



Upgrading low-grade flotation apatite tailings via selective leaching with citric acid

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ABSTRACT

Depletion of high-grade phosphate resources necessitates the recovery of phosphorus (P_2O_5) from flotation tailings. This study investigated a citric acid leaching process for selectively dissolving carbonate gangue minerals and enriching P_2O_5 in low-grade apatite flotation tailings. Mineralogical and chemical characterizations revealed an initial P_2O_5 grade of 3.20%, with calcite, dolomite, and silicate minerals identified as the predominant impurities. The effects of citric acid concentration (1–11% w/v), temperature (30–60 °C), leaching duration (20–80 min), and agitation speed (300–600 rpm) were systematically evaluated. The selected operating conditions were 3% (w/v) citric acid, 50 °C, 40 min, and 400 rpm, with a liquid-to-solid ratio of 10 mL/g. Under these conditions, the P_2O_5 grade of the solid residue increased to 9.16%, corresponding to an enrichment factor of 2.86, with a reported P_2O_5 recovery of 88.50%. The MgO grade decreased from 5.40% to 0.85%, representing an 84.26% relative decrease in grade. Higher acid concentrations and prolonged leaching were associated with lower P_2O_5 grades, suggesting reduced selectivity and possible dissolution of phosphate-bearing phases. Although citric acid leaching enhanced the P_2O_5 grade of the tailings, the resulting product should be considered a pre-enriched intermediate rather than a directly qualified feedstock for wet-process phosphoric acid production. Further beneficiation, process qualification, and techno-economic evaluation are required to assess its potential for industrial application.

Introduction

Phosphorus is an essential, irreplaceable nutrient vital for global agricultural productivity and chemical manufacturing. Phosphorus rock, primarily comprising fluorapatite [$Ca_{10}(PO_4)_6F_2$], is classified as a critical raw material due to the rapid depletion of high-grade global reserves [1, 2]. To satisfy the growing demand for phosphate fertilizers, mineral processing heavily relies on froth flotation to treat low-grade deposits [3]. However, processing complex phosphate ores containing carbonate gangues (e.g., calcite, $CaCO_3$, and dolomite, $CaMg(CO_3)_2$) presents severe technical challenges. Due

to the similar surface physicochemical properties between apatite and carbonates, flotation often yields low separation efficiency and generates vast flotation tailings containing unrecovered phosphorus (P_2O_5) [3, 4]. Discharging these tailings poses major environmental hazards, including land occupation, dust emission, and water eutrophication via nutrient leaching [5]. Simultaneously, disposing of tailings with 3–5% P_2O_5 represents a significant loss of secondary resources [6, 7]. Consequently, P_2O_5 valorization and re-enrichment from tailings have become a major focus in sustainable extractive metallurgy [2, 6, 8, 14].

Chemical leaching selectively dissolves gangue minerals to upgrade low-grade ores and tailings. Traditionally, inorganic mineral acids like sulfuric (H_2SO_4), nitric (HNO_3), or hydrochloric (HCl) acids are employed for carbonate removal [9]. Although effective, strong inorganic acids lack selectivity—leading to high apatite dissolution—consume excessive reagents, generate corrosive effluents, and damage equipment [9, 10]. Furthermore, H_2SO_4 reacts with calcium-bearing gangues to form a dense calcium sulfate (CaSO_4) precipitate layer, inhibiting mass transfer and leaching kinetics [11].

To overcome these limitations, organic acid leaching offers a green alternative [8, 12]. Low-molecular-weight organic acids, such as citric ($\text{C}_6\text{H}_8\text{O}_7$), formic (HCOOH), and acetic (CH_3COOH) acids, possess moderate acid strength ($\text{p}K_a$) and strong chelating capabilities [10, 11]. Citric acid is particularly promising due to its biodegradability, non-toxicity, and tri-carboxylic groups ($\text{p}K_{a1} = 3.13, \text{p}K_{a2} = 4.76, \text{p}K_{a3} = 6.40$) [13, 12]. Carboxylic protons (H^+) selectively dissolve carbonate minerals, while liberated citrate anions ($\text{C}_6\text{H}_5\text{O}_7^{3-}$) form soluble complexes with Ca^{2+} and Mg^{2+} , preventing surface passivation [14, 15]. While organic acids can enrich sedimentary phosphorites [10, 12], systematic optimization for low-grade flotation apatite tailings—specifically balancing carbonate dissolution and apatite preservation—remains limited [6].

