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Nickel Extraction from Morowali Laterite Ore with Chloride Acid (HCl) – Nitric Acid (HNO_3) Solution

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Abstract

Nickel is a metal of high strategic value for human needs to make stainless steel, batteries, and other products. The amount of nickel reserves in the world is about 72 % in rock oxides, and the rest is in sulfide rocks. However, only about 42 % of total nickel production in the world comes from laterite ore, while nickel sulfide is mainly produced. Research was conducted to determine the optimum nickel extraction parameters in a mixture of nitric acid and hydrochloric acid, and to determine the effect of the solvent volume ratio, temperature, and leaching time parameters on the nickel leaching results from laterite samples. This study uses the taguchi¬larger the better method with each factor having three levels of conditions, namely the volume ratio of the solvent using variations of 1: 1, 1: 2, and 2: 1 then variations in temperature of 30 °C, 60 °C, and 90 °C as well as variations in the leaching time of 3 hours, 6 hours, and 12 hours. The optimum conditions for nickel extraction are a parameter ratio of 1:1, a temperature of 90 °C, and a leaching time of 6 hours. The results of atomic absorption spectrophotometer analysis showed that the concentration of Ni^{2+} *at optimum conditions was 10.7895 ppm, and the recovery value was 2.54 %.*

Keywords: Extraction, nickel, hydrochloric acid-nitric acid solution, Taguchi

Introduction

Nickel laterite is a metallic mineral from the weathering of ultramafic rocks and the mineral enrichment process (Arifin et al., 2015). In nature, nickel is found in the form of sulfide and oxide rocks commonly called laterite. The total amount of nickel reserves in the world is about 72 %, which is in oxide rocks, and the rest is in sulfide rocks. However, only about 42 % of the world's total nickel production is sourced from laterite ores, while nickel sulfide is produced more (Purnawan et al., 2022).

The need for mineral-based chemicals at home and abroad is huge to support research processes, laboratories, industry, pharmaceuticals, stainless metal manufacturing, mixtures in stainless steel manufacturing, nickel-metal hybrid batteries, the aircraft industry, coin making, weapons plating, and various other functions (Sampath et al., 2023). This need also makes nickel very valuable and has a high selling value in the world market. At least since 1950, the demand for nickel has increased by an average of 4 % per year, and it is estimated that it will continue to increase in the next ten years (Stanković et al., 2020).

Sulfide ore types are primarily found in parts of the subtropical hemisphere, while oxide ores or laterite ores are more commonly found in the tropical hemisphere. As a tropical country, Indonesia has the world's third-largest nickel reserves in the form of laterite ore after New Caledonia and the Philippines (Chaerun et al., 2023). The issuance of the Regulation of the Minister of Energy and Mineral Resources (ESDM) No. 8 of 2015 concerning increasing the added value of minerals supports the importance of research for laterite ore processing. However, this abundant natural wealth has not been used to produce chemicals. Most of the chemicals used in Indonesia come from abroad and are sold at very high prices. If citizens process mineral ores in this country, it will significantly help save costs incurred on purchasing chemicals abroad (Aziza, 2017).

Indonesia is one of the countries with the world's largest quality nickel mining products. The Philippines and Indonesia are ranked as the top providers of nickel products. Indonesia is known as one of the countries with the world's largest nickel reserves. Around 12 % of the world's nickel reserves are in Indonesia in the form of laterite nickel ore. Indonesia's nickel-producing areas include Central Sulawesi, Southeast Sulawesi, South Sulawesi, Maluku, and Papua (Lintjewas et al., 2019).

The processing method used to extract nickel is Metallurgy, which is the study of the process used to separate the precious metal from other materials. The metallurgical method is divided into two parts: pyrometallurgy and hydrometallurgy. Hydrometallurgy is a metal extraction process

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carried out at a relatively low temperature by leaching using a chemical solution. At the same time, pyrometallurgy is a metal extraction process carried out at high temperatures (Wanta et al., 2018; He et al., 2022).

In this study, nickel analysis was carried out by processing laterite nickel ore using hydrometallurgical technology, namely the nickel extraction process using a leaching process with a specific reagent. This technology is commonly used to process nickel ore with low grades. The final result of this processing is Ni(II) nickel (Prasetyo & Prasetyo, 2015; Solihin & Firdiyono, 2018; Putera et al., 2023). An acid solvent is used to leach laterite samples. The type of acid used is a mixture of choric acid with nitric acid at a concentration of 0.5 M. Hydrochloric acid (HCl) is a chemical compound that is strong acid, consisting of chemical bonds between hydrogen atoms and chlorine atoms. In contrast, nitric acid (HNO_3) is a strong hydrogen, nitrogen, and oxygen atom solution. Acid solvents are good metal extractants (MacCarthy et al., 2016; Wahab et al., 2021).

