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Investigation of the sulfation-roasting-water leaching process for efficient recovery of nickel and cobalt from Kazakhstan's laterite ore

Kulzira Mamyrbayeva^a, Almira Kuandykova^a, Tatyana Chepushtanova^a, Yerik Merkibayev^a and Brajendra Mishra^b

^aDepartment of Metallurgy and Mineral Processing, Satbayev University, Almaty, 050013, Kazakhstan; ^bMechanical & Materials Engineering Department, Worcester Polytechnic Institute, Worcester, MA, 01609-2280, USA

ABSTRACT

The rising demand for nickel and cobalt in battery materials and high-performance alloys has renewed interest in lateritic ores, yet Kazakhstan's substantial resources remain underused due to the lack of effective extraction technologies. This study provides the first comprehensive mineralogical and processing evaluation of laterite ore from the Kempirsay deposit and demonstrates the applicability of the sulfation-roasting-water leaching (SR-WL) method to Kazakh high-iron laterites. Physicochemical analysis showed that Ni and Co are mainly hosted in Fe-bearing phases goethite, hematite, and tremolite with minor incorporation in silicates; the ore contains, (in wt.%): 1.08 Ni, 0.104 Co, and 20.2 Fe. Roasting at 500 °C with 30 wt.% H₂SO₄ generated readily leachable sulfates, and subsequent leaching at 85 °C (180 min, S: L = 1:6) extracted 93.3% Ni, 92.0% Co, and 84% Fe. Selective dissolution was achieved by roasting at 700 °C with 50 wt.% H₂SO₄, yielding 90.08% Ni and 90.01% Co while limiting Fe dissolution to 6.12%, producing a low-iron pregnant leach solution (PLS) containing, g·L⁻¹: 2.11 Ni, 0.35 Co, 2.12 Fe. Overall, the results show that the SR-WL process is a viable and newly validated pathway for converting low-grade Kazakh laterites into Ni-Co intermediates suitable for modern metallurgical supply chains.

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Laterite ore; Sulfation roasting; Water leaching; Sulfuric acid; Nickel extraction; Cobalt extraction

Étude du procédé de grillage sulfatant-lixiviation aqueuse pour une récupération efficace du nickel et du cobalt du minerai de latérite du Kazakhstan

La demande croissante en nickel et en cobalt dans les matériaux de batteries et les alliages à haute performance a renouvelé l'intérêt pour les minerais de latérite. Malgré tout, les ressources substantielles du Kazakhstan demeurent sous-utilisées en raison du manque de technologies d'extraction efficaces. Cette étude présente la première évaluation minéralogique et de traitement complète du minerai de latérite du gisement de Kempirsay et démontre l'applicabilité de la méthode de grillage sulfatant-lixiviation aqueuse (SR-WL) aux latérites kazakhes riches en fer. L'analyse physico-chimique a montré que le Ni et le Co sont principalement présents dans les phases porteuses de fer, goéthite, hématite et trévorite, avec une incorporation mineure dans les silicates; le minerai contient (% en poids): 1.08 Ni, 0.104 Co et 20.2 Fe. Le grillage à 500°C avec 30% en poids de H₂SO₄ a généré des sulfates facilement lixiviables, et la lixiviation ultérieure à 85°C (180 min, S:L = 1:6) a permis d'extraire 93.3% de Ni, 92.0% de Co et 84% de Fe. On a obtenu une dissolution sélective par grillage à 700°C avec 50% en poids de H₂SO₄, produisant 90.08% de Ni et 90.01% de Co tout en limitant la dissolution du Fe à 6.12%, produisant une solution de lixiviation enrichie (PSL) à faible teneur en fer contenant, en g·L⁻¹: 2.11 Ni, 0.35 Co et 2.12 Fe. Dans l'ensemble, les résultats montrent que le procédé de SR-WL est une méthode viable et nouvellement validée pour convertir les latérites kazakhes à faible teneur en intermédiaires Ni-Co adaptés aux chaînes d'approvisionnement métallurgiques modernes.

Introduction

The steadily increasing demand for nickel and cobalt is closely linked to their critical roles in stainless-steel production, advanced superalloys, and modern battery

technologies. The rapid expansion of low-carbon energy systems including electric vehicles and grid-scale energy storage has further intensified the need for efficient and environmentally responsible extraction routes for these

CONTACT Almira Kuandykova  a.kuandykova@satbayev.university

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metals [1–5]. As a result, improving the processing of lateritic nickel ores has become a key focus of contemporary metallurgical research.

Nickel laterites typically form layered weathering profiles in which the upper limonite horizon is dominated by Fe(III) oxyhydroxides, with nickel commonly incorporated into the structure of goethite [6–9]. Beneath the limonite zone lies the saprolite horizon, enriched in magnesium silicates and containing up to 1.5–2.5 wt.% Ni [10]. These mineralogical contrasts largely determine the processing strategy: high-grade saprolitic ores are generally treated through pyrometallurgical routes, whereas low-grade limonites are more suited to hydrometallurgical techniques [10–16]. However, the fine particle size, elevated iron content, and complex mineral assemblages typical of low-grade laterites often limit the efficiency of existing extraction technologies [17–20].

Thermal pretreatment has been widely explored as a means of enhancing nickel and cobalt recoveries. Numerous studies show that roasting with chemical modifiers such as Na_2CO_3 , NH_4HSO_4 , NaCl , Na_2SO_4 , elemental sulfur, or H_2SO_4 facilitates the formation of soluble sulfates and bisulfates that significantly improve subsequent leaching performance [21–24]. Within this context, the SR-WL process has emerged as a versatile and economically attractive hydrometallurgical option for treating lateritic ores with diverse mineralogical characteristics.

Although SR-WL has been extensively investigated for limonitic laterites, saprolites, and mixed oxide-sulfide concentrates [25], published results reveal considerable variation in the recommended roasting parameters. Studies [26–30], among others, report markedly different optimal temperatures, reagent selections, and additive dosages. These discrepancies stem from pronounced differences in ore mineralogy, serpentinization degree, chromite content, and Ni-Co distribution. The absence of convergence across these findings indicates that universal SR-WL roasting conditions have not been established; instead, each ore type requires its own tailored processing strategy. This issue is particularly relevant for the complex, chromite-rich and nickel-poor laterites of Kazakhstan, which have not yet been examined within the framework of SR-WL.

Kazakhstan hosts substantial lateritic nickel resources, including the large Kempirsay ore field, which is characterised by low nickel grades and elevated chromite content [31]. Despite their scale, these deposits have never been industrially exploited, in part due to the lack of processing technologies specifically adapted to their mineralogical features. Moreover, no prior studies

have assessed the behaviour of Kazakhstan's laterites under sulfation roasting conditions, leaving a significant knowledge gap and limiting the prospects for their development.

The aim of this study is to provide the first comprehensive evaluation of the SR-WL process applied to lateritic ores from the Kempirsay deposit. Specifically, this work investigates (1) the mineralogical characteristics governing the distribution of nickel and cobalt, (2) the phase transformations occurring under different sulfation roasting conditions, and (3) the optimisation of SR-WL parameters to maximise metal extraction. The findings establish a scientific basis for developing selective, energy-efficient, and environmentally sustainable processing routes for Kazakhstan's lateritic nickel ores.

Materials and methods

Materials

Laterite ore samples were collected from the Kempirsay deposit (Western Kazakhstan). The material was crushed in two stages: initially with a jaw crusher (Fritsch, Germany) to 1–2 cm, followed by a cone rotary crusher to achieve $80\% < 1\text{ mm}$ (P80). The crushed material was homogenised by the quartering method and used for subsequent analyses and roasting experiments.

Analytical methods

Mineralogical and chemical characterisation of the raw ore, roasted products, and leaching residues was carried out using XRD, XRF, SEM-EDS, thermal analysis (DTG/DSC), and AAS.

X-ray diffraction

XRD patterns were collected on a MiniFlex 300/600 diffractometer with Cu K α radiation (40 kV, 15 mA) in θ – 2θ geometry. Data were acquired in continuous-scan mode over 3 – 90° 2θ with a 0.02° step and a scan rate of 10°min^{-1} . The setup included an ASC-6 attachment, a K β ($\times 2$) filter, a 1.25° incident slit, a 5-mm length-limiting slit, and 6-mm receiving slits; no diffracted-beam monochromator was used. A D/teX Ultra detector recorded the signal, and phase identification was performed using EVA software and the PDF-2 database.

