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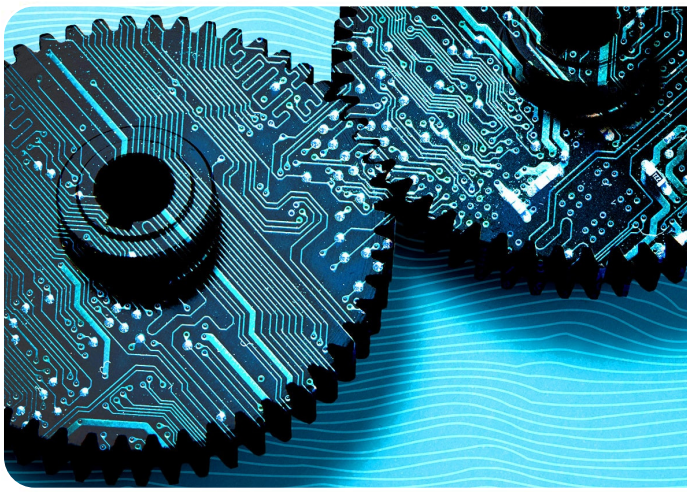

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Ma'lumot uchun, OAK Rayosatining 2024-yil 28-avgustdagi 360/5-son qarori bilan "Dissertatsiyalar asosiy ilmiy natijalarini chop etishga tavsiya etilgan milliy ilmiy nashrlar ro'yxati"ga texnika va iqtisodiyot fanlari bo'yicha "Muhandislik va iqtisodiyot" jurnali ro'yxatga kiritilgan.

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MUNDARIJA

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STUDIES ON OBTAINING AZOSUPERPHOSPHATE BY TREATING HIGH-CARBONATE, LOW-GRADE CENTRAL KYZYLKUM PHOSPHORITES WITH VARIOUS DOSAGES OF AMMONIUM SULFATE AND SULFURIC ACID

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Annotatsiya. Ushbu tadqiqotda yuqori karbonatli, past navli Central Kyzylkum fosforitlariga turli stexiometrik me’yorlarda ammoniy sulfatning sulfat kislotadagi eritmasi bilan ishlov berish orqali azosuperfosfat o‘g‘itini olish jarayoni o‘rganildi. Fosforit xomashyosi tarkibida 16,2% P_2O_5, 46,2% CaO va 17,7% CO_2 mavjudligi aniqlandi. Tajribalar laboratoriya sharoitida, xona haroratida, 93% H_2SO_4 ning 100 g miqdoriga 5–20 g ammoniy sulfat qo‘shilgan to‘rtta eritma tarkibida hamda 60–100% stexiometrik me’yorlarda olib borildi. Natijalarga ko‘ra, ammoniy sulfat miqdorini 20% gacha oshirish va kislotani 100% stexiometrik me’yorda qo‘llash natijasida o‘simlik tomonidan o‘zlashtiriladigan P_2O_5 miqdori dastlabki xomashyoga nisbatan 4,90–5,51 barobar ortdi. Granulometrik tahlillar asosida asosan 1–5 mm o‘lchamdagi, mexanik mustahkamligi kamida 1,2 MPa bo‘lgan granulalar hosil bo‘lishi tasdiqlandi. Tadqiqot natijalari mahalliy fosforit resurslari asosida yuqori samarali azosuperfosfat olishning ilmiy asoslarini belgilaydi.

Kalit so‘zlar: azosuperfosfat, past navli fosforit, Qizilqum, ammoniy sulfat, sulfat kislota, fosforit parchalanishi, mineral o‘g‘it, granulyatsiya, P_2O_5 o‘zlashtiriluvchanligi.

