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The Extraction of Nickel from Morowali Laterite Ore with Nitric Acid

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Abstract

Nickel extraction from Morowali laterite ore has been carried out with nitric acid as the solvent. This study aims to determine the effect of nitric acid concentration, temperature, and stirring time on nickel content in laterite ore originating from Morowali. Morowali laterite seeds were extracted using the leaching method using nitric acid as the solvent with various concentrations of 0.5, 1, and 2 M, temperatures of 30, 60, and 95 °C, and stirring times of 3, 6, and 12 hours. Taguchi analysis was used in this study to obtain the effect of the three parameters used. The results showed that the optimum conditions were obtained at a concentration of 2 molars nitric acid, a temperature of 95 °C, and a stirring time of 12 hours, with a nickel content of 16.469 ppm and a nickel recovery percent of 3.88%.

Keywords: Extraction, nickel, laterite ore, leaching, nitric acid

Introduction

Nickel is one of the essential elements of human life. This metal element is widely used because it has good corrosion resistance and is easy to shape but remains strong. Nickel is a vital and strategic metal. This metal is one of the primary raw materials in manufacturing stainless steel, special steel, catalysts, and batteries (Barkas, 2011). In addition, this metal also plays a vital role in several hard metal deposition processes in the form of alloys such as stainless steel.

Nickel is primarily found in the form of laterite minerals; about 60% of the world's nickel resources are available in nickel oxide, or nickel laterite (Marrero et al., 2015), while the remaining 40% is in the form of sulfide deposits. In general, laterite ores are classified into two types: limonite and saprolite (Tong et al., 2013). Saprolite ore has a nickel content of 1.5-3.0% and limonite ore of 1.0-1.5% (Thubakgale et al., 2013). Nickel ore deposits are abundant in eastern Indonesia around Sulawesi, Maluku, and parts of the island of Papua (Prasetiyo, 2008). Laterite nickel deposits in Central Sulawesi can be found in Morowali Regency and Luwuk Banggai Regency.

The challenges faced in processing mineral ores into high-purity chemicals include many components in mineral ores, making it difficult for the separation and purification process, such as cobalt and iron mixed in laterite ore. The processing of high-grade nickel ore is processed through a high-temperature process (pyrometallurgy). At the same time, hydrometallurgical methods generally carry out nickel extraction from low-grade laterite nickel ore; the hydrometallurgical process is a mineral processing process carried out at relatively low temperatures by leaching using chemical solutions. In contrast, the pyrometallurgical process is a mineral processing process carried out at high temperatures (Kyle, 2010). The pyrometallurgical process also causes environmental problems, such as air pollution due to using high temperatures. In addition, this process also requires high energy and high costs to run (Listyarini, 2017; Simate et al., 2010).

The hydrometallurgical process has the advantage of producing a much purer product than the pyrometallurgical process (Wanta et al., 2018). This technology is usually used for processing low-grade nickel ore; the outcome of this processing is nickel (Ni) (Prasetiyo, 2016).

Mubarok & Fathoni (2016) and Astuti et al. (2016) explained that theoretically, the higher the temperature, the more nickel will be obtained. In an experiment conducted by Purwanto (2003) and Wanta et al. (2018), it was also stated that the concentration of the solution used in the leaching process could affect the product produced.

The Taguchi analysis method is also used to see the effect of the parameter set in the extraction process. Nitric acid is used as a reactant in the nickel dissolution process in laterite ores. This study analyzes the effect of concentration, temperature, and stirring time on separating nickel from metal impurities using the leaching method to determine

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efficient operating conditions to produce a high % nickel extraction.

This paper is intended to describe the extraction process of laterite ore with HNO_3 using variable parameters.

Methods

The equipment used is oven, mortar, and alum, Whatman (hardened) filter paper numbers 52 and 54, Funnel, Buchner, Erlenmeyer filter flask, Vacuum pump, Filler (suction rubber), 100 mL chemical cup, magnetic stirrer, petri dish, thermometer, digital balance, measuring cup, volumetric flask, and AAS (GBC 923AA). The material used is laterite ore from Central Sulawesi Province, Morowali Regency, Bahodopi District, and nitric acid (*Merck*). All the laterite ore analyses were performed in the Laboratorium TekMIRA, Bandung Indonesia including an X-Ray Diffractometer (*Shimadzu*) to record the diffractogram of the sample, and X-Ray Fluorescence (XRF) Rigaku ZXS Primus IV to determine the elemental composition of the ore.

