Roasting-Assisted Cobalt Extraction from Saprolite Nickel Laterite through Sulphuric Acid Leaching and Cyanex 272 Separation

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Abstract: The increasing use of electric vehicles has driven demand for metals such as nickel and cobalt, which are primarily used in battery cathodes. Rising demand for battery metals drives the need for effective nickel and cobalt recovery. This study examines the upgrading of cobalt from nickel laterite ore from Morowali, Central Sulawesi, Indonesia. Laterite, common in tropical regions, is a major source of these metals. Cobalt in laterite mineral deposits is often chemically bound to iron, especially in saprolite-type ores with high magnesium content, making its extraction technically difficult. In the chemical process, magnesium oxide reacts more readily with sulphuric acid than cobalt and nickel oxides do, making the extraction process inefficient. In this study, the ore was mixed with carbon and sodium sulfate, compacted, and roasted at 700–900°C for 2 hours. The highest cobalt content (0.188 wt%) was achieved at 700°C with 5% coal, though it is likely trapped in silicates. A leaching process using 2 M H_2SO_4, followed by pH adjustment to 4.5 with NH_4OH, enabled selective precipitation of iron, achieving up to 94.27% iron removal. The selective extraction process using Cyanex 272 showed a tendency to extract cobalt. However, due to the stripping inefficiency, cobalt recovery in this study remains limited and could be further improved.

Keywords: carbon variation; cyanex 272; nickel cobalt; solvent extraction; temperature variation.

1. Introduction

Cobalt has gained popularity across academia, industry, and the media due to its essential role in lithium-ion batteries powering the rapidly growing electric vehicle industry[1)]. It is estimated that the global annual cobalt demand will increase from 50 kt/yr in 2007 to 190 kt/yr in 2050. Despite its abundant supply, Indonesia is expected to meet its needs by importing cobalt-containing products[2)]. An effective and efficient process needs to be adopted. Nevertheless, cobalt in laterite mineral deposits is frequently chemically locked in iron, especially in saprolite-type ores with an abundance of magnesium content, making its extraction technically difficult because, chemically, magnesium oxide will react more favorably with sulphuric acid than cobalt and nickel oxides, and this makes the extraction process inefficient[3,4)]. Hydrometallurgical technology, especially Atmospheric Acid Leaching (AAL), is more energy-efficient and scalable than high-pressure processes[5)]. However, the selectivity in AAL is poor because impurities, such as iron, are solubilized[6)]. The present work overcomes these shortcomings by pretreating the leachate with different roasting temperatures, adjusting the pH, and performing solvent extraction[7)].

This study aims to investigate which carbon ratio is most effective at increasing the cobalt content through the roasting process. It also aims to identify optimal leaching conditions to maximize cobalt recovery from Indonesian laterite while reducing impurity interference and obtaining purer cobalt in the end product. This study is also conducted with the hope of meeting the need for cobalt in the coming years by using a primary source, which in this

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case is the nickel laterite available in Indonesia. The main contribution of this study is to find cobalt recovery from lateritic nickel ore using Atmospheric Acid Leaching (AAL) with sulfuric acid under varying temperatures, followed by a combined purification process involving pH adjustment with NH_4OH and Cyanex 272 solvent extraction, and H_2SO_4 stripping to recover cobalt in the aqueous phase selectively.

2. Materials and Methods

2.1. Materials

Nickel laterite ore from Morowali, Sulawesi, Indonesia, is used as the main sample. Bituminous coal from a site located in Jambi, Indonesia, is used as the main reducing agent, and Na_2SO_4 from Merck is used as an additive. Lastly, the sample's binding agent is bentonite from Loba Chemie PVT LTD. The materials are processed using a planetary ball mill, a sieve shaker, a hydraulic press machine, and a muffle furnace. Furthermore, leaching was conducted with 2 M H_2SO_4 from Merck. pH adjustment was also carried out to remove impurities, especially from the iron, using 2 M NH_4OH from Merck. To perform the solvent extraction step, Cyanex 272 from Lori Industry and tributyl phosphate from Merck were also utilized. These were dissolved in naphtha from Sigma-Aldrich, which is the organic solvent used. Distilled water was used in all solution preparations and washing steps to minimize contamination and ensure consistent purity.

