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## Beneficiation of technogenic phosphorus-containing raw materials as a source of mineral fertilisers

**Abstract.** The relevance of this research arises from ongoing challenges related to rising raw material costs, geopolitical instability, and the complexity of processing phosphate rock ores with high impurity content. These factors underscore the need to develop efficient approaches for the beneficiation and processing of low-grade raw materials and waste as alternative sources of phosphorus. The aim of the study was to develop a technology for the beneficiation of technogenic phosphorus-containing materials to obtain phosphate rock concentrate suitable for fertiliser production. The research involved mineralogical, chemical, granulometric, and sedimentation analyses of the raw materials. Laboratory experiments included grinding, flotation, and magnetic separation. In addition, the properties of reagents for flotation-based beneficiation of phosphorus-containing materials were investigated. The study examined the potential for beneficiating technogenic phosphorus-containing materials derived from phosphate ore processing. Two laboratory-scale flowsheets – magnetic-flotation and flotation-magnetic – were tested, both enabling the production of commercial phosphate rock concentrate with a  $P_2O_5$  content ranging from 18.7% to 21.5%, and phosphorus recovery of 69.5% to 93%. For the first time, the significance of surface tension energy at the three-phase boundary during flotation was established, justifying the use of an anionic collector, talactam, for the effective separation of francolite from quartz. Magnetic separation, both before and after flotation, was found to enhance francolite concentration while reducing the content of glauconite and iron-bearing minerals. It was also determined that preliminary classification of the feed material at a particle size threshold of 0.16 mm reduces sludge formation during disintegration. The resulting glauconite-rich products have potential applications in the production of potash fertilisers and green pigments, although further study is required. The practical significance of this work lies in the successful implementation of the developed beneficiation technology for technogenic phosphate raw materials, which enables the production of phosphate concentrate (23.18%) suitable for the manufacture of Grade III phosphate flour fertiliser, and a glauconite product (50.71%) applicable as an enterosorbent and a source of trace elements in compound animal feeds

**Keywords:** phosphorus; francolite; glauconite; magnetic separation; flotation

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## Introduction

Phosphorus constitutes a key element in agriculture, forming a critical component of mineral fertilisers, including superphosphate, ammonium phosphate (amphos), and diammonium phosphate (diamphos). The sustainability of modern agro-industrial productivity cannot be maintained in the absence of phosphorus. However, phosphorus is a finite, non-renewable resource, with global reserves projected to be exhausted within the next 50-100 years. The resulting phosphorus deficit poses a direct threat to global food security. Consequently, intensive efforts have been directed towards the development of phosphorus recovery technologies from waste, wastewater, sludge, and phosphogypsum. Ukraine possesses no significant industrial deposits of natural phosphorus, necessitating the annual importation of over 800,000 tonnes of phosphatic fertilisers, primarily sourced from Morocco, Poland, China, and Lithuania. War, sanctions, and logistical constraints have had a marked impact on the accessibility of such imports. Attaining phosphorus self-sufficiency represents a strategic objective for Ukraine, aimed at reducing import dependency and enhancing agricultural autonomy. Ensuring a reliable supply of phosphate feedstock to Ukrainian producers of mineral fertilisers is a critical and pressing challenge. The successful resolution of this issue is vital to enabling the uptake of advanced phosphorus-processing technologies. Ukraine holds the potential to convert technogenic waste into a source of critical materials, thus reducing import reliance and entering the recycled phosphorus products market.

Within this context, research into efficient methods for processing phosphorite ores of varying degrees of enrichment is pertinent, with consideration of mineral composition, technological characteristics, and the potential for sustainable utilisation, has become increasingly relevant. Emphasis has therefore shifted towards the extraction of phosphates from tailings and low-grade ore deposits as secondary resources. Market instability, driven by geopolitical factors, rising raw material costs, and the inherent complexity of processing phosphorite ores with high impurity levels, necessitates the development of new strategies for resource utilisation, enrichment, and phosphate waste processing.

A. Proidak *et al.* (2021) identified that, due to political circumstances, Ukraine had encountered substantial challenges in supplying its fertiliser and ferrophosphorus plants with domestically sourced phosphorites and apatites. The authors conducted comprehensive studies on the mineral composition and metallurgical properties of phosphorite rocks, enabling an assessment of their suitability for the production of phosphorus-containing ferroalloys. S. Banerjee (2023) noted that the composition of phosphorite ores varies significantly depending on origin, with considerable diversity in structure, texture, and physicochemical properties. According to Y. Raiymbekov *et al.* (2023), approximately

20 million tonnes of low-grade phosphorites globally remain unprocessable under current technologies. The depletion of phosphorite deposits and the low content of phosphoric anhydride have contributed to declining global demand for phosphates, underscoring the need for advanced research on the enrichment and comprehensive utilisation of low-grade phosphates.

B. Wang *et al.* (2022) demonstrated that, due to their low phosphorus content and high proportions of accompanying materials (quartz, clay, calcite, dolomite, mica, feldspar, etc.), natural phosphate ores are unsuitable for direct application as fertilisers or for use as raw materials in phosphate fertiliser production. The efficient utilisation of phosphorites with medium or low phosphorus content is only feasible following preliminary treatment to achieve the requisite phosphorus concentration. Selection of an appropriate enrichment method depends on key parameters such as mineral texture, total phosphorus content, gangue composition, and, most importantly, the target phosphorus concentration as dictated by the intended application.

M. Sajid *et al.* (2022) reviewed phosphorite ore enrichment technologies aimed at achieving high-quality concentrates. Commonly employed enrichment techniques include flotation, electromagnetic separation, gravity separation, and magnetic separation. Global economic development has stimulated increasing demand for phosphorus-containing products. All existing high-grade phosphorite deposits have now been exhausted. This has necessitated the utilisation of low-grade phosphorites and off-spec phosphate raw materials, which remain underexplored and underutilised.

Y. Raiymbekov *et al.* (2020) provided an overview of low-grade phosphorite beneficiation practices in various countries, describing separation mechanisms during flotation enrichment and carbonate mineral decomposition using organic acids. The authors concluded that method selection depends on the mineralogical structure and chemical composition of the phosphorites. N. Abbes *et al.* (2020) enriched low-grade phosphate ore from Sra Ouertane (Tunisia) via thermal treatment, including calcination, quenching, and desliming. The applied treatment successfully increased the  $P_2O_5$  content from 20.01 wt.% to 24.24 wt.% post-calcination and to 27.24 wt.% following quenching. Furthermore, enrichment of associated rare-earth elements (Ce, La, Nd, Pr, Sm, Y) was achieved, while the Cd concentration was significantly reduced from 30 mg/kg to 14 mg/kg.

