

## **SULFURIC ACID REGENERATION FROM NICKEL LATERITE PROCESSING: TECHNOLOGIES, PROCESS CHEMISTRY, SCALE-UP CHALLENGES, AND CIRCULAR ECONOMY PERSPECTIVES**

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### **Abstract**

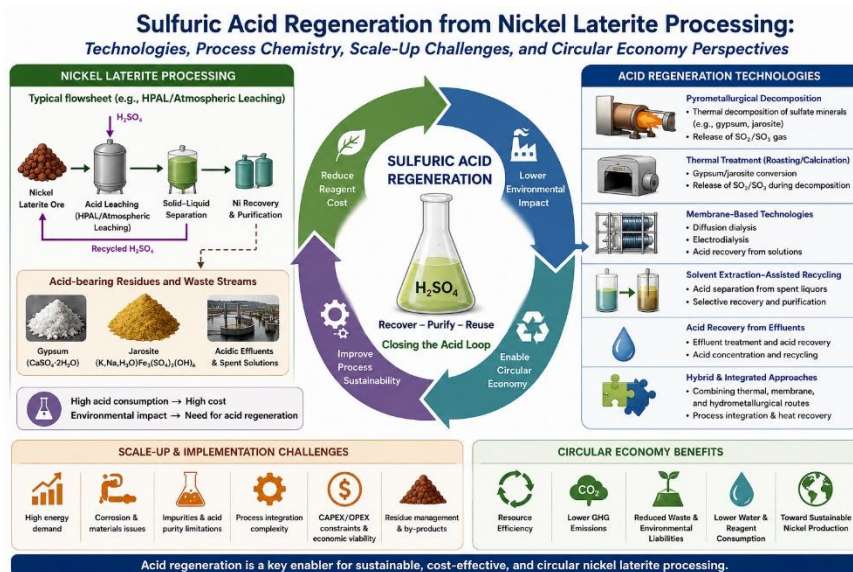
Sulfuric acid consumption represents one of the major operational and environmental challenges in nickel laterite hydrometallurgy, particularly in high-pressure acid leaching (HPAL), atmospheric leaching, and integrated purification circuits. Acid regeneration has therefore emerged as a strategic route to reduce reagent costs, minimize waste generation, improve process sustainability, and support the integration of the circular economy. This critical review analyzes the principal sulfuric acid regeneration technologies applied or proposed for nickel laterite processing, including pyrometallurgical decomposition of sulfates, thermal treatment of gypsum and jarosite residues, membrane-based systems, electrodialysis, diffusion dialysis, solvent extraction-assisted recycling, acid recovery from effluents, and hybrid integrated approaches. The review discusses fundamental process chemistry, reaction mechanisms, thermodynamic constraints, impurity behavior, corrosion challenges, energy demand, gas emissions, and integration with downstream hydrometallurgical operations. Particular emphasis is placed on industrial scalability, operational stability, acid purity limitations, residue management, and the gap between laboratory feasibility and commercial implementation. The analysis further examines the role of acid regeneration in reducing environmental liabilities, enabling residue valorization, lowering freshwater and reagent consumption, and improving the long-term sustainability of nickel laterite processing flowsheets within emerging circular economy frameworks.

**Keywords:** Nickel laterites; Sulfuric acid regeneration; Hydrometallurgy; Circular economy; Acid recycling; Process sustainability

### **Highlights**

- Critical review of sulfuric acid regeneration technologies for nickel laterite processing.
- Comparative analysis of process chemistry, scalability, and industrial integration challenges.
- Evaluation of acid recovery routes, including thermal, membrane, electrochemical, and hybrid systems.
- Discussion of circular economy opportunities through residue valorization and reagent recycling.

## Graphical abstract



## 1. Introduction

Nickel laterites have become increasingly important sources of nickel and cobalt due to rising demand from stainless steel production, batteries, and energy-transition technologies. Although sulfide ores historically dominated nickel production, lateritic ores now represent most global nickel resources and are expected to play a central role in future supply chains. However, laterite processing remains technically challenging because nickel is associated with iron oxyhydroxides and magnesium silicates, which strongly influence acid consumption, impurity dissolution, and downstream residue generation. Consequently, hydrometallurgical processing routes such as high-pressure acid leaching (HPAL), atmospheric leaching, heap leaching, and hybrid flowsheets are widely employed.

Sulfuric acid is the principal reagent used in laterite hydrometallurgy because of its high proton activity and compatibility with downstream purification circuits. In HPAL systems, sulfuric acid rapidly dissolves nickel and cobalt under elevated temperature and pressure, whereas atmospheric and heap leaching generally require longer residence times and higher cumulative acid exposure. However, only part of the acid contributes directly to valuable-metal dissolution. A substantial fraction reacts with gangue minerals containing Fe, Mg, Al, carbonates, and silicates, thereby increasing reagent consumption, sulfate formation, and operational costs.

Acid consumption varies significantly according to ore mineralogy, leaching route, temperature, pulp density, residence time, and neutralization strategy. Limonitic laterites processed by HPAL may consume 400–700 kg H<sub>2</sub>SO<sub>4</sub> per tonne of dry ore, while saprolitic ores frequently generate additional challenges due to Mg-rich silicates and MgSO<sub>4</sub> accumulation. As a result, laboratory-scale efficiencies often fail to represent industrial performance once sulfate stabilization, gypsum formation, iron hydrolysis, neutralization, and impurity buildup are considered.

The challenge extends beyond reagent consumption because unrecovered sulfuric acid is commonly converted into gypsum residues, sulfate-rich effluents, neutralized tailings, or saline process waters. Conventional neutralization effectively controls acidity but irreversibly destroys recoverable acid, transferring environmental burdens to sludge disposal and water-treatment systems. Recent studies on acid mine drainage and sulfate-rich industrial wastewater indicate that neutralization is increasingly interpreted as a treatment endpoint rather than a circular solution (Baloyi et al., 2024; Masindi & Tekere, 2020). Consequently, sulfuric acid regeneration and sulfate recycling have become increasingly important within emerging circular-hydrometallurgy strategies.

Several technologies have been proposed for sulfuric acid recovery from sulfate-rich liquors, including membrane separation, electrodialysis, solvent extraction, evaporation–crystallization, pyrometallurgical sulfate decomposition, and hybrid systems. Membrane technologies are attractive because they operate at relatively low temperatures and can selectively recover protons. However, industrial nickel laterite liquors frequently contain high concentrations of Mg, Fe, Al, silica, suspended solids, and sulfate species that promote fouling, scaling, membrane degradation, and impurity crossover. Thermal regeneration systems show similar trade-offs, since sulfate decomposition routes are chemically robust but require high temperatures, complex off-gas treatment, and elevated energy demand.

Recent studies demonstrate both technological progress and significant industrial limitations. Laboratory-scale investigations often report high acid recovery efficiencies, whereas industrial applications exhibit lower selectivity and operational instability due to multicomponent liquor chemistry and process variability. Consequently, hybrid systems integrating membrane separation, solvent extraction, evaporation, and thermal recovery are

increasingly investigated as more realistic alternatives for sulfate management, water reuse, and partial acid-loop closure.

This review critically evaluates sulfuric acid regeneration in nickel laterite hydrometallurgy, emphasizing process chemistry, industrial scalability, technical readiness, sulfate management, and circular-economy integration. The review compares neutralization and regeneration strategies, examines sulfate-rich process streams, discusses the limitations of thermal, membrane, solvent extraction, evaporation, and hybrid systems, and identifies major technological and research gaps in industrial acid recovery.

## 2. Methodology

This review was conducted as a PRISMA-informed critical review rather than as a fully systematic review, following the general principles of the PRISMA 2020 framework adapted for engineering and hydrometallurgical process analysis (Page et al., 2021). The methodological approach combined systematic literature identification and screening with critical technological evaluation focused on sulfuric acid regeneration, sulfate recycling, process integration, and circular hydrometallurgy in nickel laterite processing. The review emphasized sulfuric acid recovery technologies applicable to sulfate-rich hydrometallurgical systems, including pyrometallurgical sulfate decomposition, spray roasting, pyrohydrolysis, diffusion dialysis, electrodialysis, bipolar membrane electrodialysis (BMED), nanofiltration, membrane distillation, solvent extraction-assisted recovery, evaporation–crystallization systems, sulfur dioxide capture, sulfuric acid re-production, and hybrid process-integration strategies.

The literature survey was conducted between January and March 2026 using Scopus, Web of Science, ScienceDirect, SpringerLink, Wiley Online Library, ACS Publications, and Google Scholar. The search strategy employed combinations of Boolean operators and keywords related to sulfuric acid regeneration and nickel laterite hydrometallurgy, including “sulfuric acid regeneration”, “acid recovery”, “sulfate recycling”, “nickel laterite leaching”, “electrodialysis”, “diffusion dialysis”, “membrane acid recovery”, “pyrohydrolysis”, “thermal sulfate decomposition”, “acid mine drainage valorization”, “membrane fouling”, and “MgSO<sub>4</sub> accumulation”. Representative search strings included (“sulfuric acid regeneration” AND “nickel laterite”), (“acid recovery” AND “sulfate recycling” AND hydrometallurgy), and (“electrodialysis” OR “diffusion dialysis” OR “membrane acid recovery”) AND sulfate.

Publications from 2020–2026 were prioritized to emphasize recent technological developments and emerging circular-hydrometallurgy strategies, although earlier references were included to contextualize industrial sulfuric acid production, nickel laterite mineralogy, iron hydrolysis, gypsum formation, sulfate stabilization, and classical hydrometallurgical principles.

The screening procedure followed sequential filtering stages involving title screening, abstract evaluation, technical relevance assessment, duplicate removal, and full-text analysis. Studies lacking technical relevance, operational data, or applicability to sulfate-bearing acidic systems were excluded. Preference was given to publications reporting quantitative operational parameters such as sulfuric acid concentration, sulfate content, membrane flux, current efficiency, impurity rejection, scaling behavior, operating temperature, pH, sulfate conversion efficiency, energy consumption, or process-integration performance. The inclusion criteria prioritized peer-reviewed journal articles, industrial case studies, pilot-scale investigations, representative review articles, and theses containing quantitative physicochemical or operational information. Publications were selected when they addressed sulfuric acid recovery, sulfate separation, membrane stability, thermal sulfate decomposition, fouling mechanisms, gas-treatment systems, water reuse, or process-integration challenges associated with sulfate-rich hydrometallurgical liquors. Exclusion criteria included duplicate publications, studies lacking sufficient methodological or operational detail, purely descriptive reports without technological analysis, and publications unrelated to sulfate-bearing acidic systems.

Because direct industrial literature specifically addressing sulfuric acid regeneration in nickel laterite hydrometallurgy remains limited, the review also incorporated studies from analogous sulfate-rich industrial systems, including titanium dioxide waste acid, acid mine drainage (AMD), mining wastewater, battery-recycling liquors, vanadium leachates, copper electrolytes, and aluminum-finishing effluents. These systems were not considered directly equivalent to nickel laterite liquors; instead, their applicability was critically evaluated based on sulfate concentration, Mg load, Fe and Al concentrations, silica presence, suspended solids, acidity, operating temperature, scaling behavior, membrane fouling tendency, and process variability. Consequently, evidence transferability was qualitatively assessed according to the degree of chemical and operational similarity to nickel laterite processing conditions.

To improve evidentiary transparency and reduce interpretive overgeneralization, the reviewed literature was classified according to evidence maturity, operational realism, and

technological relevance. Industrial or commercial operational data from nickel laterite or sulfate-rich metallurgical systems were considered the highest evidence level because they reflect real process variability, impurity accumulation, scaling behavior, and long-term operational limitations. Pilot-scale investigations and demonstration studies were classified as intermediate-to-high evidence because they provide partial validation under semi-industrial conditions.

Laboratory-scale studies using real multicomponent industrial liquors were considered more representative than studies based exclusively on synthetic sulfate solutions because real liquors contain dissolved Fe, Mg, Al, silica, suspended solids, colloidal species, and variable sulfate chemistry that strongly influence process stability and recovery efficiency. Synthetic-solution studies were nevertheless included because they provide a mechanistic understanding of proton transport, sulfate speciation, membrane selectivity, thermal decomposition behavior, and fouling mechanisms under controlled conditions. However, these studies were interpreted cautiously because simplified laboratory systems frequently underestimate industrial limitations associated with scaling, impurity crossover, membrane aging, corrosion, and operational instability. The evidence-quality framework therefore included five qualitative categories: (i) industrial/commercial operational data; (ii) pilot-scale or demonstration-scale studies; (iii) laboratory-scale studies using real industrial liquors; (iv) laboratory-scale studies using synthetic sulfate solutions; and (v) conceptual or theoretical analyses.

Because the available literature exhibited substantial heterogeneity regarding liquor composition, operating conditions, process configuration, recovery targets, and performance metrics, the review did not perform a statistical meta-analysis. Instead, the methodological emphasis was placed on comparative technological evaluation, process-transfer limitations, operational constraints, industrial scalability, and critical synthesis of the available evidence.

In several cases, contradictions were identified between laboratory-scale and industrial-scale performance. Membrane systems frequently reported high acid recovery efficiencies under controlled conditions, whereas industrial liquors exhibited lower selectivity and more severe fouling due to multicomponent sulfate chemistry, scaling, silica interactions, and impurity accumulation. Similar discrepancies were observed in thermal regeneration systems, where chemically robust sulfate decomposition routes were often associated with high thermal energy demand, corrosion challenges, and complex off-gas treatment requirements.

Despite the systematic screening strategy and evidence-quality framework used in this review, several limitations must be acknowledged. Direct industrial literature on sulfuric acid regeneration in nickel laterite hydrometallurgy remains limited compared with that of other sulfate-rich process industries. Consequently, some of the available evidence comes from analogous systems, including acid mine drainage, titanium dioxide waste acid, mining wastewater, battery-recycling liquors, vanadium leachates, copper electrolytes, and acidic industrial effluents.

Although these systems provide valuable mechanistic and operational insights, their transferability to nickel laterite liquors remains constrained by differences in sulfate concentration, Mg content, Fe and Al loading, silica behavior, solids concentration, acidity, and operational variability. Another limitation is the literature's heterogeneity in operating conditions, liquor composition, membrane materials, thermal-treatment conditions, and reported performance metrics, which complicates direct quantitative comparisons among regeneration technologies. Many laboratory-scale investigations also use simplified synthetic sulfate solutions that may underestimate industrial limitations associated with fouling, scaling, impurity crossover, corrosion, membrane aging, and long-term operational instability.

Furthermore, several regeneration technologies, particularly membrane-based and hybrid systems, remain at the laboratory or pilot scale and therefore lack long-term industrial validation. Industrial operational data are also frequently proprietary or partially reported, limiting comparisons of sulfuric acid recovery efficiency, energy demand, operational stability, and economic performance. The review is also constrained by the absence of standardized sustainability indicators across the literature, since parameters such as acid recovery efficiency, freshwater reuse, sulfate purge reduction, gypsum avoidance, membrane lifetime, energy consumption, and CO<sub>2</sub> emissions are often reported inconsistently.

Finally, publication bias must be considered because successful laboratory-scale results and high recovery efficiencies are more commonly reported than operational failures or economically unfavorable cases. Consequently, the conclusions presented in this review should be interpreted as a critical synthesis of the currently available evidence rather than as universal guidelines for sulfuric acid regeneration across all nickel laterite processing systems.

Figure 1 summarizes the PRISMA-informed workflow used in this review, including literature identification, duplicate removal, screening, eligibility assessment, and final

inclusion based on technological relevance, operational applicability, and evidence maturity. The methodological strategy therefore emphasizes industrial applicability, process-transfer relevance, evidence transparency, and critical technological comparison rather than strict adherence to systematic-review standards, which was considered more appropriate given the fragmented and multidisciplinary nature of the sulfuric acid regeneration literature in nickel laterite hydrometallurgy.