This research will be optimized using the Taguchi method, which is a methodology in the field of engineering that aims to improve the quality of products and processes and can reduce costs and *resourses* to a minimum, so that with this method optimization value will be obtained and will reduce the number of experiments (Sidi & Wahyudi, 2013; Sutisna et al., 2023). This study studied the effect of leaching time parameters, HCl-HNO_3 volume ratio, and temperature conditions on the percentage increase in Ni. Each parameter has three levels, so with the Taguchi method's design, it only requires 9 experiments. If you do not use the Taguchi method, 27 experiments will be required, which can drain energy and material (Aziza, 2017).

One method for analyzing nickel content is atomic absorption spectrophotometry (AAS), which determines metal elements and metalloids by measuring light absorption with a specific wavelength by metal atoms in a free state (Tanujaya, 2017). This paper is intended to describe the extraction of nickel from Morowali laterite ore with a mixture of hydrochloric acid and nitric acid.

Methods

The main tools used in the study are *a* thermostatic hot plate, magnetic stirrer, laboratory oven, and SSA (atomic absorption spectrophotometer). The materials used as solvents in the laterite sample leaching process are 65 % nitric acid made by *E. Merck* and 37 % hydrochloric acid made by Mallinckrodt.

This research was carried out in several stages: the first stage of sample preparation and solution manufacturing. Then, the second stage carried out the leaching process according to Taguchi's design, as Aziza (2017) did. The extracted filtrate was analyzed using SSA (Atomic Absorption Spectrophotometer), and the optimal leaching parameters were determined using the Taguchi method analysis. After the optimal parameters are obtained, the next stage is an extraction process with optimal parameter conditions, and the extraction results are re-analyzed with the SSA tool.

Characteristic sampling laterit

Laterite ore originating from the Bahodopi District, Morowali Regency, Central Sulawesi Province, has reddish-brown characteristics derived from ultramafic rocks. The sample was chemically analyzed using X-ray Fluorescence (XRF) rays to determine the percentage of chemical elements and the distribution of their elements. The chemical characteristics are presented in **Table 1.**

Table 1. Chemical analysis of laterite samples (%)

Laterite	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	K_2O	TiO_2	P_2O_5
XRF	23.31	3.95	46.24	0.79	9.99	0.85	0.009	0.084	0.018
Laterite	Co_2O_3	Cr_2O_3	CuO	V_2O_3	ZnO	Lost Incandescent	Na_2O	NiO	
XRF	0.091	2.04	0.019	0.031	0.047	10.33	0.075	2.12	

Sample preparation

The laterite sample is crushed into small pieces and weighs 1 kg. It is dried in the oven at 110 ºC for ± 3 hours. The dry sample is mashed in a mortar and pestle, then sifted to a size of 80 mesh, and the result is stored in a closed container.

Solution manufacturing

34.62 mL of 37% HCl solution and 41.12 mL of 65% HNO3 solution were dissolved in a 1000 mL measuring flask. Aquades were added to the boundary mark and then homogenized, so HCl and HNO3 solutions were formed, one Liter each, at a concentration of 0.5 M, which is used as a solvent in the leaching process.

Extraction process

20 grams of laterite ore samples are put into a 1000 mL beaker. A mixture of hydrochloric acid - nitric acid solution of 0.5 M is added as much as 100 mL with a ratio of 1:1 (50:50 mL). The beaker is placed on a thermostatic hot plate magnetic stirrer at 30 ^{o}C for 3 hours. Moreover, it was repeated for acid ratios of 1:2 (33 mL: 67 mL) and 2:1 (67:33 mL) at temperatures of 600 °C and 900 °C, and for 6 hours and 12 hours by following the design of the Taguchi method in **Table 2.** Then the leaching results are filtered.