M. Derhy *et al.* (2020) reported that flotation technology had been extensively studied due to its wide application in phosphorite enrichment, with various depressants and collectors deployed to remove calcite and silica from sedimentary phosphate ores. A single-stage flotation of either phosphate or calcite was found insufficient for producing high-quality concentrates. K. Zhantasov *et al.* (2024) established that approximately

55-60% of the phosphate ore extracted and crushed comprises fine fractions below 10 mm, which are unsuitable for conventional phosphate fertiliser production. Experimental results confirmed the high efficiency of mechanical activation for fine phosphorite fractions. The proposed innovative process differs fundamentally from existing methods, as mechanochemical activation enabled direct, acid-free, and waste-free conversion into mineral phosphate fertilisers.

U. Ryszko *et al.* (2023) highlighted that enrichment processes generate substantial volumes of waste. The processing and reuse of such phosphorus production waste is currently expanding, with the goal of producing scarce products. The integration of magnetic separation and flotation methods in the enrichment of phosphorite ore waste enables the recovery of concentrates suitable for the production of mineral fertilisers. The objective of the study was to investigate the mineralogical, chemical, granulometric, and sedimentological composition and technological properties of the feedstock, thereby developing a magnetic-flotation enrichment process for technogenic phosphorus-containing materials, capable of yielding concentrates and products suitable for fertiliser production.

## Materials and Methods

A total of 18 small mineralogical samples of technogenic phosphorus-containing material were submitted to Kryvyi Rih National University for examination. A composite technological sample was prepared from these for use in process development studies. Chemical analysis was employed to determine the elemental and oxide composition of the material, including the content of primary components ( $P_2O_5$ , CaO,  $SiO_2$ ,  $Fe_2O_3$ ) and impurities affecting process properties and the suitability of concentrates for fertiliser production. Mineralogical analysis identified the constituent minerals (apatite, quartz, glauconite, etc.), which was critical for assessing the material's processing behaviour and selecting effective treatment methods.

Particle size distribution was determined through granulometric analysis, which established the optimal conditions for crushing, grinding, and enrichment, given the significant influence of size fraction on separation efficiency and recovery. Sedimentation analysis, based on the settling rate of fine particles in liquid, enabled precise estimation of particle sizes below 0.05 mm, which are known to affect material behaviour in aqueous and hydraulic enrichment processes. A technological analysis was conducted to evaluate the material's suitability for beneficiation, including laboratory-scale magnetic and flotation enrichment trials and assessment of concentrate quality indicators (e.g. yield,  $P_2O_5$  content), forming the basis for the proposed processing flowsheet.

The composite sample was washed and classified into size fractions: +1.25, -1.25+0.5, -0.5+0.25, -0.25+0.16, -0.16+0.074, -0.074+0.044, and -0.044 mm.

Representative subsamples of approximately 500 fragments were taken from each fraction using quartering, with 25% of fragments identified as free particles or composites for each mineral. Recalculation of composites into equivalent free particles was performed using a custom formula implemented in Microsoft Excel (Office XP). Grindability was evaluated using a standard method at grinding times of 5, 10, 20 and 40 minutes in a 7-litre laboratory ball mill under dry and wet conditions. The solid-to-liquid-to-ball ratio (S: L: B) was maintained at 1:0.33:10, and the mill charge was 1.6 kg (Biletsky *et al.*, 2020). Further sample preparation involved grinding to 0.16 mm (the size of the phosphorite opening) in a laboratory ball mill (model 75A-ML, Horstal Plant, Ukraine).

Magnetic susceptibility was studied in the laboratory of the Department of Mineral Processing and Chemistry, Kryvyi Rih National University, using the ponderomotive method based on the Gouy technique, as described by V. Zdeschchyts & A. Zdeschchyts (2023). The Gouy method involves measuring the gravitational force on a sample with one end in a strong magnetic field and the other in a weak field. Laboratory-scale magnetic enrichment was conducted using a rotary separator (model 259-CE, MAGNIS R&D Centre, Luhansk, Ukraine) equipped with a high-intensity magnetic field. Two materials were processed: raw material ground to 100% minus 0.16 mm (70% of class minus 0.074 mm) and the froth product from flotation.

Material with 70% of -0.074 mm and 30% solid content was fed into the separator's working zone, consisting of 200 mm high grooved plates. The matrix loading was 0.2 g/cm<sup>3</sup>. A scalping rotor, operating at one-third the magnetic induction of the axial rotors, removed magnetic particles from the plates, enhancing the recovery of iron-containing components in the magnetic product. Magnetic field induction ranged from 0.6 to 1.2 T.

Flotation tests were conducted in a mechanical-type flotation machine (model 237 FO, SCMA Lab, Kryvyi Rih, Ukraine) with cell volumes of 0.5, 1.0, and 1.5 litres, air flow rate of 1 L/m<sup>3</sup> of slurry, and impeller speed of 26.7-28.3 s<sup>-1</sup>. Test samples weighed 200, 400, or 600 g; closed-cycle tests used 150, 350, or 500 g. The flotation feed consisted of deslimed material (0.02 mm), ground to 100% of -0.16 mm, and the non-magnetic product from magnetic separation. Reagent selection and dosage followed standard phosphorite flotation practices and previous research (Oliinyk *et al.*, 2023). Anionic collectors were used: crude tall oil soap (CTOS) and talactam (RCONH(CH<sub>2</sub>)<sub>5</sub>COONa), a condensation product of tall oil with sodium salt of amino carboxylic acid. Sodium silicate acted as a depressant, and soda ash adjusted the pH (ranging from 8.7 to 10.5). Slurry solids content during agitation was 26%; mixing time was 3 minutes. Reagent consumption (kg/t): collectors – 0.5-1.7; depressants – 0.4-0.7; pH regulators – 0.5-0.8.

Optimal parameters from single flotation tests were refined in closed cycles using recirculated water, including primary, scavenger, and three to four cleaner flotation stages. Decantation was employed to obtain recycled water; tap water was used in process trials. Collector flotation activity was assessed via surface tension measurement using the ring detachment method, based on the force required to detach liquid adhering to a ring. Additionally, the impact of slurry temperature on flotation performance using talactam was studied, as this reagent is a condensation product of tall oil and amino carboxylic acid sodium salt.

Sedimentation analysis of slimes was conducted using a gravimetric method, based on differential settling velocities of particles of varying size and mineral composition. The methodology of sedimentation analysis was based on the differences in settling time of mineral particles of varying grain sizes. The essence of the method consisted in determining the variation in substance concentration at a specific depth of the suspension. During the experiments, the equal-settling coefficient for mineral particles was taken into account. The material was classified into grain size classes. For the calculation of settling time of mineral particles for each grain size class, terminal settling velocities were determined. The terminal settling velocities for the classes with a maximum grain size of 0.033 mm were calculated using Stokes' formula:

$$u = \frac{2\Delta\rho g a^2}{9\mu}, \quad (1)$$

where  $u$  – is the particle settling velocity, m/s;  $\Delta\rho = \rho_s - \rho_f$  – is the difference in densities of the dispersed phase and the medium, respectively, kg/m<sup>3</sup>;  $g$  – is the acceleration of gravity, m/s<sup>2</sup>;  $\mu$  – is the viscosity of the medium, PaS (0.0001 Pa×s);  $a$  – is the particle radius, m.

