Hydrometallurgical Recovery of Potassium from Potassium-Bearing Micas: Comparative Leaching Behavior of Biotite and Phlogopite.

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ABSTRACT

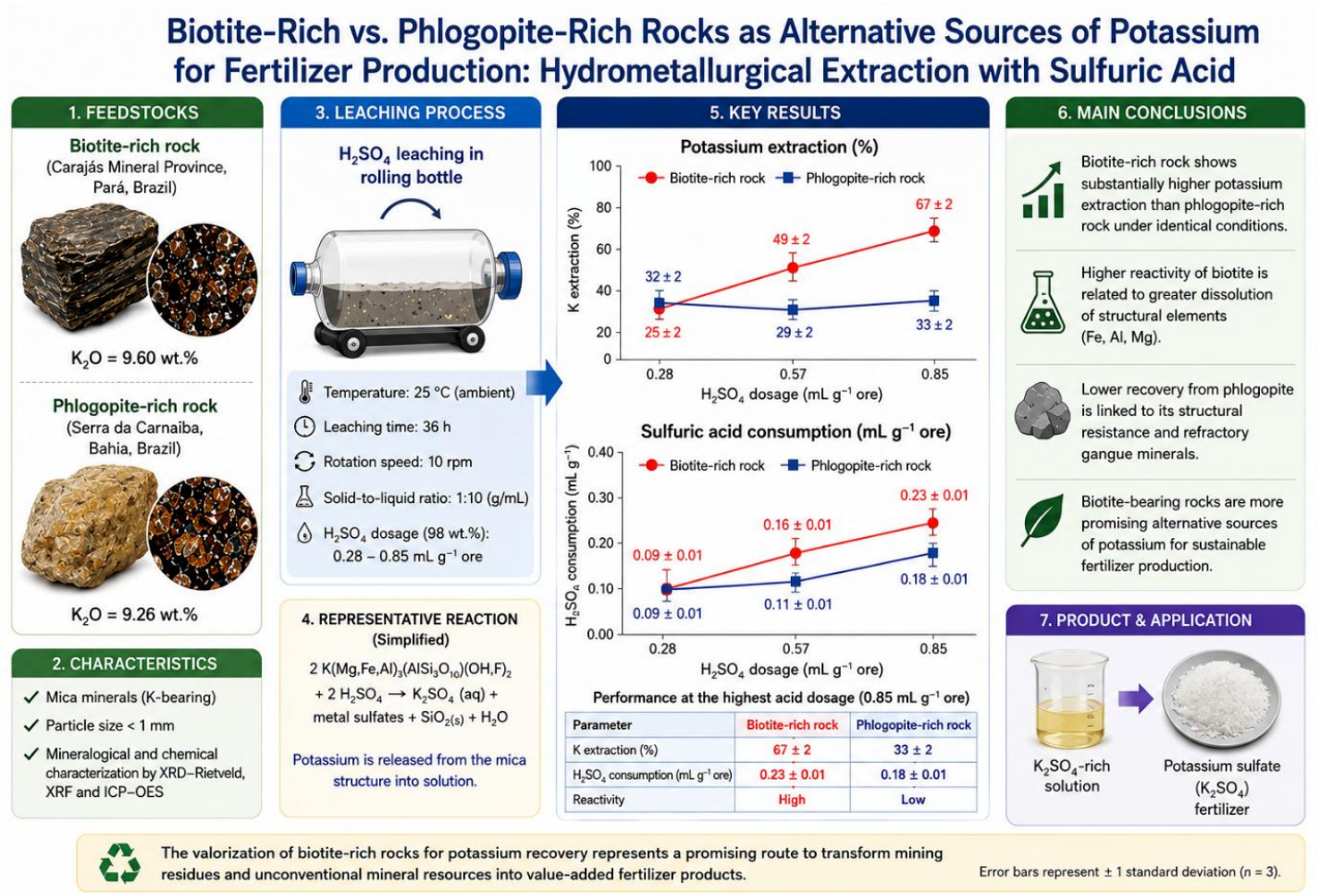
Potassium-bearing mica minerals have attracted increasing attention as alternative feedstocks for fertilizer production due to their abundance in mineral resources and mining residues. This study evaluated the hydrometallurgical extraction of potassium from biotite and phlogopite through sulfuric acid leaching under ambient conditions. Biotite-rich rock from the Carajás region (Pará, Brazil) and phlogopite-rich rock from Serra da Carnaíba (Bahia, Brazil) were characterized by mineralogical and chemical analyses prior to leaching. The materials contained 9.60 wt.% and 9.26 wt.% K_2O, respectively. Leaching experiments were conducted in a 5 L rolling bottle operated at 10 rpm for 36 h at 25 °C using particles smaller than 1 mm and H_2SO_4 dosages ranging from 0.28 to 0.85 mL g^{-1} of ore. Potassium extraction from biotite increased from 25% to 67% as acid dosage increased, whereas phlogopite exhibited significantly lower recoveries, ranging from 29% to 33%. At the highest acid dosage investigated (0.85 mL g^{-1}), sulfuric acid consumption reached 0.23 mL g^{-1} for biotite and 0.18 mL g^{-1} for phlogopite. The higher reactivity of biotite was attributed to its greater susceptibility to acid attack and dissolution of structural elements such as Fe, Al, and Mg. In contrast, the lower potassium recovery from phlogopite was associated with its lower leaching reactivity and the presence of refractory gangue minerals. The results demonstrate that biotite-bearing rocks represent a more promising alternative source of potassium for fertilizer production than phlogopite under low-temperature sulfuric acid leaching conditions. Furthermore, the process generates a potassium sulfate-rich solution and may provide a pathway to valorize potassium-rich mining residues and unconventional mineral resources.

Keywords: Hydrometallurgical processing; Biotite; Phlogopite; Potassium sulfate

Highlights

- Sulfuric acid leaching was applied to recover potassium from biotite- and phlogopite-rich samples.
- Biotite achieved a maximum potassium extraction of 67%, compared with 33% for phlogopite.
- Potassium recovery from biotite increased proportionally with sulfuric acid dosage.
- Phlogopite exhibited greater structural resistance to proton-induced dissolution.
- Structural cation dissolution (Al, Fe, and Mg) accompanied potassium release from the mica lattice.
- Biotite demonstrated superior potential as a feedstock for hydrometallurgical potassium sulfate production.

Graphical abstract



INTRODUCTION

Potassium is one of the three primary macronutrients required for plant growth and is essential for enzyme activation, osmotic regulation, photosynthesis, and crop productivity. The growing demand for agricultural fertilizers and concerns regarding the long-term availability of conventional potash resources have stimulated interest in alternative potassium-bearing minerals and secondary resources (Jena, 2021; Samantray et al., 2023). Although potassium is abundant in the Earth's crust, a significant proportion occurs within silicate mineral structures, where its low solubility limits direct agronomic utilization.

Conventional potassium fertilizers are predominantly produced from evaporitic deposits containing sylvite and carnallite. However, the geographical concentration of these deposits creates supply vulnerabilities and motivates the investigation of unconventional potassium sources, including feldspars, nepheline syenites, glauconitic rocks, and mica minerals (Jena, 2021; Jena et al., 2014). Among these materials, biotite and phlogopite have attracted particular attention because of their relatively high potassium contents and widespread occurrence in both primary mineral deposits and mining residues.

