IJCA
Indonesian Journal of Chemical Analysis
Homepage: https://journal.uii.ac.id/IJCA

Optimization of Fe Leaching Method for Indonesia Laterite Rocks using Experiment Design Central Composite Design (CCD)-Response Surface Methodology (RSM)

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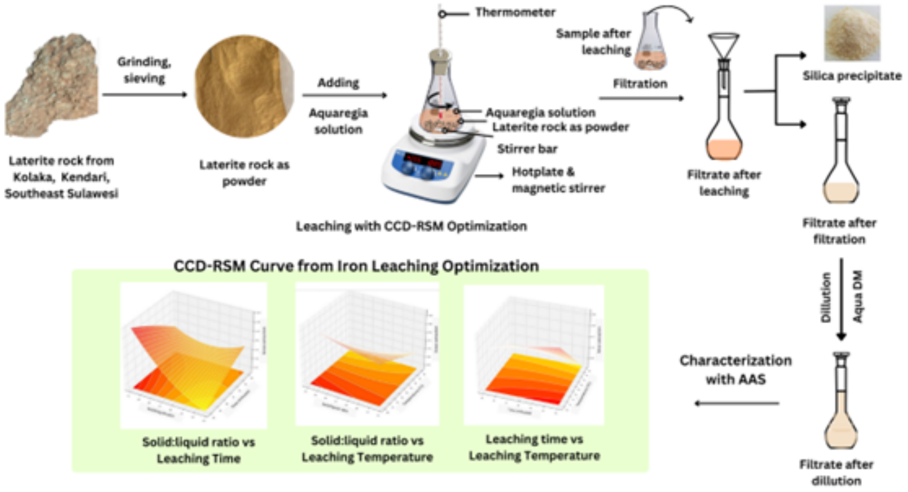
DOI: 10.20885/ijca.vol8.iss1.art5

GRAPHICAL ABSTRACT



ARTICLE INFO

Received : 14 January 2025
Revised : 20 February 2025
Published : 30 March 2025
Keywords : *Iron, leaching, laterite, CCD-RSM*

ABSTRACT

This work aims to use the experimental designs of the CCD (Central Composite Design) and RSM (Response Surface Method) to optimize the iron extraction process from laterite rocks in Kolaka, Southeast Sulawesi, Indonesia. In laterite rocks, the mineral components are identified using XRF testing. The XRF study indicates that the iron concentration of the laterite rocks is 68.91%, so the remaining metal content must be removed by leaching. Modifications in the leaching process affected the amount of iron removed, explicitly altering the S/L ratio, leaching time, and leaching temperature. Atomic Absorption Spectroscopy (AAS) measures the iron concentration and evaluates the leaching process outcomes. Using CCD-RSM to find the best process parameters, the best conditions are found at a 1:1 S/L ratio, a 30-minute leaching time, an 80°C leaching temperature, and a 19.18% iron extraction. Subsequent studies can further enhance the extraction outcomes using more ideal leaching parameters. This study makes the tests more useful by showing not only the results of the Fe extraction but also how well aqua regia works, which isn't usually used for Fe metals in a short amount of time at atmospheric pressure.

1. INTRODUCTION

Laterite rocks are among the many minerals found in Indonesia that are plentiful and different. In Indonesia, laterite rocks are distributed in Papua, Kalimantan, Sulawesi, Maluku and Halmahera [1]. The metallic mineral laterite is produced when rocks weather chemically, leading to secondary and residual enrichment of Ni, Fe, Mn, and Co elements [2]. Laterite rock has the highest iron content among other components [3]. Therefore, an efficient technique of processing iron in laterite rock is required to minimize waste generated during the iron separation process.

Hydrometallurgy and pyrometallurgical extraction techniques are two ways to treat iron in laterite rocks [4]. Pyrometallurgy is the process by which high-pressure steam precipitates the ash of a mineral. Hydrothermal technique uses a vacuum or pressure to extract a mineral's hydration phase [5]. The hydrometallurgical method is considered more economical than the pyrometallurgical method because if processed by the pyrometallurgical method, the iron produced has low quality [6]. Pyrometallurgy requires instruments with energy consumption, and maintenance costs are higher. In this study, a hydrometallurgical extraction process is used to reduce the process of metal extraction by pyrometallurgy.

