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KINETIC PROPERTIES OF NICKEL LEACHING BY ANOVA METHOD

Hydrometallurgical extraction of nickel laterite is more efficient in terms of energy consumption, because it produces less exhaust gas compared to the pyrometallurgical method. Therefore, the hydrometallurgical method can increase the extraction yield of inferior nickel laterite more. In the calculation of the analysis of variance (ANOVA), three factors are used to determine the importance of the variables and the order of the most influential variables. ANOVA test is also a form of statistical hypothesis testing where we draw conclusions based on the inferential statistical data or groups. The 'null' hypothesis of the ANOVA test is that the data are simple random from the same population, so that they have the same expected mean and variance. In addition, a study of leaching kinetics is carried out using a shrinking core model to determine the reaction rate controller. The results show that the leaching time has an important role of acid, base, and monosodium glutamate in increasing the nickel extraction rate. Based on the ANOVA results, the two most influential factors are temperature and leaching time. The ANOVA-based calculation use is more accurate than using a conventional method, such as 'excel', and it needs more development for mineral extraction in the future.

Keywords: leaching, kinetics, analysis of variance (ANOVA), Ni.

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1. Introduction: Overview of the Problem

Nickel is an important component to produce stainless steel. It is also used in super alloys, aerospace industry, high temperature alloys, and electrolytic coatings. The second largest nickel reserves in the world belong to Indonesia, meaning that this country has an important role in the supply of world nickel raw materials. Nickel ores are classified into two types, namely nickel sulphide and nickel oxide or laterite. The largest part of the nickel reserves is low-grade nickel with a Ni content of <1.8%. Nickel laterite consists of limonite with Ni content <1.5% and saprolite with Ni content >1.5%. Laterite nickel is found in Konawe Regency, Southeast Sulawesi Province. Laterite nickel in Konawe Regency, Southeast Sulawesi is a type of low-grade nickel laterite that can be processed using a hydrometallurgical route. The processing of low-grade nickel laterite consisting of limonite and saprolite with a Ni content <1.8% is still not processed domestically [1–10]. Laterite nickel ores can be classified into limonite and saprolite nickel ores [11–15].

Laterite nickel ore processing can be carried out through pyrometallurgical or hydrometallurgical routes and a combination of the two. The pyrometallurgical process treats the high grade laterite nickel ore to produce ferronickel and nickel matte. The low-grade nickel ores can be processed by hydrometallurgical processes [16]. Research on the processing of low-grade laterite nickel ore using the hydrometallurgical process continues to develop because this method has several advantages, namely low operating costs and environmentally friendly [17].

There are several methods of processing nickel laterite ore using a hydrometallurgical process, namely high-pressure acid leaching (HPAL) and atmospheric leaching (AL). Pressure/high-pressure acid leaching (PAL/HPAL) has advantages in the leaching process, but, on the other hand, this method has disadvantages such as investment costs for the equipment used, high consumption of sulphuric acid, and maintenance of equipment during operation [18–22]. One of the important studies carried out in the leaching process of laterite nickel ore is the study of leaching kinetics. The purpose of the kinetic study, among others, is to determine the step controlling the reaction rate and its kinetic model. The rate control stage is the slowest stage of all stages. The rate control step is important to know because by knowing the rate controller, it can be determined efforts to increase the leaching rate. In addition, the kinetic model can help predict the rate of the leaching process when leaching for the same ore and solution [20–22].

Laterite nickel processing generally uses sulphuric acid (H_2SO_4) as the reagent with the HPAL process, but the HPAL process cannot be used to treat low-grade nickel laterite [23]. Leaching kinetics studies laterite nickel ore using H_2SO_4 solution, which leaches laterite nickel ore on the saprolite layer with variations in temperature and leaching time variations. In this study, the nickel ore sample used was laterite nickel ore in the limonite layer by varying the leaching temperature, H_2SO_4 concentration and leaching time. The purpose of this study was to study the effect of process variables (temperature, sulphuric acid concentration, and leaching time) on the percent nickel extraction using the 3-factor analysis of variance (ANOVA) method and study the leaching process kinetics using the shrinking core model [24–29].

2. Calculation Method and Results of the Study

The statistical analysis method ANOVA (analysis of variance) belongs to the branch of inference statistics. In the literature, this method is known as different other, but similar, names. It is an extension of the Behrens–Fisher problem; so, the F-test is also used in the decision-making. Analysis of variance was firstly introduced by Sir Ronald Fisher — the father of modern statistics. In practice, analysis of variance can be a hypothesis test (more often used) or estimation (estimation, especially in the field of applied genetics). Analysis of variance or ANOVA is a multivariate analysis technique that serves to distinguish the mean of more than two groups of data by comparing the variances. Analysis of variance belongs to the category of parametric statistics. As a parametric statistical tool, to be able to use the ANOVA formula, it is necessary to first test assumptions including normality, heteroscedasticity, and random sampling.

