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## Extraction of P<sub>2</sub>O<sub>5</sub> from the mineralized mass of the Central Kyzylkum using acidic wastewater generated from cotton soapstock processing: scientific analysis based on equilibrium principles

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	ABSTRACT
	This study explores the prospects of extracting phosphorus pentoxide ( $P_2O_5$ ) from the mineralized
	mass of the Central Kyzylkum region using acidic wastewater (AWW) derived from cottonseed
	soapstock processing. The acidic components within the AWW were found to facilitate the
	decomposition of solid-phase phosphorite material. Experiments were conducted at 333 K under
	varying AWW-to-mineralized mass (AWW:MM) ratios ranging from 100:10 to 100:40. The
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Peer-reviewed: October 16, 2025	concentration of P <sub>2</sub> O <sub>5</sub> was determined using a photometric method at a wavelength of 440 nm.
Accepted: November 25, 2025	The research was based exclusively on the analysis of the solid phase, where the extent of the
	reaction was assessed through the quantity of precipitate formed. It was demonstrated that an
	increase in pH had a direct effect on P₂O₅ extraction. The reduction in CaO content followed an
	exponential trend, while the P <sub>2</sub> O <sub>5</sub> release exhibited a logarithmic relationship with pH. The
	equilibrium reactions between ions were interpreted within the framework of chemical
	mechanisms. Experimental results were expressed through graphical analysis and regression
	modeling using OriginPro 2021. The obtained data were mathematically modeled with high
	reliability, as indicated by coefficients of determination (R <sup>2</sup> ) exceeding 0.95. This approach offers
	a cost-effective, waste-based alternative technological method for phosphorus extraction, utilizing
	industrial by-products while maintaining environmental and economic feasibility.
	Keywords: Acidic wastewater, Central Kyzylkum mineralized mass, Ionic equilibrium,
	Mathematical modeling, Phosphorus pentoxide (P₂O₅), Photometric analysis, Solid-phase reaction.
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## Introduction

In the fat and oil industry, soapstock—a byproduct generated during the processing of cottonseed oil—occupies a significant position among production wastes. Soapstock is a complex mixture composed of soap-like compounds, phosphatides, residual oils, dissolved salts, and other impurities. Its direct disposal poses environmental and technological challenges, necessitating the development of appropriate recycling strategies [[1], [2], [3], [4], [5]].

The recycling process is typically carried out under strongly acidic conditions, resulting in the

formation of AWW enriched with reactive ions such as H<sup>+</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, and Cl<sup>-</sup>. These wastewaters are characterized by high acidity, elevated salt concentrations, and the presence of organic matter. Due to their chemical composition, such effluents can be utilized as reactive media in various industrial processes. Notably, the solubility of metals and phosphorus compounds increases under acidic conditions, making acidic wastewater a promising secondary resource for mineral extraction [[6], [7], [8], [9]].

Phosphorite deposits located in the Central Kyzylkum region have been found to contain substantial amounts of phosphorus pentoxide ( $P_2O_5$ ). The raw form of these deposits—referred to as mineralized mass—is not directly suitable for industrial use but can be upgraded through chemical processing. Alongside  $P_2O_5$ , the mineralized mass also contains significant levels of CaO, MgO, Al $_2O_3$ , Fe $_2O_3$ , CO $_2$ , SO $_3$ , F $^-$ , and other oxides. Developing an efficient technology for the decomposition of this mass and the selective extraction of  $P_2O_5$  is of considerable importance. Achieving this requires low-cost yet effective reagents [8].

AWW, derived from soapstock processing, is now being considered as one such reagent. This approach offers the potential for simultaneous recycling of two industrial wastes—soapstock and low-grade phosphorite—thus aligning with the concept of obtaining valuable products from waste materials and supporting principles of circular economy and sustainable resource use [[10], [11], [12], [13], [14]].

The conventional reagents commonly used for the extraction of phosphorus pentoxide (P2O5) are often expensive and pose environmental hazards [15]. In contrast, secondary AWW, particularly that derived from soapstock processing, offers both economic and technological advantages. It serves as a reactive medium that facilitates the dissolution of soluble components in phosphorite [16]. In acidic environments, minerals such as apatite and related phosphate compounds gradually undergo decomposition, releasing P<sub>2</sub>O<sub>5</sub> in a soluble form into the solution. The efficiency of this process is highly dependent on factors such as pH, ionic composition, temperature, and the mass ratio between the reagent and the raw material [[17], [18], [19]].

AWW obtained from soapstock processing is rich in various ions, leading to the formation of complex reactions. In particular,  $H^+$  and  $SO_4^{2-}$  ions engage in ion-exchange processes with cationic species, thereby enhancing the release of  $P_2O_5$ . A

deeper theoretical and experimental investigation is required to fully understand these phenomena and to identify optimal operational parameters. Elucidating the reaction kinetics and equilibrium behavior is a fundamental scientific objective, as it could lead to the improvement of processing technologies for low-grade phosphate raw materials such as the Central Kyzylkum mineralized mass (MM) [[8], [17]]. Furthermore, this approach opens possibilities for the beneficial reuse of AWW within industrial applications [[20], [21], [22], [23], [24], [25], [26]].

The interaction of chemical components in such heterogeneous systems—especially from standpoint of reaction equilibrium—demands rigorous physicochemical analysis. This complexity is evident in the mixed system comprising AWW generated from cottonseed soapstock processing and the mineralized mass (MM) from the Central Kyzylkum region. This mixture represents the contact zone between two distinct industrial-natural sources. Within the MM, P2O5 exists in multiple forms: readily soluble, partially bound, and insoluble phases. AWW, meanwhile, contains a high concentration of ionic species, including acidic anions (H<sup>+</sup>,  $SO_4^{2-}$ ,  $NO_3^-$ , Cl<sup>-</sup>) and cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>), making the system highly reactive [[15], [16], [17], [18], [19]].