Therefore, this study investigates a selective leaching process using citric acid to upgrade low-grade flotation apatite tailings ($\sim 3.20\% \text{P}_2\text{O}_5$). Operating parameters were systematically investigated and structural transformations were characterized via XRD and EDX, providing a basis for phosphorus recovery from mining waste.

Experimental

Materials and Characterization

Phosphate flotation tailings (type-III Apatite ore origin) were provided by the Institute of Industrial Chemistry, Vietnam. The raw material was homogenized and dried at 105°C to a constant weight prior to use. Analytical grade citric acid ($\text{C}_6\text{H}_8\text{O}_7$, $\geq 99.0\%$, China) and double-distilled water were used in all leaching experiments.

Mineralogical phases of the raw tailings and solid residues were identified by X-ray Diffraction (XRD, Rigaku). Elemental compositions were quantified via Energy-Dispersive X-ray Spectroscopy (EDX, JEOL).

Calculation of P_2O_5 grade and recovery

Calculation of P_2O_5 Grade and Recovery

The P_2O_5 grade was calculated from the measured phosphorus content using the stoichiometric conversion factor ($M_{\text{P}_2\text{O}_5}/(2M_{\text{P}}) \approx 2.291$):

$$w_{\text{P}_2\text{O}_5} = w_{\text{P}} \times \frac{M_{\text{P}_2\text{O}_5}}{2M_{\text{P}}}$$

The P_2O_5 recovery was calculated from the mass of P_2O_5 retained in the solid product relative to that in the initial feed:

$$R_{\text{P}_2\text{O}_5}(\%) = \frac{m_p \cdot G_p}{m_f \cdot G_f} \times 100$$

where m_f and m_p represent the dry masses of the feed and the final solid product, respectively, and G_f and G_p are their corresponding P_2O_5 grades expressed on the same mass-percentage basis. The reported recovery was checked against the measured product mass and chemical composition to ensure consistency with the overall phosphorus mass balance.

Leaching Experiments

Selective leaching of carbonate impurities was conducted in a 250 mL batch glass reactor (inner diameter 6.2 cm) equipped with a cylindrical magnetic stirrer (length 3.0 cm, radius 0.6 cm) and temperature control. In a typical run, 10.00 g of dried tailings was added to 100 mL of citric acid solution. A single-factor optimization strategy was applied to investigate the key parameters: acid concentration (1–11%), temperature (30–60°C), leaching time (20–80 min), and stirring speed (300–600 rpm). The detailed experimental matrix and sample designations are summarized in Tab 1.

Tab 1. Summary of experimental parameters and sample codification.

Experimental Parameter	Tested Conditions & Corresponding Sample Codes
Acid Concentration	$C_1=1\%, C_2=3\%, C_3=5\%, C_4=7\%, C_5=9\%, C_6=11\%$
Temperature ($^\circ\text{C}$)	$K_1=30, K_2=40, K_3=50, K_4=60$
Leaching Time (min)	$T_1=20, T_2=40, T_3=60, T_4=80$
Stirring Speed (rpm)	$R_1=300, R_2=400, R_3=500, R_4=600$

Upon completion of each reaction, the slurry was rapidly filtered under vacuum. The solid residue was washed with distilled water, dried at 105°C for 1.5 h, and weighed before phase and chemical analyses. The general leaching flowchart is illustrated in Fig. 1.

All leaching experiments were performed in duplicate to ensure reproducibility, with average values reported. The chemical composition changes, phase transformations, and calculated P_2O_5 recovery rates obtained under these varied operational conditions are systematically analyzed and discussed in the following section.