Mica minerals represent an important but underutilized potassium resource. Biotite and phlogopite commonly occur in igneous and metamorphic rocks and are frequently associated with mining wastes generated during the exploitation of gemstones, industrial minerals, and metallic ores. In Brazil, significant phlogopite-bearing deposits occur in the Serra da Carnaíba region, whereas biotite-rich rocks are found in several geological provinces, including the Carajás region (Lobato, 2009). The valorization of these resources may contribute simultaneously to fertilizer production and waste reduction.

The release of potassium from mica minerals is controlled by their crystal structure and the strong bonding of interlayer potassium ions. Consequently, the direct availability of potassium from untreated micas is generally low. Previous studies have demonstrated that potassium mobilization can be enhanced through thermal activation,

ion-exchange processes, biological weathering, and hydrometallurgical treatment (Bortun et al., 1998; Cheng et al., 2019; Song et al., 2013). Ion-exchange investigations revealed that potassium depletion rates vary among mica species and are strongly influenced by crystal chemistry and structural characteristics (Bortun et al., 1998). Similarly, lithium-ion exchange has been shown to promote potassium removal from biotite by modifying the interlayer environment and facilitating ion migration (Cheng et al., 2019).

Hydrometallurgical processing has emerged as one of the most promising routes for potassium recovery from silicate minerals because it can achieve substantial extraction efficiencies while operating under relatively moderate conditions. Sulfuric acid leaching has received particular attention due to its effectiveness in disrupting the mica structure and solubilizing potassium-bearing phases. Luo et al. (2015) reported potassium recoveries exceeding 90% from biotite under optimized sulfuric acid leaching conditions involving elevated temperature and acid concentration. More recently, Ma et al. (2020) investigated the kinetics and mechanisms of potassium dissolution from biotite in sulfuric acid solutions and demonstrated that leaching efficiency is controlled by both chemical reactions and diffusion.

Several studies have also explored alternative leaching agents and weathering mechanisms. Biological extraction using fungal species such as *Penicillium* and *Aspergillus* has been shown to enhance potassium release from biotite by producing organic acids and complexing agents (Song et al., 2013). Acidophilic microorganisms may further accelerate silicate dissolution processes by modifying local geochemical conditions and promoting mineral weathering (Dopson et al., 2009). In addition, organic acid systems have been successfully applied to the dissolution of biotite-bearing minerals, demonstrating the potential of environmentally friendly extraction routes (Zeng et al., 2021).

Despite these advances, important knowledge gaps remain regarding the comparative behavior of different mica minerals during hydrometallurgical processing. Most previous studies have focused primarily on biotite, whereas phlogopite has received considerably less attention despite its abundance in several mineral deposits and mining residues. Existing investigations suggest that phlogopite may exhibit distinct dissolution behavior due to its higher magnesium content and different crystal chemistry, which can significantly affect potassium release kinetics and extraction efficiency (Pereira, 2025; Silva et al., 2008).

Recent reviews have highlighted the need for additional experimental studies evaluating the influence of mineralogical composition, acid dosage, and leaching conditions on potassium recovery from mica-bearing materials (Jena, 2021; Samantray et al., 2023; Pereira, 2025). Comparative studies between biotite and phlogopite remain particularly scarce, limiting the development of optimized processing routes and the assessment of their potential as alternative raw materials for fertilizer production.

Therefore, the objective of this study was to evaluate the hydrometallurgical recovery of potassium from biotite and phlogopite through sulfuric acid leaching under ambient conditions. Particular emphasis was placed on comparing the leaching behavior, potassium extraction efficiency, acid consumption, and impurity dissolution characteristics of the two mica minerals in order to assess their suitability as alternative potassium sources for fertilizer production.

MATERIALS AND METHODS

Materials

Two potassium-bearing mica-rich rocks were investigated in this study. The biotite sample was collected from the Carajás region, Pará State, Brazil, whereas the phlogopite sample originated from the Serra da Carnaíba region, Bahia State, Brazil. Prior to testing, the samples were crushed, homogenized, and screened to obtain a particle size distribution of 100% passing 1 mm.

Mineralogical and Chemical Characterization

Mineralogical characterization was performed using optical microscopy. Polished sections were examined with a Leica DMLP optical microscope coupled to a digital image acquisition system. Quantitative mineralogical analysis was conducted using modal composition calculations based on the CIPW normative approach.

Chemical compositions were determined by inductively coupled plasma optical emission spectrometry (ICP-OES). Sample preparation involved complete dissolution by lithium metaborate fusion to ensure total digestion of the mineral matrix. The resulting solutions were analyzed using a PerkinElmer Optima 7300DV ICP-OES instrument.

The combined mineralogical and chemical characterization was used to identify the major potassium-bearing phases and associated gangue minerals present in the biotite- and phlogopite-rich rocks.

Sulfuric Acid Leaching Experiments

Hydrometallurgical extraction tests were carried out using sulfuric acid as the leaching reagent. Experiments were performed in a 5 L rolling bottle reactor operated at 10 rpm to maintain continuous agitation of the slurry. All tests were conducted at ambient temperature (25 °C) for 36 h of leaching.

Three initial sulfuric acid dosages were investigated according to the acid-to-ore ratio:

- 0.28 mL H_2SO_4 g^{-1} ore;
- 0.57 mL H_2SO_4 g^{-1} ore;
- 0.85 mL H_2SO_4 g^{-1} ore.

The particle size of all feed materials was maintained below 1 mm throughout the experimental program.

The principal response variables evaluated were:

- potassium extraction;
- sulfuric acid consumption;
- impurity dissolution;
- relative leaching reactivity of biotite and phlogopite.

Solid–Liquid Separation and Residue Treatment

At the end of each leaching test, the slurry was subjected to vacuum filtration to separate the pregnant leach solution from the solid residue. The filter cake was subsequently repulped in water at approximately 30 wt.% solids and filtered again to remove entrained soluble species.

The washed residues were dried at 120 °C for 6 h and weighed prior to chemical analysis. Residual elemental concentrations were used to determine the dissolution behavior of potassium and associated elements during leaching.

Determination of Acid Consumption and Potassium Extraction

Sulfuric acid consumption was calculated as the difference between the initial acid dosage and the free acid concentration remaining in solution after 36 h of leaching.

Free acidity was determined using the ammonium oxalate titration method. The concentrations of potassium and other dissolved elements were quantified using mass-balance calculations based on the chemical composition of the leach residues.

Potassium extraction was calculated according to Equation (1):

$$K(\%)=K_r/K_t*100$$

where:

- K is the potassium extraction (%);
- K_r is the mass of potassium dissolved during leaching;
- K_t is the total mass of potassium present in the initial sample.

This methodology enabled the comparative evaluation of the leaching behavior of biotite and phlogopite under identical hydrometallurgical conditions.

RESULTS AND DISCUSSION

Mineralogical Characteristics of the Phlogopite Sample

The mineralogical characteristics of the phlogopite-rich sample were investigated using optical microscopy under crossed-nicols to assess particle morphology, textural features, and potential implications for hydrometallurgical potassium recovery. The morphology and preservation of mica grains provide important information regarding mineral reactivity because potassium is located within the interlayer regions of the phyllosilicate structure. Representative photomicrographs of the investigated sample are presented in Figure 1.

