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The Extraction of Nickel from Morowali Laterite Ore with Sulfuric Acid (H_2SO_4)

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Abstract

Efficient recovery of valuable minerals from waste materials requires optimizing key processing parameters to maximize extraction efficiency and resource utilization. This research was conducted to determine the optimal extraction conditions, which were influenced by sulfuric acid concentration, temperature, and leaching time. This study used the Taguchi method with 3 factors and 3 levels: the concentration of sulfuric acid (H_2SO_4) at 0.5 M, 1 M, and 2 M; temperatures at 30 °C, 60 °C, and 95 °C; and leaching times of 3 hours, 6 hours, and 12 hours. The optimum conditions obtained from the study were a temperature of 95 °C, a leaching time of 6 hours, and an acid concentration of 2 M, with an absorbance of 0.6820. These results indicate that the Taguchi method is an efficient and reliable approach for optimizing leaching parameters, thereby contributing to the development of sustainable mineral recovery from waste materials.

Keywords: Laterite, nickel, sulfuric acid (H_2SO_4), Taguchi

Introduction

Indonesia is a country rich in natural and mineral resources. One of the mineral resources with potential for development in Indonesia is laterite nickel, as shown in data on world nickel production and reserves from the United States Geological Survey report in January 2015 (Setiawan, 2016). In terms of potential reserves, Indonesia ranks sixth, with 5% of the world's total. This shows Indonesia's important position in the world of nickel ore mining. Indonesian laterite nickel deposits are scattered across several areas, including South Sulawesi, Southeast Sulawesi, Maluku, and Papua (Solihin & Firdiyono, 2014; Konopka et al., 2022).

Global demand for metals is increasing due to the rapid development of the chemical and manufacturing industries. One of the metals widely used in industry is nickel (Wanta et al., 2018; Kruszelnicka et al., 2022). Nickel is widely used because it has good corrosion resistance, is easy to shape, yet remains strong. Nickel is a vital and strategic metal. This metal is one of the main raw materials in the manufacture of stainless steel and is primarily found as a laterite mineral. About 60% of the world's nickel resources are available as nickel oxide, commonly called nickel laterite (Marrero et al., 2015; Stanković et al., 2022).

Laterite is the result of weathering and enrichment of mafic/ultramafic rocks in the tropics; therefore, the chemical composition and mineralogy differ from one deposit to another. The weathering process in ultramafic rocks produces different nickel laterite characters and profiles (Lintjewas et al., 2019). One of the factors that influences the formation of laterite nickel deposits is the source rock (Kurniadi et al., 2017). Nickel in laterite nickel ores is associated with iron oxide and silicate minerals as a result of isomorphous substitution of iron and magnesium in their crystal structures, so that, chemically and physically, laterite nickel ores can be classified into two types, namely saprolite (silicate/hydrosilicate) ores. limonite (oxide/hydroxide). Laterite nickel ore is difficult to convert into nickel concentrate due to the isomorphic crystal structures of goethite and serpentine (Subagja et al., 2016).

At this time, 2 types of nickel ore are widely used as raw materials for producing nickel metal: sulfide and laterite ores (Bahfie et al., 2022). Over time, nickel sulphide ore reserves continue to decline, prompting some nickel producers to turn their attention to laterite ores as nickel feedstocks. Unlike sulfide ores, laterite ores are not easy to

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increase their nickel content with current technology, so various research efforts continue to be carried out to increase nickel levels in laterite (Subagja et al., 2016). New alternatives need to be developed to make nickel production from laterite ore more efficient, cost-effective, and sustainable.

High-grade nickel ore is processed through a high-temperature process (pyrometallurgy), while nickel extraction from low-grade laterite nickel ore is generally carried out by the hydrometallurgical method. 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; He et al., 2023). Pyrometallurgical processes also cause environmental problems, such as air pollution from high temperatures. In addition, this process requires high energy and high operating costs (Listyarini, 2017; Simate et al., 2010).

