ISSN 2181-8622

Manufacturing technology problems

Scientific and Technical Journal Namangan Institute of Engineering and Technology

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Volume 10
Issue 1
2025

Scientific and Technical Journal of NamIET | ISSN 2181-8622

STUDY OF THE DECOMPOSITION PROCESS OF LOCAL PHOSPHORITES USING INDUSTRIAL WASTE SULFURIC ACID

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Abstract: In this article, the chemical composition of products obtained by decomposing two differently composed samples of Central Kyzylkum phosphorite using industrial waste sulfuric acid was studied. During the research, the influence of reaction conditions, temperature, acid quantity, and reaction duration on the product composition was analyzed. Based on the obtained results, experiments confirmed the possibility of producing enriched superphosphate by processing simple phosphorite flour with waste acid. The study employed a Shimadzu UV 1900i spectrophotometer and titrimetric analysis methods.

Keywords: Phosphorite of Central Kyzylkum, waste sulfuric acid, decomposition, chemical composition, product, processing technology.

Introduction. The phosphorite mining and processing industry holds a significant position in the strategy of the Republic of Uzbekistan for the efficient use of mineral resources. Phosphorus compounds are widely used in the production of high-value products in agriculture and industrial sectors. However, the waste generated during the mining of phosphorite presents environmental challenges, and the effective processing of such waste is considered a pressing issue.

The "Development Strategy of New Uzbekistan for 2022–2026," approved by the President of the Republic of Uzbekistan, outlines objectives for the rational use of mineral resources, increasing the production of high-quality fertilizers for agriculture, and recycling industrial waste. Furthermore, the resolution adopted on July 20, 2023, titled "On measures aimed at improving the environmental situation in certain industrial sectors, reducing the negative impact of waste on the environment and public health, and efficiently using alternative energy resources," emphasizes the importance of rational use of mineral resources and the development of waste processing technologies.

The Central Kyzylkum phosphorites represent one of the main phosphorus resources in Uzbekistan and contain not only valuable phosphorus compounds but also complex mineral mixtures. Processing such phosphorites requires the implementation of highly efficient and environmentally safe technologies. In particular, using economically affordable reagents like waste sulfuric acid from industrial processes is of great importance in this field.

The phosphorite mining and processing industry is of strategic significance to Uzbekistan's agricultural and chemical industries. The phosphorus pentoxide (P_2O_5) content in phosphorite is a primary raw material for the production of phosphate fertilizers, which enhance soil fertility. The quality of phosphorite mainly depends on its P_2O_5 content: ores with a higher concentration are considered economically and ecologically efficient for processing. At the same time, the calcium oxide (CaO) content significantly affects the chemical properties of phosphorites, including their reactivity during processing.

This study also serves as a practical approach to encouraging the rational use of mineral resources in ensuring the economic stability of the Republic of Uzbekistan. The obtained results offer important technological solutions aimed at ensuring environmental safety in industry and expanding the raw material base.

In this research, two different types of Central Kyzylkum phosphorite samples were investigated for obtaining phosphorus-containing complex fertilizers through decomposition using waste sulfuric acid. The study examined key parameters such as decomposition conditions, temperature, and reagent quantities and their effect on the composition of the final product.

Chemical analysis was carried out on two types of phosphorite samples obtained from the Central Kyzylkum region. The first sample contained 20.38% P_2O_5 and 49.65% CaO, while the second sample had 22.4% P_2O_5 and 44.88% CaO. The phosphorus pentoxide content is the main indicator of a sample's suitability for agricultural and industrial applications. The close similarity in P_2O_5 values indicates that similar processing technologies can be applied. However, differences in CaO content affect the by-products formed during the reaction and their subsequent processing.

Central Kyzylkum phosphorites differ from phosphorites in other deposits due to their complex chemical composition and diverse mineralogy. Processing such ores requires the adoption of innovative and economically efficient technologies. In this study, the efficiency of decomposing phosphorites using waste sulfuric acid, the composition of the resulting products, and their dependency on reaction conditions were analyzed.