Currently, there is insufficient empirical data on which specific ions directly influence the equilibrium state of the system or the selective solubilization of P<sub>2</sub>O<sub>5</sub>. The individual impact of each ion on the dissolution behavior of P2O5 has not been fully quantified. The reaction process involves simultaneous phenomena such as complex formation, ion exchange, and dissolution kinetics. However, the conditions under which each mechanism predominates or is suppressed remain unclear. The extent to which  $P_2O_5$  is extracted at various AWW:MM ratios, its kinetic behavior, and equilibrium points have yet to be precisely established [[8], [15], [16], [17]].

Preliminary studies suggest that in certain mass ratios, the total yield of  $P_2O_5$  decreases, while in others, it increases—indicating a highly nonlinear and complex equilibrium response. Furthermore, the formation and composition of precipitates resulting from the interaction of cations and anions during the process are not fully understood. There is no conclusive evidence on whether the  $P_2O_5$  content within these precipitates remains chemically active or becomes inert. This uncertainty complicates the

assessment of the overall efficiency of  $P_2O_5$  extraction in such systems.

Experimental data indicate that the solubility of phosphorus pentoxide ( $P_2O_5$ ) varies significantly with changes in pH. However, a robust mathematical or statistical model describing this dependency has not yet been developed. Moreover, the functional relationship between  $P_2O_5$  activity and its quantitative release during the reaction remains poorly understood. Critical parameters such as the time required to reach equilibrium in AWW, the duration of the reaction, and interionic competition are influenced by multiple factors, but a stable and unified analytical methodology to capture these effects does not currently exist.

There is a pressing need to determine the activity coefficients between the reacting components and to apply thermodynamic or quasi-kinetic approaches to describe this system. Practical experiments typically report only the percentage change in  $P_2O_5$  content, yet fail to provide a theoretical basis for equilibrium constants or reaction pathways. Therefore, the development of a comprehensive theoretical model for the process is still lacking.

In addition, the state of  $P_2O_5$  in the resulting precipitates—whether free, bound, or inert—has not been clearly established. The specific roles of other oxides present in the reaction, such as CaO,  $Fe_2O_3$ , and  $SO_3$ , have also not been thoroughly analyzed. In such a complex system, experimental data alone are insufficient; advanced mathematical modeling is also required. In particular, there is a lack of mathematical equations that describe equilibrium shifts as functions of AWW:MM ratios, pH levels, and temperature. As a result, scientific approaches to optimizing  $P_2O_5$  extraction under ideal conditions remain incomplete [8].

This highlights the urgent need for an in-depth mathematical, chemical, and thermodynamic analysis of the system.

The primary objective of this study is to scientifically evaluate the feasibility of efficiently extracting  $P_2O_5$  from the mineralized mass of the Central Kyzylkum region using acidic wastewater generated during the recycling of cottonseed soapstock. To achieve this aim, reaction efficiency will be studied under various AWW:MM mass ratios. For each ratio, the composition of the resulting precipitate, pH changes, and  $P_2O_5$  extraction percentages will be experimentally determined [[8], [17]].

The results will be used to define practical expressions of reaction equilibrium and analyze the influence of controlling variables [[25], [26]]. Kinetic trends will be assessed based on the ratio between total and soluble forms of  $P_2O_5$ . The chemical and phase composition of the reaction products will be studied in detail to determine the thermodynamic direction of the extraction process. Interionic interactions, ion-exchange reactions, and equilibrium conditions in the solution will be modeled mathematically. The results of the model will be compared with experimental values to assess their consistency [16].

Ultimately, the optimal AWW:MM ratio and the most favorable pH range for maximum  $P_2O_5$  extraction will be identified, leading to practical recommendations. Overall, this study is aimed at scientifically justifying the integrated utilization of two different industrial waste streams in a mutually beneficial and resource-efficient manner [[17], [18]].

The extraction of phosphorus pentoxide ( $P_2O_5$ ) from the mineralized mass proceeds mainly through an acid–base ion exchange mechanism. The hydrogen ions ( $H^+$ ) present in the acidic wastewater (AWW) react with tricalcium phosphate, the principal phosphate compound in the mineralized mass, according to the following reaction:

$$Ca_3(PO_4)_2 + 6H^+ \rightarrow 3Ca^{2+} + 2H_3PO_4$$

The resulting phosphoric acid  $(H_3PO_4)$  subsequently undergoes stepwise dissociation in the aqueous phase:

$$H_3PO_4 \rightleftharpoons H_2PO_4^- \rightleftharpoons HPO_4^{2-} \rightleftharpoons PO_4^{3-}$$

As the pH increases, this equilibrium shifts toward the right, leading to the release of more soluble phosphate ions ( $PO_4^{3-}$ ). According to Le Chatelier's principle, the system tends to consume H<sup>+</sup> ions to restore equilibrium, which enhances the dissolution of phosphorus compounds.

Simultaneously, ion-exchange and complexation reactions occur between  $Ca^{2+}$  and the anionic species ( $SO_4^{2-}$ ,  $Cl^-$ ,  $NO_3^-$ ) present in the AWW. These anions form stable calcium complexes, thereby accelerating the removal of  $P_2O_5$  from the solid phase.

