poor phosphorites significantly worsens the technical and economic indicators of the production of phosphorus-containing fertilizers. For example, the production of 1t P_2O_5 in extractive phosphoric acid from Karatau phosphorites with a content of 24.5% P_2O_5 costs almost 2 times more than from Khibiny apatite concentrate [5].

In the Republic of Uzbekistan, phosphorites of the Central Kyzylkum are used as phosphate raw materials. These phosphorites are phosphorus-poor phosphate raw materials containing, in addition, several undesirable impurities. The average sample of phosphorite from the Jerooy-Sardara deposit contains (wt.%): 16.2 P_2O_5; 46.2 CaO; CaO: P_2O_5 = 2.85; 17.7 CO_2; 0.6 MgO; 2.9 (Fe_2O_3+Al_2O_3); 1.5 (K_2O+Na_2O); 2.65 SO_3; 1.94 F; 7.8 insoluble residue. The low content of phosphorus, the high value of calcium module (2.85), the high content of carbonates (17.7% CO_2), presence of chlorine (0.1%) make phosphorite flour of the Central Kyzylkum practically unsuitable for acid processing in order to obtain concentrated phosphorus-containing fertilizers. A high calcium modulus causes a large overrun of the acidic reagent.

Intensive exploitation of deposits of rich raw materials naturally leads to their depletion. It is

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becoming more and more difficult and expensive to extract phosphate raw materials and process them. Therefore, all over the world, there is a search for ways to reduce the cost of processes for obtaining phosphorus-containing fertilizers and to involve poor phosphate raw materials in production. Such work is being carried out in Uzbekistan. Promising in this regard are the methods of chemical, mechanical, mechanochemical, thermal, and microbiological activation of phosphate raw materials, which convert the indigestible form of P_2O_5 in the raw material into a plant-assimilable form at the lowest cost [6–8].

RESULTS AND DISCUSSION

Phosphorites of the Central Kyzylkum have become the main phosphate raw material for factories in Uzbekistan that produce phosphorus-containing mineral fertilizers. Phosphorite manifestations are found in many regions of Uzbekistan. These are Fergana, Surkhandarya, Tashkent, Navoi (Pendtikent), Central Kyzylkum, Bukharo-Khiva and Karakalpak regions. But the most promising in terms of industrial development was the Central Kyzylkum [9].

The Kyzylkum phosphorite-bearing basin covers an area of 65 thousand km2. If we assume that the industrial phosphorite content covers only 5% of this area, then the forecast reserves of phosphorites with an average total thickness of their layers of 2.5 m will be 16.25 billion tons or 1.95 billion tons of P_2O_5 (with an average content of P_2O_5 of 12%). Marly granular phosphorite ores have been discovered and studied in the Kyzyl Kum by exploration work on an area of 3000 km2. The estimated resources of phosphorites to a depth of 300 m are 10 billion tons, approximately 2 billion tons of P_2O_5, including depths available for open mining (up to 60 m). 1-1.2 billion tons of ore, 200-240 million tons of P_2O_5 [9].

The Jerooy-Sardarinskoye deposit, whose predicted resources are estimated at 2.9-3.0 billion tons of ore (550 million tons of P_2O_5), is the most studied.

A characteristic feature of granular phosphorites is the stability of the mineral composition. At all deposits, phosphorites have a three-component composition. The main mineral composing phosphate grains is fluoro carbonate apatite (francolite) with unit cell parameters a0=9.33Ǻ, c0=6.89 Ǻ, containing 33% P_2O_5, 3.5-4.0% CO_2 and up to 3% SO_3, isomorphically included in its crystal structure. The second important mineral - calcite forms cement, and is also part of the granular material of phosphorite ores. Together with francolite, they make up from 75-80 to 93-95% of the mass of the ore. A distinctive feature of the Kyzylkum phosphorites is the presence of three carbonates in them: relics of calcite, preserved from replacement by phosphate inside phosphatized shells - "endocalcite"; cement calcite - "exocalcite"; carbonate group, isomorphically included in the crystal lattice of the phosphate mineral The relationship between calcite and francolite largely determines the technological properties of ores. The third, quantitatively, the mineral component of phosphorite ores - clay substance - is usually

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included in the composition of cement. Its content is 5-25%. The predominant mineral in the clay substance is hydromica (21-87% fraction), montmorillonite (0-86%) and kaolinite (2-15%) are associated with it. Partial separation of them from phosphorite ores can be carried out by washing or dry scrubbing before firing. In addition to these main minerals, gypsum and hydro goethite are always present as an admixture in oxidized phosphorite ores, and organic matter (up to 4%) and pyrite (up to 1%) are always present in unoxidized ores. Of minerals - impurities, the content of which does not exceed tenths and hundredths of a per cent, zeolites (clinoptilolite), palygorskite, silty quartz, and others are noted. The ores of the Jerooy-Sardara deposit have the following average mineral composition, %: francolite 56.0; calcite 26.5; quartz 7.5-8.0; hydromicaceous minerals and feldspars 4.0-4.5; gypsum 3.5; goethite 1.0; zeolite less than 1; organic matter is about 0.5 [10].

