

Atmospheric Leaching Behavior and Kinetics of Nickel and Cobalt from Halmahera Limonite Ore

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Abstract. Halmahera Island in Indonesia holds great potential for nickel laterite due to its geological setting. As the product of chemical weathering, nickel laterite can be divided into saprolite and limonite. Despite containing invaluable elements like nickel and cobalt, limonite ore is seldom used in metal production. This study evaluates these two elements' leaching behavior and kinetics from the Halmahera deposit. Limonite ore was leached using 50 mL of 0.5 M sulfuric acid at an S/L ratio of 10% and 20%. The leaching process was conducted at 30°C, 50°C, and 80°C. The experimental results showed that the highest recovery for cobalt and nickel was 100% in the adequate presence of H⁺ ions. The result shows that sulfuric acid is more selective to cobalt than a nickel. The shrinking core ash diffusion model represents the leaching kinetics of cobalt, while nickel recovery follows the Kröger-Ziegler kinetics model.

Keywords: Cobalt; Kinetics; Leaching; Limonite; Nickel

1. Introduction

Natural Nickel laterite is a supergene nickel deposit formed through the chemical weathering of olivine-rich peridotite rocks under hot and humid climate conditions in tropical or subtropical zones (Xiao *et al.*, 2020; Ito *et al.*, 2021). The most crucial deposit is found around the equator, especially in locations where tectonically active plates are in contact (Van der Ent *et al.*, 2013). Given the geological setting, Indonesia owns abundant nickel deposits and is the major nickel producer globally (Ilyas, Kashiwaya, and Koike, 2016). One of the regions rich in nickel laterite is Halmahera Island, located at the Eurasian, Australian, and Philippine Sea plates (Farrokhpay *et al.*, 2019). Four laterite areas on this island are estimated to hold 277 million tons of resources (Farrokhpay *et al.*, 2019). Laterite

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accounts for 70% of the world's nickel reserve (Hosseini Nasab, Noaparast, and Abdollahi, 2020). However, only 40% of the resource has been used to fulfill global nickel demand (Botelho Junior *et al.*, 2018).

Nickel is an essential metal with good corrosion resistance, high strength, and plasticity (Ning *et al.*, 2019; Mohammadreza, Mohammad, and Ziaeddin, 2014). This metal is commonly made into alloy and stainless steel and is used in many applications, such as batteries, catalysts, and electroplating industries (Sun *et al.*, 2020; Hosseini *et al.*, 2017). Nickel is the crucial element for stainless steel, which consumes around two-thirds of its production (Xiao *et al.*, 2020). This element is also used in battery industries to enhance storage capacity and thermal stability (Purwanto *et al.*, 2021). More nickel is recovered from saprolite, the lower layer of laterite deposit, which possesses a nickel concentration of 1.5% to 3% (Elliott, Pickles, and Forster, 2016). Meanwhile, with 0.5% to 1.7% nickel, limonite is often not used in production and is discarded in dumping grounds (Sufriadin *et al.*, 2020; Elliott, Pickles, and Forster, 2016).

Despite its low nickel content, limonite also contains valuable elements like cobalt, Platinum Group Metals (PGM), rare earth elements, and scandium (Tupaz *et al.*, 2020). Cobalt is significantly enriched in the limonitic zone due to its low mobility (Sagapoa, Imai, and Watanabe, 2011). On average, limonite ore carries about 0.10% of cobalt (Li *et al.*, 2010). This element is also used mainly in battery production to boost conductivity and prevent mixing between nickel and lithium-ion (Gourley, Or, and Chen, 2020).