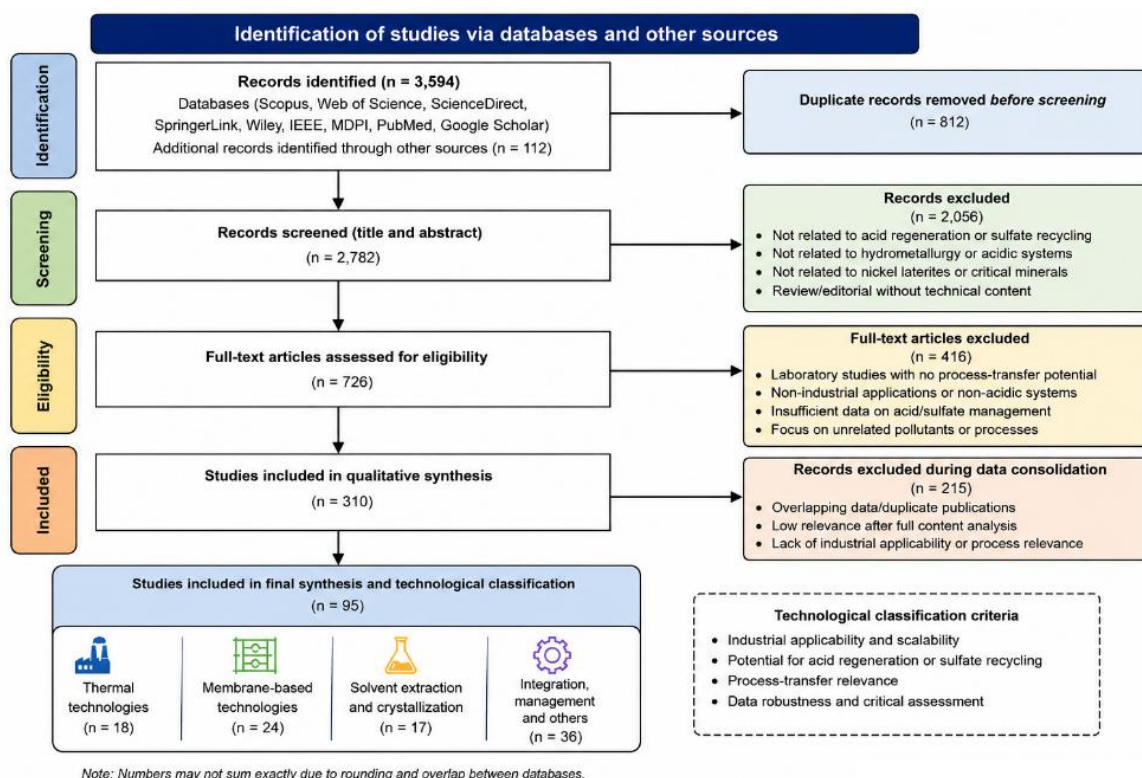


Figure 1. PRISMA-based workflow used for literature identification, screening, eligibility assessment, and inclusion of studies related to sulfuric acid regeneration and sulfate recycling in nickel laterite hydrometallurgy. Adapted from Page et al. (2021).

Figure 1 shows that the screening methodology emphasized industrial applicability and process-transfer relevance rather than strict system similarity. This broader selection strategy was necessary because industrial data specifically addressing sulfuric acid regeneration in nickel laterite circuits remains limited compared with other acidic process industries.

### 3. Nickel Laterites and Acid Consumption Mechanisms

#### 3.1. Mineralogy of Nickel Laterites

Nickel laterites are commonly classified into limonitic and saprolitic ores, although transitional zones frequently occur within the same deposit. This mineralogical distinction

strongly affects acid consumption, impurity dissolution, sulfate accumulation, residue chemistry, and downstream process integration. Consequently, ore mineralogy is one of the main factors controlling the feasibility of sulfuric acid regeneration in laterite hydrometallurgy.

Limonitic laterites mainly contain iron oxyhydroxides like goethite, hematite, and poorly crystalline Fe phases. Nickel and cobalt bind with iron minerals through adsorption or substitution and generally have lower magnesium than saprolitic laterites, which explains HPAL's popularity. Industrial HPAL operates at 240–270 °C and 3.5–5.5 MPa, quickly dissolving Ni and Co but causing iron hydrolysis and sulfate stabilization issues during neutralization.

Saprolitic laterites mainly contain Mg-rich silicates like serpentine and olivine, with nickel embedded in silicate structures rather than Fe oxyhydroxides. Despite higher nickel grades, their high magnesium content raises sulfuric acid use, as Mg silicates react strongly with acids. This leads to liquors with high MgSO<sub>4</sub>, complicating sulfate recirculation and water treatment in leaching processes.

The mineralogical differences between limonitic and saprolitic ores influence more than extraction: limonitic systems involve Fe hydrolysis, ferric residue, and gypsum, while saprolitic systems face magnesium sulfate and silica issues. These differences impact acid regeneration, affecting membrane selectivity, evaporation, scaling, and sulfate recovery, which depend on liquor composition.

Studies on mineral processing and membranes show gangue mineralogy controls impurity transport, fouling, and selectivity (Habibi et al., 2022; Manis et al., 2021). Similar issues occur in sulfate-rich systems with titanium residues and silicate wastes, where impurity buildup hampers recovery (Zhao et al., 2025; Zubarev et al., 2023).

### **3.2. Acid Consumption Reactions**

Sulfuric acid use in nickel laterite processing stems from dissolving valuable metals such as Ni and Co and from parasitic acid consumption by Fe, Mg, Al, carbonates, and silicates. Typically, parasitic acid use accounts for the majority of total acid demand.

Nickel and cobalt dissolve better in acid, especially in HPAL systems where high temperature speeds up reactions. However, gangue minerals can consume a large amount of acid without aiding metal recovery. Mg dissolution is critical because it raises acid use and MgSO<sub>4</sub> buildup in recycled liquids. Unlike iron, magnesium remains soluble over a wide pH

range, leading to the accumulation of  $MgSO_4$ , which increases ionic strength, promotes scaling, and exacerbates water-management issues.

The severity of parasitic acid use varies with ore mineralogy and conditions. HPAL systems increase metal extraction rates at high temperatures and pressures. Atmospheric and heap leaching use less energy but need longer times and more acid due to slower reactions. Lower severity does not always mean less total acid, as evidenced by comparisons between laboratory-scale efficiencies and industrial acid balances.

Similar acid-consumption processes occur in other sulfate-rich hydrometallurgical systems, affecting acid recovery due to impurity dissolution, sulfate buildup, and operational challenges in membrane systems, as shown in recent studies on vanadium slags, titanium residues, and sulfate liquors (Rajahalme et al., 2021; Tian et al., 2022; Zhang et al., 2021; Bendová & Dušek, 2022; Deng et al., 2024; Wang et al., 2020; Zhang et al., 2020).

Table 1 summarizes the main differences between limonitic and saprolitic laterites, along with their principal acid-consumption pathways and operational implications, to assess the influence of mineralogy on sulfuric acid demand and sulfate management.

Table 1. Comparative characteristics of limonitic and saprolitic nickel laterites, including dominant mineral phases, principal acid-consuming reactions, typical impurities released, operational conditions, and implications for sulfate accumulation and downstream acid recovery. Adapted from Habibi et al. (2022), Zhao et al. (2025), Zhang et al. (2021), and Jia et al. (2025)

Parameter	Limonitic laterites	Saprolitic laterites	Implication for acid regeneration
Dominant minerals	Goethite, hematite, Fe oxyhydroxides	Serpentine, olivine, Mg silicates	Different sulfate chemistry and impurity loads
Main Ni association	Adsorbed or substituted in Fe phases	Incorporated in Mg silicate structures	Controls leaching kinetics and acid efficiency
Typical Mg content	Low to moderate	High	High Mg promotes $MgSO_4$ accumulation
Main acid-consuming phases	Fe oxyhydroxides, Al phases, minor silicates	Mg silicates, serpentine, olivine	Saprolites generally increase parasitic acid use
Main impurities released	Fe, Al, Mn, sulfate	Mg, Si, Fe, sulfate	Determines downstream treatment route
Typical processing route	HPAL	Atmospheric, heap, hybrid leaching	Route affects acid recovery feasibility
Key sulfate issue	Fe hydrolysis, gypsum formation	$MgSO_4$ accumulation	$MgSO_4$ -rich liquors are harder to regenerate
Main recovery limitation	Ferric residue and gypsum load	High ionic strength and scaling	Requires different acid management strategies

Table 1 shows that acid consumption cannot be assessed solely by nickel extraction efficiency. Ore mineralogy strongly influences impurity dissolution, sulfate formation, scaling, and liquor chemistry. In limonitic systems, Fe hydrolysis and ferric residues are key concerns, whereas saprolitic systems are more affected by MgSO<sub>4</sub> buildup and silica-related issues.

Figure 2 shows the main sulfuric acid consumption pathways in nickel laterite processing, schematically illustrating the relationships among ore mineralogy, gangue dissolution, sulfate accumulation, and downstream recovery constraints.

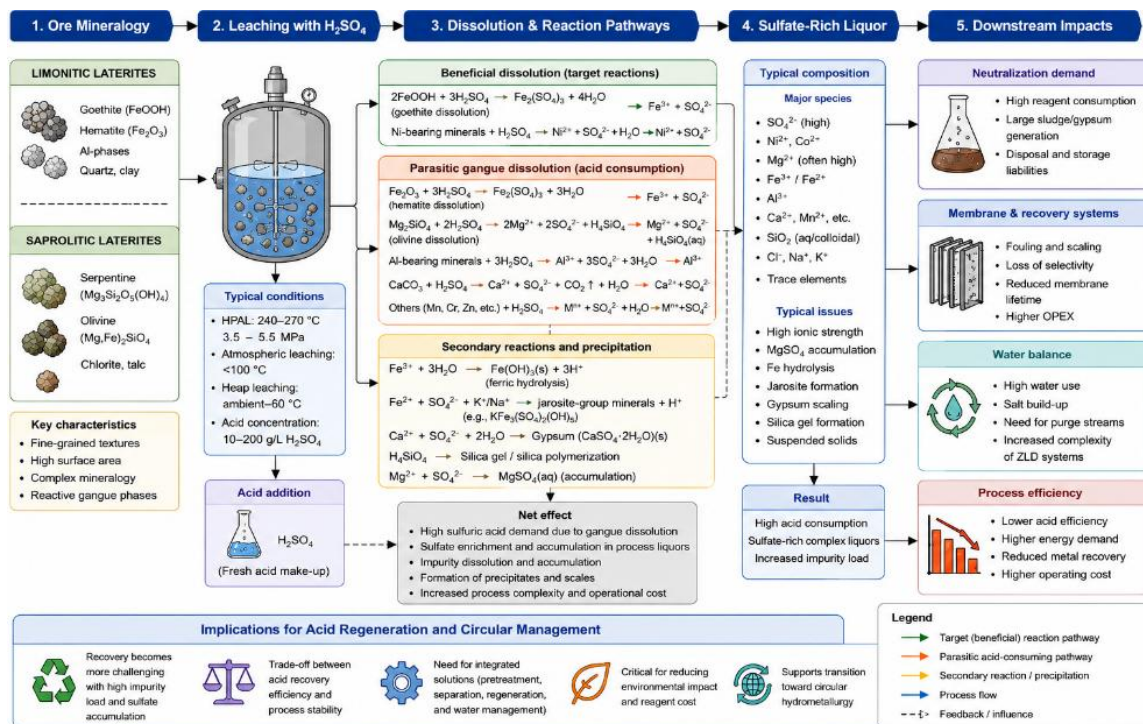


Figure 2. Main sulfuric acid consumption pathways during nickel laterite processing, including Ni/Co dissolution, parasitic gangue reactions, sulfate formation, impurity accumulation, and downstream impacts on neutralization and acid recovery systems. Adapted from Tian et al. (2022), Zhang et al. (2021), and Jia et al. (2025).

Figure 2 highlights that sulfuric acid consumption is not restricted to the leaching stage. Impurity dissolution and sulfate accumulation directly influence the demand for neutralization, membrane performance, scaling behavior, water balance, and the overall feasibility of circular acid management strategies.

#### 4. Sulfuric Acid Use in Laterite Processing Technologies

Nickel laterite hydrometallurgy involves various leaching methods, such as HPAL, tank, heap, agitated, and hybrid flowsheets, all using sulfuric acid. Their conditions affect acid efficiency and sulfate management.

HPAL is the main technology for processing limonitic laterites because it quickly dissolves Ni and Co at high temperatures (240–270 °C) and pressures (3.5–5.5 MPa) within 60–120 min, often achieving over 90% nickel recovery. However, it also dissolves gangue minerals, hydrolyzes iron, stabilizes sulfate, and consumes acid, producing sulfate-rich liquors that require large neutralization and residue management. Sulfuric acid consumption can range from 400–700 kg per ton of dry ore, depending on mineralogy and Mg content.

Atmospheric tank leaching operates at lower temperatures and pressures, typically below 100 °C and under atmospheric conditions. While this reduces energy costs and autoclave complexity, it results in slower kinetics, longer residence times, larger reactors, and increased acid consumption. These systems may experience acid loss due to gangue dissolution and silica instability. High Mg dissolution degrades water management performance because the buildup of MgSO<sub>4</sub> increases ionic strength.

Heap leaching is attractive due to its low capital and simple operation compared to HPAL. It uses acid irrigation rates of 5–15 L·m<sup>-2</sup>·h<sup>-1</sup> over several months to a year. However, acid efficiency is often limited by slow kinetics, preferential flow, ore heterogeneity, channeling, and poor liquid distribution. Long-term acid exposure can increase the dissolution of Mg silicates and secondary phases, producing sulfate-rich solutions with low nickel concentrations. Thus, lower capital cost does not necessarily mean easier sulfate management.

Agitated leaching systems, between HPAL and heap leaching, improve mass transfer and solid suspension, often achieving faster kinetics under moderate conditions. Acid demand varies with mineralogy and pulp density. Higher solids can increase productivity but also raise acid use, viscosity, and scaling. Optimizing the solid–liquid ratio is key: excessive dilution strains water management, while high pulp density may reduce efficiency and cause instability.

Hybrid flowsheets that combine atmospheric and heap leaching, HPAL, or impurity removal aim to improve acid utilization and reduce sulfate buildup. Some separate high-Mg fractions before acid leaching, others use precipitation, solvent extraction, or membranes to lower impurities. While these methods can boost acid efficiency, industrial performance varies as added process complexity demands more control.

Operational variables significantly affect sulfuric acid efficiency and sulfate production in laterite processing. Temperature accelerates metal dissolution and gangue reactions; pressure influences HPAL kinetics and phase stability; pH and Eh govern iron hydrolysis, sulfate formation, and impurity precipitation. Residence time affects acid exposure in atmospheric and heap systems. Pulp density and solid–liquid ratio impact acid distribution, viscosity, heat transfer, and neutralization. Sometimes, optimizing for nickel recovery increases sulfate and impurity levels, indicating a trade-off between extraction and acid management.

Similar challenges arise in sulfate-rich hydrometallurgical systems, including titanium residues, vanadium leachates, acidic recycling streams, and waste liquors. Research on solvent extraction, sulfate purification, and acid recovery highlights the impact of impurity buildup and sulfate chemistry on recovery feasibility (Cao et al., 2020; Chen et al., 2021). High sulfate levels and impurities also lower selectivity and complicate treatment in vanadium and titanium systems (Ding et al., 2024; Zhou et al., 2020). Acidic mining waters and rare-earth sulfate liquors show that sulfate management often limits flowsheet design (Valderrama Ángel et al., 2022). Additionally, high sulfate concentration alone cannot predict recovery, as liquor composition, ionic strength, and impurities influence separation (Zhu et al., 2023).

Table 2 summarizes the operational characteristics of the main nickel laterite leaching technologies, including sulfuric acid demand, operating conditions, impurity formation, and sulfate management.

Table 2. Comparative analysis of sulfuric acid use in nickel laterite processing technologies, including typical operating conditions, acid consumption ranges, residence time, nickel recovery, principal impurities released, and operational limitations. Adapted from Cao et al. (2020), Chen et al. (2021), Ding et al. (2024), and Valderrama Ángel et al. (2022).