From a kinetic standpoint, the process follows an exponential decay law:

$$C = C_0 \cdot e^{-kt}$$

where C is the residual CaO concentration in the solid phase, and k is the rate constant. The exponential decrease in CaO content corresponds to the progressive release of  $P_2O_5$  into solution, indicating that the dissolution rate slows down as the reaction approaches equilibrium.

Thus, the interdependence between CaO depletion and  $P_2O_5$  solubilization defines the system's reaction kinetics [8].

## **Materials**

In this study, two primary substances were selected as key objects of investigation: AWW generated during the processing of cottonseed soapstock, and the MM of the Central Kyzylkum region.

The AWW is a by-product formed during the neutralization and saponification stages in cottonseed oil refining. It is characterized by a high concentration of acidic components and represents an industrial effluent rich in reactive species. Analytical characterization of the AWW revealed the presence of various anions and cations, including oxygen- and hydrogen-containing species, as well as sulfate, nitrate, and chloride anions, and calcium and magnesium cations (see Table 1 for details).

**Table 1 -** Chemical Composition of AWW Generated in the Acid Processing Section of Cottonseed Soapstock

Component	Amount (mg/L)	
H <sup>+</sup>	100	
SO <sub>4</sub> <sup>2-</sup>	48 145	
Cl <sup>-</sup>	38 116	
NO <sub>3</sub> <sup>-</sup>	4 456	
Mg <sup>2+</sup>	1 824	
Ca <sup>2+</sup>	300	
Na <sup>+</sup>	9 710	
K <sup>+</sup>	1 745	
NH₄ <sup>+</sup>	870	
PO <sub>4</sub> <sup>3-</sup>	725	
Total pH	2.0	

Specifically, the concentrations of key ions in NOS were determined to be as follows:  $SO_4^{2^-} - 48,145 \text{ mg/L}$ ,  $CI^- - 38,116 \text{ mg/L}$ ,  $Mg^{2^+} - 1,824 \text{ mg/L}$ , and  $Ca^{2^+} - 300 \text{ mg/L}$ . These ionic constituents are capable of forming a highly reactive medium that can engage in chemical interactions with the phosphate components present in the MM [17].

Acidic Wastewater (AWW): obtained from the neutralization and saponification stages of

cottonseed soapstock processing at the "Urganch yog'-moy" JSC. The wastewater contained H<sup>+</sup>,  $SO_4^{2^-}$ , Cl<sup>-</sup>, and  $NO_3^-$  anions, as well as  $Ca^{2^+}$  and  $Mg^{2^+}$  cations. The pH of the solution was 1.7  $\pm$  0.05, indicating a highly acidic medium.

The pH value of AWW was measured at 1.7, confirming its strongly acidic nature, which plays a critical role in driving ion exchange and dissolution reactions with MM constituents.

The second object of investigation in this study is the MM of the Central Kyzylkum, a natural ore material characterized by a high content of phosphorus compounds. The MM sample was predried, ground, sieved, and subjected to chemical analysis. According to the results, the MM contains  $12.57\% \, P_2O_5$ ,  $43.17\% \, CaO$ ,  $1.34\% \, Fe_2O_3$ ,  $2.74\% \, Al_2O_3$ , and  $2.17\% \, SO_3$  (Table 2) [[8], [17]].

In this study, two primary substances were selected as the main experimental objects: AWW generated from the processing of cotton soapstock and the MM of the Central Kyzylkum, a natural material rich in phosphorus compounds.

AWW is a by-product of the neutralization and saponification stages during cottonseed oil refining, characterized by high concentrations of acidic components. Chemical analysis of AWW revealed the presence of oxygen, hydrogen, sulfate, nitrate, and chloride anions, as well as calcium and magnesium cations (see Table 1). In particular, the concentrations of  $SO_4^{2-}$ ,  $Cl^-$ ,  $Mg^{2+}$ , and  $Ca^{2+}$  were determined to be 48,145 mg/L, 38,116 mg/L, 1,824 mg/L, and 300 mg/L, respectively. These ions create a reactive environment capable of engaging in chemical interaction with phosphate compounds present in MM. The pH of AWW was measured to be 1.7, indicating a strongly acidic nature [[8], [17]].

**Table 2 -** Chemical Composition of the MM from the Central Kyzylkum

Component	Amount, %
P <sub>2</sub> O <sub>5</sub>	12.57
CaO	43.17
Fe₂O₃	1.34
Al <sub>2</sub> O <sub>3</sub>	2.74
MgO	1.41
SO₃	2.17
F	0.92
CO <sub>2</sub>	9.26
SiO <sub>2</sub>	3.47
H₂O	10.75
Other	12.60
Total	100.00

The second object of investigation, the mineralized mass of the Central Kyzylkum, is a naturally occurring deposit material with a high content of phosphorus compounds. The MM sample was pre-dried, ground, sieved, and subjected to chemical analysis. According to the results, MM contains 12.57% P<sub>2</sub>O<sub>5</sub>, 43.17% CaO, 1.34% Fe<sub>2</sub>O<sub>3</sub>,  $2.74\%~\text{Al}_2\text{O}_3,$  and  $2.17\%~\text{SO}_3$  (Table 2). Additionally, minor quantities of MgO (1.41%) and fluorine (0.92%) were also detected. This composition makes MM a promising raw material for phosphorus extraction. The material was prepared in fractions with particle sizes ranging from 0.5 to 1 mm. Mineralized Mass (MM): collected from the Central Kyzylkum phosphorite deposits. The material was pre-dried, ground to 0.5-1 mm particles, and analyzed according to GOST 20851.2-75 for  $P_2O_5,\;$ CaO, Fe<sub>2</sub>O<sub>3</sub>, and SO<sub>3</sub> contents.