Along with the increasing industrial demand for nickel and cobalt and the depletion of other resources like nickel sulfide, there is a need for processing limonite ore (Liu *et al.*, 2019; Sverdrup and Olafsdottir, 2019). The hydrometallurgy process can recover desirable elements (Sufriadin *et al.*, 2020; Wanta *et al.*, 2020; Wanta, Perdana, and Petrus, 2016). This study uses sulphuric acid to leach nickel and cobalt from Halmahera limonite ore at the atmospheric condition in which this kind of acid is commonly used for mineral leaching, i.e., vanadium from slag (Aarabi-Karasgania *et al.*, 2010), rare earth element from tailing zircon (Trisnawati *et al.*, 2020), zinc and copper from brass slag (Ahmed, Nayl, and Daoud, 2016), rare earth element from silica sand and low-grade bauxite (Kusrini *et al.*, 2020a; 2020b; 2019). According to several studies, sulfuric acid is a solvent that can provide good leaching results. It is because the dissociation of hydrogen (H^+) ions can take place completely so that the H^+ ions can be produced in greater quantities than other kinds of acids. Although some other acids, not only inorganic acid but also organic acid that is believed to be more environmentally friendly, have been studied, such as rare earth element extraction using citric acid (Prihutami *et al.*, 2021), lithium and cobalt extraction from the spent battery using acetic acid (Setiawan, Petrus, and Perdana, 2019), the kinetics of sulfuric acid leaching on Halmahera limonite ore at atmospheric condition has never been studied.

Kinetic studies are the most important part of the design process of a processing unit. By studying the kinetics of this process, the physical phenomena in a deep process are known. Thus, the process of designing and scaling up a unit can be carried out according to the most real phenomena, and design errors can be minimized. Matching the most appropriate kinetics model from a shrinking core model (Mirwan *et al.*, 2017) to the newest product diffusion developed model (Wanta *et al.*, 2022; Prihutami *et al.*, 2022), will be the objective of this study. Thus, the kinetics parameter needed for scale-up can be generated for sulfuric acid leaching on Halmahera limonite at atmospheric conditions.

2. Methods

2.1. Materials

The limonitic laterite ore used in this study was from a mining area on Halmahera

Island, Indonesia. A representative sample was obtained by coning and quartering. The dried sample was crushed, ground, ball milled, and sieved until the average size of the sample was $< 74 \mu\text{m}$. The chemical components of the selected representative sample were determined using X-ray Fluorescence (XRF, Epsilon 3XLE PANalytical). X-ray Diffraction (XRD, X'Pert 3 Powder from PANalytical, Netherlands) was also used to identify the crystalline materials in limonite ore. The morphology and elemental map of the sample were analyzed using FE-SEM/EDS Thermo Scientific Quattro S. Pregnant leached solution in this study was prepared by diluting pro analytical sulfuric acid (CAS RN of 7664-93-9, Merck, Germany) in aquadest.

2.2. Methods

Halmahera limonite ore was put into an Erlenmeyer, and 50 mL of 0.5 M sulfuric acid was added. The atmospheric leaching was done for 4 hours in an orbital shaker, as shown in Figure 1. The shaker speed was kept constant at 200 rpm, while the S/L ratio and leaching temperature varied from 30 to 80°C. The concentration of leached nickel and cobalt at any designated time (15 minutes, 30 minutes, an hour, 2 hours, and 4 hours) was determined using ICP-Optical Emission Spectrometer PQ 9000 from Analytik Jena AG, Germany.

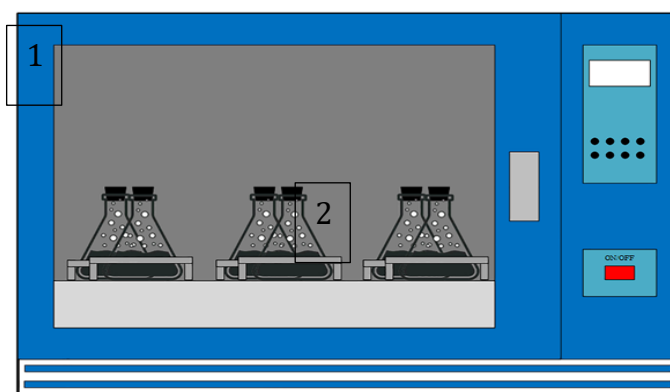


Figure 1 The experimental apparatus for nickel and cobalt leaching: (1) orbital shaker; (2) Erlenmeyer flask

Nickel recovery was calculated using Equation 1, while Equation 2 was used to calculate cobalt recovery.

$$\%Ni = \frac{2,500 \times C_{Ni} \times V}{m \times X_{Ni}} \times 100\% \quad (1)$$

$$\%Co = \frac{1,000 \times C_{Co} \times V}{m \times X_{Co}} \times 100\% \quad (2)$$

where C is the element concentration in the liquid sample (g/mL), V is the volume of sulfuric acid solution (50 mL), X is the element concentration in ore, and m is the mass of the limonite ore (g).

