Nickel Extraction from Kolaka Laterite Ore: A HCl Leaching Optimization Method Using the BBD-RSM and Recovery via Oxalate Precipitation

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ABSTRACT

This study examines the extraction of nickel from laterite ore, using HCl solution. In order to get better results, the Box-Behnken Design Response Surface Method (BBD-RSM) was applied and the laterite ore was analyzed with X-ray Fluorescence (XRF) and X-ray Diffraction (XRD). During the experiment, the concentration of HCl, the reaction time, and the temperature were changing in order to achieve the best extraction. The products were purified through a two-stage procedure, using 25% of ammonium hydroxide (NH_4OH) and oxalic acid ($H_2C_2O_4$) 2 M. The best outcome was at an 8 M concentration of HCl, an extraction time of 240 min, and 100^o C.

Keywords-nickel; laterite ore; nickel HCl leaching; Response Surface Methodology (RSM); Box-Behnken Besign (BBD)

I. INTRODUCTION

In 2023 Indonesia held the 50% of the world's nickel production, providing 1.8 million tons of nickel. The Indonesian Ministry of Energy and Mineral Resources indicate that nickel reserves reached 52% of the global total in 2020, primarily located in eastern regions, including Papua, Maluku, and Sulawesi [1]. Nickel is used in various industrial processes, including battery production, ceramic mixtures, and stainless-steel production. It is estimated that the use of nickel products as raw materials for batteries will have been increased by 30% by 2040 [2]. Nickel products are processed through smelting, which produces impure nickel because iron and other metallic elements remain in the final product. Therefore, effective processing technology is needed to obtain purer nickel [3]. Indonesia's nickel production prior to 2019 was not economically viable because nickel was mainly exported as raw ore, to China. Different processing methods are used depending on the ore's nickel content. Low-grade ores are processed hydrometallurgically in chemical solutions at comparatively low temperatures, while high-grade ores are processed pyrometallurgically at higher temperatures [4]. Hydrometallurgical processing presents significant challenges. This method is commonly used in the Caron process, High-Pressure Acid Leaching (HPAL), and Atmospheric Pressure Acid Leaching (APAL). The Caron and HPAL processes are less efficient due to their high energy and cost requirements [5]. The APAL process, which operates at atmospheric pressure, provides a more economical and effective way to refine nickel. In this process, laterite ore is extracted using sulfuric, hydrochloric, nitric, and acetic acids agents. Changing temperature, reaction time, and acid concentration significantly influences nickel recovery, in order to achieve optimal results [6]. Various lubricants are used to optimize nickel extraction. In the past, sulfuric acid was utilized to process nickel pebbles from Sorowako. During the smelting process, 4.5 M sulfuric acid is combined with nickel pebbles that contain 1.5% nickel. The mixture is stirred for 24 h at 95 °C using a magnetic stirrer set to 400 rpm. Authors in [7] showed that after 12 h of distillation, the nickel recovery rate was 90.01%, indicating ideal extraction conditions. An additional nickel extraction experiment involved mixing nickel-containing rocks (1.74%) with a solution of lactic acid, citric acid, and gluconic acid at a ratio of 1:2:3. This resulted in a total concentration of 3.18 mol/L. This process provided 25.5% nickel, with solid-to-liquid ratio of 1:10, pH at 0.5, stirring speed at 386 rpm, temperature at 75 °C, and distillation time at 120 min [8]. The variety of fluxing agents used in nickel smelting is subject to constant change. Nickel extraction was carried out utilizing an NH_4HSO_4 lubricant mixed into 1.56% nickel-containing rock. According to [9], the ideal extraction parameters were 75% NH_4HSO_4 by mass, a temperature of 115 °C, and a reaction time of 90 min. The nickel extraction percentage was 88.96%. A nickel extraction percentage of 75.0% was achieved through nickel smelting. This was accomplished by mixing nickel rock containing 1.98% nickel with $FeCl_3$ at a liquid-to-solid ratio of 1:0, a melting temperature of 150 °C, and a reaction time of 1.5 h [10]. Additionally, nickel rock extraction was performed in a 6M HCl solution containing 1.37% nickel. After 180 min, the nickel extraction percentage was 97.22% [11]. According to [12], HCl has been used as a leaching agent in nickel extraction processes. These parameters were evaluated employing the One Factor at a Time (OFAT) method and other earlier optimization techniques. However, this approach could not explain parameter interactions, which can influence optimization results. BBD and RSM have been also applied [13]. RSM identifies parameter interactions and achieves optimal results with fewer data points [14].

