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Application of MLR Method and Pearson Correlation on Nickel Extraction from Laterite Using Nitric Acid

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ABSTRACT: Indonesia is a country rich in laterite mining especially in Celebes island and northern Moluccas. Laterite is a mineral rich with precious nickel. Nitric acid leaching has been used for nickel extraction from laterite under the influence of nitric acid concentration (2.0 M, 4.0 M, and 6.0 M) and leaching temperature (65ºC, 70ºC, and 75ºC) during 60 min. at 300 rpm. The preliminary study aims to apply multiple linear regression (MLR) method to examine the dominant effect between acid concentration and leaching temperature on nickel extraction, and also Pearson correlation to study significant correlation between the variables involved (acid concentration, leaching temperature, and nickel concentration). The method and results is related to acid leaching at varied temperature set up as predictors and nickel concentration as dependent variable in the MLR equation using the enter method. The MLR method showed that acid concentration gave significant effect on nickel concentration, however, the leaching temperature showed no significant effect on nickel extraction at $t \leq 0.05$. The Pearson correlation showed acid concentration correlated significantly with nickel concentration. The Kolmogrov-Smirnov (K-S) test showed normality for distribution of nickel concentration. The acid leaching method is potential for nickel extraction from laterite using extensive range of acid concentrations and leaching temperatures to improve nickel extraction.

KEYWORD: Acid leaching, Laterite, MLR method, Nickel extraction, Pearson correlation.

1. INTRODUCTION

Nickel (Ni) is a substantial element found in laterite mineral due to its tremendous applications in modern industry and technology. Nickel application in colored pigments using titanate compound gave its superiority in brightness, opacity, and color strength (Aslan *et al*., 2022; Aslana *et al*., 2023). Application of nickel doped titanium oxide immobilized in earth clay as photocatalyst to destroy methylene blue indicator in aqueous solution showed its benefit in relation to economic cost and efficiency (Sharma *et al*., 2023). Synthesis of nickel ferrite to yield high performance electrode for supercapacitor was reported by Ilayas *et al*., (2023). On other occasion, Vinosha *et al*., (2021) investigated nickel substitution in cobalt ferrite nanoparticles to enhance magneto-optical, electrical, and acoustical properties. Furthermore, the study of nickel – based superalloys were reported for many applications using various mechanical and heat treatments to yield acceptable limit material performance (Cao *et al*., 2021; Ding *et al*., 2020; Gudivada and Pandey, 2023).

The geological history of nickel is related to laterite mineral yielded from weathering and mineral enrichment of ultramafic ore. Moreover, nickel laterite has different characterizations influenced by mineral weathering and profile of laterite zones. Limonite zone is composed of clay mineral (kaolinite), mineral oxide (magnetite, hematite, and chromite), and mineral hydroxide (goethite). Saprolite zone is composed of silicate mineral (quartz, garnierite, antigorite, enstatite, and lisardite). The depth of limonite zone is about 0 – 3 meter from surface, while the depth of saprolite zone about 3 – 9 meter. The nickel content in limonite zone is found to be 0.76 – 1.78%, while in saprolite zone 1.79 – 2.98% (Lintjewas *et al*., 2019). In addition, there is a bedrock zone, which its depth > 9 meter with insignificant nickel content < 0.5%; the very low nickel content means it has no economic value to be exploited. (Pangeran, 2022). Figure 1 shows the profile of laterite zones with rough estimation of elements sampling from North Konawe, Celebes (Pangeran, 2022).

Indonesia has reserved approximately 72 million ton nickel or about 52% total nickel in the world that means Indonesia is potential source for nickel (Pangeran, 2022). This study has used nickel laterite as the raw material from saprolite zone exploited in North Konawe, Celebes. Nickel laterite is particularly scattered in Eastern part of Indonesia, that concentrated in Central and

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Southeastern part of Celebes Island, as well as in Halmahera, Moluccas Islands. The nickel mining process starts from topsoil and overburden removal, ore loading and hauling, and then stored in stockpile port. Finally, the nickel ore was transferred from stockpile to factory for further processing like roasting followed by hydrometallurgy process such as modified acid leaching, froth floatation or its combination in mineral industry.

Laterite profile	Zone	Estimation of analysis		
		% Fe	% MgO	% Ni
	Top Soil	≥ 40	< 5	< 0.7
	Limonite	≥ 30	< 5	< 1.7
	Transition zone	20 – 30	≈ 5	≈ 1.7
	Saprolite	≤ 20	≥ 5	≥ 1.7
	Bedrock	≤ 10	20 – 30	< 0.5

Figure 1. Laterite profile with zones and analysis of elements (Pangeran, 2022)

This study has used the hydrometallurgy route addressing to low cost atmospheric pressure acid leaching for nickel extraction from laterite concentrate. Nitric acid was selected as the leaching acid. The statistical analysis applied MLR method and Pearson correlation, as well as the Kolmogorov-Smirnov normality test. The MLR method was used to determine the significant effect between acid concentrations and leaching temperatures on nickel extraction from laterite. The Pearson correlation was applied to investigate the significant correlation between the variables involved (acid concentration, leaching temperature, and nickel concentration). The K-S test was also included in this paper to examine normal distribution. Previous investigations have never reported nickel extraction from laterite mineral based on the standpoint of statistical analysis and therefore, this paper can be viewed as a novelty of this research.

2. RESEARCH METHODOLOGY

The research methodology consists of research procedure, subject characteristics, and statistical analysis. The research procedure includes mineral sampling, laterite processing, and nickel extraction. The characteristics analysis is dealing with XRD (X-Ray Diffraction) and XRF (X-Ray Fluorescence) analysis of laterite mineral. The statistical analysis includes K-S normality test, MLR method, and Pearson correlation.

2.1 Research Procedure

Laterite ore was taken from North Konawe, Celebes. Laterite ore was dried and filtered by rotary screener for selected size (200 mesh) and followed by roasted in a muffle furnace at 600^{o}C for 1h. The roasted laterite was dissolved in aqueous solution to yield laterite concentrate. An acid leaching method was used in this study. Nitric acid (99% Merck, Germany) solutions (2.0M, 4.0M, and 6.0M) were used for acid leaching at leaching temperatures (65^{o}C, 70^{o}C, and 75^{o}C) stirring at 300 rpm for 60 min. to extract nickel from laterite concentrate. The acid leaching process was conducted twice (duplo). The nickel concentration for each solution

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was determined by AAS (Atomic Absorption Spectrophotometer) technique. An Analytic Jena with acetylene/nitrous oxide flame AAS using lamp current of hollow cathode HCl lamp setting at 2 – 20 mA was applied for this work. Dahani *et al.*, (2022) applied low cost acetic acid and sulfuric acid to extract lead from galena. Figure 2 shows the flow diagram of the research procedure.