2.2. Methods

2.2.1. Roasting Method

The nickel laterite ore was processed in a ball mill and sieved to 200-mesh. The prepared materials (ball-milled ore, Na_2SO_4, and bentonite) were then mixed in the same ball mill to ensure a homogeneous mixture[8]. The mixing proportion may vary depending on the amount of coal to be applied; details are shown in Table 1. The mixed materials are then compacted using a hydraulic press to form a pelletized sample for roasting in a muffle furnace. Before roasting, the prepared samples are dried at 100 °C for 2 hours, then roasted for an additional 2 hours at 700 °C, 800 °C, and 900 °C. After roasting, the sample was cooled overnight in the furnace. The samples were characterized using X-Ray Diffraction (XRD) to identify phase transformations, X-Ray Fluorescence (XRF) to assess changes in elemental content, and Scanning Electron Microscope - Energy Dispersive Spectroscopy (SEM-EDS) to examine morphological changes.

2.2.2. Leaching Method

The approaches used in this study included an experimental study that observed the leaching behavior of pre-roasted nickel laterite ore with sulphuric acid, followed by the subsequent separation of cobalt and nickel by solvent extraction. The leaching was performed using 2 M H_2SO_4 at 50°C, 70°C, and 90°C. The solid-to-liquid ratio was 1:10, and the slurry was stirred at 200 rpm on a magnetic stirrer for 4 hours. The filtered leach slurry contained dissolved nickel and cobalt, along with several impurities, such as iron.

To selectively remove iron, the PLS was adjusted to pH 4.5 by adding 2 M NH_4OH, thereby precipitating iron hydroxides. The filtrate obtained was rich in nickel and cobalt. It was then treated by a solvent-extraction procedure in which the organic phase consisted of 15 v/v Cyanex 272, 5 v/v tributyl phosphate, and naphtha as a diluent. The aqueous and organic phases were thoroughly stirred at room temperature for 20 minutes at 200 rpm, then separated to facilitate the selective transfer of cobalt into the organic phase. The cobalt-enriched organic layer was then transferred to 1 M H_2SO_4 for back-extraction (stripping) of cobalt into the aqueous phase. Distilled water was used during the experiment to maintain solution purity and minimize contamination[9]. To assess effectiveness and progress at each step, a couple of characterization methods were adopted. The metal content of the solutions was determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), and the crystal composition of solid samples was estimated with XRD and XRF analysis. Finally, SEM-EDS provided morphological and microchemical insights into the solid residues from pH-adjusted samples.

Table 1: Sample mixing proportions

Nickel Laterite (wt%)	Coal (wt%)	Na_2SO_4 (wt%)	Bentonite (wt%)
84	5	9	2
79	10	9	2
74	15	9	2

Table 2: Initial sample XRF results

Elements (wt%)				
Ni	**Co**	**SiO_2**	**Fe_2O_3**	**MgO**
1.72	0.03	45.14	18.1	19.5
SO_3	**CaO**	**Cr_2O_3**	**Al_2O_3**	**Other**
0.01	0.9	1.08	2.41	11.16

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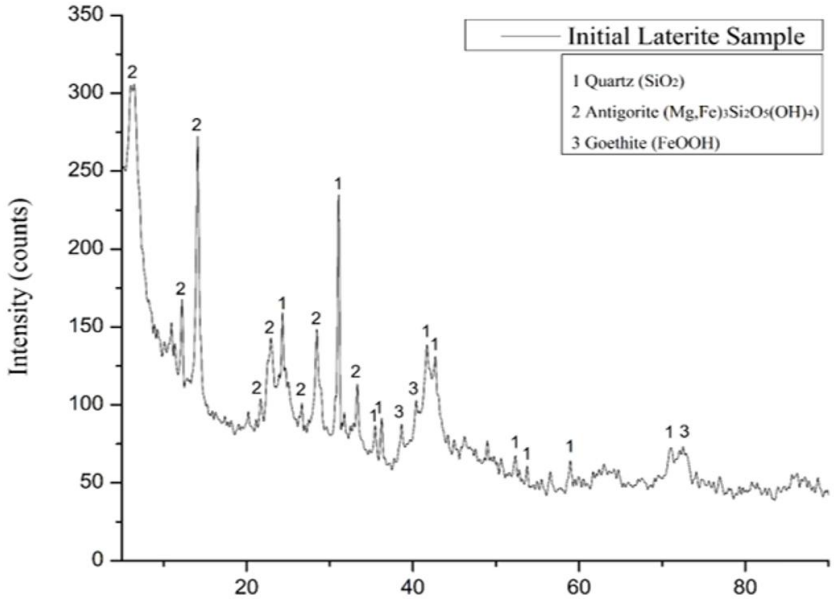