II. EXPERIMENTAL PROCESS

The chemical agents used in this study include 37% hydrochloric acid (HCl) and 25% ammonium hydroxide (NH_4OH), both from SAP Chemical. All reagents and solutions were prepared using Aqua DM as the solvent. The laterite ore shown in Figure 1 was sieved using a 200-mesh sieve to standardize the particle size [15]. Then, the laterite ore was tested using Atomic Absorption Spectroscopy (AAS, iCE 3000 series) to measure the metal content of the samples [16]. XRF, PANalytical was used to examine the nickel-containing minerals in the laterite ore samples [17], and XRD, PANalytical X'Pert PRO was deployed to determine the different phases of the minerals [18].

Fig. 1. Laterite ore from Kolaka, Sulawesi Tenggara.

The nickel content of the laterite ore was extracted through leaching. To accomplish this, 2.5 grams of the ore were mixed with 25 mL of an HCl solution at a 1:10 (w/v) ratio [19]. The leaching process involved heating the mixture at specific temperatures for designated time intervals. This process was carried out in batches, with stirring at 400 rpm using a magnetic stirrer [20]. The optimal conditions for nickel extraction were determined using the BBD method combined with RSM. BBD serves as the basis for the experimental design, and RSM is the result of optimization [21]. Python software was used to create the BBD, perform the regression analysis, and generate the 3D surface response diagrams [22]. Table I displays the variations in HCl concentration, reaction time, and temperature. Nickel extraction optimization was determined using a BBD created with Design-Expert 13 software, as portrayed in Table II.

TABLE I. VARIABLE AND FACTOR LEVELS FOR NICKEL EXTRACTION

Variable	Factor Level		
	-1	*0*	*+1*
Concentration of HCl (M, x_0)	6	8	10
Reaction time (min, x_1)	120	180	240
Temperature (°C, x_2)	80	90	100

TABLE II. BOX BEHNKEN DESIGN FOR NICKEL EXTRACTION

Run	**Concentration of HCl (M, x_0)**	**Reaction time (min, x_1)**	**Temperature (°C, x_2)**
1	-1	-1	0
2	-1	0	-1
3	-1	0	1
4	-1	1	0
5	0	0	0
6	0	-1	-1
7	0	-1	1
8	0	0	0
9	0	0	0
10	0	1	-1
11	0	1	1
12	1	-1	0
13	1	0	1
14	1	0	-1
15	1	1	0

After leaching, the nickel-HCl mixture was allowed to settle, producing a filtrate and a precipitate. The filtrate was diluted with deionized water, and 25 mL of the diluted filtrate was mixed with a 25% NH_4OH solution. Ammonium hydroxide was added until the mixture's pH reached 8. The mixture rested for 20 min to allow a precipitate to form. Then it was filtered again to separate the filtrate from the precipitate that formed. The resulting filtrate was analyzed using an AAS to determine its concentration values based on the regression equation derived from the calibration curve [23]:

$$\%\text{Extraction}= \frac{[\text{Ni}]\text{filtrate (ppm)}}{[\text{Ni}]\text{ laterite ore (ppm)}} \text{ x } 100\% \qquad (1)$$

Oxalic acid, used as a nickel-precipitating agent [24], is produced by mixing solid oxalic acid with deionized water and heating the mixture to 70°C while stirring [25]. The filtrate from the leaching process is separated from the RES-Fe and then mixed with 2 M oxalic acid until the pH reaches 2. The mixture is then filtered, and the precipitate (PRE-Ni) is collected and tested using XRF and XRD to determine the nickel content from the leaching process [26]:

$$\%\text{Recovery Ni} = \frac{[\text{Ni}]\ \text{laterite ore} - [\text{Ni}]\ \text{PRE-Ni filtrate}}{[\text{Ni}]\ \text{laterite ore}} \times 100\% \quad (2)$$

III. RESULTS AND DISCUSSION

A. Laterite Ore Characterization

XRF testing was conducted in order to identify the types and concentrations of elements in laterite nickel rock samples. The results provide qualitative data on the types of elements in the rock and quantitative data on their concentrations, as illustrated in Table III.