Аннотация. В данном исследовании изучен процесс получения удобрения азосуперфосфат путем обработки высококарбонатных низкосортных фосфоритов Central Kyzylkum раствором сульфата аммония в серной кислоте при различных стехиометрических соотношениях. Установлено, что исходное фосфоритовое сырье содержит 16,2% P_2O_5, 46,2% CaO и 17,7% CO_2. Эксперименты проводились в лабораторных условиях при комнатной температуре с использованием четырех составов раствора, содержащих 5–20 г сульфата аммония на 100 г 93% H_2SO_4, при стехиометрических нормах 60–100%. Результаты показали, что увеличение содержания сульфата аммония до 20% и применение кислоты в 100% стехиометрической норме повышает содержание усвояемого растениями P_2O_5 в 4,90–5,51 раза по сравнению с исходным сырьем. Гранулометрический анализ подтвердил образование гранул преимущественно размером 1–5 мм с механической прочностью не менее 1,2 МПа. Полученные результаты формируют научную основу для производства эффективного азосуперфосфата из отечественных фосфоритных ресурсов.

Ключевые слова: азосуперфосфат, низкосортный фосфорит, Кызылкум, сульфат аммония, серная кислота, разложение фосфорита, минеральное удобрение, грануляция, усвояемость P_2O_5.

Abstract. This study investigates the production of azosuperphosphate fertilizer by treating high-carbonate, low-grade Central Kyzylkum phosphorites with ammonium sulfate dissolved in sulfuric acid at various stoichiometric ratios. The phosphorite raw material contains 16.2% P_2O_5, 46.2% CaO, and 17.7% CO_2. Experiments were conducted under laboratory conditions at room temperature using four solution compositions containing 5–20 g of ammonium sulfate per 100 g of 93% H_2SO_4 at stoichiometric rates of 60–100%. Results showed that increasing ammonium sulfate content to 20% and applying acid at a 100% stoichiometric rate increased plant-assimilable P_2O_5 by 4.90–5.51 times compared with the initial raw material. Granulometric analysis confirmed the formation of granules predominantly in the 1–5 mm range with mechanical strength of at least 1.2 MPa. The findings provide a scientific basis for producing efficient azosuperphosphate fertilizer from domestic phosphorite resources.

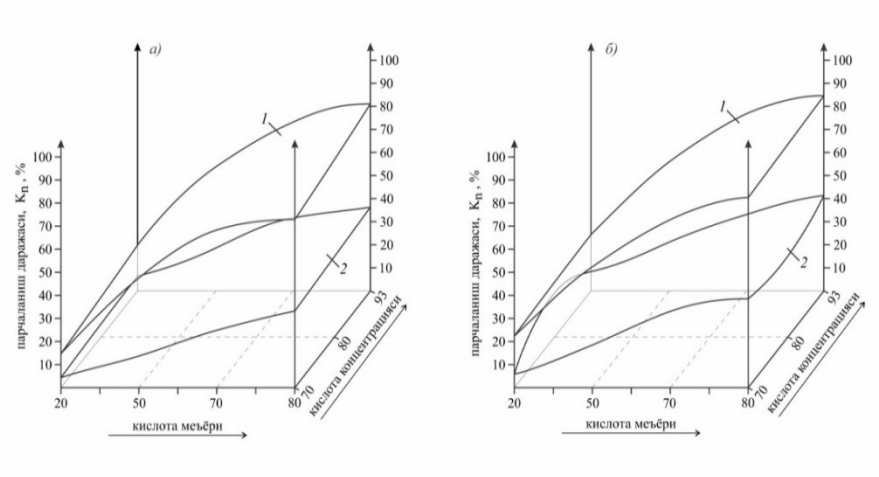
Keywords: azosuperphosphate, low-grade phosphorite, Kyzylkum, ammonium sulfate, sulfuric acid, phosphate decomposition, mineral fertilizer, granulation, P_2O_5 assimilability.

INTRODUCTION

In the Republic of Uzbekistan, reserves of raw materials required for the production of modern materials across many industrial sectors are considered sufficiently abundant. In particular, clay from the Kulatau deposit, dune sands of Tuprakkala [1], feldspar from the Sultan Uvays deposit [2–4], as well as sandstone, kaolin, and phosphorite deposits of the Khojakul raw material zone [5–9], together with talc ores of the Kazgansay deposit and Kyzylsay deposit, and the Zinelbulak deposit talc–magnesite raw materials, constitute the principal mineral resource base for the production of various industrial materials [10–14].

