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UREA-ASSISTED CONVERSION OF PHOSPHOGYPSUM INTO AMMONIUM SULFATE AND CALCIUM CARBONATE: THERMODYNAMICS, KINETICS, AND SCALE-UP CHALLENGES

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Abstract

Phosphogypsum (PG) accumulation represents one of the major environmental challenges associated with wet-process phosphoric acid production, with global stockpiles exceeding several billion tonnes and annual generation continuing to increase. Conventional PG valorization routes remain limited by impurity content, transportation costs, regulatory constraints, and low economic attractiveness. Urea-assisted conversion of phosphogypsum into ammonium sulfate has emerged as an alternative route that integrates sulfur recovery, nitrogen utilization, and calcium carbonate production within a circular-economy framework. This critical review analyzes the chemistry, thermodynamics, kinetics, impurity behavior, reactor configurations, and industrial feasibility of PG conversion using urea. Particular emphasis is placed on the role of urea hydrolysis, ammonium generation, sulfate conversion mechanisms, calcium carbonate precipitation, and impurity partitioning involving fluorine, phosphorus, rare earth elements, and radionuclides. The review also discusses process intensification strategies, including hydrothermal systems, catalytic hydrolysis, membrane integration, and reactive crystallization. Although thermodynamic analyses generally indicate favorable sulfate conversion under controlled conditions, industrial implementation remains constrained by slow hydrolysis kinetics, ammonia volatilization, impurity management, product purification requirements, and scale-up uncertainties. The logistical advantages of urea as a solid nitrogen carrier offer operational benefits over conventional ammonia-based routes. Current technological maturity remains low, particularly at pilot and continuous-operation scales, indicating substantial opportunities for integrated waste valorization and fertilizer production research.

Keywords: Phosphogypsum; Urea hydrolysis; Ammonium sulfate; Calcium carbonate; Waste valorization; Circular economy

Highlights

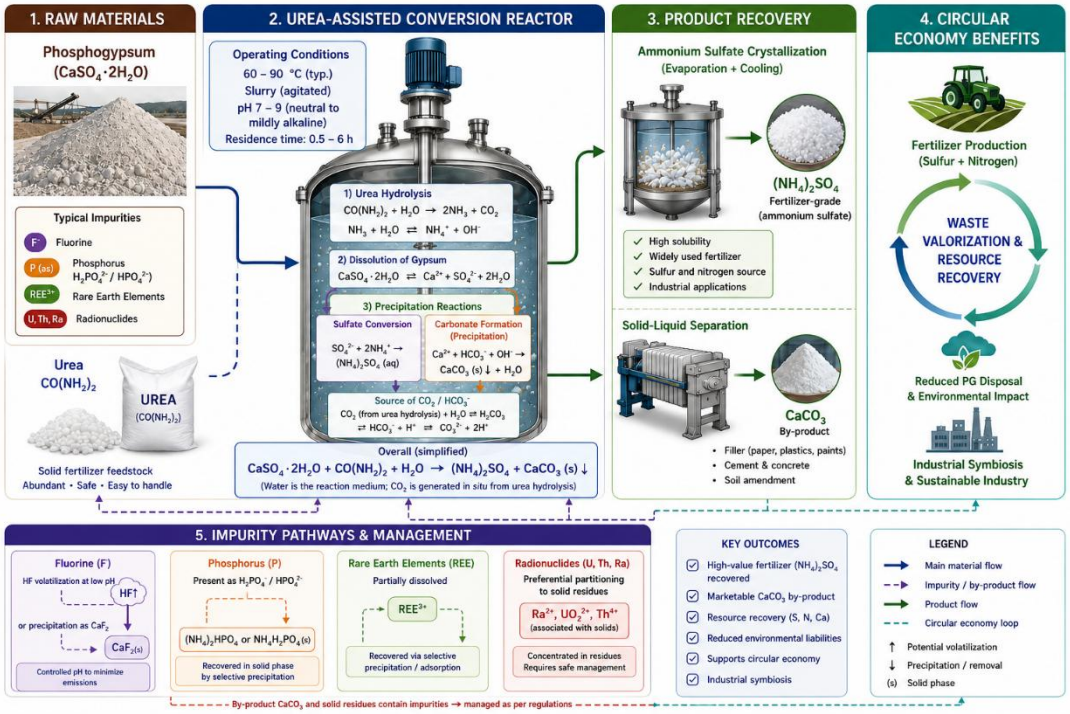
- Urea-assisted phosphogypsum conversion integrates sulfur recovery, nitrogen utilization, and calcium carbonate production within a waste valorization framework.

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- Thermodynamic feasibility is generally favorable, but industrial implementation is limited by hydrolysis kinetics, ammonia losses, and impurity management.
- Fluorine, phosphorus, radionuclides, and rare earth elements strongly influence product purity, downstream processing, and regulatory compliance.
- The solid-handling and logistics advantages of urea may reduce operational complexity compared with conventional ammonia-based ammonium sulfate routes.

Graphical abstract



1. Introduction

Phosphogypsum (PG) is the main by-product generated during wet-process phosphoric acid production, in which phosphate rock reacts with sulfuric acid to produce phosphoric acid and calcium sulfate hydrates. Approximately 4.5–5.5 t of PG are generated per tonne of phosphoric acid, with annual global production exceeding 250 million tonnes (Chernysh et al., 2021; Awad et al., 2024). Large stockpiles have accumulated worldwide, particularly in China, Morocco, the United States, and India, where phosphate fertilizer industries are highly developed (Lou et al., 2024; Maina et al., 2025).

Although calcium sulfate dihydrate is the dominant phase, PG commonly contains impurities such as residual phosphorus compounds, fluorides, organic matter, rare earth elements (REEs), and radionuclides, including uranium, thorium, and radium (Wang et al.,

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2026). These contaminants complicate reuse by affecting leaching behavior, environmental classification, product purity, and long-term stability. Radionuclides are particularly important because they are subject to regulatory restrictions in agricultural and construction applications in several countries. Environmental concerns associated with PG disposal also include acidic drainage, fluoride release, groundwater contamination, and particulate emissions. Consequently, large storage stacks require continuous monitoring and containment, increasing operational costs for phosphate producers (Awad et al., 2024). Despite decades of investigation, only a small fraction of global PG is commercially reused, highlighting the difficulty of translating laboratory-scale technologies into industrial practice (Chernysh et al., 2021).

Recent studies increasingly regard phosphogypsum as a secondary mineral resource rather than solely as industrial waste. This perspective aligns with circular-economy strategies focused on sulfur recovery, calcium utilization, and extraction of critical elements such as REEs (Maina et al., 2025). However, industrial feasibility still depends on impurity management, energy consumption, logistics, and downstream product quality.

Conventional PG valorization routes include cement production, gypsum boards, road construction, soil amendment, sulfur recovery, and chemical sulfate products. Cement and construction sectors remain among the largest consumers because calcium sulfate can partially replace natural gypsum in clinker and cement setting systems (Pliaka & Gaidajis, 2022). Agricultural applications may improve the structure of sodic soil by supplying calcium and sulfur (Xie et al., 2024). Nevertheless, impurity accumulation involving fluorides, phosphorus compounds, and radionuclides remains a major concern, particularly for long-term soil exposure. Transportation cost and high moisture content further limit the economic viability of low-value PG applications.

Alternative chemical valorization approaches aim to produce higher-value products, including sulfuric acid, ammonium sulfate, calcium carbonate, and composite materials (Shi et al., 2024). Some systems also integrate carbon mineralization and CO_2 utilization to improve environmental performance (Cui et al., 2024; Bouargane et al., 2023). In parallel, REE recovery from PG has gained attention because certain phosphate rocks contain yttrium, cerium, lanthanum, and neodymium (Meng et al., 2023). However, low REE concentrations and complex impurity matrices still hinder economic recovery at an industrial scale. Most integrated valorization systems remain limited to laboratory or pilot-scale studies due to

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operational challenges involving impurity accumulation, continuous processing, and solid–liquid separation.

Ammonium sulfate ($(NH_4)_2SO_4$) remains an important sulfur-containing fertilizer because it simultaneously supplies nitrogen and sulfur. Growing sulfur deficiency in agricultural soils has increased demand for sulfur-based fertilizers in several regions (Herrera et al., 2022). Commercial ammonium sulfate is traditionally produced through caprolactam manufacture, coke-oven gas treatment, and ammonia-neutralization systems (Gong et al., 2024). In addition to fertilizer use, ammonium sulfate is also used in water treatment, pharmaceuticals, fermentation, and flame-retardant formulations. However, conventional ammonia-based systems require handling gaseous NH_3, creating safety and storage challenges (Guo et al., 2021). Product purity is also critical because impurities such as fluorides, heavy metals, and radionuclides may restrict agricultural application (Kar et al., 2023).

Urea-assisted conversion has emerged as an alternative route in which urea acts as both a nitrogen carrier and an indirect ammonia source through hydrolysis (Avşar & Ertunç, 2025). Compared with anhydrous ammonia, urea offers important logistical advantages because it is stable, easy to transport, and compatible with existing fertilizer infrastructure (Hammas-Nasri et al., 2020). During hydrolysis, urea generates ammonia and carbonate species that promote the simultaneous formation of ammonium sulfate and calcium carbonate (Idboufrade et al., 2022). Calcium carbonate precipitation may also thermodynamically favor the conversion of sulfate by removing dissolved calcium ions.

Despite these advantages, important technical limitations remain. Urea hydrolysis may be slow under mild conditions and often requires elevated temperatures, catalysts, or hydrothermal operation (Orabi et al., 2024). Side reactions involving ammonia volatilization, biuret formation, and cyanate generation may reduce nitrogen efficiency and complicate reactor control. Product purity is additionally affected by fluorides, phosphorus compounds, and other impurities present in industrial phosphogypsum. Although recent studies have investigated hydrothermal and reactive crystallization systems (Cherrat et al., 2026), most studies remain limited to laboratory-scale reactors with simplified feed compositions.

This review critically examines urea-assisted conversion of phosphogypsum into ammonium sulfate and calcium carbonate, focusing on thermodynamics, kinetics, impurity behavior, reactor design, and industrial feasibility. Following the PRISMA-informed

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methodology proposed by Page et al. (2021), the review emphasizes sulfate equilibrium, carbonate precipitation, impurity partitioning, ammonia losses, process intensification, and scale-up limitations within circular-economy frameworks (Chernysh et al., 2021; Lou et al., 2024; Wang et al., 2026).

2. Methodology

This manuscript is a structured critical review informed by PRISMA 2020 principles rather than a full systematic review or meta-analysis. The methodology was designed to improve transparency in literature identification, screening, and thematic organization while maintaining flexibility for engineering interpretation and industrial-process evaluation (Page et al., 2021). No formal risk-of-bias assessment or meta-analytical statistical treatment was performed because the review primarily emphasizes mechanistic interpretation, process engineering, thermodynamic feasibility, and industrial applicability of urea-assisted phosphogypsum (PG) conversion to ammonium sulfate and calcium carbonate.

The literature survey covered publications related to phosphogypsum valorization, urea hydrolysis, sulfate conversion, ammonium sulfate synthesis, calcium carbonate precipitation, reactive crystallization, and waste-to-fertilizer technologies. Search terms included “phosphogypsum”, “urea hydrolysis”, “ammonium sulfate”, “gypsum conversion”, “calcium carbonate precipitation”, “sulfate recovery”, “waste valorization”, and “circular economy”. Additional searches included hydrothermal processing, catalytic hydrolysis, reactor design, radionuclide behavior, rare earth recovery, and process intensification.

The principal databases were Scopus, Web of Science, ScienceDirect, SpringerLink, and Google Scholar. Patent databases and technical industrial reports were additionally consulted to identify engineering trends not fully represented in peer-reviewed literature. The review primarily emphasized publications from 2020 to 2026 to highlight recent developments in phosphogypsum management, sulfate conversion, and circular-economy strategies (Chernysh et al., 2021; Awad et al., 2024; Lou et al., 2024). Earlier publications were selectively incorporated when essential to understanding gypsum chemistry, sulfate equilibria, ammonium sulfate crystallization, carbonate precipitation, or the mechanisms of urea hydrolysis.

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Table 1 summarizes the principal databases, search strategies, and literature-screening workflow adopted during the review process.

Table 1. Literature search and screening strategy adopted for the present review. Adapted from Page et al. (2021).

Database	Search Strategy	Search Date	Records Retrieved	Duplicates Removed	Records Screened	Final Included Studies	Main Exclusion Reasons
Scopus	"phosphogypsum" AND "urea hydrolysis"	Jan 2026	312	48	264	67	Lack of engineering relevance
Web of Science	"ammonium sulfate" AND "PG conversion"	Jan 2026	201	35	166	41	Insufficient operational data
ScienceDirect	"calcium carbonate precipitation" AND "phosphogypsum"	Jan 2026	178	29	149	36	Unrelated applications
SpringerLink	"urea-assisted sulfate conversion"	Jan 2026	96	14	82	21	Lack of thermodynamic or kinetic data
Google Scholar	Broad supplementary search	Jan 2026	420	115	305	33	Duplicated or low-quality studi

The review boundaries were defined to avoid overextension into unrelated phosphogypsum applications. Discussions involving cement chemistry, phosphate fertilizer production, or radioactive risks were only included when directly associated with ammonium sulfate production, impurity redistribution, environmental management, or process integration. Studies lacking reaction chemistry, engineering analysis, operational data, or industrial relevance were excluded. Priority was given to publications reporting thermodynamic calculations, kinetic behavior, reactor conditions, impurity partitioning, crystallization, product purity, ammonia losses, energy demand, techno-economic implications, or scale-up limitations.