TABLE III. THE MAIN ELEMENTS OF KOLAKA LATERITE ORE

Sample	**Element (%wt)**			
	Ni	Fe	Si	Mn
Laterite ore	1.91	16.28	13.13	0.16

The XRF test results show that the laterite ore contains 1.91% nickel. However, iron (Fe) still comprises the majority of the ore at 16.28%. Therefore, the laterite nickel rock was tested further using XRD. This test was conducted to identify the type of iron minerals present in the rock [26]. The aim of this analysis was to determine the most suitable method for separating nickel from the iron content in the laterite ore. According to the XRD test results, peaks were observed at 2-theta angles of 24.20°, 33.22°, 35.47°, 41.07°, 49.98°, and 57.61°, which match the structure of Fe_2O_3 [22]. The high-intensity peak aligns with the XRF characterization results showing iron oxide to be the most concentrated mineral in nickel laterite rocks. Additionally, peaks at 18.57°, 22.08°, 35.50°, and 36.44° are associated with the NiO structure. A peak noted in the XRD diffractogram at a 2-theta angle of 11.91° indicates the presence of SiO_2 in the nickel laterite rocks, bound with nickel in the form of $Ni_3Si_2O_5(OH)_4$ [24], as presented in Figure 2.

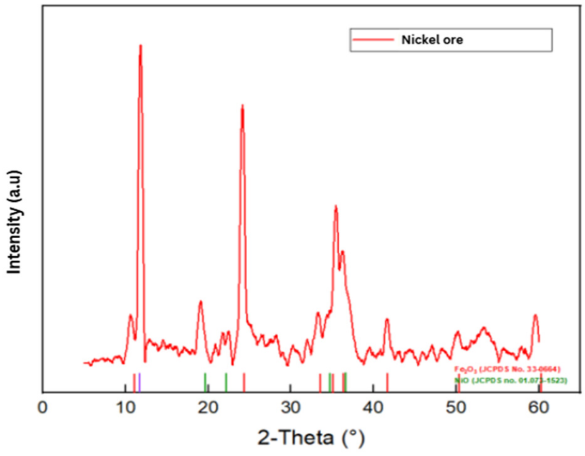

Fig. 2. XRD analysis of laterite ore.

B. Optimization of Laterite Ore Leaching Using BBD-RSM

During the leaching process, the nickel in the laterite ore dissolves and is separated from the impurities and unwanted materials. Preliminary tests showed that the laterite ore sample consisted mainly of iron. Hydrochloric acid (HCl) was used as the leaching solvent because it effectively reduces iron. When iron reacts with HCl, it is converted into iron chloride. Additionally, nickel, typically present in laterite ore as nickel oxide (NiO), dissolves in HCl, forming nickel (II) chloride:

$$Fe_2O_3\ (s) + 6HCl\ (aq) \rightarrow 2FeCl_3\ (aq) + 3H_2O\ (g) \quad (3)$$

$$NiO\ (s) + 2HCl\ (aq) \rightarrow NiCl_2\ (aq) + H_2O\ (g) \quad (4)$$

During the leaching process, 2.5 g of laterite ore were mixed with 25 mL of HCl solution in an Erlenmeyer flask, achieving a solid-to-liquid ratio of 1:10. HCl solutions with concentrations of 6 M, 8 M, and 10 M were used and the mixture was heated on a hot plate at 80 °C, 90 °C, or 100 °C for 120 min, 180 min, or 240 min. Next, the solution was mixed with 25% NH_4OH solution to precipitate the remaining iron. During this process, the pH of the solution was adjusted to 8.0 by adding 4–5 mL of 25% NH_4OH solution. The NH_4OH-treated solution was stirred with a magnetic stirrer to achieve homogeneity. At a pH of 8.0, the solution turned reddish-brown and solid particles began to form. Once homogeneity and a pH of 8 were achieved, the solution was left to stand without stirring for 20 min. Ammonium hydroxide is an effective precipitating agent for metals such as iron and aluminum:

$$FeCl_3\ (aq) + 3NH_4OH\ (aq) \rightarrow Fe\ (OH)_3\ (s) + 3NH_4Cl\ (g) \quad (5)$$

The solution was poured through filter paper and the resulting filtrate was analyzed using AAS to determine the nickel content after leaching and the iron content of the leaching water. Nickel leaching was performed using a Box–Behnken experimental design, created with Design-Expert 13 software. The experiment was performed 15 times with varying concentrations of HCl, leaching times, and temperatures, as shown in Table IV.

