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POTASSIUM RECOVERY FROM BIOTITE-RICH ROCK BY THERMAL ACTIVATION AND SULFURIC ACID LEACHING

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Abstract

Potassium-bearing silicate minerals have emerged as potential alternative resources for fertilizer production due to increasing concerns regarding the sustainability, geographic concentration, and economic vulnerability of conventional potash reserves. Among these minerals, biotite is particularly attractive because of its relatively high potassium content and widespread occurrence in mineral deposits and mining residues. However, the strong incorporation of potassium within the mica structure limits its direct agronomic use and requires mineral activation to enhance potassium availability. This study evaluated the effect of thermal activation using chloride- and sulfate-based additives on potassium recovery from a biotite-rich rock from the Carajás Mineral Province, Brazil. The material contained approximately 69 wt.% biotite and 9.6 wt.% K_2O. Two activation systems, $CaCO_3$–$MgCl_2 \cdot 6H_2O$ and $CaSO_4 \cdot 2H_2O$–Na_2CO_3, were investigated at 800 and 900 °C for 6 h, followed by sulfuric acid leaching at pH 2–3, 85 °C, and 60 min. The chloride-based route achieved potassium recoveries of up to 63%, whereas the sulfate-based system reached 41%. Thermodynamic simulations predicted the formation of soluble potassium-bearing phases, including KCl and $KCaCl_3$ in the chloride system and aphthitalite-type compounds in the sulfate system. Iron and aluminum dissolution remained below 5%, demonstrating selective potassium extraction. The results indicate that chloride-assisted thermal activation effectively promotes structural modification of biotite and enhances potassium release. The process generated potassium-rich sulfate liquors that may serve as intermediate feedstocks for subsequent potassium sulfate fertilizer production following appropriate downstream purification, concentration, and crystallization steps. These findings highlight the potential valorization of potassium-bearing mineral resources and mining residues as alternative sources of potassium for agricultural applications.

JIRRS
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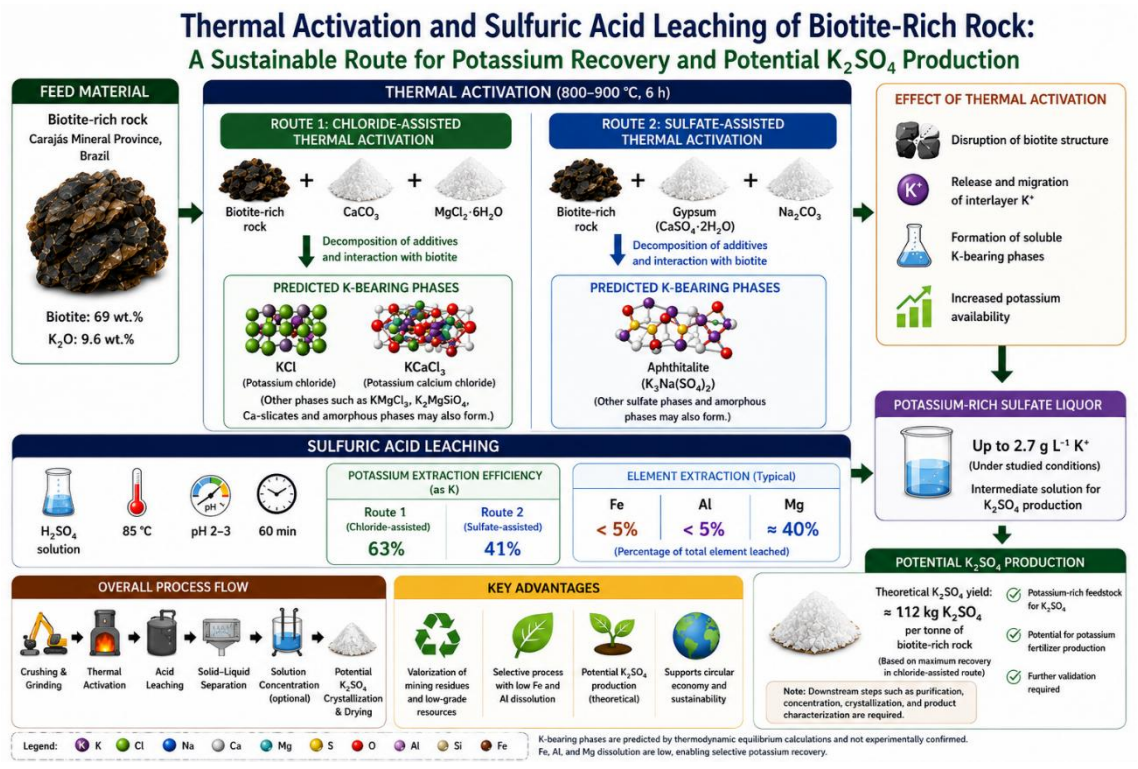
Pages: 1-41

Keywords: Biotite; Potassium recovery; Thermal activation; Sulfuric acid leaching; Potassium sulfate; Alternative fertilizers.

Highlights

- Thermal activation significantly enhanced potassium recovery from biotite-rich rock.
- Chloride-assisted roasting achieved potassium recoveries of up to 63%.
- Sulfate-assisted activation reached a maximum potassium recovery of 41%.
- Selective leaching limited iron and aluminum dissolution to less than 5%.
- Thermodynamic modeling predicted soluble potassium-bearing phases during roasting.
- Potassium-rich sulfate liquors may serve as intermediate feedstocks for K_2SO_4 production.

Graphical abstract



1. Introduction

Potassium is one of the three primary macronutrients required for plant growth and plays a fundamental role in enzyme activation, osmotic regulation, photosynthesis, carbohydrate transport, and stress resistance. Unlike nitrogen, which can be obtained from

JIRRS
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Vol: 01.08
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Pages: 1-41

atmospheric fixation, and phosphorus, which may be recycled through several biological pathways, potassium has no effective substitute in agricultural systems. Consequently, maintaining a secure and sustainable supply of potassium fertilizers is essential for global food production and agricultural productivity (Baila et al., 2025; Jena, 2021).

Current potassium fertilizer production relies predominantly on evaporite deposits containing sylvite (KCl) and carnallite ($KMgCl_3 \cdot 6H_2O$). These resources are geographically concentrated in a limited number of countries, creating supply-chain vulnerabilities and exposing agricultural markets to geopolitical and economic fluctuations. As a result, considerable attention has been directed toward developing alternative potassium sources to supplement conventional potash production (Baila et al., 2025; Jena, 2021; Harrouch et al., 2026).

Among the alternative resources investigated, potassium-bearing silicate minerals such as biotite, phlogopite, feldspar, nepheline syenite, glauconite, and verdete have received significant attention. These minerals are widely distributed throughout the Earth's crust and occur in numerous mineral deposits, low-grade ores, mine wastes, and processing residues. Their utilization could contribute to resource diversification while simultaneously supporting waste valorization and circular economy strategies (Jena, 2021; Mbissik et al., 2021; Pereira, 2025a; Pereira et al., 2026).

Biotite is a ferromagnesian mica belonging to the phyllosilicate group and is commonly represented by the generalized formula $K(Mg,Fe)_3AlSi_3O_{10}(OH,F)_2$. It occurs in a broad range of igneous and metamorphic rocks and is frequently associated with iron ore deposits, granitic intrusions, and alkaline rock complexes. Despite its relatively high potassium content, direct utilization of biotite as a fertilizer is limited by the strong bonding of potassium within the layered tetrahedral–octahedral–tetrahedral (TOT) crystal structure. Consequently, the release of potassium under natural weathering conditions is generally slow, restricting its agronomic effectiveness (Ma et al., 2020; Pereira, 2025a; Pereira & da Cunha Fonseca, 2025).

Several strategies have been proposed to enhance potassium availability from silicate minerals. These include mechanical activation, thermal treatment, alkaline roasting, chlorination, acid leaching, ion exchange, bioleaching, and combinations of

JIRRS
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DOI: 10.66104/dkjgbb35
Pages: 1-41

these approaches. Among them, thermal activation has emerged as a particularly promising route because it can modify mineral structure, destabilize potassium-bearing phases, and promote the formation of more soluble reaction products. Previous studies have demonstrated that thermal processing in the presence of suitable additives may significantly increase potassium extraction from mica-rich materials and other silicate minerals (Jena et al., 2020; Jena et al., 2022; Samantray et al., 2019; Pereira & da Cunha Fonseca, 2025).

The effectiveness of thermal activation depends strongly on temperature, additive chemistry, mineral composition, and reaction time. Chloride-bearing additives may facilitate the formation of soluble potassium chloride phases, whereas sulfate-bearing systems may generate intermediate potassium sulfate compounds. The thermodynamic stability of these phases and their subsequent behavior during leaching directly influence potassium recovery and impurity dissolution (Tanvar & Dhawan, 2022; Jena et al., 2022; Balogun et al., 2024; Xing et al., 2018).

Although several studies have investigated thermal activation of potassium-bearing silicates, significant knowledge gaps remain regarding the comparative effectiveness of different activation systems, the mechanisms governing potassium release, the selectivity of subsequent leaching processes, and the potential integration of these routes into fertilizer production chains. Furthermore, limited information is available concerning biotite-rich rocks from the Carajás Mineral Province and their suitability as alternative potassium resources (Baila et al., 2025; Harrouch et al., 2026; Pereira, 2025b).

Therefore, the objective of this study was to evaluate the effect of thermal activation using chloride- and sulfate-based additive systems on the hydrometallurgical recovery of potassium from a biotite-rich rock. Particular attention was given to thermodynamic interpretation, potassium extraction efficiency, impurity dissolution, and the potential application of the resulting potassium-rich solutions for fertilizer production.

2. Literature Review

2.1. Potassium-Bearing Silicate Minerals as Alternative Fertilizer Resources

JIRRS
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Concerns regarding the long-term security of conventional potash supplies have stimulated interest in alternative potassium-bearing minerals as potential fertilizer resources (Baila et al., 2025; Jena, 2021). Among the most widely investigated materials are feldspars, mica minerals, glauconite, nepheline syenites, and verdete, which occur abundantly in many geological settings and mining residues (Mbissik et al., 2021; Pereira et al., 2026).

Although potassium feldspars commonly contain 10–16 wt.% K_2O, potassium is strongly incorporated within a three-dimensional aluminosilicate framework, resulting in low natural reactivity and often requiring thermal, chemical, or mechanochemical activation to achieve significant extraction (Samantray et al., 2019; Jena, 2021). Nepheline syenites generally exhibit higher reactivity owing to the presence of feldspathoid minerals, which may facilitate potassium release under suitable activation conditions (Mbissik et al., 2021; Mbissik et al., 2023).