To enhance the decomposition efficiency, experiments were conducted using waste sulfuric acid. Initially, the acid used had a concentration of 52%, which was later increased to 67% through evaporation. During the phosphorite processing stage, both 52% and 67% concentrations of sulfuric acid were used. The experiments were carried out in the following sequence:

In the first experiment, 25.09 g of phosphorite containing 22.4% P_2O_5 and 10 g of thermoconcentrate were mixed with 13.07 g of waste sulfuric acid and 12.38 g of 73% phosphoric acid. To neutralize the pH during the reaction, 6 ml of ammonium hydroxide (NH_4OH) solution was added.

In the second experiment, 25.03 g of phosphorite containing 20.38% P_2O_5 was taken and mixed with 13.56 g of waste sulfuric acid and 12.66 g of phosphoric acid. To regulate the pH during the reaction, 6 ml of ammonium hydroxide solution was added. After thorough mixing, conditions were maintained to ensure complete reaction, and the product was prepared for analysis.

In the third experiment, 25.1 g of phosphorite containing 20.38% P_2O_5 was mixed with 15.11 g of waste sulfuric acid and 10.64 g of phosphoric acid. This reaction was conducted at 60°C. To bring the pH to the desired level, 10 ml of ammonium hydroxide was added. Conditions were maintained to ensure complete reaction, and the resulting product was prepared for analysis.

In the fourth experiment, 35.77 g of phosphorite with 20.38% P_2O_5 was used, and 10.94 g of waste sulfuric acid and 5.79 g of phosphoric acid were added. The mixture was stirred uniformly, and the reaction was carried out at 60°C. Then, 3 ml of ammonium hydroxide was added to adjust the pH. The composition and quality of the resulting product were analyzed.

In each experiment, the degree of phosphorite decomposition, the composition of reaction products, and pH values were monitored. Under conditions using 52% and 67% waste sulfuric acid, additional reagents (thermoconcentrate, phosphoric acid, and NH_4OH) played a significant role in enhancing reaction efficiency. The physical and chemical properties of the resulting products were analyzed, and the results are presented in Table 1.

Table 1. The main chemical composition of enriched superphosphate.

№	The chemical composition of fertilizer samples %								
	P_2O_5 umumm	P_2O_5 o'zl (lim kis)	**P_2O_5** suv.	P_2O_5 o'zl. (Tr. B)	**CaO**umum.	CaOo'zl.	CaOsuv.	Num.	SO_3
	Based on phosphorite containing 22.4% P_2O_5								
1	31,83	24,51	10,57	22,35	31,98	21,07	8,88	2,16	13,61
	Based on phosphorite containing 20.38% P_2O_5								
2	31,10	20,61	6,11	19,29	33,31	22,66	6,57	2,10	14,49
3	27,64	17,74	8,34	14,37	32,64	18,46	6,41	2,88	17,76
4	26,19	14,22	3,75	10,81	36,57	21,02	5,50	2,77	13,27

According to the experimental conditions, the concentration of sulfuric acid, the amount of added phosphoric acid, and the ammonium hydroxide solution influenced the regulation of pH levels. As a result of the experiments, with each process variation, the composition and quality of the obtained products were analyzed, and their differences were studied.

Among these, Sample 1 was selected as the most optimal variant, and its composition was thoroughly examined using physico-chemical analysis methods, yielding the following results.