TABLE IV. CONCENTRATION OF NICKEL FROM LATERITE ORE LEACHING

Run	**Coded variable levels**			**Concentration (ppm)**	**Extraction (%)**
	x_o	x_1	x_2		
1	-1	-1	0	378.61	38.58
2	-1	0	-1	463.10	47.19
3	-1	0	1	483.57	49.27
4	-1	1	0	425.55	43.36
5	0	0	0	454.75	46.34
6	0	-1	-1	402.91	41.05
7	0	-1	1	457.81	46.65
8	0	0	0	454.37	46.30
9	0	0	0	453.99	46.26
10	0	1	-1	425.74	43.48
11	0	1	1	490.01	49.93
12	1	-1	0	342.78	34.93
13	1	0	1	379.31	38.65
14	1	0	-1	398.38	40.59
15	1	1	0	366.88	37.38

The optimized results of the research provided an Ordinary Least Squares (OLS) test [27], as illustrated in Table V, which suggests that the compared data are interrelated. Based on these results, a p-value of less than 0.05 was determined, which is below the significance level ($\alpha < 5\%$). The p-value indicates the correlation between the nickel extraction response and the tested variables: HCl concentration, leaching time, and leaching temperature [28].

TABLE V. RESULT OF OLS TESTING FOR NICKEL CONCENTRATION FROM LEACHING

Dep. variable:		**Extraction (%)**	**R²**		**0.913**	
Model:		OLS	Adj. R²		0.757	
Method:		Least Squares	F-statistic:		5.845	
Date:		Mon, 23 Jun 2025	Prob(F-statistics):		0.033	
Time:		19:35:52	Log-Likelihood:		-25.28	
No. observations:		15	AIC:		70.56	
Df Residuals:		5	BIC:		77.64	
	coef	std err	t	$P>\|t\|$	0.025	0.975
const	77.565	112.573	0.689	0.521	-211.81	366.944
x_1	21.8838	7.146	3.062	0.028	3.514	40.253
x_2	0.3510	0.220	1.594	0.172	-0.215	0.917
x_3	-3.4002	2.194	-1.550	0.182	-9.039	2.238
x_4	-1.1341	0.294	-3.855	0.012	-1.890	-0.378
x_5	-0.0049	0.009	-0.520	0.625	-0.029	0.019
x_6	-0.0504	0.057	-0.891	0.414	-0.196	0.095
x_7	-0.0009	0.000	-2.725	0.042	-0.002	-5.04e-05
x_8	0.0004	0.002	0.210	0.842	-0.004	0.005
x_9	0.0216	0.012	1.835	0.126	-0.009	0.052
x_{10}	77.5650	112.57	0.689	0.521	-211.81	366.944
x_{11}	21.8838	7.146	3.062	0.028	3.514	40.253
x_{12}	0.3510	0.220	1.594	0.172	-0.215	0.917
x_{13}	-3.4002	2.194	-1.550	0.182	-9.039	2.238
x_{14}	-1.1341	0.294	-3.855	0.012	-1.890	-0.378
Omnibus:		0.106	Durbin-Watson:		2.440	
Prob (Omnibus):		0.949	Jarque-Bera (JB):		0.331	
Skew:		0.000	Prob (JB):		0.847	
Kurtosis:		2.272	Cond. No.		8.09e+06	