Table 2 summarizes the principal inclusion and exclusion criteria used in the literature selection and thematic classification.

Table 2. Inclusion and exclusion criteria were adopted for literature selection and thematic classification in the present review. Adapted from Page et al. (2021).

Category	Inclusion Criteria	Exclusion Criteria
Scope	PG conversion, urea hydrolysis, ammonium sulfate production, sulfate recovery	Unrelated sulfate or fertilizer systems

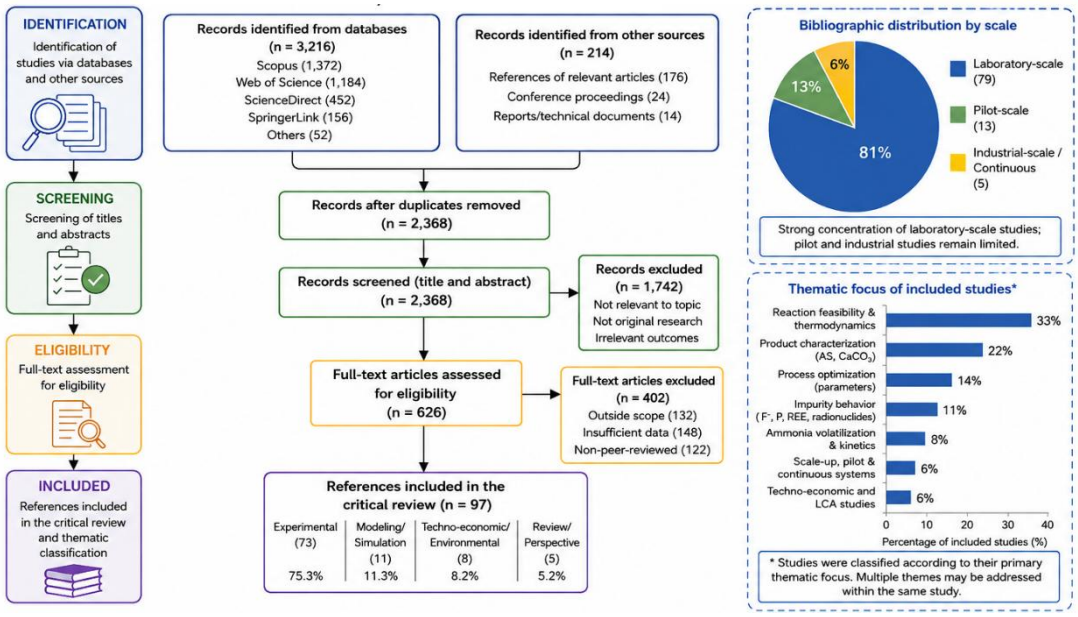
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Engineering relevance	Thermodynamics, kinetics, reactors, impurities, scale-up	Descriptive studies without engineering analysis
Data quality	Experimental, pilot, simulation, techno-economic data	Insufficient or non-reproducible data
Feedstock relevance	PG, gypsum, sulfate-rich industrial residues	Unrelated mineral systems
Industrial applicability	Continuous processing, TRL, process integration	Purely theoretical discussions without industrial relevance
Publication type	Peer-reviewed articles, patents, technical reports	Opinion papers or incomplete studies

The review emphasized contradictions and limitations reported in the literature. Many studies describe favorable thermodynamic conversion but provide limited information regarding impurity accumulation, ammonia volatilization, filtration behavior, or long-term operational stability. Therefore, this review prioritized comparative and critical analysis over isolated performance claims. Particular attention was given to differences between laboratory-scale systems and industrial implementation, especially regarding hydrolysis kinetics, slurry hydrodynamics, gas-liquid equilibrium, and impurity management.

This review used a PRISMA-based methodology from Matthew J. Page et al. (2021), detailed in Figure 1, to identify, screen, assess, and classify publications on urea-assisted phosphogypsum conversion. It aimed to identify studies on thermodynamics and sulfate conversion, and to evaluate process engineering, impurity behavior, hydrolysis kinetics, reactor design, and industrial applications. Emphasis was placed on differentiating lab-scale studies from pilot-scale, continuous processing, techno-economic, and environmental research.



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Figure 1. PRISMA-informed literature selection methodology adopted for the critical review of urea-assisted phosphogypsum conversion systems. Adapted from Page et al. (2021).

Figure 1 outlines the review's workflow for literature identification, screening, and thematic classification. Most studies are lab-scale, focusing on reaction feasibility and product analysis, whereas pilot-scale and continuous-reactor studies are less common. The data also show less research on impurity behavior, ammonia volatilization, and long-term stability compared to thermodynamic efficiency. This gap highlights the disconnect between academic research and industrial needs, especially for integrated fertilizer production.

3. Chemistry of Phosphogypsum

3.1. Formation during phosphoric acid production

Phosphogypsum (PG) is generated during wet-process phosphoric acid production, where phosphate rock reacts with sulfuric acid to produce phosphoric acid and calcium sulfate hydrates. This route dominates global phosphoric acid manufacturing due to its relatively low operational costs and high industrial maturity (Chernysh et al., 2021; Awad et al., 2024). Depending on operating conditions, calcium sulfate may crystallize as dihydrate, hemihydrate, or anhydrite, although the dihydrate form remains the most common industrial product.

The overall reaction for the dihydrate process may be represented as:

$$Ca_{10}(PO_4)_6F_2 + 10H_2SO_4 + 20H_2O \rightarrow 6H_3PO_4 + 10CaSO_4 \cdot 2H_2O + 2HF$$

The reaction is thermodynamically favorable because calcium sulfate precipitation continuously shifts the equilibrium toward the formation of phosphoric acid. Reported Gibbs free energy values generally range between approximately −350 and −500 kJ·mol−1 depending on phosphate rock mineralogy, sulfuric acid concentration, and operating temperature (Hamed et al., 2024).

Industrial wet-process reactors typically operate at 70-85 °C in dihydrate systems and 100-110 °C in hemihydrate routes (Hassoune et al., 2020). Sulfuric acid concentrations usually range from 93 to 98 wt%, while phosphoric acid concentrations may exceed 28–32 wt% P_2O_5. Residence times typically range from 4 to 8 h, depending on the reactor configuration and crystallization strategy.

Phosphate rock mineralogy strongly affects PG composition and crystallization behavior. Sedimentary ores frequently contain silica, fluorapatite, carbonates, clays, and trace

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metals that partially dissolve during acidulation (Chernysh et al., 2021). Fluoride chemistry is particularly important because fluorapatite decomposition releases HF and fluorosilicate species that partition among gaseous, liquid, and solid phases.

Several secondary reactions additionally govern impurity incorporation into PG. Fluoride may precipitate through calcium fluoride formation because CaF_2 exhibits very low solubility under acidic conditions. However, partial volatilization of HF and SiF_4 may still occur at elevated temperatures and high acidity, thereby complicating gas treatment and corrosion control (Hamed et al., 2024).

The chemistry of PG formation, therefore, depends on calcium sulfate precipitation, sulfate supersaturation, impurity partitioning, crystallization kinetics, and reactor hydrodynamics. These factors directly influence downstream valorization because impurity distribution affects ammonium sulfate purity, calcium carbonate precipitation, filtration behavior, and environmental classification.

3.2. Typical Composition

The chemical composition of phosphogypsum varies significantly depending on the phosphate ore source, acidulation conditions, crystallization route, and washing efficiency. Calcium sulfate dihydrate generally represents approximately 85–98 wt% of the material, while impurities constitute the remaining fraction (Chernysh et al., 2021; Awad et al., 2024). Moisture content after filtration commonly ranges from 10 to 25 wt%, directly affecting transport, storage, and downstream processing.

Table 3 summarizes the typical composition of phosphogypsum generated during wet-process phosphoric acid production and highlights the principal implications of major impurities for downstream PG valorization systems.

Table 3. Typical chemical composition of phosphogypsum from wet-process phosphoric acid production. Adapted from Chernysh et al. (2021), Omri et al. (2025), and Khalil et al. (2025).

Component / Property	Typical Range	Main Chemical Form	Main Implications
$CaSO_4 \cdot 2H_2O$	85–98 wt%	Calcium sulfate dihydrate	Main sulfate source for conversion
Moisture	10–25 wt%	Free/interstitial water	Affects transport and filtration
P_2O_5 (total)	0.2–2.0 wt%	Residual phosphates	Reduces ammonium sulfate purity
Water-soluble P_2O_5	0.05–0.6 wt%	H_3PO_4 / $H_2PO_4^-$	Increases acidity and impurity mobility

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Fluorides (F)	0.1–1.5 wt%	CaF_2, Na_2SiF_6, HF	Corrosion and gas-treatment issues
SiO_2	0.5–8 wt%	Quartz/silicates	Influences filtration and rheology
Al_2O_3	0.1–1.5 wt%	Aluminosilicates	Affects precipitation behavior
Fe_2O_3	0.05–1.0 wt%	Iron oxides/hydroxides	Product contamination risk
MgO	0.05–0.8 wt%	Magnesium salts	May alter carbonate precipitation
Organic matter	0.01–0.5 wt%	Residual organics	Foaming and filtration problems
$Na_2O + K_2O$	0.05–0.7 wt%	Alkali salts	Alters ionic strength
Rare earth elements (REEs)	100–1500 $mg \cdot kg^{-1}$	REE sulfates/phosphates	Potential secondary resource
Uranium (U)	5–200 $mg \cdot kg^{-1}$	Uranium phosphates/sulfates	Regulatory concern
Thorium (Th)	1–50 $mg \cdot kg^{-1}$	Thorium compounds	Radiological classification
Radium (^{226}Ra)	100–1000 $Bq \cdot kg^{-1}$	$RaSO_4$-associated phases	Environmental restriction
Particle size (d50)	20–100 µm	Fine particulate solids	Influences dissolution kinetics
pH (slurry)	2–6	Acidic aqueous phase	Controls impurity mobility

The high compositional variability of phosphogypsum significantly affects sulfate conversion, hydrolysis efficiency, impurity separation, filtration, and purification during downstream processing. Residual phosphates and fluorides are among the most important contaminants because they directly affect ammonium sulfate purity, corrosion behavior, and wastewater-treatment demand (Omri et al., 2025). Fluoride concentrations commonly range from 0.1 to 1.5 wt% and often occur as CaF_2, Na_2SiF_6, or adsorbed fluoride species.

Rare earth elements (REEs) and radionuclides additionally constitute important components of PG chemistry. Uranium concentrations commonly range between 5 and 200 mg·kg−1, while radium and thorium levels vary substantially depending on phosphate deposit origin (Khalil et al., 2025). REEs such as yttrium, lanthanum, cerium, and neodymium may accumulate due to partial transfer from phosphate rock during acidulation, creating opportunities for secondary resource recovery.

Table 4 summarizes the principal feedstock-quality parameters affecting industrial suitability of phosphogypsum for downstream valorization processes.

Table 4. Main phosphogypsum feedstock-quality parameters affecting industrial suitability for downstream conversion processes. Adapted from Awad et al. (2024), Omri et al. (2025), and Maina et al. (2025).

Parameter	Acceptable/Preferred Condition	Main Concern	Impact on Downstream Processing

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Sulfate content	High $CaSO_4 \cdot 2H_2O$ purity	Reduced conversion efficiency	Lower ammonium sulfate yield
Moisture	Moderate moisture content	Slurry instability	Filtration and transport difficulties
Fluoride concentration	Low fluoride levels	Corrosion and gas emissions	Additional purification demand
Residual phosphates	Low soluble phosphorus	Product contamination	Reduced fertilizer quality
Particle size	Controlled fine distribution	Poor filtration	Increased slurry viscosity
Organic matter	Minimal organic contamination	Foaming and instability	Separation problems
Radionuclide content	Below regulatory thresholds	Environmental restrictions	Licensing and disposal limitations
REE concentration	Moderate-to-high REE content	Variable economic value	Potential co-recovery opportunity
Ionic strength	Controlled dissolved salts	Crystallization instability	Reduced process reproducibility

The mineralogical structure of phosphogypsum influences impurity retention, as some contaminants are embedded within calcium sulfate crystals, while others are surface-adsorbed or trapped in liquid phases. Embedded impurities are harder to remove by normal washing, complicating purification.

3.3. Environmental Concerns

Environmental concerns associated with phosphogypsum storage include acidic drainage, fluoride release, radionuclide accumulation, particulate emissions, and long-term leaching behavior. Large PG stacks occupy extensive land areas and may remain environmentally unstable for decades if inadequately managed (Hassoune et al., 2020).

One of the principal environmental concerns is fluoride mobility. Under acidic conditions, fluoride may occur as dissolved F^- or HF, while low pH conditions favor HF formation and increase toxicity risks for aquatic systems and vegetation (Hamed et al., 2024). Fluoride may additionally be transported through volatilization and particulate emissions, particularly under arid conditions with poor moisture control.

Radionuclide accumulation is another major environmental issue because radionuclides such as ^{226}Ra preferentially partition into PG via sulfate co-precipitation during acidulation (Chernysh et al., 2021). Although radionuclide concentrations are relatively low, long-term

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accumulation creates environmental and regulatory concerns regarding reuse, disposal, and groundwater contamination.

Leaching behavior depends strongly on pH, mineralogy, weathering conditions, and liquid-to-solid ratio. Cui et al. (2021) reported that acidic conditions significantly increase the mobility of phosphorus and fluoride, whereas neutralization may partially reduce contaminant transport via secondary mineral precipitation. Nevertheless, formation of secondary phases may also alter long-term contaminant stability.