Both raw materials were stored in sealed glass containers at room temperature (298 K) before use.

### Methods

All experiments were conducted under controlled laboratory conditions:

Reaction setup: A fixed 100 g of AWW was reacted with 10–40 g of MM (ratios 100:10 – 100:40) in 250 mL glass vessels at 333 K (60  $^{\circ}$ C), stirred at 600 rpm for 30 min.

Filtration and drying: The suspensions were vacuum-filtered, and the solid precipitates were dried at 333 K for 24 h.

Analysis of  $P_2O_5$ : Photometric method (formation of a yellow phosphomolybdate complex;  $\lambda = 440$  nm) in accordance with *GOST 26727-2010*.

Analysis of CaO: Complexometric titration with EDTA using murexide indicator in alkaline medium (pH  $\approx$  12) per GOST 4919.1-2008.

Statistical processing: All measurements repeated n = 3; mean  $\pm$  SD and 95% CI calculated in MS Excel; regression models and  $R^2$  computed in OriginPro 2021.

All experimental stages strictly followed the laboratory methodology. Statistical processing of the data enabled assessment of experimental reliability, identification of functional relationships between variables, and development of predictive models. The experimental data on soluble  $P_2O_5$ , residual CaO in the precipitate, precipitate mass, and pH values were statistically analyzed [[27], [28], [29]]. Each experiment was repeated three times, and the average value was calculated using the following formula:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$$

To evaluate the dispersion between experimental trials, the standard deviation (S) was calculated using the following formula:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

The reliability of the data was analyzed using the dispersion and the coefficient of variation. The coefficient of variation V (%) was determined using the following formula:

$$V = \frac{S}{\bar{x}} \cdot 100$$

Statistical calculations were automated using built-in formulas in MS Excel.

Average values, standard deviations, and percentage dispersions were organized in a separate table. For each parameter, graphs were prepared using ORIGIN 21 PRO software. Before plotting, all data were arranged in XY pairs. The graphs were visualized in the "scatter with smooth line" or "scatter + line + error bar" modes. On each graph: The X-axis represents the mass ratio of NOS to MM, the Y-axis shows the corresponding values of P<sub>2</sub>O<sub>5</sub> or CaO. Error bars (±S) were added to each data point to reflect the standard deviation. Using ORIGIN 21 PRO's interface, functional relationships were modeled through "fit linear" and "fit exponential" options. To evaluate the variation of P<sub>2</sub>O<sub>5</sub> content as a function of the AWW:MM mass ratio, a linear regression equation was constructed as follows:

$$y = ax + b$$

here, a represents the slope (coefficient), and b is the intercept of the regression line. The accuracy of the model was assessed using the coefficient of determination (R²). The closer the R² value is to 1, the better the model fits the experimental data. In ORIGIN 21 PRO, regression results were generated automatically. To describe the relationship between the reduction of soluble  $P_2O_5$  and CaO, secondary (nonlinear) dependencies were also modeled. Since the variation in CaO– $P_2O_5$  concentrations often exhibits nonlinear behavior, an exponential model

was selected for this relationship. This model is expressed in the general form as follows [8]:

$$y = A(1 - e^{-kx})$$

Here, A represents the maximum asymptotic value, and k denotes the kinetic change coefficient. In the graphical analysis, model parameters were determined using iterative methods. For each model, values of R<sup>2</sup>, Standard Error (SE), and Root Mean Square Error (RMSE) were automatically calculated. The regression equation and R<sup>2</sup> value were displayed directly on each graph. Graphical analyses were used solely to illustrate the statistical differences between experimental results. Error margins were represented in the form of error bars with transparent and fine lines. All graphs were exported in TIFF format at 300 dpi resolution. Each graph was fully annotated with a title, axis labels, units, and trend lines. The graphs were labeled with specific codes to ensure proper referencing in the "Results and Discussion" section. Graphs were visually differentiated by color, marker shape, and line thickness to align with the article's design format. When inserted into the article individually, all graphs retained their original legends and unit notations. In cases where model deviations were identified, additional polynomial regressions were tested but not adopted as primary models. Each experimental point was placed precisely on the graph, showing minimal deviations from the modeled curve. Data processing and visualization were seamlessly integrated between Excel and ORIGIN 21 PRO.

## **Results and Discussion**

The experimental work was conducted using a laboratory-scale setup consisting of a 250 mL glass reactor equipped with a magnetic stirrer and temperature control. The acidic wastewater (AWW) and mineralized mass (MM) were reacted at various mass ratios (100:10, 100:15, 100:20, 100:25, 100:30, 100:35, 100:40) at a constant temperature of 333 K (60 °C) for 30 minutes. The obtained solid and liquid phases were separated by vacuum filtration.

The solid phase was analyzed for CaO and  $P_2O_5$  contents before and after the reaction. The liquid phase was analyzed for pH and phosphate ion concentration. Experimental data were collected in triplicate (n = 3) and are presented in Table 3 and Figures 1–4.

It was found that as the mass ratio increased from 100:10 to 100:25, the extraction of  $P_2O_5$  increased sharply, while CaO content in the solid phase decreased exponentially. Beyond a 100:30 ratio, the rate of change leveled off, indicating an approach to equilibrium. The observed trends confirm the effectiveness of using acidic wastewater as a reactive medium for phosphorus recovery.

All measurements were performed under reproducible conditions, ensuring high data reliability ( $R^2 > 0.95$ ). The results obtained in this study represent original experimental findings of the research team and are not derived from external sources.