Glauconite and verdete have attracted attention as alternative fertilizer feedstocks, particularly in Brazil. However, their agronomic performance is often limited by slow potassium release, necessitating activation treatmentsto improve nutrient availability (Baila et al., 2025; Jena, 2021).

Compared with feldspars and glauconitic materials, mica minerals occupy an intermediate position because their layered structures provide greater accessibility to interlayer potassium, making them attractive targets for hydrometallurgical and thermochemical processing routes (Pereira, 2025b; Pereira et al., 2026).

2.2. Biotite as a Potassium Source

Biotite is one of the most abundant potassium-bearing micas and typically contains between 7 and 10 wt.% K_2O, together with significant concentrations of Mg, Fe, Al, and Si (Ma et al., 2020; Pereira, 2025a). Potassium occurs primarily within the interlayer region of the tetrahedral–octahedral–tetrahedral (TOT) structure, where it compensates for charge imbalances generated by tetrahedral substitutions.

Although interlayer potassium is more accessible than potassium contained within feldspar structures, its release remains limited under natural weathering conditions because of the structural stability of the mica framework (Ma et al., 2020; Pereira & da

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Pages: 1-41

Cunha Fonseca, 2025). Consequently, thermal, chemical, or mechanochemical activation is generally required to enhance potassium availability and improve process economics (Pereira, 2025b; Pereira et al., 2026).

2.3. Thermal Activation of Potassium-Bearing Minerals

Thermal activation is widely recognized as an effective strategy for increasing potassium extractability from silicate minerals. Roasting promotes dehydroxylation, structural disordering, phase transformation, and enhanced alkali mobility, thereby increasing susceptibility to subsequent leaching (Tanvar & Dhawan, 2022; Pereira & da Cunha Fonseca, 2025).

The effectiveness of thermal treatment depends on mineralogy, additive chemistry, roasting temperature, residence time, and particle size. Most studies report favorable activation between approximately 700 and 1000 °C, where substantial structural modification can be achieved without excessive melting or sintering (Jena et al., 2020; Tanvar & Dhawan, 2022).

Previous investigations involving feldspars, nepheline syenites, muscovite, phlogopite, and biotite consistently demonstrate improved potassium extraction after roasting, although recovery remains strongly dependent on the specific mineralogical and reaction pathways involved (Samantray et al., 2019; Mbissik et al., 2021; Harrouch et al., 2026).

2.4. Chloride- and Sulfate-Assisted Thermal Activation

Among additive-assisted roasting approaches, chloride- and sulfate-based systems are particularly attractive because they promote the formation of soluble potassium-bearing compounds.

Chloride-assisted activation typically employs additives such as NaCl, $MgCl_2$, or $CaCl_2$. During roasting, these reagents facilitate the conversion of structurally bound potassium into soluble chloride-bearing phases that are readily dissolved during subsequent leaching (Jena et al., 2020; Jena et al., 2022; Balogun et al., 2024).

Sulfate-assisted activation follows a different reaction pathway in which sulfate donors react with potassium-bearing minerals to form potassium sulfate-bearing products

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Pages: 1-41

or intermediate sulfate phases. Although this route frequently yields lower potassium recoveries, it may offer advantages for downstream fertilizer production because potassium sulfate is a commercially valuable fertilizer (Harrouch et al., 2026; Tanvar & Dhawan, 2022).

Consequently, chloride systems generally favor extraction efficiency, whereas sulfate systems may offer benefits in fertilizer compatibility and reduced chloride management requirements (Jena et al., 2022; Harrouch et al., 2026).

2.5. Previous Studies on Biotite Processing

Several studies have investigated potassium recovery from biotite through acid leaching, thermal activation, mechanochemical treatment, ion exchange, and biological approaches. Among these methods, sulfuric acid leaching has received considerable attention because it generates potassium-rich sulfate solutions that may be further processed for fertilizer applications (Ma et al., 2020; Pereira & da Cunha Fonseca, 2025).

Experimental evidence indicates that potassium release from biotite is controlled by progressive destruction of the mica framework and by the formation of more reactive phases during activation treatments (Ma et al., 2020). Roasting prior to leaching has consistently been shown to enhance potassium recovery by increasing structural disorder and promoting the formation of more soluble potassium-bearing products (Jena et al., 2020; Jena et al., 2022).

Despite these advances, direct comparisons between chloride- and sulfate-assisted activation routes applied to biotite-rich rocks remain limited. Furthermore, relatively little information is available regarding potassium recovery from biotite-bearing materials originating from the Carajás Mineral Province.

2.6. Research Gap and Scientific Contribution

Previous studies clearly demonstrate the importance of thermal activation for enhancing potassium recovery from mica minerals. Nevertheless, uncertainties remain regarding the relative performance of chloride- and sulfate-assisted roasting systems, the mechanisms responsible for potassium redistribution during activation, and the

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Pages: 1-41

implications of these routes for subsequent fertilizer production (Baila et al., 2025; Harrouch et al., 2026).

The present work contributes to this topic by comparatively evaluating chloride- and sulfate-assisted thermal activation of a biotite-rich rock from the Carajás Mineral Province, followed by sulfuric acid leaching and thermodynamic interpretation of phase evolution and impurity behavior. Particular emphasis is placed on understanding the mechanisms of potassium liberation and assessing the potential of biotite-rich materials as alternative potassium resources.

It should be noted that part of the experimental dataset discussed in this study was previously reported by Pereira et al. (2019). The present manuscript does not seek to reproduce the original investigation but rather to provide an expanded thermodynamic and mechanistic interpretation of those results, supported by updated literature analysis and broader discussion of process implications. Accordingly, the principal contribution of this work lies in the integration of thermodynamic modeling, comparative process evaluation, and interpretation of potassium recovery mechanisms rather than in the generation of a new experimental dataset.

3. Materials and Methods

3.1. Raw Material

The material investigated was a biotite-rich rock from the Carajás Mineral Province, Pará State, northern Brazil. This region is internationally recognized for its large iron ore deposits and associated silicate-rich lithologies. The selected sample was chosen for its elevated potassium content and abundant biotite, making it a potential alternative feedstock for fertilizer production.

The biotite-rich rock was crushed, milled, and sieved prior to the thermal activation experiments. The prepared material exhibited a particle size distribution with a P80 of approximately 0.15 mm. This particle size was selected to promote adequate contact between the mineral particles and the activation additives, while enhancing heat transfer and reaction kinetics during roasting.

The sieved material was subsequently homogenized and used in all thermal activation and leaching experiments.

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Pages: 1-41

3.2. Chemical Characterization

The chemical composition of the raw material was determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). Prior to analysis, representative samples were digested using lithium metaborate fusion to ensure complete dissolution of the silicate matrix.

Major oxides, including SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, Na_2O, and K_2O, were quantified. Particular attention was given to the potassium content because it directly determines the material's theoretical fertilizer production potential.

The analyzed biotite-rich rock contained approximately 9.6 wt.% K_2O, confirming its suitability as a potential potassium source for hydrometallurgical recovery.

3.3. Mineralogical Characterization

Mineralogical characterization was performed using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS). The analyses were conducted using a JEOL JSM-35C scanning electron microscope equipped with a NORAN Voyager 3050 energy-dispersive X-ray spectrometer. These analyses were used to identify mineral phases and determine their elemental compositions.

Quantitative mineralogical analyses were additionally performed using a QEMSCAN 650 automated mineralogy system equipped with two Bruker 5030 silicon drift detector (SDD) EDS units. The instrument was operated at an accelerating voltage of 25 kV and a beam current of 10 nA under standard operating conditions. Calibration of the backscattered electron (BSE) signal was performed using internal quartz and metallic copper standards.

The modal mineral composition of the samples was determined from the automated mineralogical data. Mineral abundance calculations followed the CIPW normative procedures described by Lopes (2012).

3.4. Thermal Activation Systems

3.4.1. Thermal Activation

Thermal activation experiments were conducted using two additive-assisted roasting systems designed to promote potassium liberation from the biotite-rich rock. The

JIRRS
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Pages: 1-41

sulfate-based activation route (S_1) consisted of biotite-rich rock, gypsum ($CaSO_4 \cdot 2H_2O$), and sodium carbonate (Na_2CO_3) mixed at a mass ratio of 1.0:0.3:0.2, respectively. The chloride-based activation route (S_2) consisted of biotite-rich rock, calcium carbonate ($CaCO_3$), and magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) mixed at a mass ratio of 1.0:0.2:0.3, respectively.

The prepared mixtures were subjected to thermal treatment at 800 and 900 °C for 6 h in a Jung model 0618 laboratory muffle furnace (Jung Ltd., Brazil) operating under atmospheric conditions. These temperatures were selected based on previous studies indicating that significant structural modification of mica minerals occurs within this range while minimizing excessive melting or sintering of the material.

Following roasting, the products were allowed to cool naturally to room temperature, homogenized, and subsequently prepared for sulfuric acid leaching and chemical characterization.

3.5. Thermodynamic Simulations

Thermodynamic equilibrium calculations were performed using FactSage 8.3 (Thermfact/CRCT, Montreal, Canada and GTT-Technologies, Aachen, Germany). The Equilib module was employed to predict phase stability and equilibrium phase assemblages during thermal activation.

The calculations were conducted at atmospheric pressure (1 atm), assuming an oxidizing atmosphere represented by air. The input compositions were defined based on the chemical composition of the biotite-rich rock and the additives used in each thermal activation route.

The following FactSage databases were employed:

- FactPS database for pure substances and gaseous species;
- FToxide database for oxide phases and solid solutions;
- FTsalt database for chloride and sulfate phases.

Equilibrium calculations were performed over the temperature range of 25–1000 °C, assuming complete thermodynamic equilibrium and minimizing the total Gibbs free energy of the system. The simulations were used to identify the formation and stability of

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Pages: 1-41

potassium-bearing phases, including KCl, $KCaCl_3$, K_2SO_4, and aphthitalite, and to support the interpretation of the experimental leaching results.

3.6. Sulfuric Acid Leaching

Potassium extraction was carried out by sulfuric acid leaching of the thermally activated materials. The pH of the leaching solution was maintained between 2.0 and 3.0 to minimize iron dissolution, following the recommendations of Kalinowsk and Schweda (1996). Leaching experiments were conducted at (85 ± 5) °C for 60 min under continuous mechanical agitation.