An average sample of phosphorite from the Jerooy-Sardara deposit contains (wt%): 16.2 P_2O_5; 46.2 CaO; CaO: P_2O_5=2.85; 17.7 CO_2; 0.6 MgO; 2.9 (Fe_2O_3+Al_2O_3), 1.5 (K_2O+Na_2O); 2.65 SO_3; 1.94 F; 0.1 Cl; 7.8 insoluble residues. This raw material is poor in phosphorus, complicated for processing by the presence of a large amount of undesirable impurities. Such raw materials are unsuitable for obtaining concentrated phosphorus-containing fertilizers from it. On the world market of phosphate raw materials, phosphorus concentrates with a content of at least 33% P_2O_5 are in demand

Poor raw materials must be enriched. First of all, you need to get rid of excessive amounts of carbonates. The most common enrichment method is flotation. But Kyzylkum phosphorites, along with a high degree of carbonization, are characterized by the fine intergrowth of phosphate minerals with calcite, so attempts to enrich them using flotation did not lead to positive results [9].

Chemical processing of natural phosphates is carried out in three main ways. The most common method is the decomposition of phosphates with acids - sulfuric, nitric, phosphoric and hydrochloric.

In [11, 12], an attempt was made to enrich the ores of Jerooy-Sardara by a chemical method using a nitric acid solution of calcium and magnesium nitrates containing 12% $Ca(NO_3)2$, 10% $Mg(NO_3)_2$ and 4.06% HNO_3. The degree of extraction of carbon dioxide was 63-65%, and the transition of P_2O_5 to the liquid phase was 0.14-0.78%.

Using 15% nitric acid, selective decomposition of the carbonate part of phosphorite (more than 80%) with the smallest losses of P_2O_5 with a solution (10-11%) can be achieved at an acid rate of 80% for the CO_2 content in phosphorite, a temperature of 30-35 °C and processing time 30-40 min.

When using nitric acid with a concentration of 9% in an amount of 30% of the stoichiometry and maintaining the treatment process for 50 minutes. It is possible to reduce the CO_2 content in the ore from 17% to 8.2%. The loss of P2O5 with the liquid phase does not exceed 1%. When using sulfuric acid (3-9%), it is not possible to achieve the required degree of CO_2 removal, since a significant amount of P_2O_5 passes into the liquid phase (up to 18.34% of the initial one) [13].

In [14], for chemical enrichment of phosphorites of the Central Kyzylkum, a nitric acid extract of phosphate raw materials was used. It is shown that under optimal conditions of the process, the content of P_2O_5 phosphorite increases from 18.5 to 31.3%. The maximum content of P_2O_5, the minimum content of CO_2 (2.8%) and CaO (40.7%) in the concentrate is achieved at an H_3PO_4 concentration of 6.0%, a nitric acid extract rate of 100% and a temperature of 15 °C; interaction time (60-90 min.) does not play a significant role. The yield of P_2O_5 in the filtrate (12.3%) reaches a minimum at an H_3PO_4 concentration of 6.0%, an extraction rate of 80%, and a temperature of 15 °C. The disadvantages of chemical enrichment methods are:

1) the formation of a large amount of dilute salt solutions that cannot be drained into the sewer, but must be disposed of somehow;

2) loss of P_2O_5 with these solutions, since 100% selectivity in the extraction of carbonates cannot be achieved.

With a high content of carbonates in phosphate raw materials, as is the case in Kyzylkum phosphorites (17.7% CO_2), the best method of enrichment of such raw materials is thermal. Therefore, roasting was included in the combined technological scheme for the enrichment of phosphorites, which has already been implemented at the Kyzylkum phosphorite plant. This scheme also provides for the washing of raw materials from chlorides bringing the chlorine content in washed, dried and washed, calcined phosphorus concentrates to an acceptable rate of 0.04% [15].

Promising in the issue of enrichment of low-grade phosphorites is phosphoric acid decomposition, to obtain both unilateral and complex fertilizers.