Fig. 1: Initial sample XRD result

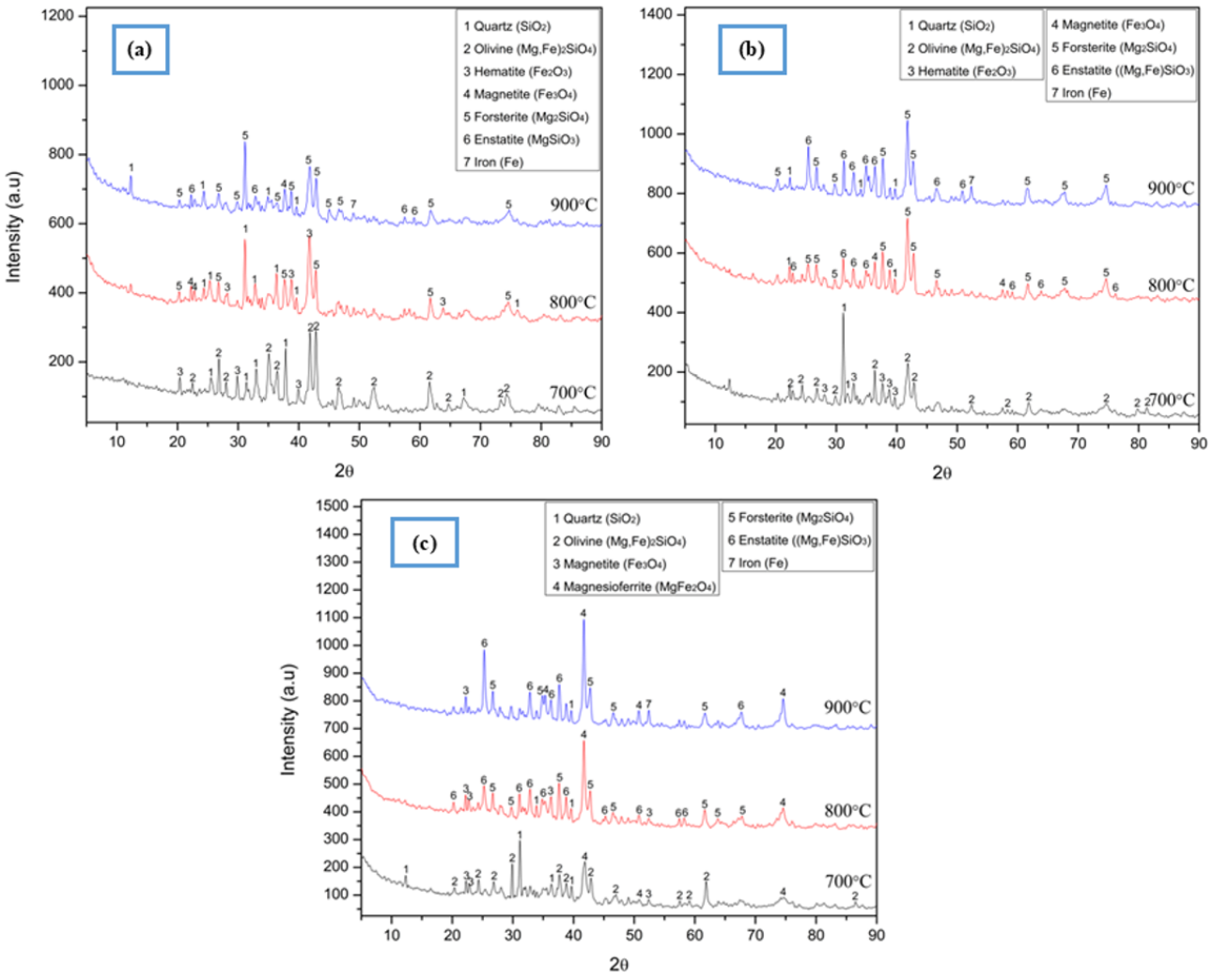


Fig. 2: Roasted samples with a) 5% coal, b) 10% coal, and c) 15% coal ratio XRD results