2.2 Characterization Analysis

This study applied XRD and XRF characterization analysis of laterite mineral. The XRD analysis investigates the existence of minerals in laterite ore. The XRD analysis applied copper with a wavelength of $K\alpha 1$ of 1.540560 and a wavelength of $K\alpha 2$ of 1.54443 as radiation source. The XRD used scanning range 2θ of $10^o – 90^o$ with scanning rate of 0.02^o/s. The XRD analysis examined the given size of laterite ore before and after roasting. The XRF analysis investigates the existence of elements in laterite mineral. The XRF analysis applied a PANalytical type Minipal 4 XRF Spectrophotometer.

2.3 Statistical Analysis

An SPSS version 22 has been conducted to run statistical analysis addressing to Kolmogorov-Smirnov normality test for one sample, multiple linear regression (MLR) with enter method, and bivariate Pearson correlation (George and Mallery, 2011). The K-S test examines the normality of a sample distribution. The MLR method assays the significance of variables affected the nickel extraction from laterite based on its standard critical value ($t \leq 0.05$). The Pearson correlation observes the degree of correlation between variables involved.

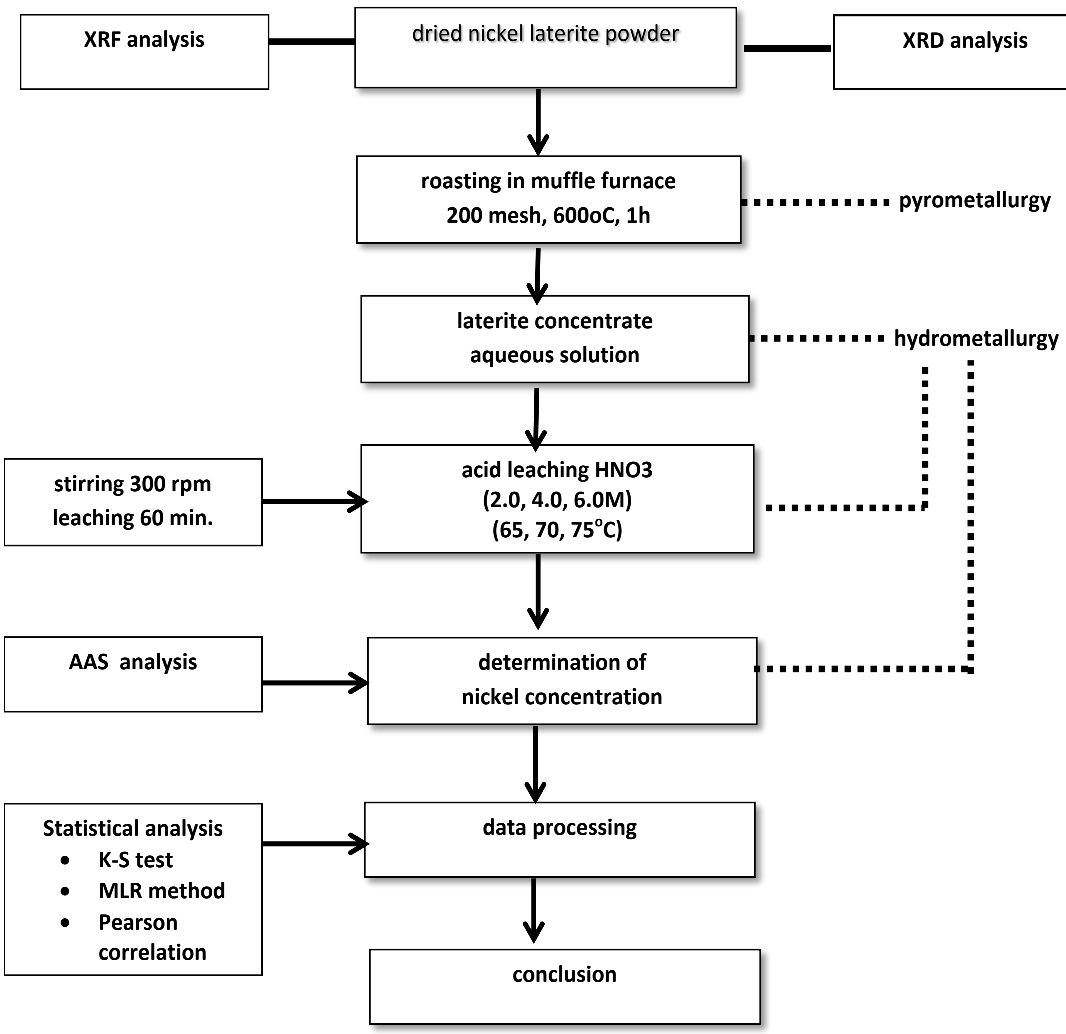

Figure 2. The flow diagram of research procedure.

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3. RESULTS AND DISCUSSION

3.1 Acid Leaching Method

Table 1 shows the results of nickel extraction from laterite mineral applying the acid leaching method with HNO_3 (2.0M, 4.0M, and 6.0M) at given leaching temperatures (65ºC, 70ºC, and 75ºC) stirred at 300 rpm for 60 min. At the moment, Table 1 shows the 4.0 M nitric acid solution at 70ºC yielded the highest result of nickel extraction from laterite concentrate (highlighted sign) that can be assumed as the optimum result of this study. It is reasonable that the concentration of 4.0M nitric acid at 70ºC is the most suitable in this case since the lower concentration (2.0M) is not yet effective to withdraw nickel from laterite matrix, on the other hand, the higher concentration (6.0M) may induce ion pair formation in aqueous solution hindering the migration of nickel ions in the solution. The lower temperature than 70ºC may result the movement of ions in aqueous solution not yet achieved the maximum speed, however, the higher temperature than 70ºC may cause solution evaporation. Therefore, the 70ºC is the most suitable condition for thermal agitation to yield the optimum result. Figure 3 shows the graphs of nickel extraction from laterite based on data Table 1 regarding the influence of given varied acid concentrations (2.0M, 4.0M, and 6.0M) and varied leaching temperatures (65ºC 70ºC, and 75ºC) on nickel extraction from laterite mineral.