During the planning of experiments, the settling time for the selected vessel was determined as the

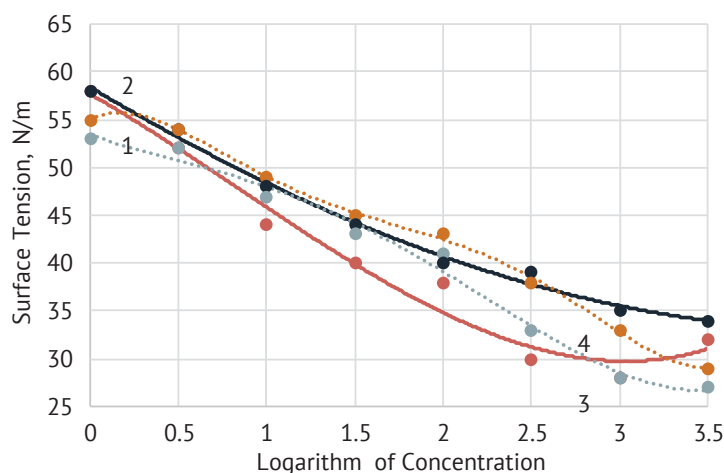
ratio of the height between the vessel markings to the calculated terminal settling velocities of the material. Distilled water was used during the experiments. In the course of sedimentation analysis using this method, samples were taken from a specific depth of the suspension at different time intervals from the start of the experiment, and the mass concentration of the substance in each sample was determined. With knowledge of the particle size and the settling height, the sampling time was calculated. The difference in mass concentrations between individual samples indicated the relative content of particles of the respective fractions in the analysed suspension. The calculations took into account the density of the solid phase in the relevant suspension: 3,800 kg/m<sup>3</sup> for intermediate products and an average of 2,600 kg/m<sup>3</sup> for waste. The settling time of a particle in the suspension ( $t_s$ ) was estimated as the time required for the particle to travel a distance equal to the vessel height ( $H$ ):

$$t_s = \frac{H}{u}. \quad (2)$$

Sedimentation analysis of the suspension by the gravimetric method was carried out using an apparatus consisting of a 1 dm<sup>3</sup> cylinder, a stirrer, and a Robinson pipette, which enabled sampling of the liquid at a constant level. A weighed portion of material of a specific grain size class (-0.045+0 mm) was placed into the vessel filled with water. The previously calculated settling time was then measured, after which the non-settled material was decanted. The experiment was repeated with the addition of clean water until the liquid above the lower mark became transparent.

## Results

The results of the study on the influence of surface tension magnitude as a function of reagent concentration were presented in Figure 1.



**Figure 1.** Surface tension as a function of reagent concentration

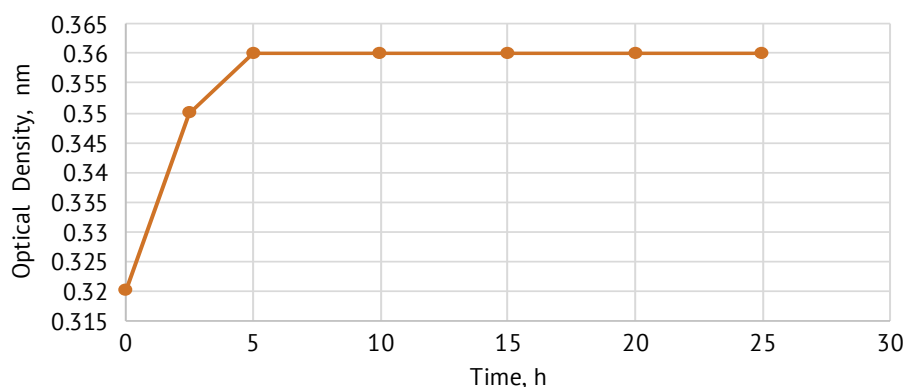
**Notes:** 1 – Talactam solution in tap water; 2 – CTOS solution in tap water; 3 – Talactam in technical water; 4 – CTOS in technical water

**Source:** authors' development

All four reagents demonstrated typical surfactant behaviour: surface tension decreased with increasing reagent concentration, confirming their capacity to accumulate at phase interfaces and reduce surface energy. In tap water, talactam produced a gradual reduction in surface tension, with the critical micelle concentration (CMC) observed around a logarithmic concentration of  $\approx 2.5$ , beyond which the curve plateaued. Surface tension reduction was moderate in this case.

In contrast, talactam in process water achieved a significantly deeper reduction in surface tension – from 60 mN/m to approximately 30 mN/m within the studied concentration range (logarithmic concentration 0

to 2.5) – followed by a gradual yet substantial decline. This behaviour was attributed to strong interactions with process water, likely influenced by the presence of calcium ions and trace impurities. Initial surface tension was slightly higher ( $\sim 58$ –60 mN/m). In both tap and process water, CTOS exhibited less efficiency than talactam, confirming talactam in process water as the most effective surfactant within this series, due to its consistent and substantial reduction of surface tension. Water type significantly influenced activity, with process water enhancing performance due to coagulation effects. The kinetics of optical density variation for talactam in tap water are shown in Figure 2.

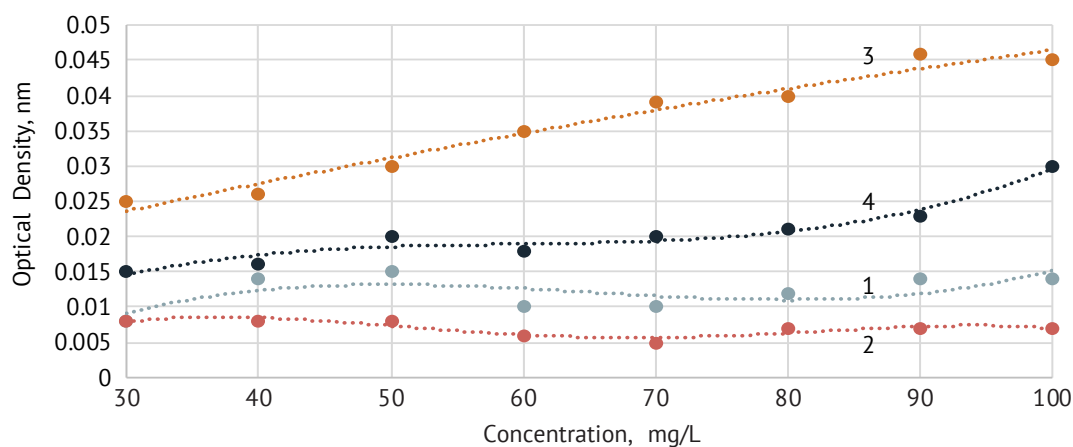


**Figure 2.** Kinetics of optical density change for talactam in tap water

Source: authors' development

A sharp increase during the initial 5 hours indicated active processes of dissolution, dispersion, or interaction between talactam and water components. Stabilisation occurred around 0.360 nm after 5 hours, indicating that the system had reached equilibrium. Stability up to 25 hours indicated the physicochemical resistance of talactam in tap water following the initial activation/

dissolution process. The observed increase in optical density was likely due to one or more of the following: dissolution or swelling of the polymer, reactions with ions or impurities, or self-aggregation of molecules. Changes in optical density – an important indicator of surfactant system stability and interactions with aqueous environments – depended on reagent concentrations (Fig. 3).