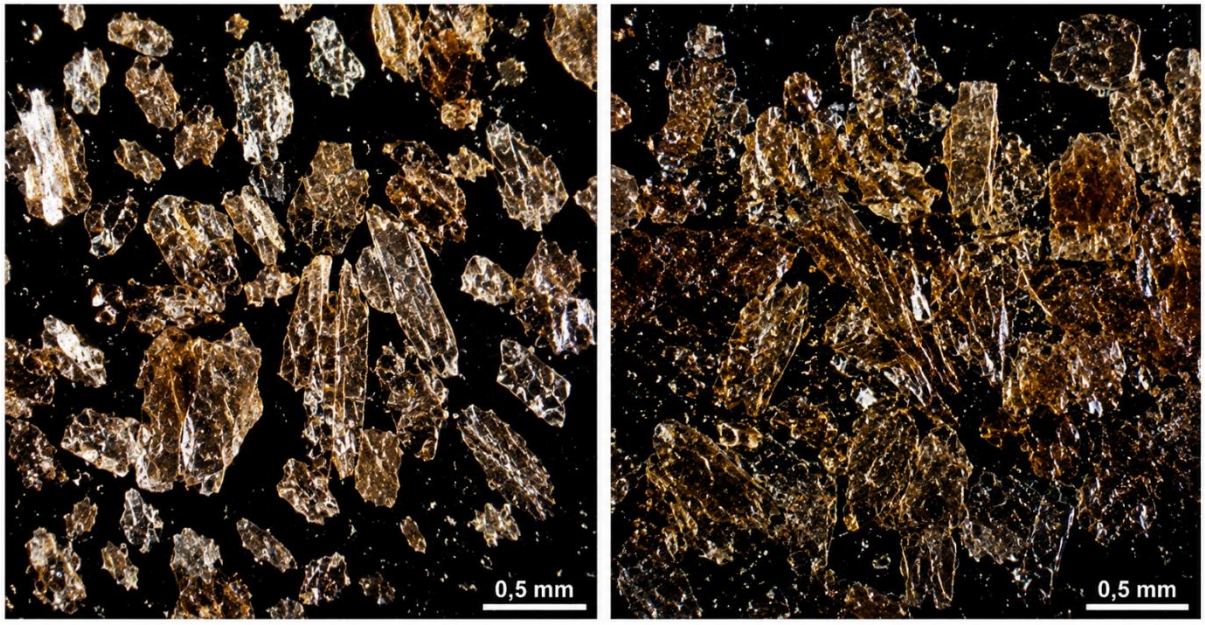


Figure 1. Optical microscopy images of phlogopite grains under crossed nicols showing brown-colored mica flakes with variable particle sizes. Adapted from: Pereira and Gomes (2021) and present study.

The photomicrographs reveal the characteristic platy morphology of phlogopite, with individual grains and aggregates displaying elongated to tabular shapes and well-developed cleavage planes. The brown-to-reddish-brown interference colors observed under crossed nicols are typical of magnesium-rich micas and reflect the optical properties associated with their layered crystal structure. The presence of particles with varying sizes and degrees of aggregation indicates a heterogeneous mineral population within the investigated sample.

The observed morphology is consistent with the crystal chemistry of phlogopite, commonly represented by the formula $KMg_3(AlSi_3O_{10})(F,OH)_2$. As a member of the mica group, phlogopite possesses a layered tetrahedral–octahedral–tetrahedral (TOT) structure in which potassium ions occupy interlayer positions between adjacent silicate sheets. The accessibility and stability of these interlayer sites play a critical role in determining potassium release during hydrometallurgical processing.

Many grains preserve their original crystal habit despite crushing and sample preparation, indicating a relatively high degree of structural integrity. Such preservation suggests that potassium remains strongly bound within the mica framework, contributing to the mineral's low natural solubility. The strong bonding of interlayer potassium and the structural stability of the magnesium-rich octahedral sheets are likely responsible for the limited potassium extraction observed during sulfuric acid leaching.

The mineralogical features identified in Figure 1 are consistent with the leaching behavior discussed later in the manuscript. Compared with biotite, phlogopite exhibited lower dissolution of structural elements and significantly lower responsiveness to increasing sulfuric acid dosage. These observations suggest that the

magnesium-rich mica framework is more resistant to proton-induced degradation, thereby restricting both structural decomposition and potassium release. Consequently, the microstructural characteristics observed in the photomicrographs provide direct mineralogical evidence for the lower hydrometallurgical reactivity of phlogopite under the conditions investigated.

Potassium Extraction by Sulfuric Acid Leaching

The effect of sulfuric acid dosage on potassium extraction from biotite- and phlogopite-rich samples was investigated to evaluate the relative leaching behavior of the two mica minerals. Because potassium is located within the interlayer sites of the mica structure, its release depends on the extent of structural degradation promoted by acid attack. Figure 2presents the potassium extraction achieved as a function of sulfuric acid dosage under identical experimental conditions.

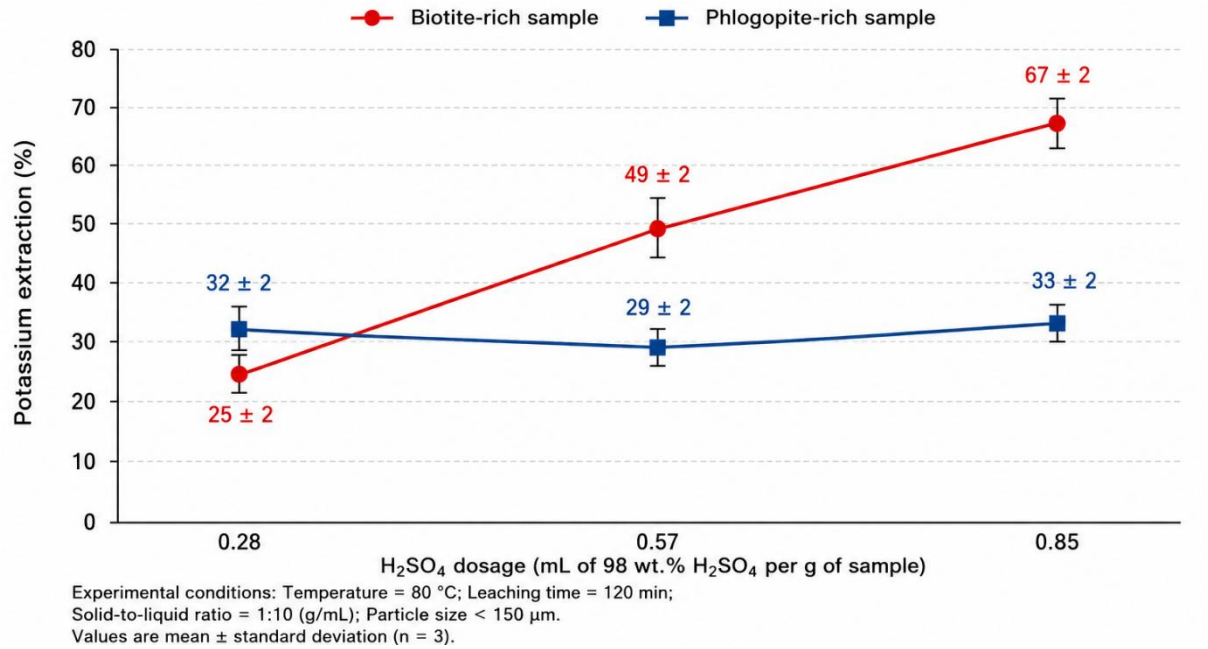


Figure 2. Potassium extraction from biotite- and phlogopite-bearing rocks as a function of sulfuric acid dosage. Adapted from: Pereira and Gomes (2021) and present study.