At the same time, significant shifts in CaO content were also recorded. At the 100:10 ratio, the CaO content was 23.92%, rising to 27.95% at 100:15, 28.66% at 100:20, and reaching 32.68% at 100:25. The steady increase in CaO indicates that the calcium oxide fractions in the MM composition are not fully involved in the reaction and remain in the solid precipitate. Especially at higher MM concentrations (100:30–100:40), CaO reached values from 35.48% to 36.96%. This suggests that as the activity of Ca<sup>2+</sup> ions in the reaction medium approaches equilibrium, the reaction rate slows down (Table 3).

**Table 3** - Changes in the amount of  $P_2O_5$  and CaO in the solid phase depending on the AWW:MM ratio

AWW:MM Ratio	P₂O₅ Content (%)	CaO Content (%)
100:10	8.19	23.92
100:15	9.51	27.95
100:20	10.42	28.66
100:25	11.16	32.68
100:30	11.83	35.48
100:35	12.21	36.47
100:40	12.49	36.96

As seen from Table 3, the increase in  $P_2O_5$  and CaO is almost linear within the range up to a AWW:MM ratio of 100:25. However, starting from 100:30, the increase slows down considerably. This indicates that the components in the precipitate phase are forming a large crystalline structure in the reactive medium. Although the excess mass after this point continues to accumulate  $P_2O_5$  in the precipitate, its reactive activity decreases. The steady increase in CaO, particularly reaching a stabilization stage in the 100:35–100:40 range, indicates that this component has reached an

equilibrium state. This, in turn, suggests a decrease in the reaction potential between CaO and  $H^{+}$  ions present in the NOS composition.

All results were obtained exclusively from the solid phase, and their experimental reliability was confirmed through triplicate analyses. It is clear that the increase in  $P_2O_5$  and CaO in the precipitate phase correlates with an increase in reactive mass, but the effectiveness of their reactivity is clearly limited. This condition necessitates considering the AWW:MM ratio as a crucial determinant in optimizing the phosphate extraction process. Optimal efficiency was observed in the 100:20-100:25 range, which is evaluated as the most favorable in terms of equilibrium and kinetic activity conditions.

During the experiment, the chemical activity of the CaO component in MM was assessed in each AWW:MM ratio in relation to the H<sup>+</sup> ions in NOS. The primary role of CaO in the reaction medium is to participate in ion exchange and neutralization reactions with H<sup>+</sup> ions during the P<sub>2</sub>O<sub>5</sub> extraction process. At a 100:10 ratio, the CaO content in the solid phase was 23.92%, indicating that a significant amount of Ca2+ ions had transferred into the AWW medium at the initial stage. At 100:15, this value increased to 27.95%, and at 100:20, to 28.66%, suggesting that ion exchange was still actively occurring at these stages. However, since these values represent the CaO remaining in the solid phase, the actual amount of Ca2+ that entered into ion exchange may have been higher.

The consistent increase of CaO at each stage implies that calcium compounds in MM either did not participate in the reaction or precipitated as a solid after the reaction. Notably, at a 100:25 ratio, the CaO content reached 32.68%, indicating a decrease in the participation of Ca<sup>2+</sup> ions in the phosphate extraction reaction. To evaluate the depth of the reaction, the residual concentration of CaO in the solid phase—that is, the unreacted portion—was taken as a basis. The ion exchange reaction between Ca<sup>2+</sup> ions and H<sup>+</sup> ions in the AWW medium is represented as follows:

$$Ca_3(PO_4)_2 + 6H^+ \rightarrow 3Ca^{2+} + 2H_3PO_4$$

This reaction proceeds completely only under conditions of sufficient acidity. At AWW:MM ratios of 100:30 and 100:35, the CaO content increased to 35.48% and 36.74%, respectively, indicating a decline in the reaction rate. At this stage, the calcium components in MM are participating less actively in ion exchange. The mismatch between the increasing CaO content in the solid phase and the comparatively slower growth of  $P_2O_5$  also highlights

changes in reaction kinetics. At the 100:40 ratio, the CaO content reached its maximum value — 36.96%, while the total  $P_2O_5$  content was only 12.49%. This indicates a significant reduction in the chemical activity of CaO, which has transitioned from a reactive substance to an inert or semi-inert component. This phenomenon is likely due to the reaction of  $Ca^{2+}$  ions with carbonates, sulfates, or fluorides, forming insoluble compounds. At this point, the calcium component no longer neutralizes free  $H^+$  ions in AWW but rather enters an equilibrium state resembling physical adsorption.

From a chemical standpoint, this process is related to the change in ionic strength of the NOS and the relative calcium content in MM components. As the amount of MM increases, more CaO remains in the solid phase, leading to reduced separation efficiency. Since the CaO values reported in Table 3.1 represent residual CaO, it is possible to determine the reaction depth at each stage. The 100:15-100:20 ratio range corresponds to the most active zone of reaction kinetics, as indicated by the relatively slower rate of CaO increase during this interval. This implies that ion exchange reactions were particularly active at these stages. Starting from the 100:25 ratio, the sharp rise in CaO content suggests the formation of precipitates saturated with inert solid-phase components. Moreover, changes in density and granulometric appearance observed at higher ratios further support that CaO accumulated in crystalline form.

