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OPTIMIZATION OF ACID DECOMPOSITION OF WASHED CALCINED PHOSPHOCONCENTRATE USING A MIXED SECONDARY SULFURIC–EXTRACTION PHOSPHORIC ACID SYSTEM: CHEMICAL AND FTIR INVESTIGATION

SHERMATOV AKRAMJON
PhD student, Namangan State Technical University, Namangan, Uzbekistan
Phone.: (0891) 180-8989, E-mail.: akramjon879@gmail.com
**Corresponding author*

SHERKUZIYEV DONIYOR
Professor, Namangan State Technical University, Namangan, Uzbekistan
Phone.: (0890) 223-3233, E-mail.: doniyor_8184@mail.ru

Abstract: This work examines the acid decomposition of washed calcined phosphoconcentrate using a mixed secondary sulfuric and extraction phosphoric acid feed (H_2SO_4:H_3PO_4 = 1:0.75) across a dosage range of 50–110 units. Products were characterized by total, citrate-soluble and water-soluble P_2O_5, free P_2O_5, total CaO and nitrogen content; structural changes were analyzed by FTIR spectroscopy. Increasing acid dosage enhanced conversion of mineral phosphorus into plant-available fractions and promoted ammonium incorporation after neutralisation, while reducing CaO content. FTIR results confirmed orthophosphate formation and ammonium-phosphate bonding, supporting the production of liquid complex fertilizer precursors. Optimal balance between available phosphorus and controlled free acidity was achieved at 85–95 units. The study provides process-relevant recommendations for up-scaling using secondary acid streams.

Keywords: Acid decomposition; Washed calcined phosphoconcentrate; Secondary sulfuric acid; Extraction phosphoric acid; Available phosphorus; Ammonium phosphate; FTIR; Process optimization.

Introduction. Phosphorus (P) is a critical nutrient for crop production; improving the agronomic availability of P from low-grade or secondary phosphate resources is a strategic objective in fertilizer engineering. Wet chemical acidulation — using sulfuric acid (H_2SO_4) or phosphoric acid (H_3PO_4), or mixtures thereof — is the core industrial route to obtain soluble P fractions from phosphate rock and concentrates (e.g., the wet-process phosphoric acid industry) and its kinetics and selectivity depend strongly on acid type, concentration and process hydrodynamics[1]. Mixed acid systems may improve dissolution kinetics and provide opportunities to valorize secondary acid streams, although the precipitation of gypsum ($CaSO_4 \cdot 2H_2O$) and the control of free acidity are persistent operational constraints. Prior work has examined phosphate dissolution in mixed H_2SO_4–H_3PO_4 systems and found optimal operating regimes that balance dissolution rate and by-product formation [2].

This study focuses on washed calcined phosphoconcentrate — a calcium-rich feedstock — treated with a mixed secondary sulfuric : extraction phosphoric acid (1:0.75) at varying dosages (50–110 units). The objectives are to: quantify P redistribution among total, citrate-soluble and water-soluble fractions; follow CaO depletion and N incorporation after ammoniation; use FTIR to resolve structural transformations; define an industrially viable acid dosage window [3].

Methodology & empirical analysis In the present study, washed and calcined phosphoconcentrate (26% P_2O_5 and 52% CaO) was used as the raw material. The sample was applied directly in the experiments without additional mechanical or chemical pretreatment. A 41% secondary sulfuric acid solution, wet-process extraction phosphoric acid, and a 25% aqueous ammonia solution were used as reagents.

Sulfuric acid and phosphoric acid were mixed in a mass ratio of 1:0.75 (H_2SO_4:H_3PO_4) to obtain a homogeneous acid mixture. The experiments were carried out using a batch method at constant room temperature. The acid dosage was varied stepwise within the range of 50–110 units according to the established process parameters.

In each experiment, the acid mixture was combined with the phosphoconcentrate, and the reaction medium was continuously stirred for 30 minutes. During this stage, phosphate minerals decomposed under the action of the acids, resulting in the formation of water-soluble phosphorus compounds and a calcium sulfate phase.