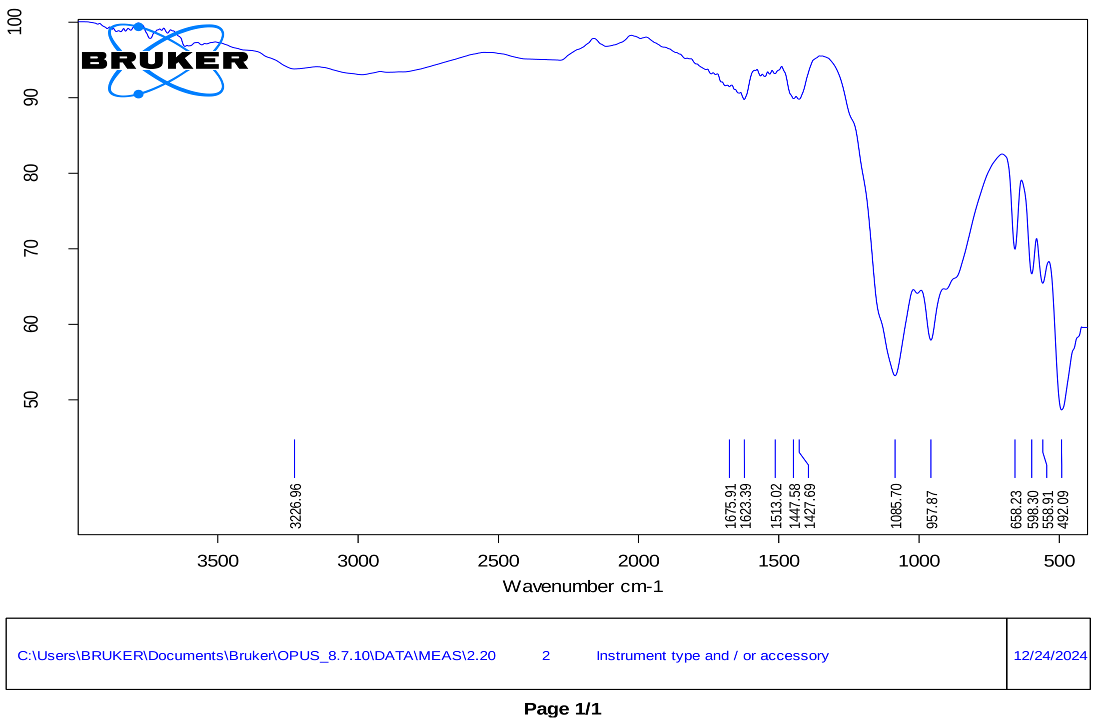

Figure 1. Results obtained in the IR spectrum of sample 1

The IR spectrum of the sample was recorded and analyzed to identify the functional groups and molecular structures present. The following absorption bands were observed, indicating the presence of various inorganic and organic components:

3236.99 cm⁻¹ – A broad absorption band associated with the stretching vibrations of hydroxyl (–OH) groups. This indicates the presence of water molecules (either adsorbed or structurally bound) or hydroxyl-containing compounds.

1631.91 cm⁻¹ – Bending (deformation) vibrations characteristic of H_2O molecules, further confirming the presence of water in the sample.

1535.19 cm⁻¹ – Stretching vibration associated with nitrate ions (NO_3^-), suggesting the inclusion of nitrogen-based fertilizer components or nitrate impurities.

1437.22 cm⁻¹ – A band corresponding to the stretching vibrations of carbonate groups (CO_3^{2-}), which indicates the presence of carbonate minerals such as calcite or dolomite.

1261.48 cm⁻¹ – A characteristic P=O stretching vibration, indicative of phosphate groups, potentially originating from phosphate compounds.

1087.70 cm⁻¹ – An intense absorption band due to the asymmetric stretching vibrations of phosphate ions (PO_4^{3-}), commonly found in phosphates and apatite minerals.

975.77 cm⁻¹ – Absorption associated with the POP bond, which may be attributed to the presence or formation of polyphosphates within the material.

877.67 cm⁻¹ – Another absorption band attributed to carbonate group vibrations, supporting the earlier identification of carbonate species.

828.82 cm⁻¹ – A vibrational band associated with aluminum-oxygen (Al–O) bonds, suggesting the presence of alumina phases or clay minerals in the sample.

669.28 cm⁻¹ – A band corresponding to vibrational modes of silicate or sulfate groups, potentially indicating the presence of silicate minerals or gypsum-like compounds.

The sample was further characterized using X-ray diffraction (XRD) with a MiniFlex 300/600 diffractometer operating at 40 kV and 15 mA. The analysis was conducted over a 2θ range of 3° to 120° under standard conditions. The diffraction pattern, presented in Figure 2, confirms the presence of crystalline phases consistent with the functional groups identified in the IR spectrum.