3. Results

3.1. Roasting Results

The sample is initially characterized, and its main minerals are identified. Quartz is the most abundant mineral, which is common in oxide minerals, especially in the saprolite region of nickel laterites. Antigorite is the second most abundant mineral, which helps enrich nickel content. Lastly, goethite serves as a host for nickel and cobalt, as evidenced by peaks 1, 2, and 3 in Figure 1, allowing both elements to be incorporated into this mineral[10].

In general, cobalt is associated as a substitute for iron in the crystalline lattice of goethite or some phyllosilicates[11]. Based on the XRF results in Table 2, we identified the key minerals present in the sample: nickel (Ni), cobalt (Co), silica (SiO_2), iron (Fe), and chromium (Cr), along with other minerals.

Due to its saprolite region, nickel enrichment may occur, composed predominantly of serpentine, goethite, and garnierite silicate minerals[12]. Serpentine is an important mineral in nickel laterite ore, such as antigorite, where it acts as a host for nickel content[9].

The roasted samples show several mineral transformations, as indicated by the XRD results in Figure 2. During roasting, the sample undergoes several chemical reactions, as shown in reactions (1)-(5).

$$(Mg,Fe,Ni,Co)_3Si_2O_5(OH)_4 \leftrightarrow (Mg,Fe,Ni,Co)SiO_3 + (Mg,Fe,Ni,Co)_2SiO_4 2H_2O \quad (1)$$

$$(Mg,Fe,Ni,Co)_3Si_2O_5(OH)_4 \leftrightarrow 3(Mg,Fe,Ni,Co)_2SiO_4 + SiO_2 + 4H_2O \quad (2)$$

$$Mg_6[SiO_4](OH)_8 \rightarrow 3Mg_2[SiO_4] + SiO_2 + H_2O \quad (3)$$

$$MgO + Fe_2O_3 \rightarrow MgFe_2O_4 \quad (4)$$

$$Na_2SiO_3 + (Mg,Fe,Ni)_2SiO_4 + CO \rightarrow Na_2Mg_2Si_2O_7 + Fe\text{-}Ni + CO_2 \quad (5)$$

Equation 1 shows the dehydroxylation of antigorite into olivine and pyroxene (enstatite) group minerals that occurs, ranging from 580 °C to 650 °C, which explains the loss of all antigorite peaks on every parameter[13].

Equation 2 shows a similar reaction of antigorite dehydroxylation, but produces free silica instead of the pyroxene group mineral, which occurs at the lowest temperature (700 °C)[14,15]. Olivine forms abundantly at 700 °C, then disappears from the pattern as temperature increases. This is explained by Equation 3, in which the serpentine mineral undergoes dehydroxylation and then recrystallizes into forsterite.

Studies suggest that its formation occurs between 810-820 °C and becomes dominant at 900 °C and higher[16,17].

Table 3: The roasted samples' XRF results

5% coal Elements (wt%)						
Temp.	**Mg**	**Si**	**Fe**	**S**	**Co**	**Ni**
700°C	24.036	32.233	30.016	4.983	0.188	4.72
800°C	22.854	32.683	29.806	5.988	0.171	4.579
900°C	22.527	32.695	30.998	4.271	0.178	4.609
10% coal Elements (wt%)						
Temp.	**Mg**	**Si**	**Fe**	**S**	**Co**	**Ni**
700°C	23.811	31.994	29.728	5.805	0.167	4.578
800°C	21.74	31.857	30.905	6.592	0.178	4.697
900°C	21.927	32.559	31.142	5.498	0.179	4.537
15% coal Elements (wt%)						
Temp.	**Mg**	**Si**	**Fe**	**S**	**Co**	**Ni**
700°C	22.411	30.667	31.871	5.999	0.181	4.964
800°C	21.912	31.313	31.364	6.53	0.18	4.697
900°C	21.121	30.527	32.543	6.276	0.185	4.882