The leaching process, which includes using acid solvents to extract metals, is typically used in research utilizing the hydrometallurgical approach [7]. Due to its complete extraction capabilities against metals like iron, nickel, and titanium, aqua regia is one of the acid solvents utilized [8]. Aqua regia acid is used in this investigation because it is thought to be highly reactive and can dissolve iron more quickly and effectively. Parameter monitoring is required to guarantee the effectiveness and success of the leaching process, which uses solvents including nitric acid, sulfuric acid, and aqua regia [9]. The metal extraction rate is one of the characteristics that must be employed [10]. Leaching time, solvent concentration, and temperature are other variables that need to be considered to optimize the leaching process [11].

Several previous experiments have been conducted on the extraction of Fe from laterite rock samples. Previous research was conducted with hydrochloric acid and the addition of aliquot 336, obtaining iron extraction results of 97.17% [12]. However, the leaching process does not use temperature, even though temperature is a factor that affects the extraction results. The research was conducted on the leaching of laterite rock with sulfuric acid using the HPAL method at temperatures of 1000-1200°C for 2 hours, achieving a Fe extraction percentage of 64.15% [13]. Another experiment was conducted with nitric acid as a leaching agent under high temperature, 180°C, pH 1.0, and time of 30 min. The experiment results obtained a percentage of Fe extraction of 99.49% [14]. However, this experiment uses high pressure and temperature, which requires high costs.

In previous studies, experiments optimized each process parameter. Earlier optimization methods like One Factor at a Time (OFAT) only evaluated specific parameters at specific periods. This method could not explain parameter interactions even though they can affect optimization results [15]. CCD (Central Composite Design) and RSM (Response Surface Method) have been employed recently. By detecting parameter interactions and utilizing fewer experimental data while still achieving the best findings, RSM increases the efficacy and efficiency of research [16]. This study employs the CCD-RSM technique to recover iron from laterite rocks with the most outstanding results.

2. EXPERIMENTAL METHODS

2.1. Material and Methods

Laterite rocks from Kendari, Kolaka, Southeast Sulawesi, SAP Chemicals' Aqua regia solvent (HCl 37% and HNO_3 65%)(1:3, v/v), and aqua demineralization as the solvent were the materials utilized in the study. Laterite rocks were sieved over a 200 µm mesh screen to standardize particle size. Atomic Absorption Spectroscopy was then used to quantify the metal concentrations in the laterite rocks, while X-ray fluorescence was used to analyze the metal content [17] qualitatively. The successful iron concentration extracted from the leaching process is further determined by characterization using Atomic Absorption Spectroscopy (AAS) [18].

2.2. Optimization of Laterite Rocks Leaching Using CCD-RSM

The leaching procedure is used to remove Fe from laterite rocks. At three distinct solid-to-liquid ratios—1:1, 1:2, and 1:3—Aqua regia solvent leaches laterite rocks. The material must be heated to a certain temperature and held there for a predetermined amount of time. The Erlenmeyer flask is swirled at 400 rpm using a magnetic stirrer during the embedding process. Table I indicates the connection among the solid-to-liquid (S/L) ratio, leaching time, and leaching temperature as they vary during this study.

TABLE I. Variables and levels of laterite rocks iron leaching

Variables	Levels		
	-1	0	1
Solid : fluid ratio (S/L, x_0)	1:1	1:2	1:3
Leaching time (minutes, x_1)	30	60	90
Leaching temperature (°C, x_2)	40	60	80

The optimization leaching of laterite rocks was designed by Central Composite design, and the experimental design with 22 runs of different variables is shown in Table II.