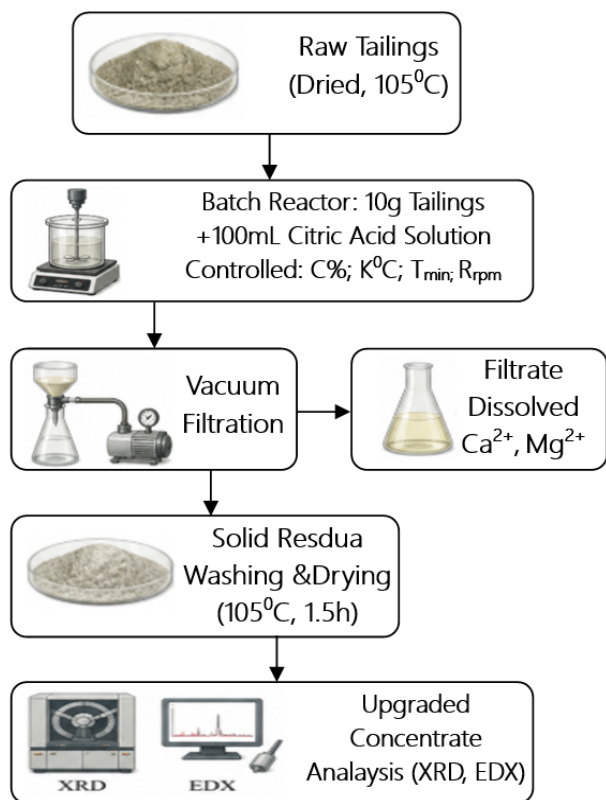


Fig. 1: Schematic diagram of the selective citric acid leaching process for phosphate tailings.

Results and discussion

Characterization of Raw Phosphate Flotation Tailings

The elemental and mineralogical compositions of the raw flotation tailings were characterized to establish a baseline. As shown in Fig. 2a, the XRD pattern reveals a complex multiphase mineral assemblage. The dominant diffraction peak at $2\theta = 26.66^\circ$ corresponds to quartz (SiO_2 , Qz). Characteristic reflections for kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$, K) appear at $2\theta = 17.86^\circ$ and 20.88° , while hematite (Fe_2O_3 , H) is identified at $2\theta = 36.58^\circ$. Fluorapatite ($\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$, A) exhibits distinct peaks at $2\theta = 26.9^\circ$ and 31.94° . Acid-soluble carbonate minerals are represented by calcite (CaCO_3 , C) at $2\theta = 50.16^\circ$ and dolomite ($\text{CaMg}(\text{CO}_3)_2$, D) at $2\theta = 59.98^\circ$.

The EDX spectrum (Fig. 2b) further corroborates these mineralogical findings, displaying major constituent elements consisting of O (53.1 wt%), C (15.4 wt%), Si (11.9 wt%), Al (7.0 wt%), Ca (3.6 wt%), and P (2.1 wt%), along with trace quantities of K (2.9 wt%), Fe (2.2 wt%), Mg (1.1 wt%), Mn (0.5 wt%), and Ti (0.2 wt%). The presence of Ca and P confirms the targeted apatite phase, whereas the substantial concentrations of C, Mg, and Ca are consistent with the carbonate impurities (calcite and dolomite). The prominent Si and Al signals further validate the abundance of silicate and aluminosilicate gangue

minerals. These combined structural and compositional insights confirm that the raw tailings contain a mix of phosphate minerals and acid-reactive carbonate phases, establishing a solid theoretical foundation for the selective dissolution of carbonates using organic citric acid.