3.2 XRD and XRF Characterization

Figure 4 and Figure 5 show the XRD presentations related to laterite mineral sampling from North Konawe, Celebes (Pangeran, 2022). Figure 4 shows the XRD presentation of laterite mineral before roasting (600ºC, 1h) attributed to sharp peaks of silicon dioxide, iron (III) oxide, and nickel sulfate as dominant minerals mostly performed by their crystalline structures. XRD examination is broadly applied in many analyses related to investigate crystalline and amorphous molecule and atomic structures. Previous study investigated the study of provskite micro oxide structure of an electrochemical cathode at 1000ºC and 1100ºC (Subardi, 2022). On other occasion, XRD analysis was applied to investigate the surface of SnO_2 crystalline synthesis by sol gel process (Arini *et al.*, 2022). Figure 5 shows the XRD presentation of laterite mineral after roasting (600ºC, 1h). With regard to Figure 4 related to XRD of laterite before roasting and Figure 5 related to XRD of laterite after roasting addressing to mineral components in laterite, it should be noted that there is a conversion of Ni_2SiO_4 (before roasting) to NiO (after roasting). The roasting process is related to change of chemical mineral addressing to nickel compound and the molecular crystal structure. It is an indication of more SiO_2 formation and nickel compound converted to NiO mineral during roasting process. The Ni_2SiO_4 known as nickel olivine is a member of silicate olivines found in igneous rock (Lin, 2001). Ni_2SiO_4 is a solid solution that has distorted hexagonal close-packed structure easily broken due to high thermal effect. Conversion of molecular chemical structure during roasting process at high temperature for several hours is very common obtained in mineral ores. Previous study (unpublished) reported SO_2 removal during roasting process of sphalerite mineral found together with galena in mineral ore.

Table 2 lists the percentage of elements in nickel laterite powder resulted from XRF analysis. Addressing to the nickel concentration in Table 2, this mineral has a tendency to high grade nickel laterite as the concentration found to be ≈ 2% (Lintjewas *et al.*, 2019). Moreover, the nickel concentration that was about 2 % indicated the laterite zone referred to transitional zone between lower laterite limonite and upper laterite saprolite (Figure 1). Previous report studied that laterite deposit in Southeastern Celebes mostly contains nickel sulfide, nickel iron, and nickel oxide as common minerals found in high grade nickel laterite (Zhang *et al.*, 2020). The report said that the nickel concentration in nickel-iron mineral was estimated to 1.99% nickel and 17.55% iron. Addressing to the nickel concentration in this study as shown by Table 2, thus, the laterite mineral used in this study mostly contains nickel iron.

3.3 Results of Statistical Analysis

Nowadays, statistical and math analysis have broad applications in other field study. Elmisaoui *et al.* (2023) applied polynomial interpolation method for the optimization of phosphate ore dissolution in phosphoric acid solution, while Arif *et al.*, (2023) used numeric simulation for the calculation of cradle deck design in shipment waterfront. This study applied three methods of statistical examination, using SPSS version 22 software, i.e. the K-S test to assay the normality of a distribution, the MLR method to assay significant predictors influenced a variable of interest, and Pearson correlation to investigate significant correlation between two variables.

3.3.1 Kolmogorov- Smirnov (K-S) Normality Test

Table 3 shows the result of K-S normality test that the nickel concentration resulting from laterite extraction by acid leaching method followed the normal distribution with values of average 32.189 and standard deviation 3.14 and that the Null Hypothesis is accepted with the value of significance 0.089 based on data of Table 1 executed by SPSS version 22. Therefore, the statistical analysis can be continued to MLR analysis and Pearson correlation.

3.3.2 Multiple Linear Regression (MLR) Method

This section discussed the results of MLR method on nickel extraction from laterite concentrate with acid concentration and leaching temperature taken as predictors or independent variables, and the nickel concentration as dependent variable in the MLR equation. Table 4 shows the MLR model with "enter" mode based on data of Table 1 executed by SPSS version 22. Table 5 shows the Analysis of Variance (ANOVA) of data Table 1 as the output of SPSS version 22. The table of ANOVA (Table 5) shows the value of significance 0.115 leading to a conclusion that both predictors (acid concentration and leaching temperature) gave no significant effect on nickel extraction from laterite ($t \leq 0.05$). However, Table 6 (the Multiple Linear Regression Coefficient) shows that acid concentration yielded significant effect on nickel extraction from laterite with its significant value of 0.041 ($t \leq 0.05$). On the other hand, Table 6 shows that leaching temperature yielded no significant effect on nickel extraction from laterite attributed to its significant value of 0.998. The remarkable high "significance" value of leaching temperature (0.998) shown by Table 6 can overcome the significant effect of acid concentration on nickel extraction from laterite, and as a result, the overall "significance" value (0.115) shown by Table 5 is the overall MLR results considering both acid concentration and leaching temperature as predictors. Nevertheless, the acid concentration yielded the significant effect on nickel extraction from laterite not necessary with the role of leaching temperature.

3.3.3 Pearson Correlation

Table 7 shows the bivariate correlation (Pearson correlation) executed by SPSS version 22 applying data Table 1. Table 7 shows moderate correlation (0.501) between leaching acid concentration and nickel concentration attributed to moderately significant effect (0.041) of acid concentration on nickel extraction shown in Table 6 (the MLR coefficient). On the other hand, Table 7 shows very weak correlation between leaching temperature and nickel concentration (- 0.01) in negative way indicated increased leaching temperature yielding decreased nickel concentration or vice versa. The very weak correlation attributed to very weak influence of leaching temperature on nickel concentration indicated by its significance value (0.998) shown in Table 6.

4. CONCLUSION

The experimental results show that leaching acid concentration yielded significant effect on nickel extraction from laterite verified by the MLR method with significance level of 0.041 ($t \leq 0.05$). This finding is supported by moderate bivariate correlation (Pearson correlation) between leaching acid concentration and nickel extraction from laterite (0.501). The K-S test shows normal distribution of nickel concentration based on Null Hypothesis (0.089 significance level). This investigation shows that statistical analysis is a valuable tool with respect to nickel extraction from laterite mineral applying acid leaching method.