The initial pulp contained 20 wt.% solids, corresponding to an approximate liquid-to-solid ratio of 4:1 (w/w). Sulfuric acid was added as required to maintain the target pH throughout the leaching period. Upon completion of the experiments, the slurry was filtered and the resulting pregnant leach solution was collected for chemical analysis.

3.7. Analytical Procedures

Chemical analyses of the raw materials, thermally activated products, and leach solutions were performed by inductively coupled plasma optical emission spectroscopy (ICP-OES) using an Optima 7300 DV instrument (PerkinElmer Inc., Waltham, MA, USA).

Prior to ICP-OES analysis, solid samples were prepared by lithium metaborate fusion to ensure complete dissolution of the silicate matrix and quantitative recovery of the target elements. The fused samples were subsequently dissolved and diluted according to standard laboratory procedures before elemental determination.

Potassium concentrations in the pregnant leach solutions were measured by ICP-OES. Additional analyses were carried out to determine the concentrations of iron, aluminum, magnesium, calcium, and sodium, allowing evaluation of potassium selectivity and impurity dissolution during the leaching process.

The solid residues obtained after leaching were dried to constant mass and weighed to assess mass losses associated with thermal activation and hydrometallurgical treatment.

JIRRS
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Vol: 01.08
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Pages: 1-41

3.8. Potassium Recovery Calculation

Potassium recovery was calculated as the percentage of potassium transferred from the thermally activated solid to the leach solution during sulfuric acid leaching. The extraction efficiency was determined according to Equation (1):

$$R_K(\%) = \frac{C_K \times V}{m_f \times w_K} \times 100$$

where:

- R_K= potassium recovery (%);
- C_K= potassium concentration in the leach solution (g L^{-1});
- V= final volume of the leach solution (L);
- m_f= mass of feed material subjected to leaching (g);
- w_K= potassium mass fraction in the feed material (g K g^{-1} feed).

When potassium content was reported as K_2O, elemental potassium content was calculated using the stoichiometric conversion factor:

$$K = 0.8301 \times K_2O$$

The calculated recovery values were used as the primary performance indicator to compare the effectiveness of the sulfate- and chloride-assisted thermal activation routes.

3.9. Experimental Reproducibility and Data Origin

To ensure transparency regarding data provenance and reproducibility, the roasting and leaching experiments discussed herein are derived from the experimental campaign reported by Pereira et al. (2019). The analytical procedures, operating conditions, and experimental protocols employed in that study are described in Sections 3.1–3.9 for completeness and to facilitate interpretation of the thermodynamic simulations and mechanistic discussion presented in this manuscript.

The thermodynamic calculations performed using FactSage 8.3 represent new analyses conducted specifically for the present study. These calculations were used to provide a mechanistic interpretation of potassium redistribution during thermal activation

JIRRS
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Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

and to identify the equilibrium phases that may be responsible for the experimentally observed extraction behavior.

Because the original experimental work was conducted as a comparative process-evaluation study, replicate measurements and detailed uncertainty analyses were not reported in the original dataset. Therefore, the recovery values presented should be interpreted as representative process-performance indicators rather than statistically validated mean values. Future investigations should include replicate testing, uncertainty quantification, and complete elemental mass balances to further strengthen process evaluation.

4. Results and Discussion

4.1. Chemical Composition of the Raw Material

The biotite-rich rock's chemical composition is crucial for assessing its potential as a potassium resource and predicting its behavior during thermal and hydrometallurgical processing. Besides potassium, major oxides affect mineral stability, roasting reactions, and leaching dissolution. The material is silicate-rich, primarily composed of iron- and potassium-bearing biotite. ICP-OES analysis results are shown in Table 1.

Table 1. Chemical composition of the biotite-rich rock used in this study. Author's own data

Oxide	Content (wt.%)
SiO_2	42.2
Fe_2O_3	12.6
K_2O	9.6
Al_2O_3	4.3
MgO	4.3
CaO	1.0
Na_2O	0.3

Table 1 presents the complete chemical composition of the biotite-rich rock investigated in this study. The material is characterized by significant concentrations of SiO_2 (42.20 wt.%), Fe_2O_3 (12.60 wt.%), K_2O (9.60 wt.%), Al_2O_3 (4.30 wt.%), MgO (4.28 wt.%), and TiO_2 (2.05 wt.%). Minor constituents include CaO, Na_2O, MnO, P_2O_5, and trace amounts of Cu, Ni, Ba, and Cr. The difference required to close the mass balance to 100 wt.% is primarily attributed to loss on ignition (LOI/PF), which reflects the presence of structural hydroxyl groups, volatile components, and other minor phases not

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Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

individually reported in the oxide composition. Consequently, the apparent deficit observed when only the major oxides are summed does not indicate missing analytical data but rather the contribution of LOI and trace constituents.

The chemical analysis confirms that the investigated rock constitutes a potassium-rich silicate resource, containing approximately 9.60 wt.% K_2O. This potassium content is comparable to that reported for several mica-rich deposits and exceeds the values commonly found in many glauconitic and other low-grade silicate materials considered for alternative potassium fertilizer production. The relatively high concentrations of SiO_2 and Fe_2O_3 are consistent with the predominance of silicate minerals and iron-bearing biotite within the mineral assemblage, whereas the presence of Al_2O_3 and MgO further supports the occurrence of trioctahedral and dioctahedral mica phases.

Although the potassium content is significant, most of the element is structurally bound within the interlayer sites of the biotite crystal lattice and is therefore only sparingly available under natural conditions. This mineralogical constraint highlights the need for pretreatment technologies that can destabilize the aluminosilicate framework and convert potassium into more reactive, soluble forms. Consequently, the combination of a relatively high K_2O content and favorable mineralogical characteristics indicates that the investigated biotite-rich rock represents a promising non-evaporitic resource for potassium recovery through thermal activation followed by hydrometallurgical processing.

4.2. Mineralogical Characteristics

The geological setting and mineralogical characteristics of the investigated material are important for understanding its suitability as an alternative potassium resource and its behavior during thermal activation and hydrometallurgical processing. The occurrence, abundance, and distribution of potassium-bearing minerals directly influence the efficiency of potassium liberation and subsequent extraction. The biotite-rich rock evaluated in this study was collected from the Carajás Mineral Province, Pará State, northern Brazil, a region recognized for its extensive mineral resources and diverse geological formations. Figure 1 illustrates the location of the study area, the sampling

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Pages: 1-41

site, a representative hand specimen of the biotite-rich rock, and the field occurrence of the investigated material.

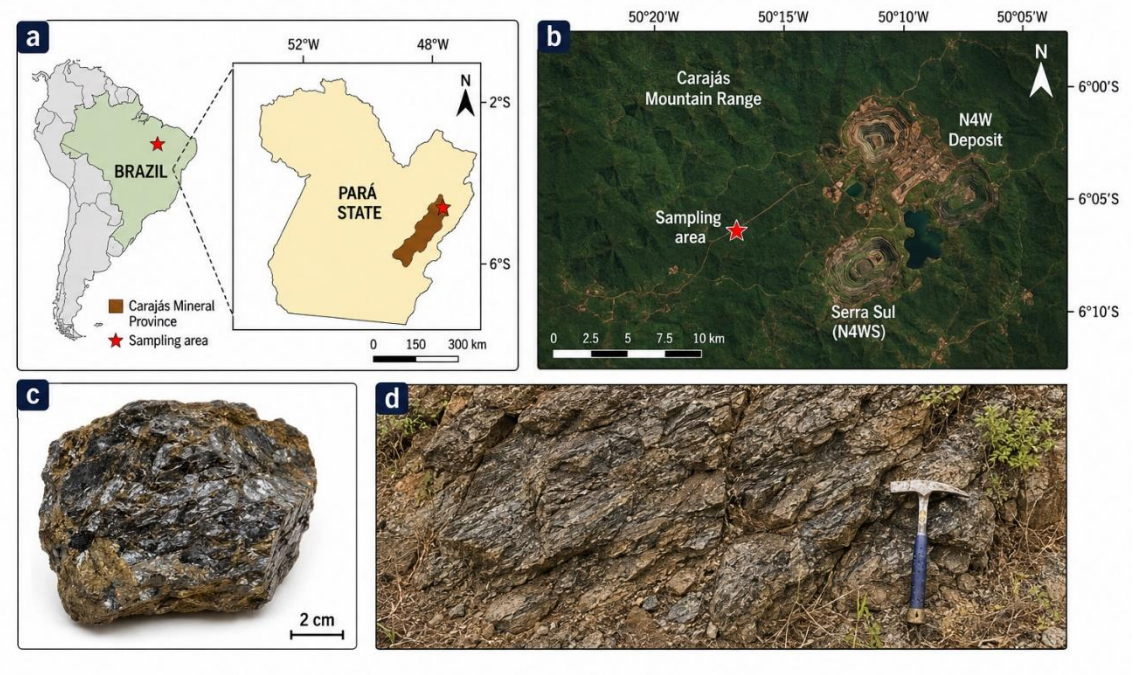

Figure 1. Location and sampling of the biotite-rich rock. (a) Geographic location of the Carajás Mineral Province in Brazil; (b) sampling area in the Carajás region (Pará State); (c) representative hand specimen of the biotite-rich rock; and (d) field outcrop showing the occurrence of the investigated material. Source: Authors' own work..

Figure 1 shows the provenance and characteristics of the studied material. The hand specimen has a dark, platy biotite-rich texture, and the outcrop displays mica-bearing rocks from the Carajás region. Visible mica flakes indicate high potassium mineral content, confirmed by chemical and mineralogical analysis. The geological and mineralogical features support using this material as feedstock for potassium recovery via thermal and hydrometallurgical processing.

The mineralogical composition of the biotite-rich rock was determined to identify the principal potassium-bearing phases and to evaluate the occurrence of gangue minerals that may affect thermal activation and leaching performance. Quantitative phase analysis provides important information on potassium distribution within the mineral assemblage and helps interpret the extraction behavior observed during subsequent processing stages. The results of the quantitative mineralogical analysis are presented in Figure 2.