Route	Typical conditions	Residence time	Acid consumption tendency	Ni recovery tendency	Main limitations
HPAL	240–270 °C; 3.5–5.5 MPa	60–120 min	High, often 400–700 kg H <sub>2</sub> SO <sub>4</sub> /t ore	High, often >90%	High CAPEX, sulfate-rich liquors, neutralization load
Atmospheric tank leaching	<100 °C; atmospheric pressure	Hours to days	Moderate to high	Moderate to high	Slow kinetics, high acid inventory, Mg dissolution
Heap leaching	Ambient to mild heating; irrigation 5–15 L·m <sup>-2</sup> ·h <sup>-1</sup>	Months to >1 year	High cumulative exposure	Variable	Channeling, slow kinetics, low PLS grade

Agitated leaching	Moderate temperature; controlled mixing	Hours	Moderate to high	Moderate	Slurry handling, scaling, pulp-density constraints
Hybrid flowsheets	Combined routes	Variable	Site-specific	Site-specific	Higher process complexity and control demand

Table 2 shows that no leaching method minimizes acid use, impurity release, sulfate, and complexity together. HPAL offers faster recovery but produces sulfate-rich liquors and requires higher neutralization. Heap and atmospheric leaching lower capital costs but often increase acid exposure and  $MgSO_4$  buildup.

In Figure 3, the main sulfuric acid utilization pathways and sulfate-generation mechanisms associated with different laterite-processing technologies are schematically illustrated to compare how operational conditions affect acid efficiency and downstream treatment requirements.

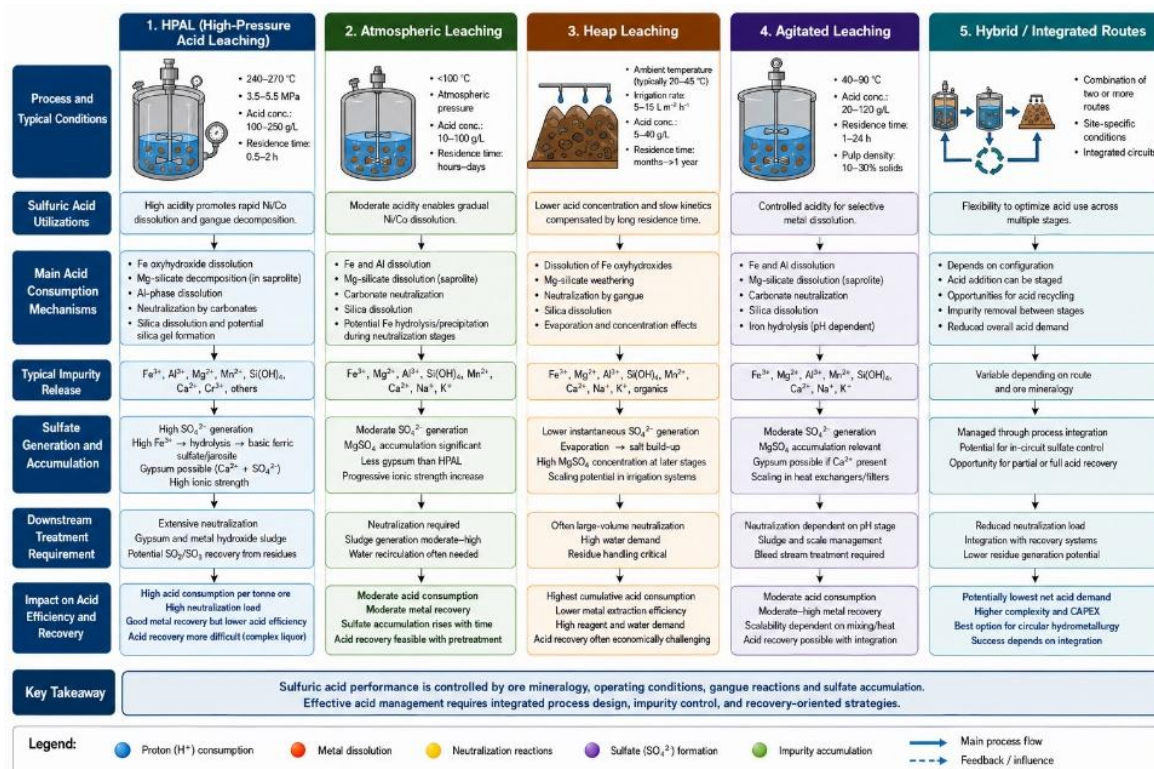


Figure 3. Comparative sulfuric acid utilization pathways in HPAL, atmospheric leaching, heap leaching, agitated leaching, and hybrid nickel laterite processing routes, including acid consumption mechanisms, impurity release, sulfate accumulation, and downstream neutralization requirements. Adapted from Chen et al. (2021), Valderrama Ángel et al. (2022), and Zhu et al. (2023).

Figure 3 highlights that sulfuric acid performance cannot be evaluated exclusively from nickel extraction efficiency. The interaction between mineralogy, operating conditions,

impurity dissolution, and sulfate accumulation strongly determines the feasibility of downstream acid recovery and circular hydrometallurgical integration.

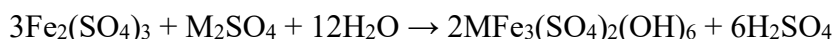
## 5. Chemistry of Sulfuric Acid Losses and Sulfate Formation

Sulfuric acid losses in nickel laterite hydrometallurgy are not restricted to primary leaching reactions. A substantial fraction of sulfur ultimately becomes stabilized in secondary sulfate phases, neutralization residues, entrained liquors, or precipitated solids generated throughout downstream processing. Consequently, total acid consumption is controlled not only by ore dissolution but also by sulfate speciation, precipitation reactions, impurity hydrolysis, and recirculating liquor chemistry.

Iron hydrolysis is one of the principal mechanisms responsible for sulfate immobilization in laterite circuits, particularly in HPAL operations processing limonitic ores. Under elevated temperature and acidic conditions, dissolved ferric iron may hydrolyze and precipitate as hematite, basic ferric sulfates, or poorly crystalline Fe-bearing phases. Although iron precipitation partially removes dissolved impurities, it also promotes the incorporation of sulfate into solid residues. In practice, ferric precipitation frequently generates acidic slurries that require further neutralization, thereby increasing gypsum formation and downstream residue production.

Jarosite formation is an important sulfate-consumption pathway. Alkali and ammonium ions react with ferric sulfate to produce jarosite minerals under acidic oxidizing conditions. These stabilize sulfate in residues, reducing free sulfuric acid for recirculation. While jarosite precipitation can improve impurity control, it increases residue mass and complicates waste management due to sulfate retention and low stability under environmental changes.

The generalized jarosite precipitation reaction may be expressed as:



Gypsum precipitation, a common outcome of sulfuric acid neutralization in laterite processing, occurs when calcium-based agents such as lime react with sulfate-rich liquors to form  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . While this reduces sulfate levels, it consumes sulfuric acid and creates large amounts of solid residue. This residue can amount to several tonnes per tonne of nickel produced, depending on ore mineralogy and strategy, and often causes gypsum scaling in pipelines and equipment, reducing operational stability and increasing maintenance needs.

Magnesium sulfate buildup is a key challenge in circular sulfuric acid management. Unlike ferric iron, magnesium is highly soluble over a wide pH range and accumulates in recycled waters. High  $\text{MgSO}_4$  levels increase ionic strength, osmotic pressure, boiling point, viscosity, and the risk of scaling. In membrane systems, these levels reduce proton selectivity, lower performance, exacerbate concentration polarization, and accelerate membrane degradation. Similar issues occur in sulfate-rich industrial streams and in wastewater treatment, where  $\text{MgSO}_4$  buildup reduces separation efficiency and increases energy use (Sheth, 2020; Zhang et al., 2020).

Aluminum hydrolysis and silica interactions cause sulfate losses and instability. Dissolved aluminum may precipitate as hydroxides, basic sulfates, or Al-bearing phases depending on pH and sulfate levels. Silica can polymerize into gelatinous precipitates that entrap sulfate-rich liquors, raising slurry viscosity, hindering filtration, and increasing fouling. Silica-rich phases can also promote the coprecipitation of Fe, Al, and sulfate, thereby reducing separation efficiency.

Sulfate speciation affects acid recovery, involving free sulfuric acid, bisulfate, sulfate ions, and metal complexes that are in dynamic equilibrium with pH, temperature, ionic strength, and metal levels. As sulfate binds more strongly to Mg, Fe, Al, Ca, or alkali metals, free acid recovery becomes more difficult. Many laboratory-scale studies oversimplify sulfate systems, missing the complexity of industrial laterite liquors.

Scaling, coprecipitation, sulfate entrainment, and fouling are particularly problematic in concentration and membrane recovery systems. Research on titanium dioxide waste, acid, and sulfate-rich liquors indicates that multicomponent sulfate chemistry often results in deposits of Fe, Ca, Mg, silica, and sulfate (Xu et al., 2023). Similar issues arise in electrodialysis, where Fe precipitation and sulfate scaling reduce membrane selectivity and increase resistance (Wang et al., 2025). Diffusion dialysis studies show that sulfate-rich liquors containing impurities are less stable over the long term than simple laboratory-scale solutions (Zhang et al., 2020).

Table 3 summarizes the main mechanisms behind sulfuric acid losses and sulfate stabilization in nickel laterite hydrometallurgy, comparing their operational impacts and effects on acid recovery.

Table 3. Main sulfuric acid loss mechanisms and sulfate stabilization pathways in nickel laterite hydrometallurgy, including dominant reactions, typical precipitated phases, operational impacts, and implications for acid recovery systems. Adapted from Pang et al. (2020), Xu et al. (2023), Wang et al. (2025), and Zhang et al. (2020).

Mechanism	Main reaction pathway	Sulfate form generated	Operational impact	Effect on acid recovery
Gangue dissolution	Fe, Mg, Al, carbonate, silicate attack	Metal sulfates	Increases acid consumption	Reduces free acid availability
Fe hydrolysis	Ferric sulfate hydrolysis	Basic ferric sulfate, hematite-associated sulfate	Residue formation	Immobilizes sulfate in solids
Jarosite formation	$\text{Fe}^{3+} + \text{alkali}/\text{NH}_4^+ + \text{sulfate}$	Jarosite-group phases	Sulfate fixation	Reduces recyclable acid
Gypsum precipitation	$\text{Ca}^{2+} + \text{SO}_4^{2-}$	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Sludge and scaling	Irreversible sulfate stabilization
$\text{MgSO}_4$ accumulation	Mg silicate dissolution	Dissolved $\text{MgSO}_4$	High ionic strength, scaling	Difficult to regenerate
Silica interaction	Polymerization/gel formation	Entrained sulfate-rich liquor	Poor filtration, fouling	Reduces separation efficiency
Membrane fouling	Precipitation/deposition	Mixed sulfate scale	Loss of flux/selectivity	Reduces long-term recovery

Table 3 shows that sulfuric acid losses go beyond gangue dissolution. Sulfate stabilization in gypsum, jarosite, ferric precipitates,  $\text{MgSO}_4$  liquors, and entrained solids often hinders circular acid use. Downstream sulfate chemistry more often impacts regeneration feasibility than primary leaching efficiency.

Figure 4 schematically illustrates the main sulfur pathways involved in sulfuric acid use, sulfate stabilization, precipitation, entrainment, and recovery limits, showing how sulfate moves from dissolved acid into solids and process streams.

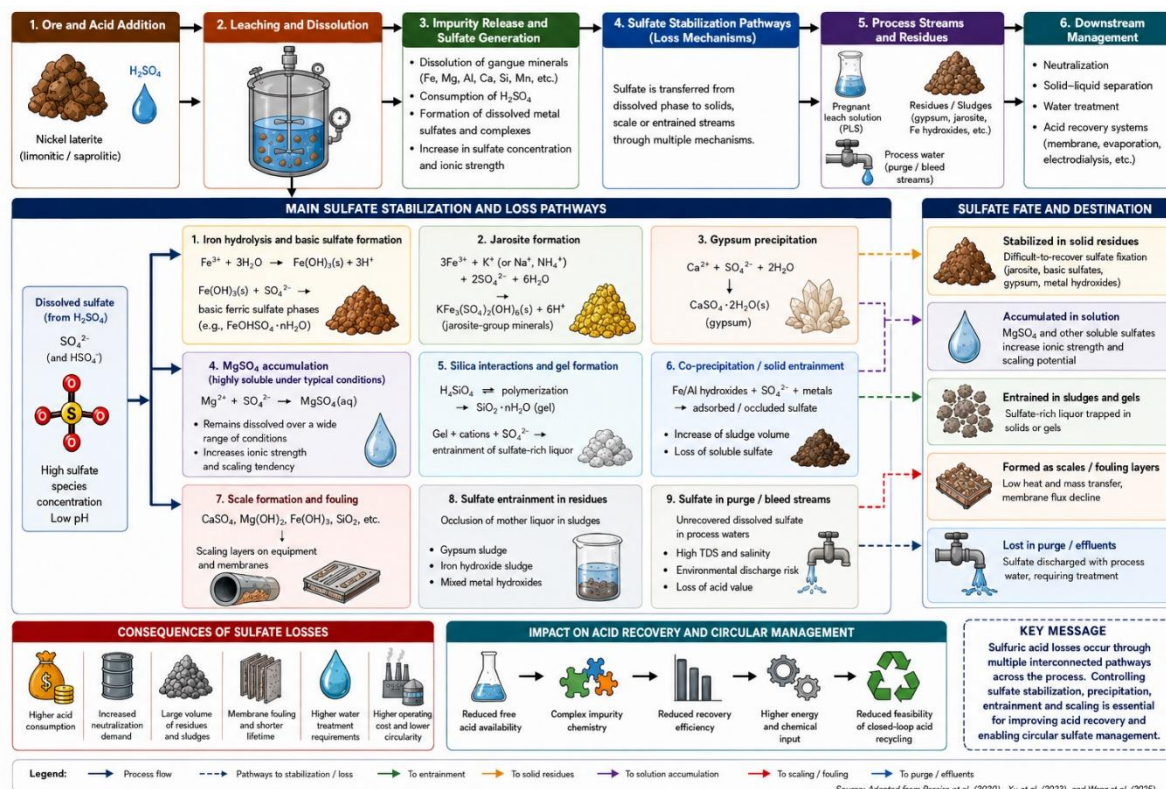


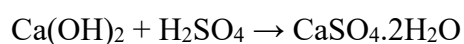
Figure 4. Main sulfuric acid loss and sulfate stabilization pathways during nickel laterite hydrometallurgy, including iron hydrolysis, jarosite formation, gypsum precipitation, MgSO<sub>4</sub> accumulation, silica interactions, fouling, and sulfate entrapment mechanisms. Adapted from Pang et al. (2020), Xu et al. (2023), and Wang et al. (2025).

Figure 4 shows that sulfuric acid losses occur across multiple stages, not just leaching. Sulfate buildup, precipitation, and impurity interactions affect scaling, membrane performance, neutralization requirements, residues, and the feasibility of circular sulfate management.

## 6. Conventional Acid Neutralization and Its Limitations

Conventional acid neutralization is the main method for controlling acidity in nickel laterite hydrometallurgy and sulfate-rich effluents. It uses low-cost, simple-to-operate materials like lime, limestone, MgO, and magnesia. However, it differs from acid regeneration by stabilizing acidity through irreversible chemical reactions, converting sulfate into solid residues or saline effluents, and increasing reagent use, sludge, and tailings management.

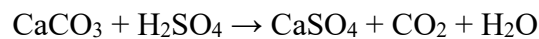
Lime neutralization is the most common industrial route due to the high alkalinity and rapid reaction kinetics of CaO and Ca(OH)<sub>2</sub>. In sulfate-rich liquors, calcium reacts readily with sulfuric acid to form gypsum according to:



This route raises pH, promotes Fe, Al, and metal precipitation but consumes sulfuric acid and creates large residues. In high-acid laterite operations, neutralization yields tons of gypsum solids per tonne of nickel, depending on ore mineralogy and water recirculation. Gypsum scaling damages equipment, raising maintenance and causing instability.

Limestone neutralization is cheaper and safer than lime, but it has slower reaction kinetics and can cause mixing issues and foaming in acidic solutions. It often requires larger reactors and longer retention times, and does not address sulfate stabilization in residues.