Most nickel production processes use High Pressure Acid Leaching (HPAL) and Atmospheric Pressure Acid Leaching (APAL). The High Pressure Acid Leaching (HPAL) process operates at 4-5 MPa and 245-270 °C. As well as a special titanium-coated autoclave. Atmospheric Pressure Acid Leaching (APAL) is operated at low temperatures and is a relatively simple process. Due to the high operating pressure and temperature requirements of HPAL, it requires a more expensive and complex tool than APAL, making APAL a cheaper alternative. However, the APAL process has several drawbacks, including low nickel-extraction reaction kinetics and production liquor containing dissolved iron and aluminum (Santoso, 2017).

Separation of nickel metal from laterite ore can be done by the extraction process. Many dissolution methods are used for nickel metal extraction, including HCl, H_2SO_4, HNO_3, and aqua Regia as solvents. In this study, nickel metal was separated from Morowali laterite ore by leaching (solid-liquid extraction). This extraction is usually used to process low-grade nickel ore, yielding nickel (Ni) as the final product using an acid solvent (Prasetyo, 2008; Zhao et al., 2022).

The extraction process in this study uses H_2SO_4 as the solvent, and the nickel separation process also uses H_2SO_4, a method known as Sulfuric Acid leaching, which is a selective separation method for extracting metals from natural minerals with H_2SO_4 as the reactant in the dissolution process. For the Sulfuric Acid leaching method, it has several advantages, including the reactant material used (H_2SO_4) is easy to obtain, relatively inexpensive, not too complicated, and when compared to using HCl, HNO_3, and aqua Regia solvents, because in solvents other than H_2SO_4, the solvent will extract more ferrous metal than nickel metal (Dubenko et al., 2020).

According to Nasab et al. (2020), the dissolution of nickel metal in laterite minerals is influenced by several conditions, including solution concentration, dissolution time, and dissolution temperature. One of them is the variation in 0.1 M solution concentration: 0.2 M, 0.3 M, 0.4 M, and 0.5 M, with 0.5 M optimal, with nickel extractable at 80% of the initial grade. Based on these studies, the higher the concentration of the H_2SO_4 solution, the higher the nickel metal content obtained. However, in this study, a lower variation was used to see the effectiveness of H_2SO_4 as a solvent.

Wanta et al. (2017), whose research used temperatures of 30 ^oC, 60 ^oC, and 85 ^oC with the highest nickel content results found at the highest temperature of 85 ^oC where the higher the use of the higher operating temperature, the higher the percentage of nickel extraction and this study used temperature variations. 30 ^oC, 60 ^oC, and 95 ^oC dissolution.

Garces-Granda (2020) reports that dissolving nickel metal in laterite minerals is carried out in several ways, including variations in solution concentration, dissolution time, and dissolution temperature. For variations in dissolution time, the optimal dissolution time is 4 hours. In this study, the dissolution times are 3, 6, and 12 hours.

The purpose of this study was to identify the effects of sulfuric acid (H_2SO_4) concentration, temperature, and leaching time on nickel extraction from laterite ore from Bahodopi, Morowali Regency, Central Sulawesi Province.

Methods

This research is an experimental study based on nickel content analysis of extraction results from laterite ore, with variations in leaching time, temperature, and sulfuric acid concentration. Meanwhile, nickel content was analyzed using SSA (Atomic Absorption Spectrophotometer) to determine the amount of nickel in each sample. This research was conducted using the Taguchi method, a new engineering approach aimed at improving product and process quality and minimizing costs and resource use.

The tools used in this research are an oven, a mortar and pestle, and Whatman filter paper (hardened) No. 52 and 54, Büchner funnel, erlenmeyer filter flax, vacuum pump, filler (rubber suction), 100 mL beaker, magnetic stirrer, petri dish, vial, thermometer, test tube, dropper, spatula, sieve, stirrer, digital balance, measuring cup, flask, aluminum foil and AAS Elmer analyst 700. The materials used in this study include laterite ore from the Bahodopi sub-district, Morowali Regency, Central Sulawesi Province, and 96.1% sulfuric acid (H_2SO_4).

