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Retrospective Bench-Scale Screening of Six Phosphate Concentrates for Wet-Process Phosphoric Acid Production

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ABSTRACT: Variability in phosphate-concentrate chemistry can affect sulfuric acid demand, phosphorus recovery, calcium sulfate formation, and solid–liquid separation during wet-process phosphoric acid (WPA) production. This study presents a retrospective, quality-controlled reanalysis of a historical bench-scale campaign comprising six coded phosphate concentrates (PQH-001 to PQH-006). Each concentrate was represented by one complete WPA bench test; accordingly, comparisons are descriptive rather than inferential. Feed P_2O_5 ranged from 27.59 to 30.55 wt%, whereas the minor-elements ratio (MER = 100 × (Al_2O_3 + Fe_2O_3 + MgO)/P_2O_5) ranged from 7.12 to 16.83%. The surviving records documented 98 wt% sulfuric acid, phosphoric acid, and gypsum streams, filtration calculations, and duplicate hydrochloric acid attack tests, but did not preserve several operating variables required for mechanistic attribution. Observed P_2O_5 recovery ranged from 94.68 to 97.99%. The highest-grade feed, PQH-004 (30.55 wt% P_2O_5), showed the lowest recovery (94.68%) and the highest residual P_2O_5 in dry gypsum (1.083 wt%), demonstrating that feed grade alone did not rank performance in this campaign. PQH-005 showed the most favorable observed combination of metrics: 97.99% recovery, 0.405 wt% P_2O_5 in dry gypsum, a filtration index of 185.3 kg h^{-1} m^{-2}, and sulfuric-acid use of 2.603 t H_2SO_4/t feed P_2O_5, approximately 1% above the CaO-only stoichiometric benchmark. A stoichiometric QA/QC audit identified the reported PQH-003 acid addition as chemically inconsistent, and its filtration result was also non-defensible because a required dry-cake term was missing. The results support a staged, multi-parameter qualification approach based on feed chemistry, stoichiometric consistency, phosphorus deportment, and filtration behavior, followed by replicated modern bench and pilot validation before industrial scale-up.

KEYWORDS: phosphate concentrate; wet-process phosphoric acid; phosphogypsum; phosphorus recovery; sulfuric acid demand; filtration

HIGHLIGHTS

- P_2O_5 grade alone did not rank bench-scale WPA performance across the six concentrates.
- The reanalysis explicitly separates CaO-only stoichiometric demand from recorded sulfuric-acid addition.
- PQH-005 showed the most favorable observed combination of recovery, gypsum P_2O_5, filtration, and acid use.
- QA/QC screening prevented interpretation of internally inconsistent PQH-003 acid and filtration data.

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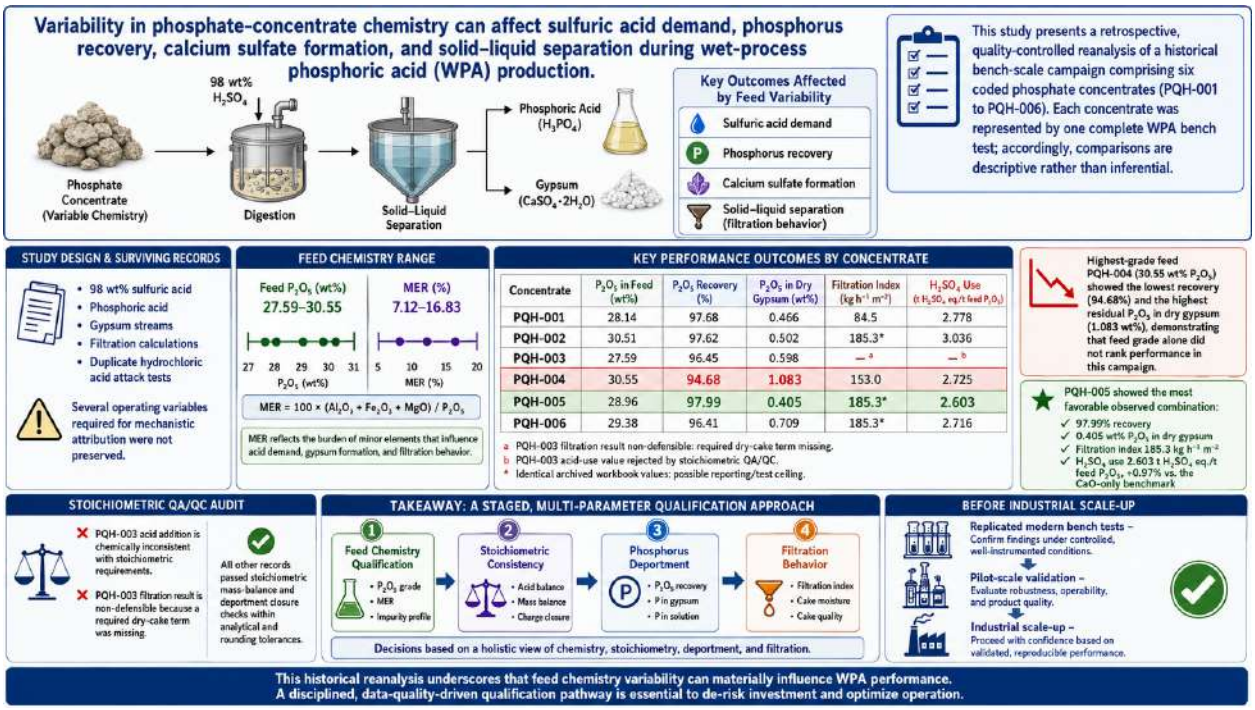
GRAPHICAL ABSTRACT



Source. *Prepared by the authors from the historical research dataset and the qualification framework developed in this study.*

1. INTRODUCTION

Wet-process phosphoric acid (WPA) is the dominant industrial route for converting phosphate resources into phosphoric acid for fertilizer manufacture and downstream phosphorus chemistry. In sulfuric-acid processes, phosphate minerals dissolve while calcium is precipitated as calcium sulfate hydrate, after which the phosphoric-acid product is separated from the gypsum-rich solid phase by filtration. Industrial performance is therefore governed by a coupled reaction–crystallization–filtration system rather than by phosphate grade alone (Becker, 1989; Peng et al., 2015).