The leaching kinetics of nickel and cobalt was determined using either the Kröger-Ziegler model (Equation 3) or the ash diffusion controls model (Equation 4); (Wanta, Perdana, and Petrus, 2016).

$$k_K \ln t = \left(1 - (1 - \alpha)^{\frac{1}{3}}\right)^2 \quad (3)$$

$$k_D t = 1 - 3(1 - \alpha)^{\frac{2}{3}} + 2(1 - \alpha) \quad (4)$$

k_k is the leaching rate constant for the Kröger-Ziegler model, t is the leaching time (min), α is the recovery value of either nickel or cobalt, and k_D is the leaching rate constant for the ash diffusion controls model.

3. Results and Discussion

3.1. Ore Characterization

The nickel content in the limonite ore from Halmahera Island, Indonesia, is 1.73%. The ore is considered middle grade, with the element content between 1.2% and 1.8% (Cao, Xue, and Duan, 2016). According to the diffractogram (Figure 2), nickel minerals are not present in the ore. Instead, limonite ore is composed mainly of goethite (α -FeOOH), with minor lizardite ($Mg_3Si_2O_5(OH)_4$) and talc ($Mg_3Si_4O_{10}(OH)_2$). Nickel is most likely hosted in goethite, indicated by the coincidence of Ni and Fe in the elemental map (Figure 3). Generally, nickel is associated with goethite through surface adsorption or Fe substitution (Butt and Cluzel, 2013). The EDS results also project the mapping of Mg, the major constituent of talc and lizardite, proving the existence of both minerals in the ore (Ersoy *et al.*, 2013; Carmignano *et al.*, 2020).

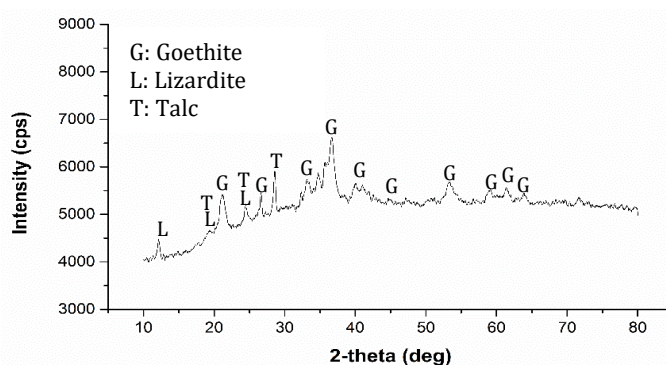


Figure 2 XRD Pattern of Limonite Ore Sample

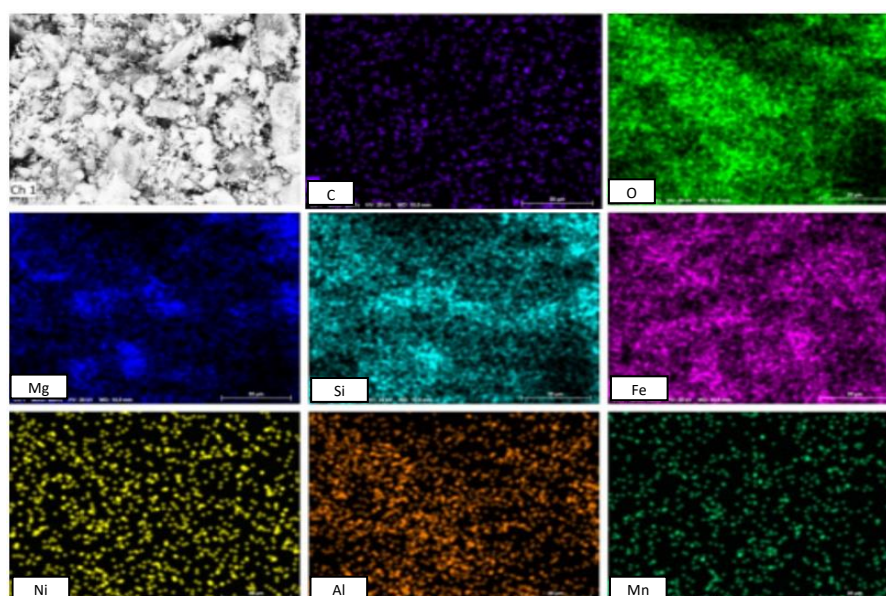


Figure 3 Elemental Mapping of Limonite Ore: Cr (dark blue); O (light green); Mg (blue); Si (cyan); Fe (purple); Ni (yellow); Al (orange); Mn (green)