**Figure 3.** Change in optical density of talactam dependent on reagent concentrations

Notes: 1 – Talactam solution in tap water; 2 – CTOS solution in tap water; 3 – Talactam solution in recycled water; 4 – CTOS solution in recycled water

Source: authors' development



Observations on talactam behaviour in recycled versus tap water revealed that in recycled water, optical density increased most significantly with increasing concentration, reaching  $\sim 0.045$  nm at 100 mg/L. This suggested active particle aggregation and the presence of colloids or impurities interacting with talactam. In tap water, optical density remained stable, with a slight peak at 40-50 mg/L. Subsequent plateauing or decline likely indicated solubility limits and stabilisation, or reduced aggregation. In recycled water, CTOS also showed increased optical density, albeit to a lesser extent than talactam, suggesting partial aggregation inhibition or lower absorbance. In tap water, CTOS exhibited minimal

changes, with optical densities in the 0.005-0.01 nm range – indicating the most stable behaviour in this environment, likely due to absence of particle aggregation. Thus, talactam in recycled water demonstrated a strong tendency to aggregate, leading to increased optical density and improved collector performance, as confirmed in subsequent trials. The chemical composition of the composite technogenic phosphorus-containing sample is shown in Table 1. The average  $P_2O_5$  content was 10.55%, with  $SiO_2$  at 35.65%, total Fe at 7.5%, CaO at 18.82%, and  $Al_2O_3$  at 5.3%. These oxides served as key evaluation criteria for phosphorite and glauconite products.

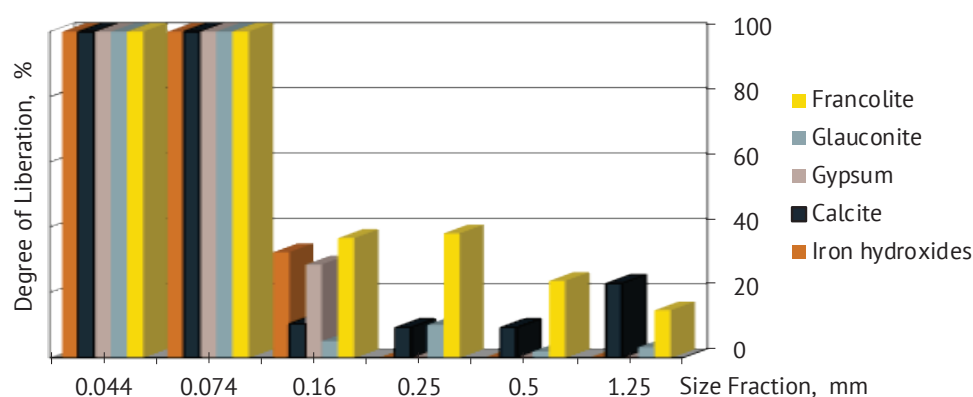
**Table 1.** Chemical composition of technogenic phosphorus-containing raw material (average values)

Material	Components, %																
	$P_2O_5$	MgO	CaO	$SiO_2$	$Al_2O_3$	$Fe_{total}$	FeO	$Fe_2O_3$	$TiO_2$	$Na_2O$	$K_2O$	MpO	F	$S_{total}$	$CO_2$	$SO_3$	LOI
Sample	10.55	2.02	18.82	35.65	5.3	7.5	1.0	10.1	0.19	0.33	2.75	0.45	1.85	1.34	2.42	0.9	0.355

Source: authors' development

Mineralogical analysis revealed that the primary phosphorus-bearing mineral, francolite, constituted 21.8% of the sample. The sample was characterised by a significant content of glauconite (38.6%) and

iron hydroxides (7%). It also contained heterogeneous clay material (9.1%) and gypsum (2.1%). The liberation characteristics of the minerals are illustrated in Figure 4.



**Figure 4.** Mineral liberation in the grain size classes of the initial sample

Source: authors' development

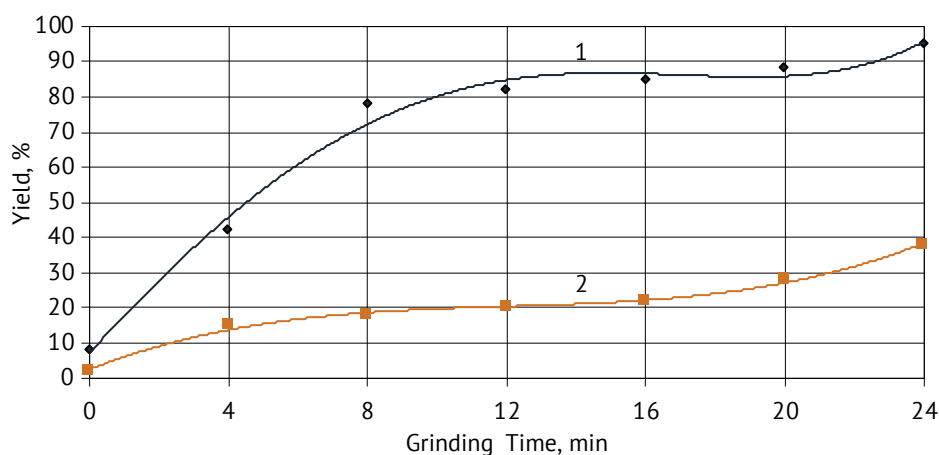
Mineralogical analysis of classified material showed that the +1.25 mm fraction comprised 15.7% free particles and 84.3% composites. Among the free particles were francolite, clay mineral, glauconite, calcite, and palaeoresidue. Rich composites (with >50% of a single mineral) outnumbered poor ones ( $\leq 50\%$ ) – 52.7% vs. 31.6%. Rich composites were primarily composed of glauconite, francolite, and clay mineral. Palaeoresidue occurred only as free fragments. The -1.25 + 0.5 mm fraction consisted of 82.2% composites (34.2% rich, 28.1% poor). Rich composites contained glauconite, francolite, and clay mineral. In contrast to previous fractions, iron hydroxides and hydromicas no longer exhibited a predominance of rich composites. Free particles (17.8%) included the same minerals

found in coarser fractions, with quartz appearing as an additional free component.

The -0.5 + 0.25 mm fraction contained 20.5% free particles and 79.5% composites. Free particles included the same components observed in coarser fractions. Rich composites significantly exceeded poor ones (67.5% vs. 12.0%). No poor magnetite composites were detected. The -0.25 + 0.16 mm fraction comprised 17.6% free fragments and 82.4% composites. All previously identified minerals were present among the free fragments, with the exception of magnetite. The gap between rich and poor composites widened to 71.2% and 11.2%, respectively. Beginning with the -0.16 + 0.074 mm fraction and continuing to finer sizes, all particles were fully liberated.