Raw-material qualification has long recognized that phosphate rocks with similar P_2O_5 contents can behave differently during acidulation. CaO and the CaO/P_2O_5 ratio establish a first-order sulfuric-acid burden, whereas carbonate minerals, Mg-, Fe-, and Al-bearing phases, silica, fluorine, chloride, organic matter, particle-size distribution, and mineral associations can influence acid consumption, slurry rheology, supersaturation, gypsum nucleation and growth, corrosion, and filtration (Al-Fariss et al., 1992; Ryszko et al., 2023; Ding et al., 2025). The minor-elements ratio (MER), commonly expressed as the combined Al_2O_3, Fe_2O_3, and MgO content relative to P_2O_5, is therefore useful as a compact impurity indicator, but it cannot represent mineralogical occurrence or all interactive effects.

The link between dissolved impurities and calcium-sulfate crystallization is particularly important because crystal morphology and size distribution directly affect cake permeability and washing. Experimental studies have shown concentration-dependent effects of ionic impurities on gypsum crystallization (Kruger et al., 2001) and have demonstrated that Si, Al, Fe, Mg, Na, and K can modify $CaSO_4$ growth and habit in phosphoric-acid media (Mu et al., 2019). More recent design-of-experiments work identified carbonate-related chemistry, F^-, Al_2O_3, and SiO_2 as important variables affecting reactive crystallization and filterability (Abdelouahhab et al., 2022). Studies of hemihydrate and hemihydrate-dihydrate systems further show that operating conditions and crystal-habit modifiers can substantially affect filtration performance and phosphorus loss (Huo et al., 2023a, 2023b).

Residual phosphorus in phosphogypsum represents both a yield penalty and a constraint on by-product utilization. Phosphorus may occur as unreacted phosphate, water-soluble species, surface-associated material, secondary precipitates, inclusions, or co-crystalline forms, and its distribution reflects coupled dissolution, precipitation, and calcium-sulfate crystallization (Dong et al., 2024; Zhao et al., 2025; Deng et al., 2026). Process intensification and source control studies increasingly seek to reduce residual P_2O_5 while improving crystal morphology and separation performance (Fang et al., 2023; Zhu et al., 2023; Shan et al., 2026; Pereira,

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2026). These developments reinforce the value of screening phosphate feeds with recovery, gypsum chemistry, and filtration metrics together.

Fluorine and chloride add further constraints. Fluorine can partition among acid, solids, and gas during WPA production and concentration, creating recovery and emission-control requirements (Peng et al., 2022). Chloride is important because WPA plants operate in highly corrosive phosphoric- and sulfuric-acid environments, and elevated chloride levels increase materials-of-construction concerns (Ryszko et al., 2023). Meanwhile, phosphogypsum valorization remains sensitive to residual phosphate, fluoride, acidity, trace contaminants, and the solid's physical properties (Tayibi et al., 2009; Li et al., 2023; Guan et al., 2024).

The present study reanalyzes a historical six-concentrate bench campaign as a quality-controlled retrospective screening dataset. The contribution is not a new mechanistic model and does not claim population-level ranking from six unreplicated tests. Instead, it evaluates how much technically defensible information can be recovered when historical industrial-style data are subjected to explicit stoichiometric reconciliation, missing-data control, and integrated interpretation. The objectives were to (i) compare feed chemistry and impurity indices; (ii) quantify observed P_2O_5 recovery, gypsum phosphorus loss, sulfuric-acid use, and filtration behavior; (iii) make the QA/QC logic transparent, particularly for inconsistent acid and filtration fields; and (iv) formulate a staged concentrate-qualification framework that defines what should be verified in replicated modern bench and pilot programs.

2. MATERIALS AND METHODS

2.1. Dataset provenance and experimental design

The study used the surviving research workbook from a bench-scale phosphoric-acid evaluation campaign dated 1 April 2023. Six final phosphate concentrates were available for complete comparative assessment: PQH-001, PQH-002, PQH-003, PQH-004, PQH-005, and PQH-006. The workbook contains sample-specific material-balance entries, chemical analyses, acid additions, gypsum and acid streams, filtration calculations, granulometric records, and duplicate hydrochloric-acid attack tests. No new laboratory experiments were performed for this manuscript.

Analyses were performed by different laboratories. The archived workbook distinguishes between a primary analytical dataset and additional results obtained from a separate laboratory. Because the available records do not provide sufficiently consistent information to unambiguously identify the laboratories, they are referred to in this study simply as the primary and secondary laboratories. Secondary-laboratory results were used only for cross-laboratory QA/QC and were not substituted for the primary dataset, except for the explicitly missing SiO_2 value for PQH-006.

Mine, beneficiation-circuit, geological, and commercial provenance were not retained in the supplied dataset. Consequently, the six concentrates must be interpreted as a historical comparative dataset rather than a statistically representative sample of global phosphate-rock types.

2.2. Bench-scale wet-process test and documentation limits

Each concentrate was processed in a bench-scale phosphoric-acid flowsheet using sulfuric acid nominally 98 wt%, phosphoric acid solution, and deionized water. Concentrate charges were approximately 300 g per test (317.23 g for PQH-005 and 304.50 g for PQH-006). Acidulation generated a phosphoric-acid liquor and a gypsum-rich cake. The recorded filtration unit had a diameter of 190 mm, corresponding to an area of 0.02835 m^2.

The surviving workbook does not document reactor temperature, agitation rate, residence time, free-sulfate control, recycle-acid composition, vacuum or pressure used during filtration, washing protocol, or filtration endpoint. It also does not preserve an experimental log proving that all of those variables were identical across the six tests. These missing details are not reconstructed or assumed. The campaign is therefore interpreted as comparative historical screening under the recorded bench protocol, not as a fully reproducible mechanistic experiment.

The workbook does not contain X-ray diffraction confirmation of the calcium-sulfate hydration state, feed mineralogical phase composition, automated mineralogy, or gypsum-crystal morphology. Accordingly, the experimental solid is described operationally as gypsum or phosphogypsum without assigning an unverified dihydrate or hemihydrate identity.

A hydrochloric acid attack test was recorded separately for each feed, with two determinations (A and B) used by the original workbook to calculate the average extraction degree. The archived file does not preserve the HCl concentration, liquid-to-solid ratio, temperature, duration, agitation, or analytical endpoint required for independent reproduction. The HCl result is therefore retained only as a supplementary historical reactivity indicator and is not treated as a surrogate for the sulfuric-acid WPA test.