Scanning electron microscopy

(SEM/EDS) Microstructure and elemental distribution were examined using a JEOL JSM-6490LA SEM (JEOL

Ltd., Japan) equipped with an integrated EDS detector. The instrument operated at a resolution of 3.0 nm.

Thermal analysis (DTG/DSC)

Thermal behaviour was investigated using a NETZSCH STA 409 PC/PG simultaneous thermal analyzer (NETZSCH GmbH, Germany) under an inert atmosphere. Measurements were conducted from room temperature to 800 °C.

Atomic absorption spectroscopy (AAS) Metal concentrations in PLS were quantified using a Shimadzu AA-7000 spectrometer (Shimadzu Corp., Japan) in flame AAS mode. Calibration curves were generated using certified standard solutions.

X-ray fluorescence (XRF)

Bulk elemental composition was determined using a Niton XL3t handheld XRF analyzer (Thermo Fisher Scientific, USA).

Combined laterite processing method

Several established hydrometallurgical and pyrometallurgical routes exist for nickel extraction from lateritic ores [1,21]. In this study, a combined SR-WL method was selected to enhance Ni and Co recovery from the Kempirsay laterite. Sulfuric acid was used as the sulfating and modifying agent due to its wide availability, proven effectiveness, and ability to convert Ni- and Co-bearing phases into water-soluble metal sulfates, thereby improving subsequent leaching performance [25,26].

Roasting procedure

Nickel laterite ore obtained from Kazakhstan was dried at 120 °C for 20 h. Thermal treatment (sulfation roasting) was performed in a SNOL-7.2/1100 muffle furnace (SNOL, Lithuania). A 100 g portion of laterite ore was mixed with concentrated sulfuric acid at an addition level of 10–50 wt. %. Roasting was conducted in the temperature range of 50–550 °C for 30–150 min. After treatment, roasted cinders were cooled to room temperature in a desiccator to prevent moisture absorption.

Leaching of roasted cinders

Leaching experiments were carried out in a thermostatically controlled glass reactor equipped with a mechanical overhead stirrer operating at 300 rpm. A suspension of roasted cinders (100 g) and distilled water was prepared in the reactor, and the temperature was

automatically maintained throughout the experiment. Experimental duration was recorded from the moment the set temperature was reached. After leaching, the slurry was vacuum-filtered, separating the PLS from the solid residue. Concentrations of Ni, Co, and Fe in the PLS were quantified by AAS (Shimadzu AA-7000). Leaching temperature ranged from 25 to 105 °C, and leaching duration from 30 to 210 min.

Results and discussion

Numerous studies have investigated the SR-WL process for laterites, saprolite ores, and mixed oxide-sulfide nickel concentrates (Table 1).

Roasting of limonitic laterites with H_2SO_4 at 700–725 °C consistently yields Ni and Co recoveries above 88–91% with minimal Fe dissolution (<3–4%), due to efficient $\text{Fe(II)} \rightarrow \text{Fe(III)}$ conversion [32, 33]. Systems employing $(\text{NH}_4)_2\text{SO}_4$ at 450–650 °C achieve similarly high Ni extraction (70–97%) through the formation of ammonium-metal sulfate complexes that enhance sulfation kinetics [34–36].

In addition, roasting of Jinchuan nickel concentrate with NH_4HSO_4 at 400 °C results in exceptionally high recoveries (95.7% Ni and 98.9% Co), while Fe is effectively removed as stable ammonium-iron sulfate precipitates, making study [37] one of the most efficient low-temperature SR-WL systems reported. The use of alkali sulfates at 700 °C promotes molten pyrosulfate formation and increases Ni extraction to about 90% [38], whereas $\text{Fe}_2(\text{SO}_4)_3$ roasting at 500 °C selectively upgrades cobalt by retaining iron in the insoluble phase [33]. Relative to these reference cases, the present study achieves comparable or superior metal recoveries while maintaining controlled dissolution of Fe, Al, and Cr, confirming that the selected roasting temperature and sulfating conditions promote selective sulfation with limited impurity carryover.

Chemical and mineralogical composition of the raw ore

Bulk chemical analysis (Table 2) shows that the Kempirsay laterite contains 1.08 wt.% Ni and 0.10 wt.% Co, classifying it as a low-grade limonitic ore [2]. Iron is the dominant metallic component (20.2 wt.%), while elevated Si and Al indicate a typical Fe–Al-silicate matrix characteristic of weathered laterites [4].

XRD analysis (Figure 1) identified goethite, hematite, quartz, clinocllore-IIb-4, and the Ni-Fe spinel trevorite as the main mineral phases. The presence of goethite and spinel phases suggests that Ni and Co predominantly substitute for Fe in oxyhydroxide and spinel

Table 1. Comparative results of SR-WL processing of nickel ores and concentrates, showing the influence of modifiers and roasting parameters on Ni and Co extraction.

References	Laterite Type/ Source	Sulfating Agent	Roasting Temperature (°C)	Extraction of Ni	Extraction of Co	Impurity Metals (Fe, Al, Cr and other)	Key Findings/Notes
[32]	a limonitic laterite ore	H ₂ SO ₄ 10–90 wt.%	700	>88%	93%	< 4% Fe. Simultaneously, about 90% Mn and Cu, 70% Mg, 45% Al, 25% Zn, 4% Cr and Ca are extracted respectively	The process demonstrates efficient separation of valuable metals from iron. Low iron dissolution; low acid consumption – 250– 500 kg/t.
[33]	a limonitic laterite ore	80 wt% H ₂ SO ₄	725	91.1%	91.5%	Al – 3%	With the increase of the roasting temperature, the conversion of Fe(II) to Fe(III) effectively prevents the leaching of iron (Fe) and chromium (Cr).
[34]	nickel sulfide concentrate	(NH ₄) ₂ SO ₄	650	90%	92%	< 1% Fe	Adding Na ₂ SO ₄ shows promise as a solution for processing nickel-bearing sulfide ores. The Na ₂ SO ₄ additive was observed to play a noticeable role in promoting the sulfation degree of valuable metals by forming liquid phases [Na ₂ Me(SO ₄) ₂] at the outermost layer, which can create a suitable dynamic environment for sulfation.
[35]	mixed oxide- sulfide nickel ore	(NH ₄) ₂ SO ₄	450	97%	-	97% Cu, and 82% Fe	Roasting temperature and particle size of the ore have greater effect on the extractions of metal
[36]	nickel oxide – sulfide mixed ore concentrate	(NH ₄) ₂ SO ₄	650	70%	89%	Fe is not detected.	Nickel oxide – sulfide mixed ore concentrate (not pure laterite, but relevant to sulfation roasting)
[37]	Jinchuan nickel concentrate.	NH ₄ HSO ₄	400	95.7%	98.9%	Iron impurities in the PLS can be removed as NH ₄ [Fe ₃ (SO ₄) ₂ (OH) ₆] precipitate	(1) The roasting temperature is lower, and the products are mainly metal-ammonium complexes; (2) High metal extractions can be obtained without adding sodium sulfate;(3) There is no problem of sodium sulfate recovery in the subsequent solution, which simplifies the process.
[38]	saprolite ore	H ₂ SO ₄ and Na ₂ SO ₄ , K ₂ SO ₄ , (NH ₄) ₂ SO ₄ and NaCl	700	90%, it was achieved by addition of 5 wt% Na ₂ SO ₄	-	-	Na ₂ SO ₄ create favourable conditions for sulfate formation of metal oxide by forming liquid pyrosulfate as an intermediate phase at elevated temperatures, which penetrated into sulphated ore, and provided more sulfuric trioxide gas to form more sulfates.
[33]	co-bearing nickeliferous laterites	Fe ₂ (SO ₄) ₃	500	Not specified	up to 73.4%	Iron (Fe) – in the insoluble phase	Demonstrated efficient cobalt upgrade with iron remaining in the insoluble phase.

structures [39, 40], which strongly influences their conversion into soluble sulfates during sulfation roasting [33, 41].