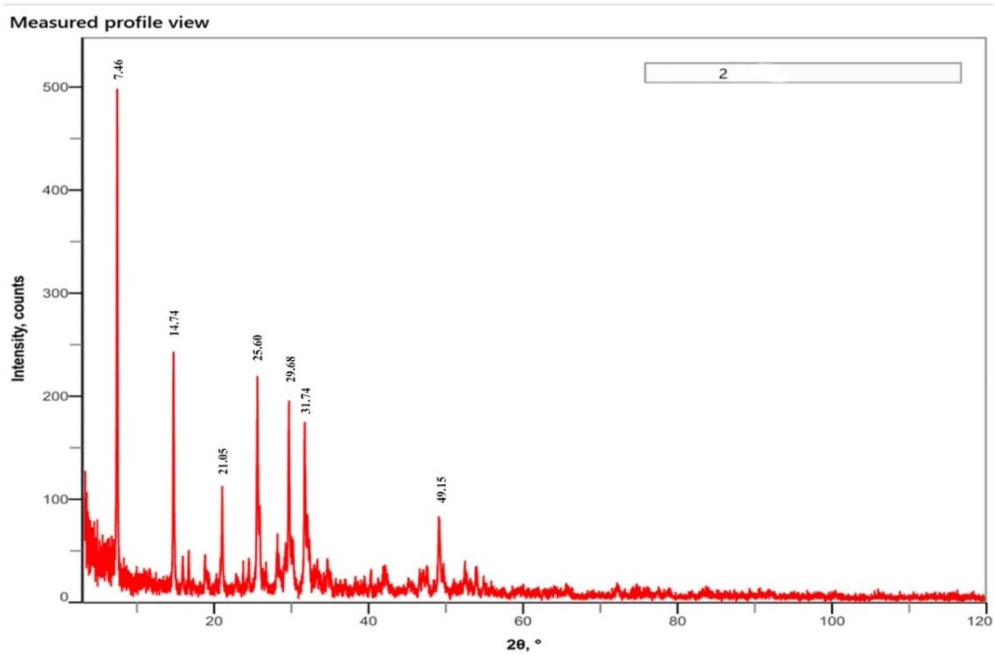

Figure 2. X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis was carried out to identify the crystalline phases present in the sample. The diffractogram revealed several prominent diffraction peaks, with the most intense reflections observed at **7.46°, 14.74°, 25.60°, 29.68°, and 31.74° (2θ)**. These peaks correspond to interplanar spacings (**d-values**) of **11.85 Å, 6.00 Å, 3.47 Å, 3.01 Å, and 2.82 Å**, respectively.

Based on the Scherrer equation, the average crystallite size was estimated to be in the range of **200–800 Å**. For instance, the crystallite size calculated for the peak at **7.46°** was **587 Å**, while that at **23.77°** was found to be **858 Å**. This range in crystallite sizes, along with the variation in full width at half-maximum (**FWHM**) values from **0.04° to 1.2°**, indicates a **polycrystalline structure** with diverse crystallite dimensions.

When compared with standard reference patterns from the **PDF-2** and **ICDD** databases, the observed diffraction peaks suggest the presence of the following mineral phases in the sample:

Montmorillonite: A strong peak at **7.46°** suggests the presence of montmorillonite, a common clay mineral in phosphorite waste that affects the physicochemical properties of soil.

Kaolinite: A peak near **14.82°** indicates kaolinite, which influences soil plasticity and sorption capacity.

Calcite: Detected at **20.88°**, calcite is a carbonate mineral known to buffer soil pH.

Quartz: A characteristic peak at **26.62°** confirms the presence of quartz, which affects the mechanical properties of soil.

Fluoroapatite: The peak at **29.42°** is attributed to fluoroapatite, a major phosphorus-bearing mineral crucial for plant nutrition.

Dolomite: Identified at **34.56°,** dolomite contributes calcium and magnesium to the soil.

Gypsum: A peak at **39.22°** reveals gypsum, which improves soil structure and aeration.

Hydroxyapatite: A peak at **45.10°** indicates hydroxyapatite, another phosphorus-bearing phase.

Anatase (TiO_2): Detected at **50.68°**, anatase is a polymorph of titanium dioxide, known for its pigment properties and potential agricultural significance.

Goethite (α-FeO(OH)): A reflection at **56.34°** suggests the presence of goethite, an iron oxyhydroxide that can influence soil color and iron availability.