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2.3. Calculated indices and performance metrics

Feed composition was expressed in wt% for major oxides and fluorine and in mg kg^{-1} for chloride. The MER was recalculated for all samples according to Eq. (1), following common WPA raw-material practice (Ryszko et al., 2023):

$$MER\ (\%) = 100 \times (Al_2O_3 + Fe_2O_3 + MgO) / P_2O_5 \quad (1)$$

The CaO/P_2O_5 mass ratio was taken from the workbook. A CaO-only stoichiometric sulfuric-acid benchmark was calculated by assuming one mole of H_2SO_4 per mole of CaO converted to calcium sulfate (Eq. (2)). This is deliberately termed a benchmark rather than a complete theoretical acid demand because other acid-consuming phases may be present.

$$CaO\text{-only } H_2SO_4/P_2O_5 = (CaO/P_2O_5) \times (98.079 / 56.077) \quad (2)$$

The archived acid-use values are internally consistent with a basis of mass of 100% H_2SO_4 equivalent added per mass of P_2O_5 contained in the concentrate charge. That basis was therefore used for reconciliation and is made explicit in Eq. (3):

$$Actual\ H_2SO_4/P_2O_5 = m(H_2SO_4,\ 100\%\ equivalent) / m(P_2O_5\ in\ concentrate\ feed) \quad (3)$$

The deviation of recorded acid addition from the CaO-only benchmark was calculated using Eq. (4). Because this quantity may include reaction with carbonates and other gangue phases, differences in free-sulfate targets, and other operating effects, it is referred to as acid addition above the CaO-only stoichiometric requirement rather than simply 'acid excess'.

$$Acid\ above\ CaO\text{-only stoichiometry}\ (\%) = 100 \times [(actual\ ratio / CaO\text{-only ratio}) - 1] \quad (4)$$

The workbook rock-use values were independently reconciled as concentrate mass required per mass of recovered P_2O_5 (Eq. (5)), which reproduces the tabulated values to rounding:

$$Rock\ use = 1 / [(feed\ P_2O_5\ mass\ fraction) \times (observed\ P_2O_5\ recovery\ fraction)] \quad (5)$$

The filtration index has dimensions of dry solids throughput per unit filter area. From the archived formula structure and the missing dry-cake term that invalidated PQH-003, it is consistent with Eq. (6):

$$Filtration\ index = m(dry\ cake) / [A(filter) \times t(filtration)] \quad (6)$$

Because the original filtration pressure/vacuum, wash protocol, cake thickness, and endpoint are unavailable, the index is used strictly as an internal throughput proxy within this campaign and should not be compared quantitatively with other filtration rigs without method harmonization.

2.4. Data-quality audit and treatment of missing values

Before sample comparison, analytical and process entries were reviewed using three criteria: stoichiometric plausibility, internal material-balance consistency, and agreement between duplicated or cross-laboratory values. The central numerical QA/QC check compared the recorded H_2SO_4/P_2O_5 ratio with the minimum CaO-only requirement calculated from feed chemistry. The resulting audit is reported explicitly in Table 3 rather than leaving the exclusion logic implicit.

For PQH-003, the workbook-derived H_2SO_4/P_2O_5 value of 1.459 was 43.1% below the CaO-only requirement of 2.566. The archived balance also indicates incompatibility between that acid entry and sulfate assigned to the gypsum stream. The reported acid-use result was therefore rejected as chemically non-defensible. The archived entries establish the inconsistency but do not provide sufficient independent stream-level information to report a defensible sulfate-closure percentage; therefore, no unsupported closure value is introduced.

The PQH-003 filtration calculation depended on an unavailable dry-cake term and therefore collapsed to zero or an invalid formula in the source workbook. That filtration result was treated as missing, not as zero performance. For PQH-006, the primary SiO_2 entry was blank/zero, whereas a secondary laboratory value of 8.22 wt% was recorded. The secondary value was used only to avoid propagating an obvious missing-value artifact into descriptive chemistry. All other primary composition values were retained.

For the four samples with paired P_2O_5 analyses, the mean primary-minus-secondary bias was +0.05 wt%, and the mean absolute difference was 0.66 wt%. These values support use of the primary dataset for comparative screening while also showing that specification-level decisions would require harmonized analytical protocols.

2.5. Statistical approach and scope of inference

The campaign contains one complete WPA process test per concentrate and therefore provides no independent within-concentrate process variance. Results are summarized using observed ranges, rankings, stoichiometric comparisons, and graphical patterns. No null-hypothesis tests, regression models, confidence intervals, or causal correlation claims are applied. Terms such as 'higher', 'lower', or 'most favorable' refer only to the observed values in this historical campaign and do not imply statistically established superiority.

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3. RESULTS

3.1. Feed chemistry and impurity burden

The six concentrates covered a relatively narrow P_2O_5 range (27.59–30.55 wt%) but a much wider range of impurity burden (Table 1). PQH-002 and PQH-006 had the lowest MER values, 7.12% and 8.35%, respectively, whereas PQH-003 had the highest MER at 16.83%. PQH-003 also contained the highest Fe_2O_3 (1.18 wt%), Al_2O_3 (2.88 wt%), SiO_2 (16.17 wt%), and chloride (852 mg kg^{-1}) of the six feeds. PQH-002 combined the highest CaO content (46.30 wt%) with relatively low Fe_2O_3 and Al_2O_3.

Table 1. Chemical composition and derived impurity index of the phosphate concentrates.

Sample	P_2O_5 wt%	CaO wt%	Fe_2O_3 wt%	Al_2O_3 wt%	MgO wt%	SiO_2 wt%	F wt%	Cl mg kg^{-1}	MER %
PQH-001	28.14	41.71	0.74	2.15	0.58	10.74	2.70	327	12.35
PQH-002	30.51	46.30	0.45	1.20	0.52	5.23	3.05	202	7.12
PQH-003	27.59	40.47	1.18	2.88	0.58	16.17	2.36	852	16.83
PQH-004	30.55	45.09	0.69	2.33	0.56	5.77	2.91	268	11.72
PQH-005	28.96	42.70	0.73	1.96	0.55	10.24	2.60	369	11.20
PQH-006	29.38	44.86	0.64	1.27	0.54	8.22	2.40	235	8.35

Note. MER = 100 × (Al_2O_3 + Fe_2O_3 + MgO)/P_2O_5. PQH-006 SiO_2 is the secondary-laboratory value because the primary field value was missing. Only reported major components are shown; totals are not intended to sum to 100 wt%. Fluorine is reported as elemental F. *Source. Prepared by the authors from the surviving historical research workbook; MER values were recalculated according to Eq. (1).*

The CaO/P_2O_5 ratios ranged from 1.467 to 1.527. This variation is process-relevant because it changes the first-order sulfuric-acid requirement independently of MER. A concentrate can therefore have relatively favorable Fe–Al–Mg chemistry yet still carry a substantial calcium-related acid burden. Figure 1 summarizes the recalculated MER values for all six concentrates.