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Table 1. Acid leaching method with HNO_3.
60 min. 300 rpm stirring.

nitric acid (molar)	temp. (ºC)	nickel conc. (mg/L)
2	65	28.11
2	70	29.12
2	75	28.10
4	65	35.54
4	70	36.56
4	75	35.55
6	65	32.21

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6	70	32.23
6	75	32.20
2	65	28.16
2	70	29.18
2	75	28.17
4	65	35.54
4	70	36.58
4	75	35.52
6	65	32.21
6	70	32.23
6	75	32.20

Table 2. Elements of nickel laterite. XRF analysis

no	element	concentration (%)
1	Si (silicon)	37.47 ± 2.67
2	Ca (calcium)	0.50 ± 0.05
3	Cr (chromium)	1.53 ± 0.13
4	Fe (iron)	44.61 ± 2.74
5	Ni (nickel)	1.95 ± 0.53

Table 3. K-S normality test of nickel concentration distribution

Null Hypothesis	Test	Sig.	Decision
The distribution of nickel conc. (mg/L) is normal with mean 32.189 and standard deviation 3.14	One-Sample Kolmogorov-Smirnov Test	0.089	Retain the null hypothesis

The significance level is 0.05

Table 4. Multiple Linear Regression Model

Variables Entered	Variables Removed	Method
leaching temperature (ºC), acid concentration (molar)	.	Enter

Dependent Variable: nickel concentration (mg/L)

Table 5. Analysis of Variance (ANOVA)[a]

Model	Sum of Squares	df	Mean Square	F	Sig.
Regression	41.963	2	20.981	2.508	.115[b]
Residual	125.495	15	8.366		
Total	167.457	17			

a. Dependent Variable: nickel concentration (mg/L)

b. Predictors: (Constant), leaching temperature (ºC), acid concentration (molar)

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Table 6. MLR Coefficient[a]

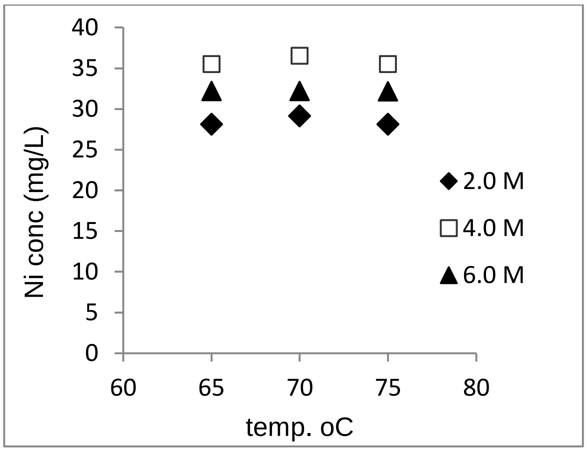
Model	Unstandardized Coefficients B	Unstandardized Coefficients Std. Error	Standardized Coefficients Beta	t	Sig.
(constant)	28.484	11.828		2.408	.029
acid concentration (molar)	.935	.417	.501	2.240	.041
leaching temperature (°C)	.000	.167	-.001	-.003	.998

a. . Dependent Variable: nickel concentration (mg/L)

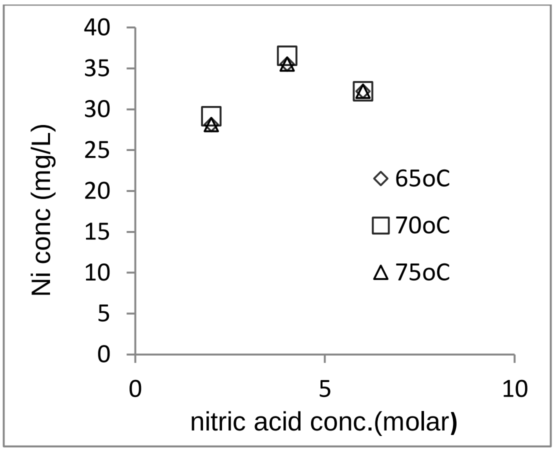
Table 7. Bivariate Correlation

		acid conc. (molar)	leaching temp. (°C)	nickel conc. (mg/L)
acid conc. (molar)	Pearson Correlation	1	.000	.501*
	Sig. (2-tailed)		1.000	.034
	N	18	18	18
leaching temp. (°C)	Pearson Correlation	.000	1	-.001
	Sig. (2-tailed)	1.000		.998
	N	18	18	18
nickel conc. (mg/L)	Pearson Correlation	.501*	-.001	1
	Sig. (2-tailed)	.034	.998	
	N	18	18	18

Note: Correlation is significant at the 0.05 level (2-tailed)



(A)



(B)

Figure 3. Nickel extraction from laterite concentrate using nitric acid leaching method stirred at 300 rpm. 60 min. (A) at varied leaching temperatures (65°C, 70°C, and 75°C) and (B) at varied leaching acid concentrations (2.0M, 4.0M, and 6.0M).

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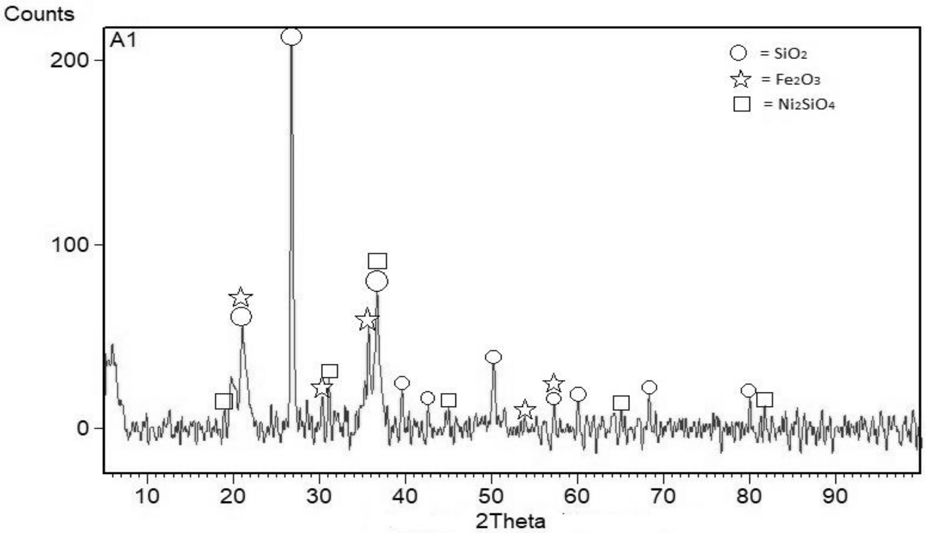