To obtain a more detailed understanding of the sample's composition and microstructure, **Scanning Electron Microscopy (SEM)** analysis was also performed. The corresponding results are presented in **Figure 3.**

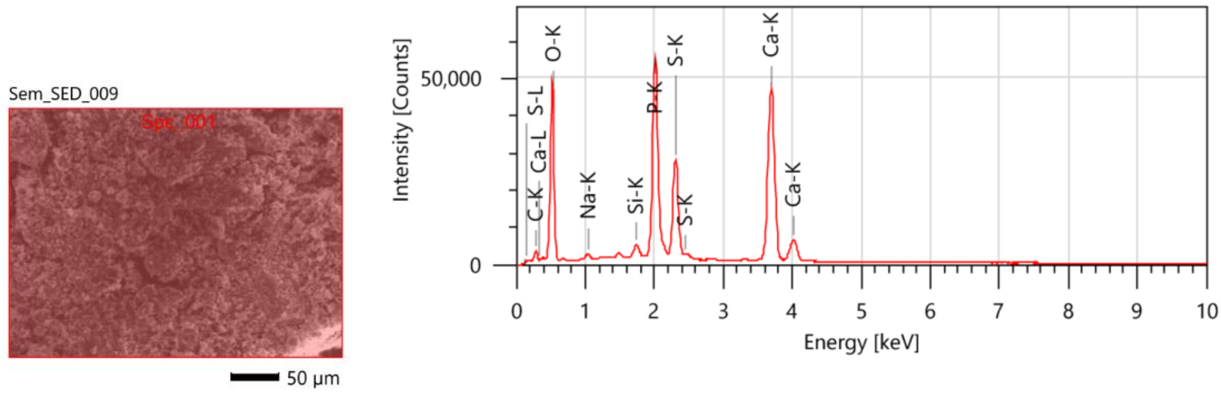

Figure 3. Scanning Electron Microscopy (SEM) Analysis

The SEM–EDS analysis of Central Kyzylkum phosphorite waste revealed that the material is rich in phosphorus (10.36%) and calcium (17.54%). These elements are essential for plant growth and development, where phosphorus plays a crucial role in strengthening root systems and participating in energy metabolism, while calcium helps regulate soil acidity and improves structural stability.

Additionally, the presence of sulfur (1.93%) and carbon (6.92%) indicates the potential to support soil microbial activity and accelerate the decomposition of organic matter. The absence of harmful heavy metals in the composition confirms that the material is environmentally safe.

Display name	Standard data	Quantification method	Result Type
Spc_001	Standardless	ZAF	Metal

Element	Line	Mass%	Atom%
C	K	6.52±0.05	10.82±0.08
O	K	52.89±0.16	65.85±0.20
Na	K	0.52±0.01	0.45±0.01
Si	K	0.66±0.01	0.47±0.01
P	K	13.34±0.04	8.58±0.02
S	K	7.16±0.03	4.45±0.02
Ca	K	18.90±0.05	9.39±0.03
Total		100.00	100.00
Spc_001			Fitting ratio 0.0141

These findings suggest that phosphorite waste can be effectively utilized in agriculture, either as a natural fertilizer or in processed form after acid treatment. This approach not only enhances crop productivity but also contributes to environmental protection by enabling the recycling of mining waste.

Further analysis was conducted on products obtained from processed samples, focusing on process efficiency and quality variation of the resulting materials. It was determined that process conditions such as sulfuric acid concentration, ammonium hydroxide addition, and temperature significantly influence product quality and yield. This research has contributed to identifying an efficient method for the decomposition of phosphorite using waste sulfuric acid.

Moreover, the addition of thermal concentrates and other chemical agents during processing played an important role in improving the final product. These methods can be optimized in the future and potentially applied to other types of phosphorite resources. Improving the process through technological optimization may lead to further enhancement in product quality and efficiency.

This study opens the way for developing a novel and promising approach to the production of phosphate fertilizers through the use of inexpensive and effective chemical agents such as waste sulfuric acid. The obtained results also expand the potential for industrial-scale processing of phosphorites and the broader application of the resulting products across different sectors. Further economic analysis will help maximize production efficiency while minimizing environmental impact.

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