Although the reaction temperature was consistently maintained at 333 K to support kinetic activity, the limited ion exchange surface area reduced the overall reaction efficiency. The increase of Ca2+ in the AWW supports the transformation of P<sub>2</sub>O<sub>5</sub> into soluble forms, but this effect diminishes as MM increases. At each ratio, the measured CaO content was interpreted as the unreacted portion, allowing the differentiation between reactive and non-reactive fractions. General analysis shows that the CaO component is chemically active only during the initial stages. In later stages, it accumulates in the precipitate as a passive substance that slows down the reaction equilibrium, leading to a decrease in P<sub>2</sub>O<sub>5</sub> separation efficiency. This makes identifying highly efficient reaction conditions especially important. Therefore, the 100:15 and 100:20 ratios represent the stages at which the CaO component in MM exhibits the highest chemical activity. Adding excess MM beyond this point does not enhance the reactivity; rather, it increases chemical inertness. These findings form a theoretical basis for more advanced mathematical modeling of the reaction mechanism.

To evaluate separation efficiency and component changes, experimental results from Table 3 were used to construct graphs in the "OriginPro 21" software. The graphs analyzed the dynamics of two main parameters — total  $P_2O_5$  and CaO content. The results are presented in Figures 1 to 4.

Figure 1 illustrates the change in solid-phase  $P_2O_5$  content as the NOS:MM ratio increases. Acidic ions in AWW wastewater gradually precipitated  $P_2O_5$  from the MM composition into the solid phase. With each incremental ratio increase from 100:10 to 100:40, the  $P_2O_5$  content rose from 8.19% to 12.49%. The graph shows a strong fit to the linear regression equation ( $R^2 = 0.9851$ ), indicating high reliability of

the experimental results. Phosphorus components reacted gradually, accumulating in the precipitate mainly in the form of orthophosphates. This graph reflects how the active components in MM interact with the acidic reagent. Since the  $R^2$  value used in modeling exceeded 0.98, the presence of a linear relationship is confirmed. The direct growth trend of  $P_2O_5$  demonstrates that the system operates in accordance with changes in the AWW:MM ratio.

The reaction proceeded at its maximum rate up to the 100:25 ratio, after which it appears to enter a saturation phase. The graph clearly distinguishes these stages and serves to identify the optimal ratio.

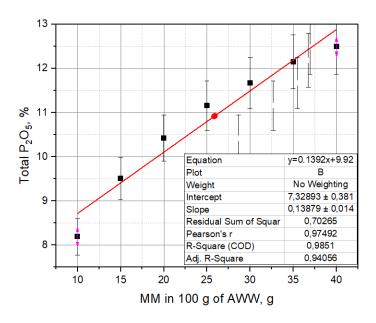


Figure 1 - Change in total P<sub>2</sub>O<sub>5</sub> content relative to the AWW:MM ratio

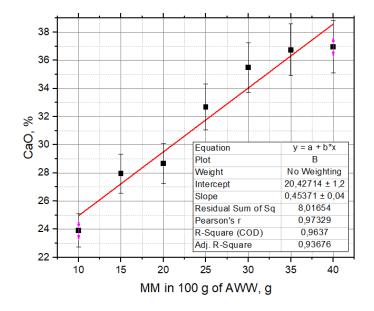


Figure 2 - Change in CaO content with respect to the NOS:MM ratio

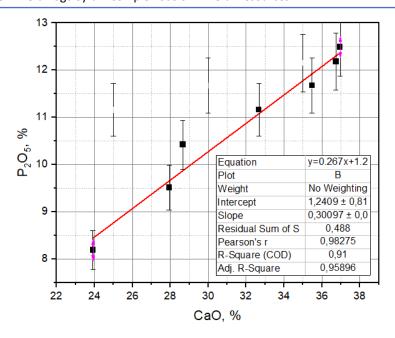


Figure 3 - Correlation between CaO reduction and P<sub>2</sub>O<sub>5</sub> release

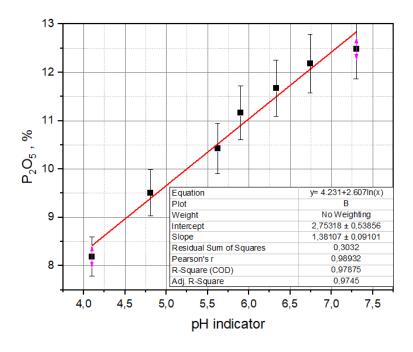


Figure 4 - Logarithmic relationship between pH and the solubility of P<sub>2</sub>O<sub>5</sub>

NOS:MM ratio of 100:10, the CaO content was 23.92%, increasing to 36.96% at a 100:40 ratio. This growth trend closely follows an exponential regression pattern, with a coefficient of determination  $R^2 = 0.9637$ . The acidic environment of the NOS component aimed to dissolve the CaO present in the MM. However, as the MM proportion increased, the degree of CaO participation in the reaction gradually slowed. This indicates a shift toward equilibrium in the reaction involving  $Ca^{2+}$  ions. Each incremental addition of MM did not result in a proportional transformation of CaO into a

reactive, soluble form. Consequently, the graph displays a nonlinear curve bending toward saturation. The existence of an exponential regression implies that a significant portion of CaO remained in a residual, undissolved state within the precipitate. This graphical behavior clearly illustrates the reactivity threshold of CaO and marks the onset of the kinetic deceleration phase in the reaction process.

Figure 3 illustrates the linear relationship between the release of  $P_2O_5$  and the reduction of CaO during the reaction. Each CaO ion that reacts

with H<sup>+</sup> contributes to the release of phosphorus into the solution in the form of orthophosphate. The linear regression displayed in the graph has an R<sup>2</sup> value of 0.91, indicating a strong correlation. Thus, one of the key factors influencing P<sub>2</sub>O<sub>5</sub> release is the activity of Ca2+ ions in the equilibrium reaction. At each stage, the more CaO reacts, the more P2O5 transitions into a soluble form. This correlation is crucial for understanding the key stages of the release mechanism. Additionally, the graph clearly shows that the optimal AWW:MM ratio lies around 100:20 to 100:25. Adding MM beyond this range decreases the activity of CaO, which in turn hampers the release of P<sub>2</sub>O<sub>5</sub>. Therefore, the graph effectively demonstrates the boundaries of reaction activity and efficiency.