The laterite ore was then weighed as much as 1000 g using a digital balance; then the ore was put in an oven at 110 ºC for 3 hours; this was done to remove the water content, then weighed again. After drying, the laterite ore was ground using a mortar and pestle, then sieved using a 200 mesh sieve. The refined laterite samples were reweighed using a digital balance.

Extraction of laterite ore using HNO_3 solution with various concentration

Two grams of fine laterite ore were weighed, put into a 1000 mL beaker, and added to 100 mL of nitric acid solution 0.5 M. Cover the surface of the glass tightly with aluminum foil so that the mixture does not evaporate. Then heat the mixture on a hot plate at 30 °C and stir using a magnetic stirrer for 3 hours. After that, the mixture was filtered using a Buchner funnel lined with filter paper and analyzed for nickel content in the filtrate using AAS. Then the same steps with variations in temperature of 60 ºC and 95 ºC and variations in leaching time of 6 hours and 12 hours.

Perform the same steps as above by changing the concentration of the nitric acid solution to 1 M and 2 M.

Preparation of a standard nickel solution of 100 ppm

Nickel nitrate $Ni(NO_3)_2.6H_2O$ was weighed as much as 0.49 grams. The nickel nitrate solid was input into a 1000 mL volumetric flask, then dissolved in 50 mL HNO_3, pour distilled water into it. In a volumetric flask up to the mark and homogenize.

Results and Discussion

The results obtained from this study are data regarding the analysis of nickel content in laterite ore on the concentration of a nitric acid solution, temperature, and nitric acid leaching time by the Taguchi method, in this study using three parameters, each of which has three different levels with the aim of reducing the number of experiments and high costs.

Table 1. Level parameters

No.	Parameters	Level I	Level II	Level III
1.	Concentration (M)	0.5	1	2
2.	Temperature (°C)	30	60	95
3.	Leaching time (hours)	3	6	12

The optimum parameter analysis result

The optimum condition was determined by using the Taguchi method (Taguchi & Clausing, 1990) by combining the optimal parameters. The S/N ratio (*Signal to Noise*) determines the factors affecting the experimental results. The results obtained for the optimum conditions were a concentration of 2 molars, a temperature of 95 °C, and a stirring or leaching time of 12 hours, which can be seen in **Figure 1**.

Data result of calculation of percent contribution

The data from the ANOVA (Analysis of Variance) conducted on the sample showed that the most significant contribution to give effect was the temperature of 44.94%, then the concentration of 10.31%, and the leaching time of 8.66%. The purpose of the ANOVA test is to determine whether the factors affect the desired results, which can be seen from the percentage contribution of each element to the total variability. The three parameters were used to contribute to the extraction or dissolution of nickel in laterite ore using nitric acid.

Table 2. Response table for the signal-to-noise ration

Level	Concentration (M)	Temperature (°C)	Leaching time (Hours)
1	22.52	21.30	22.16
2	23.31	23.84	23.84
3	24.28	24.97	24.11
Delta	1.76	3.67	1.95
Rank	3	1	2

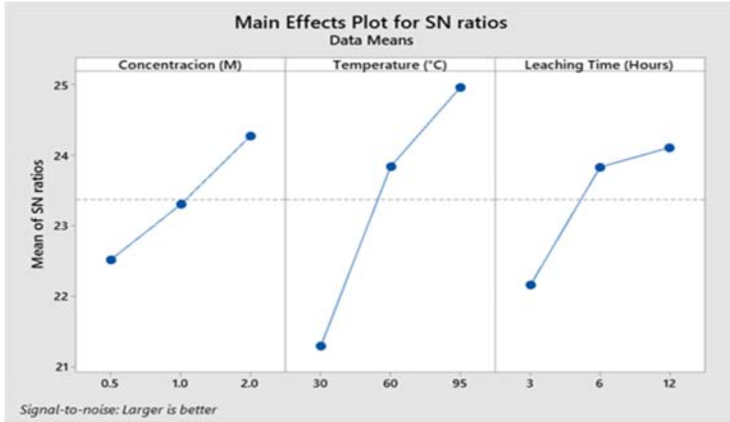

Figure 1. The results of the analysis using the Taguchi method for optimum conditions