The overall neutralization reaction may be represented as:



Magnesium hydroxide precipitation produces denser sludges and lower final volumes than calcium neutralization, attracting interest in MgO systems. Reactive MgO can improve pH control in certain hydrometallurgical processes. However, MgSO<sub>4</sub>'s high solubility across pH ranges complicates neutralization, as sulfuric acid may not form stable precipitates and sulfate-rich liquors may accumulate in recirculating water. Studies on reactive magnesia produced from magnesium sulfate suggest the potential to integrate neutralization and sulfate valorization, but industrial validation remains limited (Pereira, 2025; Pereira & Fonseca, 2025).

Conventional neutralization struggles with sulfate-rich liquors high in Fe, Al, Mn, silica, or metals, often resulting in filterable sludges, coprecipitation, and residual acidity, thereby increasing environmental risks and complicating tailings management.

Challenges in AMD treatment are common. Active neutralization with lime or limestone is reliable but produces large amounts of sludge and requires continuous reagent addition (Nguegang & Ambushe, 2025; Wibowo et al., 2023). Passive systems may cut costs but are slower and less flexible. Sulfate removal often proves more difficult than metal precipitation due to sulfate stability in water (Masindi, 2021; Riggs, 2024).

Recent studies highlight that neutralization in resource recovery from AMD and sulfate-rich wastewater isn't a circular solution. While acidity is managed, sulfuric acid is destroyed and transferred to residues, saline streams, or sludges (Mahlohla, 2024; Ryskie et al., 2021). Similar issues are seen in vanadium-containing systems and metal wastewater, where sulfate stabilization simply shifts environmental burdens to sludge disposal and residue containment (Fadaei, 2025).

Contradictory evidence exists regarding the economic attractiveness of conventional neutralization. Lime and limestone are low-cost reagents, but expenses may increase due to sludge handling, gypsum disposal, water losses, scaling, and tailings. In large hydrometallurgical operations, neutralization consumes most of the reagents and freshwater, so low reagent costs do not necessarily translate to low overall costs.

Table 4 summarizes the principal characteristics of conventional neutralization routes, including reagent consumption, sulfate stabilization mechanisms, sludge generation, operational limitations, and implications for circular acid management.

Table 4. Comparative analysis of conventional acid neutralization routes used in sulfate-rich hydrometallurgical systems, including dominant reactions, typical operating conditions, residue generation, operational limitations, and implications for sulfuric acid recovery. Adapted from Masindi (2021), Pereira (2025), Nguengang and Ambushe (2025), and Wibowo et al. (2023).

Neutralizing agent	Main reaction/product	Advantages	Main limitations	Circularity limitation
Lime/Ca(OH) <sub>2</sub>	Gypsum + metal hydroxides	Fast reaction, mature technology	High sludge volume, scaling	Destroys acid value
Limestone/CaCO <sub>3</sub>	Gypsum + CO <sub>2</sub>	Low reagent cost, safer handling	Slower kinetics, larger reactors	Produces sulfate residues
MgO	Metal hydroxides, dissolved MgSO <sub>4</sub>	Better pH control, denser sludge	MgSO <sub>4</sub> remains soluble	Does not eliminate sulfate accumulation
Reactive magnesia	Mg-based neutralization products	Potential sulfate valorization route	Limited industrial validation	Partial circularity only
Passive AMD-type systems	Precipitation and alkalinity generation	Low energy demand	Slow, less controllable	Not suitable for high-throughput laterite circuits

Table 4 demonstrates that conventional neutralization technologies prioritize acid stabilization over sulfuric acid preservation. Lime systems generally provide rapid neutralization but generate extensive gypsum residues, whereas MgO-based routes may reduce sludge density but do not eliminate sulfate accumulation. In all cases, sulfate management remains strongly dependent on downstream residue handling and water treatment capacity.

Figure 5 schematically illustrates the principal sulfur pathways associated with conventional neutralization, emphasizing how dissolved sulfuric acid is progressively converted into gypsum, hydroxide sludges, entrained sulfate phases, and tailings-associated residues.

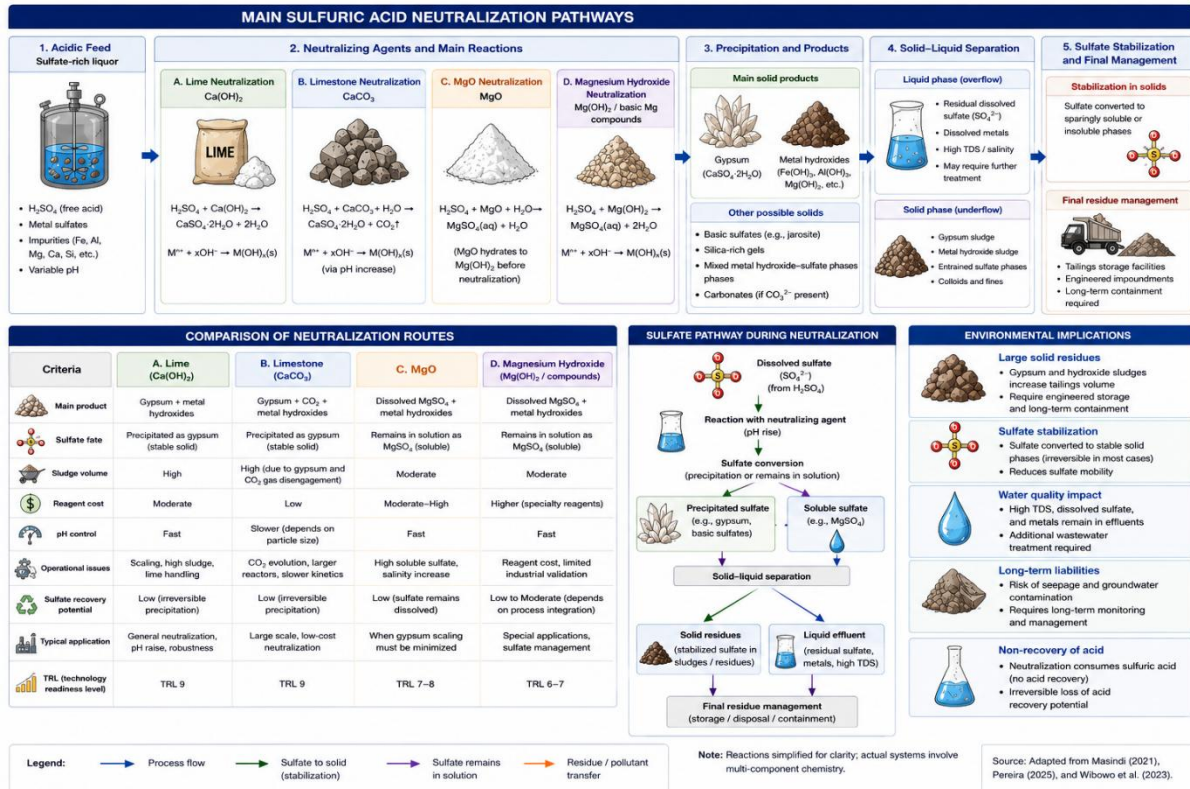


Figure 5. Main sulfuric acid neutralization pathways in sulfate-rich hydrometallurgical systems, including lime, limestone, MgO, and reactive magnesia neutralization, with emphasis on gypsum formation, sludge generation, sulfate stabilization, and downstream environmental implications. Adapted from Masindi (2021), Pereira (2025), and Wibowo et al. (2023).

Figure 5 highlights that conventional neutralization acts primarily as a sulfate stabilization strategy rather than a sulfuric acid recovery route. Although these systems remain industrially robust and operationally mature, they fundamentally convert recoverable acid into solid residues and transfer long-term environmental management to tailings storage and wastewater-treatment infrastructure.

## 7. Sulfuric Acid Regeneration Technologies

Sulfuric acid regeneration technologies aim to recover the sulfur value from sulfate-rich hydrometallurgical streams rather than permanently stabilizing sulfate through neutralization. In nickel laterite processing, regeneration systems must operate under aggressive chemical conditions, including high sulfate concentrations, elevated ionic strength, dissolved multivalent metals, silica, suspended solids, and variable acidity. Consequently, recovery performance depends not only on acid concentration but also on impurity chemistry, sulfate speciation, scaling tendency, and process integration.

No regeneration technology currently provides a universal solution for laterite circuits. Thermal systems generally tolerate chemically complex liquors but require high energy input and extensive gas treatment. Membrane systems may achieve selective proton transport under controlled conditions, yet their performance frequently declines in industrial multicomponent liquors due to fouling, scaling, and loss of selectivity. Solvent extraction, evaporation, and crystallization are therefore more effective as complementary purification or concentration stages rather than complete regeneration systems. As a result, industrial implementation increasingly favors hybrid flowsheets integrating multiple recovery mechanisms.

### **7.1. Pyrometallurgical Regeneration**

Pyrometallurgical regeneration involves thermal decomposition of sulfate compounds, recovery of  $\text{SO}_2/\text{SO}_3$ , and sulfuric acid reabsorption. Methods such as spray roasting, pyrohydrolysis, and thermal sulfate decomposition typically operate at 600–1100 °C, depending on sulfate stability. Ferric sulfate decomposes easily, while  $\text{MgSO}_4$  is more stable, needing over 1000 °C for breakdown.  $\text{MgSO}_4$  buildup impedes sulfate recycling in laterite hydrometallurgy.

Spray roasting and pyrohydrolysis systems can efficiently recover sulfur but require high thermal input, corrosion-resistant equipment, and complex off-gas treatment.  $\text{SO}_2/\text{SO}_3$  streams require dust removal, catalytic oxidation, gas cleaning, and acid absorption before being regenerated to sulfuric acid. Performance depends on sulfate composition, heat transfer, particle size, and impurities. Chlorides, alkali metals, and silica-rich solids can cause fouling and reduce cleaning efficiency.

Pyrometallurgical systems are chemically robust but face industrial limits. High temperatures increase CAPEX and energy use, and dilute sulfate streams are costly because of water evaporation before sulfate decomposition. These methods suit concentrated sulfates, complexes with sulfuric acid plants, or waste-heat recovery.

### **7.2. Membrane-Based Recovery**

Membrane-based technologies are increasingly investigated because they operate at lower temperatures and can selectively recover protons from sulfate-rich liquors. Main methods include diffusion dialysis, electrodialysis (ED), bipolar membrane electrodialysis (BMED), nanofiltration, membrane distillation, and membrane electrolysis. Laboratory-scale

studies report acid recovery rates of 70–90%, depending on the membrane, acid concentration, and impurities (Bendová & Dušek, 2022; Zhang et al., 2020).

Recent developments focus on proton-selective membranes, crosslinked structures, resistant polymers, and hybrid architectures for acidic environments (Cheng et al., 2025; Loza et al., 2023; He et al., 2024; Liu et al., 2025). BMED systems are attractive because they can produce acids and bases from sulfate streams. Nanofiltration and membrane distillation are also studied for sulfate separation and water recovery (Li et al., 2022; Ruiz-Aguirre et al., 2021).

A contradiction exists between laboratory success and industrial use. Industrial laterite liquors contain Fe, Mg, Al, silica, suspended solids, and sulfate chemistry, which lead to fouling, scaling, and membrane aging. This causes declines in proton selectivity and membrane stability. Pilot studies show membrane performance depends on pretreatment, solids removal, iron control, and scaling mitigation (Loza et al., 2022; Rodríguez, 2020). As a result, membrane systems rarely work alone in highly contaminated liquors.

### **7.3. Solvent Extraction and Acid Recovery**

Solvent extraction (SX) is mainly used for selective metal separation, not direct sulfuric acid regeneration. However, it can indirectly aid acid recovery by removing impurities and enhancing purification. Studies on titanium waste acid, scandium, vanadium, and metal-rich streams show SX can selectively remove Fe, Sc, Zn, and other metals while preserving some sulfuric acid (Cao et al., 2020; Chen et al., 2021; Zhou et al., 2020). Ionic liquids and mixed extractants are also explored for sulfate-rich acid liquors (Quijada-Maldonado et al., 2021).

SX should not be considered a universal regeneration method because organic degradation, emulsification, solvent losses, and impurity buildup can intensify under highly acidic, multicomponent conditions. It's more effective as a supplementary purification step when combined with membrane separation, precipitation, or crystallization.

### **7.4. Evaporation, Crystallization, and Concentration**

Evaporation and crystallization systems recover sulfuric acid by removing water, concentrating sulfate, or crystallizing sulfate salts. Multi-effect evaporation reduces steam use compared with single-stage systems, but energy needs remain high for dilute streams. Crystallization often focuses on  $MgSO_4$  or  $FeSO_4$  to reduce impurities.

Scaling and fouling are major OPEX limitations because sulfate solubility depends on temperature, ionic strength, and impurities. Studies on wastewater evaporation and membrane distillation show that scaling often constrains operation more than evaporation efficiency (Kalla et al., 2022; Pang et al., 2020). Similar issues occur in sulfate-rich wastewater systems, where  $MgSO_4$  buildup raises energy use and reduces stability (Ang et al., 2024; Moreira et al., 2022a).

### 7.5. Hybrid Regeneration Systems

Hybrid regeneration systems combine multiple recovery technologies to address the limitations of individual units. They are more realistic for industrial nickel laterite circuits, which require impurity removal, sulfate management, water recovery, and scaling control from sulfate-rich liquors. Common combinations include membrane separation with crystallization, electrolysis with electrodialysis, and reverse osmosis–electrodialysis pretreatment.

Recent hybrid studies demonstrate improved acid and water reuse through membrane electrolysis, sulfate separation, and mining water recovery (Han et al., 2024; Henderson et al., 2024). Integrated systems with pretreatment reduce fouling and extend membrane life (López et al., 2023; Sheth & Nath, 2020). However, concentrate management and scaling remain operational challenges (Zou et al., 2025).

Before Table 5, the principal sulfuric acid regeneration technologies are comparatively summarized to evaluate operating conditions, recovery mechanisms, industrial maturity, and major limitations associated with sulfate-rich laterite liquors.

Table 5. Comparative analysis of sulfuric acid regeneration technologies applicable to nickel laterite hydrometallurgy, including recovery mechanism, typical operating conditions, recovery efficiency ranges, principal operational limitations, and industrial integration challenges. Adapted from Bendová and Dušek (2022), Deng et al. (2024), Hou et al. (2025), and Santoro et al. (2023).

Technology	Recovery mechanism	Typical operating range	Strengths	Main limitations	TRL tendency
Thermal sulfate decomposition	Sulfate $\rightarrow$ oxide + $SO_2/SO_3$	600–1100 °C	High impurity tolerance	High energy, off-gas treatment	Medium–high
Spray roasting/pyrohydrolysis	Thermal decomposition of sulfate liquors	High temperature	Produces sulfur-bearing gas	CAPEX, corrosion, dilution penalty	Medium

Diffusion dialysis	Proton/anion transport	Ambient–moderate T	Low energy, simple operation	Fouling, metal crossover	Medium
Electrodialysis	Electric-field ion migration	Low–moderate T	Selective acid recovery	Energy demand, scaling	Medium
BMED	Salt splitting into acid/base	Low–moderate T	Acid and base generation	Membrane cost, fouling	Low–medium
Nanofiltration	Size/charge separation	Low–moderate T	Pretreatment and polishing	Limited acid selectivity	Medium
Evaporation/crystallization	Water removal/salt crystallization	Thermal	Concentrates sulfate streams	Scaling, high energy	High
Solvent extraction	Selective impurity removal	Ambient–moderate T	Improves liquor quality	Not direct acid regeneration	Medium–high
Hybrid systems	Combined mechanisms	Site-specific	More realistic industrial route	Complex integration	Medium

Table 5 shows that no single regeneration method simultaneously maximizes acid recovery, impurity tolerance, energy efficiency, and robustness. Thermal routes tolerate complex liquors; membrane systems have higher proton selectivity but are sensitive to fouling and sulfate chemistry.

Figure 6 illustrates the main sulfuric acid regeneration pathways and their interactions with impurity removal, sulfate separation, water recovery, and residue management, highlighting the differences among thermal, membrane, solvent-extraction, evaporation, and hybrid systems.