XRF analysis shows that Halmahera limonite ore contains 0.12% of cobalt. This element is mainly carried by Mn-oxyhydroxides, like asbolane and lithiophorite-asbolane

(Tupaz *et al.*, 2020; Sagapoa, Imai, and Watanabe, 2011). These minerals have poor crystallinity and thus hardly appear on diffractograms (Maciąg *et al.*, 2019; Burlet and Vanbrabant, 2015).

However, the XRF result indicates the presence of manganese oxides, which undergo coprecipitation with cobalt and other elements, such as Ni (1.73 wt%), Co (0.12 wt%), Fe (34.12 wt%), Mn (0.99 wt%), Si (3.46 wt%), and Al (1.46 wt%). Aside from Mn-oxyhydroxides, cobalt is also hosted in lizardite and some in goethite (Sufriadin *et al.*, 2020; Tang and Valix, 2004). FE-SEM/EDS analysis was also used to confirm the XRF data. FE-SEM/EDS result indicates the presence of Ni (1.72 wt%), Fe (26.17 wt%), Mg (10.80 wt%), Al (1.39 wt%), Cr (0.37 wt%), Mn (0.47 wt%), and Si (9.86 wt%) (Astuti *et al.*, 2021).

3.2. Nickel and Cobalt Leaching

The leaching process of nickel and cobalt from Halmahera limonite ore by sulfuric acid was done at 30°C, 50°C, and 80°C. Along with the increase in temperature, the recovery of both elements also increases. Higher temperature provides greater kinetic energy, which allows rapid molecular movement. The fast movement accelerates molecular diffusion and surface reaction. This trend is the same at an S/L ratio of 10% (Figure 4) and 20% (Figure 5).

Unlike temperature, the S/L ratio is inversely proportional to nickel and cobalt recovery. At 10% of the S/L ratio, sulfuric acid can recover 100% of cobalt after an hour. Meanwhile, the same value is achieved by nickel after 4 hours at 80°C. Complete recoveries are possible since there are adequate hydrogen ions (H⁺) to leach both elements from their carrier. However, a distinct case is found using a higher S/L ratio. Loading more solids into the system raises the number of leachable materials. However, the amount of H⁺ is insufficient to recover all available nickel and cobalt. At this point, leached solution selectively reacts with the host mineral that is easier to dissolve.