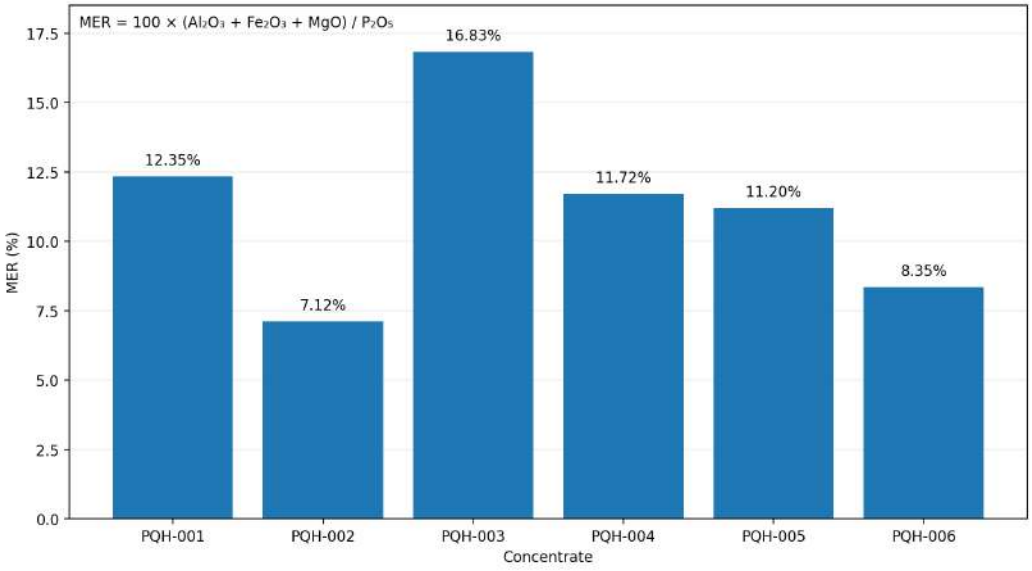

Figure 1. Minor-elements ratio (MER) calculated from feed chemistry for the six concentrates. Lower values indicate a lower combined Fe–Al–Mg burden relative to P_2O_5; MER does not capture mineralogical occurrence or all other impurities.

Source. *Prepared by the authors from the research data reported in Table 1.*

3.2. P_2O_5 recovery and supplementary HCl reactivity

Observed P_2O_5 recovery from the full WPA bench tests ranged from 94.68% to 97.99%, whereas the archived HCl attack extraction ranged from 93.64% to 98.59% (Table 2). The highest observed WPA recovery was PQH-005 (97.99%), followed by

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PQH-001 (97.68%) and PQH-002 (97.62%). PQH-004 showed the lowest recovery (94.68%) despite having the highest feed grade (30.55 wt% P_2O_5). PQH-003 showed the highest HCl extraction (98.59%) but only an intermediate WPA recovery (96.45%), confirming that the historical HCl screen did not reproduce the complete sulfuric acid/crystallization response.

Table 2. Bench-scale wet-process performance metrics after data-quality review.

Sample	HCl attack extraction (%)	Observed P_2O_5 recovery (%)	CaO/P_2O_5 ratio (t/t)	Rock use (t/t recovered P_2O_5)	H_2SO_4 use (t eq./t feed P_2O_5)	Deviation from CaO-only benchmark (%)	Filtration index (kg h^{-1} m^{-2})	P_2O_5 in dry gypsum (wt%)
PQH-001	96.64	97.68	1.482	3.638	2.778	7.17	84.5	0.466
PQH-002	93.64	97.62	1.518	3.358	3.036	14.35	185.3*	0.502
PQH-003	98.59	96.45	1.467	3.758	—	—	—	0.598
PQH-004	94.00	94.68	1.476	3.457	2.725	5.56	153.0	1.083
PQH-005	97.06	97.99	1.474	3.524	2.603	0.97	185.3*	0.405
PQH-006	95.58	96.41	1.527	3.530	2.716	1.69	185.3*	0.709

Note. H_2SO_4 use is expressed as 100% acid equivalent per mass of P_2O_5 contained in the concentrate feed. 'Acid above CaO-only stoichiometry' is the deviation from Eq. (2), not a complete thermodynamic acid-efficiency measure. PQH-003 H_2SO_4 use and filtration were excluded by QA/QC. *The three identical 185.3 values may indicate a workbook reporting/test ceiling; this cannot be confirmed from the surviving documentation and the values are therefore retained but not interpreted as precise equality. The recorded PQH-003 acid ratio is shown in Table 3 solely for QA/QC diagnosis; it is excluded from the comparative performance dataset in Table 2. *Source. Prepared by the authors from the surviving historical research workbook and the present QA/QC reanalysis.*

Figure 2 illustrates the absence of a simple monotonic relation between feed P_2O_5 grade and observed recovery. The two highest-grade feeds, PQH-004 and PQH-002, occupy markedly different recovery positions, while the highest observed recovery occurred for PQH-005 at an intermediate grade. The plot is descriptive only; no regression is fitted because n = 6 and each point represents one process test.