Analysis of variance is a procedure that tries to apply this portion of variance to each group of independent variables. This technique compares several variables simultaneously to minimize the possibility of error. The advantage of using analysis of variance is that it can perform comparisons for many variables. The purpose of the analysis of variance is to find the independent variables in the study, how they interact and affect the treatment. The lateritic nickel ore composition used in this determination was determined using an x-ray fluorescence (XRF) instrument. Categories of low-grade nickel ore with the results of XRF analysis are presented in Table 1 [15].

Table 1 shows that laterite nickel ore samples were dominated by Fe and Si with grades of 9.403% and 8.077%, respectively. The elemental content of Ni in this sample is 1.021%. In addition to using XRF, nickel ore samples were also analysed using x-ray diffraction (XRD) to determine the mineral content in the sample. The results of the XRD analysis are presented in Fig. 1. The results of the XRD analysis show that the dominant compounds in this research sample are magnetite (Fe_3O_4), coesite (SiO_2), goethite (FeOOH), and liebengergite (Ni_2SiO_4) [15]. Determination of the order of the variables that most influence the

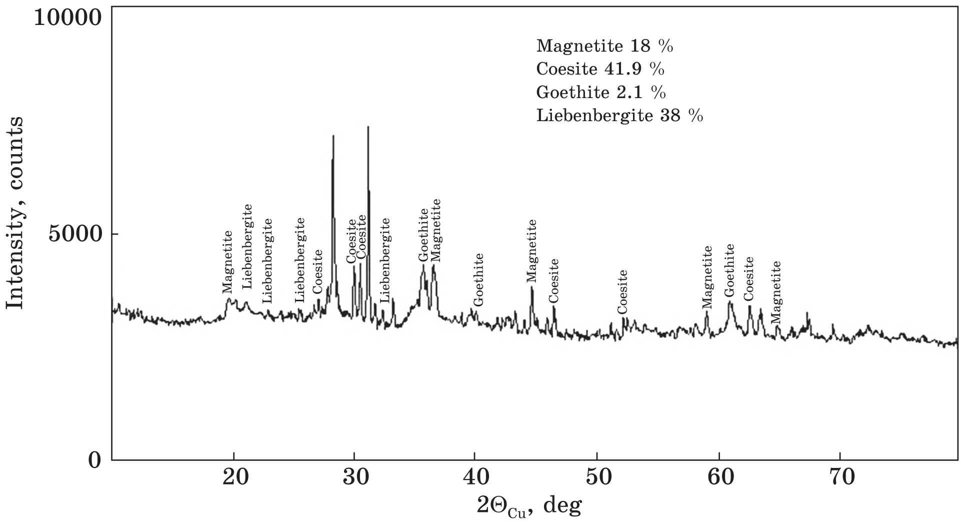

Fig. 1. XRD analysis results of laterite nickel ore [15]

percent nickel extraction using ANOVA 3 factors. If the P-value > 0.05 or F-value $< F_{0.05v1,v2}$, then H_0 is accepted, that indicates that there is no effect of these variables on the results. H_0 is rejected if P-value < 0.05 or F-value $> F_{0.05v1,v2}$, that indicates that there is a significant effect of the variable on the evaluated results (percent of Ni extraction). The results of the 3-factor ANOVA calculation are presented at '0' — the factor (from ANOVA program) is not affecting in leaching process [15].

Based on '0', all variables and interactions between variables have a significant influence on the percent nickel extraction. This is indicated by the P-value < 0.05, while determining the order of the most influential variables based on the magnitude of the difference in the value of F-value to $F_{0.05v1,v2}$. F-value obtained from the calculation, while the value of $F_{0.05v1,v2}$ obtained from the distribution table F, where v_1 is the degree of freedom of a factor and v_2 is the degree of freedom from error. The results of the 3-factor ANOVA calculation presented at '0' show the order of the variables that have the most influence on the percent nickel extraction, namely sulfuric acid concentration, temperature, leaching time, leaching time temperature interaction, sulphuric acid concentra-