At the same time, the country also possesses substantial reserves of raw materials for the production of mineral fertilizers, which are essential for the growth and development of agricultural crops. These include phosphorites of the Kyzylkum basin, particularly the Jer-Sardora deposit, Karakat deposit, and Yetimtog deposit deposits, with an average chemical composition (wt.%): 16.2 P_2O_5; 46.2 CaO; CaO:P_2O_5 = 2.85; 17.7 CO_2; 0.6 MgO; 2.9 (Al_2O_3 + Fe_2O_3); 1.5 (Na_2O + K_2O); 2.65 SO_3; 0.12 Cl; 1.94 F; and 7.8% insoluble residue. In addition, the Guliob deposit located in Sariosiyo District, as well as phosphorite deposits in the Republic of Karakalpakstan, may also be regarded as important raw material sources [15–17].

In this regard, the authors of references [15–16] carried out studies on high-carbonate, low-grade phosphorites of Central Kyzylkum. Their research involved treating the raw material with sulfuric acid of 70–93% concentration under continuous stirring conditions. The process was performed using acid dosages in the range of 20–80%, depending on both



acid concentration and stoichiometric ratios (Figure 1).

Figure 1. Dependence of the degree of phosphorite decomposition on sulfuric acid concentration and stoichiometric ratio: (a) low-grade phosphorite; (b) unbeneficiated phosphorite.

1 – assimilable form of P_2O_5; 2 – water-soluble form.

According to the conclusions of the authors, when phosphorite samples were treated with 70% H_2SO_4, an increase in the stoichiometric ratio of acid from 20% to 80% resulted in a rise in the plant-available phosphorus content of the product from 1.63% to 6.30%, while the degree of decomposition increased by 4.95 times.

When H_2SO_4 concentrations of 80% and 93% were used, with the stoichiometric ratio varied within the range of 20–80%, the assimilable phosphorus content in the product increased from 1.85% to 6.42% and from 2.10% to 7.36%, respectively. At the same time, the degree of decomposition increased by 4.66 and 4.34 times, respectively.

Based on the data presented in this article, scientific studies on the decomposition of high-carbonate phosphorites of Central Kyzylkum using concentrated sulfuric acid, as well as ammonium sulfate in combination with sulfuric acid, are considered.

LITERATURE REVIEW

The efficient utilization of low-grade phosphorite resources has become an important scientific and industrial objective due to the growing demand for mineral fertilizers and the depletion of high-grade phosphate reserves. In Uzbekistan, substantial phosphorite deposits are concentrated in the Central Kyzylkum region, where many ores are characterized by high carbonate content and relatively low P_2O_5 concentration. These characteristics complicate direct processing by conventional acid decomposition methods and necessitate the development of improved activation technologies. Previous studies by Atashev E. and Tadjiev S. demonstrated the possibility of obtaining azosuperphosphate from low-grade Central Kyzylkum phosphorites through sulfuric acid treatment, while increasing acid concentration enhanced phosphorus

availability in the final product.

Further investigations reported that the introduction of ammonium sulfate into acidic systems improves phosphorite decomposition kinetics and promotes the formation of ammonium phosphate compounds with higher agronomic efficiency. X-ray diffraction and IR-spectral analyses of processed phosphorites confirmed structural changes in apatite minerals and the formation of new soluble phosphate phases. International studies on phosphate beneficiation also indicate that combined acid-salt activation reduces diffusion resistance, improves nutrient solubility, and enhances granulation properties of fertilizers.

However, the existing literature provides limited information regarding the optimal dosage of ammonium sulfate in concentrated sulfuric acid media for treating high-carbonate phosphorites. Therefore, further research aimed at determining rational reagent ratios, decomposition efficiency, and product granulometric properties remains scientifically and practically significant.

RESEARCH METHODOLOGY

The study employed both chemical and modern physicochemical analytical methods. The nitrogen content in the samples was determined in accordance with GOST 30181.4–94 by reducing nitrate nitrogen to ammonium nitrogen using Devarda's alloy in the presence of sodium hydroxide, followed by titration of excess acid with sodium hydroxide using appropriate indicators [19, 20].