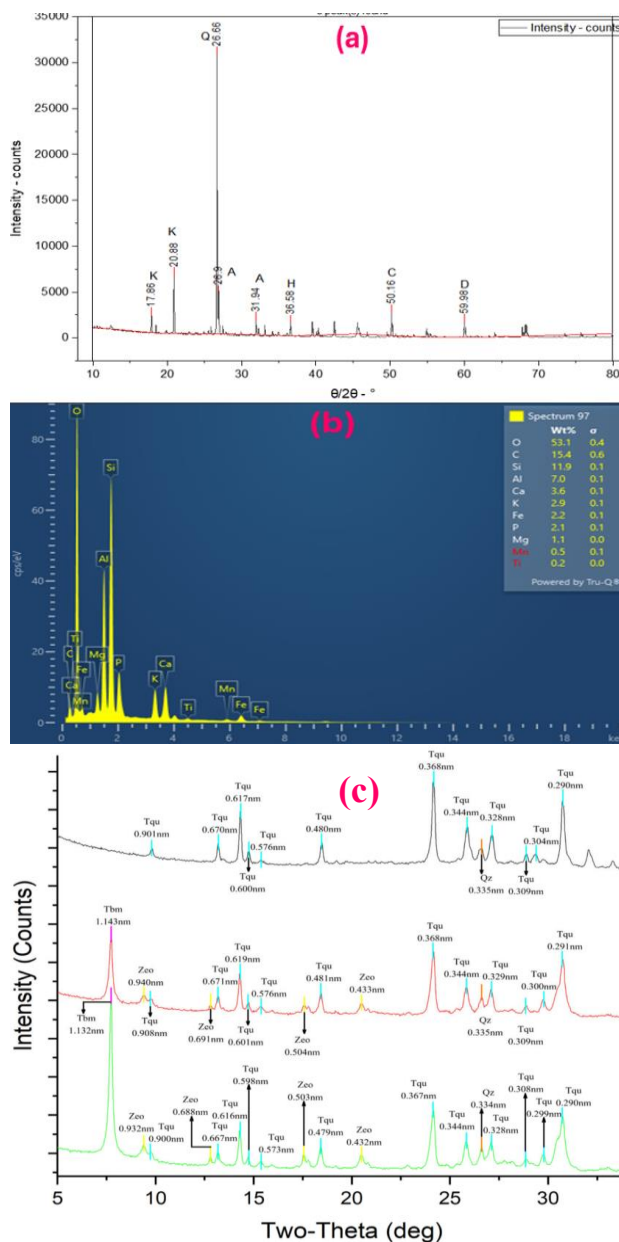


Fig. 2: (a) XRD pattern and (b) EDX spectrum of the raw phosphate flotation tailings (represented by the gray curve), (c) comparative X-ray diffraction (XRD) patterns and phase identification of the three samples shown via the gray, red, and green curves.

Furthermore, a comparative phase analysis among the three distinct sample states (Fig. 2c: the raw tailings represented by the gray curve, and the leaching residues represented by the red curve and green curve, respectively) demonstrates noticeable structural evolution. The XRD patterns exhibit diffraction

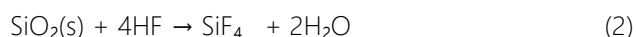
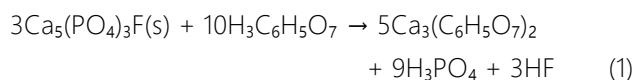
reflections assigned to turquoise ($\text{CuAl}_6(\text{PO}_4)_4(\text{OH})_8 \cdot 4\text{H}_2\text{O}$, Tqu), quartz (SiO_2 , Qz), tobermorite ($\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$, Tbm), and zeolite (Al-Si-O framework, Zeo). The quartz peak occurs at $d \approx 0.335$ nm ($2\theta \approx 26 \div 27^\circ$), while the prominent tobermorite-related reflection appears at $d \approx 1.132 \div 1.143$ nm in the green and red curves, respectively. Additional reflections assigned to zeolite occur at $d \approx 0.432 \div 0.433$ nm and $0.503 \div 0.504$ nm. These differences indicate variations in crystalline phase characteristics among the samples, reflecting the impact of the processing conditions.