TABLE II. Central composite design for laterite rocks iron leaching

Run	Solid:liquid ratio (x_0)	Leaching time (x_1)	Leaching temperature (x_2)	Run	Solid:liquid ratio (x_0)	Leaching time (x_1)	Leaching temperature (x_2)
0	-1	-1	-1	11	0	0	0
1	-1	1	-1	12	0	-1	0
2	-1	-1	1	13	0	1	0
3	-1	1	1	14	0	0	-1
4	1	-1	-1	15	0	0	1
5	1	1	-1	16	-1	0	0
6	1	-1	1	17	1	0	0
7	1	1	1	18	0	0	0
8	0	0	0	19	0	0	0
9	0	0	0	20	0	0	0
10	0	0	0	21	0	0	0

After leaching, the Fe and aqua regia mixture is filtered to generate filtrates and sediments. The filtrate from the leaching is diluted with aqua demineralization in a 100 mL measuring flask. AAS measures the Fe concentration following the leaching process by creating a calibration curve of the Fe standard solution. After introducing the filtered filtrate samples into the AAS and measuring the absorbance, Equation 1 was used to get the percentage of Fe extraction [19].

$$\text{Iron extraction (\%)} = \frac{\text{Iron concentration after leaching (ppm)}}{\text{Iron concentration in laterite rocks (ppm)}} \text{ x } 100\% \quad (1)$$

3. RESULTS AND DISCUSSIONS

3.1. Characterization of Laterite Rocks

XRF analysis is performed on the laterite rocks before the leaching procedure to determine their chemical composition [20]. Figure 1 displays the XRF analysis results.

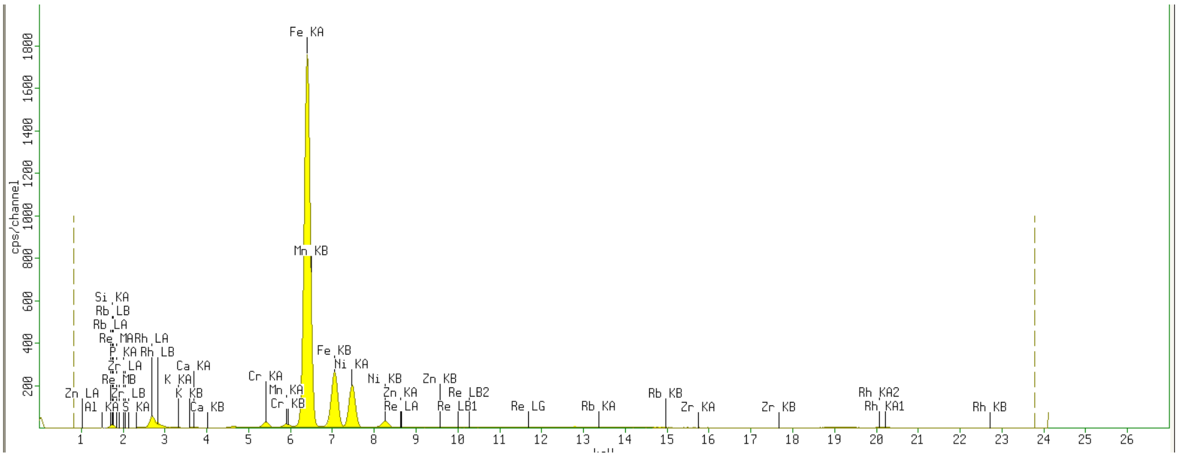


Figure 1. XRF analysis of Kendari laterite rock

Iron is the most prevalent metal in laterite rocks, with the largest peak on the XRF result curve compared to other elements. The rock's iron content was determined to be 68.91% based on the results of XRF analysis. Most laterite is formed from iron-rich rocks such as hematite and goethite, which are red, yellow, or brown [21].

Additional testing of laterite rocks is done using XRD. XRD analysis is performed to identify the mineral. The Sepuluh Nopember Institute of Technology Surabaya's Department of Materials Engineering and Metallurgy conducts XRD testing. 2θ: 20-90° is the angle at which XRD testing occurs. The XRD data show several peaks, some of which are rather intense. The peak indicates that iron is present as Fe_2O_3 and Fe_3O_4. At 2θ = 24.23°, 33,32°, 35,73°, and 72.09°, which is the peak of Fe_2O_3 [22]. Furthermore, Fe_3O_4 exhibits peaks at 2θ = 30.19° and 53.36° [23]. The XRD diffractogram for laterite rock is shown in Figure 2.