JIRRS
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Pages: 1-41

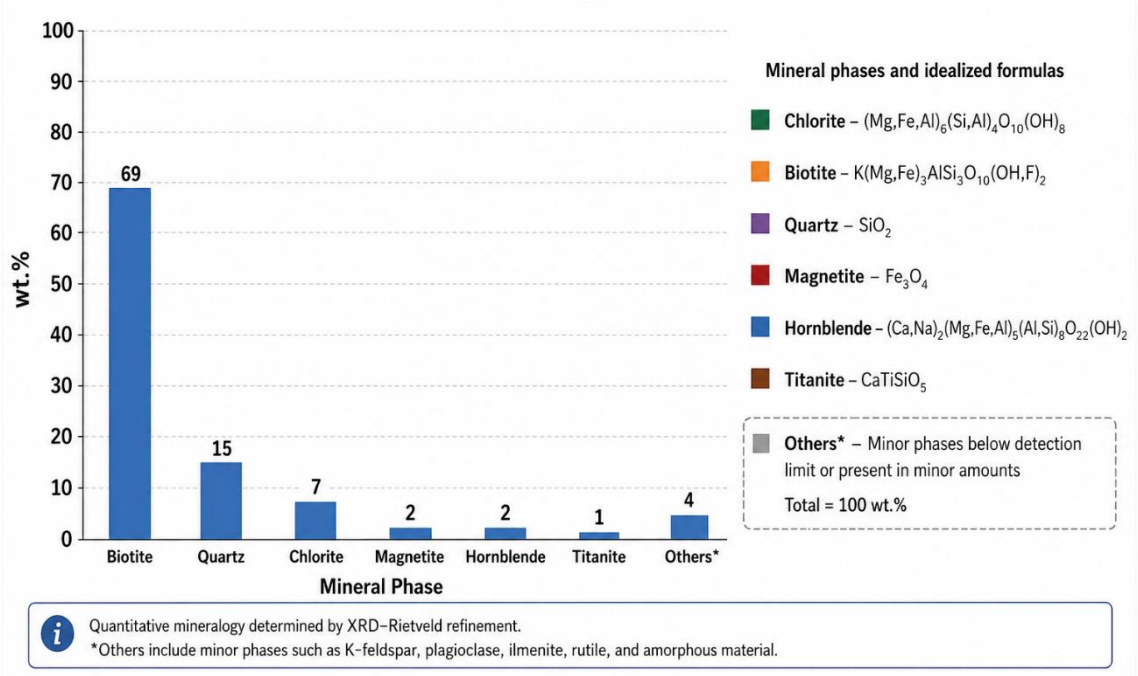

Figure 2. Quantitative mineralogical composition determined by QEMSCAN automated mineralogy.. Biotite is the dominant mineral phase (69 wt.%), followed by quartz (15 wt.%), chlorite (7 wt.%), magnetite (2 wt.%), hornblende (2 wt.%), titanite (1 wt.%), and minor accessory phases (4 wt.%). Adapted from: Present study.

The analysis confirms that biotite is the dominant mineral, making up about 69 wt.% of the rock, consistent with a high potassium content, indicating that most potassium is in biotite. Quartz is the main gangue mineral at 15 wt.%, and chlorite accounts for around 7 wt%.

Minor quantities of magnetite, hornblende, titanite, and accessory minerals were also identified. The presence of magnetite is consistent with the elevated Fe_2O_3 content observed in the chemical analysis, whereas chlorite contributes additional magnesium and aluminum to the overall composition. Collectively, these phases explain the major oxide distribution reported in Table 1.

Biotite's dominance favors potassium recovery since potassium resides in its interlayer positions within the TOT structure. Therefore, thermal activation that destabilizes this framework can significantly boost potassium extraction during sulfuric acid leaching.

JIRRS
Journal International Review of Research Studies
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Pages: 1-41

Optical microscopy was used to investigate the textural characteristics and mineral associations of the biotite-rich rock. The morphology, grain-size distribution, and intergrowth relationships among mineral phases provide important information on the accessibility of potassium-bearing minerals and their response to thermal activation and acid leaching. Particular attention was given to the occurrence of biotite, which hosts most of the potassium in the investigated material. Representative photomicrographs obtained under crossed nicols are presented in Figure 3.

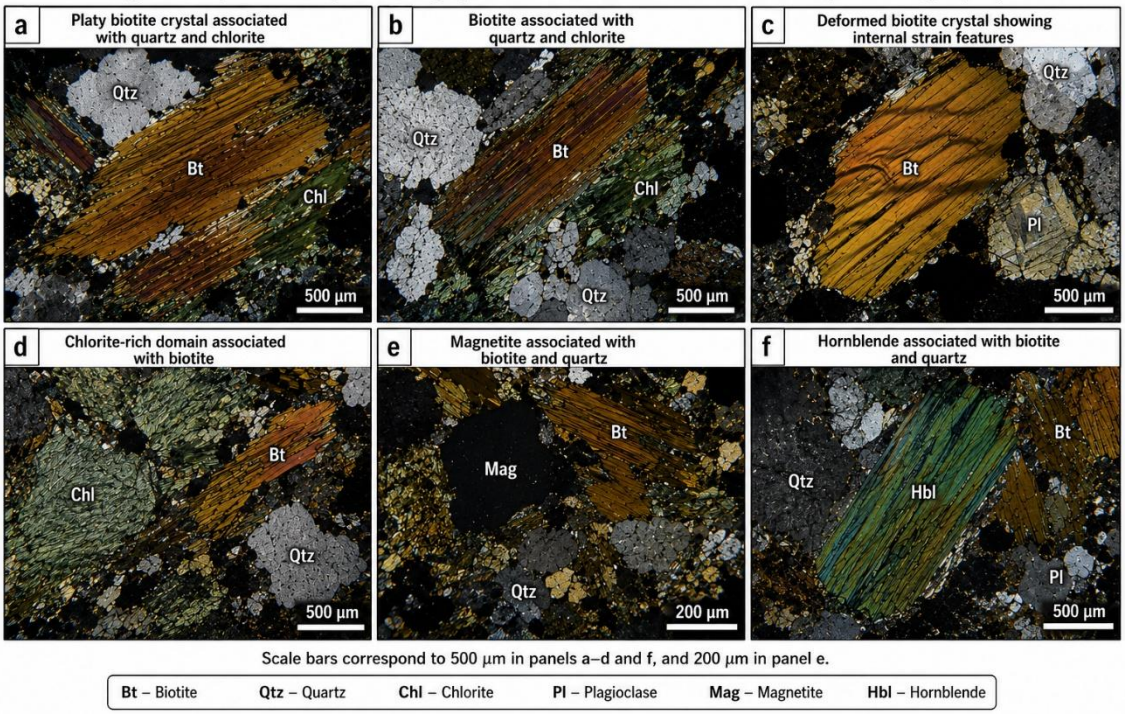

Figure 3. Representative optical microscopy images of the biotite-rich rock under crossed nicols. (a) Biotite grain exhibiting the characteristic platy morphology of mica minerals; (b) intergrowth of biotite with quartz and chlorite; (c) biotite crystal displaying well-developed cleavage and undulose extinction; (d) chlorite associated with biotite; (e) magnetite occurring within the mineral assemblage; and (f) hornblende associated with biotite and quartz. Mineral abbreviations: Bt = biotite; Qtz = quartz; Chl = chlorite; Mag = magnetite; Hbl = hornblende; Pl = plagioclase. Adapted from: Present study

The optical microscopy observations confirm the predominance of biotite within the investigated rock and reveal the characteristic platy morphology and well-developed basal cleavage typical of mica minerals. Most grains exhibit elongated to tabular habits, reflecting the layered tetrahedral–octahedral–tetrahedral (TOT) crystal structure that

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

hosts potassium within interlayer positions. The brown to dark-brown interference colors observed under crossed nicols are consistent with the optical properties of iron-bearing biotite.

Biotite occurs both as discrete crystals and as intergrown aggregates associated with quartz, chlorite, hornblende, magnetite, and minor accessory minerals. The occurrence of these phases indicates a heterogeneous mineral assemblage, which may influence local reaction pathways and dissolution rates during thermal activation and subsequent sulfuric acid leaching. Quartz constitutes the principal gangue mineral, whereas chlorite and hornblende contribute additional magnesium and aluminum to the system.

The mineralogical observations are consistent with the chemical composition and quantitative XRD results, which identified biotite as the dominant mineral phase and the primary host of potassium. Because potassium is structurally incorporated within the interlayer sites of the mica lattice, its release depends on the extent of structural destabilization achieved during thermal treatment. The preservation of crystal morphology in the untreated material suggests that potassium remains strongly bound within the original framework, explaining the limited availability expected under direct leaching conditions.

Consequently, thermal activation plays a critical role in enhancing potassium recovery by promoting structural modification of the biotite lattice and facilitating the transformation of structurally bound potassium into more reactive and soluble forms. The petrographic evidence therefore provides an important mineralogical basis for interpreting the potassium-extraction behavior observed during subsequent hydrometallurgical experiments.

4.3. Thermodynamic Predictions of Roasting Reactions

Thermodynamic simulations were conducted to identify the most likely reaction products formed during thermal activation and to provide a mechanistic framework for interpreting the experimental leaching results. Particular attention was given to the

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

formation of soluble potassium-bearing phases, as these compounds largely determine the efficiency of subsequent potassium extraction.

4.3.1. Chloride-Based Activation System

The chloride-based system consisted of biotite mixed with calcium carbonate and magnesium chloride hexahydrate. Under roasting conditions, several reactions become thermodynamically favorable, promoting destabilization of the biotite structure and redistribution of potassium into chloride-bearing phases.

Thermodynamic simulations were performed to investigate the phase transformations occurring during chloride-assisted thermal activation of the biotite-rich rock and to identify the mechanisms responsible for enhanced potassium recovery. Particular attention was given to the redistribution of potassium from the original mica structure into chloride-bearing compounds, as these phases largely determine subsequent leaching efficiency. The predicted equilibrium phase assemblages as a function of temperature are presented in Figure 4.