Effect of Leaching Parameters on Carbonate Removal and P_2O_5 Grade

Effect of Citric Acid Concentration on Leaching Efficiency

The influence of citric acid concentration (1–11% w/v) on the selective leaching of carbonate gangue minerals from the apatite flotation tailings was evaluated at 35 °C for 60 min under continuous agitation at 500 rpm. As illustrated in Fig. 3a, the P_2O_5 grade in the solid residue exhibited a non-monotonic trend with increasing acid concentration. Elevating the citric acid concentration from 1% to 3% enhanced the P_2O_5 content from 5.50% (sample C1) to a maximum value of 6.18% (sample C2). At relatively low acid dosages ($\leq 3\%$), citric acid preferentially dissolved carbonate gangue phases (calcite and dolomite) and surface oxides without inducing substantial dissolution of the fluorapatite phase, thereby enriching phosphorus within the solid residue.

Conversely, further increasing the acid concentration from 5% to 11% (samples C3–C6) resulted in a pronounced reduction in the residual P_2O_5 grade, which declined to 3.28% at 5% (C3), 0.30% at 7% (C4), 0.19% at 9% (C5), and 0.14% at 11% (C6). Excessive proton (H^+) activity and high citrate ligand concentrations promoted non-selective chemical attack on the fluorapatite crystal lattice and silicate matrix according to Eqs. (1) and (2):



This loss of leaching selectivity led to severe phosphorus dissolution into the aqueous phase. Consequently, 3% (w/v) citric acid was selected as the optimal reagent concentration for subsequent investigations to balance carbonate removal, phosphate preservation, and reagent consumption.