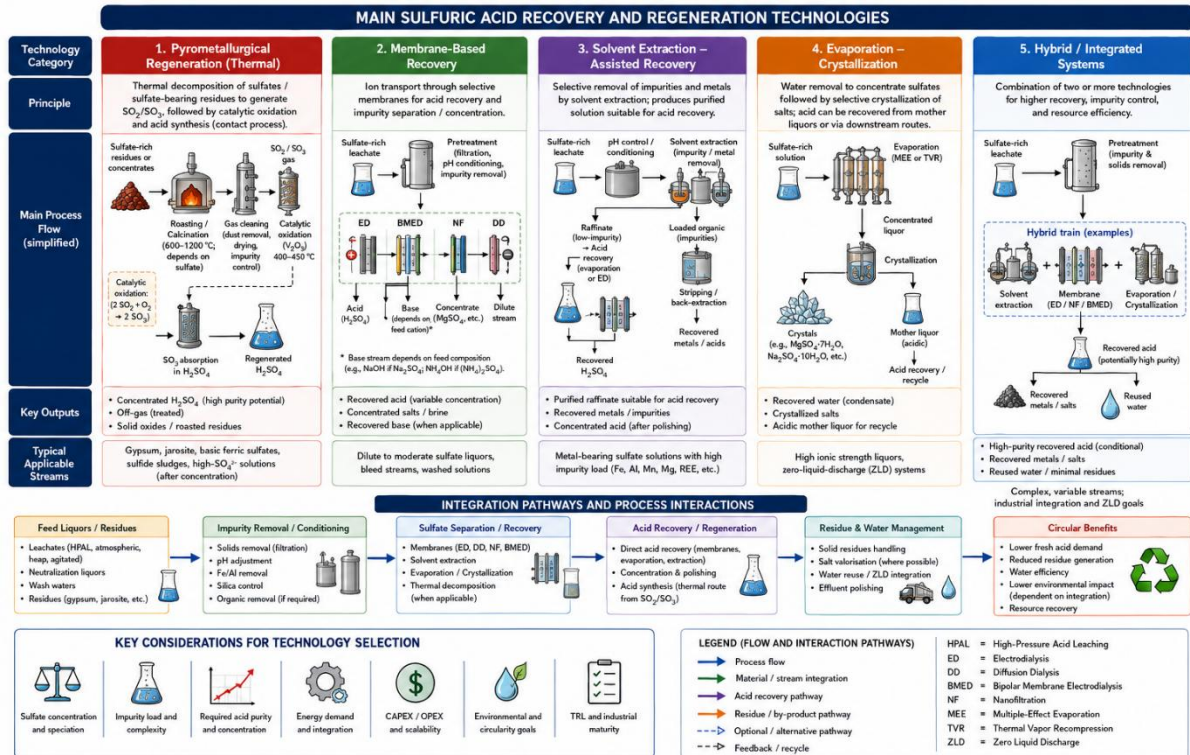


Figure 6. Main sulfuric acid regeneration technologies and integration pathways for sulfate-rich nickel laterite liquors, including pyrometallurgical regeneration, membrane-based recovery, solvent extraction, evaporation–crystallization, and hybrid recovery systems. Adapted from Hou et al. (2025), Liu et al. (2020), and Santoro et al. (2023).

Figure 6 highlights that sulfuric acid regeneration in nickel laterite hydrometallurgy should be interpreted as a multi-stage process-integration challenge rather than a single-unit operation, because sulfate speciation, impurity accumulation, scaling behavior, water balance, and residue generation collectively determine industrial feasibility.

## 8. Sulfur Dioxide Capture and Sulfuric Acid Re-Production

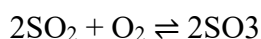
Sulfur dioxide capture and sulfuric acid re-production are mature methods for large-scale sulfur recovery. In pyrometallurgy, sulfur gases from roasting, smelting, or regeneration can be processed to produce sulfuric acid. Similarly, in nickel laterite hydrometallurgy, sulfate-rich residues or thermal regeneration often generate concentrated  $\text{SO}_2$  streams suitable for acid production.

Principal  $\text{SO}_2$  sources in sulfuric acid regeneration include thermal decomposition of metal sulfates, spray roasting, pyrohydrolysis, sulfide roasting, and combustion of sulfur fuels or residues. The stability and concentration of  $\text{SO}_2$  in the gas phase are crucial for process

feasibility. Plants need continuous, stable SO<sub>2</sub> streams as fluctuations or dilution reduce efficiency and raise costs.

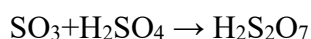
After SO<sub>2</sub> generation, gas cleaning is crucial before catalytic oxidation and acid absorption. Off-gases contain dust, metal particulates, impurities, halides, arsenic, selenium, fluorides, chlorides, and condensables that can poison catalysts or contaminate final acid. Gas cleaning systems usually include cyclones, electrostatic precipitators, scrubbers, mist eliminators, cooling, and drying towers before catalytic conversion.

The oxidation of SO<sub>2</sub> to SO<sub>3</sub> is generally performed over vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>)-based catalysts according to:



This reaction is exothermic and thermodynamically favored at lower temperatures, although reaction kinetics require intermediate operating ranges typically between 400–450 °C in industrial converters. Multi-bed catalytic reactors with intermediate cooling are commonly employed to maximize conversion efficiency while controlling temperature rise. Under optimized industrial conditions, SO<sub>2</sub> conversion efficiencies above 98–99% may be achieved in modern sulfuric acid plants.

Following catalytic oxidation, SO<sub>3</sub> is absorbed into concentrated sulfuric acid to produce additional H<sub>2</sub>SO<sub>4</sub>:



Direct water hydration is avoided industrially due to acid mist and poor absorption; instead, SO<sub>3</sub> is absorbed into concentrated sulfuric acid in packed towers.

Double Contact Double Absorption (DCDA) technology is the industry standard for high-efficiency sulfuric acid production. It involves gas streams undergoing intermediate absorption between catalytic stages, boosting SO<sub>2</sub> conversion and reducing emissions. Modern plants can exceed 99.5% SO<sub>2</sub> conversion. These systems are ideal for concentrated, continuous off-gases of sulfur, such as those from smelting or thermal regeneration.

Sulfuric acid plants, though mature, struggle with nickel laterite hydrometallurgy. Many produce sulfate-rich liquids instead of sulfur gases, needing extra thermal stages for sulfur recovery, which raises energy use, CAPEX, and complexity. Variability in sulfate content, intermittent operation, and high moisture reduce regeneration efficiency and threaten plant stability.

Impurity control is vital in sulfuric acid production from smelters, as trace contaminants such as As, Se, Hg, Cl, and F can poison catalysts, contaminate the acid, cause corrosion, and shorten equipment lifespan. Similar issues occur in sulfate decomposition systems involving industrial residues, where impurity volatility and particulate carryover increase gas-cleaning requirements (Remmen et al., 2020). Sometimes, impurity removal is among the highest costs in sulfur recovery systems.

A key limitation is the mismatch between sulfur generation and sulfuric acid demand. While producing sulfuric acid is cost-effective with continuous SO<sub>2</sub> generation, many sulfate-rich laterite streams are dilute, intermittent, or contaminated. Therefore, integrating sulfate decomposition and acid production is only feasible under specific conditions.

Table 6 summarizes the main stages of sulfur dioxide capture and sulfuric acid production, comparing operating conditions, industry maturity, efficiency, and integration challenges.

Table 6. Main stages involved in sulfur dioxide capture and sulfuric acid re-production, including SO<sub>2</sub> generation source, operating conditions, conversion mechanisms, principal impurities, industrial performance, and operational limitations. Adapted from Truong (2023) and industrial sulfuric acid production practices

Stage	Function	Typical condition	Key limitation	Industrial relevance
SO <sub>2</sub> generation	Produces sulfur-bearing gas	Roasting, smelting, sulfate decomposition	Requires concentrated feed	Essential for acid plant integration
Gas cooling	Reduces gas temperature	Before cleaning	Condensation/corrosion	Protects downstream equipment
Dust removal	Removes particulates	Cyclones, filters	ESP, Fine particulate carryover	Prevents catalyst poisoning
Gas scrubbing	Removes impurities	Wet or dry scrubbing	Wastewater generation	Controls halides and volatile species
Drying	Removes moisture	Drying tower	Acid mist control	Required before conversion
Catalytic oxidation	SO <sub>2</sub> → SO <sub>3</sub>	~400–450 °C	Catalyst poisoning	Core DCDA step
SO <sub>3</sub> absorption	Produces H <sub>2</sub> SO <sub>4</sub>	Concentrated acid tower	Mist formation	Final acid production
Acid purification	Controls contaminants	Product conditioning	As, Se, Cl, F, Hg	Determines acid quality

Table 6 shows that sulfuric acid re-production is only mature when sulfur-bearing off-gases are concentrated, continuous, and well-cleaned before catalytic oxidation. Gas cleaning

and impurity control often pose greater operational challenges than SO<sub>2</sub> oxidation, especially in complex metallurgical systems with volatile contaminants and sulfate particulates.

In Figure 7, the principal sulfur pathways associated with SO<sub>2</sub> generation, gas cleaning, catalytic oxidation, and sulfuric acid recovery are schematically illustrated to emphasize the complexity of integrating thermal regeneration systems with industrial acid plants.

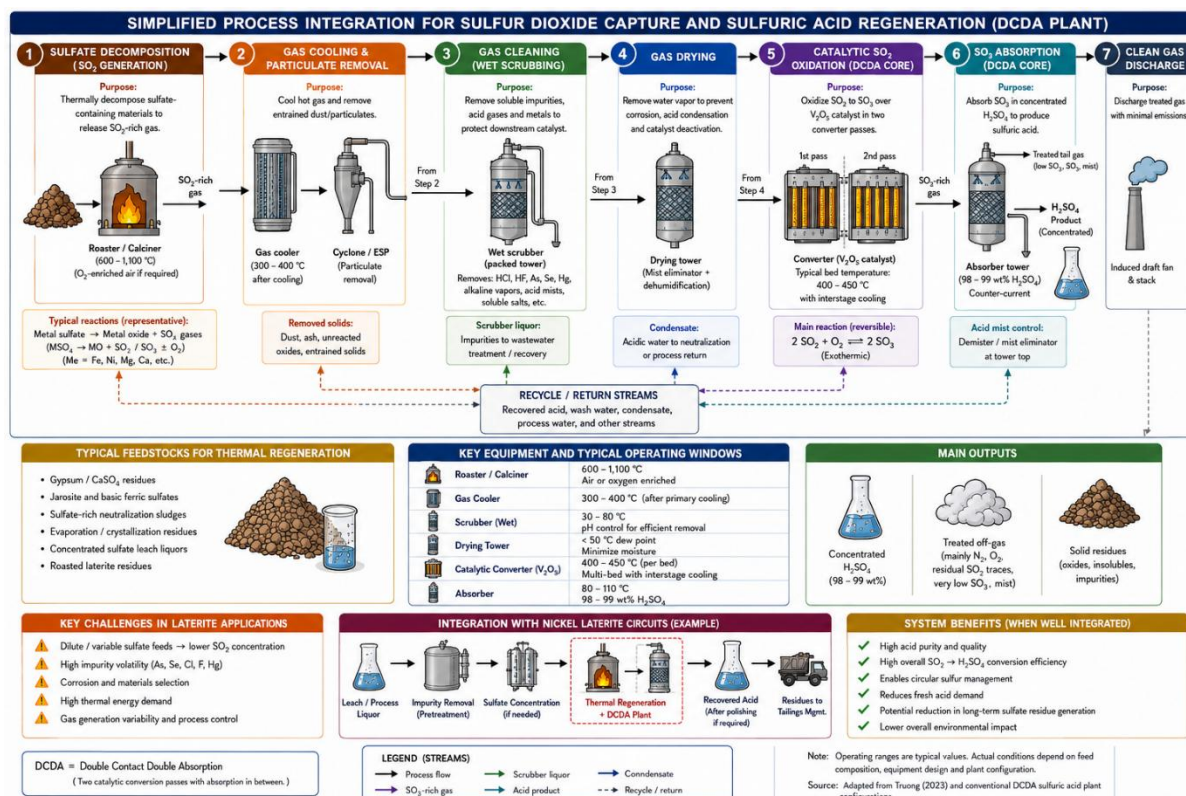


Figure 7. Simplified process integration for sulfur dioxide capture and sulfuric acid re-production, including sulfate decomposition, gas cleaning, catalytic SO<sub>2</sub> oxidation, SO<sub>3</sub> absorption, and sulfuric acid regeneration pathways. Adapted from Truong (2023) and conventional DCDA sulfuric acid plant configurations.

Figure 7 shows that sulfuric acid reproduction relies on integrated gas-handling and impurity-control systems. While DCDA technology is robust and efficient, its use in nickel laterite hydrometallurgy is limited by sulfate dilution, variable gas composition, impurity volatility, and the high thermal energy required for sulfate decomposition.

## 9. Process Integration and Circular Hydrometallurgy

Sulfuric acid regeneration in nickel laterite hydrometallurgy must be evaluated at the flowsheet level, considering interactions among leaching, separation, impurity removal, sulfate stabilization, water reuse, energy demand, and residue management. Circular hydrometallurgy

therefore requires integration between multiple unit operations rather than implementation of a single regeneration technology.

### 9.1. Circular Economy Framework for Sulfuric Acid Management

In sulfate-rich hydrometallurgical systems, the concept of circular economy should not be interpreted exclusively as complete sulfuric acid recirculation or total elimination of waste streams. Different circularity strategies involve distinct operational objectives, technological limitations, and environmental implications. Consequently, it is important to distinguish among sulfuric acid recovery, recycling, reuse, sulfate valorization, closed-loop operation, partial-loop operation, and burden shifting when evaluating regeneration technologies for nickel laterite processing.

Sulfuric acid recovery generally refers to the chemical or physical separation of recoverable acid or sulfate species from process streams. Recovery may involve membrane separation, thermal sulfate decomposition, electrodialysis, evaporation–crystallization, or sulfur dioxide capture followed by sulfuric acid re-production. In contrast, sulfuric acid recycling refers to the reintegration of recovered acid into the hydrometallurgical flowsheet for reuse in leaching, washing, or process-conditioning operations. Acid reuse may also occur without complete regeneration, for example through cascade utilization of partially spent acidic streams in lower-acidity process stages.

Sulfate valorization involves converting sulfur-rich residues into products like gypsum, magnesium sulfate, or reusing industrial by-products, instead of returning them to the hydrometallurgical circuit. It can support circularity through industrial symbiosis, even if full sulfuric acid regeneration isn't feasible (Kalupahana et al., 2025; López et al., 2023).

Closed-loop systems try to minimize sulfuric acid use and sulfate discharge via recirculation but rarely close completely because sulfur accumulates in precipitates, gypsum, MgSO<sub>4</sub>-rich liquors, and purge streams. Most rely on partial strategies like sulfate purges, bleed-stream treatment, impurity precipitation, or water replacement to stay stable.

Burden shifting transfers impacts rather than reduces them, such as water recirculation increasing MgSO<sub>4</sub> buildup, scaling, and energy use. Neutralization may control acidity but move sulfur to residues. Circular hydrometallurgy should be viewed as managing sulfur, water, impurities, energy, and residues across the entire process, not just acid recovery efficiency.

Closed acid-loop strategies aim to cut external sulfuric acid use by recycling sulfate streams, reducing reagent consumption, freshwater, sulfate discharge, and gypsum byproduct. Full acid closure is rare in industrial laterite processing since sulfur gradually builds up in ferric precipitates, gypsum, liquors, and  $\text{MgSO}_4$ -rich streams. Sustainability depends on partial sulfur recovery with purge streams and impurity control.

Water balance, acid recovery, and energy use are crucial in sulfate-rich systems. Evaporation, crystallization, thermal regeneration, and membrane separation raise energy demand, while water recirculation concentrates sulfate and impurities. Higher ionic strength causes scaling, fouling, osmotic effects, and increased membrane resistance, risking long-term stability, especially in Mg-rich laterite systems with magnesium sulfate buildup.

Integration with neutralization circuits is essential because complete sulfate recovery is rarely feasible at an industrial scale. Partial recovery strategies can lower gypsum formation and reagent use. Selective sulfate removal before lime neutralization, controlled bleed-stream treatment, and impurity precipitation can improve acid recovery and reduce sludge and sulfate discharge.