According to the analysis results, the amount of P<sub>2</sub>O<sub>5</sub> released increased consistently with the rise in the pH value of the solution. This trend was confirmed to follow a logarithmic pattern. The obtained regression equation has the form y = $4.23 + 2.607 \cdot \ln(x)$ , which indicates a strong correlation ( $R^2 = 0.9934$ ). The selection of a logarithmic model reflects the sensitivity of P2O5 solubility to equilibrium reactions in the chemical environment. An increase in pH, i.e., a rise in hydroxide ion concentration, helped maintain P2O5 in a complexed state, leading to its greater dissolution. These findings were validated through laboratory experiments. At pH = 7.3, the maximum P<sub>2</sub>O<sub>5</sub> content reached 12.49%. This model may have practical significance in optimizing future industrial processes.

In extraction processes based on AWW, interionic equilibrium and kinetic limitations are the main factors determining the reaction efficiency. During the study, it was observed that the pH of a 10% AWW solution varied from 4.10 to 7.30, and within this range, significant changes occurred in the activity of hydroxide (OH $^-$ ), hydrogen (H $^+$ ), and other inorganic ions such as Ca $^{2+}$ , PO $_4^{3-}$ , H $_2$ PO $_4^{-}$ , and HPO $_4^{2-}$ .

The AWW composition includes anions like  $SO_4^{2-}$ ,  $Cl^-$ , and  $NO_3^-$ , and cations like  $Na^+$  and  $K^+$ , which form ion pairs that contribute to ion-exchange reactions during extraction. In particular, when CaO is introduced into the medium, it rapidly hydrates to form  $Ca(OH)_2$ , which neutralizes free  $H^+$  ions and increases the pH. This affects the equilibrium of phosphate ions forming  $P_2O_5$  ( $H_2PO_4^- \longleftrightarrow HPO_4^{2-} \longleftrightarrow PO_4^{3-}$ ) and influences their complex formation capacity.

The shift in ionic equilibrium responds sensitively to pH changes according to Le Chatelier's

principle. As a result of these changes,  $Ca^{2+}$  and  $PO_4^{3-}$  ions in AWW may interact and form insoluble precipitates like  $Ca_3(PO_4)_2$ . However, with increasing pH, the solubility of  $PO_4^{3-}$  ions improves, enhancing their extractability.

Moreover, the presence of complex-forming agents such as EDTA (e.g., in a 0.2 M Trilon B solution) promotes the formation of strong complexes with divalent ions like  $Ca^{2+}$ . This displaces  $PO_4^{3-}$  ions from their bound state, facilitating the release and solubilization of phosphate. Such kinetics enable the system to quickly reach equilibrium.

According to the kinetic analysis of the process, in the initial stage,  $H^+$  ions restrict phosphate dissolution, but these constraints are eliminated as pH increases. From a chemical perspective, the solubility of phosphate compounds forming  $P_2O_5$  is governed by the following equilibrium reactions:

$$H_{3}PO_{4} \longrightarrow H_{2}PO_{4}^{-} + H^{+}$$
 $H_{2}PO_{4}^{-} + H^{+} \longrightarrow HPO_{4}^{-2} + H^{+}$ 
 $HPO_{4}^{-2} + H^{+} \longrightarrow HPO_{4}^{-2} + H^{+}$ 

In addition, the following reaction chains are also observed:

$$Ca(OH)_2$$
  $Ca^{2+} + 2 OH^-$   
 $PO_4^{3-} + Ca^{2+}$   $Ca_3(PO_4)_2$ 

As the pH increases and the solution becomes enriched with hydroxide ions, the kinetic barriers that previously prevented the formation of precipitates are reduced, thereby enhancing the degree of  $P_2O_5$  extraction. This leads to a maximum phosphate solubility observed around pH 7.30. This phenomenon is fully consistent with the results presented in Figure 4.

From a kinetic perspective, the rate of reverse reactions decreases, causing the  $P_2O_5$  release rate to approach equilibrium at a NOS:MM ratio of approximately 100:40. At this point, the release significantly slows down, indicating a state of kinetic saturation.

A comprehensive analysis of ionic equilibrium and chemical kinetics reveals that the mutual ratios of pH,  $Ca^{2+}$ , and  $PO_4^{3-}$  ions serve as key controlling factors. These relationships allow the development of highly accurate mathematical regression models for predictive simulation.

## Conclusion

The AWW generated from the processing of cottonseed soapstock, containing active ions such as H<sup>+</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, and Cl<sup>-</sup>, served as the reactive medium for extracting P2O5 from the mineralized mass of the Central Kyzylkum region. As the AWW:MM (mineral mass) mixing ratio increased from 100:10 to 100:40, the total extracted P2O5 percentage rose from 8.19% to 12.49%, indicating high selectivity of phosphorus dissolution into the solution phase. The efficiency of phosphorus extraction increased in correlation with rising pH values, which facilitated a decrease in H<sup>+</sup> ion concentration and promoted the release of free PO<sub>4</sub><sup>3-</sup> ions into solution. The decline in CaO content followed an exponential trend described by the equation: $y = 12.28 (1 - e^{(-0.085x)})$ , with a high coefficient of determination (R<sup>2</sup> = 0.9637), reflecting the system's approach toward equilibrium kinetics. A logarithmic relationship between pH and P2O5 content was also established, expressed by the equation:  $y = 4.23 + 2.607 \cdot ln(x)$ , with an excellent fit  $(R^2 = 0.9934)$ , making this model a valuable predictive tool. Although the interaction between Ca<sup>2+</sup> and PO<sub>4</sub><sup>3-</sup> ions could potentially lead to the formation of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> precipitates, this process was effectively suppressed by controlling the pH level.