Figure 2 demonstrates a marked contrast in the leaching behavior of biotite and phlogopite under sulfuric acid leaching conditions. Potassium extraction from the biotite-rich sample increased significantly with increasing acid dosage, rising from 25% at 0.28 mL H_2SO_4 g^{-1} to 67% at 0.85 mL H_2SO_4 g^{-1}. This nearly threefold increase indicates that sulfuric acid effectively promotes the structural decomposition of biotite, thereby facilitating the progressive release of potassium from the mica framework.

The strong positive response of biotite to increasing acid dosage suggests that proton attack progressively destabilizes the aluminosilicate structure, exposing additional potassium-bearing sites and enhancing mineral dissolution. This behavior is consistent with previous investigations reporting the susceptibility of biotite to acid attack and the gradual breakdown of its crystal lattice during sulfuric acid leaching (Luo et al., 2015; Ma et al., 2020).

In contrast, phlogopite exhibited considerably lower responsiveness to increasing sulfuric acid dosage. Potassium extraction remained within a narrow range, from approximately 32% at the lowest dosage to 33% at the highest, with a slight decrease at the intermediate dosage. The limited variation indicates that increasing acid concentration alone is insufficient to substantially increase potassium release from phlogopite under the conditions investigated.

The different extraction trends stem from the crystal chemistry differences between biotite and phlogopite. Biotite's iron-rich octahedral sheets are more prone to proton-induced dissolution, weakening the structure and freeing potassium. Phlogopite's magnesium-rich structure resists sulfuric acid, maintaining lattice integrity and

limiting potassium access. Similar behavior in potassium-bearing silicates highlights structural stability as a key factor in potassium recovery (Bortun et al., 1998; Pereira, 2025).

The results clearly demonstrate that mineralogical composition strongly influences potassium extraction efficiency. Under the leaching conditions investigated, biotite exhibited substantially greater chemical reactivity and potassium recovery than phlogopite. These findings confirm the superior potential of biotite as a feedstock for hydrometallurgical potassium recovery and subsequent production of potassium sulfate fertilizer. Although phlogopite remains a potential alternative potassium source, additional process intensification strategies, such as finer grinding, elevated temperatures, thermal activation, or extended leaching times, may be required to achieve economically attractive recoveries.

Sulfuric Acid Consumption

Sulfuric acid consumption is a critical parameter for evaluating the technical and economic feasibility of hydrometallurgical potassium recovery processes. Although higher acid dosages generally promote greater mineral dissolution and potassium extraction, they also increase reagent consumption and operating costs. Therefore, understanding the relationship between sulfuric acid dosage and actual acid consumption is essential for optimizing process efficiency. Figure 3 presents the sulfuric acid consumption measured during the leaching of biotite and phlogopite as a function of acid dosage..

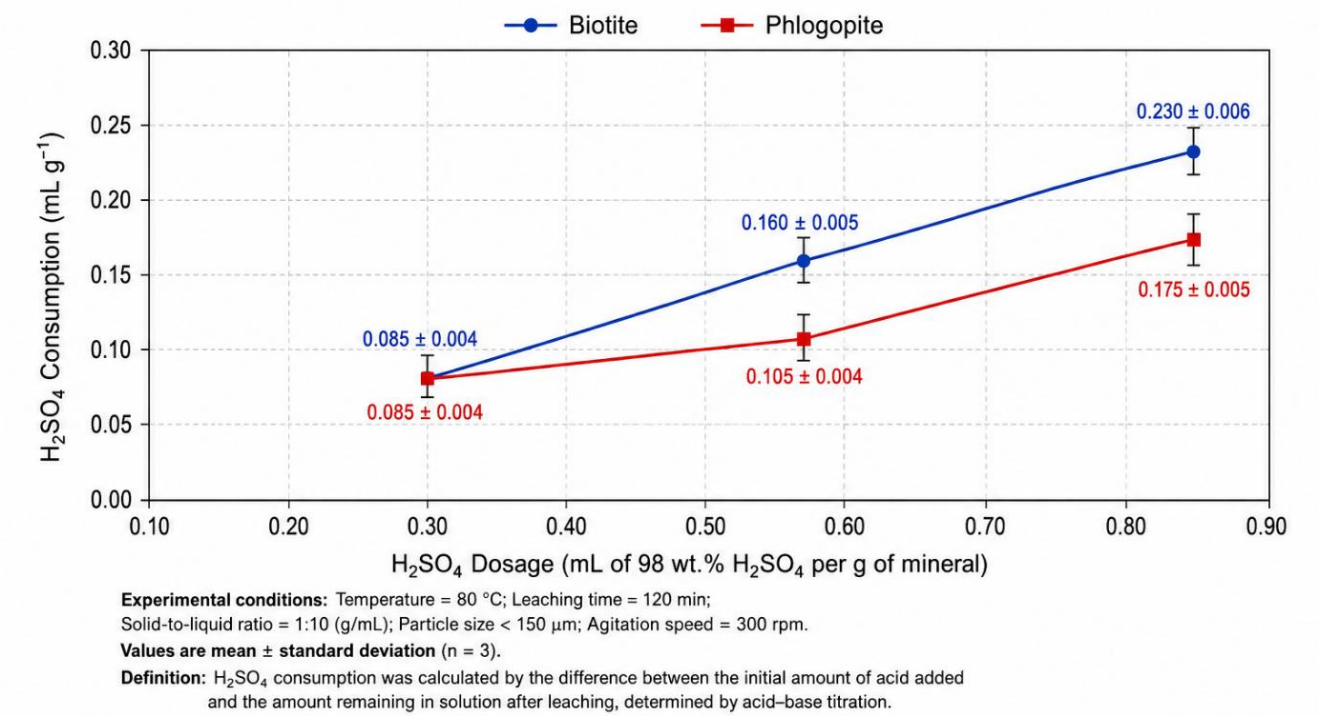


Figure 3. Sulfuric acid consumption as a function of sulfuric acid dosage for biotite and phlogopite samples. Adapted from: Pereira and Gomes (2021) and present study

Figure 3 illustrates the relationship between sulfuric acid dosage and acid consumption during the leaching of biotite and phlogopite. In both cases, acid consumption increased with increasing reagent dosage, reflecting the progressive dissolution of mineral phases and the occurrence of acid–mineral reactions throughout the leaching process. However, clear differences were observed between the two minerals, indicating distinct levels of chemical reactivity and potassium-release behavior.

For biotite, sulfuric acid consumption increased from approximately 0.085 mL g^{-1} at the lowest dosage to 0.230 mL g^{-1} at the highest dosage investigated. This increase closely paralleled the rise in potassium extraction observed in Figure 3, suggesting that the additional acid was effectively utilized to degrade the biotite lattice structure and release potassium. The substantial dissolution of Al, Fe, and Mg further supports the occurrence of extensive framework decomposition, indicating that potassium extraction proceeded through progressive breakdown of the mica structure rather than through simple ion-exchange reactions.