Integrated methods like membrane separation, evaporation, and crystallization are increasingly used in mining-water valorization, wastewater reuse, and ZLD systems to reduce discharge and manage sulfate, despite energy and scaling challenges (Aksenov et al., 2020; López et al., 2023). Similar trends are seen in battery recycling and effluents, where impurity control often outweighs acid recovery (Li et al., 2026; Pincay-Pilay & Carrasco, 2025).

Recent studies also indicate that hybrid systems integrating membrane separation, solvent extraction, and water recovery may improve operational flexibility and reduce sulfate discharge (Moreira et al., 2023). However, these systems remain highly sensitive to feed variability, fouling, and concentrate management. As a result, many proposed “closed-loop” systems still require purge streams to maintain impurity balance and operational stability.

Table 7 summarizes strategies for circular sulfuric acid management, including their goals, requirements, benefits, and limitations. It shows that circular hydrometallurgy relies on coordinated management of sulfur, impurities, water, and energy. Systems focused only on acid recovery can become unstable without controlling scaling, impurities, and water balance.

Table 7. Main process integration strategies for circular sulfuric acid management in nickel laterite hydrometallurgy, including closed acid loops, sulfate recycling, hybrid recovery systems, industrial

symbiosis, and ZLD-related approaches. Adapted from Aksenov et al. (2020), Kalupahana et al. (2025), López et al. (2023), and Moreira et al. (2023)

Strategy	Objective	Main requirement	Integration	Advantage	Limitation
Closed loop acid	Reduce fresh acid demand	Acid recovery	+	Lower reagent cost	Complete closure rarely feasible
Partial acid recovery	Recover acid from selected streams	Bleed-stream treatment		Practical and flexible	Does not eliminate sulfate discharge
Water recirculation	Reduce freshwater use	Salt and impurity control		Lower water demand	MgSO <sub>4</sub> accumulation
Hybrid recovery	Combine separation steps	Pretreatment membrane/thermal system	+	Better robustness	Higher complexity
Neutralization integration	Reduce gypsum/sludge	Pre-removal of sulfate/impurities		Lower residue generation	Neutralization still required
ZLD-related systems	Minimize liquid discharge	Evaporation crystallization	+	Strong water recovery	High energy and scaling
Industrial symbiosis	Reuse sulfate streams externally	Nearby compatible industries		Waste valorization	Site-specific feasibility

Table 7 demonstrates that circular hydrometallurgy depends on coordinated management of sulfur, impurities, water, and energy. Systems optimized exclusively for acid recovery may become operationally unstable if scaling, impurity accumulation, and water balance are not simultaneously controlled.

Figure 8 shows the integration relationships among leaching, acid regeneration, water recovery, neutralization, and residue management, and it is schematically illustrated to emphasize that sulfuric acid recovery must be evaluated through full-process integration.

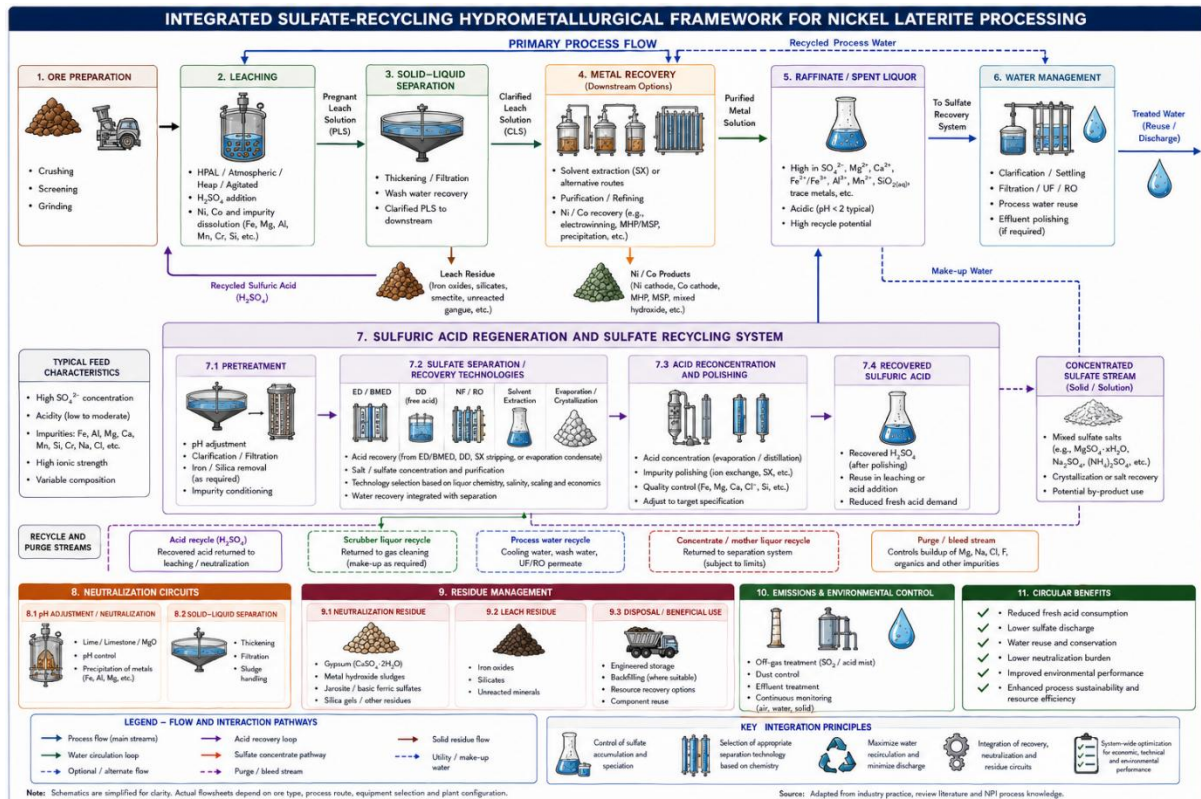


Figure 8. Integrated circular hydrometallurgical framework for nickel laterite processing, showing interactions between leaching, sulfuric acid regeneration, sulfate recycling, water recovery, neutralization circuits, and residue management pathways. Adapted from Kalupahana et al. (2025), López et al. (2023), and Moreira et al. (2023).

Figure 8 highlights that sulfuric acid regeneration is fundamentally an integration challenge that involves the simultaneous control of sulfate accumulation, impurity buildup, water recirculation, and residue stabilization throughout the hydrometallurgical flowsheet.

## 10. Environmental and Sustainability Challenges

Environmental performance has become a critical factor in evaluating sulfuric acid management in nickel laterite hydrometallurgy. Acid consumption is not only an operational cost; sulfate-rich streams are also directly associated with water consumption, waste generation, atmospheric emissions, and long-term environmental liabilities. Consequently, sulfuric acid regeneration must be evaluated not only by acid recovery efficiency but also by energy demand, sulfate discharge, residue stability, membrane lifetime, and secondary waste generation.

Sulfate-bearing effluents pose a major environmental challenge in laterite processing. Even after removing dissolved metals, sulfate concentrations may remain high enough to affect

groundwater quality, aquatic systems, and water reuse. In many cases, sulfur is transferred to saline wastewater or gypsum-rich residues during neutralization rather than being effectively recovered. Similar limitations are observed in acid mine drainage (AMD) treatment, where sulfate management is often more difficult than metal precipitation (Baloyi et al., 2024; Ngerem et al., 2026).

Atmospheric emissions associated with thermal regeneration and sulfuric acid production also raise sustainability concerns. Acid mist, SO<sub>2</sub>, and SO<sub>3</sub> emissions may contribute to acidification, corrosion, and occupational exposure risks. Although modern gas-cleaning systems and DCDA plants significantly reduce emissions, environmental performance still depends strongly on sulfur capture efficiency, energy consumption, and operational stability.

Tailings management and sulfate stabilization remain important challenges because neutralization generates gypsum sludge, ferric precipitates, and sulfate-bearing residues requiring long-term storage. Furthermore, extensive water recirculation tends to concentrate MgSO<sub>4</sub>, dissolved salts, and impurities, thereby increasing scaling, osmotic resistance, and treatment complexity.

The carbon footprint is another critical parameter when comparing regeneration technologies. Thermal routes frequently provide robust sulfur recovery but require high energy input, whereas membrane systems reduce thermal demand but increase electricity consumption, chemical use, and membrane replacement. Secondary waste generation must also be considered because membrane concentrates, degraded organics, and low-value sulfate salts may require additional treatment (Hegab et al., 2020; Ordóñez et al., 2022). Consequently, sustainability assessments should prioritize long-term environmental stability rather than short-term acid recovery efficiency alone (Shin, 2020).

Table 8 summarizes key environmental and sustainability challenges in sulfuric acid management for nickel laterite hydrometallurgy, highlighting their drivers, impacts, and mitigation limits.

Table 8. Main environmental and sustainability challenges associated with the use and regeneration of sulfuric acid in nickel laterite hydrometallurgy, including sulfate effluents, SO<sub>x</sub> emissions, gypsum generation, water consumption, carbon footprint, and secondary waste formation. Adapted from Baloyi et al. (2024), Hegab et al. (2020), Ngerem et al. (2026), and Pincay-Pilay and Carrasco (2025).

Challenge	Main source	Environmental impact	Mitigation route	Remaining limitation
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Sulfate effluents	Neutralized process waters	Salinity, groundwater impact	Recovery, controlled	purge	Sulfate is difficult to remove
SO <sub>x</sub> and acid mist	Thermal regeneration, acid plants	Atmospheric emissions, corrosion	Gas cleaning, DCDA		Requires stable gas feed
Gypsum generation	Lime/limestone neutralization	Tailings volume increase	Sulfate pre-removal		Acid value destroyed
Tailings burden	Neutralization residues	Long-term storage liability	Better residue washing/stabilization		Persistent sulfate phases
Water consumption	Washing, slurry transport, cooling	Freshwater demand	Recirculation		Salt buildup
Carbon footprint	Thermal and electrochemical systems	Energy-related emissions	Waste heat, renewable electricity		Energy intensity remains critical
Secondary wastes	Membrane concentrates, spent solvents	Additional disposal need	Hybrid treatment		Burden transfer risk
Membrane replacement	Fouling/aging	Material and cost impact	Pretreatment, cleaning		Long-term durability uncertain

Table 8 demonstrates that environmental performance depends on multiple interconnected variables rather than acid recovery efficiency alone. Technologies minimizing sulfate discharge may simultaneously increase energy demand, concentrate generation, or scaling intensity.

Figure 9 schematically illustrates the main environmental pathways of sulfuric acid use, regeneration, sulfate stabilization, emissions, residue generation, and water recirculation, highlighting their interconnected impacts in laterite hydrometallurgy.

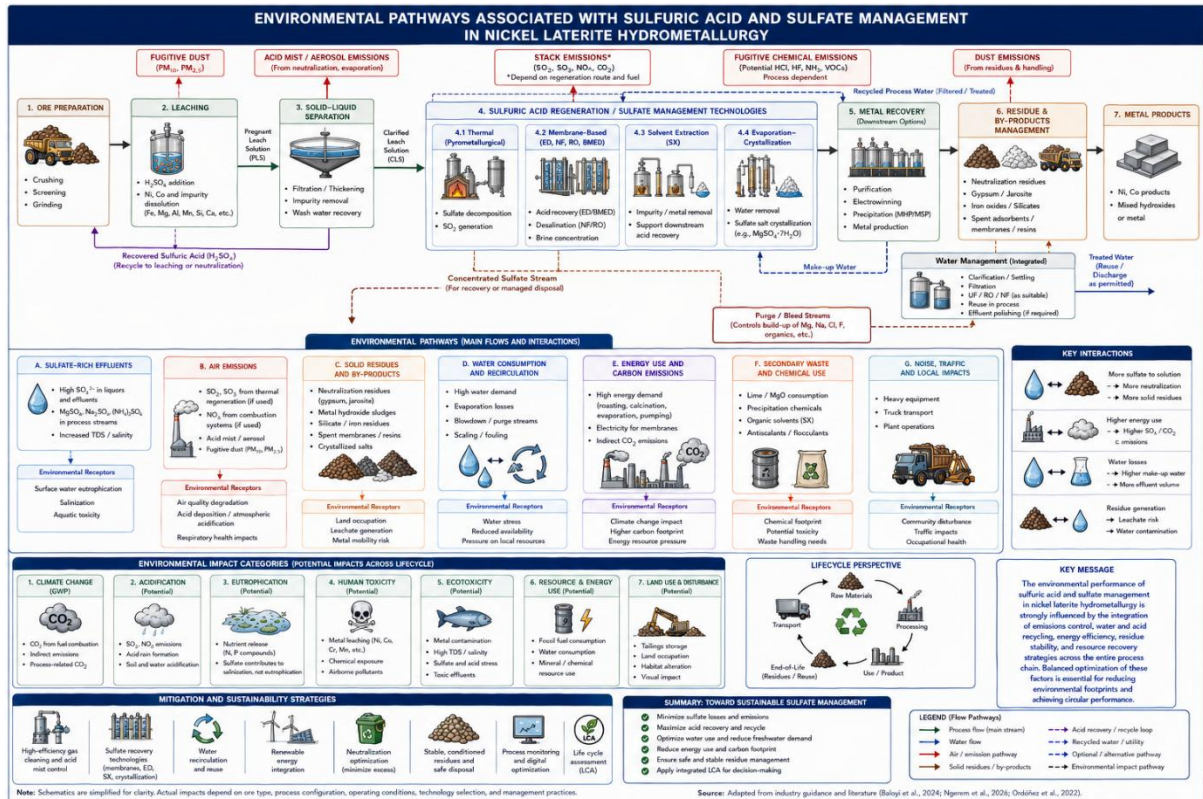


Figure 9. Environmental pathways associated with sulfuric acid management in nickel laterite hydrometallurgy, including sulfate effluents, SO<sub>x</sub> emissions, gypsum disposal, water recirculation, secondary waste generation, and carbon-emission contributions from regeneration technologies. Adapted from Baloyi et al. (2024), Ngerem et al. (2026), and Ordóñez et al. (2022).

Figure 9 shows that sulfuric acid management should be assessed through an integrated life cycle analysis rather than individual recovery metrics, because sulfate stabilization, emissions, residues, water reuse, and energy use together shape environmental sustainability.

## 11. Thermodynamic, Kinetic, and Predictive Modeling

Thermodynamic, kinetic, and predictive models play an important role in evaluating sulfuric acid regeneration in nickel laterite hydrometallurgy. Equilibrium calculations, sulfate stability diagrams, membrane transport models, CFD simulations, reactive transport analysis, and AI-assisted prediction are increasingly used to estimate acid recovery, precipitation, scaling, and process feasibility. However, industrial regeneration systems cannot be fully described by equilibrium thermodynamics because most industrial operations occur under non-ideal and transient conditions.

Thermodynamic models are widely applied to predict sulfate stability, gypsum formation, ferric hydrolysis, sulfate complexation, and MgSO<sub>4</sub> accumulation under different

operating conditions. Nevertheless, these approaches frequently overestimate industrial separation efficiency because many sulfate-rich systems do not reach true equilibrium during operation. In laterite liquors containing silica, suspended solids, and multicomponent ions, factors such as residence time, mixing, supersaturation, and reaction kinetics strongly influence performance.

Kinetic modeling is particularly important because precipitation, membrane fouling, sulfate crystallization, gypsum scaling, and metal hydrolysis evolve dynamically through nucleation, diffusion, adsorption, and transport processes. In membrane systems, proton transport may initially appear highly selective but gradually declines due to fouling layers, concentration polarization, and membrane aging.

Electrochemical and membrane transport models are especially relevant for electrodialysis and bipolar membrane systems. Recent studies demonstrate that acid recovery depends strongly on ionic transport phenomena rather than simple equilibrium partitioning (Falina et al., 2023; He et al., 2024). Water transport, osmotic drag, membrane hydration, and multivalent ions significantly influence proton selectivity and recovery efficiency. Although laboratory-scale membrane performance is often promising, industrial systems frequently suffer from scaling, iron precipitation, silica fouling, and membrane degradation (Jia et al., 2025; Zhu et al., 2024).