The distribution of minerals across size classes was non-uniform. The -0.5 mm fractions were the most enriched in francolite, due to the low selectivity of fine-grained ore enrichment. Grinding to -0.16 mm resulted in sharp reductions in coarse fractions and a marked increase in fines. Fine grinding maximised mineral

liberation at 0.16 mm, though it generated a significant volume of slimes. Hence, pre-classification using classifiers and hydrocyclones prior to grinding is recommended. The formation rate of the minus 0.16 mm fraction is shown in Figure 5, with rapid formation occurring within the first 16 minutes, followed by a sharp decline.



**Figure 5.** Kinetics of formation of -0.16 mm and -0.044 mm size fractions during sample grinding in a ball mill

**Notes:** 1 – Formation curve of the -0.16 mm size fraction; 2 – Formation curve of the -0.044 mm size fraction

**Source:** authors' development

Formation of the <0.16 mm fraction followed a polynomial equation with an approximation reliability of 0.9894:

$$R^+ = 0.0008 t^4 - 0.0236 t^3 - 0.188 t^2 + 10.743 t + 7.1234, \quad (3)$$

where  $R^+$  – retained portion on the 0.16 mm sieve, %;  $t$  – grinding time, min. Formation of the -0.044 mm fraction followed a polynomial with approximation reliability of 0.9954:

$$R^+ = 0.0082 t^3 - 0.2969 t^2 + 3.871 t + 2.4762, \quad (4)$$

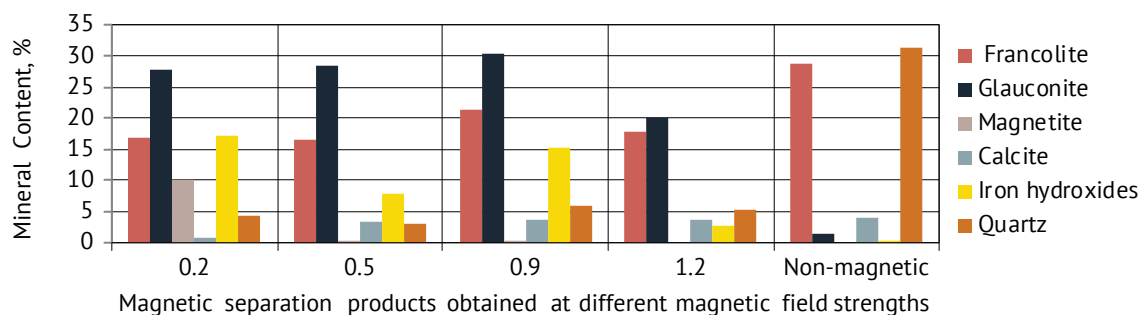
where  $R^+$  – retained portion on the 0.044 mm sieve, %;  $t$  – grinding time, min.

Detailed mineral-petrographic analysis of screened products clarified the grinding mechanism. The first phase involved intense disintegration of aggregates, liberating francolite grains. This was followed by disaggregation of intergrowths and grinding of liberated mineral grains. Due to its low hardness, francolite was prone to overgrinding and transitioned into the -0.044 mm fraction. During the study of raw material comminution, it was established that, in order to increase the degree of mineral intergrowth liberation, release of phosphorite and glauconite, and to reduce the content of refractory slimes, a closed grinding circuit with high circulating loads had to be employed.

Sedimentation analysis of post-grinding slimes demonstrated that the  $P_2O_5$  content in the +0.02 mm

and -0.02 mm fractions was evenly distributed, allowing the +0.02 mm fraction to be included in enrichment processes and thereby increasing overall  $P_2O_5$  recovery in the final product. The material, with slimes removed (-0.02 mm), was subjected to magnetic and flotation enrichment. Chemical analysis confirmed the sample as a high-carbonate feedstock with elevated iron content. Studies of magnetic susceptibility showed that glauconite and siderite exhibited weak magnetic properties, while phosphates and quartz were non-magnetic. The difference in susceptibility between glauconite/siderite and quartz/phosphates was sufficient for selective recovery in a high-intensity magnetic field.

The mineral distribution in magnetic separation products was found to be independent of feed composition, with magnetic products enriched in glauconite, iron hydroxides, and magnetite in varying ratios. Grinding to -0.16 mm did not significantly reduce phosphorus losses in magnetic products, indicating an isomorphic association between glauconite and phosphate. In non-magnetic products, an increase in non-magnetic mineral phases (calcite, quartz, clay, barite, palaeoresidue) was observed, depending on the material fed into the separator. The presence of these phases in magnetic products resulted from intergrowths with iron-rich minerals. Francolite was detected in both magnetic and non-magnetic products, mainly as intergrowths with glauconite. The distribution of minerals in the products of magnetic separation of the ground ore was presented in Figure 6.

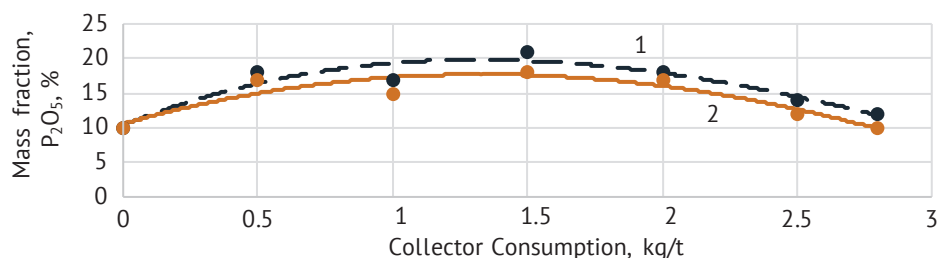


**Figure 6.** Mineral Distribution across magnetic separation products of raw material ground to 100% passing 0.16 mm  
**Source:** authors' development

The clay mineral content in magnetic products of ground materials increased from 3.3% to 19.2% with rising magnetic field induction (0.2 T to 1.4 T). In non-magnetic products, the content reached 30.0 – 44.0%. In intermediate products, content varied from 12.2% to 20.1%. Glauconite distribution in magnetic products peaked at 75.4% at 0.9 T and then declined to 52.2% at 1.4 T. In non-magnetic products, glauconite content was 1.3%.

Magnetic separation of ground feedstock enabled glauconite to be separated into a distinct product. However, due to residual francolite in magnetic products, additional separation of the flotation froth was

performed. In the non-magnetic product, francolite content was 28.8%, compared to 16.6–21.2% in magnetic products. Magnetic separation of the froth product improved the francolite concentration in the flotation fraction by removing glauconite. At 0.9 T, the magnetic field reduced total iron content in the froth phosphate product from 8–11% to 2–3%. Analysis of the flotation froth product from material ground to 100% -0.16 mm showed that the  $P_2O_5$  content in the phosphorite concentrate ranged from 18.7% to 21.5%, with  $P_2O_5$  recovery in the froth product ranging from 69.5% to 93%. The relationship between  $P_2O_5$  content and collector dosage (talactam and CTOS) is shown in Figure 7.