Effect of Reaction Temperature

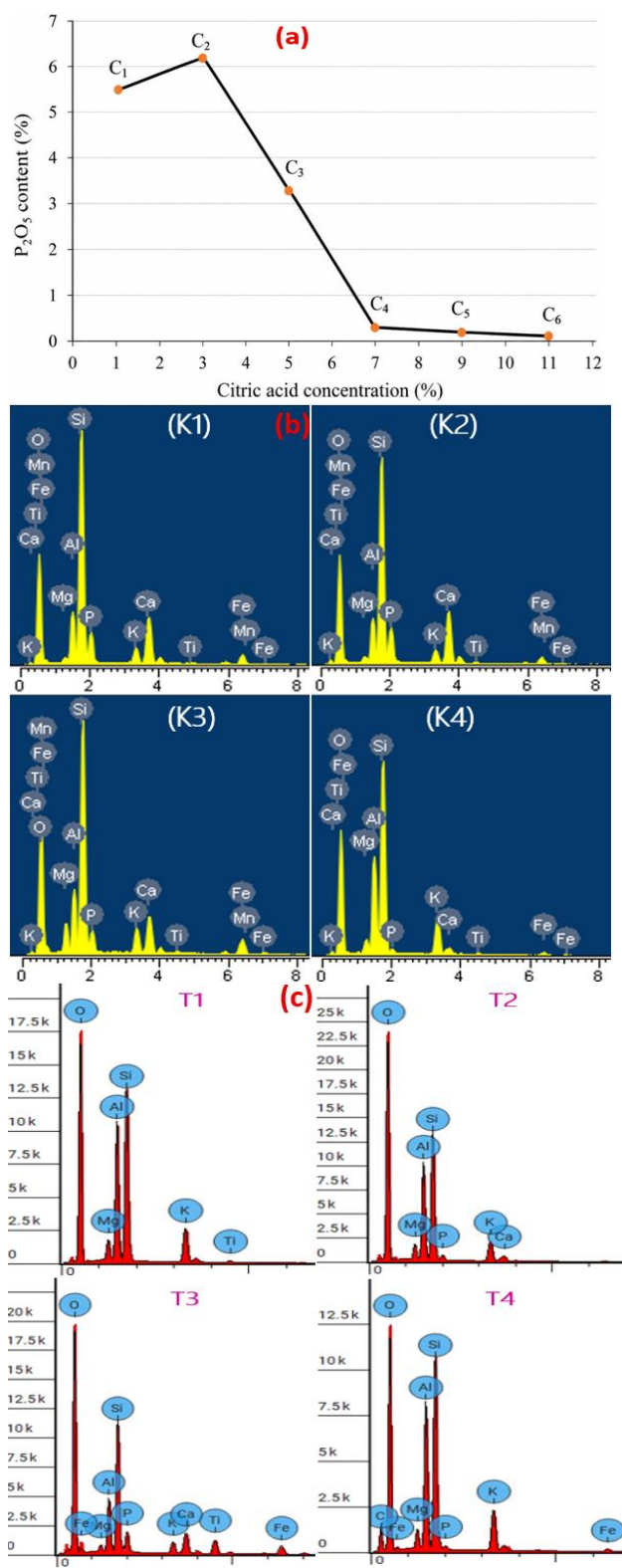


Fig. 3: (a) Effect of citric acid concentration on the P_2O_5 content of the leached residue; (b) EDX spectra of solid residues obtained at different reaction temperatures (K1: 30 °C, K2: 40 °C, K3: 50 °C, and K4: 60 °C); and (c) EDX spectra of solid residues obtained after various leaching durations (T1: 20 min, T2: 40 min, T3: 60 min, and T4: 80 min).

The temperature dependence of the leaching process was investigated across the range of 30–60 °C using 3% (w/v) citric acid for 60 min at an agitation speed of 500 rpm. The elemental variations of the resulting solid residues were monitored via EDX analysis (Fig. 3b, samples K1–K4), from which the corresponding P_2O_5 grades were determined. Increasing the reaction temperature from 30 °C (K1) to 50 °C (K3) progressively elevated the residual P_2O_5 content from 5.24% (K1) to 6.85% at 40 °C (K2), reaching a maximum of 8.68% at 50 °C (K3). As reflected in the EDX spectra (Fig. 3b), the relative intensities of the P and Ca peaks were well preserved from K1 to K3 relative to the Si and Al gangue signals, indicating enhanced selective removal of carbonate impurities. Higher thermal energy accelerates molecular diffusion and interfacial reaction kinetics, promoting the preferential dissolution of carbonate and oxide gangue phases.

However, raising the temperature further to 60 °C (sample K4) resulted in a decline in the P_2O_5 grade to 6.25%, accompanied by a noticeable attenuation of the P and Ca characteristic peaks relative to the Si and Mg signals in the K4 spectrum (Fig. 3b). At elevated temperatures (> 50 °C), once accessible surface carbonates are depleted, increased thermal activation promotes the partial co-dissolution of the apatite phase. Furthermore, solution evaporation at higher temperatures alters the effective acid concentration and destabilizes the solid-liquid equilibrium [6]. Therefore, 50 °C was established as the optimal reaction temperature.

Effect of Leaching Duration

The influence of leaching duration was evaluated from 20 to 80 min under the established conditions (3% w/v citric acid, 50 °C, 500 rpm), with the corresponding EDX spectra of the solid residues (samples T1–T4) presented in Fig. 3c. Extending the reaction time from 20 min (T1) to 40 min (T2) markedly increased the calculated P_2O_5 grade from 3.89% (T1) to a peak value of 9.02% (T2). In the EDX spectrum of sample T1 (Fig. 3c), the P and Ca peaks remained barely discernible against the dominant O, Si, and Al signals, indicating incomplete carbonate dissolution within 20 min. At 40 min (T2), distinct P and Ca signals emerged alongside a reduced Mg peak, confirming effective exposure and enrichment of the fluorapatite phase as surface carbonates were selectively leached.

Beyond 40 min, however, the P_2O_5 grade decreased to 5.48% at 60 min (T3) and 5.43% at 80 min (T4). Once the reactive carbonate gangue was exhausted, prolonged acid exposure led to secondary attack on the apatite phase, diminishing phosphorus retention in the solid product, as evidenced by the weakened P and Ca signals

in the T4 spectrum (Fig. 3c). Consequently, a leaching duration of 40 min was selected as the optimal reaction time to maximize carbonate removal while minimizing phosphate loss and energy consumption.