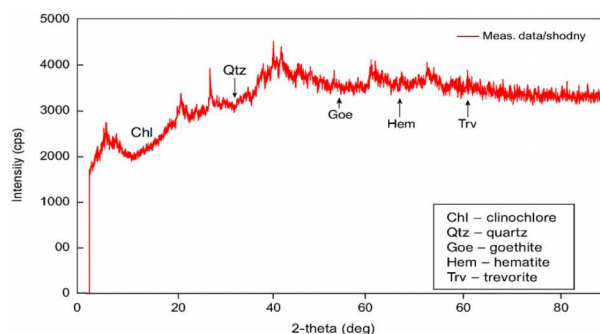
SEM-EDS analysis (Figure 2a-b) reveals aggregated particles with heterogeneous and partially porous textures characteristic of limonitic laterites [40]. The

Table 2. Chemical composition of laterite ore analyzed by XRF (wt%).

NiO	CoO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	MnO	Cr ₂ O ₃	CaO	Na ₂ O	L.O.I.
1.37	0.13	2.65	28.88	2.10	28.88	2.47	0.92	0.59	1.08	30.93

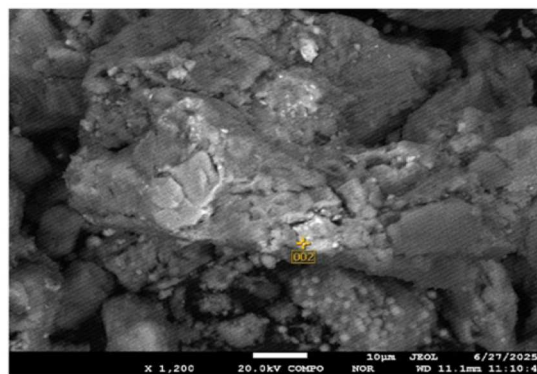
analysis of first sample (Figure 2 a, point 002) shows relatively low Fe content (5.39 wt.%) and elevated concentrations of Si (13.68 wt.%) and Ce (27.05 wt.%). Nickel is present at 0.74 wt.%, a typical value for many lateritic ores, indicating that Ni is likely chemically bound to or isomorphically substituting Fe and Mg ions within the crystal lattice of Fe-hydroxides (goethite-type) and silicate minerals, thereby forming a Fe-Ni lateritic matrix [41]. Despite the presence of a characteristic Fe-O-Si-Ni matrix, the dominant element in this sample is cerium (27.05 wt.%), reflecting significant enrichment in rare-earth phases. The Fe-Si-Ni matrix likely acts as a host for rare-earth mineralisation. Such an association is characteristic of the formation of rare-earth silicate-phosphate phases typically observed in the upper, more weathered zones of lateritic profiles, where rare-earth elements migrate and become fixed within siliceous matrices in the form of Ce-phosphates (monazite, CePO₄) or Ce-silicates (Ce₂Si₂O₇). In contrast, second sample (Figure 2a, point 001) exhibits high concentrations of Fe (35.88 wt.%) and O (39.35 wt.%), indicating the presence of Fe-bearing phases such as goethite (FeO(OH)), hematite (Fe₂O₃), or a mixture of Fe oxyhydroxides typical of laterite systems. Additionally, measurable amounts of Ni (3.29 wt.%), Cu (3.66 wt.%), and Zn (3.15 wt.%) were detected, suggesting the formation of a Ni-Fe carbonate-hydroxide association, commonly found in oxide-type lateritic ores.

Taken together, the XRF, XRD, and SEM-EDS data indicate that Ni and Co are predominantly hosted within iron oxyhydroxides, Fe-silicates, and spinel-type phases a mineralogical arrangement known to influence their reactivity and extraction behaviour during the SR-WL process [42, 43].

**Figure 1.** XRD pattern of the ore.

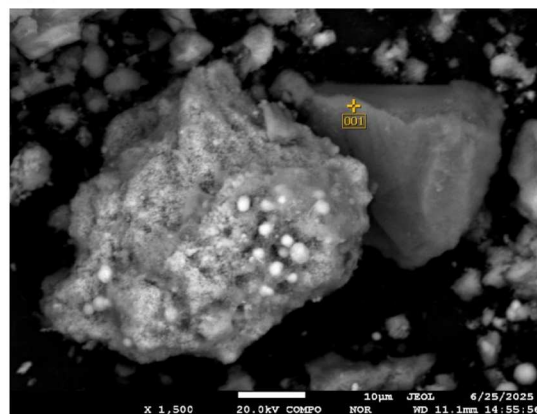
Roasting

Thermodynamic analysis of the Ni-S-O (Figure 3a), Co-S-O (Figure 3b), and Fe-S-O (Figure 3c) systems was performed to determine the temperature and SO₃ partial pressure ranges favourable for selective sulfation during roasting. To determine the optimal conditions for sulfate formation and to understand the behaviour of the main components of laterite ore during roasting with sulfuric acid, a thermodynamic analysis was performed using HSC Chemistry 10.0 software. The analysis was conducted in a temperature range of 0–800 °C, with the partial pressure of ($p(\text{SO}_3)$) as a variable and the oxygen partial pressure held constant at a low value: $P(\text{O}_2(\text{g})) = 1 \times 10^{-20}$ bar.



O	Al	Si	P	Ca	Fe	Ni	Ce
47.27	3.45	13.68	1.44	0.98	5.39	0.74	27.05

(a)



O	Mg	Al	Si	Ca	Fe	Ni	Cu	Zn
39.35	0.99	0.92	7.48	5.28	35.88	3.29	3.66	3.15

(b)

Figure 2. SEM images of the raw laterite ore and corresponding elemental composition (wt.%): (a) analysis area 002; (b) analysis area 001.

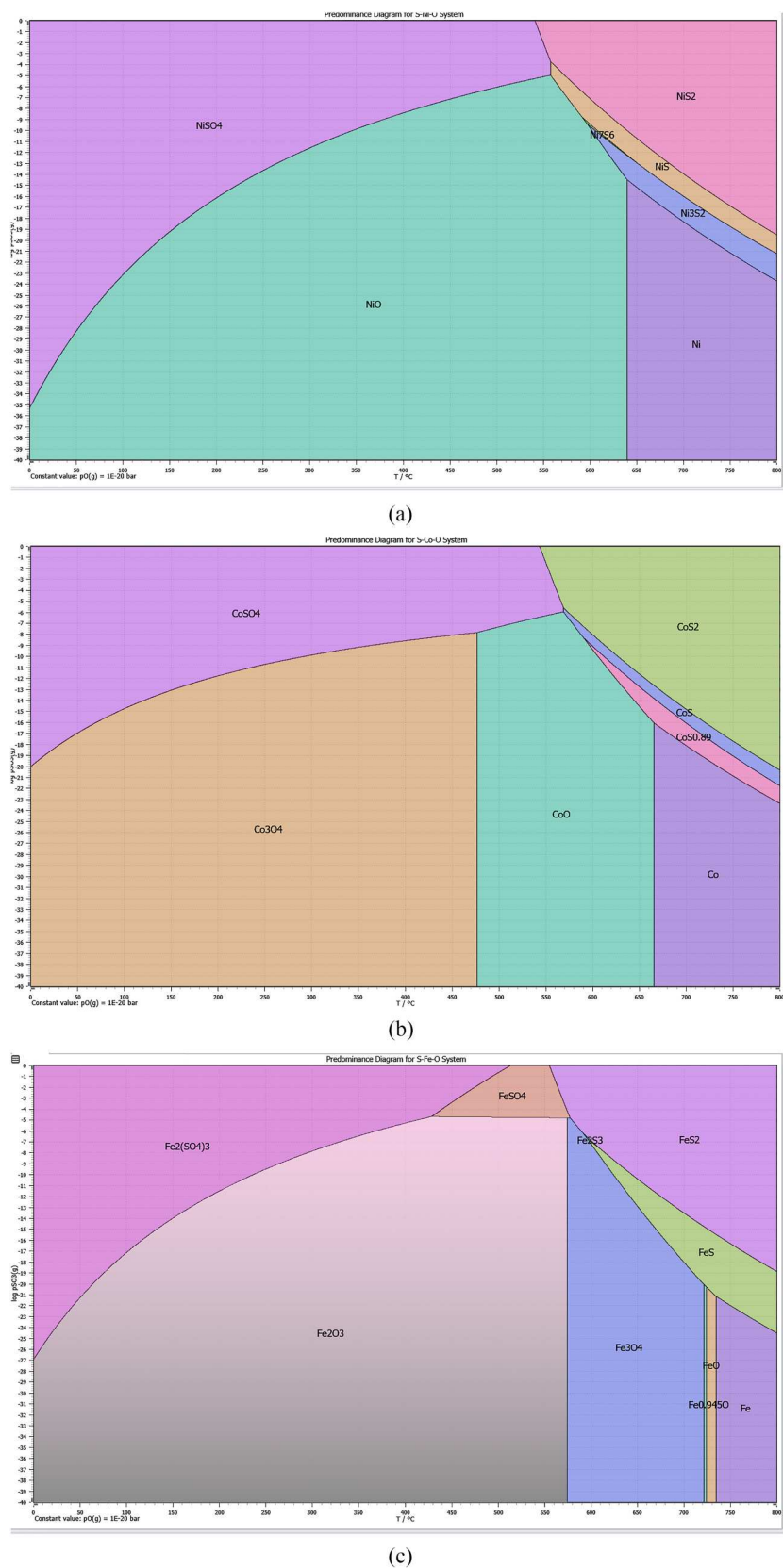


Figure 3. Diagrams of $\log p(\text{SO}_3)$ - $f(T)$ dependencies for Fe-S-O (a), Co-S-O (b) and Fe-S-O (c) systems in the temperature range from 0 to 1000 °C.