CFD, reactive transport, and AI-assisted models are increasingly used to simulate industrial hydrodynamics, concentration gradients, fouling evolution, and process instability. However, many predictive models remain limited by the scarcity of long-term industrial data and the frequent use of simplified laboratory-scale solutions rather than complex industrial liquors.

Table 9 summarizes the main predictive methods for sulfuric acid regeneration and sulfate management, covering their scope, applicability, advantages, and limitations.

Table 9. Comparative analysis of thermodynamic, kinetic, electrochemical, CFD, reactive transport, and AI-assisted modeling approaches applied to sulfuric acid regeneration and sulfate management in hydrometallurgical systems. Adapted from Falina et al. (2023), He et al. (2024), Jia et al. (2025), Tekinalp et al. (2023), Wright and Kanan (2025), and Zhu et al. (2024).

Modeling approach	Main application	Strength	Limitation	Best use
Thermodynamic equilibrium	Sulfate stability, precipitation	Identifies feasible phases	May overpredict performance	Screening of operating windows

Kinetic modeling	Reaction and precipitation rates	Captures transient behavior	Requires kinetic data	kinetic	Scaling and hydrolysis prediction
Electrochemical modeling	ED/BMED systems	Describes ion migration	Sensitive to membrane parameters	to	Proton transport analysis
Membrane transport models	Flux, selectivity, resistance	Predicts membrane performance	Fouling difficult to model		Design and optimization
CFD	Mixing, RTD, dead zones	Captures hydrodynamics	Computationally intensive		Reactor/module design
Reactive transport	Coupled chemistry and flow	Realistic process behavior	Data-intensive		Continuous sulfate circuits
AI/ML	Pattern recognition, control	Useful for dynamic prediction	Needs industrial datasets	for industrial	Fouling and process monitoring

Table 9 shows that no single predictive framework fully captures industrial acid regeneration systems. Thermodynamic models predict sulfate stability, while kinetic and transport models better describe fouling, membrane aging, and hydrodynamics.

Figure 10 schematically shows the hierarchy and interactions among thermodynamic, kinetic, transport, electrochemical, and AI-assisted frameworks, highlighting how industrial acid regeneration relies on coupled physicochemical and hydrodynamic phenomena.



Figure 10. Hierarchy of predictive approaches applied to sulfuric acid regeneration systems, including thermodynamic equilibrium, kinetic modeling, electrochemical transport, CFD, reactive transport simulation, and AI-assisted process prediction. Adapted from Falina et al. (2023), Jia et al. (2025), and Tekinalp et al. (2023).

Figure 10 highlights that industrial sulfuric acid regeneration cannot be accurately evaluated using equilibrium diagrams alone because fouling evolution, membrane degradation, impurity transport, and hydrodynamic limitations frequently dominate long-term process performance.

## 12. Comparative Critical Analysis

The evaluation indicates that no single sulfuric acid management technology simultaneously maximizes acid recovery, minimizes environmental impact, reduces energy demand, and maintains operational robustness under all conditions. Performance strongly depends on sulfate concentration, impurity composition, water balance, and energy availability, while laboratory-scale efficiencies frequently decline in complex industrial liquors.

Regeneration and neutralization fulfill different industrial functions. Neutralization remains operationally robust, technologically mature, and relatively simple at industrial scale, rapidly handling process variability but irreversibly converting sulfuric acid into gypsum and sulfate-bearing residues. In contrast, regeneration technologies preserve sulfur value and reduce sulfate discharge, although they generally require higher CAPEX, stricter operational control, and greater sensitivity to fouling and scaling. Consequently, regeneration should be viewed as complementary to neutralization rather than as a complete replacement.

Thermal regeneration and membrane recovery are primary methods for sulfuric acid recovery. Pyrometallurgical regeneration handles sulfate-rich streams with high Fe, Mg, silica, and solids, especially when linked to sulfuric acid plants or waste-heat systems. These require high temperatures, complex off-gas treatment, and corrosion-resistant materials. Membrane systems work at lower temperatures and show high proton selectivity in dilute-to-moderate sulfate streams, but industrial use faces challenges like fouling, silica buildup, iron precipitation, polarization, and membrane aging. Laboratory tests show 70–90% acid recovery, but real-world results are often lower (Janiszewska et al., 2020; Wu et al., 2023).

Industrial flowsheets increasingly favor semi-integrated hybrid approaches combining membrane separation, solvent extraction, crystallization, thermal regeneration, water reuse, and controlled neutralization. Although membrane systems can recover acid from moderate-

sulfate streams when impurity levels remain controlled (Knežević et al., 2022), higher impurity concentrations reduce selectivity and operational stability (Ge et al., 2020). Conventional neutralization and sulfuric acid plants are fully mature, whereas selective electrodialysis and hybrid recovery systems remain at pilot or early industrial scale. Consequently, high laboratory recovery efficiency does not necessarily guarantee industrial feasibility.

Table 10 summarizes the main sulfuric acid management strategies for nickel laterite hydrometallurgy, based on operational, environmental, economic, and technological criteria.

Table 10. Comparative critical assessment of sulfuric acid management technologies for nickel laterite hydrometallurgy, including regeneration and neutralization routes, evaluated according to acid recovery, energy demand, emissions, CAPEX, OPEX, TRL, impurity tolerance, operational flexibility, and environmental implications. Adapted from Agarwal and Pandey (2023), Ibrahim et al. (2024), Janiszewska et al. (2020), Moreira et al. (2022a), and Wu et al. (2023).

Criterion	Neutralization	Thermal regeneration	Membrane recovery	Solvent extraction	Evaporation/crystallization	Hybrid systems
Acid recovery	None	High if gas recovery integrated	Moderate to high	Indirect	Indirect	Moderate to high
Energy demand	Low–moderate	High	Low–moderate	Moderate	High	Variable
CAPEX	Low–moderate	High	Moderate	Moderate	Moderate–high	High
OPEX	Reagent/slugge driven	Energy driven	Membrane/electricity driven	Solvent driven	Energy/scale driven	Site-specific
Impurity tolerance	High	High	Low–moderate	Moderate	Low–moderate	Moderate
TRL	High	Medium–high	Medium	Medium–high	High	Medium
Main limitation	Gypsum/slugge	Energy/of f-gas treatment	Fouling and aging	Not direct regeneration	Scaling	Integration complexity
Best application	Final acidity control	Concentrated sulfate streams	Pretreated dilute/moderate acid streams	Impurity removal	Concentration/salt recovery	Industrial sulfate management

Table 10 demonstrates that technology selection depends strongly on sulfate concentration, impurity chemistry, water balance, and integration strategy. Thermal systems

generally exhibit greater robustness toward complex sulfate streams, whereas membrane systems may achieve higher selectivity in cleaner liquors.

Figure 11 schematically illustrates the principal trade-offs among sulfuric acid management routes, emphasizing the relationships among acid recovery efficiency, operational robustness, impurity tolerance, energy demand, and industrial applicability.

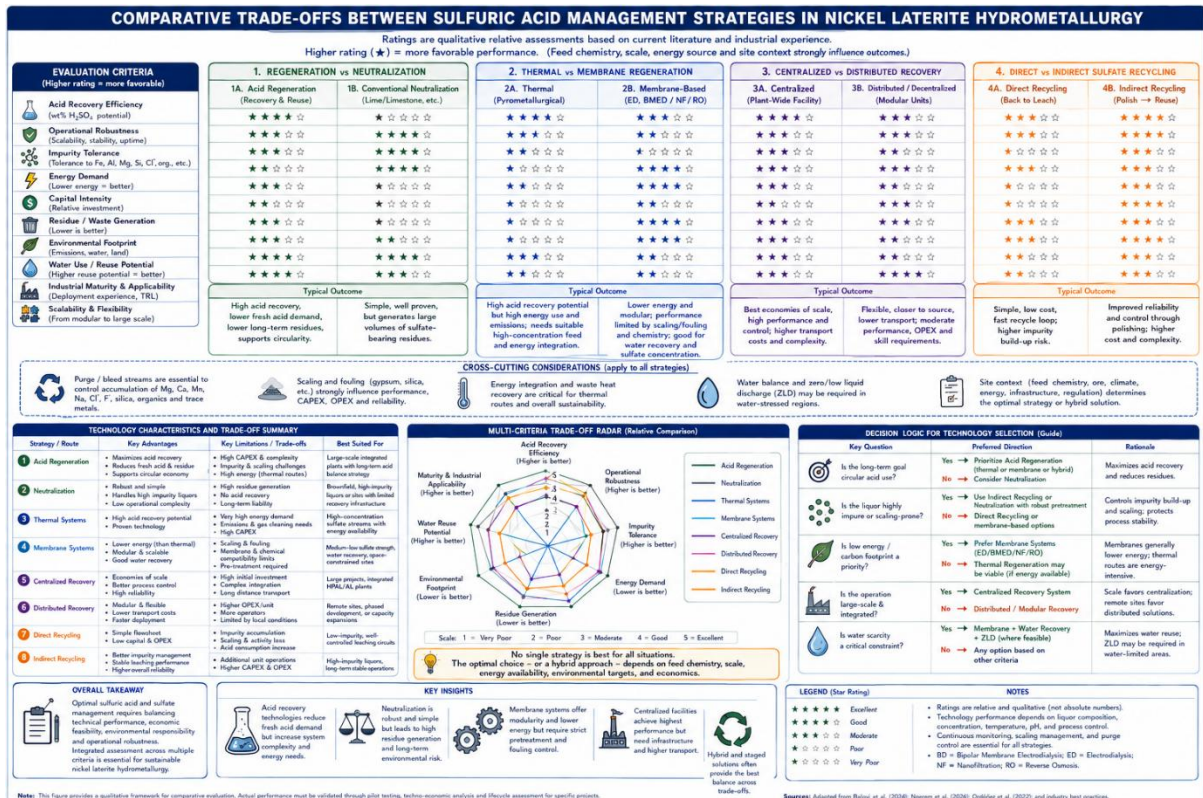


Figure 11. Comparative trade-offs between sulfuric acid management strategies in nickel laterite hydrometallurgy, including regeneration versus neutralization, thermal versus membrane systems, centralized versus distributed recovery, and direct versus indirect sulfate recycling pathways. Adapted from Fanimoghdam et al. (2022), Ibrahim et al. (2024), and Moreira et al. (2022a).

Figure 11 shows sulfuric acid management balances multiple criteria, not just maximizing one. High-acid-recovery technologies may require more energy or handle impurities poorly, while neutralization systems can stabilize acidity but produce sulfate residues and pose environmental challenges.

### 13. Industrial Case Studies

Industrial implementation challenges in sulfuric acid regeneration for nickel laterite hydrometallurgy include sulfate accumulation, membrane lifespan, purge-stream management, scaling, and economic feasibility. Although many regeneration technologies show promising

laboratory-scale performance, industrial validation remains limited, and feasibility should therefore be evaluated using pilot-scale or commercial sulfate-rich systems with comparable operational complexity.

HPAL operations such as Moa Bay, Murrin Murrin, Coral Bay, Ramu, and Ravensthorpe consume large quantities of sulfuric acid during the processing of limonitic laterite. Acid consumption may reach several hundred kilograms of H<sub>2</sub>SO<sub>4</sub> per tonne of ore and is strongly influenced by ore mineralogy, Mg dissolution, iron hydrolysis, and neutralization requirements. Consequently, sulfuric acid represents both a major operational cost and an environmental challenge due to sulfate-rich residues and sludges.

Despite the maturity of HPAL itself, most regeneration technologies remain at intermediate technology readiness levels (TRLs). Conventional neutralization and sulfuric acid plants operate at TRL 9, whereas electro dialysis, membrane recovery, and hybrid systems generally remain between TRL 4 and 7. Industrial experience at Ravensthorpe demonstrated that actual acid demand may differ significantly from laboratory predictions because of ore heterogeneity, scaling, and hydrodynamic limitations.

Studies from analogous sulfate-rich industries provide additional insight. Hybrid membrane systems in steel-wire production achieved partial acid recovery, although fouling and concentrate management remained problematic (Loza et al., 2022). Similar limitations were observed in mining-water recovery systems, battery recycling, and aluminum-finishing wastewater treatment, where impurities, scaling, and membrane degradation reduced long-term stability (Guo et al., 2025; Moreira et al., 2022a; Wu et al., 2023; Yuzer et al., 2022). Industrial evidence therefore indicates that sulfuric acid recovery alone is insufficient without integrated water recovery, impurity control, neutralization, and residue management.

Table 11 summarizes key industrial and pilot case studies on sulfuric acid management and regeneration, covering process type, sulfate strategy, performance, maturity, and limitations.

Table 11. Representative industrial and pilot-scale case studies related to sulfuric acid management and regeneration in nickel laterite processing and analogous sulfate-rich industrial systems, including process characteristics, recovery strategy, TRL, operational performance, and principal limitations. Adapted from Loza et al. (2022), Moreira et al. (2022a), Moreira et al. (2023), Santoro et al. (2023), Wu et al. (2023), and Yuzer et al. (2022).

System/case	Process type	Acid/sulfate issue	Recovery strategy	TRL	Key limitation
Moa Bay	HPAL laterite	High acid demand, sulfate residues	Neutralization, acid supply control	9	Limited sulfur closure
Murrin Murrin	HPAL laterite	Acid logistics, residue generation	Integrated acid management	9	Neutralization burden
Coral Bay	HPAL laterite	Sulfate-rich liquors	Water reuse and neutralization	9	Gypsum/residue management
Ramu	HPAL laterite	Acid supply and sulfate balance	Integrated process control	9	Partial recycling only
Ravensthorpe	Laterite hydrometallurgy	Ore variability, scaling	Process modification	8–9	Scale-up sensitivity
Coated steel wire wastewater	Acidic industrial liquor	Spent acid	DD + ED hybrid system	6–7	Fouling/concentrate management
Gold mining wastewater	Acidic sulfate wastewater	Acid + metal-bearing stream	Membrane distillation + SX	4–6	Scaling and concentrate disposal
Battery recycling wastewater	H <sub>2</sub> SO <sub>4</sub> /NiSO <sub>4</sub> stream	Acid and metal recovery	DD/ED	4–6	Impurity load
Aluminum finishing wastewater	Acidic industrial effluent	Acid recovery and water reuse	Electrodialysis	6–8	Membrane degradation
Copper mining waters	Sulfate-rich mining water	Water and acid valorization	Integrated membranes	4–7	Feed variability

Table 11 shows that mature sulfuric acid systems mainly use neutralization and integrated production rather than full regeneration. Membrane and hybrid systems perform well in pilots but face long-term fouling, impurities, scaling, and concentrate-handling issues.

Figure 12 outlines key industrial paths for sulfuric acid in nickel laterite processing and similar systems, comparing their maturity and regeneration complexity.