After completion of the reaction, the resulting mass was partially neutralized with 25% aqueous ammonia to adjust the pH to 4.0–4.5. The neutralization step was carried out to convert free phosphoric acid into ammonium phosphate species and to reduce the free acidity of the product. The free phosphorus content in the mixture was determined analytically after ammonia addition.

Subsequently, the entire reaction mass was dried at 80 °C for 48 hours without prior separation of solid and liquid phases. The drying process was conducted to reduce moisture content, ensure the formation of a stable solid product, and obtain a solid mineral fertilizer.

The chemical composition of the dried samples (total P_2O_5, water-soluble P_2O_5, available forms, residual CaO, and free acidity) was determined using standard analytical methods, and the process efficiency was evaluated.

Chemical composition: Total P_2O_5 by standard colorimetric digestion; citrate-soluble P_2O_5 (citrate extraction), water-soluble P_2O_5 (water extraction) were performed following recognized agronomic methods (specify standard methods used). Free P_2O_5 (free acidity) was determined by titration. CaO and N (Kjeldahl / elemental analyzer) were quantified by standard methods[5-8].

Results.

Table 1: Comprehensive chemical composition of products obtained at different acid dosages (H_2SO_4:H_3PO_4 = 1:0.75)

Acid Dosage (units)	Total P_2O_5 (%)	Citrate-soluble P_2O_5 (%)	Water-soluble P_2O_5 (%)	Free P_2O_5 (%)	Total CaO (%)	Citrate-soluble CaO (%)	Water-soluble CaO (%)	SO_3 (%)	N (%)
50	27.82	13.71	5.69	5.58	39.63	18.50	3.10	9.85	6.20
55	27.27	14.01	6.12	6.10	38.94	18.10	3.25	10.10	6.48
60	26.61	14.77	6.74	6.63	37.63	17.45	3.55	10.42	6.85
65	26.21	15.63	7.27	7.16	36.74	16.80	3.85	10.75	7.14
70	25.85	16.32	7.52	7.69	36.05	16.10	4.05	11.10	7.45
75	25.19	16.96	7.93	8.22	35.37	15.40	4.35	11.55	7.95
80	24.27	17.34	8.51	8.87	34.74	14.60	4.75	12.10	8.35

Acid Dosage (units)	Total P_2O_5 (%)	Citrate-soluble P_2O_5 (%)	Water-soluble P_2O_5 (%)	Free P_2O_5 (%)	Total CaO (%)	Citrate-soluble CaO (%)	Water-soluble CaO (%)	SO_3 (%)	N (%)
85	23.87	18.04	8.94	9.40	33.06	13.20	5.10	12.65	8.65
90	23.46	18.72	9.34	9.93	32.59	12.60	5.40	13.05	8.95
95	22.91	19.25	9.56	10.46	31.22	11.80	5.65	13.40	9.25
100	22.39	19.31	9.71	10.85	30.59	11.20	5.80	13.85	9.05
105	22.29	19.46	9.82	11.38	30.28	10.90	5.95	14.20	9.48
110	22.17	19.54	9.85	11.91	29.91	10.50	6.05	14.55	9.65

FTIR: Spectra were recorded in the 4000–400 cm^{-1} region using a Bruker FTIR spectrometer (transmission mode or ATR as applicable); peak positions were assigned to phosphate (PO_4^{3-}), hydroxyl/water bands, and ammonium features according to literature assignments. FTIR is a rapid, reliable technique for P speciation in solids and has been used for biochar, digestates and phosphate minerals [9-10].

Transformation of Phosphorus Fractions. Acid dosage had a clear and systematic influence on phosphorus redistribution within the system. As the dosage increased from 50 to 110 units, total P_2O_5 slightly decreased. This reduction does not indicate phosphorus loss; rather, it reflects the transformation of mineral-bound phosphorus into more soluble and reactive forms.

The most meaningful change was observed in citrate-soluble and water-soluble P_2O_5 fractions. Citrate-soluble P_2O_5 increased steadily, with the sharpest growth between 60 and 90 units. This interval corresponds to the most active stage of mineral decomposition. In practical terms, this means that within this range the acid system effectively breaks down calcium phosphate structures and converts them into plant-available forms.