According to the predominance diagrams, NiSO_4 and CoSO_4 are thermodynamically stable only at temperatures below 500–550 °C, provided that SO_3 partial pressure exceeds $\sim 10^{-12}$ – 10^{-11} bar. At higher temperatures (>600 °C), both sulfates decompose to oxides or sulfides regardless of sulfur pressure [44].

In contrast, iron sulfates exhibit significantly narrower stability fields. $\text{Fe}_2(\text{SO}_4)_3$ is stable only below ~ 450 °C, while FeSO_4 exists between 450–550 °C at elevated SO_3 partial pressure. Outside this interval, iron forms oxides (Fe_2O_3 , Fe_3O_4) or sulfides, which inhibit Ni and Co release due to structural incorporation [45].

Thus, thermodynamic modeling indicates that the optimal temperature window for selective formation of NiSO_4 and CoSO_4 , while suppressing excessive iron sulfation, lies between 400–500 °C [44, 45].

Thermal decomposition behaviour of the laterite

The TG-DTA results (Figure 4) confirm that the key transformations required for effective sulfation occur within the same temperature range predicted thermodynamically.

The first mass-loss event (80–250 °C) corresponds to dehydration. The major transformations occur between 250–370 °C, including the dehydroxylation of goethite and the breakdown of clinocllore-IIb-4, accompanied by formation of amorphous Mg-Si phases. Above 400 °C, crystalline silicates begin to form, while hematite becomes the dominant iron oxide phase [45, 46].

These transformations create reactive surfaces and free $\text{Ni}^{2+}/\text{Co}^{2+}$ sites previously bound in silicate-goethite structures, facilitating subsequent sulfation. The cumulative 15% mass loss by 800 °C is consistent with decomposition reactions described for laterites.

Phase composition after sulfation roasting

XRD analysis of the H_2SO_4 -roasted ore (Figure 5) directly confirms the formation of sulfate phases predicted by thermodynamic modeling. The roasted product contains NiSO_4 (nickel sulfate), MgSO_4 (magnesium sulfate), $\text{Al}_2(\text{SO}_4)_3$ (millosevichite), and $\text{Fe}_2(\text{SO}_4)_3$ (mikaite), along with SiO_2 (quartz).

This phase assemblage demonstrates that at roasting temperatures of 450–500 °C and sufficient SO_3 generation, nickel and cobalt are converted into soluble sulfates. The presence of a limited amount of $\text{Fe}_2(\text{SO}_4)_3$ indicates that iron sulfation is partially suppressed, consistent with the Fe-S-O diagram (Figure 3c).

Importantly, hematite is not the dominant phase after roasting at these conditions, indicating that Ni

and Co were not encapsulated in iron oxides, which would otherwise reduce leaching efficiency.

Table 3 shows the parameters, including the initial (TI) and final decomposition temperatures (TF), corresponding peak temperatures (TP) and maximum weight loss rates, and thermodynamic data.

The thermal analysis of laterite after roasting at 500 °C reveals that laterite ore undergoes significant stage transformations of its main ore-bearing and waste rock minerals, in addition to the decomposition of goethite. These changes are supported by X-ray diffraction analyses (Figure 5).

During sulfation roasting, the transformation of the laterite proceeds through three coupled stages. First, structural dehydroxylation of goethite and decomposition of clinocllore create an open, reactive surface that exposes finely dispersed Ni-Co bearing domains. In the presence of SO_3 generated in situ from H_2SO_4 decomposition, Ni and Co readily convert into their corresponding sulfates (NiSO_4 , CoSO_4), which are thermodynamically favoured within the 350–500 °C stability window. Iron, by contrast, exhibits a narrower sulfate stability field; FeSO_4 partially forms but rapidly oxidises or transforms into $\text{Fe}_2(\text{SO}_4)_3$ or hematite depending on local $p\text{SO}_3$. As a result, Ni and Co undergo preferential sulfation, while a significant portion of Fe remains in less soluble oxide or spinel forms.

Integration of the thermodynamic predictions (Figure 3), thermal decomposition behaviour (Figure 3), and phase assemblages after roasting (Figure 5) demonstrates that the temperature range of 450–500 °C provides the most favourable balance for selective sulfation. At these temperatures, NiSO_4 and CoSO_4 are stable and abundant, while extensive formation of iron sulfates or their decomposition products is avoided. Higher temperatures (>500 °C) shift all sulfate stability fields toward higher $p\text{SO}_3$ and promote rapid breakdown of NiSO_4 , CoSO_4 , and FeSO_4 , whereas lower temperatures (<350–400 °C) limit the conversion of Ni and Co into soluble sulfate phases. Therefore, the chosen roasting interval ensures maximum formation of leachable Ni- and Co-sulfates while minimising unwanted side reactions.

Microstructural evidence of sulfate formation

SEM-EDS of the samples roasted at 500 °C with 30% mass H_2SO_4 addition (Figure 6a) shows a pronounced morphological transformation.

EDS analysis (Figure 6c) reveals sulfur associated with Fe, Al, Mg, and Ni, confirming the formation of metal sulfates, fully consistent with the XRD results. The block-like crystallites correspond to recrystallized

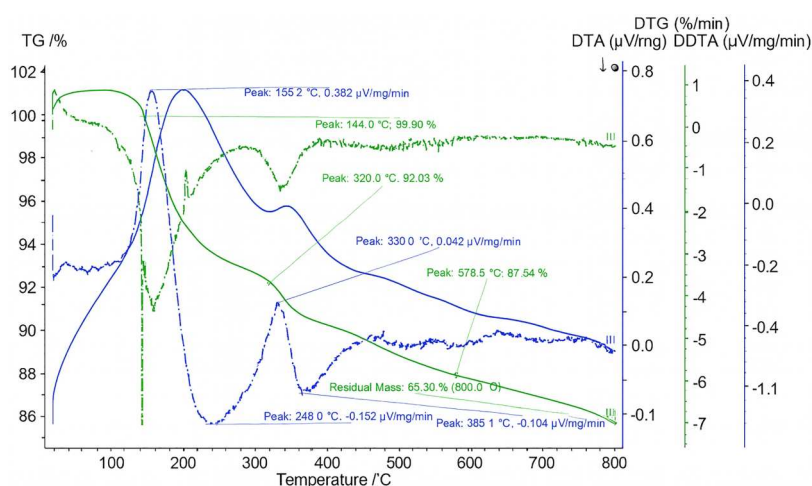


Figure 4. Derivatogram of laterite ore samples.

sulfate phases such as $\text{Fe}_2(\text{SO}_4)_3$, $\text{Al}_2(\text{SO}_4)_3$, MgSO_4 and NiSO_4 . Cobalt sulfate is not visible in EDS due to its trace concentration, but its formation is thermodynamically expected under the applied roasting conditions [45, 47].

These structural and compositional changes increase the reactivity of the roasted material and promote rapid dissolution of Ni and Co during water leaching. The observed sulfate stability at ≤ 500 °C agrees with the known decomposition thresholds of divalent and trivalent metal sulfates reported in the literature [42].