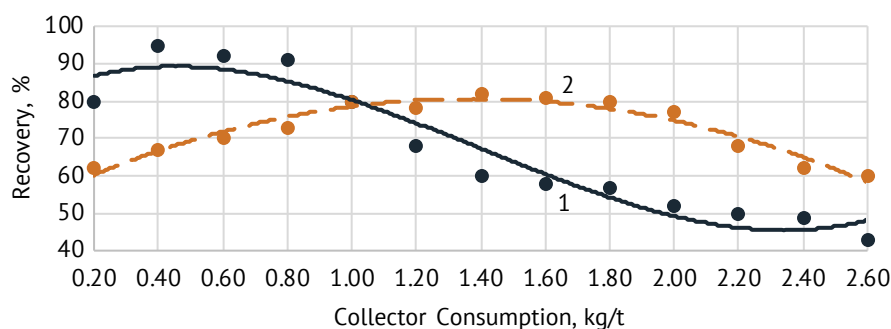
**Figure 7.** Dependence of mass fraction of phosphorus on collector consumption

**Notes:** 1 – Talactam; 2 – CTOS

**Source:** authors' development

Analysis of the flotation results of the ore indicated that the mass fraction of  $P_2O_5$  in the concentrate varied from 15.7 to 21.0%. The dependence of

phosphorus oxide recovery in the concentrate on the collector consumption (tall oil amide and CTOS) was presented in Figure 8.



**Figure 8.** Dependence of phosphorus oxide recovery into concentrate on collector consumption

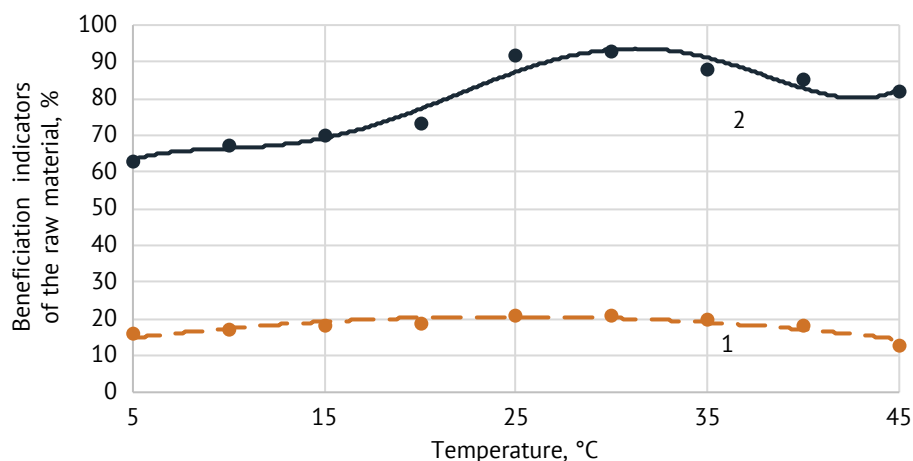
**Notes:** 1 – Talactam; 2 – CTOS

**Source:** authors' development



Analysis of the flotation results of the ore demonstrated that  $P_2O_5$  recovery in the concentrate varied from 93% to 69.5%. Further analysis demonstrated that increasing collector dosage above 2.0 kg/t impaired selectivity, causing not only phosphate but also quartz to report to the froth. With minimal collector dosage,  $P_2O_5$  content increased sharply while yield decreased. As collector dosage increased, concentrate yield rose

but quality declined. Talactam consumption was 40% lower than CTOS. Thus, talactam demonstrated superior collecting performance and selectivity, and was selected as the primary reagent in the recommended scheme. The results of the study on the influence of temperature on the selectivity of the flotation beneficiation process of technogenic phosphorus-bearing material were presented in Figure 9.



**Figure 9.** Dependence of beneficiation performance indicators of phosphorus-containing technogenic raw material on pulp temperature

**Notes:** 1 – change in mass fraction of phosphorus oxide in concentrate; 2 – change in phosphorus oxide recovery into concentrate

**Source:** authors' development

Analysis of the research results revealed that during beneficiation of the sample within the temperature range of 5 to 45°C. Low temperatures negatively impacted technogenic phosphorus-containing material recovery due to reduced bubble attachment strength on a mineral surface. At a suspension temperature in the range 0-15°C,  $P_2O_5$  recovery in the concentrate was 20-60%, with  $P_2O_5$  content at 15-18%. Using CTOS under the same conditions yielded only 10-30% recovery at 12-16%  $P_2O_5$ . Talactam mitigated the negative impact of low temperatures. Between 20°C and 35°C, recovery increased to 93%, with  $P_2O_5$  content at 20-21%.

Mineralogical analysis of flotation products from material ground to 100% -0.16 mm showed that glauconite (39.9-46.7%), iron hydroxides (4.2-6.9%), gypsum, and barite concentrated in the cell product. The froth product exhibited a 3-5-fold increase in francolite content – an advantageous outcome. However, clay mineral, quartz, calcite, and palaeoresidue were

also present. These may be removed during chemical treatment. The froth product is recommended for further magnetic separation to eliminate glauconite and enhance phosphorite concentrate quality. The cell product represented tailings of the process, with  $P_2O_5$  content at 4.25%. In flotation tests using the non-magnetic product (100% of the -0.16 mm size class;  $P_2O_5$  content: 12.93%), a phosphorite concentrate with 21%  $P_2O_5$  was obtained, meeting third-grade phosphorite flour standards, with a yield of 47.03%.  $P_2O_5$  recovery in the froth was 76.41%. The cell product had 5.76%  $P_2O_5$ .

Based on the conducted research, two variants of the beneficiation flowsheet were developed: magnetic-flotation and flotation – magnetic, both providing the production of phosphorite concentrate and glauconite product. The results of ore beneficiation under laboratory conditions according to the proposed flowsheets were presented in Table 2.

**Table 2.** Results of beneficiation of technogenic phosphorus-containing raw material

Flowsheet operation	Product name	Yield, %	Mass share, %		Recovery, %	
			P <sub>2</sub> O <sub>5</sub>	F <sub>total</sub>	P <sub>2</sub> O <sub>5</sub>	Fe <sub>total</sub>
Option 1						
Magnetic Separation	Magnetic (Glauconite Product)	50.71	8.24	12.07	39.60	81.60
	Non-magnetic	49.29	12.93	2.80	60.40	18.40
Flotation	Concentrate	23.18	21.0	1.70	46.15	5.25

Table 2. Continued

Flowsheet operation	Product name	Yield, %	Mass share, %		Recovery, %	
			P <sub>2</sub> O <sub>5</sub>	F <sub>total</sub>	P <sub>2</sub> O <sub>5</sub>	Fe <sub>total</sub>
	Tailings	26.11	5.76	3.78	14.25	13.15
	<b>Feed</b>	100.0	10.55	7.50	100.0	100.0
Option 2						
Flotation	Concentrate Middlings	41.92	19.28	5.98	76.60	33.40
	Tailings	58.08	4.25	8.60	23.40	66.60
Magnetic Separation	Magnetic (Glaucinite Product)	18.79	17.20	10.60	30.64	26.56
	Concentrate	23.12	20.97	2.22	45.96	6.84
	<b>Feed</b>	100.0	10.55	7.5	100.0	100.0