Environmental impacts associated with PG storage involve complex interactions between impurity composition, hydrological conditions, atmospheric exposure, and mineral transformations. Figure 2 summarizes the principal pathways associated with acidic drainage, fluoride mobility, radionuclide accumulation, groundwater contamination, and particulate transport.

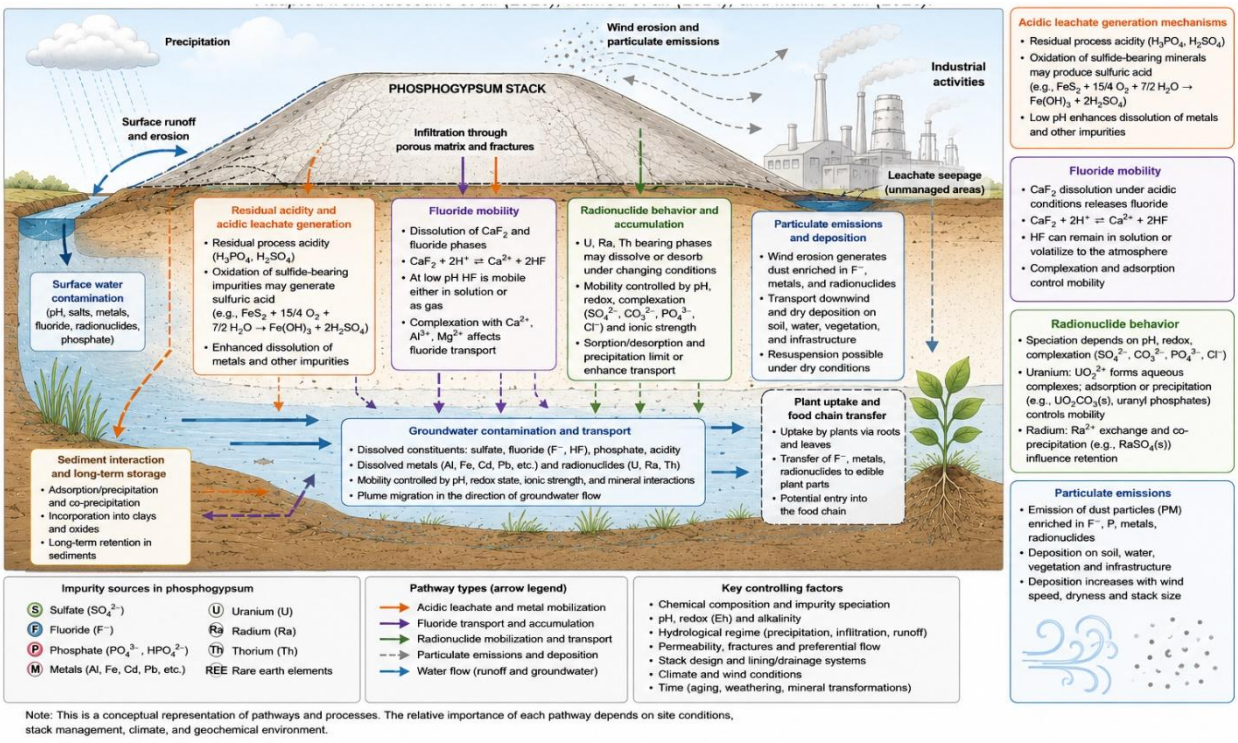


Figure 2. Main environmental pathways associated with phosphogypsum storage and impurity release, including fluoride mobility, radionuclide accumulation, acidic drainage, and particulate emissions. Adapted from Hassoune et al. (2020), Hamed et al. (2024), and Maina et al. (2025).

Figure 2 summarizes how phosphogypsum impacts during long-term storage depend on bulk composition, hydrological conditions, impurity speciation, particle transport, and mineral

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formation. These factors influence urea-assisted systems by affecting impurity redistribution, ammonium sulfate purity, calcium carbonate quality, and wastewater management.

4. Urea Chemistry and Hydrolysis

4.1. Properties of urea

Urea ($CO(NH_2)_2$) is a widely produced nitrogen compound, mainly used in fertilizer manufacturing and industrial nitrogen management. Annually, global production exceeds 180 million tonnes, primarily via the Haber–Bosch process using ammonia and carbon dioxide (Sergeev et al., 2020). Urea has high nitrogen content (46 wt%), low toxicity, high water solubility, and good storage stability compared to anhydrous ammonia. These traits make urea especially suitable for decentralized nitrogen delivery and sulfate conversion.

Under ambient conditions, urea forms colorless hygroscopic crystals with melting points around 132–135 °C. Its water solubility increases with temperature from about 545 g·L−1 at 25 °C to over 1,000 g·L−1 near 80 °C (Swify et al., 2023). High solubility promotes uniform hydrolysis and crystallization in ammonium sulfate production, but excessive supersaturation can lead to unwanted secondary reactions during heating.

Urea's thermodynamic stability depends on temperature. At ambient conditions, it remains metastable and hydrolyzes slowly without catalysts. Its standard Gibbs free energy of formation is about −197 kJ·mol−1, showing moderate stability (Dörnhöfer et al., 2020). Hydrolysis becomes more favorable at higher temperatures or with catalytic assistance.

Industrial urea handling offers operational advantages over gaseous ammonia, such as simpler storage without pressurized or cryogenic vessels, lowering infrastructure and transport risks. This is especially useful for phosphogypsum conversion in regions without large ammonia infrastructure. Nonetheless, controlling urea decomposition is crucial to prevent ammonia volatilization, biuret formation, and fouling in reactors.

The technological readiness level (TRL) of industrial urea production is fully mature, usually TRL 9. In contrast, using urea as an indirect ammonia source for phosphogypsum conversion is less mature, typically at TRL 3-5, depending on the reactor setup and process integration. Most studies are limited to laboratory or pilot-scale testing without long-term data.

4.2. Hydrolysis mechanisms

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Urea hydrolysis is central to ammonium production in phosphogypsum conversion. In water, the overall reaction is:

$$CO(NH_2)_2 + H_2O \rightarrow 2NH_3 + CO_2$$

Under standard conditions, the Gibbs free energy change for this reaction is mildly negative, ranging from −13 to −20 kJ·mol−1, depending on temperature and speciation assumptions (Yahya et al., 2021). Despite favorable thermodynamics, hydrolysis kinetics are slow at room temperature due to the high activation energy of the uncatalyzed reaction.

In aqueous systems, ammonia from hydrolysis quickly reaches equilibrium with ammonium ions.

$$NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^-$$

This equilibrium depends on pH and temperature. Under acidic conditions, NH_4^+ favors ammonium sulfate; at alkaline pH and higher temperatures, ammonia volatilization increases, reducing nitrogen recovery (Yurekli et al., 2024).

Hydrolysis typically involves intermediate carbamate species. Proposed mechanisms include water attacking the urea's carbonyl carbon, forming carbamic acid, which then decomposes into ammonia and carbon dioxide (Matsubara & Yamada, 2020). These reactions are often represented as:

$$CO(NH_2)_2 + H_2O \rightarrow NH_2COOH + NH_3$$

followed by:

$$NH_2COOH \rightarrow NH_3 + CO_2$$

The decomposition of carbamic intermediates is thermodynamically favorable, as the Gibbs free energy decreases with increasing temperature. However, the transient stability of carbamate species may affect local pH gradients and carbonate speciation during sulfate conversion.

Catalytic hydrolysis speeds up ammonia production. Enzymatic urease is most efficient under mild conditions, lowering the activation energy for rapid conversion at near-ambient temperatures (Arias et al., 2025). However, industrial phosphogypsum systems rarely use biological catalysts due to stability issues in acidic, saline, or hydrothermal environments.

Hydrothermal and catalytic systems dominate current engineering. Elevated temperatures (70-150°C) boost hydrolysis, but excessive heat can increase ammonia

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volatilization and secondary reactions (Yurekli et al., 2024). Most sulfate hydrolysis technologies are still at TRL 3–5 due to limited evidence of long-term stability, catalyst durability, and impurity control.

4.3. Thermal decomposition pathways

Urea's decomposition is complex, involving multiple reactions influenced by temperature, residence time, and water. Below 120 °C, hydrolysis and dissolution dominate. From 130 to 160 °C, melting and condensation become more significant (Dörnhöfer et al., 2020).

One of the principal secondary reactions involves biuret formation:

$$2CO(NH_2)_2 \rightarrow NH_2CONHCONH_2 + NH_3$$

Biuret formation becomes thermodynamically favorable at higher temperatures, releasing ammonia. Gibbs free energy becomes more negative above about 140 °C, encouraging condensation during prolonged heating (Sergeev et al., 2020). Biuret buildup is undesirable as it lowers nitrogen availability and may disrupt ammonium sulfate crystallization.

Further thermal treatment may produce cyanuric acid, ammeline, melamine, and polymeric carbon–nitrogen compounds.

$$6CO(NH_2)_2 \rightarrow C_3H_6N_6 + 3CO_2 + 6NH_3$$

These reactions occur above 180–200 °C in the presence of limited water, potentially causing reactor fouling, increased viscosity, and solid deposits in hydrothermal systems.

Ammonia volatilization is another critical limitation during thermal treatment:

$$NH_4^+ \rightleftharpoons NH_3(g) + H^+$$

The Gibbs free energy for ammonia volatilization favors higher temperatures and alkaline conditions, leading to nitrogen losses in poorly controlled systems. In phosphogypsum processes, ammonia losses impact ammonium sulfate yield and economics.

Current thermal decomposition control systems are mature in fertilizer production (TRL 8–9), but their integration into phosphogypsum systems is underdeveloped and limited to experimental reactors.

4.4. Factors affecting hydrolysis

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Temperature mainly affects urea hydrolysis. Rates increase exponentially from 40 to 150 °C due to Arrhenius behavior, with activation energies typically 45-90 kJ·mol−1 depending on catalysts (Yahya et al., 2021). Higher temperatures boost ammonia production but also increase volatilization and secondary decomposition.

pH influences ammonia/ammonium balance and carbonate chemistry. Acidic conditions stabilize NH_4^+ and reduce ammonia loss, whereas alkaline conditions increase ammonia volatilization (Yurekli et al., 2024). In phosphogypsum systems, pH gradients from sulfate dissolution and carbonate precipitation can affect nitrogen recovery.

Catalysts significantly alter hydrolysis kinetics. Urease enzymes exhibit high reaction rates under mild conditions but suffer from thermal instability, limiting their industrial use. Metal oxides, transition-metal catalysts, and alkaline systems improve hydrolysis under hydrothermal conditions (Chamoli et al., 2024). However, catalyst poisoning by fluoride, phosphate, or metal impurities is not well studied in industrial phosphogypsum feeds.

Mass transfer limitations are more significant in concentrated slurries and high-solids systems, where particle size, slurry viscosity, gas-liquid transport, and mixing affect hydrolysis rates and ammonia distribution (Broumidis et al., 2025). Poor mixing can cause supersaturation zones, leading to secondary precipitation or incomplete conversion of sulfate.

The conversion of phosphogypsum using urea involves complex reactions like hydrolysis, carbonate formation, and thermal decomposition. Figure 3 illustrates key pathways, including primary hydrolysis, carbamate formation, carbonate generation, and secondary condensation. It also shows how temperature, pH, water, pressure, and residence time affect ammonia production and by-products. The scheme emphasizes the competition between the desired ammonium pathway and undesired decomposition routes that form biuret, cyanuric acid, melamine, and other products. Understanding these networks is crucial for optimizing sulfate conversion, reducing ammonia loss, controlling carbonate precipitation, and enhancing purity in phosphogypsum processing.

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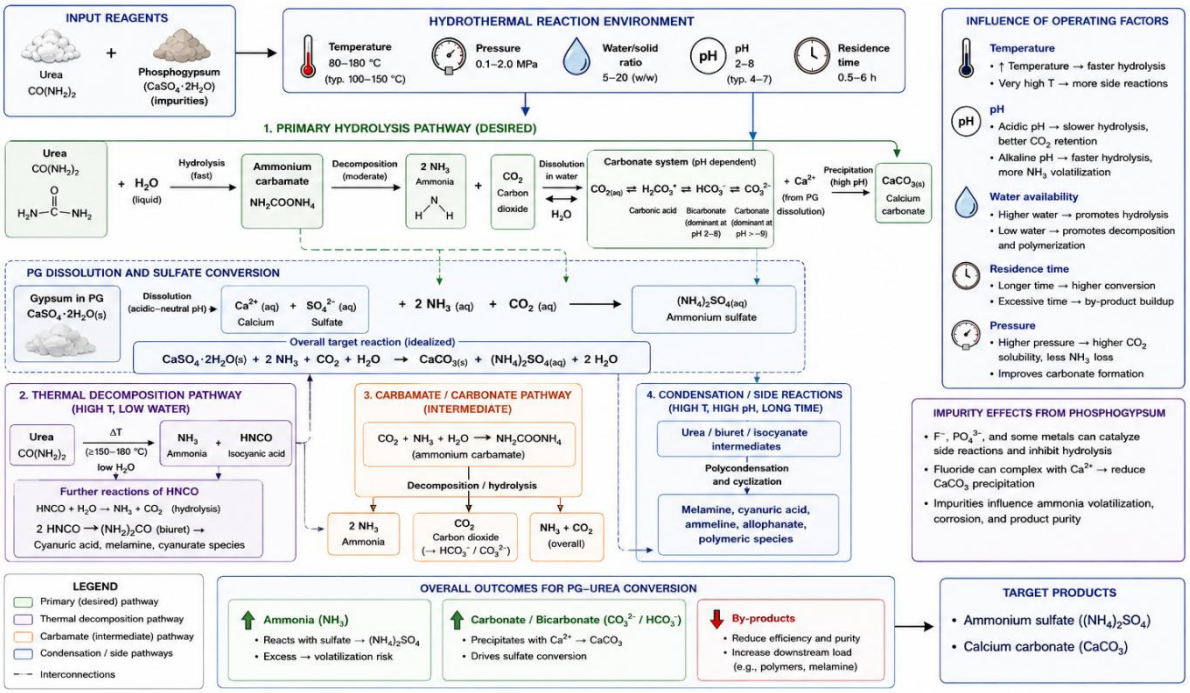


Figure 3. Main hydrolysis and thermal decomposition pathways of urea during phosphogypsum conversion, including ammonia generation, carbonate formation, biuret formation, and secondary condensation reactions. Adapted from Sergeev et al. (2020), Yahya et al. (2021), and Hopsort (2023).