Goethite undergoes dehydroxylation to form Fe at 900 °C. The step-by-step reduction proceeds from goethite, hematite, magnetite, and wüstite, then to iron, over a temperature range of around 750-950 °C[18]. Interestingly, magnesioferrite peaks are observed at a coal ratio of 15%. The formation of magnesioferrite is described by Equation 5. Magnesium oxide, formed by antigorite dehydroxylation, reacts with hematite from goethite, forming magnesioferrite efficiently at 800 °C[19]. Na_2SO_4 is utilized as an additive. At 800 °C, it will form new phases such as Na_2SiO_3 and ferrous forsterite, as can be seen in Equation 5. Both of these will react with the available CO and form an Fe-Ni alloy[20]. From the XRF results shown in Table 3, the cobalt content is highest at 700 °C with a 5% coal ratio. If we analyze the peaks, cobalt might be incorporated into hematite or olivine, indicating that some of it is trapped within the silicate phase and might affect the leaching process[21].

By visually inspecting the SEM image shown in Figure 3, we can see that the two have different morphologies. The initial sample comprises heterogeneous structures composed of irregularly shaped particles. Its grains are also relatively fine, with particles of varying sizes. Meanwhile, the roasted sample shows a noticeable change in morphology. The roasting process leads to structural changes, resulting in larger, more clustered particles, and the composition of the roasted sample is shown in Table 4. Roasting can also increase the surface area of the laterite sample, thereby disrupting mineral lattices and making more surface area accessible for leaching or other extraction processes[22]. Furthermore, based on the EDS results, we can see a reduction in oxygen content and a slight increase in nickel content.

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Table 4: Roasted samples XRF results

Element	Sample (wt%)	
	Initial	Roasted
O	38.5	27.52
Na	-	2.6
Mg	20.5	14.57
Al	1.29	2.01
Si	28.59	30.07
S	-	2.21
Ca	0.4	0.66
Cr	0.46	1.09
Fe	8.67	14.18
Ni	1.53	1.77

3.2. Leaching Results

The results of XRF analysis of the leaching residues in Table 5 indicate that an increase in the leaching temperature promotes metal leaching. The final levels of nickel and cobalt in the residue were 2.384 wt% and 0.137 wt%, respectively, indicating better leaching capability at higher temperatures[23,24]. There was also a lower concentration of iron and magnesium, which strengthens the argument for the speed of matrix breakdown and the release of metals as temperature increases.

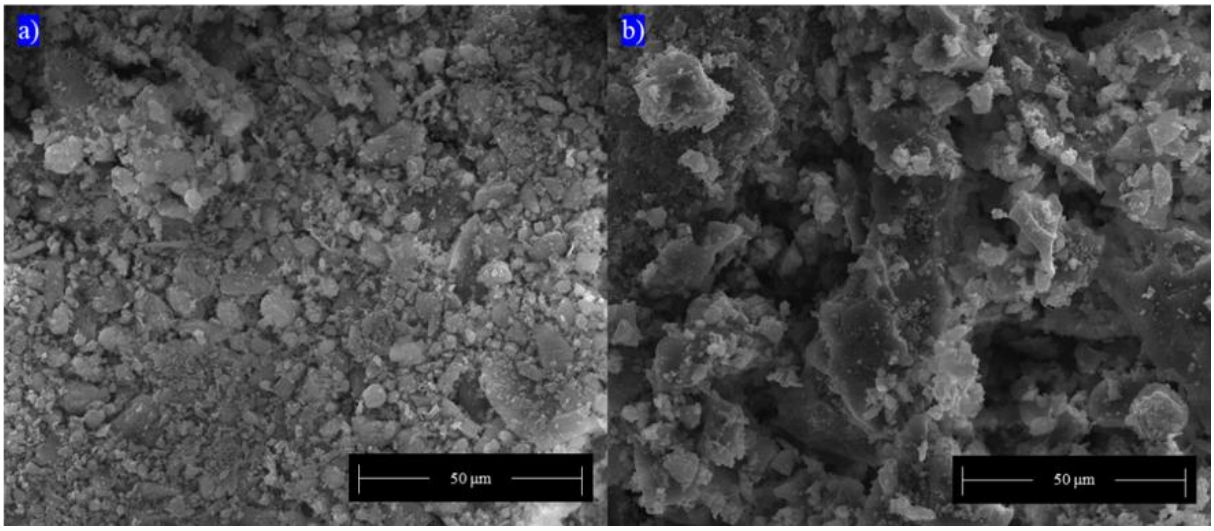


Fig. 3: SEM results of a) initial sample and b) roasted sample (900ºC-15%)