According to GOST 20851.2–75, all forms of P_2O_5 in the samples (total, assimilable, and water-soluble) were determined by the differential photometric method. The analyses were carried out at a wavelength of $\lambda = 440$ nm using a KFK-3-01 "30M3" photocolorimeter [20, 21].

Calcium and magnesium oxides were determined in accordance with GOST 24596.4–81 using a complexometric titration method with Trilon B solution [22]. Iron and aluminum oxides in phosphorites were determined according to GOST 22275–90. During the analysis, volumetric flasks and graduated cylinders complying with GOST 1170 were used. Nitric acid (density 1.4 g/cm³, GOST 4461), sulfuric acid (density 1.84 g/cm³, GOST 4204), hydrochloric acid (GOST 3118), and acetic acid (GOST 61) were utilized. For iron determination, 30 cm³ of filtrate was transferred into a 300 cm³ flask, 2 cm³ of sulfosalicylic acid was added as an indicator, the solution was neutralized with ammonia, and then titrated with 0.05 N Trilon B solution [23].

The quantitative determination of sulfur in the samples was carried out according to GOST 24024.12–81, based on the turbidity formed by the interaction of sulfates with barium ions and comparison with a standard solution. The procedure employed laboratory glassware (GOST 25336–82), volumetric flasks and pipettes (GOST 1770–74), nitric acid (GOST 3118–77), barium chloride (GOST 4108–72), and distilled water (GOST 6709–72).

The fluorine content in the samples was determined according to GOST 24596.7–81 using a fluoride-selective electrode, either after separation by distillation or by direct measurement. In this study, fluorine concentration was measured directly in solution using a fluoride-selective electrode after decomposition with sulfuric acid, without prior removal of fluorine [24].

The CO_2 content in all samples was determined according to GOST 13455–91 using a volumetric method, in which carbon dioxide released during carbonate decomposition was absorbed and measured [25].

The pH of the medium was determined in accordance with GOST 24596.5–81. For this purpose, 10% suspensions of samples in distilled water were prepared, and pH values were measured using a METTLER TOLEDO FE20/EL20 pH meter [26].

Moisture content in mineral fertilizer samples was determined according to GOST 20851.4–75 by drying at 100–105 °C to constant weight in a Memmert drying oven [27]. The static strength of granules was determined according to GOST 21560.2–82 [28].

In addition, raw materials and obtained products were analyzed using X-ray diffraction (XRD) and infrared (IR) spectroscopy methods. XRD patterns were obtained using a Shimadzu XRD-6100 under CuKα radiation (Ni filter, $\lambda = 1.54178$ Å, 30 mA, 30 kV). The scanning parameters included a detector rotation speed of 4°/min, step size of 0.02° (ω/2θ), and a scanning range from 4° to 80°, with a sample rotation speed of 30 rpm. The obtained diffractograms were analyzed by comparison with the ASTM database ("The American Mineralogist Crystal Structure Database", ICSD) and reference tables compiled by Mikheev and Giller [29–32].

Infrared spectroscopic analyses were performed using the Shimadzu IRTracer-100 in the spectral range of 4000–400 cm^{-1}, with a signal-to-noise ratio of 60,000:1 and a scanning speed of 20 spectra per second. The obtained spectra were interpreted by comparison with standard mineral spectral databases [32–36].

ANALYSIS AND RESULTS

The studies were initially carried out using sulfuric acid with a concentration of 93%, while the stoichiometric amount

was varied within the range of 60–100%. The 100% stoichiometric amount of sulfuric acid was calculated based on the complete decomposition of the principal components present in low-grade phosphorite of Central Kyzylkum (P_2O_5 – 1.61; CO_2 – 2.23; Fe_2O_3 – 0.61; Al_2O_3 – 0.96).