Source: authors' development

According to the first variant, magnetic beneficiation of the ground material to 100% of the -0.16 mm size class was proposed at the beginning of the flowsheet. This enabled the removal of iron-bearing minerals from the phosphorus-bearing product and, consequently, reduced the amount of material fed to flotation. Magnetic separation yielded a glauconite concentrate with a mass fraction of P<sub>2</sub>O<sub>5</sub> of 8.24% and 4.07%, and a phosphorite concentrate with a mass fraction of P<sub>2</sub>O<sub>5</sub> of 21.0%. In the second variant, magnetic beneficiation was applied to the concentrate obtained from flotation of the ground material. Magnetic separation produced a glauconite concentrate with a mass fraction of P<sub>2</sub>O<sub>5</sub> of 17.2%, and a phosphorite concentrate with a mass fraction of P<sub>2</sub>O<sub>5</sub> of 20.97%. Therefore, the first flowsheet variant (magnetic – flotation) proved to be more efficient and offered several advantages. Firstly, it allowed a 50.71% reduction in flotation load due to early recovery of the glauconite product during the initial stage of processing the technogenic phosphorus-bearing material. Secondly, phosphorus recovery in the concentrate was higher by 0.19%.

## Discussion

The obtained results were compared with those presented in scientific publications addressing similar aspects of natural and technogenic phosphorite ore beneficiation. The choice of enrichment methods in these studies was largely determined by the mineral composition of the initial feedstock.

H. Amar *et al.* (2022) demonstrated that the increasing demand for phosphate ore, driven by the depletion of high-grade reserves, has rendered the mining and processing of low-grade phosphates a necessity for sustainable production. The phosphate industry, identified as a major contributor of mining waste, consists of three main operational stages. The first involves ore extraction and separation from accompanying minerals and was sent to the beneficiation plant. In the second process, phosphate rock was concentrated using physical and chemical beneficiation methods. The resulting phosphate concentrate was then delivered to processing plants for the production of phosphoric acid and

various types of fertilisers. Enrichment generates substantial volumes of tailings composed of fine fractions, which represent an environmental burden and disrupt landscapes. Numerous studies have examined sedimentary low-grade phosphate deposits (<16% P<sub>2</sub>O<sub>5</sub>) to raise phosphate quality to market specifications (~30% P<sub>2</sub>O<sub>5</sub>). Applied processes have ranged from simple mechanical treatments (crushing, grinding and classification) to complex operations such as flotation and leaching. Pre-enrichment stages have included attrition, scrubbing, washing, and desliming to eliminate clays and fine silicates. Flocculation was found to be highly effective, achieving sedimentation levels of 81.3% in just 15 seconds using anionic flocculants. Polyacrylamide flocculants showed superior performance, achieving 37 times faster sedimentation than natural settling.

R. Dabbebi *et al.* (2023) explored the reuse of phosphate mine waste (PMW) across various sectors. Their study highlighted the potential of PMW as raw material for multiple applications within sustainable management frameworks. The authors emphasised that the variable mineralogical composition and elemental content of PMW, dependent on deposit origin and enrichment methods, made it suitable for diverse uses. Secondary utilisation of phosphate waste is a complex process requiring detailed examination of the physical and mineralogical properties of tailings. The most efficient phosphate recovery method identified was direct flotation using anionic fatty acid collectors. PMW was also recognised as a rich source of heavy metals and rare-earth elements (REEs). Elements such as Cd, Cr, Mn, Mo, Ni, Pb, U, V, and Zn, along with phosphorus, were extracted from phosphate slimes. Zinc and nickel were successfully recovered via leaching. REEs were most effectively recovered using flotation.

In a study by J. Guo *et al.* (2024), it was emphasised that the successful beneficiation of phosphate ore containing phosphates, apatite, calcite, and dolomite was based on a thorough understanding of its mineralogy, mineral surface properties, distribution, and mineral liberation. Due to the relatively low P<sub>2</sub>O<sub>5</sub> content and high impurity levels, most ores were unsuitable for direct acidulation and required pre-enrichment to

produce suitable concentrates. Applied methods included flotation, attrition cleaning, desliming, electrostatic separation, magnetic separation, gravity concentration, and calcination. In direct flotation, anionic collectors were used differentially, with optimum pulp alkalinity at pH 9.5 maintained by  $\text{Na}_2\text{CO}_3$ . Phosphate ores (53–63  $\mu\text{m}$ ) from Rajasthan and Madhya Pradesh (India) underwent two-stage high-gradient magnetic separation following calcination and flotation. The use of sodium oleate and sodium metasilicate yielded a final concentrate containing 31.5%  $\text{P}_2\text{O}_5$  and 8.8%  $\text{SiO}_2$ , with an overall  $\text{P}_2\text{O}_5$  recovery of 65.0%.

A. Kareeva *et al.* (2023) investigated Chilisai low-grade phosphorites through a comprehensive study of their physicochemical properties. Techniques employed included chemical and energy-dispersive analysis, FTIR spectroscopy, X-ray diffraction, and mineralogical analysis. The presence of glauconite, a hydromica composed of iron and aluminium silicate, quartz, and potassium oxides, was identified alongside dolomite. The latter was often misidentified as calcite; however, its crystal structure, with calcium and magnesium ion mobility along three axes, enabled distinction. The results offered new insights into the unique characteristics of Chilisai phosphorites, such as REE traces and ion mobility in glauconite and dolomite, which are critical for identifying novel phosphorus-bearing feedstocks.

A. Mahmoud *et al.* (2024) studied low-grade phosphate ore from Abu Tartur in Egypt's New Valley Governorate. Estimated at 980 million tonnes at 30% phosphate content, it is one of the country's largest deposits. Phosphate extraction was performed using gravity, magnetic, and flotation enrichment. Chemical composition ( $\text{P}_2\text{O}_5$ ,  $\text{CaO}$ ,  $\text{SiO}_2$ ,  $\text{SO}_2$ ,  $\text{Fe}_2\text{O}_3$ ) was determined via X-ray diffraction. The flotation process was negatively affected by fine fractions <0.074 mm, prompting mechanical impurity removal using modern classifiers. This improved  $\text{P}_2\text{O}_5$  content from 21.27% to 22.38%. Magnetic separation with a Dings cross-belt separator increased  $\text{P}_2\text{O}_5$  content to 25.25% with 90.3% recovery. Flotation of the non-magnetic product using a mixed reagent system (oil and oleic acid) produced a concentrate with 28.29%  $\text{P}_2\text{O}_5$  and 86.14% recovery.