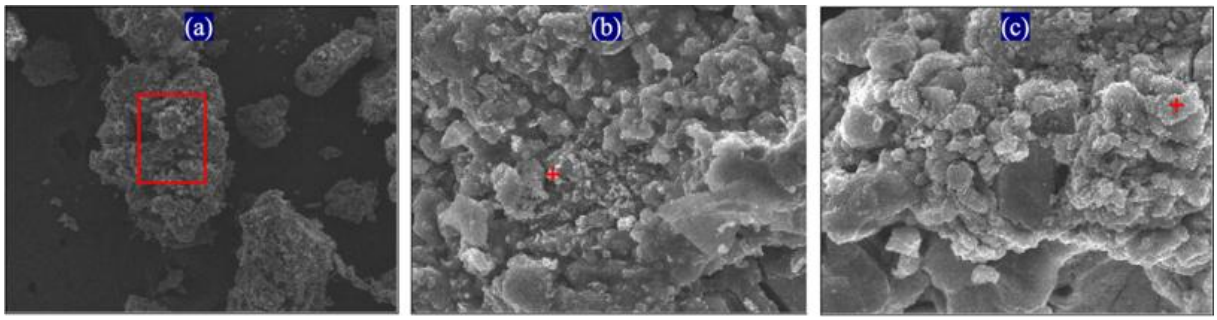


Fig. 4: SEM EDS residues in pH adjustment; (a) 50°C leached PLS, (b) 70°C leached PLS, (c) 90°C leached PLS

Table 5: XRF results of samples' residue leached at 50°C, 70°C, 90°C

Temp.	Element Content (wt%)				
50ºC	Ni	Co	Si	Fe	Mg
	4.088	0.176	44.507	33.675	12.079
	S	Ca	Cr	Al	Other
	0.735	1.785	1.776	0.592	0.587
70ºC	Ni	Co	Si	Fe	Mg
	3.519	0.160	50.196	31.557	9.294
	S	Ca	Cr	Al	Other
	0.516	1.827	1.805	0.580	0.546
90ºC	Ni	Co	Si	Fe	Mg
	2.384	0.137	44.507	26.000	6.315
	S	Ca	Cr	Al	Other
	0.419	2.028	1.974	0.604	0.502

ICP-OES in Table 6 also confirmed that increasing leaching temperature significantly increases nickel dissolution, with concentrations rising from 551.8 ppm at 50°C to 1536.17 ppm at 90°C. Iron, magnesium, and manganese also increased with temperature, indicating higher mineral breakdown. Cobalt remained below detection limits, possibly due to its low initial content, higher acid consumption by Mg, or entrapment in silicates[25]. A possible reaction is as follows:

$$(Mg,Ni,Co)_3Si_2O_5(OH)_4 + 3H_2SO_4 \rightarrow (3Mg^{2+}/Ni^{2+}/Co^{2+}) + 2H_4SiO_4 + 3SO_4^{2-} + H_2O \quad (6)$$

Figure 4 shows the distribution of Fe observed in the highlighted areas of the SEM images, consistent with the ICP-OES data, which shows a significant decrease in Fe concentration in the solution after pH adjustment. Meanwhile, Table 7 shows SEM-EDS results for samples taken from three parts of the solid that was adjusted to pH 4.5 using NH_4OH. Analyses show that each sample contains a considerable amount of iron in the residue. This clearly shows that Fe precipitation was present during pH adjustment.