The decomposition of minerals contained in natural phosphates with phosphoric acid occurs according to the following main reactions:

$$Ca_5F\ (PO_4)_3 + 7H_3PO_4 + 5H_2O\ =\ 5Ca\ (H_2PO_4)_2\, H_2O\ +\ HF$$

$$MgCa(CO_3)_2 + 4H_3PO_4 =\ Ca\ (H_2PO_4)_2\, H_2O\ +\ Mg(H_2PO_4)_2\, H_2O\ +\ 2CO_2$$

$$R_2O_3 +\ 2H_3PO_4 + H_2O\ =\ 2[RPO_4.2H_2O]$$

Phosphoric acid decomposition of phosphate raw materials underlies the production of a single phosphorus fertilizer - double superphosphate, both by the in-line and by the chamber method. This type of fertilizer is one of the cheapest concentrated phosphate fertilizers, suitable for use on any soil and for all crops [16].

The paper [17] studied the physical and mechanical properties and chemical composition of off-balance phosphorus-containing raw

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materials - mineralized mass, in order to process it into one-sided phosphorus [18-23], as well as complex phosphorus-containing fertilizers [24-32].

The rheological parameters, as well as the water-insoluble part, were studied [33]. Mineralized mass was used as raw material (composition, wt. %: 14.33 P_2O_5; 43.66 CaO; 1.19 MgO; 1.38 Fe_2O_3; 1.18 Al_2O_3; 2.22 SO_3; 14.70 CO_2; 1. 75 F; 13.23 n.o.) - waste from the production of washed calcined phosphorus concentrate and extraction phosphoric acid (EPA) of Almalyk JSC "Ammophos-Maxam" composition (wt.%): 18.43 P_2O_5; 0.21 CaO; 0.30 MgO; 0.44 Fe_2O_3; 0.79 Al_2O_3; 1.71F; 1.47 SO_3. The number of initial components was taken based on the mass ratios of EPA: FS from 100:15 to 100:30.

Samples of ammophosphate that meet the requirements of agriculture were obtained, and their rheological properties were also studied [24]

In order to study the water-insoluble part of ammophosphate fertilizers, namely its chemical composition, because the difference between the total and water-soluble forms of P_2O_5 in the products was a significant achievement of the goal after the interaction of the extraction of phosphoric acid with the mineralized mass under the conditions [24], the pulp was quickly filtered under a vacuum of 550- 600 mmHg Art. on a Buchner funnel using one layer of filter paper. The precipitate remaining on the filter was washed with hot water until neutral according to the indicator paper. The washed precipitate was dried with filter paper in an oven at 105 °C. The dried precipitate was weighed and analyzed for the content of total, digestible, and water-soluble forms of P_2O_5. The results are shown in Table 1.

Table 1. Composition of the water-insoluble part of ammophosphate fertilizers

Mass ratio EPA: FS	Mass of dry sediment, gr.	P_2O_5 content, wt. %				$\frac{P_2O_{5усв}}{P_2O_{5общ}}$, in 2% lim. acid, %	$\frac{P_2O_{5усв}}{P_2O_{5общ}}$, by 0.2 M tril. B, %
		P_2O_5tot.	P2O5usv. in 2% lim. Kis-te	P_2O_5usv. by 0.2 M tril. B	P_2O_5aq.		
100:15	8.6	16.70	8.99	7.42	0.32	53.83	44.43
100:20	11.4	17.89	8.27	6.96	0.28	46.23	38.90
100:25	15.8	21.49	8.96	7.49	0.10	41.69	34.85
100:30	22.2	22.11	8.94	6.96	-	40.43	31.48

From the data in the table, one can observe the presence of trace amounts of water-soluble P2O5, but this indicates insufficient washing of the precipitate. The main result of the study is that in the water-insoluble part of the fertilizer, 40-54% of phosphorus is in the form digestible for plants

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in relation to its total content. This suggests that when phosphoric acid interacts with the mineralized mass, the phosphate mineral is activated. Activation is facilitated by the intensive release of CO2 from the phosphate mineral, which destroys its structure. Upon activation, unreacted phosphates differ significantly in structure from the original ones. Their grains are etched with acid, have a porous structure and are 15-30 times smaller than the original ones. Therefore, although they are water insoluble, their P2O5 is available to plants. That is why ammophosphates belong to the so-called

Conclusion

Suitable for nitric acid decomposition are natural phosphates that do not contain significant amounts of calcium carbonate, magnesium carbonate and silicates, iron and aluminium compounds. All these impurities hinder the processing of phosphates and degrade the quality of fertilizers. Particularly harmful are impurities of iron-containing minerals that are easily decomposed by acids, such as glauconite, and limonite, for example. Promising in this matter is the enrichment of low-grade phosphorites with phosphoric acid. Fertilizers obtained as a result of phosphoric acid decomposition have a high concentration of the nutrient component, as well as positive physical and mechanical properties.

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