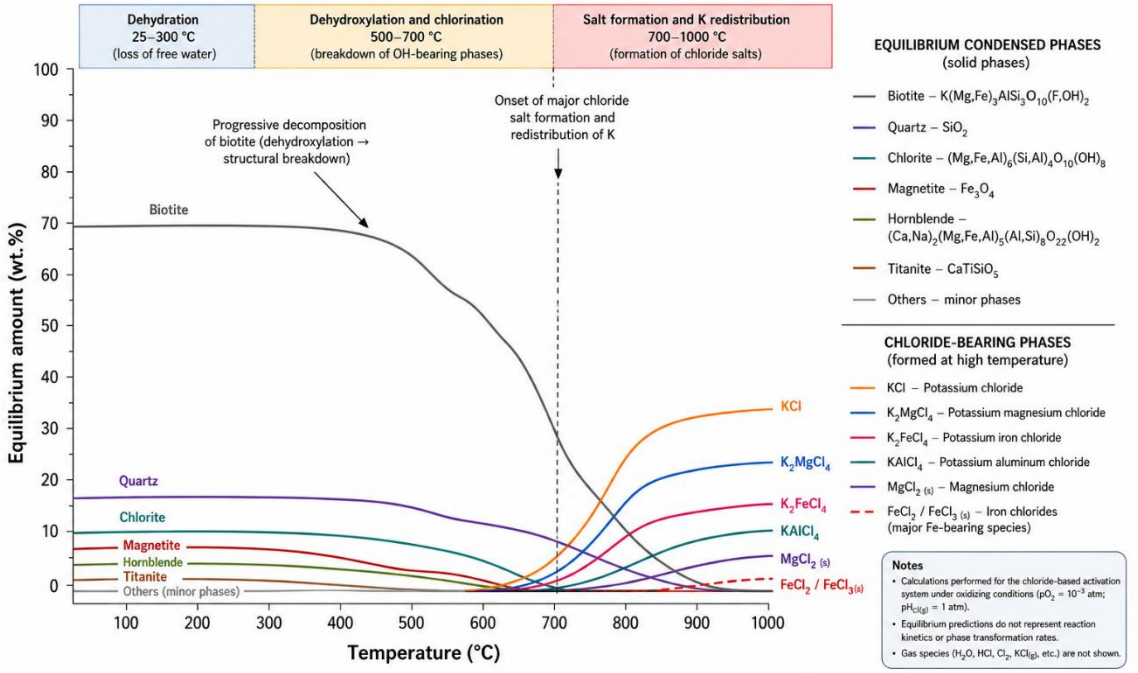

Figure 4. FactSage-predicted equilibrium phase assemblages for the chloride-based activation system (biotite-rich rock + $CaCO_3$ + $MgCl_2 \cdot 6H_2O$) as a function of temperature. The simulations indicate progressive destabilization of the biotite structure and redistribution of

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

potassium into chloride-bearing phases, including KCl and mixed potassium chlorides, at elevated temperatures. Adapted from: Present study based on thermodynamic equilibrium simulations.

Figure 4 presents the thermodynamic equilibrium predictions for the chloride-assisted activation system and demonstrates the progressive redistribution of potassium during roasting. The simulations indicate that increasing temperature promotes destabilization of the original biotite structure and favors the formation of potassium-bearing chloride phases.

Thermodynamic equilibrium calculations predict that KCl is a major potassium-bearing phase, along with mixed potassium–calcium chloride phases such as $KCaCl_3$. The high solubility of these predicted phases relative to structurally bound potassium in biotite may contribute to the experimentally observed higher potassium recoveries during sulfuric acid leaching.

The formation of chloride-bearing potassium phases provides a thermodynamic explanation for the high potassium extractions observed experimentally. During roasting, potassium migrates from interlayer sites within the mica structure and reacts with chloride species generated from the decomposition of $MgCl_2 \cdot 6H_2O$. As a result, structurally bound potassium is converted into more reactive, readily soluble compounds, thereby increasing its availability for acid leaching.

In addition to potassium-bearing chlorides, the simulations predict the formation of calcium silicates and magnesium-rich reaction products. The generation of these phases consumes portions of the aluminosilicate matrix and contributes to the progressive breakdown of the original mineral framework. This structural reorganization facilitates potassium liberation by weakening the crystal lattice and promoting the redistribution of potassium into secondary phases.

Overall, the thermodynamic results support the proposed activation mechanism, in which chloride-assisted roasting enhances potassium recovery through a combination of biotite destabilization, potassium migration, and formation of highly soluble chloride-bearing compounds. These predictions are consistent with the experimental observations

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

and confirm the effectiveness of chloride additives for improving potassium extraction from biotite-rich rocks.

4.3.2. Sulfate-Based Activation System

Thermodynamic equilibrium calculations were also performed for the sulfate-based activation system to evaluate the phase transformations that occur during roasting and to identify the mechanisms responsible for potassium mobilization. Unlike the chloride-assisted route, in which potassium is converted into chloride-bearing compounds, the sulfate-based system promotes the formation of sulfate phases via reactions involving calcium sulfate and alkali species released from the biotite structure. The predicted equilibrium phase assemblages as a function of temperature are presented in Figure 5.

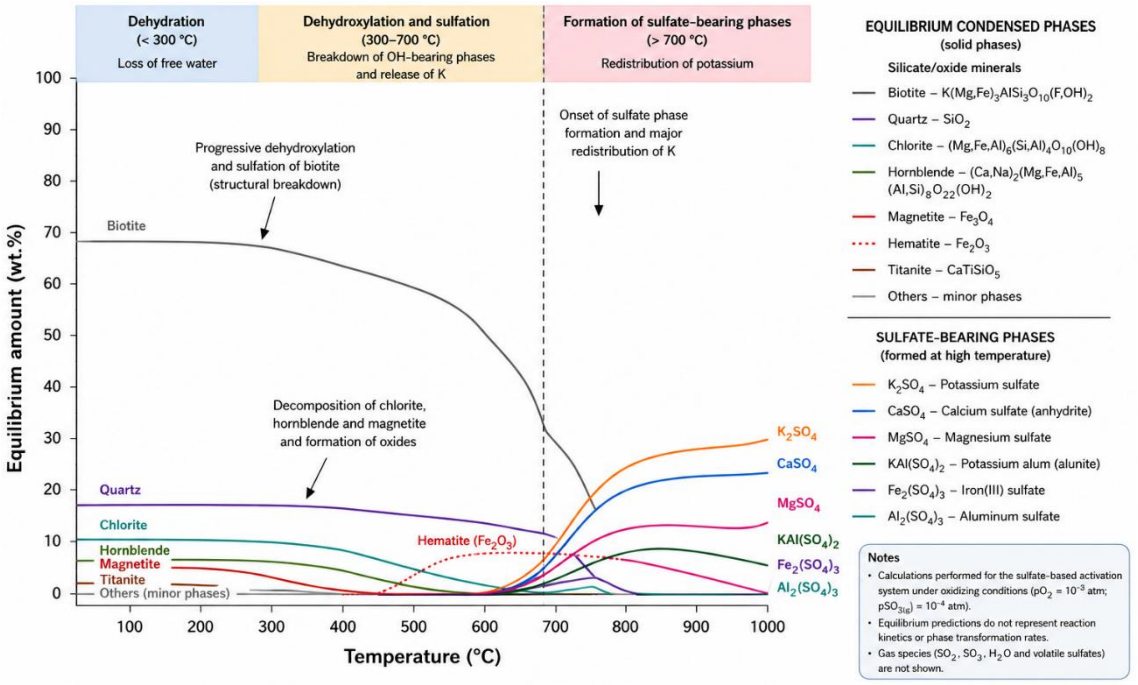

Figure 5. FactSage-predicted equilibrium phase assemblages for the sulfate-based activation system as a function of temperature. The simulations indicate progressive destabilization of the biotite structure and the formation of sulfate-bearing phases, including K_2SO_4, $KAl(SO_4)_2$, $Fe_2(SO_4)_3$, $Al_2(SO_4)_3$, and $MgSO_4$, at elevated temperatures. Adapted from: Present study based on thermodynamic equilibrium simulations.

Figure 5 illustrates the thermodynamically predicted evolution of mineral phases during sulfate-assisted thermal activation. The simulations indicate that biotite remains

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

relatively stable at lower temperatures, whereas progressive decomposition occurs as temperature increases. Above approximately 700 °C, the original mica structure becomes increasingly unstable, promoting the redistribution of potassium and other structural elements into secondary sulfate-bearing phases.

Thermodynamic equilibrium calculations predict K_2SO_4 as a major potassium-bearing phase at elevated temperatures. Additional sulfate-bearing compounds, including $KAl(SO_4)_2$, $MgSO_4$, $Fe_2(SO_4)_3$, and $Al_2(SO_4)_3$, are also predicted to form during roasting. The higher solubility of these predicted phases than that of structurally bound potassium in biotite may contribute to the enhanced potassium recoveries observed during subsequent sulfuric acid leaching.

Simultaneously, the progressive disappearance of biotite and the formation of calcium-bearing silicates indicate extensive restructuring of the original aluminosilicate matrix. The consumption of silicate phases and the generation of sulfate compounds contribute to weakening the crystal framework and facilitating potassium liberation. The formation of K_2SO_4 is particularly important because it is a directly marketable fertilizer, providing an attractive pathway for potassium valorization.

Overall, the thermodynamic results suggest that sulfate-assisted roasting enhances potassium availability by converting structurally bound potassium into soluble sulfate compounds. The predicted phase transformations are consistent with the experimentally observed increase in potassium extraction after thermal activation and support the feasibility of producing potassium sulfate-rich leach solutions from biotite-bearing rocks.

4.3.3. Mechanistic Interpretation and Evidence Limitations

The thermodynamic simulations, mineralogical observations, and leaching results collectively support the interpretation that thermal activation enhances potassium recovery through progressive destabilization of the biotite structure and redistribution of potassium into more soluble reaction products. However, the proposed mechanisms should be interpreted within the limitations of the available evidence.

The formation of potassium-bearing phases such as KCl, $KCaCl_3$, K_2SO_4, and aphthitalite-type compounds is primarily supported by thermodynamic equilibrium

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

predictions and indirect elemental evidence obtained from the roasted products. Although these observations are consistent with the extraction behavior observed experimentally, they do not constitute definitive phase identification. Direct confirmation would require complementary post-roasting characterization techniques such as X-ray diffraction, Raman spectroscopy, Fourier-transform infrared spectroscopy, or quantitative phase refinement.

Consequently, the present results support the interpretation that potassium migration and phase transformation play important roles in the activation process, but the precise reaction pathways and intermediate phases remain subjects for future investigation.

4.4. Effect of Thermal Activation on Potassium Recovery

Thermal activation was performed to promote structural modification of the biotite-rich rock and increase potassium availability during subsequent sulfuric acid leaching. The effectiveness of this pretreatment depends on the formation of soluble potassium-bearing phases, the extent of crystal lattice disruption, and the stability of reaction products generated during roasting.

The experimental results demonstrated that thermal activation significantly enhanced potassium extraction compared with the expected behavior of untreated biotite. Both activation systems improved potassium availability; however, clear differences were observed between the chloride-based and sulfate-based routes.

The chloride-containing system consistently produced higher potassium recoveries than the sulfate-containing system at both investigated temperatures. This behavior is consistent with the thermodynamic predictions presented previously, which indicated the formation of highly soluble potassium chloride phases during roasting.