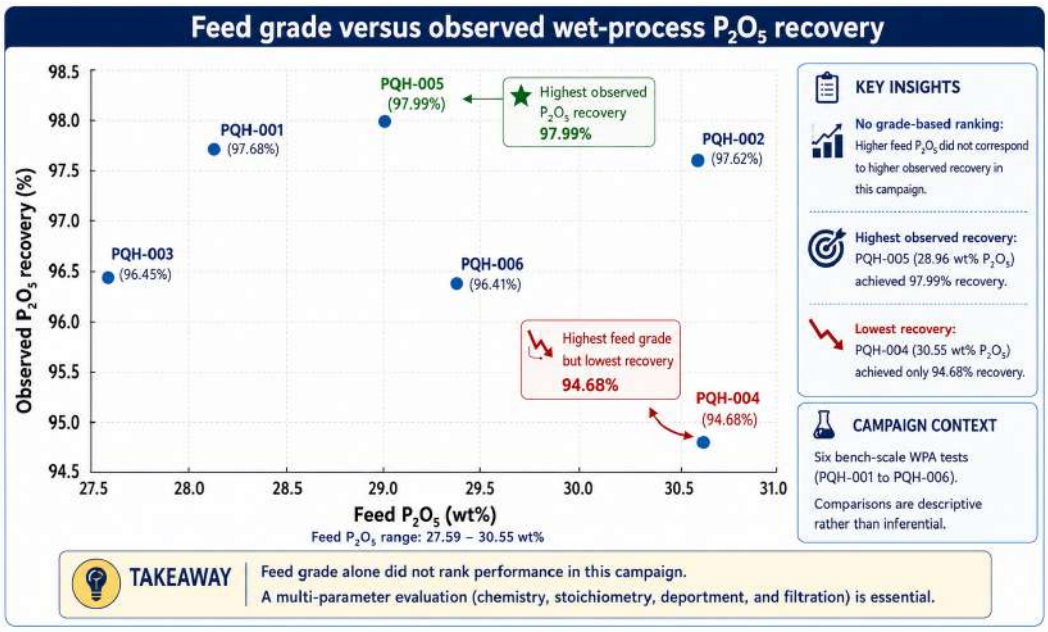

Figure 2. Feed P_2O_5 grade versus observed P_2O_5 recovery. Each point represents one historical process test; no inferential fit is shown.

Source. *Prepared by the authors from the research data reported in Table 2.*

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3.3. Sulfuric-acid demand and QA/QC audit

Among the five samples with valid acid-use entries, recorded consumption ranged from 2.603 to 3.036 t H_2SO_4/t feed P_2O_5. The CaO-only benchmark distinguishes the acid burden associated with calcium from additional recorded acid above that baseline. PQH-005 was closest to the CaO-only benchmark (+0.97%), followed by PQH-006 (+1.69%), PQH-004 (+5.56%), and PQH-001 (+7.17%). PQH-002 had the largest positive deviation (+14.35%) and the highest recorded acid use despite its low MER and high observed P_2O_5 recovery. The complete stoichiometric and filtration QA/QC audit is summarized in Table 3.

Table 3. Stoichiometric QA/QC audit of sulfuric acid and filtration entries.

Sample	CaO/P_2O_5 ratio (t/t)	CaO-only H_2SO_4/P_2O_5 (t/t)	Recorded H_2SO_4/P_2O_5 (t/t)	Deviation from CaO-only benchmark (%)	Acid-data status	Filtration status
PQH-001	1.482	2.592	2.778	+7.17	Valid	84.5; valid
PQH-002	1.518	2.655	3.036	+14.35	Valid	185.3; valid, repeated high value
PQH-003	1.467	2.566	1.459	−43.14	Rejected: below minimum CaO-only requirement	Missing dry-cake term; rejected
PQH-004	1.476	2.582	2.725	+5.56	Valid	153.0; valid
PQH-005	1.474	2.578	2.603	+0.97	Valid	185.3; valid, repeated high value
PQH-006	1.527	2.671	2.716	+1.69	Valid	185.3; valid, repeated high value

Note. The recorded PQH-003 acid ratio is shown solely for QA/QC diagnosis and is excluded from the comparative performance dataset in Table 2. The PQH-003 filtration result was rejected because the required dry-cake term was missing.

Source. *Prepared by the authors from the historical research workbook and the stoichiometric QA/QC calculations described in Eqs. (2)–(4).*

Table 3 provides a quantitative reason for rejecting PQH-003: the recorded acid ratio is approximately 43% below the amount required to react stoichiometrically with the measured CaO alone. Treating that value as evidence of exceptional acid efficiency would therefore be chemically misleading. By contrast, positive deviations in the other samples are retained as observed process descriptors; they should not be attributed to a single mechanism, as carbonate, Mg/Fe/Al-bearing phases, free-sulfate targets, and operating conditions were not fully documented.

3.4. Gypsum phosphorus loss and filtration

Residual P_2O_5 in dry gypsum ranged from 0.405 to 1.083 wt%. PQH-005 had the lowest observed residual phosphorus (0.405 wt%), while PQH-004 had the highest (1.083 wt%) and also the lowest observed WPA recovery. Recovery and gypsum P_2O_5 are not statistically independent because both arise from the same phosphorus balance, but the gypsum analysis is operationally useful because it localizes a major yield-loss pathway to the solid by-product. These solid-phase phosphorus losses are compared in Figure 3.

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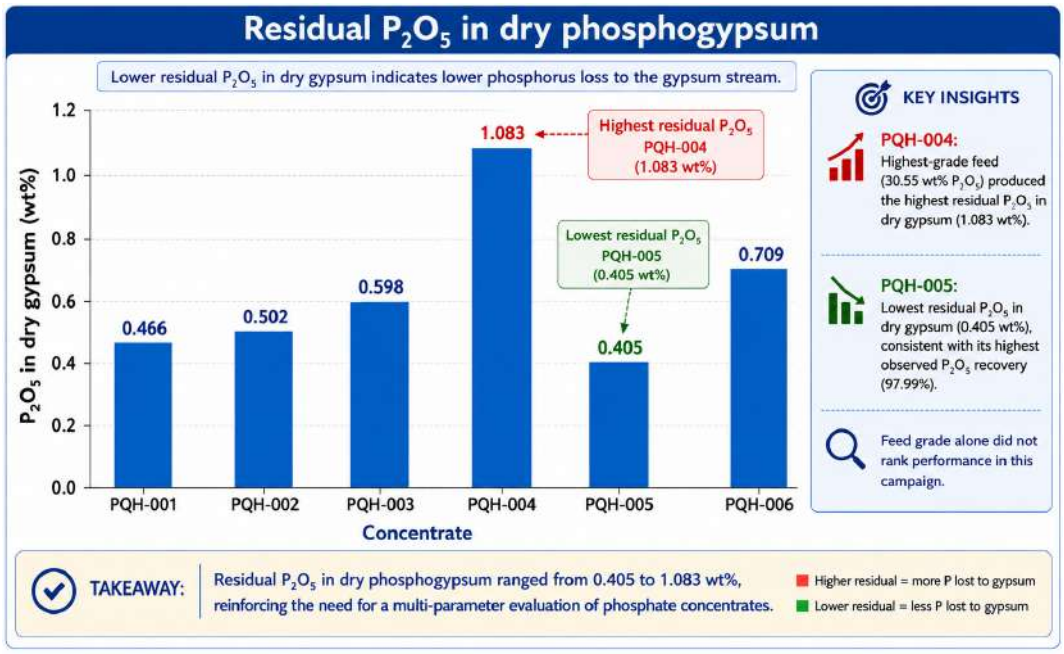

Figure 3. Residual P_2O_5 concentration in dry phosphogypsum from the six historical bench tests.