Effect of Agitation Speed

The influence of hydrodynamics on carbonate dissolution was systematically investigated across a stirring speed range of **300–600 rpm** (3% wt/v citric acid, **T = 50°C**, **t = 40 min**, liquid-to-solid ratio = **10 mL/g**). Elevating the stirring speed from **300 rpm (R₁)** to **400 rpm (R₂)** markedly improved the P_2O_5 grade in the solid residue from **8.02%** to a peak value of **9.16%**. This enhancement is attributed to the reduction in liquid-solid boundary layer thickness around the mineral particles at higher shear rates, which minimizes external mass transfer resistance and promotes the dissolution kinetics of carbonate gangues.

However, a further increase in agitation speed to **500 rpm (R₃)** and **600 rpm (R₄)** yielded a negligible change in the P_2O_5 content (**9.11%**). This plateau suggests a regime shift in the leaching process from external mass-transfer diffusion control to chemical reaction kinetic control above **400 rpm**. Furthermore, excessive agitation (≥ 500 rpm) induced severe fluid splashing and mechanical instability within the reactor setup. Consequently, **400 rpm** was identified as the optimal agitation speed for subsequent experiments.

Summary of Optimal Leaching Conditions and Process Efficiency

The single-factor experiments identified 3% (w/v) citric acid, 50 °C, 40 min, a liquid-to-solid ratio of 10 mL/g, and an agitation speed of 400 rpm as the selected operating conditions. Among the tested agitation speeds, 400 rpm produced the highest reported P_2O_5 grade of 9.16%. The concentration, temperature, and duration experiments independently identified favorable conditions for phosphate enrichment, while the agitation experiment provided the highest grade under its specific test conditions.

These results indicate that the performance of citric-acid leaching depends on balancing gangue dissolution against phosphorus retention. However, P_2O_5 grade alone does not quantify phosphate recovery or the mass-based removal of impurities. Therefore, the overall performance should also be assessed using the solid mass yield, P_2O_5 recovery, and relevant impurity concentrations. The selected conditions should be regarded as the preferred operating regime within the investigated experimental ranges rather than as a universally optimal condition.

Phase and elemental evolution under optimal conditions

The solid residue obtained under optimal conditions (3% wt/v citric acid, 50 °C, 40 min, L/S = 10 mL/g, 400 rpm) was characterized.

Tab. 2: Elemental composition (wt%) of raw tailings and upgraded concentrate (UC) under optimal conditions.

Sample	Raw Tailings	UC
P ₂ O ₅	3.20	9.16
CaO	48.50	51.20
MgO	5.40	0.85
SiO ₂	18.20	22.40
Fe ₂ O ₃	2.10	1.95
Al ₂ O ₃	1.80	1.65
Loss on Ignition (LOI)	20.80	4.10

Note on Table 2: Oxides and P₂O₅ were calculated from elemental analyses and stoichiometric conventions. LOI was measured by calcination at 950 °C.

The MgO content decreased from **5.40%** to **0.85%** (representing an **84.26%** relative decrease in MgO

grade), while LOI dropped from **20.80%** to **4.10%**. The XRD and EDX results provide indirect evidence of preferential carbonate dissolution during citric acid leaching. The disappearance or marked reduction of diffraction reflections assigned to calcite and dolomite, together with the decrease in MgO content and LOI, is consistent with the removal of carbonate-rich gangue. Meanwhile, the persistence of fluorapatite reflections indicates that the apatite crystalline phase remained detectable after treatment. However, these solid-phase characterizations do not independently establish the complete preservation of apatite or quantify phosphorus dissolution into the liquid phase. The proposed mechanism should therefore be interpreted as a plausible explanation of observed changes rather than definitive proof of selective dissolution.

Comparative Performance Analysis

To highlight the efficacy and practical advantages of the proposed process, the leaching performance and separation metrics obtained in this work were benchmarked against previously reported leaching technologies for low-grade phosphate ores and flotation tailings (Table 3).

Tab. 3: Comparative performance of various leaching agents for upgrading low-grade phosphate ores/tailings.