Table 3. Analysis of Variance

Source	DF	Seq SS	Contribution (%)	Adj SS	Adj MS	F-Value	P-Value
Concentration (M)	2	36.09	10.31	36.09	18.043	2.86	0.081
Temperature (°C)	2	157.30	44.94	157.30	78.648	12.45	0.000
Leaching Time (Hours)	2	30.29	8.66	30.29	15.147	2.40	0.117
Error	20	126.33	36.09	126.33	6.316		
Total	26	350.01	100.00				

Characteristics of laterite ore samples

The characteristics of the laterite ore samples in this study were of the limonite taken from the Bahodopi District, Morowali Regency, Central Sulawesi Province, which had reddish-brown features. Laterite ore samples were analyzed using X-ray diffraction (XRD). The results of XRD analysis found that the mineral composition of laterite ore consisted of Trevorite, Clinochlore, Lizardite, Talc, Magnetite, Enstatite, Riebeckite, and Goethite. The purpose of this XRD analysis is to determine the compounds contained in laterite ore samples.

Sample testing using X-ray Fluorescence (XRF) was also carried out to determine the metal elements in this laterite ore quantitatively. The results of sample testing using XRF show that laterite rock ore is dominated by iron (Fe) at 46.24%, while for nickel (Ni), it is only 2.12%. The result shows that laterite ore has a higher iron content than its nickel content.

Laterite ore preparation

The limonite laterite ore used contains water on the ore surface. Before the experiment, the laterite ore was dried in an oven for 3 hours at a temperature of 110 °C. The purpose of the drying is to reduce the water content in the laterite ore, then the limonite nickel ore is ground and enriched to obtain ore with a size fraction of 200 mesh. The water content on the surface of limonite nickel ore is determined by calculating the difference in weight of limonite nickel ore before and after being put into the drying oven; this aims to determine what percentage of water content is contained in laterite ore.

Table 4. Laterite ore XRF analysis results

No.	Sign		Laterite Ore
1.	SiO_2	%	23.31
2.	Al_2O_3	%	3.95
3.	Fe_2O_3	%	46.24
4.	MgO	%	9.99
5.	Cr_2O_3	%	2.04
6.	NiO	%	2.12

Effect of nitric acid concentration on nickel levels

Acid concentration is one of the factors that affect the leaching process. In this study, the concentrations of nitric acid used were 0.5, 1, and 2 M. The analysis results using the Taguchi method that has been carried out show that the percentage recovery of nickel content increases with increasing acid concentration. Where the higher the concentration of nickel content increases. Nitric acid was used for the higher nickel content obtained. It can be seen from the SN (*Signal to Noise*) value in **Table 2**; at a concentration of 0.5 M, the result is 22.52, then the results continue to increase at a concentration of 1 M by 23.31 and the highest nickel yield at a concentration of 2 M is 24.28. Thus, the most optimum nickel leaching ratio is at a concentration of 2 M. The results obtained are influenced by several factors, one of which is the pH value, where the acidity of a solution is more significant. , then the solution is better for use in the metal ion leaching process; this is because the solution is more acid (pH is getting smaller) and tends to have more H^+ to dissolve nickel into $Ni(OH_3)_2$ as the aqueous phase (*aq*) (Lintjewas et al., 2019).

$$NiFe_2O_{4(s)} + 2H = Ni^+ + Fe_2O_{3(s)} + H_2O_{(l)} \quad (1)$$
$$Ni_{(s)} + 2H^+ = Ni^{2+} + H_2O_{(l)} \quad (2)$$
$$2Ni.SiO_{2(s)} + 4H^+ = 2Ni^{2+} + SiO_{2(s)} + H_2O_{(l)} \quad (3)$$
$$NiO_{(s)} + 2HNO_{3(aq)} = Ni(NO_3)_{2(aq)} + H_2O_{(l)} \quad (4)$$