Source. *Prepared by the authors from the research data reported in Table 2.*

Figure 4 compares the filtration indices for the five samples with valid dry-cake data. Valid filtration indices ranged from 84.5 to 185.3 kg h^{-1} m^{-2}. PQH-001 had the lowest index despite 97.68% observed P_2O_5 recovery, illustrating that favorable chemical recovery can coexist with unfavorable solid–liquid separation. PQH-004 gave 153.0 kg h^{-1} m^{-2}, whereas PQH-002, PQH-005, and PQH-006 each carried the identical workbook value of 185.3 kg h^{-1} m^{-2}. Because the repeated high values may reflect a reporting or method ceiling, they are treated as a high-performance class within this dataset rather than evidence of precise equality.

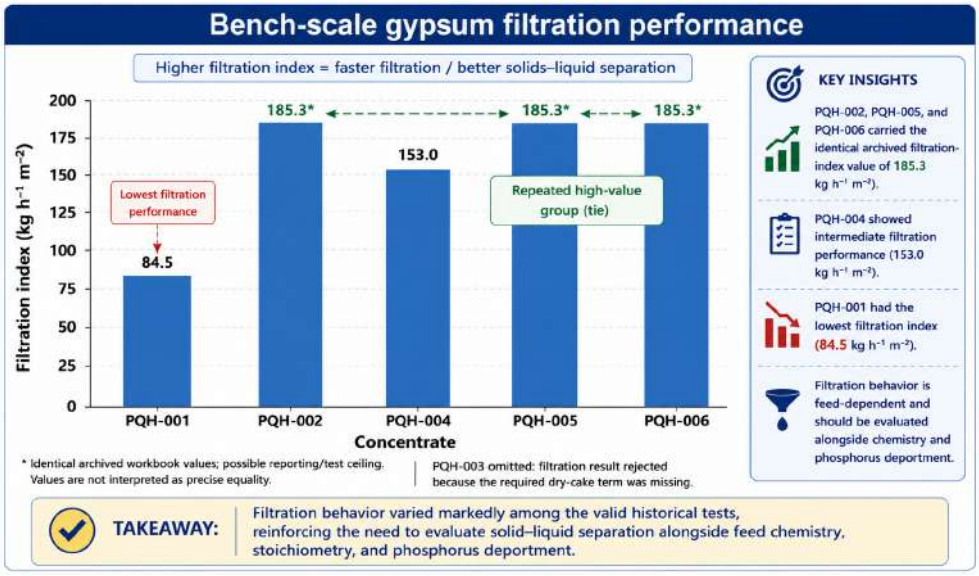

Figure 4. Filtration index for samples with valid dry-cake data. PQH-003 is omitted. Asterisks identify the three identical 185.3 kg h^{-1} m^{-2} workbook values, which may reflect a reporting/test ceiling that cannot be confirmed from the archived method documentation.

Source. *Prepared by the authors from the research data reported in Table 2 after QA/QC exclusion of the invalid PQH-003 filtration result.*

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3.5. Cross-laboratory check and integrated observed performance

For the four feeds with duplicate laboratory characterization, P_2O_5 showed good overall agreement: the mean primary-minus-secondary bias was +0.05 wt%, the mean absolute difference was 0.66 wt%, and the maximum absolute difference was 0.81 wt%. CaO showed a larger systematic difference, with primary values averaging 1.45 wt% lower than the secondary results. Fe_2O_3 and Al_2O_3 showed positive primary-laboratory biases of approximately 0.16 and 0.44 wt%, respectively. These discrepancies matter for compliance with absolute specification requirements and reinforce the need for standardized analytical protocols in purchasing or blending programs.

Within the six historical tests, PQH-005 showed the most favorable observed combination of metrics: the highest recovery, the lowest P_2O_5 in dry gypsum, a filtration index in the repeated high-value group, and recorded H_2SO_4 use close to the CaO-only benchmark. PQH-002 also combined high observed recovery, low MER, and a high filtration index, but its recorded acid addition was 14.35% above the CaO-only benchmark. PQH-001 combined high recovery with the lowest filtration index. PQH-006 had a low MER and a high filtration index, but a somewhat lower recovery. PQH-004 illustrates the risk of grade-only selection. PQH-003 cannot be fully compared because two operational metrics failed QA/QC. These descriptions are campaign-specific observations, not statistical rankings of intrinsic concentrate quality.

4. DISCUSSION

4.1. Why feed P_2O_5 grade alone was insufficient

The principal empirical pattern is that feed P_2O_5 grade did not rank the observed WPA responses over the tested range. This is consistent with industrial raw-material assessment, where grade determines how much phosphate is carried into the plant but does not specify the acid burden of calcium- and carbonate-bearing phases, impurity dissolution, slurry behavior, gypsum supersaturation, or phosphorus partitioning (Becker, 1989; Al-Fariss et al., 1992; Ryszko et al., 2023).

PQH-004 is the clearest example: 30.55 wt% P_2O_5 would appear attractive on a grade basis, yet this single bench test showed 94.68% recovery and 1.083 wt% P_2O_5 in dry gypsum. PQH-005 contained 28.96 wt% P_2O_5 but showed 97.99% recovery and 0.405 wt% P_2O_5 in dry gypsum. Current mechanistic literature identifies several possible pathways for phosphorus retention, including incomplete decomposition, adsorption, inclusion, secondary phosphate precipitation, and co-crystallization with calcium sulfate (Dong et al., 2024; Zhao et al., 2025; Deng et al., 2026). Those mechanisms were not measured in the present samples; they are cited only as plausible explanations for why bulk grade alone cannot determine recovery.