To investigate the conditions required for selective sulfation, additional roasting experiments were performed at 700 °C, and the resulting calcine was characterised by SEM coupled with EDS analysis and compared directly with the material roasted at 500 °C. Increasing the roasting temperature to 700 °C induced pronounced changes in both phase composition and microstructure. SEM observations revealed the transition from a finely porous, sulfate-rich matrix at 500 °C to a denser, partially sintered microstructure dominated by iron oxide phases at 700 °C. These morphological changes are consistent with the thermodynamic

instability of iron sulfate, which undergoes rapid decomposition above ~ 580 °C. EDS mapping confirmed this transformation, showing a strong enrichment of Fe and O and a significant reduction in sulfur within the matrix, while Ni-bearing domains retained detectable levels of sulfur. This observation aligns with the higher thermal stability of NiSO_4 relative to $\text{Fe}_2(\text{SO}_4)_3$, enabling Ni and Co to remain partially in sulfate form under the same roasting conditions.

Leaching behaviour of the roasted laterite cinder

The efficiency of Ni and Co extraction from thermally activated laterite is primarily governed by the sulfatization temperature, sulfuric acid concentration, and roasting duration. As shown in Figure 7a, the optimal roasting conditions were identified as 500 °C for 60 min with 30 wt% H_2SO_4 , which yielded maximum recoveries of 93.3% Ni, 91.9% Co, and 80.5% Fe. Increasing the sulfatization temperature to 700 °C under the same acid dosage resulted in a pronounced decline in metal extraction most notably a sharp reduction in Fe dissolution from 87% to 5%, which significantly improved Ni, Co selectivity. However, at these elevated temperatures the thermal decomposition of metal sulfates becomes substantial, decreasing Ni extraction from 93% to 75% and Co extraction from 91% to 45%. These trends reflect the greater thermal

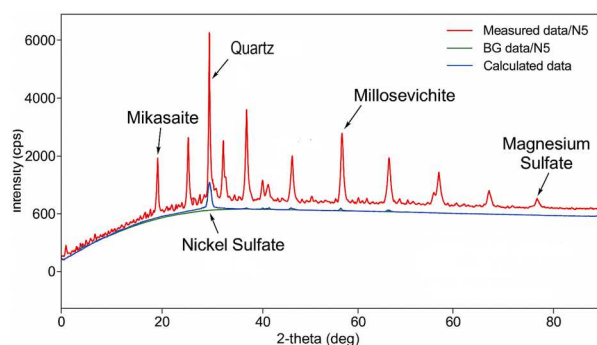


Figure 5. XRD pattern of the ore after roasting at 500 °C.

Table 3. Thermal decomposition of laterite in the temperature range of 20–800 °C.

Stages	Stage Decomposition range (°C)		Peak temperature (°C)	$\mu\text{V/mg/min}$
	T_{init}	T_{final}		
1	80.0	248.0	155.2	0.382
2	248.0	365.0	330.0	0.042

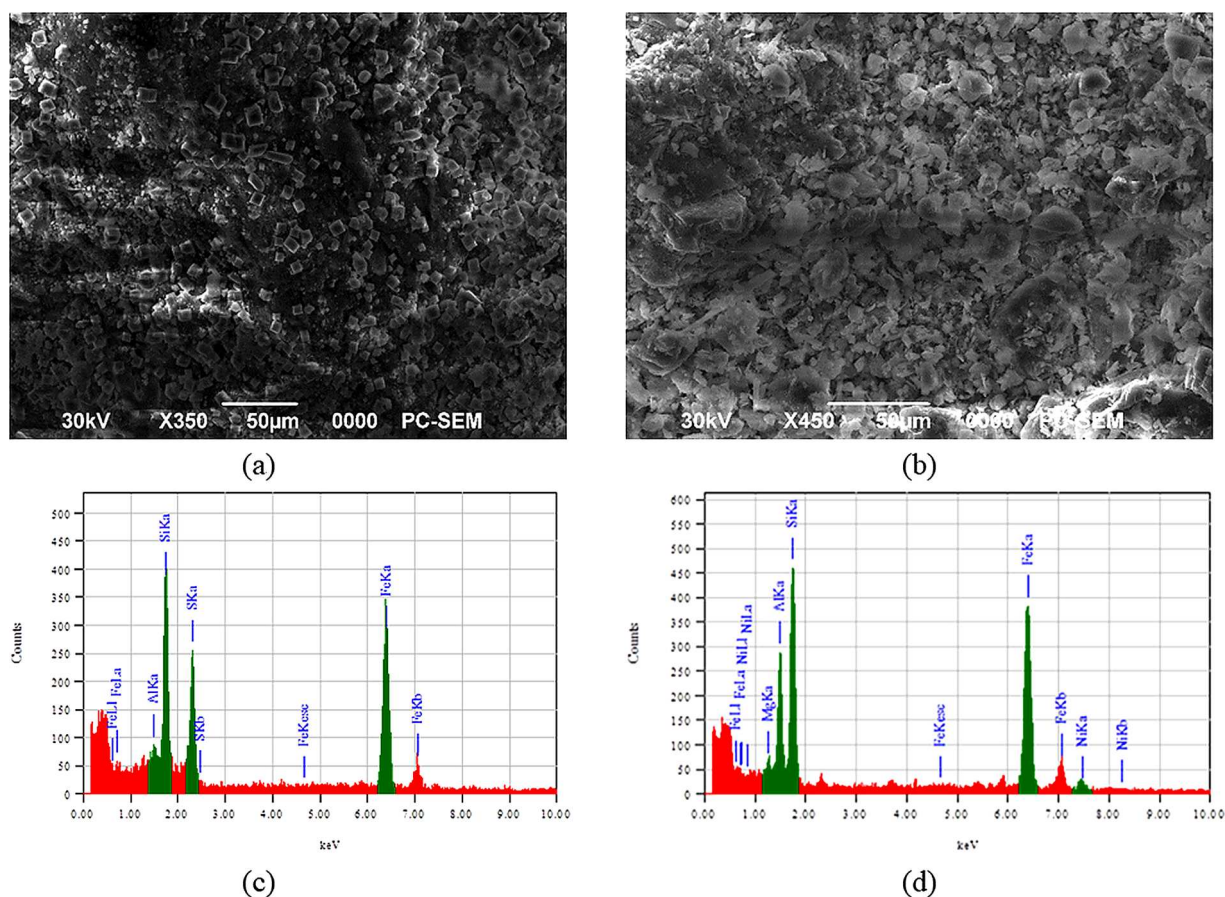


Figure 6. SEM images and EDS spectra of the ore after roasting at 500 °C (a, c) and 700 °C (b, d).

stability of NiSO_4 and CoSO_4 relative to FeSO_4 , the latter decomposing readily into Fe_2O_3 and SO_3 , leaving much of the iron locked in refractory oxide structures.

To evaluate the effects of acid dosage and leaching duration on the maximum extraction of metals from the calcine produced at 500 °C, the influence of increasing sulfuric acid concentration (30–50 wt.%) (Figure 7b) and leaching time from 30 to 180 min (Figure 7c) was systematically investigated. As shown in Figure 7b, raising the acid concentration from 10 to 30 wt.% substantially enhanced extraction from 43.4% to 93.3% for Ni, from 59% to 92% for Co, and from 46% to 84% for Fe. Further increasing the dosage from 30 to 50 wt.% led to only marginal improvements in Ni and Co extraction (0.5% and 1%, respectively), but increased Fe dissolution from 80.4% to 84%, indicating diminishing returns and reduced process selectivity.

Increasing the roasting duration of laterite at 500 °C with the addition of 30 wt.% H_2SO_4 from 30 to 60 min led to a sharp increase in the extraction of all metals into PLS, whereas extending the roasting time from 60 to 180 min produced no further improvement. This behaviour indicates that the sulfation reactions of Ni, Co, and Fe are effectively completed within the first

hour of roasting. At 500 °C, the system reaches a quasi-equilibrium state in which the available reactive surface is fully converted to the corresponding metal sulfates, and the supply of SO_3 generated from acid decomposition becomes insufficient to drive additional reactions. Under these optimised conditions, the extraction efficiencies reached 93.3% for Ni, 92% for Co, and 84% for Fe.