Leaching Agent	Feed Ore / Tailing Type	Feed P ₂ O ₅ (%)	Product P ₂ O ₅ (%)	MgO Removal (%)	Feed P ₂ O ₅ Recovery (%)	Ref.
Sulfuric Acid (H ₂ SO ₄)	High-calcium ore	15.20	28.50	65.40	72.10	[9]
Nitric Acid (HNO ₃)	Carbonate-rich phosphate	18.10	29.30	82.00	78.50	[11]
Formic Acid (HCOOH)	Sedimentary phosphate	14.50	25.20	88.60	81.00	[10,12,14]
Acetic Acid (CH ₃ COOH)	Flotation tailings	11.80	22.40	74.30	79.80	[6, 16]
Citric Acid (C ₆ H ₈ O ₇)	Phosphate tailings	15.84	30.10	81.50	82.50	[6, 7]
Citric Acid (C ₆ H ₈ O ₇)	Flotation Tailings (Type-III)	3.20	9.16	84.26	88.50	This Work

Conventional mineral acids (H₂SO₄, HNO₃) rapidly dissolve carbonates; however, their non-selective nature leads to severe co-dissolution of the phosphate matrix (apatite) and generates hazardous secondary wastes, such as phosphogypsum [7, 9, 16]. Conversely, weak organic acids provide controlled dissociation (pK_a) and enhanced leaching selectivity. Among them, triprotic citric acid (C₆H₈O₇; pK_{a1} = 3.13, pK_{a2} = 4.76, pK_{a3} = 6.40) exhibits significant advantages over monoprotic acetic or formic acids due to its strong multidentate chelating capability. The liberated citrate anions (C₆H₅O₇³⁻) form highly stable, soluble complexes with dissolved Ca²⁺ and Mg²⁺ ions, efficiently driving

carbonate dissolution under ambient conditions without requiring severe operational environments [15, 16, 19]. This selective chelation capability effectively prevents surface passivation and minimizes the co-dissolution of phosphate phases [17, 18, 19].

As detailed in Table 3, a critical breakthrough of this study lies in successfully treating an exceptionally challenging, ultra-low initial feed grade (3.20% P₂O₅) derived from industrial flotation tailings—significantly lower than the feed grades (11.80–18.10% P₂O₅) typically investigated in previous studies [6, 7, 9, 14]. Despite this severe compositional limitation, the optimized citric acid process nearly tripled the P₂O₅ concentration to

9.16%, while achieving a reported P_2O_5 recovery of 88.50%. This result compares favorably with the organic-acid leaching studies listed in Table 3, subject to consistent calculation methods and experimental bases [6, 14, 17]. Furthermore, the relative decrease in MgO grade (84.26%, from 5.40% to 0.85%) indicates substantial MgO-grade reduction, consistent with the removal of Mg-bearing gangue minerals, particularly dolomite. Consequently, selective citric acid leaching represents a highly efficient and promising approach for upgrading low-grade industrial phosphate flotation tailings [14, 16, 17].

Conclusion

This study evaluated selective citric acid leaching for upgrading low-grade phosphate flotation tailings (Type III). Under the selected operating conditions (3% w/v citric acid, 50 °C, 40 min, a liquid-to-solid (L/S) ratio of 10 mL/g, and an agitation speed of 400 rpm), the P_2O_5 grade increased from 3.20% to 9.16%, corresponding to an enrichment factor of 2.86, with a reported overall P_2O_5 recovery of 88.50%. The results indicate that citric acid leaching can enrich phosphate in an ultra-low-grade secondary resource, although comparisons with conventional mineral acids and previous organic-acid leaching studies should be based on consistent performance metrics and operating conditions. The MgO grade decreased from 5.40% to 0.85%, representing an 84.26% relative decrease in grade. However, this grade reduction should not be interpreted as an 84.26% mass-based MgO removal efficiency unless it is confirmed by a solid mass balance. The resulting upgraded material should therefore be regarded as a pre-enriched intermediate rather than a directly qualified feedstock for wet-process phosphoric acid production. Further beneficiation, blending with higher-grade phosphate materials, and process-specific qualification tests may be required before industrial utilization. The practical feasibility of the process also depends on citric acid consumption, solution recycling, wastewater management, and overall operating costs, which warrant further investigation.

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