Table 1. **Composition of laterite nickel ore [6]**

Element	Fe	Si	Ni	Al	Ca	Cr	Mn
Rate, %	9.403	8.077	1.021	1.159	0.968	0.340	0.175
Element	Co	S	P	V	Sb	Cd	Zn
Rate, %	0.063	0.042	0.022	0.011	0.035	0.022	0.009

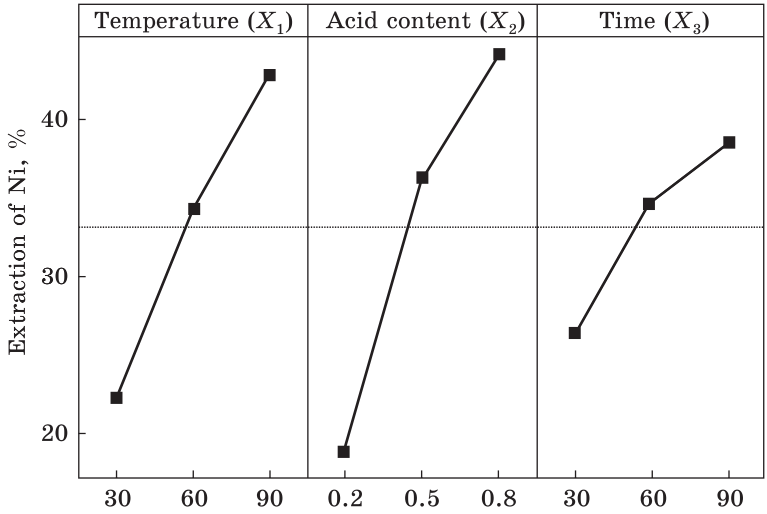

Fig. 2. Effect of leaching variable on the Ni percent extraction [15]

tion temperature interaction, temperature–sulphuric acid concentration–leach time interaction, and leaching time temperature interaction [30].

ANOVA analysis of 3-factors to determine the degree of freedom (DF), sum of square (SS), mean of square (MS), *F*- and *P*-value. The results of the 3-factor ANOVA analysis are presented in Table 2 [15].

The results of the 3-factor ANOVA analysis on the percent nickel extraction were used to determine the significance of the effect of the variable on the percent nickel extraction. If the *P*-value of the variable <0.05, then the variable is significant to the response. Table 2 shows that all variables are significant to the percent Ni extraction [15].

The determination of the most influential variable can be seen from the graph presented in Fig. 2. The steeper the line formed, the greater the effect of the variable. On the other hand, the more sloping the line is, the smaller the effect of this variable. The order of variables that most influence the percent nickel extraction is acid concentration, temperature and leaching time. These three variables show a positive effect on the percent nickel extraction [15].

Table 2. **Analysis of variance percent nickel extraction [15]**

Variable	DF	DS	MS	*F*-value	*P*-value
X_1	2	3801.5	1900.75	853.84	0.000
X_2	2	6101.9	3050.95	1370.52	0.000
X_3	2	1407.2	703.62	316.07	0.000
Error	47	346.6	7.37		
Total	53	11657.2			

Regression analysis is one of the data analysis techniques in statistics that is often used to examine the relationship between several variables and predict a variable [31]. Regression analysis is an analytical method that can be used to analyse data and draw meaningful conclusions about the relationship between variable dependence on other variables [32]. The regression equation reads as

$$Y = A + BX \tag{1}$$

with Y — predictable variable (predictant/dependent), A — constant, B — regression coefficient, X — estimator variable (predictor/independent).

The Ni extraction percent data (in Table 2) are then determined from the regression equation model — equations for different acid concentration c_{acid} [15]:

$$y_1 = -13.91 + 0.3413X + 0.2043X_2 \ (c_{\text{acid}} = 0.2 \text{ molar}), \tag{2}$$

$$y_1 = 3.63 + 0.3413X + 0.2043X_2 \ (c_{\text{acid}} = 0.5 \text{ molar}), \tag{3}$$

$$y_1 = 11.52 + 0.3413X + 0.2043X_2 \ (c_{\text{acid}} = 0.8 \text{ molar}). \tag{4}$$

Regression analysis is a mathematical model that can be used to determine the pattern of the relationship between two or more variables. The main purpose of regression analysis is to make an estimate of a variable (dependent variable) if the value of the variable associated with it (independent variable) has been determined. Regression analysis involves the study of the relationship between a variable Y, which is called the response variable or the dependent variable, namely the variable whose existence is influenced by other variables. Variable X is a predictor variable or independent variable, namely the independent variable (not influenced by other variables). Testing the linearity of the regression equation is done by graphing the relationship between the percent extraction of the experimental results and the percent extraction of the predicted results to determine the value of the correlation coefficient (R^2) [15].