It is well known that ammonium sulfate reacts with monocalcium phosphate formed in the system, resulting in the formation of ammonium dihydrogen phosphate ($NH_4H_2PO_4$). As a consequence of this reaction, a significant amount of calcium sulfate is formed in the composition of the samples.

$$(NH_4)_2SO_4 + Ca(H_2PO_4)_2 \cdot H_2O + H_2O - 2NH_4H_2PO_4 + CaSO_4 \cdot 2H_2O$$

Taking this into account, in order to determine the efficiency of phosphorite decomposition using mineral salts, ammonium sulfate in amounts ranging from 5 to 20 g, as well as its acidic and aqueous solutions, was used together with 100 g phosphorite samples. The experiments were carried out under laboratory conditions in a 5 L vertical glass reactor equipped with an OS20-S overhead stirrer.

To determine the optimal conditions for decomposition using ammonium sulfate dissolved in 93% sulfuric acid, the studies were conducted by varying the ratios of compositions prepared from ammonium sulfate and sulfuric acid.

Ammonium sulfate was dissolved in 93% sulfuric acid, and four different samples were prepared. The compositions of these samples are presented in Table 1.

Table 1

Composition of Samples Based on Sulfuric Acid and Ammonium Sulfate

No.	Sample	Amount of (NH□)□SO□ in 100 g of 93% H□SO□, g	Solution composition, %		
			H□SO□	(NH□)□SO□	H□O
1	Sample 1	5	88.35	5.0	6.65
2	Sample 2	10	83.70	10.0	6.30
3	Sample 3	15	79.05	15.0	5.95
4	Sample 4	20	74.40	20.0	5.60

The decomposition processes of phosphorites using the above-mentioned solutions were carried out under laboratory conditions at room temperature with continuous stirring of the phosphorite and acidic ammonium sulfate solution for 20 minutes.

When the dosage of the first composition was increased from 60% to 100%, the degree of decomposition with respect to the plant-available form of phosphorus pentoxide (P_2O_5 assimilable) increased from 45.90% to 79.10%, while for the water-soluble form (P_2O_5 water-soluble) it increased from 20.81% to 50.83%.

When the second composition was used, the degree of decomposition with respect to the plant-available form of P_2O_5 increased from 46.80% to 81.30%, while the water-soluble form increased from 0.75% to 1.25%.

For the third and fourth compositions, when the dosage was increased from 60% to 100%, the water-soluble P_2O_5 increased from 1.07% to 2.30%, while the plant-available P_2O_5 reached 83.52% and 86.13%, respectively, corresponding to an increase of 1.72 times.

It was also determined that the initial assimilable form of phosphorus pentoxide present in the raw material increased by 2.73 and 4.39 times after the decomposition process, depending on the solution dosage (60–100%).

All analyses were carried out in accordance with established regulatory standards and procedures. The results of these analyses are presented in Figure 2.

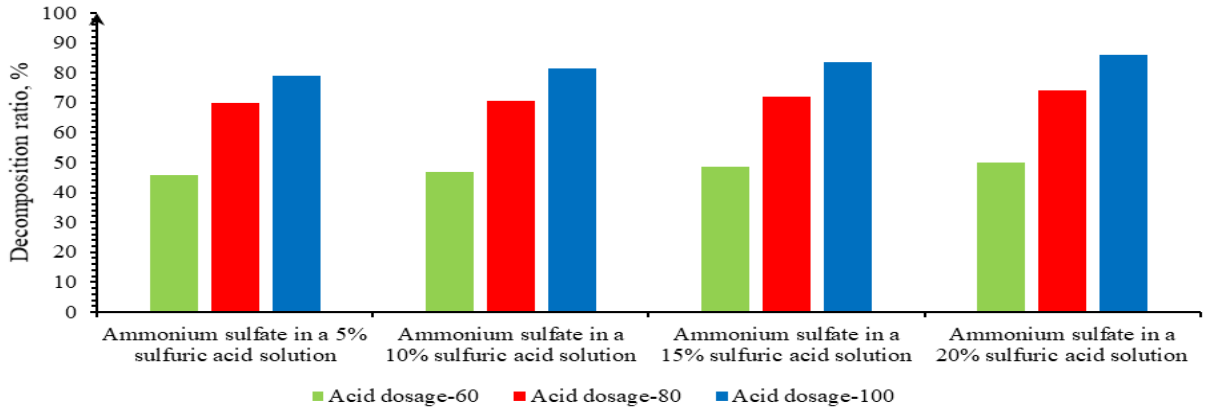


Figure 2. Degree of decomposition of low-grade phosphorite samples obtained by treatment in ammonium sulfate solutions dissolved in sulfuric acid.