4.2. Impurity burden and the limits of bulk chemistry

MER was useful as a compact Fe–Al–Mg indicator but did not uniquely rank observed process performance. PQH-002 had the lowest MER and high recovery, PQH-006 had the second-lowest MER but lower recovery, and PQH-005 had a moderate MER yet the most favorable observed integrated profile. This is expected because MER collapses three oxide concentrations into one number and omits Si, F, chloride, carbonate-related chemistry, particle characteristics, and mineral associations. Experimental studies show that impurity effects on calcium-sulfate crystallization are concentration-dependent and interactive rather than strictly monotonic (Kruger et al., 2001; Mu et al., 2019; Abdelouahhab et al., 2022).

The absence of feed mineralogy is therefore a major interpretive limit. Identical bulk CaO or P_2O_5 values can arise from different apatite, carbonate, silicate, and Fe/Al mineral assemblages, each with distinct dissolution kinetics and acid consumption. The current dataset can establish observed contrasts among the six tests but cannot attribute those contrasts to specific mineral phases. Modern validation should include XRD and automated mineralogy together with a harmonized particle-size basis.

4.3. Acid demand and process trade-offs

The revised acid analysis separates two concepts that are often conflated. The CaO/P_2O_5 ratio quantifies an intrinsic first-order calcium burden, whereas the recorded addition above the CaO-only benchmark captures all additional acid associated with the actual archived test. The latter is not synonymous with chemical inefficiency because phosphate concentrates may contain acid-consuming carbonates and Mg-, Fe-, or Al-bearing phases, and WPA operation also depends on the chosen free-sulfate condition. This distinction is consistent with industrial descriptions of phosphate-rock evaluation and with modern studies showing that associated minerals and dissolved impurities influence acidolysis and downstream behavior (Al-Fariss et al., 1992; Zhao et al., 2025).

PQH-002 illustrates why a low MER cannot substitute for an acid-demand test. Its recorded H_2SO_4/P_2O_5 ratio was 3.036, 14.35% above the CaO-only benchmark, whereas PQH-005 was only 0.97% above that benchmark. At plant scale, a persistent

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difference of this magnitude could affect reagent cost, sulfate balance, heat release, water balance, and gypsum generation. The historical dataset does not explain why the difference occurred, but it demonstrates that a purchasing specification based solely on P_2O_5 and MER may miss economically important process behavior.

PQH-003 demonstrates the complementary value of QA/QC. A reported acid ratio 43% below the CaO-only requirement is chemically incompatible with complete reaction of the measured CaO. Rejecting that entry, rather than treating it as an exceptional performance, is a reproducibility safeguard applicable to retrospective industrial datasets in general.

4.4. Filtration and phosphorus loss to gypsum

Calcium-sulfate crystal size, aspect ratio, aggregation, and cake structure are central to filtration and washing. Controlled studies have linked impurity chemistry and supersaturation to $CaSO_4$ morphology (Peng et al., 2015; Mu et al., 2019; Abdelouahhab et al., 2022), while recent work in hemihydrate and hemihydrate-dihydrate systems shows that crystal habit can materially change filtration strength and phosphorus loss (Huo et al., 2023a, 2023b). High-shear process intensification has likewise demonstrated that mixing and mass transfer can affect both phosphate conversion and phosphogypsum crystallization (Shan et al., 2026). These studies provide possible mechanisms for the observed filtration differences but do not prove the mechanism in the present historical samples because crystal morphology and operating conditions were not preserved.

From an engineering-screening perspective, PQH-001 would warrant caution if judged only by recovery: its filtration index was less than half the repeated 185.3 kg h^{-1} m^{-2} values. Conversely, PQH-005 combined low solid-phase phosphorus loss with a high filtration index. Such combined metrics are more relevant to full-scale verification than recovery alone because filtration can constrain throughput and washing efficiency.

Residual phosphorus also affects phosphogypsum quality. Recent studies and reviews emphasize that P and F impurities limit direct reuse and motivate source control or purification strategies (Fang et al., 2023; Li et al., 2023; Guan et al., 2024; Zhu et al., 2023). Lower P_2O_5 in the gypsum-rich solid is therefore favorable for both phosphorus yield and potential by-product quality, although actual reuse specifications require additional contaminant and radiological characterization that is not available here.

4.5. Role and limitations of the HCl attack screen

The HCl attack test differentiated the samples but did not reproduce the WPA ordering. PQH-003 had the highest HCl extraction but not the highest WPA recovery, while PQH-002 had the lowest HCl extraction but a high WPA recovery. This divergence is unsurprising because an HCl attack does not reproduce sulfuric-acid stoichiometry, calcium-sulfate nucleation and growth, phosphate co-precipitation, or cake filtration. Because the archived HCl protocol is incomplete, the present evidence supports only a limited conclusion: the historical HCl value may serve as a supplementary reactivity screen, but it should not be used alone as a WPA acceptance criterion.

4.6. Integrated concentrate-qualification framework

The most transferable contribution of the reanalysis is a staged qualification framework that prevents a single feed-grade number from dominating the decision. The framework intentionally separates feed descriptors, stoichiometric consistency, observed process response, and solid–liquid separation before a concentrate is advanced to replicated validation or blending studies (Table 4 and Figure 5). It is a decision structure, not a set of universal numerical acceptance limits.

Table 4. Proposed staged screening framework for phosphate concentrates intended for WPA production.

Screening variable	Preferred direction	Primary process relevance	Interpretive caution
Feed P_2O_5	Higher	Lower concentrate consumption per unit P_2O_5	Grade alone does not predict recovery or filtration
CaO/P_2O_5	Lower, all else equal	Lower first-order H_2SO_4 burden	Mineralogical form of Ca is not represented
MER	Lower, all else equal	Lower combined Fe–Al–Mg burden	Omits Si, F, Cl, carbonate, mineral associations
SiO_2, F, Cl and trace contaminants	Within site-specific limits	Crystallization, gas handling, corrosion, product quality	Effects depend on speciation and process conditions

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Screening variable	Preferred direction	Primary process relevance	Interpretive caution
Acid above CaO-only benchmark	Lower after valid QA/QC	Reagent/sulfate-balance indicator	Not equivalent to inefficiency; other acid sinks may be real
Observed WPA P_2O_5 recovery	Higher	Phosphorus yield	Requires replication for precision
P_2O_5 in dry gypsum	Lower	Lower solid-phase P loss and better by-product quality	Loss mechanisms require mineralogy/speciation
Filtration/washing performance	Higher throughput; effective washing	Filter area, throughput, soluble-P recovery	Method must be standardized; ceiling effects possible