Taguchi's analysis in experiments on nitric acid concentration levels showed that the higher the concentration, the higher the SN value. This phenomenon is related to the mechanism of the chemical reaction that occurs; the higher the concentration of nitric acid causes, the number of hydrogen ions (H^+) formed in the acid dissociation stage also, increase the number of H^+ resulting in an increase in activity at the proton attack stage (Equation 2). The H^+ reacts with the NiO compound contained in the nickel laterite sample. The more H^+ that responds, the more nickel(II) (Ni^{2+}) ions formed due to the proton attack reaction will also increase. Thus, the formation of nickel nitrate products $Ni(NO_3)_2$ will also increase (Wanta et al., 2017).

Effect of temperature on nickel levels

Temperature is an important parameter that considerably influences nickel extraction (Luo et al., 2010). Measurements at three temperature levels are 30, 60, and 95 °C to measure the effect of leaching temperature on nickel extraction. The results of the experiments carried out show that the acquisition of nickel content continues to increase with increasing temperature; this can be seen in **Table 2**, where the results of the addition of the SN (*Signal to Noise*) value at a temperature of 30 °C are 21.30 and then continue to increase at a temperature of 60 °C, of 23.84. The highest nickel content was obtained at 95 °C at 24.97. It shows that the higher the temperature, the more nickel can be extracted from laterite ore (Fathoni & Mubarok, 2015). It can happen because the increased operating temperature causes the energy between molecules to be higher, so the frequency of collisions between molecules will increase. This increase in the frequency of crashes causes the reaction for the formation of nickel nitrate products also to be greater (Astuti et al., 2016).

Temperature is very influential on the leaching reaction kinetics; the results of this study prove that the leaching temperature conditions greatly determine the extraction results, where the higher the operating temperature used, the percentage of nickel extraction will also increase.

Temperature also affects the leaching reaction kinetics; based on the Arrhenius equation, the higher the temperature, the higher the reaction rate constant. The higher the temperature will cause the movement of each molecule to be faster, and the possibility of the molecules colliding and reacting will also be more significant, so the opportunities for nickel leaching will be more excellent (Gustiana, 2018).

Effect of leaching time (stirring) on nickel levels

The leaching time also influences stages of the laterite ore leaching process. The length of the leaching process or leaching that takes place is one factor that plays an essential role in optimizing nickel collection in the ore laterite. Variations in stirring time for 3 hours, 6 hours, and 12 hours were carried out to determine the effect of leaching time on nickel collection.

The results can be seen in Figure 1, which shows that the recovery of nickel content increases with increasing stirring or leaching time in nickel extraction. The more extended the leaching time used, the higher the nickel content obtained; this can be seen from the acquisition of SN values. (*Signal to Noise*) in **Table 2**, where the leaching time for 3 hours is 22.16, then it continues to increase at the 6-hour leaching time of 23.84, and the highest nickel content value is obtained at the 12-hour leaching time with a value of 24.11. It also happened from previous research conducted by Wahab et al. (2021), where the optimum or highest nickel leaching time obtained was 3 hours, from the variation of time used, namely 2 hours, 2.5 hours, and 3 hours. The nickel extraction yield also increased with increasing stirring time.

It happens because the longer stirring time causes the contact between the sample and the solvent to last longer so that the solubility increases; this indicates that with increasing time, the connection between the nickel laterite sample and the solvent in the leaching process becomes more optimum. As a result, the more optimal the contact of the sample with the solvent, the better the nickel metal-binding reaction by the solvent (Permana et al., 2020).

Conclusion

The optimum conditions for extracting nickel from Morowali laterite ore using nitric acid were obtained at a concentration of 2 molar nitric acids, a temperature of 95 °C, and a leaching or stirring time of 12 hours, with a nickel content obtained of 16.469 ppm, and a nickel recovery percent of 3.88%.

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