Water-soluble P_2O_5 followed a similar upward trend. The gradual increase indicates that the product gains faster nutrient release capability, which is particularly important for liquid or rapidly available fertilizers.

Free P_2O_5, on the other hand, increased almost linearly across the entire dosage range. While this confirms stronger acid action and deeper mineral conversion, it also highlights a technological limitation: excessive dosage increases free acidity without proportionally increasing agronomic efficiency. After approximately 100 units, the benefit in available phosphorus becomes marginal compared to the rise in free acidity.

Calcium Redistribution and Sulfate Formation. The behavior of calcium provides additional insight into the decomposition mechanism. Total CaO content consistently decreased as acid dosage increased. This confirms that calcium phosphate minerals were progressively dissolved.

In parallel, the increase in soluble calcium fractions and SO_3 content suggests the formation of calcium sulfate species. At moderate dosages (around 80–95 units), calcium breakdown was most intensive. This indicates that the system reached a chemically active regime where proton attack on Ca–O–P bonds was highly effective.

However, at higher dosages, although calcium dissolution continued, the rate slowed. This may be related to surface passivation by sulfate phases or reduced availability of reactive mineral surfaces — a common phenomenon in acid digestion systems.

From a process engineering perspective, this behavior is important. It suggests that there is a practical limit beyond which additional acid no longer improves mineral conversion efficiency.

Nitrogen Incorporation and Formation of Ammonium Phosphate Species. Nitrogen content increased progressively after ammoniation, confirming the formation of ammonium-containing species. The presence of NH_4^+ functional groups in the FTIR spectrum supports this conclusion. This step is critical for transforming the product into a complex fertilizer rather than a simple acid-treated phosphate slurry. The gradual rise in nitrogen content demonstrates that neutralization was effective and that phosphorus was successfully stabilized in ammonium phosphate forms.

Interestingly, slight fluctuations in nitrogen content at higher dosages may indicate equilibrium competition between free acid and ammonium ions. In practical production conditions, this balance must be carefully controlled to avoid excess free acidity.

FTIR Evidence of Structural Transformation FTIR spectroscopy provided structural confirmation of the chemical trends observed in the compositional data.

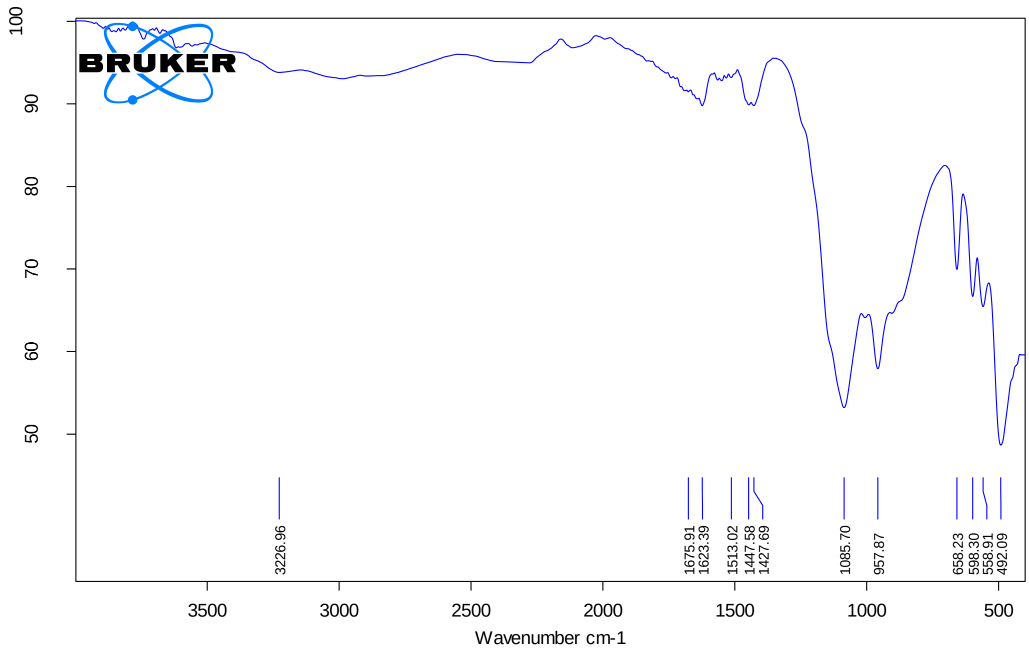

Figure 1. FTIR spectrum of product obtained after treatment of washed calcined phosphoconcentrate with H_2SO_4:H_3PO_4 (1:0.75) and subsequent partial ammoniation. Key bands are annotated (see Table 2 for peak assignments)