Source. *Developed by the authors from the present reanalysis and the literature discussed in Sections 4.1–4.6.*

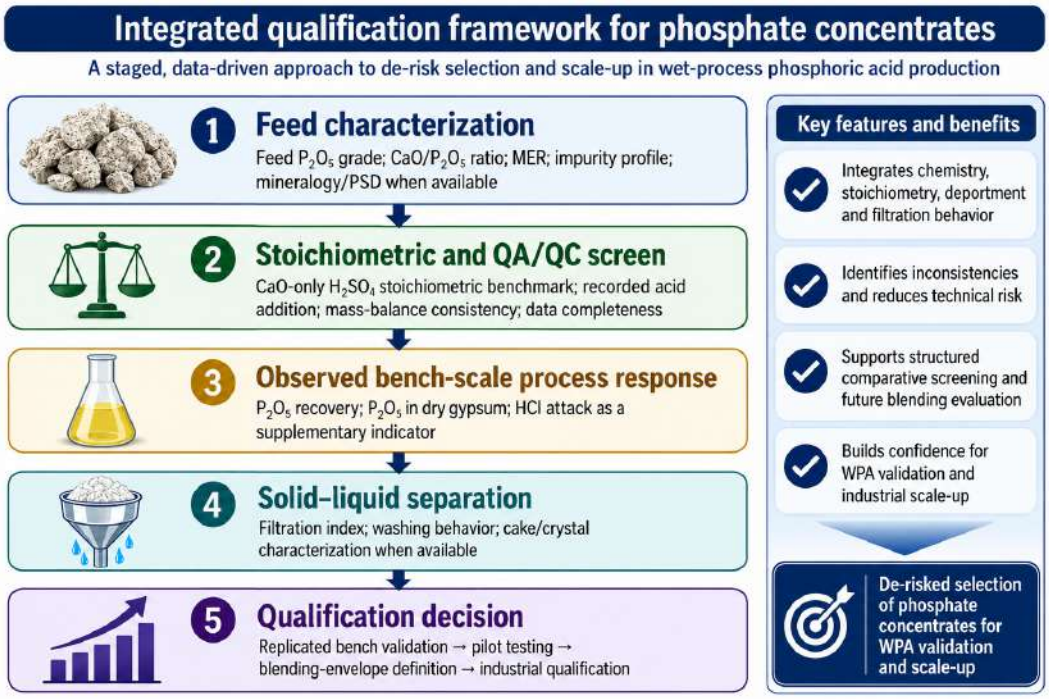

Figure 5. Integrated qualification framework developed from the present quality-controlled reanalysis and the literature discussed in this study. Numerical acceptance limits are intentionally not assigned because they are deposit-, process-, and plant-specific.

Source. *Developed by the authors from the present quality-controlled reanalysis and the literature discussed in Sections 4.1–4.6.*

The framework also provides a rational basis for blending studies. A high-grade concentrate with poor filtration or high recorded acid addition may potentially be blended with another feed that provides a more favorable overall response, but impurity effects are not necessarily additive. Mixture-design or factorial experiments are therefore required before a blending envelope can be defined. The present six-sample campaign generates screening hypotheses; it does not provide a validated blending model.

4.7. Study limitations and recommended validation work

The principal limitation is the absence of replicated full WPA tests. Experimental precision, within-sample variability, and statistical significance of small differences cannot be estimated. A second limitation is incomplete documentation of reaction temperature, residence time, agitation, free sulfate, recycle-acid composition, filtration pressure/vacuum, wash conditions, and experimental equality across the six tests. Third, feed mineralogy, common-basis particle-size distributions, gypsum phase identity, and gypsum-crystal morphology are unavailable. These limitations prevent causal attribution and direct scale-up.

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The unknown geological and beneficiation provenance of the coded samples further restricts generalization. The six concentrates should not be interpreted as representative of global phosphate resources. Likewise, the cross-laboratory naming inconsistency in the archived workbook means that the secondary analyses should be used only as QA/QC context unless provenance can be recovered.

A modern follow-up program should include randomized replicated bench tests; XRD and automated mineralogy of feeds; particle-size distributions on a common basis; ICP-OES/IC with certified reference materials; explicit free-acid and free-sulfate measurements; verified sulfuric-acid mass balances; gypsum phase and crystal-size/morphology characterization; filtration resistance, cake moisture, and wash-efficiency measurements; and a designed experiment varying temperature, residence time, acid addition, and recycle-acid composition. After bench replication, selected concentrates and blends should progress to pilot-scale verification before industrial qualification.

5. CONCLUSIONS

A quality-controlled retrospective reanalysis of six historical bench-scale phosphate-concentrate tests showed that feed P_2O_5 grade alone did not rank observed wet-process phosphoric-acid performance. Across a feed-grade range of 27.59–30.55 wt% P_2O_5, observed recovery ranged from 94.68% to 97.99%; the highest-grade concentrate showed the lowest recovery and the highest P_2O_5 concentration in dry gypsum.

PQH-005 showed the most favorable observed combination of metrics in this campaign, with 97.99% P_2O_5 recovery, 0.405 wt% P_2O_5 in dry gypsum, a high filtration index, and recorded sulfuric-acid use approximately 1% above the CaO-only stoichiometric benchmark. PQH-002 combined high recovery and low MER but had the largest positive deviation from the CaO-only acid benchmark among valid tests. PQH-001 showed that high observed recovery can coexist with poor filtration, whereas PQH-004 showed that high feed grade can coexist with relatively poor phosphorus deportment. PQH-003 could not be fully compared because its acid and filtration fields failed QA/QC.

For concentrate qualification, the evidence supports an integrated screening sequence combining feed chemistry, CaO-based stoichiometry, recorded acid addition, P_2O_5 recovery, residual P_2O_5 in gypsum, and standardized filtration behavior. These variables define a defensible validation program, but replicated modern bench tests and pilot-scale confirmation are required before industrial scale-up or statistically supported ranking of concentrates.

Declarations

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Author contributions: Conceptualization, methodology, literature synthesis, critical analysis, visualization, and writing were performed by the author.

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