However, this roasting regime did not provide selective extraction of the target metals. As shown in the corresponding figures, selective dissolution of Ni and Co is achieved only at higher roasting temperatures 700 °C and above, where the thermal decomposition of iron sulfates to oxides effectively suppresses Fe dissolution during leaching. To further optimise the process, additional studies were conducted to determine the optimal sulfuric acid concentration capable of promoting sufficient sulfate formation while simultaneously enhancing the extraction of the target metals under high-temperature sulfatization conditions.

Systematic variation of the acid dosage between 30 and 70 wt.% H_2SO_4 at a roasting temperature of 700 °C (Figure 7d) revealed a pronounced influence of acid concentration on sulfate stability and, consequently, on the

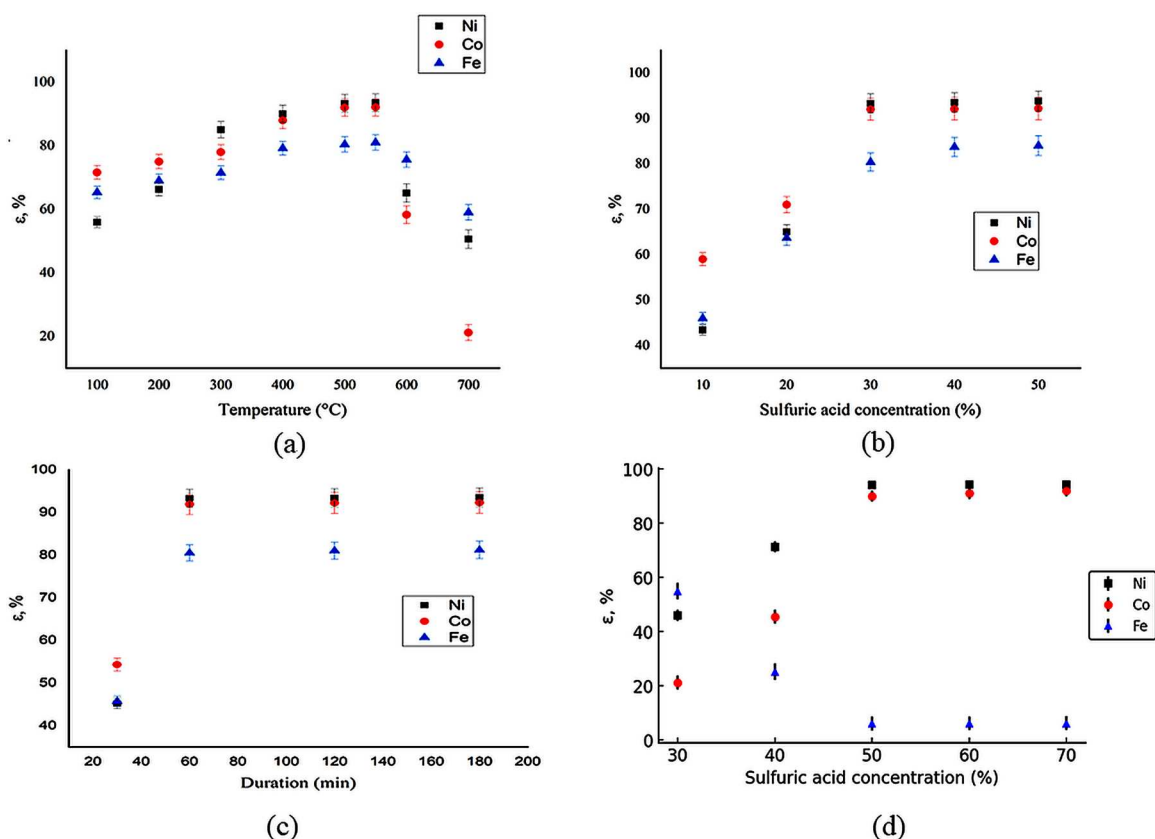


Figure 7. Dependence of metal extraction on (a) the roasting temperature; (b) the roasting duration; (c) the concentration of acid added to the quality of the activator at 500 °C, (d) the concentration of acid added to the quality of the activator at 700 °C.

extraction behaviour of the metals. At lower dosages (30 wt.%), the extent of sulfation at 700 °C was insufficient to compensate for the rapid thermal decomposition of metal sulfates, leading to substantial losses of Ni and Co into refractory oxide phases. Increasing the acid concentration enhanced the availability of SO_3 during roasting and improved the stability of NiSO_4 and CoSO_4 under high-temperature conditions.

An optimal balance was achieved at 50 wt.% H_2SO_4 , where sufficient sulfation occurred to preserve Ni and Co in extractable sulfate forms while iron sulfate was largely decomposed to hematite and magnetite. Under these conditions, high extraction efficiencies of 90.08% Ni and 90.01% Co were achieved, whereas Fe dissolution remained suppressed at 6.12%, confirming the strong selectivity of the process. Additional increases in the acid dosage beyond 50 wt.% yielded no measurable improvement and, in some cases, promoted partial agglomeration or densification of the calcine, which hindered further conversion.

Overall, these results highlight the critical role of roasting temperature in controlling the selective sulfatization behaviour of laterites. Higher temperatures promote the thermal decomposition of iron sulfate into oxide phases, thereby suppressing Fe dissolution during

leaching and enhancing the relative selectivity of Ni and Co extraction.

The optimal roasting conditions for the sulfation of Ni, Co, and Fe were established as 500 °C for 60 min with 30 wt.% H_2SO_4 (cinder 1), whereas selective sulfation of nickel and cobalt was achieved at 700 °C for 60 min with 50 wt.% H_2SO_4 (cinder 2).

To determine the optimal aqueous leaching conditions for cinders 1 and 2, the effects of temperature, S: L ratio, and leaching duration were systematically investigated. The experiments showed that 85 °C provides the highest extraction efficiencies (Figure 8a), while further increases in temperature resulted in only marginal improvements. The S: L ratio exerted a strong influence (Figure 8c): at S: L = 1:6, metal extraction reached 93.3% Ni, 92% Co, and 80.4% Fe.

Leaching kinetics (Figure 8b) demonstrated rapid initial dissolution. Increasing the leaching time from 30 to 180 min raised Ni extraction from 39% to 93.3% and Co extraction from 31.5% to 92%. Beyond 180 min, the dissolution rate slowed due to reduced reactive surface area and partial particle passivation, whereas Fe extraction increased disproportionately. Therefore, 180 min was selected as the optimal leaching duration.

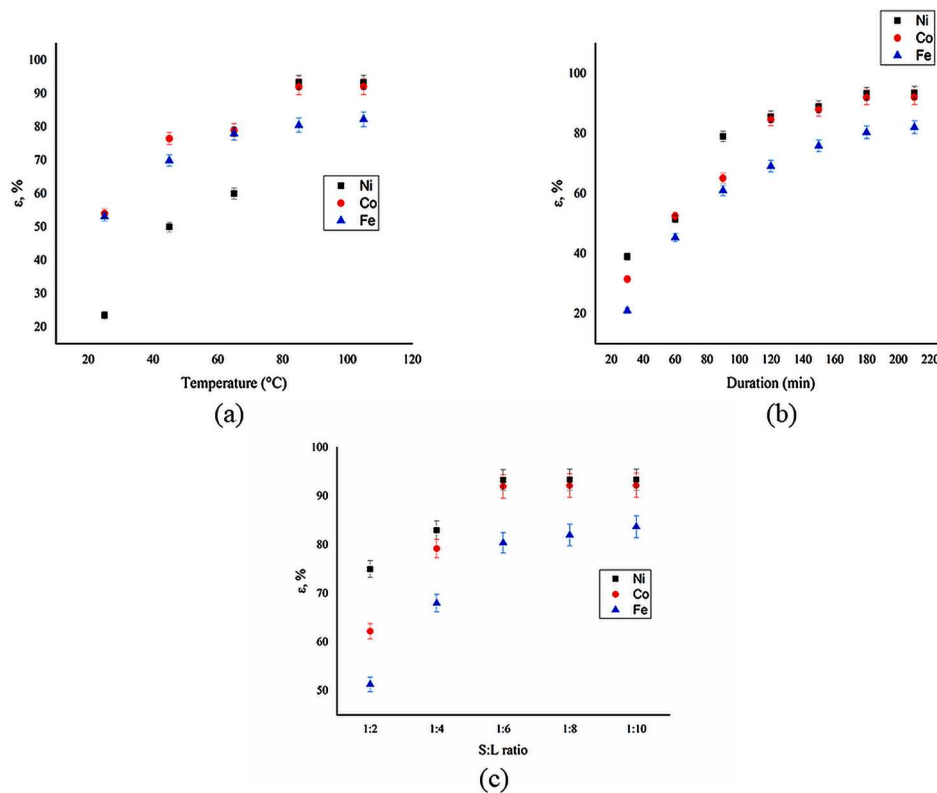


Figure 8. Effect of leaching parameters on metal extraction from roasted laterite cinder at 500°C: (a) leaching temperature; (b) leaching duration; (c) S: L ratio.