Phlogopite exhibited different behavior. Acid consumption increased from approximately 0.085 mL g^{-1} to 0.175 mL g^{-1} as sulfuric acid dosage increased; however, the corresponding improvement in potassium extraction was relatively limited. Although additional acid promoted some dissolution of structural components, particularly magnesium and aluminum, potassium recovery increased only marginally. This behavior suggests that a significant fraction of the acid was consumed by reactions involving structural cations, gangue minerals, and secondary alteration products rather than by the efficient liberation of potassium from the phlogopite lattice.

The divergence between acid consumption and potassium recovery becomes particularly evident at higher acid dosages. While biotite exhibited both increased acid consumption and substantially higher potassium extraction, phlogopite consumed additional acid without achieving comparable improvements in recovery. This observation indicates that the phlogopite structure is more resistant to proton-induced degradation and that structural stability and diffusion limitations may restrict potassium release under the investigated conditions.

These results demonstrate that acid consumption alone is not a reliable indicator of process performance and must be evaluated together with potassium extraction and impurity dissolution data. From a process-design perspective, biotite exhibits superior reagent utilization efficiency because the increase in acid consumption is accompanied by a proportional increase in potassium recovery. Consequently, biotite provides a more favorable balance between extraction performance and reagent consumption, reinforcing its potential as a feedstock for hydrometallurgical potassium recovery and potassium sulfate fertilizer production.

Dissolution of Associated Elements

The dissolution behavior of structural elements provides important information about the mechanisms controlling potassium release from mica minerals. Because potassium occupies interlayer sites in the crystal structure, its extraction is typically accompanied by partial dissolution of framework cations such as Al, Fe, and Mg. Therefore, evaluating the extraction of these elements can provide valuable insights into the extent of mineral decomposition and the relative reactivity of biotite and phlogopite during sulfuric acid leaching. The extraction percentages of aluminum, iron, and magnesium as a function of sulfuric acid dosage are presented in Figure 4.

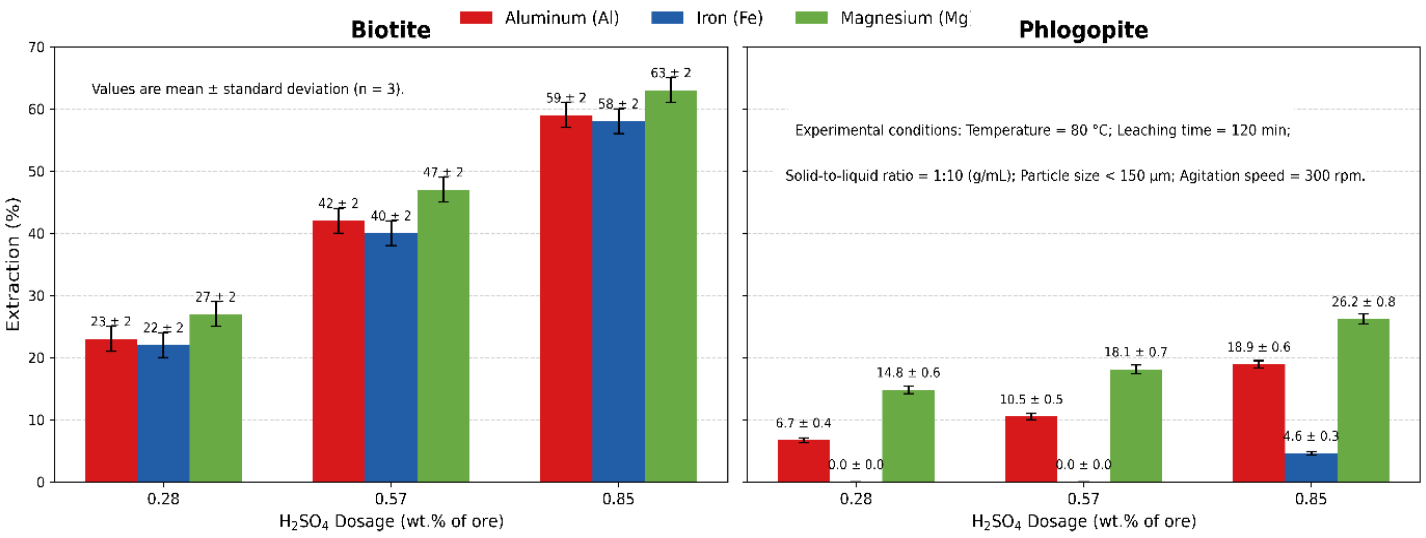


Figure 4. Dissolution of Al, Fe, and Mg as a function of sulfuric acid dosage during leaching of biotite and phlogopite samples. Adapted from: Pereira and Gomes (2021) and Present study.

Figure 4 illustrates the dissolution behavior of the principal structural elements present in biotite and phlogopite during sulfuric acid leaching. The results reveal marked differences in the chemical reactivity of the two mica minerals, reflecting their distinct crystal chemistries and acid resistance.

For biotite, the extraction of Al, Fe, and Mg increased consistently with increasing sulfuric acid dosage, reaching approximately 59%, 58%, and 63%, respectively, at the highest dosage investigated. The close correspondence between the extraction trends of these structural elements and potassium recovery indicates that leaching proceeds through progressive degradation of the biotite crystal framework. The substantial dissolution of Fe, Al, and Mg

suggests that potassium release is associated with the breakdown of the tetrahedral–octahedral–tetrahedral (TOT) structure rather than with a simple ion-exchange mechanism.

In contrast, phlogopite exhibited significantly lower dissolution of all evaluated elements. Magnesium showed the highest extraction, increasing from approximately 15% to 26%, while aluminum extraction increased from about 7% to 19% as acid dosage increased. Iron extraction remained negligible at lower acid dosages and reached only about 5% at the highest dosage investigated. These results indicate that the phlogopite structure is considerably more resistant to sulfuric acid dissolution than biotite, limiting both structural degradation and potassium release.

The pronounced difference in iron extraction between the two minerals is particularly significant. In biotite, iron constitutes an integral component of the octahedral sheets and is readily mobilized during acid attack, contributing to destabilization of the crystal lattice. In the phlogopite-bearing sample, however, a substantial fraction of the iron occurs in refractory accessory minerals such as hematite, magnetite, and ilmenite, which exhibit much lower solubility under the leaching conditions employed. Consequently, iron contributes less to the overall dissolution process and has a limited influence on potassium extraction.

The dissolution of Al, Fe, and Mg also indicates that potassium recovery is accompanied by the formation of sulfate-rich solutions containing multiple dissolved metal species. Depending on solution composition and downstream processing conditions, these species may subsequently form secondary products such as ferric sulfate, aluminum sulfate, magnesium sulfate, and mixed potassium-aluminum sulfate compounds.

Overall, the results demonstrate that mineralogical composition strongly influences leaching performance. Biotite exhibits substantially greater chemical reactivity, more extensive structural decomposition, and higher potassium recovery than phlogopite. These findings confirm the superior suitability of biotite as a feedstock for hydrometallurgical potassium recovery and highlight its potential as an alternative raw material for potassium sulfate fertilizer production.

Kinetic Considerations

Although the experimental program was conducted at a fixed leaching time of 36 h, the observed extraction trends provide valuable information regarding the mechanisms governing potassium dissolution from biotite and phlogopite. The leaching of potassium-bearing silicates is generally described using heterogeneous reaction models in which proton diffusion, surface reactions, and structural degradation occur simultaneously (Azimov & Bushmin, 2007, Han).