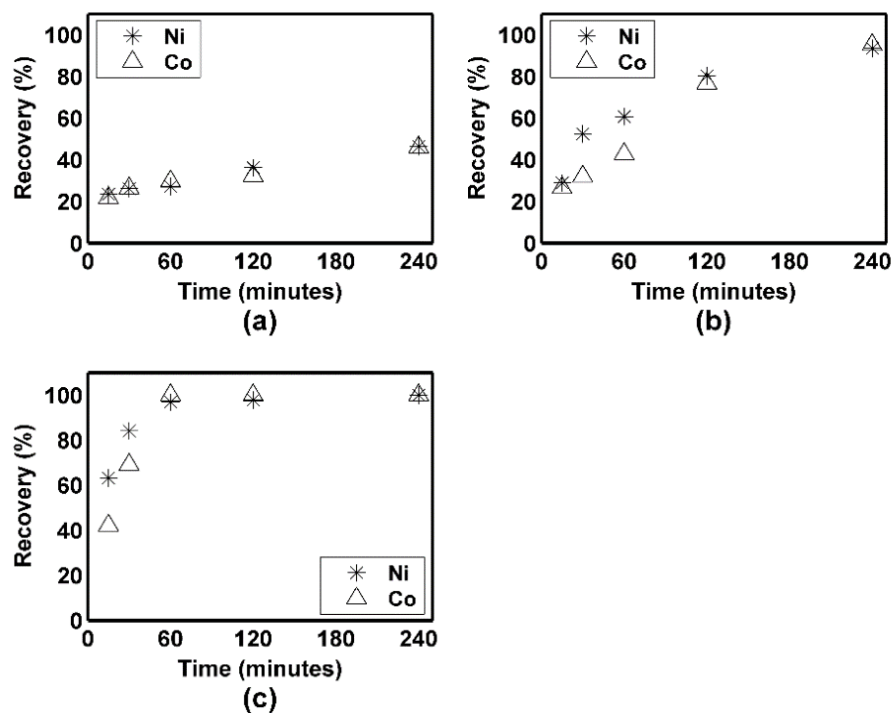


Figure 4 Nickel and Cobalt Recovery using an S/L Ratio of 10% at a) 30°C, b) 50°C, and c) 80°C

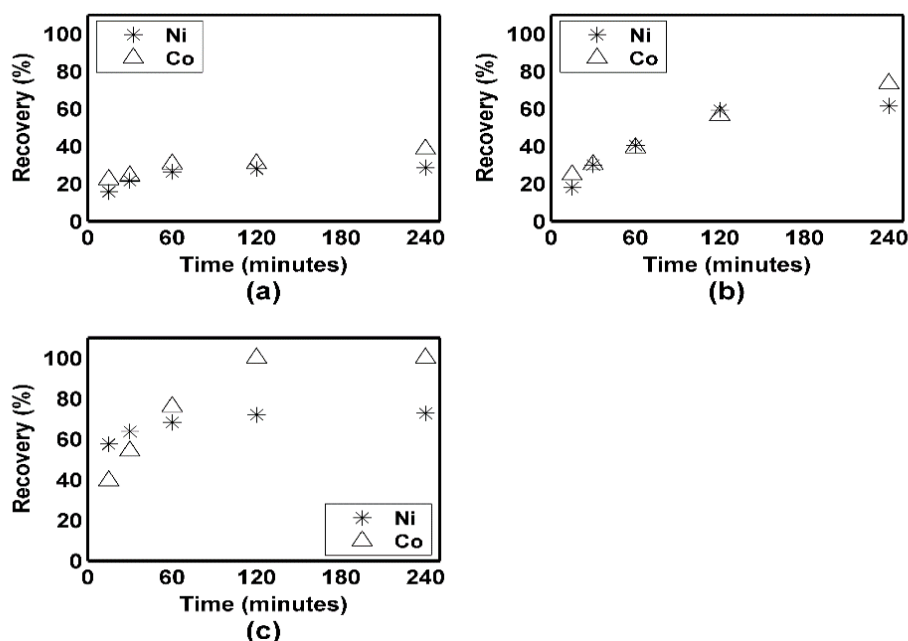
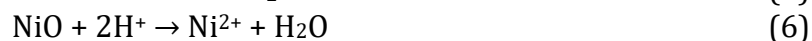


Figure 5 Nickel and Cobalt Recovery using an S/L Ratio of 20% at a) 30°C, b) 50°C, and c) 80°C

Using 20% of the S/L ratio at any temperature, the leaching process shows that cobalt recovery increases proportionally with time. Meanwhile, the recovery of nickel initially surges but slows down after some time. At 80°C, the leaching of nickel does not surpass 72.72% even after 4 hours, while cobalt achieves complete recovery only after 2 hours. This phenomenon indicates that sulfuric acid dissolves cobalt-rich minerals, like lizardite and manganese oxide, more quickly than nickel-rich minerals (Astuti *et al.*, 2016). As goethite is the main component in Halmahera limonite ore, a great amount of acid will be needed to dissolve said mineral and recover nickel, according to Equations 5 and 6 (Senanayake *et al.*, 2011).



3.3. Leaching Kinetics

Knowing the leaching kinetics of elements will be useful for the industrialization process. In this study, leaching kinetics are evaluated using a high S/L ratio. It is assumed that the rate-controlling step would be diffusional mass transfer through an ash layer. According to the data processing result, the recovery of cobalt and nickel from Halmahera limonite ore follows this assumption. However, these two elements have a slightly different phenomena during the leaching process.