Figure 4. XRD presentation of nickel laterite sampling before roasting

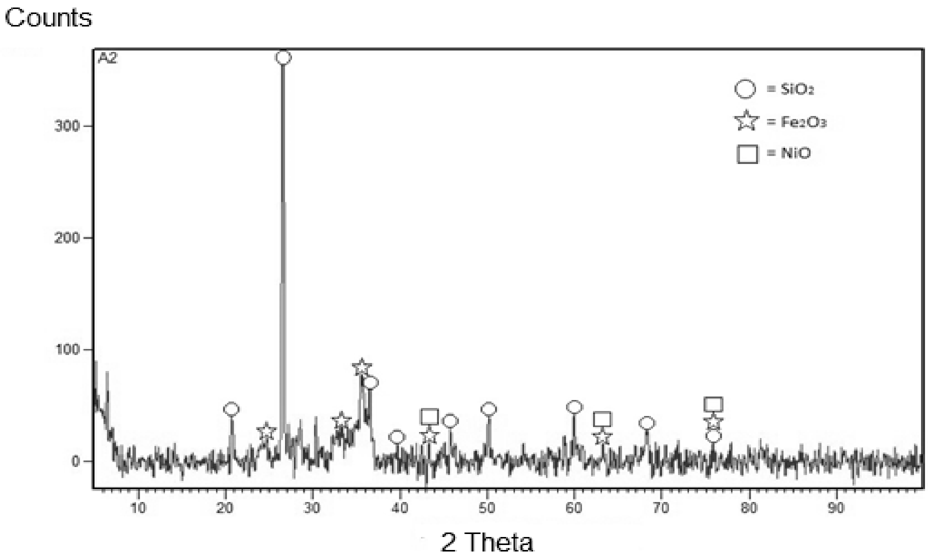

Figure 5 . XRD presentation of nickel laterite sampling after roasting. Roasting 600ºC, 1h

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ABSTRACT: Indonesia is a country rich in laterite mining especially in Celebes island and northern Moluccas. Laterite is a mineral rich with precious nickel. Nitric acid leaching has been used for nickel extraction from laterite under the influence of nitric acid concentration (2.0 M, 4.0 M, and 6.0 M) and leaching temperature (65°C, 70°C, and 75°C) during 60 min. at 300 rpm. The preliminary study aims to apply multiple linear regression (MLR) method to examine the dominant effect between acid concentration and leaching temperature on nickel extraction, and also Pearson correlation to study significant correlation between the variables involved (acid concentration, leaching temperature, and nickel concentration). The method and results is related to acid leaching at varied temperature set up as predictors and nickel concentration as dependent variable in the MLR equation using the enter method. The MLR method showed that acid concentration gave significant effect on nickel concentration, however, the leaching temperature showed no significant effect on nickel extraction at $t \leq 0.05$. The Pearson correlation showed acid concentration correlated significantly with nickel concentration. The Kolmogrov-Smirnov (K-S) test showed normality for distribution of nickel concentration. The acid leaching method is potential for nickel extraction from laterite using extensive range of acid concentrations and leaching temperatures to improve nickel extraction.

KEYWORD: Acid leaching, Laterite, MLR method, Nickel extraction, Pearson correlation.

1. INTRODUCTION

Nickel (Ni) is a substantial element found in laterite mineral due to its tremendous applications in modern industry and technology. Nickel application in colored pigments using titanate compound gave its superiority in brightness, opacity, and color strength (Aslan *et al*., 2022; Aslana *et al*., 2023). Application of nickel doped titanium oxide immobilized in earth clay as photocatalyst to destroy methylene blue indicator in aqueous solution showed its benefit in relation to economic cost and efficiency (Sharma *et al*., 2023). Synthesis of nickel ferrite to yield high performance electrode for supercapacitor was reported by Ilayas *et al*., (2023). On other occasion, Vinosha *et al*., (2021) investigated nickel substitution in cobalt ferrite nanoparticles to enhance magneto-optical, electrical, and acoustical properties. Furthermore, the study of nickel – based superalloys were reported for many applications using various mechanical and heat treatments to yield acceptable limit material performance (Cao *et al*., 2021; Ding *et al*., 2020; Gudivada and Pandey, 2023).

The geological history of nickel is related to laterite mineral yielded from weathering and mineral enrichment of ultramafic ore. Moreover, nickel laterite has different characterizations influenced by mineral weathering and profile of laterite zones. Limonite zone is composed of clay mineral (kaolinite), mineral oxide (magnetite, hematite, and chromite), and mineral hydroxide (goethite). Saprolite zone is composed of silicate mineral (quartz, garnierite, antigorite, enstatite, and lisardite). The depth of limonite zone is about 0 – 3 meter from surface, while the depth of saprolite zone about 3 – 9 meter. The nickel content in limonite zone is found to be 0.76 – 1.78%, while in saprolite zone 1.79 – 2.98% (Lintjewas *et al*., 2019). In addition, there is a bedrock zone, which its depth > 9 meter with insignificant nickel content < 0.5%; the very low nickel content means it has no economic value to be exploited. (Pangeran, 2022). Figure 1 shows the profile of laterite zones with rough estimation of elements sampling from North Konawe, Celebes (Pangeran, 2022).

Indonesia has reserved approximately 72 million ton nickel or about 52% total nickel in the world that means Indonesia is potential source for nickel (Pangeran, 2022). This study has used nickel laterite as the raw material from saprolite zone exploited in North Konawe, Celebes. Nickel laterite is particularly scattered in Eastern part of Indonesia, that concentrated in Central and

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Southeastern part of Celebes Island, as well as in Halmahera, Moluccas Islands. The nickel mining process starts from topsoil and overburden removal, ore loading and hauling, and then stored in stockpile port. Finally, the nickel ore was transferred from stockpile to factory for further processing like roasting followed by hydrometallurgy process such as modified acid leaching, froth floatation or its combination in mineral industry.

Laterite profile	Zone	Estimation of analysis		
		% Fe	% MgO	% Ni
	Top Soil	≥ 40	< 5	< 0.7
	Limonite	≥ 30	< 5	< 1.7
	Transition zone	20 – 30	≈ 5	≈ 1.7
	Saprolite	≤ 20	≥ 5	≥ 1.7
	Bedrock	≤ 10	20 – 30	< 0.5

Figure 1. Laterite profile with zones and analysis of elements (Pangeran, 2022)

This study has used the hydrometallurgy route addressing to low cost atmospheric pressure acid leaching for nickel extraction from laterite concentrate. Nitric acid was selected as the leaching acid. The statistical analysis applied MLR method and Pearson correlation, as well as the Kolmogorov-Smirnov normality test. The MLR method was used to determine the significant effect between acid concentrations and leaching temperatures on nickel extraction from laterite. The Pearson correlation was applied to investigate the significant correlation between the variables involved (acid concentration, leaching temperature, and nickel concentration). The K-S test was also included in this paper to examine normal distribution. Previous investigations have never reported nickel extraction from laterite mineral based on the standpoint of statistical analysis and therefore, this paper can be viewed as a novelty of this research.