One of the most widely applied approaches for describing mineral dissolution is the shrinking core model (SCM). According to this framework, acid attack initially occurs on the external surface of the particle and progressively advances toward the particle interior. As dissolution proceeds, an unreacted core remains surrounded by a partially altered product layer. Depending on the mineralogical characteristics and operating conditions, the overall leaching rate may be controlled either by interfacial chemical reactions or by diffusion through the product layer (Clemens et al., 1987; Han et al., 2023).

The proportional increase in potassium extraction observed for biotite suggests that the leaching process was not limited by rapid surface passivation. Instead, progressive structural breakdown likely controlled potassium release. Potassium extraction increased from 25% to 67% as sulfuric acid dosage increased from 0.28 to 0.85 mL g^{-1}, indicating that additional acid continuously exposed new reactive surfaces and promoted further destruction of the mica structure (Kalinowski & Schweda, 1996).

In contrast, phlogopite exhibited only a marginal increase in potassium extraction despite increased acid dosage. This behavior suggests that diffusion limitations and structural resistance may play a more important role in controlling dissolution. Partial alteration layers formed during leaching may hinder proton transport to unreacted regions, reducing the effectiveness of additional acid addition (Lin & Clemency, 1981a; Kuwahara & Aoki, 1995).

The layered crystal structure of mica minerals strongly influences their leaching behavior. Both biotite and phlogopite possess a 2:1 phyllosilicate structure composed of tetrahedral–octahedral–tetrahedral (TOT) sheets separated by potassium-rich interlayers. Potassium extraction, therefore, requires disruption of the crystal lattice and the liberation of interlayer cations. The higher reactivity of biotite may be attributed to the presence of iron-bearing octahedral sites that are more susceptible to proton attack than the magnesium-rich structure of phlogopite (Madakkaruppan et al., 2019; Shao et al., 2023).

The simultaneous dissolution of Fe, Al, and Mg observed during leaching supports a mechanism involving progressive structural degradation rather than simple ion exchange. If ion exchange were the dominant mechanism, potassium could be released without substantial dissolution of framework elements. Instead, the extraction of multiple structural cations indicates that sulfuric acid promotes partial decomposition of the aluminosilicate lattice (Lin & Clemency, 1981b, Ma et al., 2014).

Although definitive kinetic modeling requires experiments performed at multiple temperatures, particle sizes, and reaction times, the present results suggest that biotite dissolution is primarily controlled by chemical attack on the mineral structure, whereas phlogopite dissolution is more strongly influenced by structural resistance and diffusion constraints. Future studies should focus on quantitatively fitting shrinking-core and mixed-control kinetic models to determine activation energies and identify the dominant rate-controlling mechanisms.

Qualitative Thermodynamic Considerations

The dissolution of biotite and phlogopite in sulfuric acid is fundamentally governed by thermodynamic driving forces associated with proton attack on the aluminosilicate framework. Under acidic conditions, hydrogen ions destabilize the mica structure, promoting the release of interlayer potassium together with structural cations such as Fe, Mg, and Al into solution (Song et al., 2025a).

A simplified representation of biotite dissolution may be expressed as:

$$\text{Biotite} + H_2SO_4 \rightarrow K^+ + Fe^{2+}/Fe^{3+} + Mg^{2+} + Al^{3+} + \text{dissolved silica} + \text{sulfate species}$$

Similarly, phlogopite dissolution may be represented as:

$$\text{Phlogopite} + H_2SO_4 \rightarrow K^+ + Mg^{2+} + Al^{3+} + \text{dissolved silica} + \text{sulfate species}$$

Although these equations do not represent balanced reactions, they illustrate the principal species generated during sulfuric acid attack on mica minerals. Potassium released from the interlayer positions remains dissolved in the sulfate-rich solution, creating favorable conditions for the subsequent production of potassium sulfate (K_2SO_4). Simultaneously, dissolved iron, aluminum, and magnesium may occur as free ions or as sulfate complexes depending on solution composition, pH, ionic strength, and oxidation state.

From a qualitative thermodynamic perspective, the observed differences between biotite and phlogopite can be associated with their distinct crystal chemistries. Biotite contains significant amounts of iron in octahedral positions, whereas phlogopite is predominantly magnesium-rich. The higher extraction efficiencies observed for biotite suggest that its crystal framework is less resistant to proton-induced destabilization under the investigated conditions. Progressive dissolution of Fe-bearing octahedral sites may facilitate structural weakening and enhance potassium release. In contrast, the Mg-rich phlogopite structure appears more resistant to acid attack, limiting both framework degradation and potassium extraction.

The dissolution process also involves competing acid-consuming reactions. Sulfuric acid is not consumed exclusively by potassium release but also by the dissolution of structural cations and accessory minerals. Consequently, the overall system consists of a complex network of simultaneous acid–mineral reactions involving mica degradation, sulfate formation, silica release, and impurity dissolution.

The sulfate-rich leach solutions generated during the experiments are expected to contain dissolved potassium together with varying concentrations of Fe, Al, and Mg sulfates. Under appropriate concentration and purification conditions, potassium sulfate may subsequently crystallize from solution. However, the presence of dissolved

impurities may influence crystallization behavior and product quality, requiring additional downstream purification steps.

The thermodynamic interpretations presented herein should be considered qualitative because no equilibrium modeling or speciation calculations were performed. Therefore, the stability of dissolved species, sulfate complexes, secondary precipitates, and potential reaction products was not quantitatively evaluated. Future investigations should incorporate thermodynamic modeling using software packages such as HSC Chemistry, FactSage, PHREEQC, or equivalent tools to determine equilibrium phase assemblages, predict sulfate stability, evaluate solution speciation, and quantify the influence of acid dosage on potassium recovery.

Despite these limitations, the experimental results are consistent with previous studies reporting that increased availability of sulfuric acid promotes greater destabilization of mica structures and enhances potassium extraction from potassium-bearing silicates (Luo et al., 2015; Ma et al., 2020; Song et al., 2025b). Nevertheless, thermodynamic favorability alone does not guarantee efficient extraction, as kinetic constraints may significantly limit the extent of mineral dissolution at ambient temperatures.

Comparative Assessment of Alternative Potassium-Bearing Minerals

The search for alternative potassium sources has expanded considerably due to concerns regarding long-term supply security and the uneven geographical distribution of conventional potash deposits. Several potassium-bearing silicate minerals have therefore been investigated as potential raw materials for fertilizer production (Jena, 2021; Samantray et al., 2023).

Table 1 compares the principal characteristics of selected potassium-bearing minerals reported in the literature.

Table 1. Comparison of selected alternative potassium-bearing minerals for fertilizer production. Adapted from: Jena (2021), Samantray et al. (2023), and references therein.

Mineral	Typical K_2O (%)	Typical Recovery (%)	Main Limitation
Biotite	8–10	25–95	Acid consumption
Phlogopite	8–10	20–40	Low reactivity
Glauconite	6–8	20–60	Slow dissolution kinetics
Verdete	7–14	10–70	Mineralogical variability
Feldspar	10–16	5–50	High structural stability

Among these minerals, biotite exhibits one of the most favorable combinations of potassium content and hydrometallurgical reactivity. The present study demonstrated that potassium extraction from biotite reached 67% under ambient conditions, significantly higher than that achieved from phlogopite.