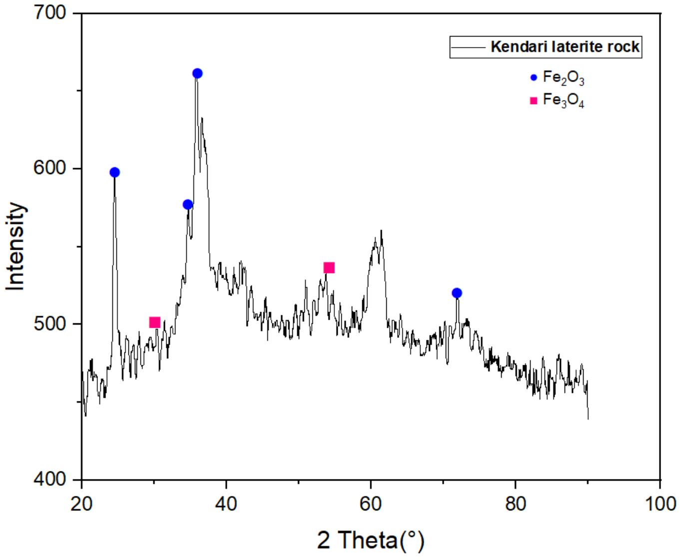


Figure 2. XRD diffractogram for laterite rock

2.3. Optimization of Laterite Rocks Leaching Using CCD-RSM

The leaching method uses aqua regia to separate iron from laterite rocks because this acid is considered very reactive and can dissolve iron quickly and efficiently. This method was carried out with parameters varying in the ratio of fluid solids (S/L), leaching time (minutes), and leaching temperature (°C). These parameters are used because they can affect the quality and amount of iron obtained. This variation aims to determine the effect on iron extraction efficiency [24].

The selection of S/L ratio variations in this study was at S/L ratios of 1:1, 1:2, and 1:3. The selection of these variations was made due to the experimental approach in exploring the effect of the ratio on reaction efficiency. The variation of the S/L ratio 1:1 in that variation was chosen to represent the influence of using a relatively high solvent concentration so that the leaching process could proceed more quickly while still considering the potential waste of chemicals. The variation of the S/L ratio 1:2 provides a good balance between solvent strength and solvent volume, allowing the metal to dissolve efficiently while avoiding excessive dilution. At the same time, the more dilute conditions in the leaching process can increase the diffusion of metal into the solution for an S/L ratio of 1:3.

The selection of leaching times of 30, 60, and 90 minutes is based on optimal conditions from previous studies. The study on the leaching of iron from laterite rocks using sulfuric acid showed optimal results in 60 minutes [25]. Nitric acid is also used as a leaching agent for iron and achieves the same optimum leaching time of 60 minutes, with a Fe extraction percentage of 56.53% [26]. The optimum leaching time reached 60 minutes based on previous research, which was used as the study's middle-value variation (level 0). In comparison, other variations of soaking time were chosen around those optimum level conditions. Leaching times of 30 and 90 minutes were aimed at testing the effect of decreasing and increasing leaching time on leaching results by knowing any significant changes in conditions outside the optimum.

The leaching temperature in the atmospheric acid leaching method for extracting iron typically did not rise over 95°C [27]. This is the main factor in selecting variations at 40, 60, and 80°C temperatures in this study. Temperature variation selection is also supported by previous research, which says that the optimum Fe leaching conditions are in the range of 60-90°C with a percentage of Fe reaching 70% [28]. In contrast, the selection of other temperature variations is a testing factor chosen within that optimum condition range.

Using a mortar and pestle, laterite rock is first crushed into powder for leaching. To achieve a consistent size, nickel ore powder is sieved via a 200-mesh sieve [19]. Better contact between the laterite rock particles and the Aquaregia solution can be achieved by continuously swirling at 400 rpm. Continuous stirring speeds up iron dissolving by causing the solid particles to constantly move and come into contact with the solvent.

The Aqua regia leaching method dissolves iron in laterite rock by forming iron ion complexes in solution. Aqua regia, a mixture of HNO_3 and HCl in a 1:3 ratio, can dissolve heavy metals like iron (Fe).