2. RESEARCH METHODOLOGY

The research methodology consists of research procedure, subject characteristics, and statistical analysis. The research procedure includes mineral sampling, laterite processing, and nickel extraction. The characteristics analysis is dealing with XRD (X-Ray Diffraction) and XRF (X-Ray Fluorescence) analysis of laterite mineral. The statistical analysis includes K-S normality test, MLR method, and Pearson correlation.

2.1 Research Procedure

Laterite ore was taken from North Konawe, Celebes. Laterite ore was dried and filtered by rotary screener for selected size (200 mesh) and followed by roasted in a muffle furnace at 600°C for 1h. The roasted laterite was dissolved in aqueous solution to yield laterite concentrate. An acid leaching method was used in this study. Nitric acid (99% Merck, Germany) solutions (2.0M, 4.0M, and 6.0M) were used for acid leaching at leaching temperatures (65°C, 70°C, and 75°C) stirring at 300 rpm for 60 min. to extract nickel from laterite concentrate. The acid leaching process was conducted twice (duplo). The nickel concentration for each solution

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was determined by AAS (Atomic Absorption Spectrophotometer) technique. An Analytic Jena with acetylene/nitrous oxide flame AAS using lamp current of hollow cathode HCl lamp setting at 2 – 20 mA was applied for this work. Dahani *et al*., (2022) applied low cost acetic acid and sulfuric acid to extract lead from galena. Figure 2 shows the flow diagram of the research procedure.

2.2 Characterization Analysis

This study applied XRD and XRF characterization analysis of laterite mineral. The XRD analysis investigates the existence of minerals in laterite ore. The XRD analysis applied copper with a wavelength of $K\alpha1$ of 1.540560 and a wavelength of $K\alpha2$ of 1.54443 as radiation source. The XRD used scanning range 2 θ of $10^o – 90^o$ with scanning rate of 0.02^o/s. The XRD analysis examined the given size of laterite ore before and after roasting. The XRF analysis investigates the existence of elements in laterite mineral. The XRF analysis applied a PANalytical type Minipal 4 XRF Spectrophotometer.

2.3 Statistical Analysis

An SPSS version 22 has been conducted to run statistical analysis addressing to Kolmogorov-Smirnov normality test for one sample, multiple linear regression (MLR) with enter method, and bivariate Pearson correlation (George and Mallery, 2011). The K-S test examines the normality of a sample distribution. The MLR method assays the significance of variables affected the nickel extraction from laterite based on its standard critical value ($t \leq 0.05$). The Pearson correlation observes the degree of correlation between variables involved.

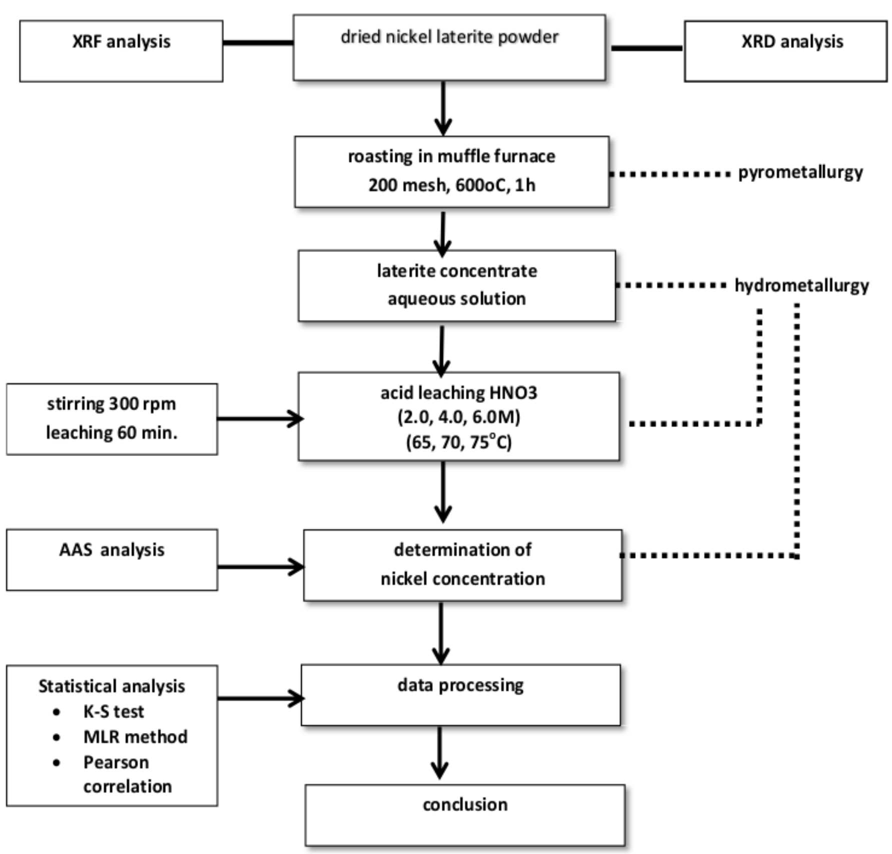

Figure 2. The flow diagram of research procedure.

3. RESULTS AND DISCUSSION

3.1 Acid Leaching Method

Table 1 shows the results of nickel extraction from laterite mineral applying the acid leaching method with HNO_3 (2.0M, 4.0M, and 6.0M) at given leaching temperatures (65°C, 70°C, and 75°C) stirred at 300 rpm for 60 min. At the moment, Table 1 shows the 4.0 M nitric acid solution at 70°C yielded the highest result of nickel extraction from laterite concentrate (highlighted sign) that can be assumed as the optimum result of this study. It is reasonable that the concentration of 4.0M nitric acid at 70°C is the most suitable in this case since the lower concentration (2.0M) is not yet effective to withdraw nickel from laterite matrix, on the other hand, the higher concentration (6.0M) may induce ion pair formation in aqueous solution hindering the migration of nickel ions in the solution. The lower temperature than 70°C may result the movement of ions in aqueous solution not yet achieved the maximum speed, however, the higher temperature than 70°C may cause solution evaporation. Therefore, the 70°C is the most suitable condition for thermal agitation to yield the optimum result. Figure 3 shows the graphs of nickel extraction from laterite based on data Table 1 regarding the influence of given varied acid concentrations (2.0M, 4.0M, and 6.0M) and varied leaching temperatures (65°C 70°C, and 75°C) on nickel extraction from laterite mineral.