The sequential leaching and precipitation of iron to pH 4.5 with NH_4OH efficiently removed iron but left nickel and cobalt in solution, as shown in Table 8. A substantial decrease in iron levels was observed across all samples, with the removal percentages at 90 °C reaching 94.27%, as indicated in Table 9. This has shown the preferential character of Fe precipitation at relatively high pH (>3), as Fe is prone to form insoluble hydroxide at pH >3[26]. In the meantime, nickel and cobalt stayed in solution. As shown in Table 10, the results after solvent extraction and stripping have low contents of Nickel and Cobalt.

Table 6: ICP-OES results of PLS leached at 50°C, 70°C, and 90°C

Elements	Temperatures (°C)		
	50 (ppm)	70 (ppm)	90 (ppm)
Ni	551.8	834.464	1536.17
Co	Under value	Under value	Under value
Fe	2274.37	3903.39	7670.88
Si	249.285	437.153	784.408
Mg	2470.86	2696.61	3089.79
Mn	67.333	100.617	181.621
Ca	225.432	204.148	231.154

Table 7: ICP-OES results of PLS leached at 50°C, 70°C, and 90°C

Elements	Temperatures (°C)		
	50 (wt%)	70 (wt%)	90 (wt%)
C	7.350	7.250	-
O	42.530	48.350	28.850
Mg	-	2.320	5.200
Al	1.100	2.320	1.450
Si	35.080	23.630	48.470
Fe	13.930	15.040	15.460
Ni	-	-	0.570

Table 8: ICP-OES results of PLS leached at 50°C, 70°C, and 90°C

Elements	Temperatures (°C)		
	50 (ppm)	70 (ppm)	90 (ppm)
Ni	196.333	295.294	525.162
Co	2.449	3.937	4.743
Fe	298.649	329.676	439.906
Si	54.816	62.828	56.951
Mg	1452.37	1634.49	1932.42
Mn	94.156	20.847	46.014
Ca	88.499	77.942	87.863

Table 9: Decreasing amount of Fe in each sample

Temperature in Each PLS (°C)	Decreasing Amount (%)
50	86.869
70	91.554
90	94.265

Table 10: ICP-OES results of sample after solvent extraction and stripping

Elements	Temperatures (°C)		
	50 (ppm)	70 (ppm)	90 (ppm)
Ni	1.666	1.736	5.186
Co	Under value	Under value	Under value
Fe	26.240	33.110	105.605
Si	13.841	14.429	13.913
Mg	4.503	5.956	31.370
Mn	Under value	Under value	Under value
Ca	Under value	Under value	Under value

4. Discussion

4.1. Roasting Discussion

The roasting process was conducted using a muffle furnace as the reduction medium. As the muffle furnace operated under non-inert conditions and did not control the oxygen atmosphere, reoxidation may occur during the cooling stage due to the presence of oxygen within the furnace environment[27]. This can hinder the reduction process and lead to lower overall extraction[28]. One example is the reduction of the goethite phase at 900 °C with a 15% coal ratio; magnetite is still present, but its peak intensity may be slightly lower than at 800 °C. This decrease is likely due to its partial reduction to metallic iron. Despite its persistence alongside metallic iron, it suggests an incomplete reduction by the non-inert atmosphere. A non-inert atmosphere might also lead to loss of Na_2SO_4[29]. The decomposition of Na_2SO_4 in the presence of carbon and oxygen can lead to the direct evolution of gaseous sulfur

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oxides. This indicates significant potential for sulfur loss from the system as SO gases in the muffle furnace, therefore reducing the amount of sulfur available for sulfidation reactions[30]. Inert furnaces such as tube furnaces can be utilized as a process improvement and potentially improve both nickel and cobalt content, as well as liberate them from silicate or other phases that could be a problem (trapping the contents) during the leaching process[31].

4.2. Leaching Discussion

The results of the leaching experiments at different temperatures (50°C, 70°C, and 90°C) showed an obvious trend: the higher the temperature, the more effective the extraction of nickel and cobalt from the saprolite-type laterite ore. This observation is consistent with basic knowledge of leaching kinetics, in which high temperatures catalyze reaction rates and diffusion, thereby increasing metal solubility[32]. These results are consistent with a previously published experiment, which showed that extraction becomes more efficient in atmospheric acid-leaching systems as temperature increases. Still, the extent of leaching is not as great as that observed with high-pressure extraction techniques such as HPAL[33].