The leaching process of iron from laterite rocks with aqua regia, which is made from a mixture of hydrochloric acid and nitric acid, results in oxidation reactions and the formation of soluble complexes. Aqua regia can act as a strong solvent for iron due to the presence of nitric acid, which has a high oxidizing ability. Iron will be oxidized to Fe^{2+} or Fe^{3+} ions, which bind to chloride ions (Cl-) from HCl. From the binding of these ions, complex compounds in the form of $FeCl_2$ and $FeCl_3$ are formed, increasing iron solubility in the solution. In addition, nitrogen dioxide gas is also formed as a byproduct of the reaction due to the reduction of nitric acid as it becomes an oxidizing agent. The process is based on the chemical equation from Equation 2.

$$\mathrm{Fe_{(s)} + 4HNO_{3(aq)} + 12HCl_{(aq)} \rightarrow 3FeCl_{2(aq)} + 4NO_{2(g)} + 4H_2O_{(l)}} \quad (2)$$

The sample's concentration or content of iron (Fe) is measured with AAS. It can calculated using the regression line equation $Y = 0.0133x - 0.0021$ obtained from the calibration curve of the $Fe(NO_3)_3$ standard solution. From the regression line equation, Y is the absorbance of the sample. By substituting the absorbance value (Y) of each sample, we will obtain a value of (X), which is the concentration of iron (Fe) in each sample and also obtain percent extraction by calculating using Equation 1. The results of iron concentration and %Iron extraction are shown in Table III. From the data in Tabel III, the concentration of iron (Fe) in Run 2 with a fluid-solid ratio (S/L) of 1:1, the leaching times for 30 minutes, and the temperature of 80°C indicate the level of the highest iron extraction percentage, with the value of 19.18%. In the CCD test design, replication was carried out 3 times using the same test variations in experiments 19, 20, and 21. Testing with this replication can ensure the reliability and consistency of the experiments. In addition, regression testing was also conducted to validate the research data. From the three replications, there are 2 data in the 19th and 21st experiments with almost similar values, namely the acquisition of Fe extraction percentages of 16.74% and 16.67%. Besides, the p-value is below 5%, so there is no significant difference, and the results are considered consistent.

TABLE III. Iron concentration and percentage of iron extraction from leaching process

Run	Iron concentration (ppm)	Iron Extraction (%)	Run	Iron concentration (ppm)	Iron Extraction (%)
0	3.68	14.73	11	4.16	16.64
1	4.17	16.67	12	4.37	17.50
2	4.80	19.18	13	4.28	17.14
3	4.74	18.95	14	4.05	16.21
4	4.29	17.14	15	4.63	18.52
5	4.19	16.75	16	4.48	17.89
6	4.60	18.41	17	4.26	17.03
7	4.52	18.08	18	4.22	16.87
8	4.67	18.70	19	4.19	16.74
9	4.67	18.64	20	4.52	18.09
10	4.45	17.81	21	4.17	16.67

Response surface analysis was then used to optimize the extraction results, which were developed using the Central-Composite experimental design made with Design Expert version 13 software. This stage aimed to identify the ideal range for the iron leaching tests on laterite rocks [29]. The optimized research results were the source of the Ordinary Least Squares (OLS) findings. If the OLS test finds a connection, it can mean that the two data sets are related. According to the OLS test, the p-value is less than 0.05, lower than the 5% significance level. The correlation between the iron extraction percentage response and the research variables (solid: liquid ratio, leaching period, and leaching temperature) is shown by the p-value [30]. The polynomial equation's statistical parameters are displayed in Table IV.

TABEL IV. OLS regression for iron leaching from laterite rocks

Dep. Variable :	Iron concentration	**R-squared :**	0.713
Model :	OLS	**Adj. R-squared :**	0.498
Method :	Least Squares	**F-statistic :**	3.319
Date :	Tue, 02 Jul 2024	**Prob (F-statistic) :**	0.0282
Time :	15:48:52	**Log-Likelihood :**	-18.397
No. Observations :	22	**AIC :**	56.79