3.2 XRD and XRF Characterization

Figure 4 and Figure 5 show the XRD presentations related to laterite mineral sampling from North Konawe, Celebes (Pangeran, 2022). Figure 4 shows the XRD presentation of laterite mineral before roasting (600°C, 1h) attributed to sharp peaks of silicon dioxide, iron (III) oxide, and nickel sulfate as dominant minerals mostly performed by their crystalline structures. XRD examination is broadly applied in many analyses related to investigate crystalline and amorphous molecule and atomic structures. Previous study investigated the study of provskite micro oxide structure of an electrochemical cathode at 1000°C and 1100°C (Subardi, 2022). On other occasion, XRD analysis was applied to investigate the surface of SnO_2 crystalline synthesis by sol gel process (Arini *et al*., 2022). Figure 5 shows the XRD presentation of laterite mineral after roasting (600°C, 1h). With regard to Figure 4 related to XRD of laterite before roasting and Figure 5 related to XRD of laterite after roasting addressing to mineral components in laterite, it should be noted that there is a conversion of Ni_2SiO_4 (before roasting) to NiO (after roasting). The roasting process is related to change of chemical mineral addressing to nickel compound and the molecular crystal structure. It is an indication of more SiO_2 formation and nickel compound converted to NiO mineral during roasting process. The Ni_2SiO_4 known as nickel olivine is a member of silicate olivines found in igneous rock (Lin, 2001). Ni_2SiO_4 is a solid solution that has distorted hexagonal close-packed structure easily broken due to high thermal effect. Conversion of molecular chemical structure during roasting process at high temperature for several hours is very common obtained in mineral ores. Previous study (unpublished) reported SO_2 removal during roasting process of sphalerite mineral found together with galena in mineral ore.

Table 2 lists the percentage of elements in nickel laterite powder resulted from XRF analysis. Addressing to the nickel concentration in Table 2, this mineral has a tendency to high grade nickel laterite as the concentration found to be ≈ 2% (Lintjewas *et al*., 2019). Moreover, the nickel concentration that was about 2 % indicated the laterite zone referred to transitional zone between lower laterite limonite and upper laterite saprolite (Figure 1). Previous report studied that laterite deposit in Southeastern Celebes mostly contains nickel sulfide, nickel iron, and nickel oxide as common minerals found in high grade nickel laterite (Zhang *et al*., 2020). The report said that the nickel concentration in nickel-iron mineral was estimated to 1.99% nickel and 17.55% iron. Addressing to the nickel concentration in this study as shown by Table 2, thus, the laterite mineral used in this study mostly contains nickel iron.

3.3 Results of Statistical Analysis

Nowadays, statistical and math analysis have broad applications in other field study. Elmisaoui *et al*. (2023) applied polynomial interpolation method for the optimization of phosphate ore dissolution in phosphoric acid solution, while Arif *et al*., (2023) used numeric simulation for the calculation of cradle deck design in shipment waterfront. This study applied three methods of statistical examination, using SPSS version 22 software, i.e. the K-S test to assay the normality of a distribution, the MLR method to assay significant predictors influenced a variable of interest, and Pearson correlation to investigate significant correlation between two variables.

3.3.1 Kolmogorov- Smirnov (K-S) Normality Test

Table 3 shows the result of K-S normality test that the nickel concentration resulting from laterite extraction by acid leaching method

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followed the normal distribution with values of average 32.189 and standard deviation 3.14 and that the Null Hypothesis is accepted with the value of significance 0.089 based on data of Table 1 executed by SPSS version 22. Therefore, the statistical analysis can be continued to MLR analysis and Pearson correlation.

3.3.2 Multiple Linear Regression (MLR) Method

This section discussed the results of MLR method on nickel extraction from laterite concentrate with acid concentration and leaching temperature taken as predictors or independent variables, and the nickel concentration as dependent variable in the MLR equation. Table 4 shows the MLR model with "enter" mode based on data of Table 1 executed by SPSS version 22. Table 5 shows the Analysis of Variance (ANOVA) of data Table 1 as the output of SPSS version 22. The table of ANOVA (Table 5) shows the value of significance 0.115 leading to a conclusion that both predictors (acid concentration and leaching temperature) gave no significant effect on nickel extraction from laterite ($t \leq 0.05$). However, Table 6 (the Multiple Linear Regression Coefficient) shows that acid concentration yielded significant effect on nickel extraction from laterite with its significant value of 0.041 ($t \leq 0.05$). On the other hand, Table 6 shows that leaching temperature yielded no significant effect on nickel extraction from laterite attributed to its significant value of 0.998. The remarkable high "significance" value of leaching temperature (0.998) shown by Table 6 can overcome the significant effect of acid concentration on nickel extraction from laterite, and as a result, the overall "significance" value (0.115) shown by Table 5 is the overall MLR results considering both acid concentration and leaching temperature as predictors. Nevertheless, the acid concentration yielded the significant effect on nickel extraction from laterite not necessary with the role of leaching temperature.

3.3.3 Pearson Correlation

Table 7 shows the bivariate correlation (Pearson correlation) executed by SPSS version 22 applying data Table 1. Table 7 shows moderate correlation (0.501) between leaching acid concentration and nickel concentration attributed to moderately significant effect (0.041) of acid concentration on nickel extraction shown in Table 6 (the MLR coefficient). On the other hand, Table 7 shows very weak correlation between leaching temperature and nickel concentration (- 0.01) in negative way indicated increased leaching temperature yielding decreased nickel concentration or vice versa. The very weak correlation attributed to very weak influence of leaching temperature on nickel concentration indicated by its significance value (0.998) shown in Table 6.

4. CONCLUSION

The experimental results show that leaching acid concentration yielded significant effect on nickel extraction from laterite verified by the MLR method with significance level of 0.041 ($t \leq 0.05$). This finding is supported by moderate bivariate correlation (Pearson correlation) between leaching acid concentration and nickel extraction from laterite (0.501). The K-S test shows normal distribution of nickel concentration based on Null Hypothesis (0.089 significance level). This investigation shows that statistical analysis is a valuable tool with respect to nickel extraction from laterite mineral applying acid leaching method.