Figure 3 shows the reaction pathways controlling urea during hydrothermal sulfate conversion. It emphasizes the narrow operational window needed to maximize ammonium while reducing ammonia volatilization and condensation. Current systems are at TRL 3–5, with long-term operation, impurity tolerance, and thermal stability still under demonstration for industrial use.

5. Thermodynamics of PG–Urea Conversion

5.1. Global reactions

The thermodynamic feasibility of converting phosphogypsum (PG) with urea depends on coupled equilibria involving urea hydrolysis, sulfate exchange, ammonium formation, carbonate speciation, and calcium carbonate precipitation. Unlike conventional ammonia-neutralization routes for ammonium sulfate production, PG–urea systems rely on indirect ammonia generation via hydrolysis, coupled with equilibrium displacement driven by calcium carbonate precipitation (Hammas-Nasri et al., 2020).

The overall idealized reaction may be represented as:

$$CaSO_4 \cdot 2H_2O + CO(NH_2)_2 + H_2O \rightarrow (NH_4)_2SO_4 + CaCO_3$$

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Thermodynamic analyses by Avşar et al. (2022a) and Idboufrade et al. (2022) indicate that the process becomes favorable because urea hydrolysis generates ammonia while calcite precipitation continuously removes dissolved calcium from solution. Reported Gibbs free energy values near ambient conditions are moderately negative, generally ranging between approximately −25 and −60 kJ·mol−1 depending on carbonate activity, ionic strength, and aqueous speciation.

The conversion mechanism does not occur through a single reaction step because several coupled equilibria develop simultaneously. Urea hydrolysis initially generates ammonia and carbon dioxide, followed by the formation of ammonium sulfate and the precipitation of calcium carbonate. Table 5 summarizes the principal reactions governing PG–urea conversion systems and their thermodynamic relevance.

Table 5. Main reactions involved in PG–urea conversion systems and their thermodynamic/kinetic relevance. Adapted from Avşar et al. (2022a), Brahma and Saikia (2020), and Goncharik et al. (2020).

Reaction	Main Role	Thermodynamic Characteristic	Main Limitation
Urea hydrolysis	NH_3 generation	Thermodynamically favorable at elevated temperature	Slow kinetics
Ammonium sulfate formation	Sulfate recovery	Favorable under ammonium-rich conditions	Dependent on hydrolysis efficiency
Calcium carbonate precipitation	Equilibrium displacement	Strongly negative Gibbs free energy	Scaling and fouling
CO_2 hydration/speciation	Carbonate generation	pH-dependent equilibrium	Carbonate availability
Ammonia volatilization	Nitrogen loss	Favored at high temperature and pH	Reduced nitrogen efficiency

Calcium carbonate precipitation is a principal thermodynamic driving force because calcite formation continuously shifts the sulfate dissolution equilibrium, promoting further phosphogypsum conversion. Several modified approaches, including ammonium bicarbonate, catalytic hydrolysis, and CO_2 enrichment, have been proposed to improve conversion efficiency and ammonia utilization (Avşar & Ertunç, 2025). However, most thermodynamic analyses still rely on simplified equilibrium models that inadequately represent industrial impurity accumulation, ionic competition, gas-liquid partitioning, and slurry hydrodynamics.

5.2. Gibbs Free Energy Analysis

Thermodynamic studies consistently indicate that the conversion of PG to ammonium sulfate and calcium carbonate is energetically favorable under controlled operating conditions.

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However, reaction feasibility depends strongly on temperature, pH, ionic strength, carbonate activity, gas-liquid equilibrium, and precipitation behavior (Brahma & Saikia, 2020).

Calcite precipitation plays a critical role because the removal of dissolved calcium continuously promotes the dissolution of sulfate. Under standard conditions, calcite formation exhibits a strongly negative Gibbs free energy of approximately −48 kJ·mol−1, favoring spontaneous crystallization in supersaturated systems (Bouargane et al., 2020). Although aragonite and vaterite may transiently form depending on supersaturation and temperature, calcite remains thermodynamically dominant at equilibrium.

Urea hydrolysis is also thermodynamically favorable, but the process remains kinetically limited under mild conditions. Gibbs free energy values become progressively more negative as temperature increases because entropy contributions associated with gaseous species become more significant (Goncharik et al., 2020). This behavior explains the substantially improved hydrolysis efficiency observed under hydrothermal conditions.

Carbonate speciation strongly influences equilibrium stability because hydrolysis-generated CO_2 partitions among dissolved CO_2, bicarbonate, and carbonate ions according to pH-dependent equilibria. Acidic conditions favor dissolved CO_2 and bicarbonate species, thereby reducing the availability of carbonate for calcite precipitation. In contrast, near-neutral and mildly alkaline conditions improve carbonate formation and calcium removal, although excessive alkalinity may simultaneously increase ammonia volatilization.

Kholmurodov et al. (2024) reported that equilibrium simulations frequently predict sulfate conversion efficiencies above 85–95% under ideal conditions. Nevertheless, these predictions often overestimate industrial performance because they neglect diffusion limitations, incomplete hydrolysis, impurity accumulation, solids transport, and slurry rheology. Consequently, thermodynamic favorability alone does not guarantee industrial feasibility.

5.3. Speciation Analysis

Speciation behavior strongly influences sulfate conversion efficiency, ammonium stabilization, carbonate precipitation, and impurity partitioning during PG–urea processing. The system involves coupled equilibria among NH_3/NH_4^+, $CO_2/HCO_3^-/CO_3^{2-}$, Ca^{2+}, SO_4^{2-},

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phosphate species, and dissolved impurities, thereby generating highly dynamic chemical conditions (Ennaciri et al., 2023).

Ammonia speciation is particularly important because gaseous NH_3 losses directly reduce nitrogen recovery. Below pH 7.5, ammonium ions dominate, whereas alkaline conditions increasingly favor the formation of gaseous ammonia, especially at elevated temperatures (Laaboubi et al., 2022). Consequently, industrial operation requires balancing carbonate availability with nitrogen retention.

Calcium speciation additionally influences precipitation pathways because dissolved calcium may react competitively with carbonate, fluoride, phosphate, and sulfate species. The low solubility of CaF_2 may promote partial fluoride immobilization, although dissolution may still occur under acidic conditions (Laaboubi et al., 2023). Phosphate adsorption onto calcite surfaces may further modify crystal morphology and precipitation kinetics.

Ionic strength also substantially affects equilibrium behavior because concentrated ammonium sulfate and recycled process streams deviate considerably from ideal solution behavior. Guo et al. (2021) observed that elevated ionic strength may reduce apparent carbonate activity and alter ammonium sulfate crystallization dynamics, particularly under high-solids conditions.

5.4. Effect of Temperature

Temperature strongly affects hydrolysis kinetics, equilibrium displacement, gas-liquid partitioning, and precipitation during PG–urea conversion. Most systems operate between approximately 60 and 150 °C, depending on reactor configuration and hydrolysis strategy (Yuan et al., 2024).

Increasing temperature accelerates urea hydrolysis, phosphogypsum dissolution, and carbonate precipitation under hydrothermal conditions. Conversion efficiencies frequently increase from below 50% under ambient conditions to above 80–90% at temperatures exceeding approximately 100 °C (Laasri, 2024). Nevertheless, excessive temperatures may reduce nitrogen efficiency because ammonia volatilization becomes progressively more favorable under alkaline conditions.

Temperature additionally affects calcium carbonate polymorphism. Although calcite remains the thermodynamically stable phase, elevated temperatures may transiently favor

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aragonite formation because of kinetic effects (Pyagai et al., 2022). Crystal morphology subsequently influences downstream filtration and solid-liquid separation performance.

Table 6 summarizes the principal differences between thermodynamic feasibility and industrial feasibility in PG–urea conversion systems.

Table 6. Comparison between thermodynamic feasibility and industrial feasibility in PG–urea conversion systems. Adapted from Kholmurodov et al. (2024), Maina et al. (2025), and Wang et al. (2026).

Aspect	Thermodynamic Prediction	Industrial Limitation
Sulfate conversion	Frequently >85–95%	Reduced by mass-transfer and kinetic limitations
Urea hydrolysis	Favorable at elevated temperature	Slow kinetics and ammonia losses
Calcite precipitation	Strong equilibrium displacement	Scaling and fouling
Carbonate availability	Improved at alkaline pH	NH_3 volatilization increases
Continuous operation	Stable equilibrium assumed	Slurry instability and impurity accumulation
Impurity behavior	Often neglected in simulations	Strongly affects product quality and operation

Recent hydrothermal investigations indicate that optimal operating conditions generally fall between approximately 80 and 130 °C, where hydrolysis remains efficient, and ammonia losses remain manageable (Laasri et al., 2025). However, most systems remain confined to batch laboratory reactors at intermediate technological maturity (TRL 3–5). In contrast, continuous industrial systems that integrate impurity control, gas recovery, and crystallization stability have not been sufficiently demonstrated.

6. Kinetics and Reaction Mechanisms

6.1. Urea hydrolysis kinetics

Despite being thermodynamically favorable, urea hydrolysis is kinetically limited under mild conditions. The uncatalyzed hydrolysis reaction:

$$CO(NH_2)_2 + H_2O \rightarrow 2NH_3 + CO_2$$

presents moderately negative Gibbs free energy values between −13 and −20 kJ·mol−1, depending on temperature and speciation (Yahya et al., 2021). However, spontaneous hydrolysis at ambient conditions remains slow due to activation energies of 50 to 90 kJ·mol−1 in non-catalytic systems.

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Hydrolysis kinetics follow pseudo-first-order or mixed behavior based on temperature, catalyst, and pH (Matsubara & Yamada, 2020). Below ~40 °C, reaction rates are slow, taking hours to produce substantial amounts of ammonia. Between 80-150 °C, hydrolysis speeds up due to increased molecular mobility and water dissociation.

Several studies report Arrhenius behavior for urea decomposition and hydrolysis. Yahya et al. (2021) observed exponential increases in ammonia above 70 °C, while Yurekli et al. (2024) showed that reaction rates are strongly affected by local pH and ionic strength in sulfate systems. Increased ionic strength may lower water activity and alter carbamate stability, impacting conversion rates.

Hydrolysis mechanisms proceed through transient carbamic intermediates:

$$CO(NH_2)_2 + H_2O \rightarrow NH_2COOH + NH_3$$

followed by:

$$NH_2COOH \rightarrow NH_3 + CO_2$$

The decomposition of carbamic acid becomes more favorable with increasing temperature, as the entropy contribution from gas formation decreases the Gibbs free energy. However, intermediate stability may locally alter carbonate speciation and pH in concentrated phosphogypsum slurries.

Catalytic systems modify reaction kinetics significantly. Urease catalysts can greatly boost hydrolysis rates under near-ambient conditions (Hopsort, 2023). However, enzymatic systems are unsuitable for industrial phosphogypsum conversion due to fluoride, acidity, metals, and hydrothermal conditions that deactivate enzymes. Most current hydrolysis relies on thermal acceleration rather than enzymes.

Urea hydrolysis for fertilizer synthesis is highly mature (TRL 8–9). In contrast, integrated systems with phosphogypsum are less developed (TRL 3–5) due to ongoing issues with stability, ammonia recovery, and tolerance to impurities.

6.2. Dissolution of phosphogypsum

Phosphogypsum dissolution influences sulfate availability and the kinetics of ammonium sulfate formation. Its dissolution reaction is represented as:

$$CaSO_4 \cdot 2H_2O \rightleftharpoons Ca^{2+} + SO_4^{2-} + 2H_2O$$

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Under standard conditions, the dissolution of calcium sulfate has a moderately positive Gibbs free energy because gypsum's low solubility limits it. However, ongoing calcium removal via carbonate precipitation shifts the equilibrium, increasing dissolution and coupling the kinetics (Bouargane et al., 2020).

Phosphogypsum dissolves via surface-controlled mechanisms at low supersaturation and diffusion-controlled mechanisms at higher solids concentration (Pyagai et al., 2022). Rates increase with temperature due to enhanced ionic mobility and surface reactivity under hydrothermal conditions. Excessive sulfate may hinder dissolution by saturation effects.

Particle size distribution affects dissolution; fine particles dissolve faster due to a larger surface area (Ennaciri et al., 2023). However, very fine materials can raise slurry viscosity and hinder mixing during reactor operation.

Impurity distribution complicates dissolution. Phosphate residues, fluoride, silica, and organic matter may coat crystals, thereby lowering dissolution rates. Cherrat et al. (2026) found that industrial phosphogypsum dissolves more slowly than purified gypsum because impurities alter surface morphology and mass transfer.

Temperature influences phase stability; above 100–120 °C, partial transformations among gypsum, hemihydrate, and anhydrite can occur, affecting dissolution because of their differing solubilities.

Most dissolution studies are limited to lab batch systems (TRL 2–4), with scarce industrial data under continuous hydrothermal operation and impurity-rich feeds.