The effectiveness of thermal activation can be attributed to two simultaneous mechanisms. First, roasting promotes partial destruction of the original mica framework, weakening the interlayer bonding of potassium ions. Second, potassium is redistributed into newly formed reaction products exhibiting substantially greater solubility than the

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

parent mineral. These combined effects significantly improve the accessibility of potassium during acid leaching.

The results demonstrate that thermal activation constitutes a critical step for transforming biotite-rich materials into more reactive feedstocks suitable for hydrometallurgical potassium recovery.

4.5. Potassium Extraction Results

The primary objective of the thermal activation process was to maximize potassium recovery while minimizing impurity dissolution. The potassium extraction results obtained after sulfuric acid leaching are presented in Figure 6.

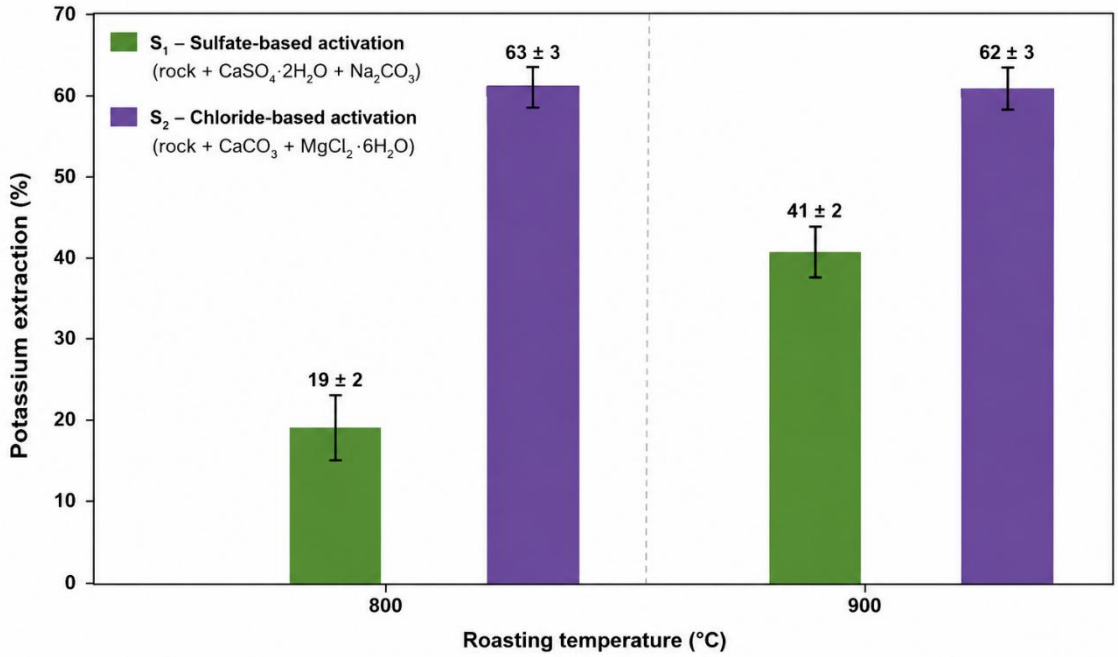

Figure 6 Potassium extraction from thermally activated biotite-rich rock as a function of activation system and roasting temperature.Adapted from: Pereira et al. (2019).

It should be noted that the experimental data presented in Figure 6 were previously reported by Pereira et al. (2019) and are reproduced herein to support the expanded thermodynamic interpretation and mechanistic discussion developed in the present study. Accordingly, the values shown should be regarded as previously published experimental results reanalyzed within a broader scientific framework.

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

Figure 6 clearly demonstrates the influence of additive chemistry on potassium recovery. The chloride-based activation route achieved the highest extraction efficiencies, reaching approximately 63% potassium recovery after roasting at 800 °C. At 900 °C, potassium extraction remained high, although it decreased slightly to approximately 62%.

In contrast, the sulfate-based activation system exhibited lower potassium recoveries across the temperature range investigated. Potassium extraction increased from approximately 19% at 800 °C to approximately 41% at 900 °C, indicating that higher temperatures favor the formation of more reactive potassium-bearing sulfate phases.

The superior performance of the chloride route can be explained by the formation of potassium chloride and mixed potassium-calcium chloride compounds during roasting, as predicted by the thermodynamic equilibrium calculations presented in Section 4.3. These phases are highly soluble and readily dissolve during sulfuric acid leaching. In contrast, the sulfate route appears to generate potassium-bearing sulfate phases that exhibit lower reactivity under the leaching conditions investigated.

The results also reveal a distinct temperature sensitivity for the two activation systems. While potassium recovery in the sulfate route increased by approximately 116% when the roasting temperature was raised from 800 to 900 °C, the chloride route exhibited a slight reduction of approximately 1.6%. This behavior suggests that temperatures above the optimum range may promote phase transformations, partial volatilization of potassium-bearing species, or sintering, which reduces potassium accessibility.

Overall, the results demonstrate that chloride-assisted thermal activation at approximately 800 °C provides the most effective route for potassium recovery from the investigated biotite-rich rock, achieving approximately 63% potassium extraction and confirming the favorable role of chloride-bearing phases in enhancing potassium availability during subsequent hydrometallurgical processing.

4.6. Dissolution of Iron, Aluminum, and Magnesium

The dissolution behavior of structural and impurity elements provides valuable information regarding process selectivity and the potential need for downstream solution purification. Excessive dissolution of iron and aluminum is generally undesirable because

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

these elements increase purification requirements and may negatively affect fertilizer production.

The extraction of iron, aluminum, and magnesium following thermal activation and sulfuric acid leaching is presented in Figure 7.

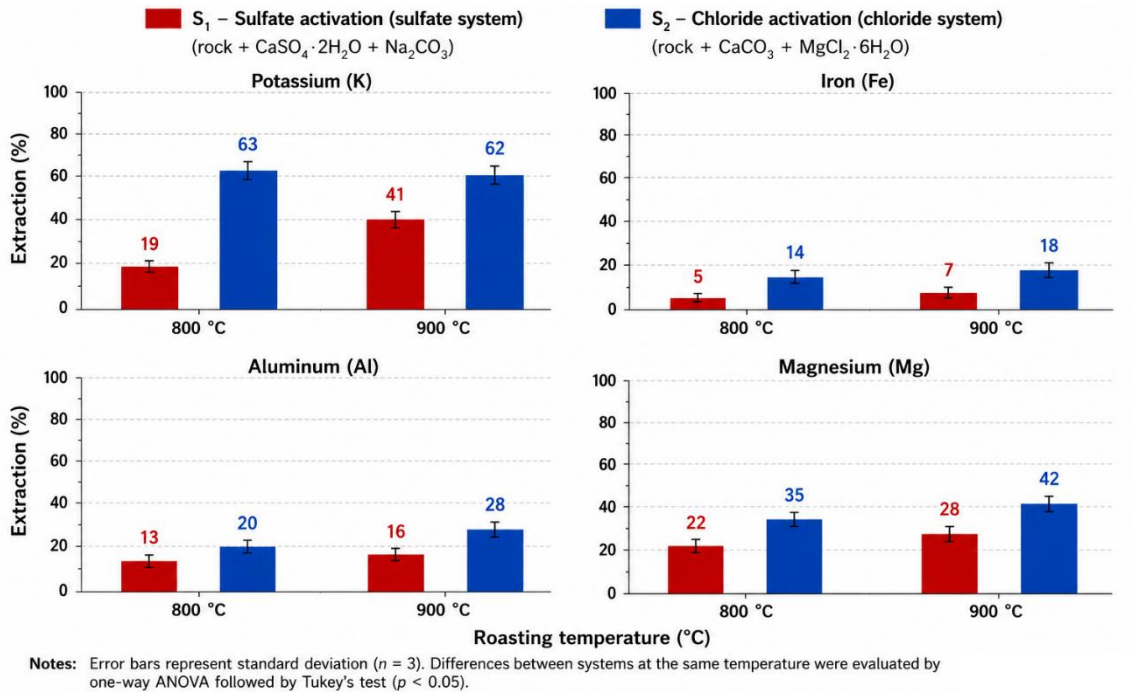

Figure 7. Extraction of potassium, iron, aluminum, and magnesium following thermal activation and sulfuric acid leaching. Adapted from: Pereira et al. (2019).

The results demonstrate that maintaining the leaching pH between 2 and 3 was highly effective in suppressing iron and aluminum dissolution. Iron extraction remained below approximately 5%, while aluminum extraction was generally negligible throughout the experimental program.

The low recovery of these impurities is particularly important because the investigated biotite-rich rock contains significant quantities of iron-bearing minerals and aluminum-bearing silicates. Under more aggressive leaching conditions, substantially higher impurity dissolution would be expected.

Magnesium exhibited different behavior. In the chloride-based activation system, magnesium extraction reached approximately 43%, reflecting its participation in thermal reaction pathways and subsequent dissolution during leaching. Although magnesium

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

contributes to solution chemistry, its presence is generally less problematic than iron or aluminum and may even provide agronomic benefits in some fertilizer applications.

The selective dissolution observed in this study indicates that potassium can be recovered efficiently while maintaining relatively low impurity concentrations in the pregnant leach solution. This characteristic represents a significant advantage for subsequent fertilizer production processes.

4.7. Mechanism of Potassium Redistribution During Thermal Activation

The experimental results, thermodynamic simulations, and SEM–EDS observations collectively provide a consistent explanation for the mechanism responsible for enhanced potassium recovery during chloride-assisted thermal activation. The process involves a sequence of physicochemical transformations that progressively destabilize the biotite structure, promote potassium migration from interlayer positions, and convert structurally bound potassium into highly soluble chloride-bearing compounds.

Biotite is a layered aluminosilicate mineral characterized by a tetrahedral–octahedral–tetrahedral (TOT) structure in which potassium occupies interlayer sites that maintain electrostatic balance between adjacent silicate sheets. Under ambient conditions, these potassium ions are strongly retained within the crystal lattice, resulting in low natural solubility and limited potassium availability. Consequently, direct acid leaching of untreated biotite generally yields relatively low extraction efficiencies.

During thermal activation, $MgCl_2 \cdot 6H_2O$ undergoes progressive dehydration and decomposition, releasing chloride species that interact with the mineral matrix. Simultaneously, elevated temperatures weaken the original biotite structure and promote the breakdown of chemical bonds linking potassium to the aluminosilicate framework. As structural degradation proceeds, potassium becomes increasingly mobile and migrates from interlayer positions toward newly formed reaction zones.