Df Residuals :	12	**BIC :**	67.70
Df Model :	9		
Covariance Type :	nonrobust		

	coef	**std err**	**t**	**P>|t|**	**[0.025]**	**[0.975]**
Const	16.1932	3.955	4.095	0.001	7.577	24.810
x1	-7.5015	8.053	-0.932	0.370	-25.048	10.045
x2	0.0172	0.069	0.251	0.806	-0.132	0.167
x3	0.0440	0.141	0.311	0.761	-0.264	0.352
x4	0.8932	5.516	0.162	0.874	-11.125	12.911
x5	0.0316	0.026	1.217	0.247	-0.025	0.088
x6	0.0737	0.039	1.893	0.083	-0.011	0.159
x7	-7.324e-05	0.001	-0.145	0.887	-0.001	0.001
x8	00004	0.000	-0.986	0.344	-0.001	0.001
x9	-4.979e-05	0.001	-0.044	0.966	-0.003	0.002

Ordinary Least Square (OLS) results indicate that while the model's R-squared value (0.713) is pretty good, several of the input variables employed as estimators do not significantly affect the iron percentage, as indicated by a lower R-squared adjusted value (0.498). However, because the value is less than 0.05, the Prob value (F-statistic) is 0.0694, indicating that several input variables substantially impact output variables at the 5% significance level. The second-order polynomial equations obtained can be seen in Equation 3.

$$Z = 16.193245337022578 - 7.501492X_0 + 0.017235X_1 + 0.043960X_2 + 0.893211X_0^2 + 0{,}031602X_0X_1 + 0{,}073741X_0X_2 - 0{,}000073X_1^2 - 0{,}000439X_1X_2 - 0{,}000050X_2^2 \tag{3}$$

The variation in test parameters has several different types of significance that can be determined from the p-value. Variations of test parameters play a significant role in Fe leaching if the p-value is <0.05. If the p-value is in the range of 0.05-0.1, it is pretty substantial. If the p-value is more than 0.1, the test parameters do not significantly affect Fe leaching [31].

Table IV has several test constants (const, x1-x9). The test constants are variables included in the regression model to observe their effect on the response variable. Those variables are the main parameters used in testing or the interaction between several main factors used in the research. Const. x1-x9 are the variables from the polynomial regression from Equation 3, respectively. From the regression results, const. x6 has a positive regression coefficient, indicating that const. x6 is a factor that enhances iron leaching [32]. Const. x6 is the interaction of the main variables, namely X0 and X1, which are the S/L ratio and temperature. Thus, the higher the interaction between S/L and temperature, the more the iron extraction efficiency increases. In addition, the P>|t| value, which is the same as the p-value of const. x6, also has a value of 0.083, which supports that const. x6 plays a quite significant role in iron leaching.

Based on the regression results, the R^2 value of 0.713 indicates that the resulting study can represent 71.3% variation after Fe extract extraction. This suggests that the data is highly accurate and provides a good correlation between the test variables and the percentage of Fe extraction.

However, the low adjusted R^2 value (0.498) indicates that some variables in the model do not contribute significantly to the prediction of Fe extraction. Furthermore, from Table IV, const. x4 and x6 have high coefficients (0.8932 and 0.0737), representing data on the effect of the S/L ratio and the interaction between the s/l ratio and temperature, indicating that the test variables influence Fe extraction. While other variables still do not have a significant effect, the Fe extraction rate is still low, 19.28%. This research provides future optimization opportunities by adjusting the S/L ratio, leaching time, and temperature. Data from this study can be the basis for further development related to the Fe leaching process from laterite rocks using aqua regia.

The RSM curve for this study is presented in Figure 3. From the RSM curve, the optimum area is marked by the yellow area. In addition, the concave curvature of the curve also indicates the optimum area. Figure 3a indicates the vital connection between the solid: liquid ratio (S/L) and the leaching time for Fe extraction. The proportion of Fe extraction increases dramatically after 30 minutes of leaching time. The extraction percentage is optimum at an S/L ratio of 1:1 and reduced significantly, as seen by the ratio being more than 1:1. This can also be observed from the yellow-colored area of the RSM curve, indicating the optimum value for Fe extraction.