Table 2. Research design according to Taguchi

No	Race HCl – HNO_3	Time (hours)	Temperature (°C)
1	1: 1	3	30
2	1: 1	6	60
3	1: 1	12	90
4	1: 2	6	30
5	1: 2	12	60
6	1: 2	3	90
7	2: 1	12	30
8	2: 1	3	60
9	2: 1	6	90

Nickel analysis

The filtrate obtained from *leaching is* analyzed by measuring the absorbance value of Ni(II) ions and then calculating the concentration through the relationship of the Ni(II calibration curve. Nickel metal analysis was performed twice to obtain more accurate data. The SSA instrument used is GBC 932 AA. The specifications parameters for nickel metal analysis are a special LTL2-Ni cathode hollow lamp, acetylene gas fuel (C_2H_2), oxygen support (O_2), and a wavelength of 330.3 nm.

After the Nickel metal was analyzed quantitatively with SSA, data processing was carried out with a data analysis technique referring to Aziza (2017), namely the analysis of optimal leaching parameters using the Taguchi method "larger the better". The steps to determine the optimal conditions with the Taguchi method are, in the first step, make a Taguchi design or Orthogonal Array matrix = L9 3^3 based on the extraction parameters used, namely 3 factors and 3 levels with 9 stages of experimentation (Sidi & Wahyudi, 2013; Pérez-Portugal et al., 2023). The second step is to enter the data from the analysis of the processed nickel in the form of the average absorbance value into the "Taguchi Method Design" worksheet made in the first step. Furthermore, the final step is to analyze the experimental data using the Taguchi method to determine the optimal parameters for extracting nickel.

Results and Discussion

Data from nickel analysis

Before analyzing Ni(II) levels, a calibration curve of the Ni(II) standard solution was first made to compare with several concentrations, namely 0.1, 0.5, 1, and 2 mg / L. The results of the calibration curve of the nickel standard solution obtained a value of y = 0.0602x + 0.01335 with R2 = 0.9982.

Based on the calibration curve equation, the average nickel concentration data from the results of SSA analysis were obtained 2 times presented in **Table 3.**

Table 3. Results of quantitative analysis of nickel with Taguchi design

No	Race (HCl-HNO_3)	T (°C)	t (hours)	Concentration average (mg/L)	Abs average
1	1:1	30	3	8.696	0.5375
2	1:1	60	6	8.534	0.5270
3	1:1	90	12	10.452	0.6425
4	1:2	30	6	10.122	0.6230
5	1:2	60	12	7.454	0.4620
6	1:2	90	3	8.617	0.5320
7	2:1	30	12	8.313	0.5165
8	2:1	60	3	9.970	0.6135
9	2:1	90	6	8.715	0.5380

Optimal parameter analysis

Optimal conditions are those where the highest amount of nickel is obtained. This can be determined by analyzing the Taguchi method on the experimental data in **Table 3** above to combine optimal condition parameters. The results obtained for optimal conditions are at an acid mixture ratio of 1:1, a temperature of 90 °C, and a leaching time of 6 hours. The optimal combination of parameters obtained from the Taguchi analysis is presented in **Figure 1.**

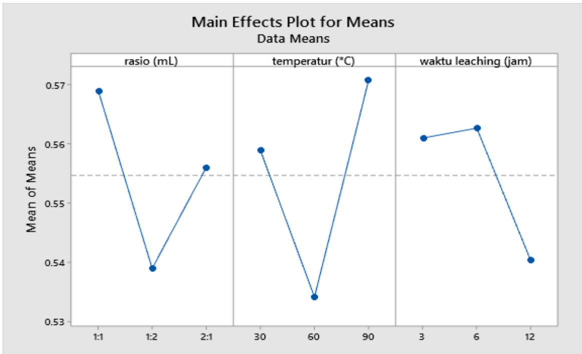

Figure 1. Optimal leaching parameters

The experiment's results will allow modeling the relationship between the factors and the characteristics being studied, i.e., the relationship between the metal Ni's absorbance value and the variation in the level of the leaching condition, where the greater the Ni's absorbance, the more optimal the parameter.

Optimal nickel analysis

Nickel analysis at the optimum parameters was based on the results of laterite leaching with an acid ratio of 1:1, temperature of 90 °C, and 6 hours using an SSA tool with an absorbance value of 0.757. Then, with the equation of the relationship of the Ni-calibration curve, it is known that the nickel concentration is 10.789 ppm. From the results of optimal nickel analysis using the Taguchi method, there has been an increase in the amount of nickel extraction from the results of the first nickel analysis in **Table 3** with a difference of 3.22 %, so it can be said that nickel analysis at 1:1 ratio condition, temperature 90 °C, and leaching time of 6 hours has significantly extracted more nickel. Then, with these results, the percentage of nickel recovery was determined from the comparison of the results of the Ni analysis in the sample (solid) with the results of the optimal nickel analysis (liquid), which was 2.54 %.