The thermodynamic simulations predict the formation of potassium chloride (KCl) and mixed potassium–calcium chloride phases, including $KCaCl_3$, over the temperature range investigated. These compounds exhibit substantially greater solubility than the original mica and therefore represent thermodynamically favorable sinks for

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

liberated potassium. The conversion of structurally bound potassium into chloride-bearing phases is considered the primary mechanism responsible for the enhanced extraction efficiencies observed experimentally.

Direct evidence supporting this mechanism is provided by the SEM–EDS analyses shown in Figure 8. The composition of the unaltered biotite grain is characterized by significant potassium concentrations, whereas the reacted grain exhibits severe potassium depletion accompanied by calcium enrichment. The potassium concentration decreases from approximately 8.6 wt.% in the original biotite to less than 1 wt.% after roasting, while calcium increases markedly. These compositional changes indicate that potassium was removed from the original crystal structure during thermal treatment and that extensive chemical modification of the mineral matrix occurred.

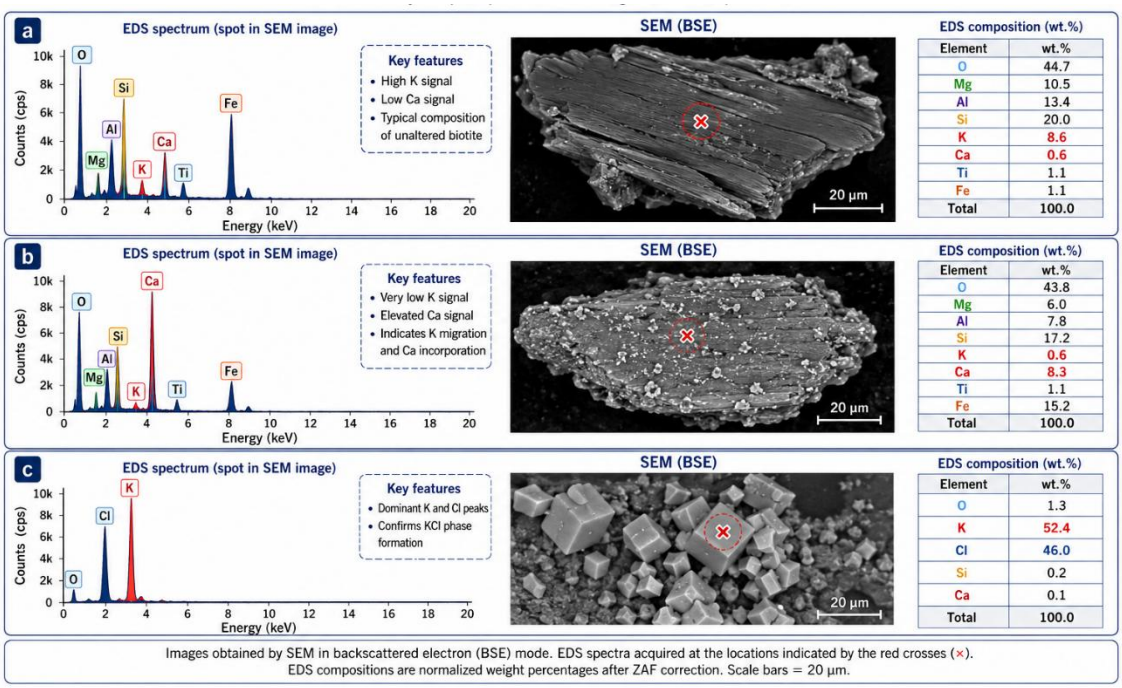

Figure 8. SEM–EDS evidence of potassium redistribution during chloride-assisted thermal activation of biotite-rich rock. (a) Unaltered biotite grain showing the characteristic composition of the original mineral; (b) reacted biotite grain displaying potassium depletion and calcium enrichment after thermal treatment; and (c) KCl-rich crystals precipitated during roasting. EDS compositions are normalized weight percentages after ZAF correction. Scale bars = 20 µm. Adapted from: Present study.

Additional confirmation is provided by the occurrence of cubic KCl-rich crystals identified after roasting. The EDS spectra of these particles are dominated by potassium

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

and chlorine peaks, and the measured compositions closely approach the theoretical stoichiometry of potassium chloride. The presence of these crystals demonstrates that potassium migrated from the biotite lattice and subsequently reacted with chloride species generated during additive decomposition. This observation represents direct microstructural evidence for the potassium redistribution process predicted by the thermodynamic calculations.

The formation of calcium-bearing silicates and magnesium-rich reaction products further contributes to the liberation of potassium. These phases consume portions of the original aluminosilicate matrix and promote structural reorganization of the roasted material. As a result, the crystal framework becomes increasingly destabilized, facilitating potassium release and enhancing the accessibility of residual potassium during subsequent sulfuric acid leaching.

The proposed mechanism is consistent with the previously reported extraction results. The chloride-assisted activation route achieved potassium recoveries exceeding 60%, substantially higher than those obtained using sulfate-based activation. The superior performance of the chloride system can therefore be attributed to its ability to transform structurally bound potassium into highly soluble chloride-bearing compounds while simultaneously destabilizing the original biotite structure.

Overall, the results demonstrate that potassium recovery from biotite-rich rocks is governed by a coupled mechanism involving thermal decomposition of the mica lattice, migration of potassium from interlayer sites, formation of secondary chloride-bearing phases, and enhanced dissolution during hydrometallurgical treatment. The integration of thermodynamic modeling, leaching experiments, and SEM–EDS characterization provides a coherent description of the processes that control potassium liberation and supports the development of chloride-assisted thermal activation as an effective route for producing potassium fertilizers from unconventional mineral resources.

4.8. Process Selectivity and Impurity Behavior

The simultaneous achievement of high potassium recovery and low impurity dissolution is one of the most significant findings of this investigation. Many

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

hydrometallurgical processes capable of extracting potassium from silicate minerals also promote extensive dissolution of aluminum, iron, and silica, increasing downstream processing complexity.

In the present study, thermal activation modified potassium speciation without causing excessive mobilization of undesirable elements during leaching. This behavior suggests that the process selectively targets potassium-bearing reaction products while leaving a substantial fraction of the gangue minerals relatively unaffected.

The chloride-based activation route showed high selectivity. Although it achieved the highest potassium recovery, iron and aluminum extraction were limited. This suggests potassium is liberated mainly through transforming interlayer potassium into soluble chloride phases, not full mica dissolution.

From a process-engineering perspective, such selectivity offers several advantages:

- Reduced reagent consumption during purification.
- Lower risk of secondary precipitate formation.
- Simplified downstream fertilizer production.
- Improved environmental performance.
- Potential reduction in overall operating costs.

The resulting pregnant leach solutions contained approximately 2.7 g L^{-1} of potassium under the conditions investigated. Although this concentration may be insufficient for direct fertilizer crystallization, counter-current leaching, solution recycling, or concentration steps could substantially increase potassium content and improve process economics.

The combination of thermal activation and sulfuric acid leaching offers a promising way to convert biotite-rich minerals into potassium solutions for fertilizer production.

4.9. Comparative Assessment of Chloride- and Sulfate-Assisted Activation Routes

The experimental results demonstrate that both activation systems improved potassium availability relative to the untreated material, although significant differences

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

were observed in extraction performance and potential process implications. The chloride-assisted route achieved the highest potassium recovery, reaching approximately 63%, whereas the sulfate-assisted route attained a maximum recovery of approximately 41% under the investigated conditions. However, process selection should not be based solely on potassium recovery, as each activation strategy offers distinct advantages and limitations that may affect industrial implementation.

To facilitate comparison, Table 2 summarizes the principal technical characteristics, potential benefits, and operational challenges associated with the chloride- and sulfate-assisted activation routes.

Table 2. Comparative assessment of chloride- and sulfate-assisted activation routes.

Criterion	Chloride Route	Sulfate Route
Potassium recovery	High	Moderate
Formation of soluble K phases	Excellent	Good
Corrosion potential	High	Low
Chloride management	Required	Minimal
Off-gas treatment requirements	Higher	Lower
Fertilizer compatibility	Requires chloride control	Directly compatible with K_2SO_4 production
Process complexity	Moderate	Lower
Environmental burden	Potentially higher	Potentially lower

The superior performance of the chloride-assisted route is consistent with the thermodynamic interpretation that roasting promotes the formation of highly soluble potassium-bearing chloride phases. These reaction products are more readily dissolved during sulfuric acid leaching, resulting in higher potassium recoveries. Nevertheless, industrial implementation of chloride-based systems would require careful consideration of corrosion, refractory compatibility, management of chloride-containing process streams, and appropriate off-gas treatment systems.

In contrast, the sulfate-assisted route produced lower potassium recoveries but may offer advantages related to downstream fertilizer production. Because sulfate species are already incorporated into the process chemistry, this route may be more readily integrated into potassium sulfate production flowsheets and may reduce challenges associated with chloride management. Furthermore, the lower corrosion potential and

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

potentially reduced environmental burden may partially offset the lower extraction efficiency observed experimentally.

Therefore, the results suggest that the chloride-assisted route is technically advantageous when maximum potassium recovery is the primary objective, whereas the sulfate-assisted route may offer benefits related to process integration, fertilizer compatibility, and operational simplicity. Consequently, the selection of the most appropriate activation strategy should consider not only extraction efficiency but also economic, environmental, and product-quality requirements.

4.10. Fertilizer Production Potential and Remaining Development Requirements

Based on the measured potassium content of approximately 9.6 wt.% K_2O and the maximum recovery achieved in the chloride-assisted activation route, the process theoretically has the potential to generate approximately 112 kg of K_2SO_4 per tonne of biotite-rich rock processed.

However, it is important to emphasize that this value represents a theoretical production potential rather than demonstrated fertilizer manufacture. The present study evaluated potassium recovery into solution but did not experimentally investigate downstream operations required for commercial fertilizer production.

Additional processing stages would be necessary, including:

- solution purification;
- chloride management;
- sulfate adjustment;
- concentration and evaporation;
- potassium sulfate crystallization;
- product drying;
- product quality characterization;
- agronomic performance evaluation;
- compliance with fertilizer regulations.

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

Therefore, the results should be interpreted as evidence of the potential suitability of the process for potassium sulfate production rather than direct demonstration of fertilizer manufacture.