The strong band near 1085 cm^{-1} confirmed the dominance of orthophosphate groups in the final product. Bands in the 950–960 cm^{-1} region indicated the presence of partially converted calcium phosphate species. The appearance of N–H bending vibrations in the 1515–1425 cm^{-1} range clearly demonstrated ammonium incorporation after neutralization.

The broad O–H stretching band near 3200–3300 cm^{-1} revealed hydrated phases, which are typical for acid-treated phosphate systems. Importantly, the absence of strong apatite-specific signatures suggests that most of the original mineral structure was transformed within the optimal dosage range. Taken together, FTIR results validate the chemical analysis and confirm that the mixed acid system successfully restructures the mineral matrix into more soluble phosphate forms.

Process Optimization and Industrial Implication. From an engineering standpoint, the objective of the process is not merely to achieve maximum mineral dissolution, but to ensure optimal efficiency in producing a high-quality solid fertilizer. At low acid dosages (50–70 units), the decomposition of phosphate minerals remains incomplete. As a result, the content of available phosphorus does not increase sufficiently, and a relatively high proportion of undecomposed calcium compounds remains in the product. Although acid consumption is economically lower in this range, the agrochemical quality of the resulting solid fertilizer is not optimal.

Within the 80–95 unit range, the process enters a highly efficient regime. In this interval, phosphate mineral decomposition is significantly enhanced, leading to increased levels of water-soluble and plant-available P_2O_5. The transformation of calcium compounds proceeds effectively, and the ammoniation stage remains stable, with free acidity maintained at a controllable level. After drying, the product forms a stable solid phase with favorable physical properties. This range represents a technologically and economically balanced operating window.

At acid dosages above 100 units, free acidity increases sharply, while the additional gain in plant-available phosphorus becomes marginal. Under such conditions, excess acid may increase the hygroscopicity of the product and negatively affect storage stability. From both economic and safety perspectives, the additional acid input cannot be justified.

Therefore, the 85–95 unit interval can be considered the most rational industrial operating range for the production of solid mineral fertilizer. Furthermore, the use of secondary sulfuric acid enhances resource efficiency and promotes waste utilization. With proper control of acid feed rate, pH, and residence time in a continuous mixing system, the process shows strong potential for industrial-scale production of solid phosphate fertilizers.

Conclusions. The present study demonstrates that the mixed secondary sulfuric–extraction phosphoric acid system is an effective and technically viable approach for converting washed calcined phosphoconcentrate into a highly reactive fertilizer intermediate.

Systematic variation of acid dosage revealed a clear transformation pattern in the mineral matrix. As the dosage increased, calcium phosphate structures progressively decomposed, resulting in a significant rise in citrate- and water-soluble phosphorus fractions. This confirms that the acid system successfully disrupts Ca–O–P bonds and redistributes phosphorus into more plant-available forms.

From a process engineering perspective, the 85–95 unit acid dosage interval represents the most rational operational window. Within this range: phosphorus availability reaches near-maximum levels, calcium decomposition is highly effective, free acidity remains controllable, nitrogen incorporation is stable, additional acid consumption does not significantly improve product performance.

This balance is critical for industrial scalability, where economic efficiency, safety, and product stability are as important as chemical conversion. Furthermore, the utilization of secondary sulfuric acid enhances resource efficiency and supports sustainable fertilizer production strategies. The integration of such mixed-acid systems into continuous reactor configurations has strong potential for industrial implementation.

In summary, the study confirms that controlled mixed-acid treatment combined with optimized ammoniation can serve as a robust pathway for producing high-efficiency liquid complex fertilizers from calcined phosphate resources. Future work should focus on kinetic modeling, reactor-scale validation, and long-term stability assessment of the produced formulations.

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