X. Zhang *et al.* (2022) developed an effective desliming method for siliceous phosphate ore. MLA and SEM-EDS microscopy characterised mineral components and morphology. The effect of Falcon centrifuge parameters (rotation speed, water pressure, feed rate) on separation efficiency was evaluated. Orthogonal experimental design was used to establish a predictive model. Laboratory tests confirmed that at 64.73 Hz, 0.012 MPa, and 30.51 mL/s, maximum  $\text{P}_2\text{O}_5$  recovery of 86.02% was achieved at 27.45%  $\text{P}_2\text{O}_5$ . Most valuable mineral loss occurred in the -0.045 mm fraction. Optimisation experiments identified 0.045–0.09 mm as the optimal particle size range. By implementing these recommendations, flotation feed optimisation reduced grinding costs.

M. Derqaoui *et al.* (2021) reviewed reagents for direct froth flotation of low-grade phosphates. Mixed collectors were found to provide better apatite selectivity and recovery than fatty acids, which required selective depressants. Apatite flotation efficiency was shown to depend not only on reagent type but also on adsorption mechanisms and solution chemistry. Surfactants such as dodecylaspartate and dodecylglutamate, containing two and three carbon atoms between carboxyl groups respectively, exhibited micellisation coefficients 4–5 times higher than dodecylglycinate. Hydrogen bonding promoted denser molecular packing at the air–water interface and within micelles. Formation of intermolecular complexes led to lower surface tension. Internal chelation was advantageous for calcium-bearing surfaces. While dodecylaminomalonnate and dodecylaspartate showed strong adsorption on hydroxyapatite, dodecylglutamate adsorbed weakly. On non-specific surfaces, the three surfactants displayed similar adsorption behaviour.

Analysis of the current study in comparison with previous works confirmed the relevance of technogenic phosphorus-containing feedstock beneficiation and validated the proposed process flowsheet, comprising: closed-circuit grinding with high circulating loads to minimise slime formation; high-intensity magnetic separation to reduce flotation load; flotation using a complex anionic collector (talactam) to ensure selectivity and performance.

## Conclusions

The conducted research confirmed the effectiveness of utilising phosphorite ore processing waste to produce concentrates suitable for the manufacture of third-grade phosphorite flour. The glauconite products obtained following francolite removal were found suitable for use in the production of potassium fertilisers and green pigments.

It was established that fine grinding of technogenic phosphorus-containing material resulted in significant slime formation. To minimise this, disintegration should be preceded by classification using hydrocyclones and classifiers, with a particle size cut-off of 0.16 mm. Experimental studies demonstrated that magnetic separation of feed material with 100% -0.16 mm reduced total iron content in the non-magnetic product from 8–11% to 2–3%, while increasing  $\text{P}_2\text{O}_5$  content by a factor of 1.18–2.63.

Flotation trials demonstrated the possibility of producing phosphorite concentrates with  $\text{P}_2\text{O}_5$  contents ranging from 18.7% to 21.5% and recoveries between 69.5% and 93%. Magnetic separation of flotation froth products enabled partial enhancement of francolite concentration in the froth fraction and a significant reduction in glauconite and iron hydroxide content. However, the advisability of magnetic separation post-flotation should be confirmed via detailed techno-economic

assessments, incorporating downstream chemical processing considerations.

Based on the processing of technogenic phosphorus-containing samples with initial  $P_2O_5$  contents of 10.55% and 5.45%, market-grade phosphorite concentrates were obtained with  $P_2O_5$  contents of 18.7-21.5% and recoveries of 69.5-93%.

Two laboratory-verified flowsheet variants were developed for feedstock enrichment: magnetic-flotation and flotation-magnetic. Both enabled production of phosphorite concentrate and glauconite product. In the first variant, magnetic separation performed at the head of the circuit, produced glauconite concentrates with  $P_2O_5$  contents of 8.24% and 4.07%, and phosphorite concentrate with 21.0%  $P_2O_5$ . In the second variant, magnetic separation followed flotation, producing glauconite concentrate with 17.2%  $P_2O_5$  and phosphorite concentrate with 20.97%  $P_2O_5$ .

For the first time, it was demonstrated that, during technogenic phosphorus-containing material flotation,

interfacial surface tension energy at the interfaces of the three-phase system must be considered. This finding substantiates the recommendation of talactam, an anionic collector, for the selective separation of francolite from quartz, and defines the optimal operational conditions for the technological process. The questions examined in this study require further investigation. The results obtained are consistent with findings in related research and may serve as a foundation for more in-depth exploration of REE recovery from technogenic phosphorus-containing materials.

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## Conflict of Interest

None.

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## Збагачення техногенної фосфорвміщуючої сировини як джерела отримання мінеральних добрив

● **Анотація.** Актуальність досліджень пов'язана з невирішеними питаннями зростання вартості сировини, геополітичної нестабільності та складності переробки фосфоритових руд із високим вмістом домішок, що обумовлює необхідність пошуку ефективних підходів до збагачення та переробки низькосортної сировини й відходів як альтернативних джерел фосфору. Мета дослідження – розробка технології збагачення техногенної фосфорвмісної сировини для одержання фосфоритового концентрату, придатного для виробництва добрив. При виконанні досліджень використано мінералогічний, хімічний, гранулометричний та седиментаційний аналізи складу сировини. При виконанні лабораторних експериментів використані подрібнення, флотація та магнітна сепарація. Досліджено властивості реагентів для розробки флотаційної технології збагачення фосфорвмісної сировини. У роботі досліджено можливості збагачення техногенної фосфорвмісної сировини, отриманої в результаті переробки фосфоритової руди. Запропоновано дві лабораторно апробовані технологічні схеми – магнітно-флотаційну та флотаційно-магнітну, що забезпечують одержання товарного фосфоритового концентрату з масовою часткою  $P_2O_5$  від 18,7 до 21,5 % при вилученні фосфору 69,5–93 %. Уперше встановлено необхідність урахування енергії поверхневого натягу на межі розділення трьох фаз під час флотації, що дозволило обґрунтувати доцільність застосування аніонного збирача – талактаму – для ефективного розділення франколіту та кварцу. Проведення магнітної сепарації як до, так і після флотації сприяло підвищенню вмісту франколіту та зменшенню вмісту глауконіту і залізовмісних мінералів. Встановлено, що попередня класифікація сировини за граничною крупністю 0,16 мм дозволяє зменшити утворення шламів при дезінтеграції. Отримані глауконітові продукти можуть бути потенційно використані для виробництва калійних добрив та зелених пігментів, однак це потребує подальших досліджень. Практична значимість роботи полягає в тому, що впровадження у виробництво розробленої технології збагачення техногенної фосфоритової сировини дозволило отримати фосфатний концентрат в кількості 23,18 %, який придатний для виготовлення мінерального добрива – фосфатного борошна III-го сорту, та глауконітовий продукт в кількості 50,71 %, що використовується як ентеросорбент та джерело мікроелементів у складі комбікормів

● **Ключові слова:** фосфор; франколіт; глауконіт; магнітна сепарація; флотація