During the study, the granulometric composition of azosuperphosphate fertilizer samples obtained using the third and fourth compositions, which demonstrated the highest efficiency, was investigated. The results of this analysis are presented in Table 2.

Table 2

Granulometric composition of azosuperphosphate obtained using ammonium sulfate solutions in sulfuric acid at different dosages.

№	Solution dosage, %	>5 mm, %	3–5 mm, %	1–3 mm, %	<1 mm, %
Ratio $(NH_4)_2SO_4$:H_2SO_4, 0,15:1					
1	60	-	17	45	38
2	80	-	13	42	35
3	100	2	28	43	20
Ratio $(NH_4)_2SO_4$:H_2SO_4, 0,2:1					
1	60	-	20	43	30
2	80	3	27	50	20
3	100	2	40	47	11

With an increase in the amount of ammonium sulfate in sulfuric acid, positive results were observed in granule formation. In particular, for samples obtained at a ratio of $(NH_4)_2SO_4$:H_2SO_4 = 0.2:1 and a solution dosage of 60%, the fraction of granules larger than 5 mm was 2%, granules of 3–5 mm accounted for 40%, those of 1–3 mm constituted 47%, and particles smaller than 1 mm made up 11%.

It was also observed that, as the stoichiometric dosage of these solutions increased from 60% to 100%, the size of the resulting granules increased accordingly. For example, at a ratio of $(NH_4)_2SO_4$:H_2SO_4 = 0.15:1 and a dosage range of 60–100%, the proportion of granules larger than 5 mm increased to 2%, the 3–5 mm fraction increased by 13%, and the 1–3 mm fraction increased by 5%, while the fraction of particles smaller than 1 mm decreased by 20%.

This trend was similarly maintained for the ratio of $(NH_4)_2SO_4$:H_2SO_4 = 0.2:1.

CONCLUSIONS AND RECOMMENDATIONS

Based on the above experimental results, the following conclusions can be drawn. To achieve complete decomposition of fluorocarbonate apatite and calcite minerals present in phosphorites, 63.88 g of sulfuric acid is required. In other words, for 13.94 g of P_2O_5 contained in 100 g of low-grade, high-carbonate phosphorite, 52.11 g of SO_3 ions are needed. Thus, if the ratio of sulfate ions to phosphorite (SO_3/P_2O_5) is lower than 3.68, the degree of decomposition decreases and the amount of plant-available P_2O_5 in the resulting product remains low. Conversely, when this ratio exceeds 3.68, not only the plant-available form of P_2O_5 but also its water-soluble form appears in the product.

The use of a 20% solution of ammonium sulfate in sulfuric acid leads to disruption of the crystal lattice of low-grade Central Kyzylkum phosphorite particles. As a result, diffusion resistance decreases and the reaction proceeds more readily. In other words, the ammonium sulfate medium activates the phosphorite, reducing the activation energy by 20–30%.

Introducing ammonium sulfate in an amount of 20% relative to the mass of low-grade Central Kyzylkum phosphorite, together with the use of highly concentrated acids (93–96%) and a full stoichiometric dosage (100%), increases the plant-available form of P_2O_5 (initially 1.52%) by 4.90 to 5.51 times.

The application of ammonium sulfate in the production of azosuperphosphate promotes the agglomeration of fine particles during granulation. It forms a thin layer on the surface of phosphate particles, thereby improving the mechanical strength of the product. During granule formation, fine particles compact and form larger aggregates, making it possible to obtain products containing 20–36% of 3–5 mm granules and 45–52% of 1–3 mm granules. The granules exhibit a mechanical strength of not less than 1.2 MPa and a bulk density of approximately 1.42 g/cm³.

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