The higher the temperature, acid concentration and leaching time, the higher the nickel extraction percentage: the trend of the results of this study is in accordance with the results of previous studies [33]. The higher the temperature, the greater the nickel recovery. This is because the higher the temperature it will increase the movement of the reacting species thereby increasing the reaction products. The same trend also occurs in the increase in acid concentration, namely increasing the acid concentration increases nickel recovery. The increase in acid concentration causes an increase in H^+ ions in the solution, which will react with the nickel ore and leach the nickel contained in the ore. The increase in leaching time also increases nickel recovery. The leaching time relates to the contact time between the leaching agent and the nickel ore. The longer the leaching time, the contact time to react between the leaching agent and nickel ore also increases. Based on this trend, the percent recovery of nickel can be increased by increasing the acid concentration, leaching temperature, and leaching time [15].

The kinetic study used in this research based on the shrinking core model with the following equations [1, 15, 16]:

$$K_f t = x \quad (5)$$

— diffusion through fluid film layers;

$$K_d t = 1 - 3(1 - x)0.67 + 2(1 - x) \quad (6)$$

— diffusion through the unreacted solid product layer;

$$K_r t = 1 - 0.33(1 - x) \quad (7)$$

— interface reaction; here, x is the nickel recovery value, t is the leaching time, K_f, K_d and K_r are the leaching rate constants. The leaching process rate controller was determined from the linearity of the leaching regression curve according to Eqs. (5)–(7). The linearity of the regression curve was determined from the value of the regression curve that was closest to 1. The linear regression curves were for diffusion through the fluid film layer, diffusion through the unreacted solid product layer, and interfacial reactions [15].

Values for each controller of the leaching process rate of laterite nickel ore using H_2SO_4 solution at various concentrations. The value of R_2 indicates that the control of the laterite nickel ore leaching process using H_2SO_4 is diffusion through the unreacted solid product layer. The results of this study are in accordance with the results of a study that conducted a study of the kinetics of nickel ore leaching using sulphuric acid solution [33]. Increasing the reaction rate can be done by increasing the stirring intensity because it can reduce the thickness of the unreacted solid product [33]. In addition, it can also be done by increasing the temperature because increasing the temperature will increase the rate of diffusion of the reacting species.

In the leaching process, several kinetic models were tested to fit the experimental data [1, 34]:

$$kt = 1 - (1 - R)^{1/3}, \quad (6)$$

$$R = (1 - (1 - kt)^3)R_{\max} \quad (7)$$

— the shrinking core model (chemical reaction control);

$$kt = 1 - (1 - R)^{1/3} - 1, \quad (8)$$

$$R = (1 - (1 + kt)^3)R_{\max} \quad (9)$$

— the diffusion and transport model; here, k — reaction rate constant, t — leach time, R — gain of Ni at time, $R_{\max}$ — maximum Ni gain on each leach. (For comparison, see also diffusional kinetics models for Ni-based alloys in Refs. [35–40].)

In kinetic modelling, non-linear Eqs. (6) and (7) are used instead of linear equations. The mean relative error (ARE) in Eq. (5) can be evaluated from the following expression [1]:

$$\mathrm{ARE}(\%) = \frac{100}{n-1}\sum_{i=0}^{n}\left(\frac{R_{\mathrm{exp}} - R_{\mathrm{mod}}}{R_{\mathrm{exp}}}\right)^2, \qquad (10)$$

where R_{max} — maximum Ni gain on each leach, R_{exp} — the gain of Ni over time as a result of the experiment, and R_{mod} — gain of Ni over time as model result.

Generally, the shrinking core model fails to model the experimental data in this study, because the process control stage is not only one stage, and the interface-diffusion is more suitable for the experimental data. This shows that the dissolution of Ni can be controlled by diffusion and mass transfer model. The calculation of the activation energy (E_{a}) using the Arrhenius Eq. (6) and the reaction rate constant (k) obtained from the modelling, with the information A, T, and R, respectively, namely the frequency factor, temperature, and gas constant. The activation energy in this case is 24.6 kJ/mol. Low activation energy values can be controlled through the mass transfer and small temperature effects, because if the temperature is small, the activation energy value will remain large so that the exponential value is close to zero. This means that the particles do not have enough energy to react, and the reaction does not occur. The Arrhenius equation can be seen in the following Eq. (11) [34]:

$$k = Ae^{-\frac{E_a}{RT}} \text{ or } \ln k = \ln A - \frac{E_a}{RT}. \qquad (11)$$