Under the optimised conditions (85 °C, S: L = 1:6, 180 min), the PLS 1 contained $2.5 \text{ g}\cdot\text{L}^{-1}$ Ni, $0.325 \text{ g}\cdot\text{L}^{-1}$ Co, and $22 \text{ g}\cdot\text{L}^{-1}$ Fe. During leaching, increased viscosity and the formation of a white precipitate were observed, attributable to hydrolysis of Fe- and Al-bearing species [48], which limits Fe solubility.

Notably, the optimal leaching parameters identified for cinder 1 were also effective for cinder 2. Leaching of cinder 2 under these conditions produced a highly selective PLS with the following composition (g L^{-1}): 2.11 Ni, 0.35 Co, 2.12 Fe.

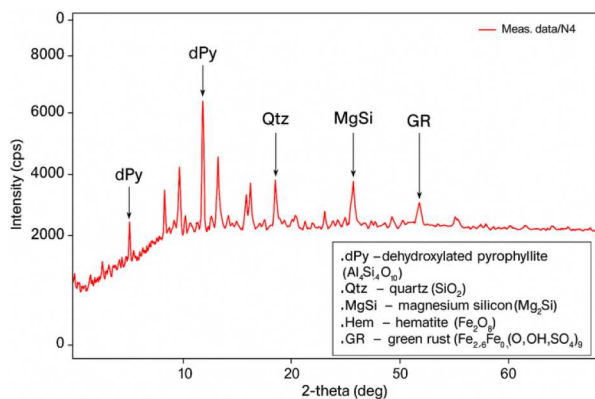


Figure 9. XRD pattern of the solid residue obtained after leaching.

Physico-chemical analysis of the solid residue after leaching cinder 1 (Figure 9) indicates that mineral dissolution was not complete. The residue contains Fe_2O_3 , SiO_2 , $\text{Al}_2\text{Si}_4\text{O}_{10}$, Mg_2Si , and $\text{Fe}_{3.6}\text{Fe}_{0.9}(\text{O},\text{OH},\text{SO}_4)_9$. In addition, the formation of sulfate hydroxides and layered complex iron sulfates was observed.

SEM-EDS analysis of the cinder 1 after leaching (Figure 10 a, b) shows that the crystalline phases are heterogeneous in shape, size, and spatial distribution. Residual phases such as hematite, water-insoluble aluminum and calcium sulfates, and silica are still present in the sample. Although quantitative calculations confirmed that leaching was not fully complete, neither nickel nor cobalt was detected by EDS. This apparent discrepancy is attributed to the presence of a small fraction of Ni and Co that remained insoluble, likely trapped within the interior of ore-bearing mineral phases and/or adsorbed on aluminum sulfate, calcium phases, and other gangue minerals.

SEM-EDS analysis of the cinder 2 leached under optimal conditions (Figure 10 c, d) shows that Fe persists in the form of stable Fe-oxide aggregates, confirming that these phases remain largely undissolved and are retained in the solid residue after leaching.

The experiments demonstrated that the thermochemical treatment of laterite roasting with sulfuric

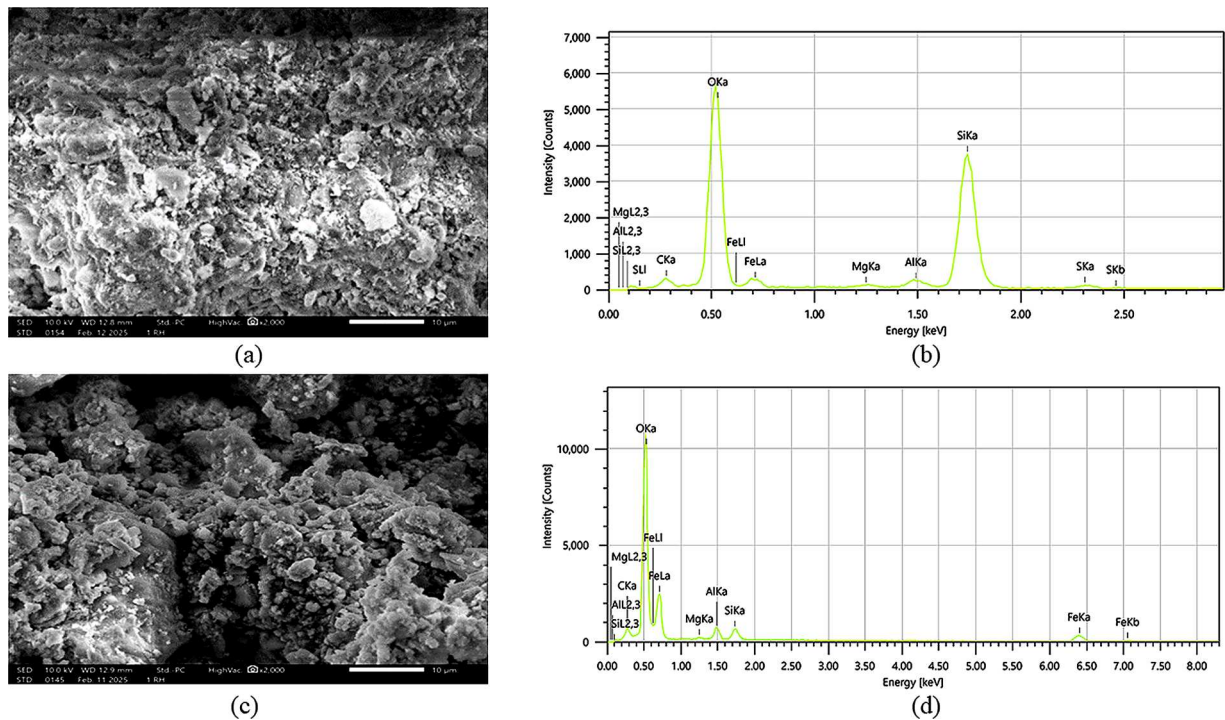


Figure 10. SEM images and EDS spectra of the solid residue after leaching cinders 1(a, b) and 2 (c, d).

acid followed by leaching results in pronounced colour changes (Figure 11). The initial brown laterite (a) transforms into an intense reddish-brown cinder after roasting (b), and the residue obtained after leaching appears grayish (c), while the cake of the leached calcine at 700 °C shown in (d) is characterised by a fine, light-gray powdery texture. These colour transformations reflect the phase and chemical changes occurring in the material. During roasting, sulfation and subsequent decomposition of nickel- and cobalt-bearing iron minerals lead to the formation of intensely coloured sulfate salts (Fe, Ni, Co sulfates) and hematite. During the leaching step, the dissolution and removal of these sulfates and iron compounds into the PLS result in the grayish appearance of the final solid residue.

Comparative evaluation of the SR-WL performance with similar studies

The present study demonstrates the technical feasibility of the SR-WL process for the beneficiation of lateritic ores from the Kempirsay deposit, enabling high recoveries of both nickel and cobalt.