Influence of acid ratio

Based on the results of the analysis of the Taguchi method, it is known that the most optimal nickel spinning ratio is at the level of 1:1. The results obtained are influenced by several factors, one of which is the pH condition, where the greater the acidity level of a solution, the better the solution is to be used in the process of leaching metal ions, this is because more acidic solutions (the smaller the pH) tend to have more H^+ ions to dissolve nickel into $Ni(OH)_2$ as the aqueous phase. So that the reaction that occurs in the laterite nickel leaching process is an acid-base reaction, with the possibility of reaction similarities that can occur from the laterite leaching process, according to Li et al. (2020), are as follows:

$$NiFe_2O_{4(s)} + 2H^+ = Ni^{2+} + Fe_2O_{3(s)} + H2O_{(l)} \quad (1)$$

$$NiO_{(s)} + 2H^+ = Ni^{2+} + H_2O_{(l)} \quad (2)$$

$$2NiO \cdot SiO_{2(s)} + 4H^+ = 2Ni^{2+} + SiO_{2(s)} + H_2O_{(l)} \quad (3)$$

Theoretically, the acidity level of the three acid ratios used in this study will be the same: pH = 0.3. This is because the concentration is always 0.5 M, and the number of hydrogen ions possessed by HCl and HNO_3 compounds is also the same value, namely a = 1 in strong acids. In acidic mixtures with a ratio of 1:1 (50:50 mL), the solution may have a higher acidity level than other ratios. This result can be caused by diluting the acid solution; it is possible that the researcher is not accurate in reading the numbers, and the tools used are not specific (makeshift ones), so the solution used can have a slightly different concentration than desired. Other factors can also affect leaching efficiency, such as different stirring rate intensities or other compounds that can affect the diffusion ability of Ni(II) ions (Peng et al., 2023).

Temperature Effect

The highest nickel extraction results in the temperature factor are obtained when using a temperature of 90 °C, as shown in Figure 1, where, when using a temperature of 30 °C to 60 °C, the percentage of nickel melting produced decreases. This means that using 30 °C and 60 °C temperatures is not significant in increasing the value of nickel collection. A similar tendency also occurs in experiments conducted by Astuti et al. (2016) and Wanta et al. (2016). Based on previous research by Chou et al. (1977), Georgiou & Papangelakis (1998), Whittington & Muir (2000), and Ali et al. (2023), the optimum temperature of the nickel leaching process ranges from 270- 280 °C. However, in this study, it is not possible to use this temperature due to the limitations of the tools used in the research, so that if it is above 90 °C, the results of nickel extraction will continue to increase to the optimal limit. Temperature affects the kinetics of the spinning reaction, based on the Arrhenius equation; the higher the temperature, the higher the constant of the reaction rate (Carvalho-Silva et al., 2019). The higher the temperature, the faster the movement of each molecule will be, and the possibility of molecules colliding and reacting will also increase, so the chance of nickel precipitation will increase (Taylor et al., 2023).

Effects of leaching time

The length of the leaching process is also an important factor in the optimal nickel collection. To determine the effect of the rolling time in nickel collection, time variations were made for 3 hours, 6 hours, and 12 hours. The results obtained can be seen in Figure 1, where the optimal nickel turning time is at the level of 6 hours. Sudibyo et al. (2017) also obtained this optimal condition. After this time, the extraction results tend to decrease within 12 hours (not showing significant results). Based on previous research by Tian et al. (2023), the optimum time for nickel polishing is about 180 minutes or 3 hours. This is influenced by the kinetics of chemical reactions, in the form of the presence of heat-resistant minerals (e.g. lizardite and goethite) that are highly resistant to acids, thus preventing the extraction of nickel from the existing mineral lattice, and the influence of the solvent used to achieve a state of equilibrium on the amount of metals extracted (Febriana et al., 2018).

Conclusions

The optimal conditions of the laterite nickel *leaching* process using a mixture of hydrochloric acid and nitric acid were obtained at a ratio parameter of 1:1, a leaching time of 6 hours, and a temperature of 90 °C, with nickel analysis results of 10.789 ppm and nickel recovery of 2.54 %.

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