After leaching, the pH was adjusted to 4.5 with 2 M NH_4OH, and a considerable amount of iron precipitated. Maximum Fe removal was observed at 90°C, with ~94.27%. This was a critical step that enhanced cobalt selectivity to solvent extraction. However, the percentage of Fe removed in this research was slightly lower than that reported by Rao et al., who nearly precipitated all iron under optimal conditions at higher temperatures[34]. The current results may be explained by the comparatively short precipitation period (15 minutes) and by the lack of high-temperature treatment during pH adjustment. Despite this, the overall ion selectivity for iron over nickel was high, due to the formation of insoluble hydroxides at pH > 3, whereas nickel and cobalt remain soluble in acidic sulfate solutions. Such precipitation played a key role in obtaining a cleaner leach solution during cobalt extraction. This was followed by solvent extraction, using Cyanex 272, TBP, and naphtha to selectively transfer cobalt to the organic phase and nickel to the aqueous phase. Such behavior is consistent with previous observations of the pH-dependent selectivity of Cyanex 272 for Co^{2+} [35]. However, the stripping step with 1 M H_2SO_4 did not yield satisfactory cobalt recovery, indicating that the stripping equilibrium was not adequately driven under the explored conditions. This result suggests the need to further optimize it, for instance, by increasing the acid concentration or using an alternative stripping agent, to complete the recovery cycle. Additionally, it should be noted that the primary difficulty of this study lies in the nature of the feed, which is a low-grade saprolite laterite ore. This fact essentially limits the achievable recovery efficiency compared to research studies that may use higher-grade feeds. Furthermore, the lack of literature directly addressing the use of Cyanex 272 to separate cobalt from a saprolite ore matrix indicates a research gap; thus, the results obtained in this study offer insights into the potential and challenges of this underexplored hydrometallurgical system.

5. Conclusion

A sequential treatment strategy of obtaining cobalt from nickel laterite saprolite-type ore was proposed in the present study: reduction roasting–sulphuric acid leaching–pH adjustment–solvent extraction. After roasting and acid leaching, the samples showed reduced impurity levels, and the extraction of nickel and cobalt from the leach solution was the focus of this work. At 700 °C, a 5% reduction in coal is sufficient to raise the cobalt grade to 0.188%, about 6 times that of the untreated ore, demonstrating the feasibility of the thermal activation process as a means of improving cobalt utilization, despite the encapsulation in silicates, presumably limiting its exploitation. Leaching with 2 M H_2SO_4, followed by pH adjustment to 4.5 with NH_4OH, enabled selective precipitation of iron, achieving up to 94.27% iron removal while keeping Ni and Co in solution, thereby improving the selectivity of the next extraction process. Cyanex 272 and TBP were used for solvent extraction using naphtha, and efficiently transferred cobalt into the organic phase. However, cobalt could not be stripped back into the aqueous phase under the conditions tested, highlighting the need for further optimization in the stripping process.

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Nomenclature

Symbol	Description
AAL	Atmospheric acid leaching
Al_2O_3	Alumina
CaO	Calcium oxide
Co	Cobalt
Co^{2+}	Cobalt ion in 2+ oxidation state
Cr_2O_3	Chromium (III) oxide
Fe_2O_3	Hematite
Fe-Ni	Ferronickel
H_2SO_4	Sulphuric acid
HPAL	High pressure acid leaching
ICP-OES	Inductively coupled plasma–optical emission spectroscopy
kt/yr	Kilotones per year
Mg	Magnesium

MgO	Magnesium oxide
Mn	Manganese
Na_2SiO	Sodium silicate
Na_2SO_4	Sodium sulfate
NH_4OH	Ammonium hydroxide
Ni	Nickel
^oC	Degree celcius
ppm	Parts per million
PLS	Pregnant leach solution
rpm	Revolutions per minute
SEM-EDS	Scanning Electron Microscopy with Energy Dispersive Spectroscopy
SiO_2	Quartz
SO	Sulfur oxide
S/L	Solid to liquid ratio
TBP	Tributyl phosphate
v/v	Volume/volume percentage
wt%	Weight percent
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

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