In this study, the p-value was 0.033, corresponding to the probability (F-statistic). This suggests that the tested variables are related to the nickel extraction rate. Additionally, the coefficient (R^2) test was measured, with a higher R^2 value indicating that the generated model is consistent with the data, presenting a strong association between the test factor and the response. The OLS test results provided a model equation that can be used to determine extraction percentage based on different test variations. This regression model indicates the response of %Extraction (Z):

$$Z = 77{,}5650 + 21{,}8838x_0 + 0{,}3510x_1 - 3{,}4002x_2 - 1{,}1341x_0^2 - 0{,}0049x_0x_1 - 0{,}0504x_0x_2 - 0.0009x_1^2 + 0{,}0004x_1x_2 + 0{,}0216x_2^2 \quad (6)$$

where x_0, x_1, and x_2 are the concentration of HCl (M), leaching time (min), and leaching temperature (°C), respectively. Meanwhile, Z is the response from these factors, which is the %Extraction. The coefficient of determination (R^2) obtained from the equation is 0.913. A value of R^2 approaching 1 indicates that the data fit is quite good [28]. Figure 3 shows the 3D surface response diagram, presenting the relationship between the test variables and the resulting nickel concentration from the leaching process. This diagram compares the nickel concentration resulting from the leaching process on the Z-axis with two test variables, while holding the other variable constant at level 0 [27]. The 3D graph displays several colored areas, with blue areas representing the lowest nickel concentration resulting from leaching, while red areas indicate an increasing nickel concentration. Figure 3 (a) is a 3D curve diagram demonstrating the interaction between HCl concentration (M) and solution temperature (°C) on leached nickel concentration. The curve shows that the optimal HCl concentration for nickel extraction is between 7.75 and 10 M. The HCl concentration of 8.75 M is at the center of the red area. However, at a concentration of 6 M, leaching is not optimal, so the curve is blue. At concentrations greater than 10 M, the leaching rate decreases as the curve turns slightly red. The optimal temperature for nickel leaching, however, is 92.5–100 °C, as shown by the solid red curve. The temperature can be increased to over 100 °C, as indicated by the dashed curve. This suggests that the leaching process requires heating to higher temperatures.

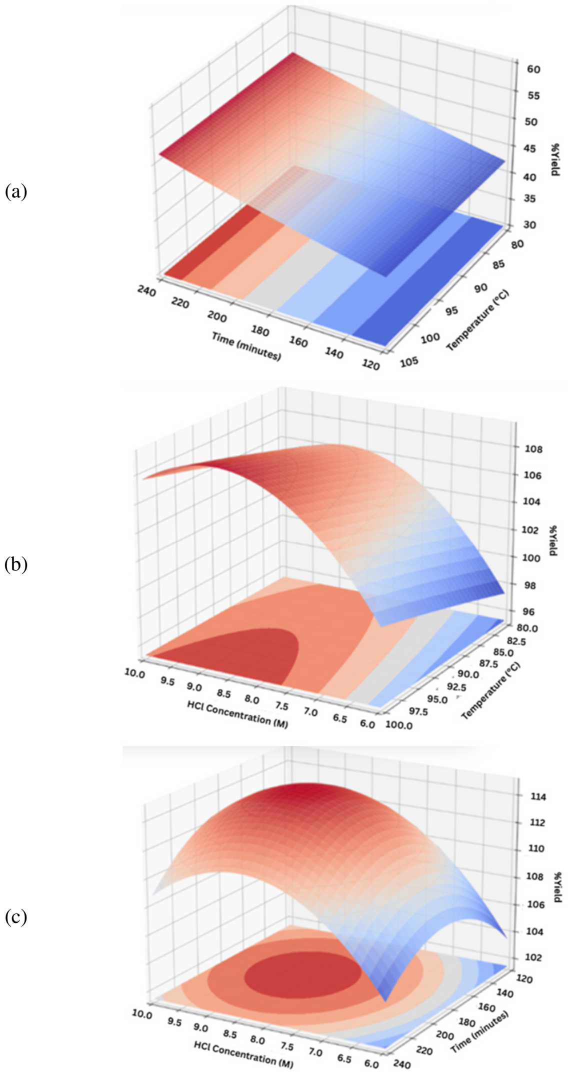

Fig. 3. BBD-RSM 3D curve shows/showing the effect of %Extraction Ni: (a) leaching time versus temperature, (b) HCl concentration versus temperature, (c) HCl concentration versus leaching time.