6.3. $CaCO_3$ precipitation mechanisms

Calcium carbonate precipitation is one of the principal kinetic and thermodynamic driving forces governing phosphogypsum conversion. The precipitation reaction:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(s)$$

presents strongly negative Gibbs free energy values under standard conditions, commonly near −48 to −60 kJ·mol−1 depending on polymorph and ionic environment (Czaplicka & Konopacka-Łyskawa, 2020). Precipitation continuously removes dissolved calcium, thereby promoting further dissolution of phosphogypsum.

$CaCO_3$ usually precipitates via nucleation, growth, agglomeration, and polymorphic transformation. Vaterite often forms initially under high supersaturation due to lower

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nucleation barriers, despite lower stability (Namsaraev et al., 2020). Over time, it transforms into calcite through dissolution–reprecipitation.

Temperature and pH influence polymorph selection, primarily favoring calcite. Aragonite can form kinetically at high temperatures or ionic strength (Yi et al., 2021). Crystal shape impacts filtration; needle-like or porous precipitates reduce cake permeability.

Impurities in phosphogypsum alter precipitation. Phosphate adsorbs onto calcite, inhibiting growth; fluoride affects nucleation or promotes secondary reactions (Khumalo et al., 2025). Magnesium and organics stabilize metastable forms and influence particle aggregation.

Supersaturation control remains one of the most important engineering challenges. Excessively rapid precipitation may generate ultrafine particles difficult to separate industrially, whereas insufficient supersaturation reduces sulfate conversion efficiency. Consequently, precipitation kinetics must be balanced against mixing intensity, hydrolysis rate, and dissolution behavior.

Reactive precipitation systems for carbonate formation remain predominantly at TRL 3–5 in PG–urea applications because continuous crystallization control and impurity management have not been sufficiently validated under industrial conditions.

6.4. Mass transfer limitations

Mass transfer limits significantly impact PG–urea conversion due to hydrolysis, dissolution, precipitation, and gas-liquid equilibria in concentrated slurries. Many experiments show that transport constraints have a greater influence on conversion efficiency than thermodynamics (Liu et al., 2021).

Gas-liquid transfer limits are vital for the distribution of CO_2 and ammonia. CO_2 from hydrolysis must dissolve to form carbonate before calcium carbonate can form. Ammonia volatilization can lower nitrogen recovery if gas-liquid equilibrium isn't properly managed.

Slurry viscosity affects hydrodynamics and mixing; high solids content leads to poor mixing, concentration gradients, and supersaturation (Ma et al., 2023). This heterogeneity may lead to incomplete dissolution of phosphogypsum, uneven carbonate precipitation, or ammonia losses.

Diffusion limits around particles affect conversion rates. Calcium carbonate precipitating on phosphogypsum particles can block sulfate release and lower mass transfer

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coefficients (Zhang et al., 2025). Similar passivation occurs in other sulfate systems with heterogeneous precipitation.

Particle aggregation affects reactor performance. Xu et al. (2025) state that agglomeration changes local hydrodynamics, settling, and filtration during sulfate processing. Agglomerates trap liquids and impurities, reducing ammonium sulfate purity.

PG–urea conversion depends on thermodynamics, kinetics, and transport in multiphase slurries. Figure 4 shows mechanisms like phosphogypsum dissolution, urea hydrolysis, carbonate precipitation, gas–liquid transport, and solid–liquid separation in hydrothermal conversion. Diffusion, mixing, bubble transport, precipitation, and hydrodynamics influence sulfate conversion and product quality. Boundary-layer transport, carbonate saturation, ammonia volatilization, and particle aggregation often impact reactor performance more than thermodynamics. Under high-solids conditions, transport limits can cause scaling, filtration issues, impurity buildup, and process instability.

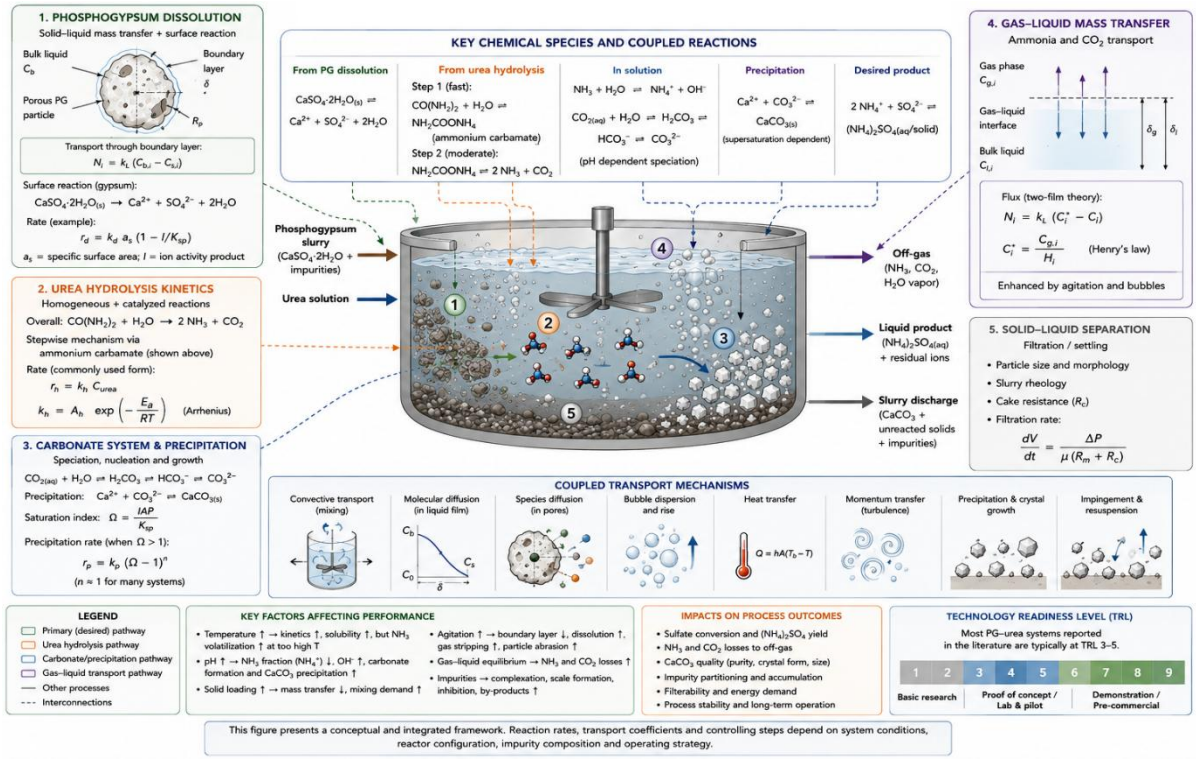


Figure 4. Coupled kinetic and mass transfer mechanisms governing phosphogypsum dissolution, urea hydrolysis, carbonate precipitation, and gas-liquid transport during PG–urea conversion. Adapted from Liu et al. (2021), Zhang et al. (2025), and Xu et al. (2025).

Figure 4 shows how chemical kinetics and transport affect PG–urea systems. Performance depends on thermodynamics, hydrodynamics, diffusion, precipitation, and gas-

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liquid equilibrium. Most systems are at TRL 3–5 because long-term continuous operation under realistic slurry conditions is still unproven.

7. Process Configurations and Reactor Design

7.1. Batch systems

Batch reactors are the most common in lab-scale phosphogypsum studies because of their flexibility and ease of controlling processes. They typically use mechanically stirred glass or stainless-steel reactors at temperatures between 60 and 150 °C and at atmospheric or moderate pressure (Hammas-Nasri et al., 2020; Idboufrade et al., 2022).

In batch operation, phosphogypsum, urea, and process water are introduced simultaneously, enabling hydrolysis and sulfate conversion in a closed volume. The main reaction remains:

$$CaSO_4 \cdot 2H_2O + CO(NH_2)_2 + H_2O \rightarrow (NH_4)_2SO_4 + CaCO_3$$

The global Gibbs free energy stays negative under controlled hydrothermal conditions because calcium carbonate precipitation shifts the equilibrium toward sulfate dissolution. However, batch reactors require longer residence times, typically 1 to 8 hours, due to the slow rate of urea hydrolysis (Patil et al., 2023).

Batch systems allow independent control of temperature, pH, reagent ratio, and solids loading, benefiting impurity partitioning and carbonate polymorphism studies. However, scale-up challenges arise at larger volumes due to difficulties in controlling gas-liquid equilibrium, supersaturation, and slurry homogeneity.

Several studies report incomplete sulfate conversion in poorly mixed batches due to carbonate depletion and passivation layers on phosphogypsum (Nogqwazi, 2020). Filtration may worsen as prolonged reactions produce fine precipitates and wider particle sizes.

Batch PG–urea systems are generally TRL 3–4. While lab efficiencies above 80–90% are reported under optimal conditions, industrial continuous processing and long-term stability are not yet sufficiently demonstrated.

7.2. Continuous stirred reactors

Continuous stirred-tank reactors (CSTRs) enable steady-state operation and continuous product removal, bridging lab studies and industrial use. They are fed phosphogypsum slurry,

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urea solution, and process streams into agitated reactors with controlled residence times (Guo et al., 2021).

Continuous operation can enhance thermal efficiency and process integration by easing heat recovery and slurry circulation. However, it adds complexity with solids handling, crystallization, and slurry rheology. Sulfate conversion hinges on sufficient mixing and the avoidance of dead zones (Costamagna et al., 2020).

Hydrolysis and precipitation reactions occur simultaneously inside the reactor:

$$CO(NH_2)_2 + H_2O \rightarrow 2NH_3 + CO_2$$

followed by carbonate precipitation:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(s)$$

Both reactions are thermodynamically favorable under hydrothermal conditions, with negative Gibbs free energy encouraging spontaneous progress. However, industrial performance depends mainly on kinetic and hydrodynamic factors, not just on equilibrium thermodynamics.

Continuous reactors may reduce ammonia losses compared to open-batch systems, as the enclosure enables gas recovery and pressure control. However, solids buildup and carbonate scaling are key challenges. Zhang et al. (2025) noted that localized precipitation near feed zones can change hydrodynamics and decrease effective residence time.

Current continuous PG–urea systems are mostly at TRL 4-5, with limited pilot demonstrations, especially with industrial phosphogypsum containing fluoride, phosphate, and radionuclide impurities.

7.3. Hydrothermal/autoclave systems

Hydrothermal and autoclave reactors are increasingly studied because high temperatures and pressures accelerate urea hydrolysis and phosphogypsum dissolution. Typical conditions are 100-180 °C under autogenous pressure, with some studies exploring nearly 200 °C (Yuan et al., 2024).

Hydrothermal conditions boost reaction kinetics by increasing water dissociation, ionic mobility, and dissolution. Elevated temperatures make urea hydrolysis more favorable due to entropy effects from ammonia and carbon dioxide, often leading to higher conversion efficiencies than atmospheric conditions.

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Laasri et al. (2025) reported sulfate conversion efficiencies over 90% under optimized hydrothermal conditions with controlled urea dosage and residence times below 2 h. The improved kinetics are mainly due to faster carbonate formation and better phosphogypsum dissolution. However, hydrothermal systems also increase ammonia volatilization risk if pressure isn't properly managed.

Autoclave systems provide several operational advantages:

- improved hydrolysis kinetics;
- reduced reaction time;
- better gas containment;
- enhanced conversion efficiency.

These systems have higher CAPEX due to the use of pressure-resistant materials, insulation, and gas seals. Corrosion resistance is vital as fluoride and acids can speed material degradation under hydrothermal conditions.

Scale formation is a major limitation, with calcium carbonate precipitation creating dense deposits on internal surfaces and heat-transfer zones, reducing thermal efficiency and increasing maintenance requirements (Khumalo et al., 2025). Higher solids loading complicates slurry handling due to increased viscosity and particle agglomeration.

Current hydrothermal PG–urea systems generally correspond to TRL 4–5 because pilot-scale demonstrations exist, but long-term industrial operation remains limited.

7.4. Fluidized/slurry reactors

Fluidized and slurry reactors are gaining interest for their potential to enhance gas-liquid-solid contact and reduce supersaturation. In these systems, particles stay dispersed via gas injection, agitation, or recirculation (Liu et al., 2021).

Fluidized systems can partly reduce passivation during phosphogypsum dissolution by continuously renewing reactive surfaces through particle motion. Improved mixing enhances carbonate distribution and reduces localized precipitation, leading to more uniform sulfate conversion than in traditional batch reactors.

Hydrodynamic conditions impact reactor performance. Excess gas velocity can enhance ammonia stripping, whereas poor mixing can cause sedimentation and incomplete conversion.

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Laasri et al. (2025) found that moderate slurry turbulence boosts overall efficiency by reducing diffusion limits around phosphogypsum particles.

The coupled precipitation reaction:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(s)$$

remains thermodynamically favorable throughout reactor operation. However, precipitation morphology depends on local hydrodynamics and supersaturation gradients. Fine carbonate particles from turbulent conditions may hinder downstream filtration and separation.

Fluidized-bed and slurry reactors can be partially integrated with gas recovery systems for ammonia recycling and CO_2 management. However, industrial use is limited by an insufficient understanding of large-scale hydrodynamic stability in impurity-rich slurry conditions.

Current maturity levels are approximately TRL 3–5, with variations depending on operational complexity and scale.

7.5. Process intensification

Process intensification strategies address limitations in conventional PG–urea systems through methods such as ultrasound-assisted hydrolysis, microwave heating, membrane integration, reactive crystallization, catalytic hydrolysis, and advanced slurry circulation systems (Ma et al., 2023).