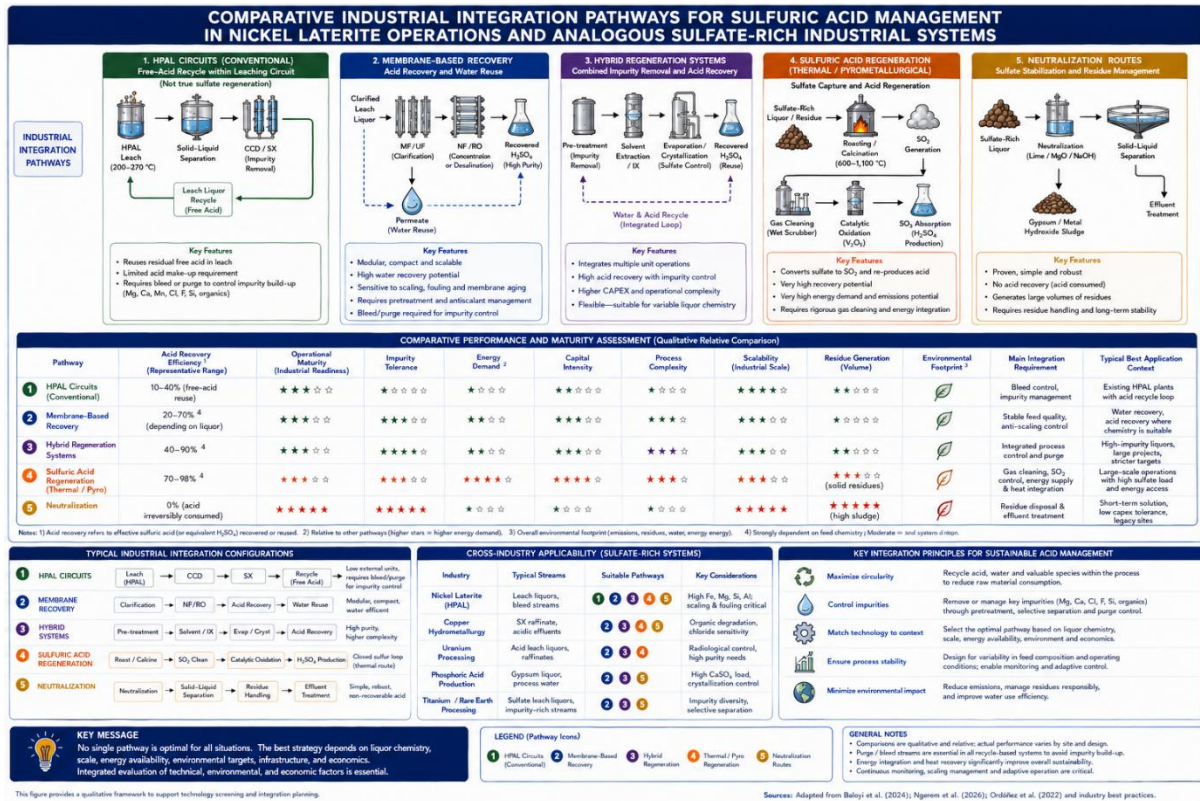


Figure 12. Comparative industrial integration pathways for sulfuric acid management in nickel laterite operations and analogous sulfate-rich industrial systems, including HPAL circuits, membrane-based recovery, hybrid regeneration systems, sulfuric acid re-production, and neutralization routes. Adapted from Loza et al. (2022), Santoro et al. (2023), and Moreira et al. (2023).

Figure 12 shows that sulfuric acid regeneration depends on process integration, impurity control, and stability. Technologies with high laboratory-scale recovery often face scale-up issues such as sulfate buildup, membrane aging, and changes in liquor composition.

## 14. Research Gaps and Future Perspectives

Despite advances in sulfuric acid recovery, challenges in nickel laterite hydrometallurgy persist. Many regeneration systems, designed for cleaner streams, struggle with liquors high in Mg, Fe, Al, silica, solids, and variable acid concentrations. Future research should prioritize integrated industrial operability and long-term stability over isolated laboratory-scale tests.

A key unresolved issue is regenerating MgSO<sub>4</sub>-rich liquors, as managing sulfate buildup reduces acid recovery, causes scaling, and destabilizes water systems. Existing separation methods fall short, and no mature technology efficiently regenerates sulfuric acid

from concentrated  $\text{MgSO}_4$  streams in laterite processing. Recovering magnesium sulfate from dilute liquors is also difficult due to poor membrane selectivity, fouling, and the use of costly thermal methods.

Chemically resistant membranes are vital for research, but long-term exposure to acidic sulfate liquors with Fe, silica, chlorides, and oxidants degrades their structure and selectivity. Despite recent advances (Ibrahim et al., 2024), aging, swelling, fouling, and loss of proton selectivity under industrial conditions remain poorly understood. Fouling control is challenging; many studies focus on recovery efficiency but neglect scaling, gypsum deposits, colloidal transport, and hydrodynamic instability during extended use (Xie et al., 2026).

Hybrid process design is a key research area. Stand-alone technologies rarely meet all industrial criteria. Future systems will combine membrane separation, precipitation, crystallization, electrochemical recovery, thermal concentration, and water recirculation in integrated flowsheets. The challenge is optimizing these combinations to balance acid recovery, energy use, impurity tolerance, and flexibility.

Electrification and electrochemical acid/base generation offer potential advances, with studies showing that concentrated acids and bases can be electrochemically produced from neutral salts at high current density and moderate energy use (Wright & Kanan, 2025). These systems aid sulfate management and metal recovery in battery recycling and industries (Garrido et al., 2025; Sun et al., 2025). Challenges include membrane durability, energy consumption, and scale-up obstacles.

Integration with zero liquid discharge (ZLD) systems is a key future direction. Growing environmental restrictions and water scarcity will boost interest in sulfate-management systems that combine acid recovery, water reuse, crystallization, and residue minimization. AI and dynamic process control may also become vital for predicting fouling, membrane degradation, sulfate buildup, and operational issues with real-time data.

Another opportunity links sulfuric acid recovery with critical mineral recycling. Battery recycling, copper refining, rare earth processing, and mining wastewater produce sulfate-rich streams that could serve as secondary sulfur resources rather than waste. This convergence may boost modular sulfur-management technologies.

Table 12 summarizes the main research gaps and future directions for sulfuric acid regeneration in nickel laterite hydrometallurgy, considering technological maturity, industrial relevance, operational barriers, and development potential.

Table 12. Principal research gaps and future technological directions for sulfuric acid regeneration and sulfate management in nickel laterite hydrometallurgy, including  $MgSO_4$  regeneration, selective recovery from dilute liquors, membrane durability, hybrid systems, electrification, ZLD integration, and AI-assisted process control. Adapted from Garrido et al. (2025), Ibrahim et al. (2024), Sun et al. (2025), and Wright and Kanan (2025).

Research gap	Current limitation	Future direction	Industrial relevance
MgSO <sub>4</sub> -rich liquor regeneration	MgSO <sub>4</sub> remains highly soluble and difficult to split	Selective separation, electrochemical routes	Critical for saprolitic laterites
Dilute acid recovery	Low acid strength increases energy and selectivity challenges	Improved membranes and hybrid concentration	Important for bleed streams
Membrane durability	Fouling, swelling, oxidation, aging	Chemically resistant and self-cleaning membranes	Essential for scale-up
Fouling control	Silica, Fe, gypsum, colloids	Pretreatment, monitoring, dynamic cleaning	Determines long-term uptime
Hybrid flowsheets	Stand-alone units underperform	Integrated membrane + SX + crystallization	Most realistic near-term route
Electrification	Energy and membrane constraints	Renewable-powered ED/BMED	Supports decarbonization
ZLD integration	High energy and scaling	Selective recovery before final concentration	Relevant under strict water rules
AI-assisted control	Lack of industrial datasets	Predictive control and fouling diagnostics	Supports stable operation
Critical-mineral recycling integration	Fragmented acid recovery systems	Shared acid/sulfate loops	Supports circular hydrometallurgy
Industrial validation	A few long-term pilot datasets	Demonstration plants and techno-economic studies	Required for commercial adoption

Table 12 shows that future sulfuric acid regeneration progress will rely more on integrated advances in fouling control, impurity tolerance, energy use, membrane durability, and operational stability than on isolated recovery efficiency improvements.

Figure 13 depicts key future sulfuric acid management pathways, emphasizing circular hydrometallurgy, electrification, water reuse, and mineral recycling.

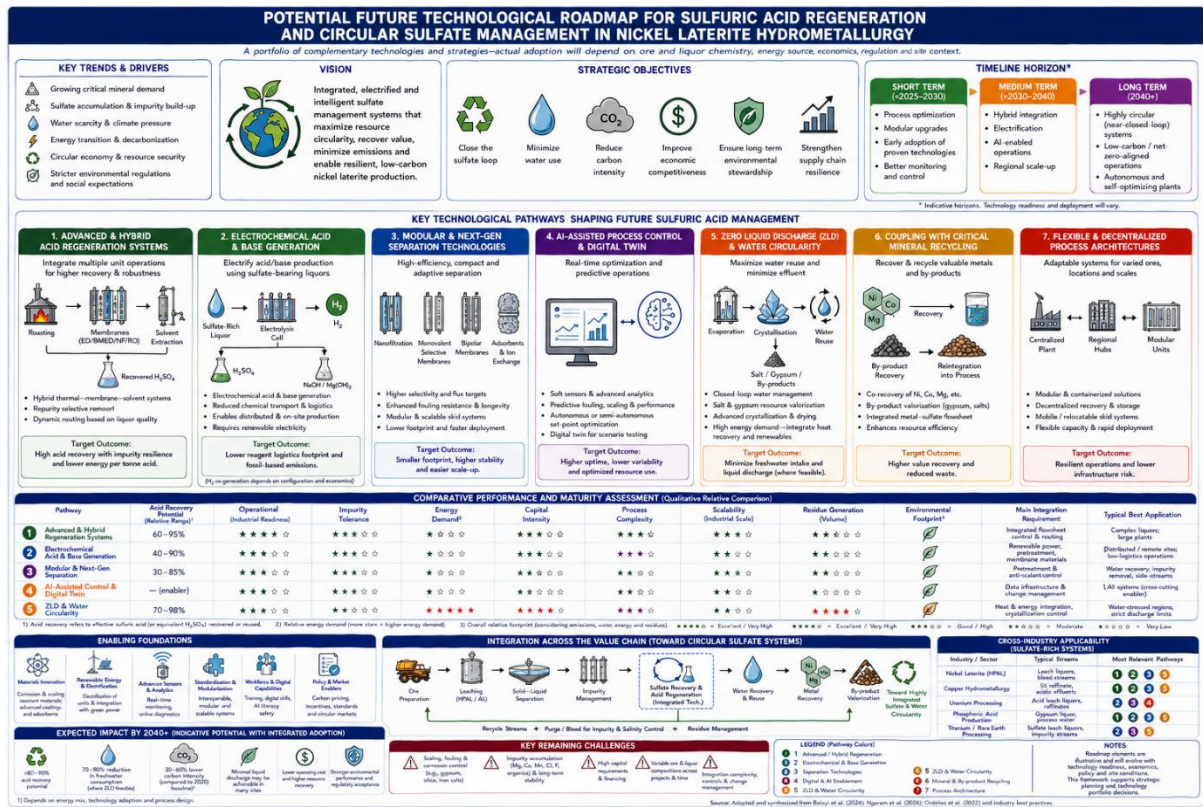


Figure 13. Future technological roadmap for sulfuric acid regeneration and circular sulfate management in nickel laterite hydrometallurgy, including hybrid recovery systems, electrochemical acid/base generation, AI-assisted control, modular membrane technologies, ZLD integration, and coupling with critical-mineral recycling. Adapted from Garrido et al. (2025), Lin et al. (2023), Sun et al. (2025), and Wright and Kanan (2025).

Figure 13 indicates that future sulfuric acid management systems will likely evolve toward integrated, adaptive flowsheets rather than isolated regeneration units. Electrification, hybrid separation technologies, modular process design, and AI-assisted operation may improve sulfate circularity, although challenges related to scaling, impurity accumulation, membrane lifetime, and industrial economics remain unresolved.

Future research on sulfuric acid regeneration in nickel laterite hydrometallurgy should prioritize industrial applicability over laboratory-scale recovery efficiency alone. Although numerous studies report promising acid recovery under controlled conditions, significant uncertainties remain about long-term operational stability, scaling behavior, impurity accumulation, and economic feasibility in industrial sulfate-rich liquors.

One of the principal research priorities involves pilot-scale and industrial-scale validation of regeneration technologies under realistic operating conditions. Many membrane, electro dialysis, and hybrid recovery systems have only been evaluated using synthetic or simplified laboratory solutions, which frequently underestimate fouling, silica deposition, iron precipitation, membrane degradation, and concentrate-management challenges. Long-duration pilot campaigns using real industrial liquors are therefore necessary to evaluate membrane lifetime, cleaning frequency, operational stability, and process economics under fluctuating feed conditions.

Additional research is needed on  $\text{MgSO}_4$  accumulation and sulfate management in recirculating laterite circuits. Magnesium sulfate remains a major barrier to closed-loop sulfuric acid recycling because its high solubility progressively increases ionic strength, osmotic resistance, scaling tendency, and water-treatment complexity. Future studies should therefore investigate selective Mg removal, sulfate valorization routes, hybrid crystallization systems, and controlled purge strategies to improve long-term process stability.

Dynamic process modeling also represents an important research frontier. Current thermodynamic models frequently overestimate regeneration performance because they neglect transient hydrodynamics, non-equilibrium precipitation, fouling evolution, and multicomponent transport phenomena. Consequently, future work should increasingly integrate CFD, reactive transport simulation, electrochemical transport models, and sulfate-speciation analysis into coupled predictive frameworks.

Artificial intelligence and machine-learning tools also show significant potential for process monitoring, fouling prediction, and operational optimization. However, most existing AI-assisted studies remain based on simplified laboratory datasets. The development of reliable predictive systems will therefore require long-term industrial operational databases incorporating sulfate concentration, impurity levels, membrane resistance, scaling evolution, water balance, and energy consumption.

Finally, future industrial flowsheets will likely depend on semi-integrated hybrid systems combining membrane separation, thermal regeneration, crystallization, water recovery, impurity control, and selective neutralization. As a result, future research should focus less on identifying a universal regeneration technology and more on understanding how

different recovery mechanisms can be integrated into operationally stable and economically viable sulfate-management strategies for complex nickel laterite circuits.

## 15. Conclusions

Nickel laterite hydrometallurgy relies heavily on sulfuric acid-based methods such as HPAL, atmospheric leaching, and hybrid sulfate systems. While these enable large-scale nickel and cobalt production, they generate sulfate-rich liquors and residues, along with environmental issues. Managing sulfuric acid affects not only reagent use but also water needs, impurity buildup, residue stability, energy use, and overall sustainability.

Conventional neutralization is reliable, robust, and mature. Lime-, limestone-, and Mg-based systems stabilize acidic liquors and metals but are not circular because they irreversibly consume sulfuric acid and transfer sulfur into residues. As environmental and water-reuse restrictions tighten, the limitations of neutralization strategies will grow more significant.

Sulfuric acid regeneration and sulfate recycling can significantly reduce acid use, neutralization, sulfate discharge, freshwater needs, and environmental impact. However, industrial viability depends on sulfate levels, impurities, membrane stability, fouling, hydrodynamics, energy, and plant integration. Laboratory-scale, cost-effective technologies may face challenges with industrial liquors containing Mg, Fe, Al, silica, solids, and variable sulfate levels.

Thermal regeneration systems tolerate complex sulfate streams and integrate with sulfuric acid plants and waste-heat recovery systems. They require high-energy, high-temp materials and complex off-gas treatment. Membrane technologies can offer good acid selectivity in dilute to moderate sulfate conditions but suffer from fouling, scaling, aging, and impurity crossover. Solvent extraction, crystallization, and electrochemical systems are better seen as supplementary purification or concentration steps rather than standalone regeneration solutions.

The review also highlights that sulfuric acid regeneration should not be evaluated as an isolated unit operation. Industrially viable systems will likely depend on coordinated integration between leaching, impurity removal, sulfate management, water recovery, residue stabilization, and energy optimization. Complete sulfur closure appears unlikely under most current industrial laterite conditions, particularly in  $\text{MgSO}_4$ -rich systems. Consequently, partial

acid recovery combined with controlled purge streams and hybrid process integration presently represents the most realistic pathway toward improved circularity.

Future progress will depend on advances in chemically resistant membranes, fouling control, hybrid regeneration systems, electrification, AI-assisted process control, and integrated water–acid–energy management. Greater availability of long-term industrial datasets will also be essential, as many current technologies are validated primarily under laboratory or pilot-scale conditions.

Overall, sulfuric acid regeneration in nickel laterite hydrometallurgy should be interpreted as a gradual transition toward more integrated and circular sulfate management rather than a complete replacement for conventional neutralization. The most technically plausible future route is likely to involve hybrid systems that can partially recover the sulfur value while improving operational flexibility, reducing environmental burden, and supporting broader circular hydrometallurgy strategies.

## Declarations

### Author Contributions

Antonio Clareti Pereira: Conceptualization, methodology, literature investigation, formal analysis, visualization planning, writing—original draft preparation, writing—review and editing.

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### Conflicts of Interest

The author declares no conflicts of interest related to this work.

### Data Availability Statement

No new experimental data were generated during this study. All information analyzed in this review was obtained from publicly available scientific literature

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