Figure 3(b) shows the optimal leaching time range, indicated by the red curve. This range is from 220 min to 240 min, while, the leaching temperature, ranging from 80 °C to 100 °C, does not affect the color of the curve. The results reveal that there is no significant interaction between temperature and time. Leaching time significantly influences the color of the curve, which changes from blue to red as time increases. In contrast, the temperature's influence appears uniform, as indicated by the consistent red curve across all tested temperature variations. Figure 3 (c) presents the relationship between the HCl concentration and leaching time. The curve shows a perfectly round optimal range in the middle. The selected HCl concentration and leaching time are based on these results. The optimal HCl concentration is between 7.5 M and 9.25 M, and the optimal leaching time is between 160 min and 220 min. The three 3D curves of the leaching results indicate that the ideal conditions for each test factor are HCl concentrations between 7.75 M and 10 M, leaching times between 160 min and 240 min, and leaching temperatures above 100 °C. The variations in HCl concentration and leaching time used in the study were sufficient to achieve optimal conditions. However, the variation in leaching temperature was insufficient, as the selected range did not produce a red curve. This suggests that temperature had only a minimal impact on the leaching results.

C. Nickel Recovery

In this experiment, nickel was precipitated using oxalic acid. The BBD-RSM test results determined which samples to use for precipitation. The selected sample had the highest nickel concentration, particularly in run 11, with/that is, a concentration of 490.01 ppm. 15 ml of this solution were used in the subsequent precipitation process. Run 11's solution, which had an initial pH of 9, was treated with 2 M oxalic acid as a nickel precipitant. This continued until the pH dropped to 2, at which point a white precipitate of solid nickel oxalate began to form [29]. This reaction is [30]:

$$Ni^{2+}_{(aq)} + C_2O_4^{2-}{}_{(aq)} + 2H_2O_{(aq)} \rightarrow NiC_2O_4 \cdot 2H_2O_{(s)} \qquad (7)$$

The formed precipitate was poured through Whatman filter paper to separate the filtrate, and was dried in an oven at 60°C for 24 h. The resulting solids were characterized using XRF and XRD, and their elemental composition was determined, as shown in Table VI.

TABLE VI. THE DIFFERENCE OF THE MAIN ELEMENTS FROM LATERITE ORE AND PRE-NI FROM XRF

Sample	**Element (%wt)**			
	Ni	Fe	Si	Mn
Laterite ore	1.91	16.28	13.13	0.16
PRE-Ni	59.50	0.96	6	0

According to the XRF results, the nickel content of the two samples—one of initial nickel laterite ore and the other of residue—differs significantly. The testing of the initial nickel laterite ore and the PRE-Ni revealed results of 1.91% and 59.50%, respectively. XRD also characterized the PRE-Ni, as depicted in Figure 4. These results show a change in the peak in the 2-theta region for laterite rocks and leached solids, indicating the presence of nickel oxalate precipitate. Peaks at 18.50°, 29.03°, 35.32°, 37.64°, and 43.66° are present, which correspond to the peaks of $NiC_2O_4 \cdot 2H_2O$ [30]. Thus, the solids formed from the leaching process contain nickel oxalate.

Based on the obtained results, the solid was identified as a type of bunsenite. Figure 5 displays the results of a comparative analysis of XRD tests of laterite ore and PRE-Ni, demonstrating the differences in phases formed by the two samples.