Microwave-assisted systems speed up hydrolysis through rapid heating and localized energy transfer. Wang et al. (2023) found faster ammonia production under microwave heating than under conventional heating at the same temperatures. However, issues with energy efficiency and large-scale electromagnetic field distribution remain.

Ultrasound-assisted processing can boost dissolution and precipitation by reducing particle clumping and improving mixing. Cavitation may also renew phosphogypsum surfaces and boost carbonate nucleation (Dong et al., 2022). However, too much cavitation can produce ultrafine precipitates that hinder filtration.

Catalytic hydrolysis systems are a key route for intensification. Metal oxide catalysts, alkaline promoters, and heterogeneous surfaces can lower hydrolysis activation energy (Yurekli et al., 2024). However, catalyst poisoning by fluoride and phosphate impurities remains poorly understood in industrial phosphogypsum feeds.

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Membrane-integrated systems aim to improve ammonia recovery and water recirculation. Xu et al. (2025) suggest gas-permeation membranes can reduce nitrogen losses and boost sulfate conversion, but fouling and scaling are key issues in slurry environments.

Various reactor types for urea-assisted phosphogypsum conversion aim to optimize sulfate efficiency, kinetics, impurity control, energy, and scalability. Figure 5 shows the main types reported in the literature, including batch, continuous stirred-tank, hydrothermal autoclaves, slurry-loop, and intensified processes. Strategies like improved mixing, gas–liquid contact, heat integration, crystallization control, and hydrothermal methods address kinetic and transport issues. The trade-offs between reaction performance and scalability are noted: hydrothermal and intensified systems provide faster kinetics and higher sulfate conversion, while slurry reactors are more scalable. Most concepts are still at intermediate maturity, with challenges like fouling, corrosion, and downstream purification.

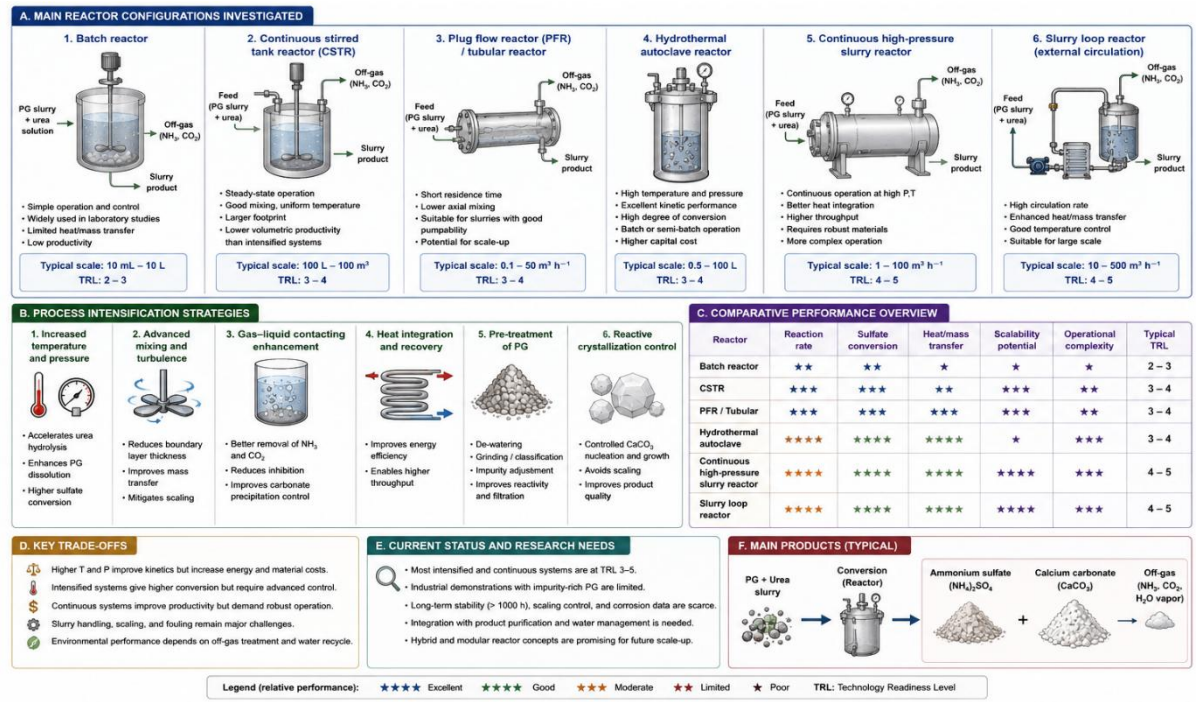


Figure 5. Main reactor configurations and process intensification strategies for PG–urea conversion systems, including batch, continuous, hydrothermal, slurry, and intensified processing routes. Adapted from Ma et al. (2023), Wang et al. (2023), and Sharma et al. (2025).

Figure 5 summarizes reactor setups for converting phosphogypsum with urea. Hydrothermal and intensified systems offer better kinetics, while continuous slurry systems

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may scale more effectively. Most intensified setups are at TRL 3-5 due to limited demonstrations involving impurity-rich phosphogypsum, stability, and purification.

8. Impurity Behavior and Product Purity

8.1. Fluorine behavior

Fluorine affects phosphogypsum conversion by influencing corrosion, ammonia stability, purity, and emissions. During wet-process phosphoric acid production, fluorapatite releases fluoride species that are incorporated into phosphogypsum as CaF_2, Na_2SiF_6, adsorbed fluoride, or residual fluorides (Cui et al., 2021).

Under acidic hydrolysis, fluoride may stay dissolved as HF or fluorosilicate complexes.

$$F^- + H^+ \rightleftharpoons HF$$

At low pH, the equilibrium favors the formation of HF because hydrofluoric acid dissociates only weakly. Although thermodynamically favored in acids, fluoride partitioning depends on calcium availability and ionic strength (Luo et al., 2022).

Calcium fluoride precipitation may partially immobilize fluoride:

$$Ca^{2+} + 2F^- \rightarrow CaF_2(s)$$

This reaction shows a strongly negative Gibbs free energy due to the low solubility of CaF_2. However, fluoride retention is incomplete in highly acidic or hydrothermal systems since fluoride speciation varies with temperature and pH.

Fluoride impacts urea hydrolysis and ammonium sulfate purity. Xiang et al. (2022) found that dissolved fluoride can change carbonate precipitation and disrupt calcite nucleation. It may also poison hydrolysis catalysts and worsen metal corrosion, especially under hydrothermal conditions.

Volatile fluoride compounds pose operational issues. Elevated temperatures boost HF and SiF4 gas-phase fluoride transport, needing dedicated scrubbing systems (Xiao et al., 2024). Thus, fluoride management is key for product quality, environmental compliance, and equipment longevity.

Current fluoride-control systems in sulfate conversion are mature (TRL 7–9), but integrated fluoride management for PG–urea systems is at TRL 3–5 due to limited understanding of impurity redistribution during hydrolysis and carbonate precipitation.

8.2. Phosphorus behavior

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Residual phosphorus compounds are key impurities impacting sulfate conversion and ammonium sulfate crystallization. In phosphogypsum, phosphorus occurs as adsorbed phosphoric acid, calcium phosphates, or as acidic liquids trapped during phosphoric acid production (Zhang et al., 2021).

Under hydrolysis, phosphate species dissolve based on pH-dependent equilibria.

$$H_3PO_4 \rightleftharpoons H_2PO_4^- + H^+$$

followed by:

$$H_2PO_4^- \rightleftharpoons HPO_4^{2-} + H^+$$

The Gibbs free energy for phosphate dissociation becomes more favorable at higher pH, promoting phosphate mobility in neutralized systems. However, calcium concentration still heavily influences phosphate because secondary calcium phosphate may precipitate.

Phosphate species can adsorb onto calcium carbonate, inhibiting crystal growth. Luo et al. (2022) found that phosphate adsorption alters calcite morphology and slows precipitation by blocking nucleation sites. These interactions may reduce sulfate conversion efficiency and affect downstream filtration.

Phosphorus contamination lowers ammonium sulfate purity by trapping phosphate species, affecting fertilizer quality. Xiang et al. (2022) found higher phosphorus reduces purity and increases washing needs.

In hydrothermal systems, phosphate redistribution is complex due to simultaneous dissolution and precipitation. Cherrat et al. (2020) shown that phosphorus partitioning depends on temperature, residence time, and calcium. Optimizing the process involves balancing sulfate conversion with impurity stabilization.

Most phosphorus-removal methods are at TRL 3–5 for integrated PG–urea systems, as continuous purification with industrial phosphogypsum remains unproven.

8.3. REE behavior

Rare earth elements in phosphogypsum, including yttrium, cerium, lanthanum, and neodymium, are gaining attention as they could enable additional valorization during sulfate conversion, depending on the phosphate rock origin.

REE distribution is influenced by acidulation chemistry and sulfate precipitation. Some REEs incorporate into calcium sulfate crystals; others adsorb onto surfaces or are trapped in

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residual acids (Khalil et al., 2025). During PG dissolution, REE release depends on dissolution kinetics and impurity binding.

REE sulfate dissolution may occur according to reactions such as:

$$REE_2(SO_4)_3 \rightleftharpoons 2REE^{3+} + 3SO_4^{2-}$$

The Gibbs free energy for REE sulfate dissolution varies with ionic strength and sulfate activity due to the variable solubility of lanthanide sulfates. High sulfate levels can stabilize REE complexes, while carbonate-rich conditions may cause secondary precipitation.

Carbonate precipitation introduces additional complexity because REEs may co-precipitate with calcite or form discrete carbonate phases:

$$2REE^{3+} + 3CO_3^{2-} \rightarrow REE_2(CO_3)_3(s)$$

Feng et al. (2025) noted that carbonate-rich environments can immobilize REEs during PG conversion, reducing recovery. Conversely, pH control may enable partial REE separation before carbonate precipitates.

Although REE recovery could improve process economics, most studies are limited to lab-scale systems. Smerigan and Shi (2025) note that industrial viability is uncertain due to low REE concentrations and separation challenges in impurity-rich sulfate systems.

Current REE recovery in PG–urea processes is at low TRL levels, mostly between TRL 2 and TRL 4.

8.4. Radionuclides

Radionuclide accumulation is a key environmental and regulatory issue associated with the use of phosphogypsum. During phosphate rock acidulation, radionuclides such as 226Ra, uranium, and thorium preferentially partition into phosphogypsum via sulfate co-precipitation (Chernysh et al., 2021).

Radium behavior is particularly important because Ra2+ presents chemical similarity to calcium:

$$Ra^{2+} + SO_4^{2-} \rightarrow RaSO_4(s)$$

Radium sulfate precipitation has a highly negative Gibbs free energy because of $RaSO_4$'s low solubility. As a result, radium stays bound to phosphogypsum solids during storage and chemical conversion.

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Hydrothermal conditions can redistribute radionuclides between solid and liquid phases. Hamed et al. (2024) found that acidic dissolution mobilizes uranium and thorium, whereas radium remains associated with sulfate or carbonate solids. This impacts ammonium sulfate purity and wastewater management.

Hassoune et al. (2020) noted radionuclide partitioning varies with pH and carbonate levels, as uranium can form soluble carbonate complexes in alkaline conditions. Stability depends on radionuclide concentration and chemical speciation.

Current radionuclide management in phosphate industries is mature (TRL 7–9), but radionuclide behavior during PG–urea conversion is less understood, around TRL 2–4 (Maina et al., 2025).

8.5. Product quality

Product quality hinders PG–urea conversion in industry, as fertilizer-grade ammonium sulfate requires strict control of impurities such as fluorides, phosphorus, metals, and radionuclides, thereby limiting its agricultural use (Orabi et al., 2024).

Ammonium sulfate purity depends on crystallization, washing, and impurity partitioning during hydrolysis. Cherrat et al. (2026) found purities of 90–95 wt% achievable under ideal lab conditions, but industrial feedstocks often yield lower purities due to impurity carryover.

Calcium carbonate quality depends on precipitation kinetics and impurities. Pyagai et al. (2022) found fluoride and phosphate adsorption can change particle morphology, reducing filler suitability. Fine particles may also hinder filtration and drying.

Table 7 summarizes impurities in ammonium sulfate and calcium carbonate during PG–urea conversion, comparing sources, behavior, partitioning, impacts, and purification challenges.