Limitation Regarding Residue Characterization

The present study focused on evaluating the comparative potassium extraction behavior of biotite-rich and phlogopite-rich rocks under sulfuric acid leaching conditions. Chemical characterization of the feed materials was performed using ICP-OES, while mineralogical compositions were estimated from petrographic and mineralogical analyses, which revealed biotite and phlogopite as the dominant potassium-bearing phases.

Based on the initial mineralogical composition, the high Fe_2O_3 content of the biotite-rich sample (12.6 wt.%) combined with the low abundance of magnetite (2%) suggests that a significant fraction of iron is structurally incorporated within biotite. This observation is consistent with the higher dissolution of Fe, Al, and Mg observed during leaching and supports the interpretation that potassium release is associated with the progressive breakdown of the biotite structure.

For the phlogopite-rich sample, the mineralogical assemblage consisted predominantly of phlogopite (approximately 65%), together with quartz, chlorite, hematite, ilmenite, and minor magnetite. The lower potassium extraction efficiency observed during leaching is therefore attributed to the greater chemical stability

of the Mg-rich phlogopite structure and the presence of refractory accessory minerals that do not contribute to potassium release.

It should be noted that post-leaching residue characterization by X-ray diffraction (XRD), scanning electron microscopy (SEM), or energy-dispersive spectroscopy (EDS) was not performed in the present work. Consequently, the proposed dissolution mechanisms should be regarded as interpretations based on feed mineralogy, solution chemistry, and extraction behavior rather than direct evidence of mineralogical transformations. Future studies should include detailed characterization of leach residues to identify secondary phases, quantify residual potassium-bearing minerals, and further elucidate the mechanisms controlling potassium extraction from biotite and phlogopite.

Glauconite and verdete have attracted considerable attention in Brazil due to their abundance and their direct application potential as remineralizers. However, their potassium release rates are generally slow and often require thermal or chemical activation to achieve commercially attractive recoveries (Zeng et al., 2021).

Feldspars possess high potassium contents but are characterized by extremely stable crystal structures, making potassium extraction technically challenging and often economically unattractive. Consequently, mica minerals remain among the most promising unconventional sources of potassium for hydrometallurgical processing.

Sulfuric Acid Consumption and Process Efficiency

Sulfuric acid consumption represents one of the most important parameters affecting the technical and economic viability of potassium recovery processes. In the present study, acid consumption increased with increasing sulfuric acid dosage for both minerals, reflecting greater dissolution and impurity extraction.

To evaluate process efficiency, potassium recovery must be interpreted together with reagent consumption. Although biotite consumed more acid than phlogopite, the additional reagent usage resulted in substantially greater potassium extraction. Consequently, the overall acid utilization efficiency of biotite was superior to that of phlogopite.

The relationship between potassium extraction and acid consumption highlights the importance of optimizing reagent dosage. Excessive acid addition may increase operating costs without producing proportional increases in potassium recovery. This effect is particularly evident for phlogopite, where increased acid consumption yielded only marginal improvements in extraction efficiency.

A useful indicator for future process optimization is the ratio between potassium recovered and sulfuric acid consumed. Higher values indicate more efficient reagent utilization and lower operating costs. Based on the present results, biotite exhibits a significantly more favorable balance between potassium recovery and acid consumption than phlogopite.

From an industrial perspective, sulfuric acid consumption is expected to be a principal contributor to operating costs. Therefore, optimization strategies should focus on maximizing potassium recovery while minimizing reagent use by improving control of particle size, temperature, acid concentration, and residence time.

Overall, the results demonstrate that biotite not only yields higher potassium recovery but also offers superior process efficiency in terms of acid consumption, reinforcing its suitability as a feedstock for hydrometallurgical potassium production.

Potential Production of Potassium Sulfate

The production of potassium sulfate (K_2SO_4) represents one of the most attractive downstream applications of the sulfuric acid leaching process investigated in this study. Unlike conventional potash production, which is largely based on potassium chloride (KCl), the present route generates sulfate-rich pregnant leach solutions containing dissolved potassium ions. Consequently, potassium recovery and fertilizer production can potentially be integrated into a single hydrometallurgical process.

Potassium sulfate is considered a premium fertilizer because it supplies both potassium and sulfur while avoiding the addition of chloride to soils. This characteristic is particularly important for chloride-sensitive crops such as tobacco, potatoes, fruits, and several horticultural species. The increasing demand for specialty fertilizers has therefore stimulated interest in alternative routes for producing K_2SO_4 from non-conventional potassium resources.

During sulfuric acid leaching, potassium ions are released from the mica structure and remain in solution together with sulfate ions. Under suitable concentration, purification, and crystallization conditions, these ions can subsequently be recovered as potassium sulfate.

To evaluate the fertilizer production potential of the investigated materials, theoretical K_2SO_4 yields were estimated from the measured K_2O contents and the corresponding potassium extraction efficiencies obtained during leaching. The results are summarized in Table 2.

Table 2. Theoretical potassium sulfate production from biotite and phlogopite. Adapted from: Present study

Feed Material	K_2O Content (%)	Potassium Recovery (%)	Theoretical K_2SO_4 Production (kg t^{-1})
Biotite	9.60	67	~122
Phlogopite	9.26	33	~57

Table 2 demonstrates that biotite has substantially greater potential for potassium sulfate production than phlogopite under the investigated conditions. Owing to its higher potassium extraction efficiency, biotite could theoretically generate approximately 122 kg of K_2SO_4 per tonne of ore processed, whereas phlogopite would yield approximately 57 kg t^{-1}. This difference reflects both the higher leaching recovery and the superior overall process efficiency of biotite compared with other potassium-bearing feedstocks.

These values represent theoretical maximum yields and do not account for purification losses, crystallization efficiency, potassium retained in process residues, or other operational limitations. Nevertheless, the results highlight the potential of mica-bearing minerals as alternative raw materials for sulfate fertilizer production and indicate that improvements in leaching performance, particularly for phlogopite, could further enhance fertilizer output and process economics.

The presence of dissolved Fe, Al, and Mg in the pregnant leach solution may require additional purification steps before fertilizer-grade potassium sulfate can be produced. Conventional hydrometallurgical operations such as pH adjustment, selective precipitation, solvent extraction, or ion exchange may be employed to reduce impurity concentrations and improve product quality.

Overall, the results suggest that sulfuric acid leaching of biotite and phlogopite may provide a viable route not only for potassium extraction but also for the production of value-added potassium sulfate fertilizers from unconventional mineral resources and mining residues.

Preliminary Economic Considerations

The economic viability of potassium recovery from mica minerals depends on the balance between reagent consumption, mineral processing costs, product value, and feedstock availability. Although a detailed techno-economic analysis is beyond the scope of the present study, several important factors can be identified.

The principal operating costs are expected to include crushing, grinding, sulfuric acid consumption, solid–liquid separation, solution purification, and potassium sulfate recovery. Among these variables, sulfuric acid consumption is likely to be one of the most significant contributors to operating expenditure. Consequently, optimization of acid utilization is essential for process competitiveness.

Biotite demonstrated superior economic potential because it achieved higher potassium extraction with only a moderately higher acid consumption than phlogopite. The additional potassium recovered per unit of sulfuric acid consumed may compensate for the increased reagent requirement. In contrast, the limited extraction

observed for phlogopite suggests that larger quantities of ore would be required to produce equivalent amounts of potassium fertilizer.