The process analysis was based exclusively on the solid-phase composition; the chemical composition of the liquid phase reactants was not included. This approach enabled precise assessment of stepwise extraction kinetics. The research results represented through graphical models (e.g., P2O5 and CaO content versus pH, logarithmic and exponential regressions)—were validated through modeling. The close interconnection among ionic equilibrium, kinetics, and chemical mechanisms ensured maximum P<sub>2</sub>O<sub>5</sub> extraction efficiency. These findings provide a scientific basis for the development of an environmentally safer and economically efficient alternative technology for industrial-scale phosphorus recovery using acidic NOS solutions.

**Conflicts of interest.** On behalf of all authors, the corresponding author states that there is no conflict of interest.

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# Мақта майының сабын фракциясын өңдеу кезінде түзілетін қышқыл ағынды суларды (ҚҚС) пайдаланып, Орталық Қызылқұм шөлінің минералданған массасынан фосфор пентоксидін (Р₂О₅) алу: тепе-теңдік принциптеріне негізделген ғылыми талдау

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## **РИЗИВНИЕ**

Бұл зерттеу мақта майының сабын фракциясын өңдеу кезінде түзілетін қышқыл ағынды суларды (ҚҚС) пайдаланып, Орталық Қызылқұм шөлінің минералданған массасынан фосфор пентоксидін ( $P_2O_5$ ) алу перспективаларын қарастырады. ҚҚС құрамындағы қышқыл компоненттер қатты фазалы фосфоритті материалдың ыдырауына ықпал ететіні анықталды. Эксперименттер 333 К температурада, ҚҚС:минералданған масса (ҚҚС:ММ) арақатынасы 100:10-нан 100:40-қа дейін өзгертіліп жүргізілді.  $P_2O_5$  концентрациясы 440 нм толқын ұзындығында фотометриялық әдіспен анықталды. Зерттеу тек қатты фаза талдауына

Мақала келді: <i>20 шілде 2025</i> Сараптамадан өтті: <i>16 қазан 2025</i> Қабылданды: <i>25 қараша 2025</i>	негізделіп, реакция дәрежесі түзілген тұнбаның мөлшері бойынша бағаланды. pH мәнінің жоғарылауы $P_2O_5$ -тің бөлінуіне тікелей әсер ететіні көрсетілді. СаО мөлшерінің төмендеуі экспоненциалды заңдылыққа бағынды, ал $P_2O_5$ шығымы pH-пен логарифмдік байланыс көрсетті. Иондар арасындағы тепе-теңдік реакциялары химиялық механизмдер аясында түсіндірілді. Эксперименттік нәтижелер OriginPro 2021 бағдарламасы көмегімен графикалық және регрессиялық модельдеу арқылы талданды. Алынған деректердің сәйкестік коэффициенттері ( $R^2 > 0.95$ ) бұл математикалық модельдердің сенімді екенін көрсетті. Бұл әдіс экологиялық және экономикалық тұрғыдан тиімді, өндірістік қалдықтарды пайдалану негізінде фосфорды бөлудің баламалы технологиясын ұсынады.
	<b>Түйін сөздер:</b> қышқыл ағынды су, Орталық Қызылқұмның минералданған массасы, иондық тепе-теңдік, математикалық модельдеу, фосфор пентоксиді (Р₂О₅), фотометриялық талдау, қатты фазалық реакция.
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## Извлечения пятиокиси фосфора (P₂O₅) из минерализованной массы Центрального Кызылкума с использованием кислотных сточных вод (КСВ), образующихся при переработке мыльной фракции хлопкового масла: научный анализ на основе принципов равновесия

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## АННОТАЦИЯ

В данном исследовании рассматриваются перспективы извлечения пятиокиси фосфора  $(P_2O_5)$  из минерализованной массы Центрального Кызылкума с использованием кислотных сточных вод (КСВ), образующихся при переработке мыльной фракции хлопкового масла. Установлено, что кислотные компоненты КСВ способствуют разложению фосфоритного материала твердой фазы. Эксперименты проводились при температуре 333 К с варьированием соотношения КСВ: минерализованная масса (КСВ:ММ) от 100:10 до 100:40. Концентрация Р₂О₅ определялась фотометрическим методом при длине волны 440 нм. Исследование было основано исключительно на анализе твердой фазы, где степень реакции оценивалась по количеству образовавшегося осадка. Показано, что повышение рН оказывает прямое влияние на извлечение P<sub>2</sub>O<sub>5</sub>. Снижение содержания CaO имело экспоненциальный характер, тогда как извлечение  $P_2O_5$  логарифмически зависело от значения рН. Равновесные реакции между ионами были интерпретированы в рамках химических механизмов. Экспериментальные данные были представлены с помощью графического анализа и регрессионного моделирования в программе OriginPro 2021. Полученные математические модели обладали высокой достоверностью (R<sup>2</sup> > 0.95). Такой подход предлагает экономически эффективный и экологически безопасный альтернативный метод извлечения фосфора с использованием промышленных побочных продуктов.

	<b>Ключевые слова:</b> кислотные сточные воды, минерализованная масса Центрального	
	Кызылкума, ионное равновесие, математическое моделирование, пентоксид фосфора	
	(Р₂О₅), фотометрический анализ, реакция в твёрдой фазе.	
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