Kinetic studies carried out for up to 12 hours at three different temperatures, namely 30, 55, and 80 °C with constant variables of $p\mathrm{H} = 9$, monosodium glutamate (MSG) concentration of 1 M, and pulp density of 50 g/L showed that maximum leaching efficiency could be achieved in 120 minutes for Zn and 4 hours for Cu at 30 °C. At higher temperatures, the gain reaches saturation in a relatively short time. The acquisition of Cu was delayed by about 1 hour. This is because in the

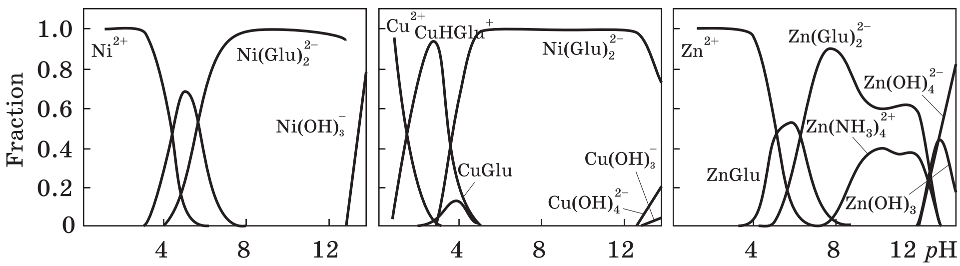

Fig. 3. Ni (left), Cu (middle), and Zn (right) distributions as a function of $p\mathrm{H}$ [1]

early stages of leaching the aqueous phase is reductive which inhibits the oxidation and complexation of Cu by reagents. During leaching, the aqueous phase becomes oxidative, favouring the oxidation and complexation of Cu [34] (see also Fig. 3).

The results of the leaching laterite ore with variations in temperature and leaching time are presented in Table 1. The percent Ni extraction data was performed with a 3-factor ANOVA analysis to determine the degree of freedom, sum of square, mean of square, *F*-value and *P*-value. The results of the 3-factor ANOVA analysis are presented in Table 2. The results of the 3-factor ANOVA analysis on the percent nickel extraction were used to determine the significance of the effect of the variable on the percent nickel extraction. If the *P*-value of the variable <0.05, then the variable is significant to the response. The significant result is in the leaching time due to the *P*-value <0.05.

3. Brief Summary

The percentage of nickel recovered in the leaching process is influenced by variations in temperature and leaching time. The increase in temperature and leaching time caused an increase in the percentage of leached nickel extraction. From the 3-factor ANOVA analysis, it is known that all variables are significant to the percent nickel extraction with the most influential variables are temperature and leaching time. The results of the leaching kinetics analysis showed that the control of the rate of the leaching process, namely diffusion through the solid product layer. The calculation use ANOVA is more accurate than using conventional method such as 'excel', and it needs more developing for mineral extraction in future.

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КІНЕТИЧНІ ВЛАСТИВОСТІ ВИЛУГОВУВАННЯ НІКЕЛЮ МЕТОДОМ ДИСПЕРСІЙНОГО АНАЛІЗУ (ANOVA)

Гідрометалурґійне добування латериту нікелю є більш ефективним з точки зору споживання енергії, оскільки виділяє менше вихлопних газів у порівнянні з пірометалурґійним методом. Таким чином, гідрометалурґійний метод може ще більше збільшити видобуток низькоякісного нікелевого латериту. У розрахунку за методом дисперсійного аналізу (ANOVA) використовуються три чинники для визначення важливости змінних і порядку найбільш впливових змінних. Метод ANOVA також є тестуванням (перевіркою) статистичної гіпотези, де робляться висновки на основі статистичних даних або груп. «Нульова» гіпотеза тесту ANOVA полягає в тому, що дані є простими випадковими для однієї сукупности; тому вони мають однакове очікуване середнє значення та дисперсію. Крім того, для визначення реґулятора швидкости реакції проводять дослідження кінетики вилуговування з використанням моделі «усадного ядра». Результати показали, що час вилуговування відіграє важливу роль кислоти, основи та моноглутамату натрію у збільшенні швидкости добування нікелю. Виходячи з результатів дисперсійного аналізу, двома найбільш впливовими чинниками є температура та час вилуговування. Використання розрахунку на основі методу ANOVA є більш точним, аніж використання традиційного методу, такого як ‘excel’, і потребує подальшого розвитку для видобутку корисних копалин у майбутньому.

Ключові слова: вилуговування, кінетика, дисперсійний аналіз (ANOVA), Ni.

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