The cobalt leaching is seen in Figure 6a to fit the shrinking core model. Experiments at three different temperatures result in R^2 values close to 1. The leaching rate constants increase around seven times from 30°C to 50°C and from 50°C to 80°C, indicating that high temperature favors cobalt recovery. On the other hand, nickel leaching kinetics suits the Kröger-Ziegler model with all R^2 values above 0.92 (Figure 6b). For all temperatures, the leaching of nickel is seen to approach equilibrium.

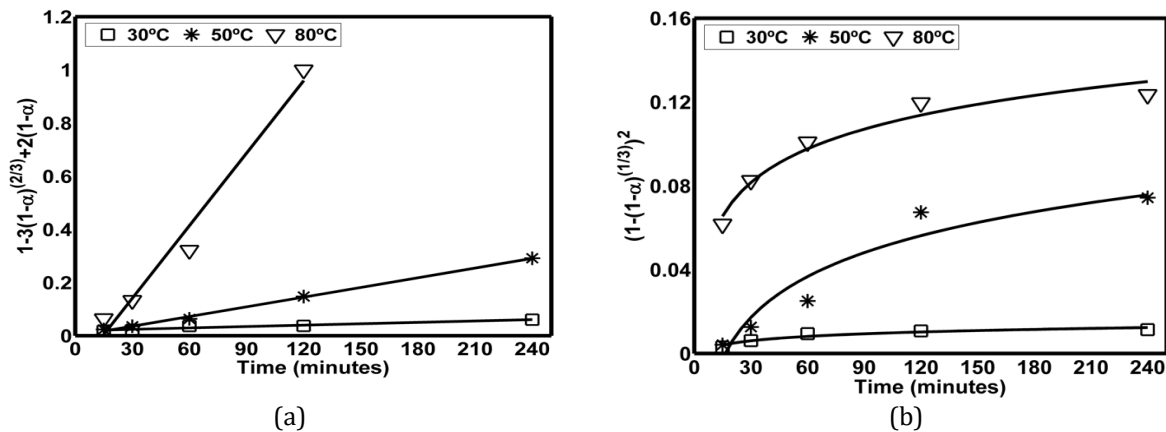


Figure 6 (a) Shrinking Core Ash Diffusion Model for Cobalt Leaching at Various Temperatures, and (b) Kröger-Ziegler Model for Nickel Leaching at Various Temperature

One thing that differentiates the shrinking core model from the Kröger-Ziegler model is that its effective diffusion coefficient is assumed to be constant (Levenspiel, 1999). In the Kröger-Ziegler model, the effective diffusion coefficient is inversely correlated with leaching time (Prihutami *et al.*, 2021). In a condition that lacks acid, the leaching of limonite leaves behind unreacted goethite (Sahu, Kavuri, and Kundu, 2011). This ash layer thickens with time, hinders molecular diffusion, and lowers nickel's diffusion coefficient, which mainly exists in goethite. This phenomenon does not happen to cobalt as it is mainly hosted in much more leachable minerals.

Table 1 Kinetics Model Parameters

Temperature (°C)	Cobalt		Nickel	
	k_D (min ⁻¹)	R ²	k_K (min ⁻¹)	R ²
30	1.74×10^{-4}	0.9323	3.04×10^{-3}	0.9232
50	1.21×10^{-3}	0.9977	2.81×10^{-2}	0.9215
80	9.10×10^{-3}	0.9748	2.32×10^{-2}	0.9630

4. Conclusions

In this work, temperature positively affects nickel and cobalt recovery, unlike the S/L ratio. At a high S/L ratio, the system lacks H⁺ activity, and sulfuric acid selectively leaches more cobalt than a nickel. Due to the formation of an ash layer (unreacted goethite), the leaching kinetics is controlled by molecular diffusion through the ash layer. However, the host mineral greatly determines the leaching phenomenon. Thus, cobalt and nickel have two different kinetics models. The shrinking core model suits cobalt leaching well, while the Kröger-Ziegler model best describes the nickel leaching phenomenon. Furthermore, in future work, the mathematical models will be explored further so that kinetic information which describes the actual physical phenomena of this leaching process can be obtained. The model can then be applied to the design extractor.

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