Additionally, the RSM curve demonstrates that a S/L ratio above 1:1 (e.g., 1:2 and 1:3) results in a notable decline in the percentage of Fe extraction. This is because an excessively high solvent ratio can dilute the Fe ions released from laterite rocks in the solution, decreasing the effectiveness of the ongoing dissolving process. A low concentration of iron ions in the solvent may prevent the reaction's optimum leaching from occurring [33]. From the S/L ratio, the variation of solid and liquid has differences in aqua regia volume, thus providing accurate information on the effectiveness of aqua regia use in the leaching process. The amount of solids is fixed because more solids will potentially increase the presence of other confounding materials besides iron contained in rocks that can interfere with the leaching process [34].

Figure 3b shows the correlation between the leaching temperature and the solid: liquid ratio (S/L). At temperatures above 40°C, the percentage of Fe extracted escalated [35]. Since high temperatures aim to break down and dissolve difficult-to-extract particles into the leaching liquid, higher leaching temperatures can hasten the leaching rate [36]. High temperatures can also speed up metal corrosion [37]. Additionally, higher temperatures can make iron ions more soluble in the solution, enabling more iron to be drawn out of laterite rocks and added to the solvent [38]. However, the Fe extraction percentage significantly decreases when the S/L ratio rises above 1:1 (e.g., 1:2 and 1:3). This is because an excessively high solvent ratio can dilute the Fe ions released from the laterite rocks in the solution, decreasing the effectiveness of the subsequent dissolution process. A low concentration of iron ions in the solvent may prevent the reaction's optimal leaching from occurring [39].

Figure 3c indicates the correlation between the leaching time and leaching temperature. From the curve, it was found that the leaching time during the first 30 minutes with a temperature increase above 40°C can accelerate Fe extraction. However, leaching time longer than 30 minutes often has a much lower extraction percentage. This can be explained by the fact that a more extended period may induce materials to deteriorate or key extraction components to be lost, which could lower the extraction percentage [40]. Temperatures above 40°C significantly raise the percentage of iron removed. Raising the temperature can potentially accelerate chemical processes and the extraction percentage [41]. This is consistent with the theory of chemical reactions, which states that temperature directly affects reaction rate.

Overall, all test parameters have a strong interaction relationship, namely the S/L ratio, leaching time, and leaching temperature. High temperatures can accelerate the leaching reaction. On the other hand, an excessively high S/L ratio can hinder the leaching process due to the dilution effect that impedes the leaching process. In addition, leaching time is also a test factor that influences the process, so it must be optimized to prevent a decrease in efficiency due to material degradation.