Extraction parameters and level

In using the Taguchi method, testing parameters with different levels are needed. The research design table for this study is presented in **Table 1.**

Table 1. Research design according to Taguchi

Parameter	Condition level		
	I	II	III
Acid Concentration (M)	0.5	1	2
Temperature (°C)	30	60	95
Leaching Time (Hour)	3	6	12

Orthogonal array (AO)

The Taguchi method uses a special set of matrices, called Orthogonal Arrays, to determine which combination of factors and levels to use in efficient experiments and to analyze experimental data (Soejanto, 2009). The orthogonal array for the experimental design in this study is shown in **Table 2.**

Table 2. *Orthogonal array* matrix

No	Acid Concentration (M)	Temperature (°C)	Time (Hour)
1	0.5	30	3
2	0.5	60	6
3	0.5	95	12
4	1	30	6
5	1	60	12
6	1	95	3
7	2	30	12
8	2	60	3
9	2	95	6

Sample preparation

Sample preparation aims to make the sample ready for testing. The sample used in this study is laterite rock originating from the Morowali area. The procedure for sample preparation is to weigh 1000 grams of the sample using a digital balance, then heat or dry the weighed sample in an oven at 110 °C for 3 hours to remove the water content. After that, the dried samples were ground in a mortar and pestle, then sieved through a 200-mesh sieve.

Extraction process

The extraction process to be carried out in this study will use three factors: temperature, time, and acid concentration. These three factors will affect the extraction results from each sample. 20 grams of fine laterite were weighed, placed in a 1000 mL beaker, and 100 mL of 0.5 M sulfuric acid (H_2SO_4) solution was added. Cover the glass surface tightly with aluminum foil to prevent the mixture from evaporating.

Heat the mixture on a hot plate at room temperature and stir using a magnetic stirrer for 3 hours. The mixture was filtered using a Büchner funnel lined with filter paper. Perform steps 1-4 for temperature variations of 60 °C and 95 °C, time variations of 6 hours and 12 hours, and concentration variations of 1.0 M and 2.0 M.

After leaching and filtration, the nickel content in the filtrate was analyzed by AAS, and the results were recorded. The data obtained from the sample analysis using the AAS tool are processed to determine the optimum conditions and the percent recovery of nickel.

Results and Discussion

Nickel level analysis

Nickel analysis using AAS on samples extracted from nickel in laterite ore, influenced by 3 factors: temperature, leaching time, and sulfuric acid (H_2SO_4) solution, using the Taguchi method. The results of the analysis are shown in **Table 3.**

Table 3. Results of the analysis of nickel levels

No.	Acid Concentration (M)	Temperature (°C))	Leaching Time (Hour)	Absorbance	Concentration (ppm)
1	0.5	30	3	0.4120	15.5172
2	0.5	60	6	0.3775	14.1954
3	0.5	95	12	0.5725	21.6667
4	1	30	6	0.5215	19.7126
5	1	60	12	0.5430	20.5364
6	1	95	3	0.5645	21.3602
7	2	30	12	0.3945	14.8467
8	2	60	3	0.5710	21.6092
9	2	95	6	0.6820	25.8621

Determination of optimum conditions

The optimum condition is the one in which the analyzed nickel content is highest. Based on **Figure 1,** the optimum conditions obtained from the 9 analytical data points were then used to analyze nickel samples. The results of nickel extraction under the optimum conditions are shown in **Table 4.**

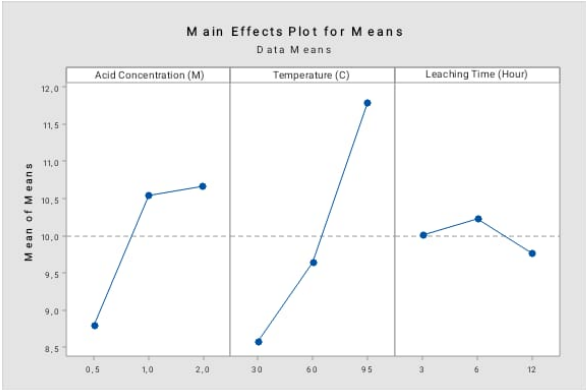

Figure 1. Results of determining optimum conditions

Table 4. Results of optimum nickel concentration

Acid Concentration (M)	Temperature (°C)	Leaching Time (Hour)	Absorbance	Concentration (ppm)
2	95	6	0.6820	25.8621

The average absorbance under the optimum conditions was 0.6820, the average concentration was 25.8621 ppm, and the percent recovery of nickel was 6.01%.