Although elevated roasting temperatures promote the transformation of iron-bearing phases into hematite, magnetite and effectively reduce the iron content in the PLS, insufficient amounts of sulfating agent lead to significant retention of nickel and cobalt within refractory Fe-rich matrices. When the optimal acid dosage is supplied, however, adequate sulfate formation is maintained, enabling the selective extraction of nickel

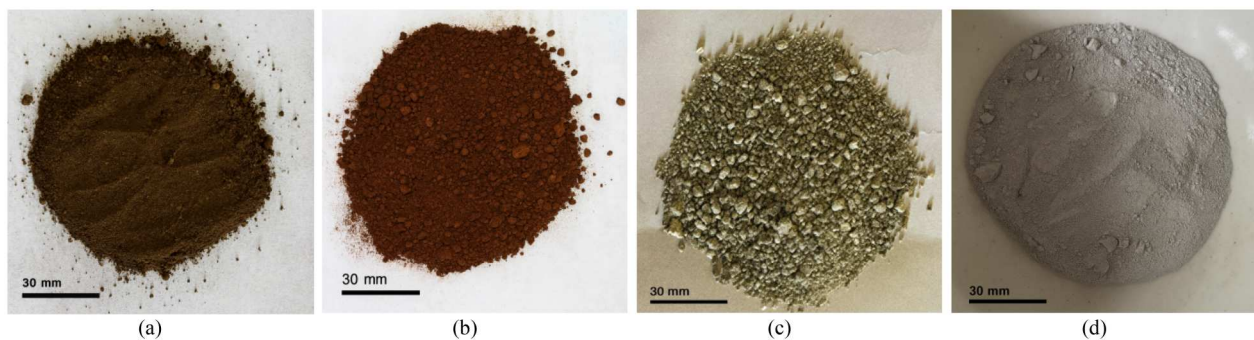


Figure 11. Visual changes in laterite samples throughout the SR-WL process: (a) raw laterite; (b) roasted product after sulfation; (c) solid residue after leaching cinder 1 and (d) solid residue after leaching cinder 2.

and cobalt. This trade-off highlights the need for further optimisation of the roasting stage, which will be addressed in future investigations.

A comparison with previously published sulfation-roasting and leaching studies shows that the optimised SR-WL process developed in this work achieves notably higher metal recoveries while operating under comparatively milder conditions, including a substantially lower acid consumption. In this study, extraction efficiencies of 93.3% Ni, 92% Co, and 84% Fe were achieved, which exceed or match most reported values for ores of similar grade. At the same time, the selective roasting condition, 700 °C, 50 wt.% H₂SO₄, 1 h, provides a markedly improved balance between nickel recovery and impurity dissolution. The improved performance reflects the thermodynamic interplay between sulfate

formation and decomposition, the distinct stability fields of Fe-, Ni-, and Co-sulfates, and the controlled evolution of the oxide-silicate matrix. Compared with the roasting temperatures commonly reported in previous SR-WL studies (Table 1), the conditions used in this work fall within the established operational window while providing a favourable balance between nickel extraction and impurity dissolution.

Iron removal and ni/Co separation strategy

The significant iron burden in both PLS 1 (22 g·L⁻¹) and PLS 2 (2.12 g·L⁻¹) necessitates a robust purification stage prior to nickel and cobalt recovery. To achieve this, the proposed flowsheet incorporates sodium jarosite precipitation using NaOH as the neutralising agent. The process is maintained at 90–95 °C and pH 1.5–1.8, with a residence time of 3–4 h. Controlling the reaction within this specific operating window is essential to promote the growth of coarse, filterable crystals and to strictly minimise the co-precipitation of valuable metals [49]. Under optimised sodium jarosite precipitation conditions, no measurable losses of nickel or cobalt were observed, confirming the selectivity and metallurgical efficiency of the iron removal stage.

Following iron removal, selective separation of Ni and Co is achieved using solvent extraction with Cyanex 272. Updated studies confirm that Cyanex 272 remains the most selective extractant for Co²⁺ over Ni²⁺ in sulfate systems, especially under controlled pH conditions and with the application of kinetic modifiers [50, 51]. The cobalt-rich organic phase can then be stripped and processed via electrowinning to obtain high-purity cobalt metal.

The remaining raffinate, already depleted in cobalt, can be further treated for nickel recovery using ion-exchange sorbents. Modern sorption technologies demonstrate high selectivity toward Ni²⁺ and are increasingly applied for nickel purification from complex sulfate liquors [52, 53], allowing the production of high-purity nickel salts or metal by electrolysis.

The process of extraction of nickel and cobalt using the technology of SR-WL with further processing of the PLS using the methods of chemical precipitation of iron, selective separation of nickel and cobalt by solvent extraction is effective. The recommended combined technological scheme for processing laterites from the Kempirsay deposit is shown below (Figure 12).

In addition to Ni and Co extraction, the SR-WL approach offers the potential for integrated valorisation of several valuable by-products. Depending on process conditions, the residue and intermediate streams may serve as sources of iron oxide powders or metallic iron

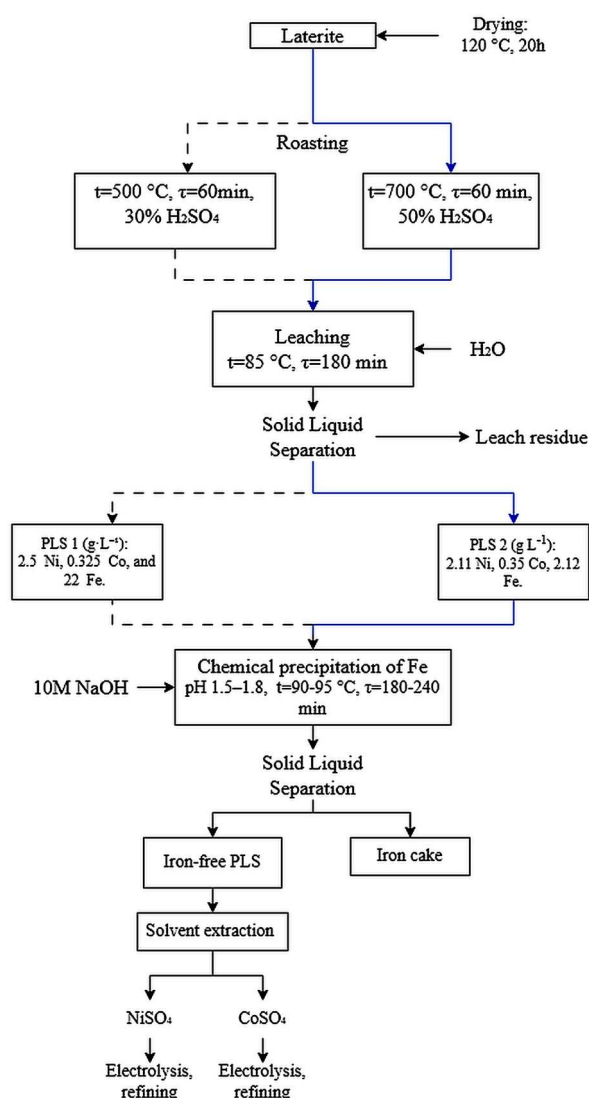


Figure 12. Proposal processing flowsheet for Kempirsay laterite ore.

[13, 54], magnesium compounds [55], aluminum sulfates, as well as trace metals such as chromium and rare-earth elements (REEs). The co-recovery of these components may further enhance the economic attractiveness and resource efficiency of the proposed flowsheet.

Conclusions

Based on the initial experimental data obtained during the processing of laterite ores from the Kempirsay deposit using the SR-WL method, the following key conclusions can be drawn:

The laterite ore is characterised by low nickel and cobalt grades and a goethite-trevorite-silicate mineral assemblage, typical of high-iron laterites.

Thermal analysis conducted under a nitrogen atmosphere revealed a sequential removal of free and structurally bound water, followed by structural transformations of silicate and hydroxide phases above 400 °C. These findings provided a basis for selecting a roasting temperature that promotes effective sulfation while minimising the decomposition of refractory phases.

Roasting at 500 °C with 30 wt% H₂SO₄ facilitated the formation of readily leachable sulfate phases of nickel, cobalt, and iron. Under optimised leaching conditions (solid-to-liquid ratio 1:6, 85 °C, 180 min), high recoveries of Ni (93.3%), Co (92%), and Fe (84%) were achieved. The resulting PLS contained 2.5 g L⁻¹ Ni, 0.325 g L⁻¹ Co, and 22 g L⁻¹ Fe.

For selective extraction of nickel and cobalt, roasting at 700 °C with 50 wt% sulfuric acid proved effective, producing a low-iron PLS containing 2.11 g L⁻¹ Ni, 0.35 g L⁻¹ Co, and 2.12 g L⁻¹ Fe.

The SR-WL process demonstrates clear technical feasibility for the treatment of high-iron laterites from the Kempirsay deposit. Future research should focus on optimising the recovery of all valuable metals from lateritic ore through the use of advanced sulfating reagents and additional physicochemical treatments, as well as on assessing the performance and economic viability of the process under pilot-scale and industrial operating conditions.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

All data generated or analyzed during this study are included in this published article.

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