Table 7. Main impurities affecting ammonium sulfate and calcium carbonate quality during PG–urea conversion, including impurity sources, partitioning behavior, operational effects, and purification challenges. Adapted from Wu et al. (2026), Luo et al. (2022), Xiang et al. (2022), and Xiao et al. (2024)

Impurity	Main Source in PG	Main Chemical Forms	Main Partitioning Pathway	Main Operational Effects	Main Purification Challenges
Fluorine (F)	Fluorapatite	CaF_2, HF, Na_2SiF_6, F^-	Liquid phase, $CaCO_3$ surface	Corrosion, ammonia loss,	Difficult removal under acidic conditions

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	decomposition		adsorption, volatile HF	gas-treatment demand	
Phosphorus (P)	Residual phosphoric acid	H_3PO_4, $H_2PO_4^-$, Ca phosphates	Ammonium sulfate liquor, calcite adsorption	Reduced fertilizer purity, altered crystal growth	Co-precipitation and adsorption
Sulfate excess	Incomplete conversion	SO_4^{2-}, HSO_4^-	Residual mother liquor	Increased ionic strength, scaling	Water recirculation limitations
Silica (SiO_2)	Gangue minerals	Quartz, silicates	Solid residue	Filtration resistance, slurry viscosity	Fine-particle separation
Iron (Fe)	Phosphate ore impurities	Fe^{3+}, Fe hydroxides	Liquid and solid phases	Product discoloration, precipitation interference	Selective precipitation control
Aluminum (Al)	Aluminosilicates	Al^{3+}, Al hydroxides	Solution and solids	Increased sludge formation	pH-sensitive precipitation
Magnesium (Mg)	Dolomitic gangue	Mg^{2+}, Mg carbonates	Dissolved phase	Alters $CaCO_3$ polymorphism	Difficult selective separation
Sodium/Potassium	Process water and ore	Na^+, K^+ salts	Ammonium sulfate liquor	Crystal habit modification	High solubility prevents removal
Organic matter	Phosphate ore residues	Humic/organic compounds	Liquid phase and solids	Foaming, filtration instability	Adsorption and oxidation requirements
Rare earth elements (REEs)	Phosphate rock	REE sulfates/phosphates	Carbonate co-precipitation or liquor	Potential resource opportunity	Low concentration/selectivity
Uranium (U)	Natural radionuclides	Uranyl species	Solution and carbonate solids	Regulatory restrictions	Speciation-dependent mobility
Thorium (Th)	Natural radionuclides	Th compounds	Solid residue	Waste classification concerns	Low solubility but persistent
Radium (^{226}Ra)	Sulfate co-precipitation	$RaSO_4$-associated phases	Solid phase accumulation	Radiological management	Difficult removal from solids
Chlorides	Process water contamination	Cl^- salts	Liquid phase	Corrosion acceleration	Membrane sensitivity
Heavy metals (Cd, Pb, Zn)	Ore impurities	Metal ions/sulfates	Liquor and precipitates	Toxicity and product contamination	Multi-stage purification needed

Table 7 shows that the behavior of impurities in PG–urea systems is complex, with contaminants affecting hydrolysis, crystallization, filtration, corrosion, and environmental

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compliance. Impurity partitioning often affects purification costs and product quality more than it does sulfate conversion efficiency.

9. Downstream Processing and Separation

9.1. Solid-liquid separation

Solid-liquid separation is crucial in PG–urea systems, as filtration impacts ammonium sulfate purity, calcium carbonate recovery, water recirculation, and economics. Post-hydrolysis and sulfate conversion, the slurry typically includes ammonium sulfate solution, calcium carbonate, residual phosphogypsum, and impurity-rich particles (Pyagai et al., 2022).

Filtration behavior depends on carbonate polymorphism, particle size, slurry viscosity, and impurity adsorption. Calcite-rich systems usually filter better than vaterite-dominated ones because calcite forms denser, more permeable filter cakes (Orabi et al., 2024). However, excessive supersaturation creates ultrafine particles that lower permeability and raise filtration resistance.

Cake formation partly follows Darcy-type filtration, with resistance rising as particle size decreases and compressibility increases. In sulfate systems, phosphate and fluoride impurities can alter aggregation and lower settling rates. Zhang et al. (2025) found that high ionic strength promotes agglomeration, creating heterogeneous cakes with variable moisture.

The principal precipitation reaction governing solid generation remains:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(s)$$

Because the Gibbs free energy for calcite precipitation is strongly negative, precipitation occurs rapidly once carbonate supersaturation is reached. However, excessively rapid nucleation may produce poorly filterable solids and raise downstream separation costs.

Hydrocyclones, pressure filters, vacuum belt filters, and centrifuges are proposed for PG–urea systems. Vacuum filtration is attractive for its lower CAPEX and simplicity, but fine carbonate particles often reduce throughput. Centrifugal systems may boost solids recovery but usually increase energy use and maintenance.

Most downstream solid-liquid separation systems from the mineral and fertilizer industries are mature (TRL 7–9). However, integrated separation for PG–urea slurries with high impurities and mixed carbonate polymorphs is still at TRL 4–6.

9.2. Crystallization of ammonium sulfate

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Ammonium sulfate crystallization is the main purification and recovery step in PG–urea systems. After separation, the liquor contains dissolved ammonium sulfate and residual impurities like phosphates, fluoride, metals, and trace organics (Guo et al., 2021).

Crystallization typically occurs via cooling, evaporation, or combined systems. Supersaturation control is vital because ammonium sulfate nucleation and growth are sensitive to temperature, ionic strength, and impurities. The main reaction can be shown as:

$$2NH_4^+ + SO_4^{2-} \rightarrow (NH_4)_2SO_4(s)$$

The Gibbs free energy of ammonium sulfate crystallization becomes increasingly favorable as solution supersaturation increases. However, excessive supersaturation may promote secondary nucleation and the formation of ultrafine crystals, thereby reducing filtration efficiency and the quality of fertilizer-grade product.

Crystal morphology impacts handling and drying. Xu et al. (2025) found that controlled cooling produces larger, more uniform crystals with better flow and less moisture retention. Rapid evaporation leads to irregular crystals and the entrapment of impurities.

Impurity incorporation is a major crystallization challenge, with fluoride and phosphate species adsorbing onto ammonium sulfate surfaces or trapping in crystal agglomerates. Gong et al. (2024) noted that this issue worsens in concentrated sulfate solutions with limited washing.

Evaporative crystallization systems need significant energy because water removal is energy-intensive. Wang et al. (2023) estimated that downstream evaporation could be one of the biggest energy consumers in sulfate conversion systems, especially with dilute feed liquors after hydrolysis and washing.

Industrial ammonium sulfate crystallization tech are mature (TRL 8–9). However, crystallization with phosphogypsum conversion is at TRL 4–5 due to impurity-rich liquors and fluctuating sulfate levels, which complicate stable operation.

9.3. Purification strategies

Purification strategies are vital for producing fertilizer-grade ammonium sulfate and valuable calcium carbonate. Major impurities to control include fluorides, phosphorus, metals, organics, and radionuclides (Wu et al., 2026).

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Washing operations are a simple purification method used after carbonate filtration and ammonium sulfate crystallization. While water washing can remove some impurities, excessive washing increases water use and generates wastewater that requires treatment.

Fluoride removal frequently relies on calcium precipitation reactions:

$$Ca^{2+} + 2F^{-} \rightarrow CaF_2(s)$$

The negative Gibbs free energy of CaF_2 formation favors fluoride immobilization when calcium is available. However, Xiao et al. (2024) reported that fluoride removal declines under strongly acidic conditions because HF formation becomes thermodynamically favored.

Phosphate removal often uses selective precipitation with calcium, magnesium, or aluminum salts. Luo et al. (2022) found that partial phosphate removal prior to ammonium sulfate crystallization improved crystal purity and reduced the amount of washing required. However, additional precipitation stages increase reagent use and sludge production.

Activated carbon adsorption, membrane filtration, and ion-exchange systems have been studied for impurity control. Xiang et al. (2022) showed that membrane-assisted purification can lower dissolved fluoride and phosphate and partially recover water. However, membrane fouling and scaling are major limitations in concentrated sulfate solutions.

Purification requirements differ based on the final product. Fertilizer-grade ammonium sulfate tolerates some impurities, whereas industrial- and electronic-grade materials demand stricter purification. Calcium carbonate impurity tolerance also varies depending on its use, whether in cement, fillers, or agriculture.

Conventional sulfate purification methods are mature (TRL 7–9), but integrated purification for PG–urea systems stays between TRL 4 and 6 due to poorly understood impurity interactions under hydrothermal sulfate conversion.

9.4. $CaCO_3$ applications

The commercial viability of PG–urea conversion depends on the potential uses of the precipitated calcium carbonate by-product. Depending on its purity, morphology, and particle size, recovered $CaCO_3$ may be used in cement, fillers, paper, plastics, paints, wastewater treatment, or soil amendment (Czaplicka & Konopacka-Łyskawa, 2020).

Calcite is the most desired polymorph for industrial uses due to its higher stability and filtration skills. The precipitation reaction:

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$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(s)$$

remains thermodynamically favorable due to negative Gibbs free energy for calcite formation, but impurity incorporation may lower market value.

Nogqwazi (2020) reported that carbonate purity influences filler suitability as fluorides, phosphates, and residual sulfates can alter optical and rheological properties. High-purity PCC applications require stricter control of purification and crystallization.

In cement and construction, tolerance to impurities is higher. Khumalo et al. (2025) found carbonate from phosphogypsum can partially replace limestone in some cement systems, but radionuclide content and stability are key regulatory issues.

Particle morphology influences commercial use. Bao et al. (2024) showed that controlled precipitation can produce tailored crystal shapes for fillers or adsorption. However, maintaining morphology control in industrial impurity-rich conditions is challenging.

Downstream processing in PG–urea systems is vital for sulfate conversion, product recovery, impurity control, water management, and residue stabilization. Figure 6 shows an integrated flowsheet including slurry prep, hydrothermal conversion, separation, crystallization, calcium carbonate recovery, gas treatment, and wastewater management. It illustrates the link between impurity removal and product purification, such as fluoride removal, radionuclide stabilization, and solid separation. The diagram highlights integration of off-gas treatment, water recirculation, and residue management, affecting environmental and operational costs. Though units are mature, challenges like scaling, fouling, impurity buildup, and stability persist.

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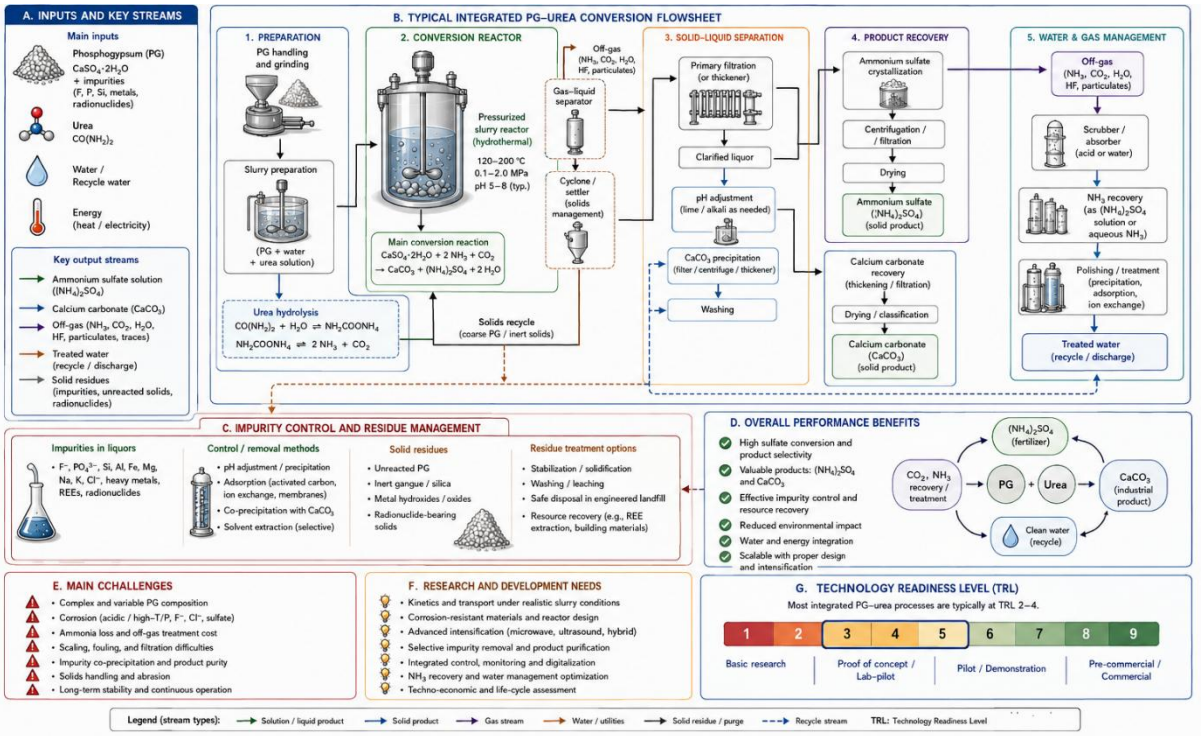


Figure 6. Downstream processing routes for PG–urea conversion systems, including filtration, crystallization, purification stages, and calcium carbonate utilization pathways. Adapted from Xu et al. (2025), Wu et al. (2026), and Bao et al. (2024).

Figure 6 summarizes key downstream operations in PG–urea systems, showing that separation often dictates process feasibility. Impurity control, filtration, crystallization, and water recirculation impact costs, efficiency, and product marketability. While individual units are mature, their integration in continuous systems is mainly at mid-tech stage (TRL 4–6).

10. Techno-Economic Feasibility and Scale-Up Challenges

Conventional ammonium sulfate is mainly produced via ammonia neutralization, caprolactam by-products, and flue-gas desulfurization, and is highly technologically mature (TRL 8–9) due to widespread industrial use (Hammas-Nasri et al., 2020). Conversely, PG–urea conversion systems remain mostly at TRL 3-5, as most research remains in labs or pilot stages. Although thermodynamic analysis shows promising sulfate conversion under hydrothermal conditions, economic success depends more on process integration, stability, impurity control, energy use, and reactor performance than on conversion efficiency alone.

One key advantage of PG–urea systems is that they use solid urea rather than gaseous ammonia. Urea, which is globally traded, stable under ambient conditions, and compatible with existing fertilizer infrastructure (Sergeev et al., 2020), contrasts with conventional ammonia-

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neutralization systems that often require pressurized storage, vaporization, gas scrubbing, and safety infrastructure, thereby increasing CAPEX and complexity (Swify et al., 2023). Urea-assisted conversion reduces dependence on external ammonia infrastructure by generating ammonia in situ via hydrolysis. However, slower hydrolysis kinetics often require higher temperatures and longer residence times for effective sulfate conversion (Avşar & Ertunç, 2025).