4.11. Comparison with Other Alternative Potassium Sources

The development of alternative potassium fertilizers requires not only efficient extraction technologies but also a clear understanding of how different potassium-bearing minerals compare in terms of potassium content, mineralogical characteristics, processability, and recovery potential. In this context, the performance of the biotite-rich rock investigated in the present study can be evaluated against other unconventional potassium resources that have been proposed for fertilizer production.

Potassium feldspars represent one of the largest potassium reservoirs in the Earth's crust and typically contain between 10 and 16 wt.% K_2O. However, potassium is incorporated within a highly stable three-dimensional aluminosilicate framework, which generally requires aggressive thermal, chemical, or mechanochemical activation to achieve significant extraction. Consequently, despite their relatively high potassium contents, feldspars often exhibit lower reactivity than mica minerals during hydrometallurgical processing.

Glauconite and verdete have attracted considerable interest, particularly in Brazil, because of their large reserves and potential application as slow-release fertilizers. These materials typically contain between 5 and 10 wt.% K_2O and may respond favorably to thermal activation. Nevertheless, potassium release is frequently limited by mineralogical constraints and strongly depends on roasting conditions and additive selection.

Nepheline syenites and feldspathoid-rich rocks have also been investigated as alternative potassium resources. Although some deposits exhibit favorable reactivity, their potassium contents are often lower than those observed in biotite-rich materials, and their geographic distribution is more restricted.

Among the alternative silicate resources reported in the literature, biotite occupies a particularly favorable position because it combines relatively high potassium content with a crystal structure that is more susceptible to thermal destabilization than feldspar-based systems. The approximately 63% potassium recovery achieved through chloride-

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

assisted activation in the present study falls within the upper range reported for many potassium-bearing silicate minerals and demonstrates the potential competitiveness of biotite as a non-evaporitic potassium resource.

Overall, the comparison indicates that biotite-rich rocks represent a technically attractive feedstock for potassium recovery, particularly in regions where conventional potash resources are unavailable and where mining operations generate potassium-bearing silicate residues that could be valorized through integrated mineral processing and fertilizer production strategies.

4.12. Environmental and Industrial Implications

The recovery of potassium from biotite-rich rocks presents several potential environmental and industrial advantages. One of the most significant benefits is the possibility of transforming low-value mineral resources, mine wastes, and processing residues into value-added fertilizer feedstocks. Such an approach is consistent with circular-economy principles and may contribute to improved resource efficiency within the mining sector.

The selective behavior observed during sulfuric acid leaching is also environmentally favorable. Potassium extraction was achieved while maintaining relatively low dissolution of iron and aluminum, thereby reducing impurity transfer to process solutions and potentially simplifying downstream purification requirements. Lower impurity dissolution may reduce reagent consumption, decrease waste generation, and minimize the environmental burden associated with effluent treatment.

From an agricultural perspective, generating potassium-rich sulfate solutions is particularly attractive because potassium sulfate is widely regarded as a premium fertilizer for chloride-sensitive crops. In addition to supplying potassium, potassium sulfate provides sulfur, an essential plant nutrient, which can reduce the need for supplementary sulfur fertilizers.

Industrial implementation of the process, however, would require careful consideration of several technical challenges. In the chloride-assisted route, the management of chloride-bearing species may necessitate corrosion-resistant materials, appropriate refractory selection, and control of potential chloride-containing emissions.

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

These factors could increase capital and operating costs relative to sulfate-based alternatives.

Furthermore, thermal activation constitutes the most energy-intensive stage of the proposed process and is likely to represent a major contributor to overall environmental impacts and operating expenditures. Consequently, future evaluations should include detailed assessments of energy consumption, greenhouse-gas emissions, and process integration opportunities to determine the overall sustainability of the technology.

Despite these challenges, the results suggest that thermal activation followed by selective sulfuric acid leaching may provide a promising route for converting potassium-bearing silicate resources into useful agricultural products while simultaneously supporting resource diversification and waste valorization.

4.13. Future Perspectives

The results obtained in this study highlight several opportunities for future research and technological development. Although thermal activation significantly improved potassium recovery from the investigated biotite-rich rock, additional work is required to validate the proposed mechanisms, optimize process conditions, and assess industrial feasibility.

A priority research area involves the experimental validation of the thermodynamically predicted reaction products. Future studies should combine post-roasting X-ray diffraction, Raman spectroscopy, FTIR, and quantitative phase analysis to confirm the formation of potassium-bearing phases predicted by the equilibrium simulations and to improve understanding of potassium redistribution during thermal activation.

Further optimization of roasting and leaching conditions is also required. Variables such as additive dosage, roasting temperature, residence time, particle size, sulfuric acid concentration, and solid-to-liquid ratio should be systematically investigated to maximize potassium recovery while minimizing energy consumption and reagent requirements.

Another important research direction concerns the development of complete fertilizer-production flowsheets. The present work demonstrates the generation of

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

potassium-rich process liquors but does not address downstream operations such as solution purification, concentration, potassium sulfate crystallization, product characterization, or agronomic performance testing. These steps will be essential for assessing the commercial viability of the proposed route.

Pilot-scale demonstration represents an additional challenge. Most studies involving potassium recovery from mica minerals remain restricted to laboratory-scale investigations. Pilot-scale testing would provide valuable information regarding reactor design, additive recycling, process control, residue management, and overall process integration.

Finally, comprehensive techno-economic and life-cycle assessments will be necessary to determine whether biotite processing can compete with conventional potash production and other alternative potassium sources. Such evaluations should consider not only potassium recovery but also fertilizer quality, energy requirements, environmental performance, resource availability, and regional agricultural demand.

Collectively, these research directions may contribute to the development of more sustainable and diversified potassium supply chains and enhance the utilization of potassium-bearing silicate resources in future fertilizer-production systems.

5. Limitations of the Study

The present study has several limitations that should be considered when interpreting the results and assessing the broader applicability of the proposed process.

First, part of the experimental dataset discussed herein was previously reported by Pereira et al. (2019), whereas the present work focuses primarily on expanded thermodynamic interpretation, mechanistic analysis, and process implications. Consequently, the main novelty of this manuscript resides in the integration of thermodynamic modeling, updated literature analysis, and broader technological assessment rather than in the generation of a completely new experimental dataset.

Second, thermal activation experiments were restricted to two roasting temperatures (800 and 900 °C) and a single residence time of 6 h. Additional investigations covering wider temperature ranges, additive ratios, and reaction times would provide a more comprehensive understanding of process optimization.

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

Third, sulfuric acid leaching was conducted under one principal set of operating conditions (pH 2–3, 85 °C, 60 min, and 20 wt.% solids), limiting kinetic interpretation and preventing rigorous evaluation of the effects of temperature, acidity, solid-to-liquid ratio, and particle size on potassium extraction.

Fourth, the equilibrium phases predicted by FactSage were not fully validated by post-roasting X-ray diffraction (XRD), Raman spectroscopy, FTIR, or quantitative phase refinement. Consequently, potassium-bearing phases such as KCl, $KCaCl_3$, K_2SO_4, and aphthitalite-type compounds should be regarded as thermodynamically predicted phases supported by indirect evidence rather than definitively confirmed reaction products.

Fifth, downstream fertilizer-production steps, including solution purification, potassium sulfate crystallization, product-quality assessment, agronomic performance evaluation, and regulatory compliance testing, were not experimentally investigated. Therefore, the present work demonstrates the potential for generating potassium-rich process liquors rather than direct production of fertilizer-grade potassium sulfate.

Finally, the conclusions are based on a single biotite-rich rock containing approximately 69 wt.% biotite and 9.6 wt.% K_2O. Variations in mineralogy, alteration degree, Fe/Mg ratio, gangue composition, and texture may significantly influence process performance. Accordingly, direct extrapolation of the present results to other mica-bearing rocks, mining residues, or potassium-bearing silicate resources should be undertaken with caution.

6. Conclusions

This study evaluated the recovery of potassium from a biotite-rich rock via thermal activation followed by sulfuric acid leaching and provided an expanded thermodynamic and mechanistic interpretation of the experimentally observed potassium extraction behavior.

The results indicate that additive-assisted thermal activation substantially enhances potassium availability relative to the untreated material. Among the investigated activation systems, the chloride-assisted route employing $CaCO_3$ and $MgCl_2·6H_2O$ achieved the highest potassium recovery, reaching approximately 63% after roasting at 800 °C for 6 h, whereas the sulfate-assisted route based on $CaSO_4·2H_2O$ and Na_2CO_3

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

achieved a maximum recovery of approximately 41% after roasting at 900 °C. These results suggest that chloride-assisted activation is more effective under the investigated conditions.

Thermodynamic equilibrium calculations support the interpretation that the improved performance of the chloride route is associated with the formation of soluble potassium-bearing chloride phases, whereas the sulfate route favors the formation of potassium sulfate-bearing compounds such as aphthitalite-type phases. However, these phase assignments should be regarded as thermodynamic predictions supported by indirect evidence rather than definitively confirmed reaction products.

Sulfuric acid leaching exhibited favorable selectivity, extracting up to 63% of the potassium while maintaining iron and aluminum dissolution below approximately 5%. This behavior suggests that potassium recovery can be achieved without extensive dissolution of the entire aluminosilicate matrix, potentially reducing downstream purification requirements.

The investigated biotite-rich rock, containing approximately 9.6 wt.% K_2O and 69 wt.% biotite, represents a potentially attractive non-evaporitic potassium resource. The results support the interpretation that thermal activation can convert part of the structurally bound potassium into more soluble forms suitable for subsequent hydrometallurgical recovery.

Based on the maximum potassium recovery achieved in the chloride-assisted route, the process has the theoretical potential to generate approximately 112 kg of K_2SO_4 per tonne of biotite-rich rock processed. Nevertheless, fertilizer production was not experimentally demonstrated in the present study, and additional stages such as solution purification, concentration, crystallization, product characterization, and agronomic evaluation would be required before fertilizer-grade potassium sulfate could be produced.

Overall, the results highlight the potential of thermal activation and selective sulfuric acid leaching as a route for the utilization of potassium-bearing silicate resources and mining residues. Future work should focus on experimental validation of thermodynamically predicted phases, optimization of roasting and leaching conditions, potassium sulfate crystallization, pilot-scale testing, and comprehensive techno-economic

JIRRS
Journal International Review of Research Studies
Received: 01/05/2026 - **Accepted:** 12/06/2026
Vol: 01.08
DOI: 10.66104/dkjgbb35
Pages: 1-41

and environmental assessments to determine the practical feasibility of the proposed process.

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