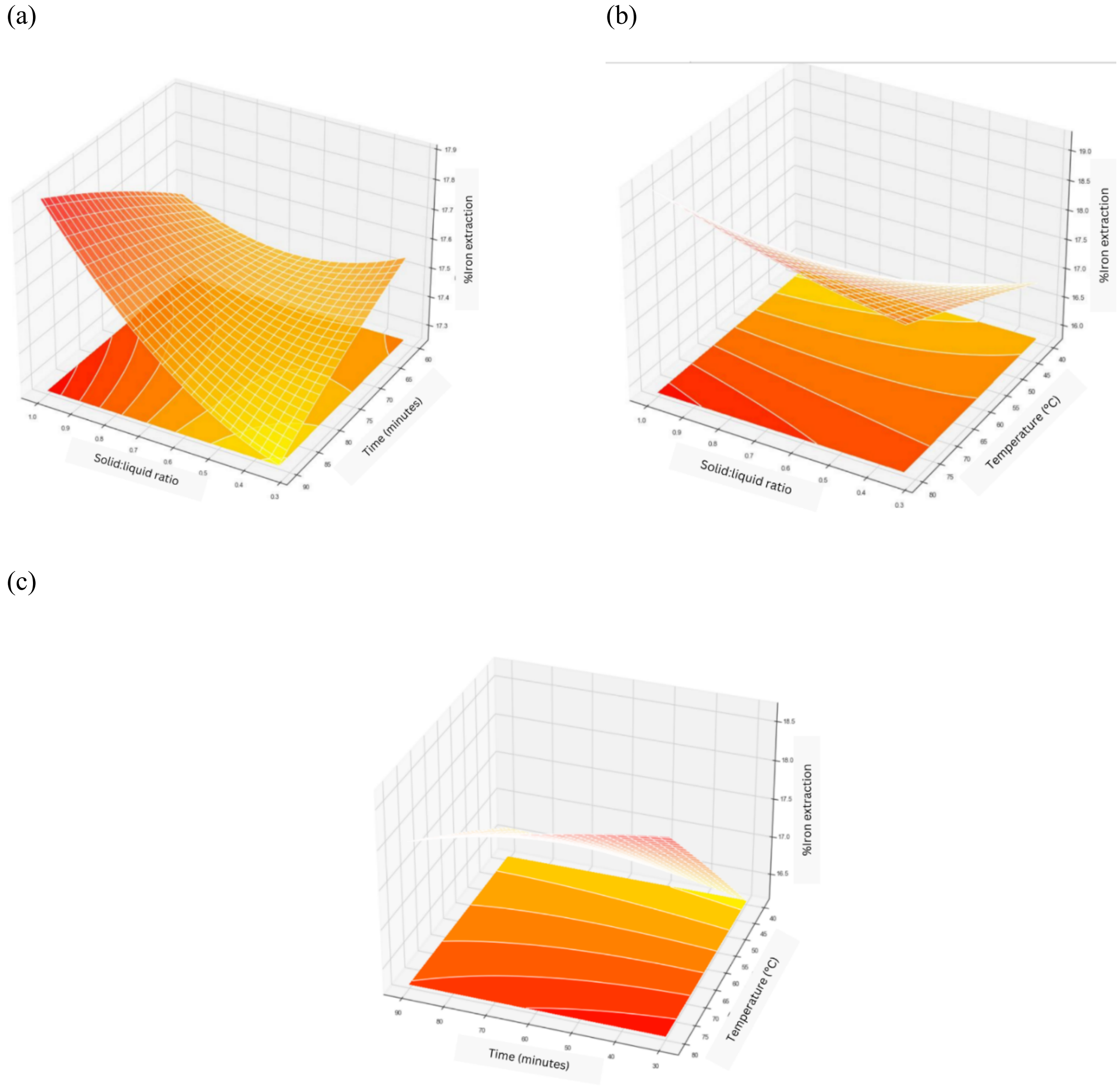


Figure 3. RSM curve laterite rocks iron leaching

The research results show that using aqua regia as a leaching agent impacts the leaching results of iron from laterite rock. Generally, aqua regia is rarely employed in the leaching process of iron from laterite rocks. Aqua regia is used to extract high-value metals such as gold and platinum. However, aqua regia has the potential for the Fe leaching process because aqua regia has very strong solubility, making it easier for Fe metal to be released by forming stable Fe complexes, as shown in the chemical equation from Equation 2. Aqua regia is a compelling mixture for dissolving precious metals, including iron, in laterite rocks [10].

The optimum results of the leaching process in this study were obtained at an S/L ratio of 1:1, a leaching time of 30 minutes, and a temperature of 80°C with a percentage of Fe that was successfully obtained at 19.18%. Although the percentage of Fe leached is low, this figure is sufficient for the initial stage in assessing the feasibility of the process and the leaching parameters of Fe from laterite rock. In addition, this study does not require high pressure in its process, making it more efficient, with a leaching time of 30 minutes and low temperature. This result can also be a reference in further optimization, such as adjusting operating conditions, using additional reagents, or modifying other test parameters, such as increasing the S/L ratio, temperature, and leaching time to improve the percentage of Fe extraction further. The following laboratory-scale tests can provide validation of theories and assumptions that are useful for potential future research innovations in the extraction process.

4. CONCLUSIONS

Using the CCD-RSM experiment design, a leaching method was used to separate iron elements (Fe) in laterite rocks. According to the study's findings, ideal conditions are achieved at a fluid-solid ratio (S/L) of 1:1, leaching time of 30 minutes, and leaching temperature of 80°C. The iron extraction (Fe) percentage with the highest figure was 19.18%. These results show how well the CCD-RSM technique works to maximize iron leaching from laterite rock, which can reduce the likelihood of hazardous waste and increase iron extraction efficiency.

Acknowledgement

The authors are grateful to the Institut Teknologi Sepuluh Nopember for providing financial support for this work under the Publication Writing and Intellectual Property Rights Incentive Program (PPHKI) 2025.

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