Energy consumption limits PG–urea conversion. Hydrothermal systems operate at 80-150 °C to speed hydrolysis and sulfate conversion (Yuan et al., 2024). Thermal demand mainly comes from slurry heating, pressure, evaporation, crystallization, drying, agitation, and circulation. Reactors can achieve over 90% sulfate conversion but raise OPEX due to steam and crystallization costs (Laasri et al., 2025). Heat integration and waste-energy recovery are proposed to improve economics (Zeng et al., 2026).

CAPEX and OPEX are influenced by reactor design and impurity management. Hydrothermal systems need pressure-resistant vessels, corrosion-resistant alloys, gas seals, and heat exchangers, raising initial costs (Kar et al., 2023). Operating costs mainly involve urea, thermal energy, solids handling, reagents, maintenance, and wastewater treatment. Removing fluoride and phosphorus often adds purification steps, making reagent use site-specific (Gong et al., 2024).

Scale-up limitations remain unresolved because most available studies rely on simplified laboratory systems operating under controlled feeding conditions. Under industrial operation, slurry rheology, hydrodynamic heterogeneity, solids transport, ammonia volatilization, filtration instability, and carbonate scaling become considerably more severe (Maina et al., 2025). Continuous operation additionally introduces uncertainty regarding crystallization stability and long-term process reliability (Lou et al., 2024). Laboratory conversion efficiencies may therefore overestimate industrial performance because many systems assume ideal mixing conditions and simplified impurity distributions (Wang et al., 2026).

Calcium carbonate precipitation further complicates industrial operation. Although carbonate formation thermodynamically promotes the conversion of sulfate, uncontrolled precipitation may cause severe fouling and scaling in reactors, pipelines, and heat exchangers

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(Chen et al., 2025). Scaling control and solids management may therefore become major operational constraints during continuous processing.

Table 8 summarizes the principal techno-economic factors affecting industrial implementation of PG–urea conversion systems.

Table 8. Main techno-economic factors affecting industrial implementation of PG–urea conversion systems, including reactor configuration, energy demand, operational limitations, and technological maturity. Adapted from Kazanc et al. (2024), Chen et al. (2025), and Maina et al. (2025).

Process / Reactor Configuration	Main Advantages	Main Limitations	Main OPEX/CAPEX Drivers	Main Scale-Up Challenges	Typical TRL
Batch reactor	Simple operation and flexibility	Long residence time	Heating, labor, vessel cost	Slurry heterogeneity	3–4
CSTR	Continuous operation	Scaling and solids accumulation	Mixing and maintenance	Fouling and RTD effects	4–5
Hydrothermal reactor	High conversion efficiency	High pressure and corrosion	Steam and pressure systems	Scaling and safety	4–5
Slurry-loop reactor	Improved heat transfer	Complex hydrodynamics	Pumping and abrasion	Solids transport stability	4–5
Membrane-integrated process	Improved impurity control	Membrane fouling	Replacement and cleaning	Long-term stability	4–5
Conventional NH_3 neutralization	Mature industrial route	Requires NH_3 infrastructure	Ammonia supply and storage	Mainly logistical	8–9

Table 8 demonstrates that the principal industrial barriers are not exclusively thermodynamic. Process economics are strongly controlled by energy integration, reactor durability, solids handling, scaling control, and long-term operational stability. While several individual unit operations are technologically mature, their integration into stable continuous phosphogypsum-conversion systems remains insufficiently validated under industrial conditions.

11. Environmental Sustainability and Circular-Economy Implications

The conversion of phosphogypsum (PG) into ammonium sulfate and calcium carbonate has attracted increasing attention within circular-economy frameworks because it simultaneously promotes waste valorization, sulfur recovery, and fertilizer production (Lou et al., 2024). Conventional phosphogypsum management still relies predominantly on large

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storage stacks, which are associated with acidic drainage, fluoride release, radionuclide accumulation, groundwater contamination, and long-term environmental liabilities (Chernysh et al., 2021). Consequently, PG conversion technologies may help reduce residue accumulation while generating commercially valuable products from industrial waste streams.

Environmental performance, however, depends strongly on impurity stabilization and process integration. In PG–urea systems, sulfate contained in calcium sulfate is recovered as ammonium sulfate fertilizer, while calcium is converted into precipitated calcium carbonate. Although thermodynamic analyses indicate favorable sulfate conversion under hydrothermal conditions, environmental benefit is not determined exclusively by conversion efficiency. Poor impurity management may redistribute fluorides, phosphorus compounds, radionuclides, and dissolved metals among liquid, gaseous, and solid streams rather than effectively mitigating environmental risk (Awad et al., 2024). Consequently, impurity partitioning and downstream treatment remain critical for sustainable industrial implementation.

Carbonate precipitation may additionally provide limited carbon-mineralization benefits because calcite formation contributes to partial CO_2 incorporation. Nevertheless, PG–urea systems should primarily be regarded as waste-valorization technologies rather than dedicated carbon-capture processes. Their environmental footprint remains highly dependent on thermal energy consumption because hydrothermal reactors, evaporation systems, and crystallization stages require substantial heat input (Kazanc et al., 2024). Fossil-derived energy sources may partially offset the environmental advantages associated with sulfate recovery and carbonate formation. For this reason, heat integration, waste-energy recovery, and external CO_2 utilization have been proposed as strategies to improve overall sustainability (Ma et al., 2023; Kang et al., 2022).

Resource efficiency represents another important advantage of integrated PG conversion. Sulfur recovery reduces dependence on virgin sulfate resources while generating fertilizers compatible with existing agricultural infrastructure (Guo et al., 2021). Water recirculation may additionally reduce freshwater demand, although impurity accumulation involving fluorides, phosphates, and dissolved salts frequently limits complete reuse in closed-loop systems (Dong et al., 2022). Therefore, environmental sustainability should be evaluated through integrated mass and energy balances rather than sulfate conversion efficiency alone (Gong et al., 2024).

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Regulatory acceptance remains one of the principal barriers to large-scale implementation because environmental regulations governing phosphogypsum reuse differ substantially among countries. Radionuclide redistribution under hydrothermal conditions remains insufficiently characterized, while fluoride emissions frequently require gas-scrubbing systems and dedicated wastewater treatment infrastructure (Hamed et al., 2024; Hassoune et al., 2020). These regulatory uncertainties continue to limit commercialization of PG-derived products despite increasing interest in circular-economy strategies.

Table 9 summarizes the principal environmental and sustainability-related variables affecting the long-term implementation of PG–urea conversion systems.

Table 9. Qualitative sensitivity ranking of environmental and sustainability-related variables affecting PG–urea conversion systems. Adapted from Kazanc et al. (2024), Gong et al. (2024), Maina et al. (2025), and Wang et al. (2026).

Variable	Main Environmental Concern	Relative Sensitivity
Energy demand	Carbon footprint and thermal emissions	High
Impurity management	Product contamination and environmental compliance	High
Water recirculation	Salt and contaminant accumulation	Medium–High
Fluoride emissions	Gas-treatment and atmospheric control	High
Wastewater treatment	Dissolved contaminants and discharge limitations	Medium–High
PG variability	Reproducibility and environmental consistency	Medium–High
Regulatory approval	Commercial acceptance and environmental licensing	High
REE recovery integration	Additional resource valorization potential	Medium
$CaCO_3$ marketability	Sustainable by-product utilization	Medium

Overall, PG–urea conversion should be regarded as an emerging sustainability-oriented waste-management strategy with significant circular-economy potential but still limited long-term industrial validation. Future environmental viability will depend on effective impurity stabilization, energy integration, regulatory standardization, and development of reliable continuous-operation systems capable of meeting industrial environmental requirements.

12. Limitations of the Review

This review highlights the scientific and engineering potential of urea-assisted phosphogypsum (PG) conversion, but several limitations must be acknowledged. First, the available literature remains strongly dominated by laboratory-scale studies conducted under simplified conditions using controlled feed compositions, relatively low impurity

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concentrations, and short operational periods. Consequently, many reported thermodynamic and kinetic performances may not accurately represent industrial operation under high-solids slurry conditions and variable phosphogypsum composition (Maina et al., 2025; Lou et al., 2024).

The scarcity of pilot-scale and continuous-operation studies additionally limits the review. Most available publications focus on reaction feasibility, sulfate conversion, and product characterization, whereas long-term reactor stability, slurry rheology, fouling, gas–liquid equilibrium control, and impurity accumulation remain insufficiently investigated. Technological readiness level (TRL) classifications presented throughout the review were inferred from reported operational evidence and should therefore be interpreted as approximate engineering estimates rather than standardized industrial certifications.

Another limitation involves the heterogeneity of phosphogypsum itself. Industrial PG composition varies substantially according to phosphate ore source, sulfuric acid conditions, crystallization route, washing efficiency, and impurity distribution. This variability complicates direct comparison among studies and may partially explain contradictory results reported for sulfate conversion, calcium carbonate precipitation, impurity redistribution, and ammonium sulfate purity.

The review also relied primarily on publications indexed in major scientific databases and on English-language technical reports. Although patent databases and industrial documents were consulted, unpublished industrial operational data remain limited. In addition, no formal risk-of-bias assessment or meta-analytical statistical treatment was performed because the objective of the manuscript was critical engineering interpretation rather than quantitative meta-analysis.

13. Future Research Directions

Future research should prioritize pilot-scale and continuous-operation validation because current technological maturity generally remains between TRL 3 and TRL 5 despite favorable thermodynamic performance. Industrial implementation requires stable operation under variable phosphogypsum composition, realistic impurity loads, and high-solids slurry conditions (Wang et al., 2026). Continuous hydrothermal reactors, intensified crystallization

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systems, and integrated ammonia-recovery units represent particularly important research directions.

Kinetic limitations associated with urea hydrolysis also require further investigation. Although the hydrolysis reaction is thermodynamically favorable, conversion rates remain slow under mild conditions because the activation energy is relatively high. Catalytic hydrolysis, therefore, represents a major opportunity for process intensification. However, catalyst poisoning caused by fluoride, phosphate, and dissolved metals under industrial conditions remains poorly understood and requires systematic investigation.

Impurity management constitutes another major research priority. Fluoride and phosphorus redistribution strongly affect ammonium sulfate purity, corrosion, wastewater treatment, and environmental compliance (Luo et al., 2022). Future studies should therefore investigate impurity stabilization, selective removal, gas-treatment systems, and dynamic impurity partitioning during continuous operation. Standardized characterization protocols for industrial phosphogypsum would also improve process comparability and scale-up reliability.

Integrated valorization strategies involving rare earth element (REE) recovery may improve process economics, although most current REE recovery systems remain below TRL 4. Additional work is needed to evaluate selective extraction, impurity interactions, and downstream purification under realistic sulfate-conversion conditions.

Finally, future investigations should expand techno-economic analysis, life-cycle assessment (LCA), and dynamic process simulation. Many current studies emphasize sulfate conversion efficiency while neglecting energy integration, wastewater management, the demand for purification, and the marketability of ammonium sulfate and calcium carbonate products. Advanced modeling approaches involving artificial intelligence, reactive transport simulation, and digital process optimization may become increasingly important for industrial implementation and process-control strategies.

14. Conclusions

Urea-assisted conversion of phosphogypsum into ammonium sulfate and calcium carbonate offers a promising method for waste valorization, sulfur recovery, and fertilizer production in circular economies. The process involves phosphogypsum dissolution, urea hydrolysis, ammonium sulfate crystallization, and calcium carbonate precipitation within a

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coupled system. Thermodynamics indicates a favorable equilibrium, as carbonate precipitation drives the conversion of sulfate forward. The main reaction remains feasible under controlled hydrothermal conditions with adequate removal of carbonate and calcium.

Despite favorable thermodynamics, the process faces kinetic constraints. Urea hydrolysis rates are slow under mild conditions, often requiring high temperatures, hydrothermal reactors, or catalysts to achieve acceptable conversion, thereby increasing energy use and reactor complexity. Additionally, ammonia volatilization, slurry rheology, and passivation effects may lower sulfate conversion during continuous operation. Thus, industrial performance depends on thermodynamics, coupled transport phenomena, crystallization, and hydrodynamics.

Impurity management remains a key challenge in industry, with issues such as fluoride, phosphorus compounds, radionuclides, dissolved metals, and rare earth elements affecting product quality, corrosion, wastewater, and regulatory compliance. Impurity redistribution during hydrolysis and carbonate precipitation remains poorly understood, particularly in industrial phosphogypsum. Many laboratory studies report high conversion efficiencies in simplified systems that may not reflect real industrial conditions. As a result, product purification becomes a crucial economic and operational concern.

The industrial potential of PG–urea conversion remains significant, especially in regions with rising phosphogypsum disposal liabilities and high fertilizer demand. A key advantage is urea's use as a stable solid nitrogen carrier. Unlike traditional ammonia-based sulfate production, urea simplifies storage, transportation, and handling without relying on pressurized NH_3 infrastructure. These benefits are especially relevant for decentralized or medium-scale plants linked to phosphoric acid production.

Current technological maturity is low, with most integrated PG–urea systems at TRL 3-5 due to limited long-term validation, continuous data, and industrial stability. Studies mainly use lab batch reactors under controlled conditions, with limited evaluation of scale-up, fouling, impurity buildup, and process integration.

Future development opportunities are significant. Key research includes catalytic hydrolysis, continuous reactors, impurity management, REE recovery, membrane purification, and energy integration with industrial facilities. Pilot validation and techno-economic assessment are vital for converting PG–urea from a lab concept to a viable industry technology.

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With proper process integration and impurity control, this route could reduce waste, produce fertilizer, and improve resource efficiency in phosphate industries.

Declarations

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Conflicts of Interest

The author declares no conflict of interest.

Data Availability Statement

No new experimental data were generated during this study. All information analyzed in this review was obtained from published literature sources.

Author Contributions

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

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