The availability of low-cost raw materials represents another critical factor. Many biotite- and phlogopite-bearing rocks occur in mining residues, waste rock piles, and low-grade deposits that currently possess little or no economic value. The utilization of such materials could significantly reduce feedstock costs while simultaneously contributing to waste valorization and environmental management.

Compared with conventional potash production from sylvite and carnallite deposits, hydrometallurgical processing of mica minerals uses a lower-potassium feed. However, the process may become attractive in regions lacking access to conventional potash resources or where mining residues are readily available.

From a sustainability perspective, the proposed route offers additional benefits through resource diversification, reduced dependence on imported fertilizers, and the potential integration of mining waste utilization with fertilizer production. Future studies should therefore include detailed techno-economic assessments incorporating capital costs, reagent consumption, energy requirements, product quality, and market value.

State of the Art and Future Perspectives (2021–2026)

Research on alternative potassium resources has expanded considerably over the last decade, driven by growing concerns about fertilizer security, resource sustainability, and the utilization of low-grade mineral resources. Recent reviews have highlighted the growing importance of silicate minerals as potential sources of potassium fertilizers (Jena, 2021; Samantray et al., 2023).

The current state of the art indicates that several processing routes are available for the recovery of potassium from mica minerals, including thermal activation, ion exchange, biological weathering, and hydrometallurgical leaching. Among these approaches, sulfuric acid leaching remains one of the most effective methods because it can achieve relatively high potassium recoveries while operating under technically feasible conditions.

Before discussing future research opportunities, it is useful to situate the present work within recent advances in the recovery of potassium from potassium-bearing silicate minerals. As shown in Table 3, considerable progress has been made in understanding the leaching behavior of biotite and other unconventional potassium resources. Previous investigations have explored sulfuric acid leaching, kinetic modeling, and broader assessments of alternative potash sources, while recent review papers have highlighted both the opportunities and limitations associated with silicate-based fertilizers. The comparison presented in Table 5 summarizes key contributions from the literature and illustrates how the present study expands current knowledge by directly comparing biotite and phlogopite under identical hydrometallurgical conditions.

Table 3. Selected studies on potassium recovery from potassium-bearing silicate minerals. Adapted from: Jena (2021), Samantray et al. (2023), Pereira (2025), and present study.

Study	Mineral	Process	Main Outcome
Luo et al. (2015).	Biotite	Sulfuric acid leaching	>90% potassium recovery under optimized conditions
Ma et al. (2020).	Biotite	Kinetic analysis	Chemical reaction and diffusion mechanisms identified
Jena (2021)	Multiple sources	Review	Comprehensive assessment of alternative potash resources
Samantray et al. (2023).	Silicate minerals	Review	Evaluation of fertilizer potential and extraction technologies
Pereira (2025)	Phlogopite	Review	Identification of research gaps and agronomic opportunities
Present study	Biotite and phlogopite	Sulfuric acid leaching	Comparative evaluation under ambient conditions

Table 3 demonstrates the evolution of research from fundamental leaching investigations toward broader assessments of alternative potassium resources and sustainable fertilizer production. Luo et al. (2015) reported high potassium recoveries from biotite under optimized sulfuric acid leaching conditions, whereas Ma et al.

(2020) provided mechanistic insights into the kinetic controls governing potassium dissolution. More recent reviews by Jena (2021), Samantray et al. (2023), and Pereira (2025) emphasized the strategic importance of unconventional potassium sources and identified key technological challenges that still limit industrial implementation.

Within this context, the present study contributes new information by directly comparing the hydrometallurgical behavior of biotite and phlogopite under identical ambient-temperature leaching conditions. The results confirm the superior reactivity of biotite and provide experimental evidence supporting its greater suitability as a feedstock for potassium recovery and potential potassium sulfate production. Furthermore, integrating kinetic, thermodynamic, and process-efficiency considerations helps bridge the gap between laboratory-scale investigations and future industrial applications.

Despite recent progress, several scientific and technological challenges remain unresolved. First, detailed kinetic studies are still required to quantify activation energies and identify rate-controlling mechanisms under different operating conditions. Second, thermodynamic modeling tools such as HSC Chemistry, FactSage, and PHREEQC could provide valuable insights into solution chemistry, sulfate formation, and impurity behavior.

Another important research direction involves optimizing potassium sulfate recovery from pregnant leach solutions. Although potassium extraction has been widely investigated, comparatively fewer studies have addressed downstream purification and fertilizer production. Integrated flowsheets combining leaching, impurity removal, and crystallization therefore represent a promising area for future development.

Additional opportunities exist in the utilization of mining residues containing mica minerals. Such materials may provide low-cost feedstocks while simultaneously contributing to circular economy strategies and waste reduction. The combination of resource recovery and environmental remediation may significantly improve the overall sustainability of potassium production.

Finally, future investigations should incorporate pilot-scale testing and comprehensive techno-economic analyses. These studies will be essential for determining whether hydrometallurgical processing of mica-bearing materials can become a commercially viable complement to conventional potash production. The results obtained in the present work indicate that biotite represents a particularly promising candidate for such developments due to its superior leaching performance and higher potassium recovery.

CONCLUSIONS

This study evaluated the hydrometallurgical recovery of potassium from biotite-rich and phlogopite-rich rocks through sulfuric acid leaching under ambient conditions and demonstrated significant differences in the behavior of the two mica-bearing materials.

The main conclusions are summarized as follows:

1. **Biotite exhibited substantially higher potassium extraction than phlogopite under all investigated conditions.** Potassium recovery from biotite increased from 25% to 67% as sulfuric acid dosage increased from 0.28 to 0.85 mL g^{-1}, whereas phlogopite achieved only 32–33% extraction.
2. **The higher reactivity of biotite was associated with greater dissolution of structural Fe, Al, and Mg**, indicating that potassium release is accompanied by progressive destabilization of the mica framework.
3. **Phlogopite displayed significantly greater resistance to sulfuric acid attack**, which limited potassium extraction despite its comparable K_2O content. This behavior is consistent with the higher chemical stability of Mg-rich mica structures.
4. **Sulfuric acid consumption increased with increasing acid dosage for both materials.** Although biotite consumed slightly more acid, its substantially higher potassium recovery resulted in superior overall process performance.
5. **The leach solutions generated during the experiments provide a potential basis for downstream potassium sulfate production.** Theoretical mass balance calculations indicate production potentials of approximately 122 kg K_2SO_4 t^{-1} for biotite and 57 kg K_2SO_4 t^{-1} for phlogopite under the investigated conditions.

6. **The results demonstrate that biotite-rich rocks represent a more promising feedstock for hydrometallurgical potassium recovery than phlogopite-rich rocks**, particularly when sulfuric acid leaching is conducted at ambient temperature.
7. **The conclusions of the present study are limited to the experimental conditions investigated** (25 °C, 36 h, particle size <1 mm, and sulfuric acid dosages between 0.28 and 0.85 mL g^{-1}). Additional studies are required before extrapolating the results to other operating conditions or industrial applications.

Future work should focus on residue characterization, kinetic modeling, thermodynamic simulation, optimization of potassium sulfate recovery, pilot-scale validation, and comprehensive techno-economic assessment to further evaluate the industrial feasibility of potassium production from mica-bearing resources.

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