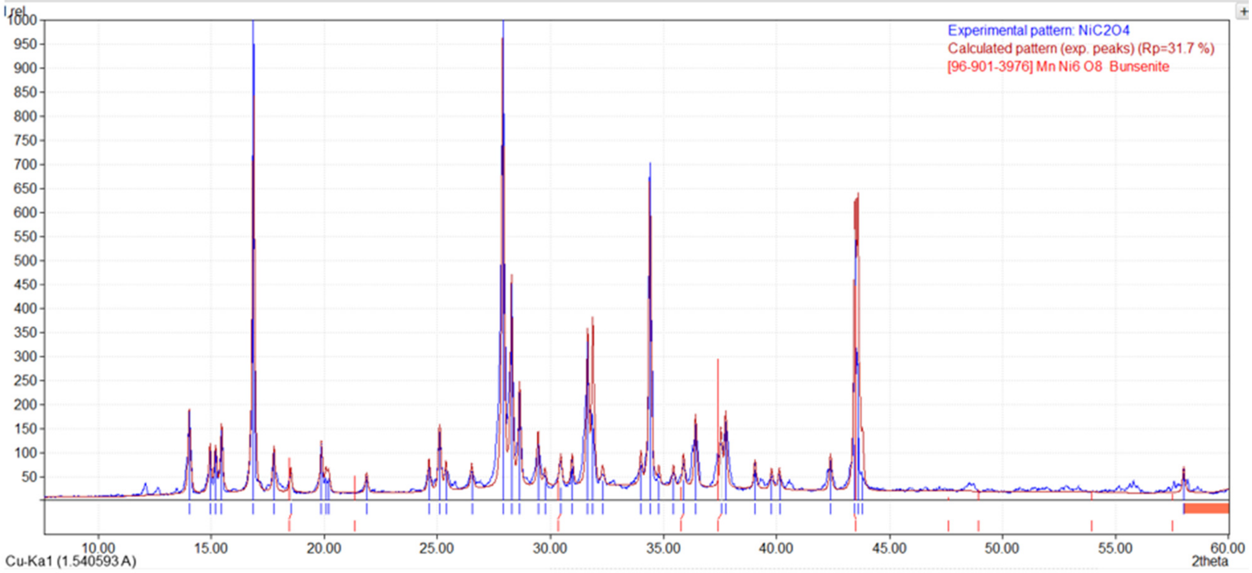

Fig. 4. XRD analysis result of PRE-Ni with Match! software.

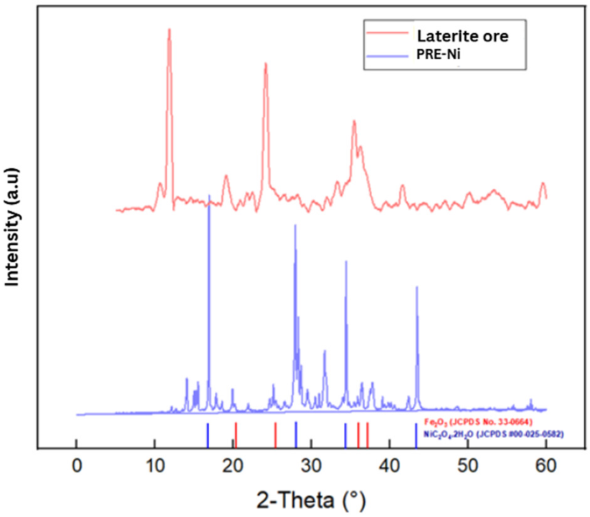

Fig. 5. Comparative analysis of XRD testing of laterite ore versus PRE-Ni.

The XRD results showed that impurity peaks, such as iron (Fe) and silicon (Si), disappeared from the PRE-Ni sample. These peaks were replaced by ones resembling nickel oxalate. The PRE-Ni filtrate was then characterized, using AAS to determine the remaining nickel concentration in the solution. The results showed that the filtrate's free nickel concentration was 13.08 ppm. The initial nickel concentration in the laterite rock was 981.43 ppm, with both solutions having a volume of 100 mL. The calculations revealed that the nickel recovery percentage is 98.66%, yielding a high nickel recovery rate.

IV. CONCLUSIONS

Nickel extraction from laterite rocks used various types of acids, such as sulfuric and nitric acids, under high-pressure conditions, showing limitations regarding the effect of using HCl under Atmospheric Pressure Acid Leaching (APAL) conditions. This study examined the use of HCl to extract Kolaka laterite rock under APAL conditions, optimizing the process by deploying the Box-Behnken Design Response Surface Method (BBD-RSM). The variables examined were HCl concentration, leaching time, and leaching temperature. The optimal conditions for nickel extraction were: an HCl concentration of 8 M, a leaching time of 240 min, and a leaching temperature of 100°C. This method produced a nickel concentration of 490.01 ppm and a recovery rate of 98.66%. The X-ray Diffraction (XRD) results supported these findings, indicating that the leached solid had a phase similar to that of the nickel oxalate solid, indicating a successful extraction process. The novelty of this study lies in the usage of HCl under APAL conditions and the BBD-RSM method for extraction optimization, which is still rarely used. Unlike previous works, this study provides a statistically validated model to predict nickel yield and determine leaching parameters with high accuracy, contributing to the development of laterite rock hydrometallurgical processes in Indonesia, providing information not only on nickel extraction from laterite rocks, but also on the development of research on nickel extraction and purification from rocks under non-extreme conditions. This is because the research was conducted under non-extreme conditions: not too acidic or alkaline, not at very high temperatures.

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