This research presents an alternative method for analyzing nickel content in laterite rock or soil using extraction. The leaching process, or solid-liquid extraction, is commonly used to treat low-grade nickel ore, yielding a final product of nickel (Ni) using an acid solvent. The nickel laterite leaching process has several important variables that can affect the nickel recovery from leaching, namely solution concentration, solvent temperature, and solvent time, which in this study used acid concentrations of 0.5 M, 1 M, and 2 M, temperatures 30 °C, 60 °C, and 95 °C, and leaching time for 3 hours, 6 hours, and 12 hours.

The nickel separation process using H_2SO_4 solvent, also known as the sulfuric acid leaching method, is a selective method for extracting metals from natural minerals, with H_2SO_4 as the reactant in the dissolution step. Javanshir et al. (2018) conducted a series of leaching experiments using sulfur and hydrochloric acid as solvents. Conditions such as temperature (90 °C), time (2 hours), stirring speed (300 rpm), and S/L ratio (0.25 g/cm^3) remained constant during the experiment. The best conditions for maximum recovery were obtained by using H_2SO_4. Therefore, sulfuric acid solution was used for nickel extraction in a comprehensive study.

Effect of acid concentration

The sulfuric acid (H_2SO_4) concentrations in this study were 0.5 M, 1 M, and 2 M. The acid concentration was determined to identify the optimal concentration for extracting laterite ore. Based on the research conducted, optimal nickel extraction occurs at an acid concentration of 2 M. This is because increasing the acid concentration increases the concentration of H+ ions in solution, which react with nickel ore and leach the nickel it contains. thereby increasing the activity of H^+ ions in solution. According to Le Chatelier's principle, increasing the acid concentration in the reaction of Ni-containing material with acid will shift the reaction to the right.

Top et al. (2020) stated that increasing acid concentration in atmospheric leaching can increase metal extraction from laterite ores and cause greater dissolution of nickel. According to Li et al. (2020), the possible reactions involving this metal are given in equations 1, 2, and 3.

$$NiFe_2O_{4(s)} + 2H^+ \rightarrow Ni^{2+} + Fe_2O_{3(s)} + H_2O_{(l)} \quad (1)$$

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

$$NiO_{(s)} + 2H^+ \rightarrow Ni^{2+} + H_2O_{(l)} \quad (3)$$

Temperature effect

Temperature is an important parameter and has a considerable influence on the nickel extraction process (Luo et al., 2010). To determine the effect of leaching temperature on nickel collection, experiments were conducted at 30 °C, 60 °C, and 95 °C. Based on the research results, the highest temperature is 95 °C, indicating that higher temperatures will also yield a higher nickel percentage. Similar results were also observed in the study by Wanta et al. (2017), which used temperatures of 30 °C, 60 °C, and 85 °C, with the highest nickel content at 85 °C. Using higher temperatures increases the likelihood of molecular collisions, so the stages of nickel product formation will also be higher.

Wahab et al. (2021) also stated that higher temperatures are associated with greater nickel recovery. This is because the higher the temperature, the greater the movement of the reacting species, thereby increasing the amount of reaction product. This is in accordance with the research of Meshram et al. (2019), which reports an optimum temperature of 270 °C for the nickel leaching process, while the previous research of Whittington & Muir (2000) reports an optimum temperature of 280 °C.

Effect of leaching time

The duration of the leaching process is an important factor in determining optimal conditions. To determine the effect of leaching time on nickel content analysis, the analysis was carried out at 3, 6, and 12 hours. The results obtained were the leaching time at which the highest nickel content was observed: 6 hours. These results indicate that the obtained nickel extraction optimization is neither the fastest nor the longest. Meanwhile, based on research by Faris et al. (2023), the optimum nickel extraction time is 180 minutes (3 hours), after which the % nickel recovery percentage remains constant. The kinetics of chemical reactions influence this; the presence of heat-resistant minerals (e.g., lizardite and goethite), which are highly acid-resistant, prevents the extraction of nickel from the lattice. The minerals present and the solvent used reach an equilibrium state with respect to the amount of metal extracted.

Conclusions

Based on the research conducted, the optimal conditions were a concentration of 2 M Sulfuric Acid (H2SO4), a temperature of 95 °C, and a leaching time of 6 hours. The average concentration was 25.8621 ppm, and the percent recovery of nickel was 6.01%.

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