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Table 1. Acid leaching method with HNO_3.
60 min. 300 rpm stirring.

nitric acid (molar)	temp. (°C)	nickel conc. (mg/L)
2	65	28.11
2	70	29.12
2	75	28.10
4	65	35.54
4	70	36.56
4	75	35.55
6	65	32.21

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6	70	32.23
6	75	32.20
2	65	28.16
2	70	29.18
2	75	28.17
4	65	35.54
4	70	36.58
4	75	35.52
6	65	32.21
6	70	32.23
6	75	32.20

Table 2. Elements of nickel laterite. XRF analysis

no	element	concentration (%)
1	Si (silicon)	37.47 ± 2.67
2	Ca (calcium)	0.50 ± 0.05
3	Cr (chromium)	1.53 ± 0.13
4	Fe (iron)	44.61 ± 2.74
5	Ni (nickel)	1.95 ± 0.53

Table 3. K-S normality test of nickel concentration distribution

Null Hypothesis	Test	Sig.	Decision
The distribution of nickel conc. (mg/L) is normal with mean 32.189 and standard deviation 3.14	One-Sample Kolmogorov-Smirnov Test	0.089	Retain the null hypothesis

The significance level is 0.05

Table 4. Multiple Linear Regression Model

Variables Entered	Variables Removed	Method
leaching temperature (°C), acid concentration (molar)	.	Enter

Dependent Variable: nickel concentration (mg/L)

Table 5. Analysis of Variance (ANOVA)[a]

Model	Sum of Squares	df	Mean Square	F	Sig.
Regression	41.963	2	20.981	2.508	.115[b]
Residual	125.495	15	8.366		
Total	167.457	17			

a. Dependent Variable: nickel concentration (mg/L)

b. Predictors: (Constant), leaching temperature (°C), acid concentration (molar)

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Table 6. MLR Coefficient[a]

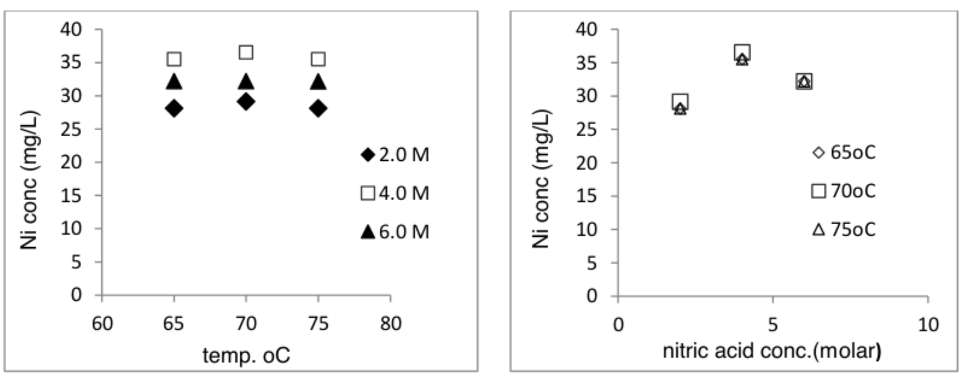
Model	Unstandardized Coefficients B	Unstandardized Coefficients Std. Error	Standardized Coefficients Beta	t	Sig.
(constant)	28.484	11.828		2.408	.029
acid concentration (molar)	.935	.417	.501	2.240	.041
leaching temperature (°C)	.000	.167	-.001	-.003	.998

a. . Dependent Variable: nickel concentration (mg/L)

Table 7. Bivariate Correlation

		acid conc. (molar)	leaching temp. (°C)	nickel conc. (mg/L)
acid conc. (molar)	Pearson Correlation	1	.000	.501*
	Sig. (2-tailed)		1.000	.034
	N	18	18	18
leaching temp. (°C)	Pearson Correlation	.000	1	-.001
	Sig. (2-tailed)	1.000		.998
	N	18	18	18
nickel conc. (mg/L)	Pearson Correlation	.501*	-.001	1
	Sig. (2-tailed)	.034	.998	
	N	18	18	18

Note: Correlation is significant at the 0.05 level (2-tailed)



(A) (B)

Figure 3. Nickel extraction from laterite concentrate using nitric acid leaching method stirred at 300 rpm. 60 min. (A) at varied leaching temperatures (65°C, 70°C, and 75°C) and (B) at varied leaching acid concentrations (2.0M, 4.0M, and 6.0M).

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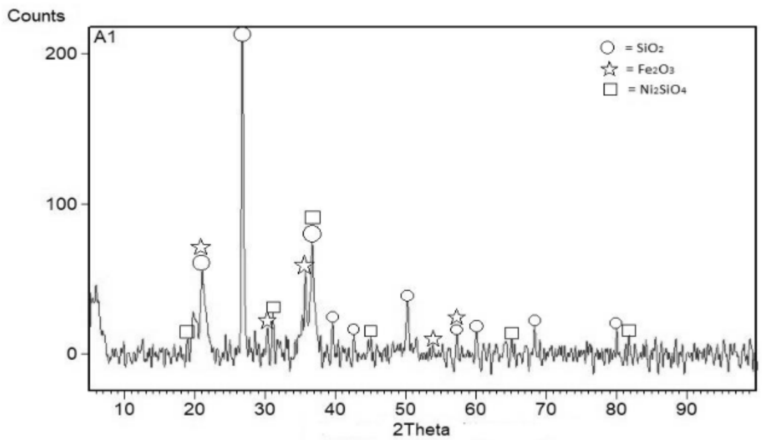

Figure 4. XRD presentation of nickel laterite sampling before roasting

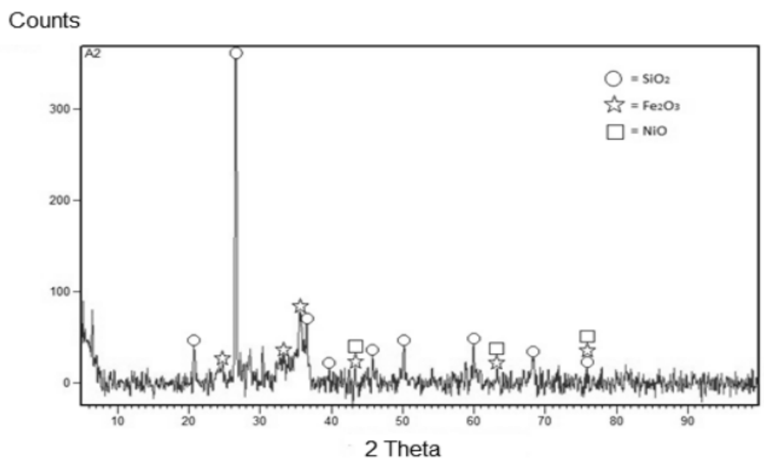

Figure 5 . XRD presentation of nickel laterite sampling after roasting.
Roasting 600°C, 1h

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