



## OPTIMIZATION OF LATERITE ORE GRINDING PROCESS USING BALL MILL WITH RESPONSE SURFACE METHOD

Gyan Prameswara\*, Flaviana Yohanala Prista Tyassena, Idi Amin, Husnul Hatimah

Mineral Chemical Engineering, Makassar ATI Polytechnic

Jl. Sunu 220, Kota Makassar, Indonesia 90211

\*E-mail: [gyan@atim.ac.id](mailto:gyan@atim.ac.id)

Received: 29-06-2022, Revised: 04-07-2022, Accepted: 31-12-2022

### Abstract

This study used a CCD (central composite design) of RSM to determine the dependence and interaction between several operating conditions that affect the grinding process using a ball mill, such as the number of balls, grinding duration, and rotational speed, on particle size at 80% product mass ( $P_{80}$ ) and mineral liberation (response surface method). The grinding process was carried out in a cylindrical ball mill with a diameter and length of 18.6 cm and 21.5 cm, respectively, as well as a steel ball with a diameter of 2.5 cm and a weight of 100 grams/ball. A sieve aperture of 180-600 microns is used to analyze the particle size distribution. The optimum data for the grinding process was obtained with the smallest response value of  $P_{80}$  (513.294  $\mu\text{m}$ ). It was known that the number of balls and grinding duration significantly affected the reduction of the  $P_{80}$  value in the sample. The model that can describe the influence of process variables on the  $P_{80}$  value was obtained with good accuracy. The elemental concentration and the XRD (x-ray diffraction) pattern were used to determine the mineral content of the sample. Minerals with a lower hardness scale are more easily liberated and exposed. The initial material's  $P_{80}$  value was 1560.89  $\mu\text{m}$ , while the  $P_{80}$  grinding process was reduced to 513.29  $\mu\text{m}$  under optimal conditions.

**Keywords:** CCD (central composite design), grinding, RSM (response surface method), laterite, nickel

## 1. INTRODUCTION

Nickel is a metal currently attracting concern due to its numerous applications, including its use in battery cathodes. The growth of the EV (electric vehicle) industry sets the stage for the rising use of high-capacity batteries. In response to this demand, the nickel refining process from primary and secondary sources must be carried out. Because saprolite resources are depleted and limonite is abundant, the investigation is primarily focused on nickel laterite discovered in limonite. Indonesia has 1.576 million tons of laterite reserves, the third-largest after New Caledonia and the Philippines [1]-[2]. The topic of the nickel refining process becomes quite intriguing to be addressed as a result of these various concerns. The nickel concentration of limonite ore ranges between 0.8 and 1.5 percent. The low nickel content is also essential when creating an optimum processing method. One of these processes is the use of a ball mill for grinding.

The preliminary laterite processing is critical for the following process stage. The grinding process is the first step in separating liberated minerals from undesired minerals and increasing surface area. Grinding consumes a significant amount of energy in the initial stages of the mineral processing process. The grinding process can consume up to 70% of the energy in a mineral processing plant [3]-[4]. Typically, the grinding step occurs towards the end of the comminution process. A ball mill is a type of grinding machine that uses a combination of impact and abrasion to reduce particle size [5]. The key to successful mineral processing is to create optimal operating conditions.

Considerable research has been conducted to investigate mineral grindability. However, few have investigated the optimization of the grinding process itself, particularly for nickel laterite. Velázquez et. al., [6] investigated the grindability of nickel laterite with various serpentine combinations. Tong et. al., [7] used a stirred mill

DOI : 10.14203/metalurgi.v37i3.655

© 2021 Metalurgi. This is an open access article under the CC BY-NC-SA license (<https://creativecommons.org/licenses/by-nc-sa/4.0/>)

Metalurgi is Sinta 2 Journal (<https://sinta.ristekbrin.go.id/journals/detail?id=3708>) accredited by Ministry of Research & Technology, Republic Indonesia

to examine the selective comminution of goethite from saprolite. Petrakis et. al., [8] used the population balance approach to examine the effect of the number of balls on particle size reduction in the ball mill during a selective grinding process on low-grade laterite. Based on the research that has been done, there hasn't been much discussion about optimization with the RSM (response surface method) in the grinding process of laterite ore using a ball mill. The goal of this study is to see how the number of balls, grinding duration, and ball mill rotational speed affect the size of 80 percent of the particles that pass through the grinding process ( $P_{80}$ ). The particle distribution before and after milling was investigated using a sieve shaker with an aperture size range of 180-600  $\mu$ m.

## 2. MATERIALS AND METHODS

### 2.1 Mineral Characterization

The laterite ore utilized in the research was obtained from Morowali, Central Sulawesi. Elemental analysis was carried out using an XRF (x-ray fluorescence) Rigaku Primini Benchtop with a 5-minute scan duration. Table 1 shows the findings of the analysis.

Mineral phase analysis was also performed using an XRD (x-ray diffraction) SmartLab using Cu-K $\alpha$  radiation in the  $2\theta$  range of 3-90 $^\circ$ .

Table 1. The elemental concentration of laterite ore (wt.%)

Ni	Co	Si	Al	Fe	Ca
1.90	0.16	9.36	2.22	13.82	0.44
Mg	Li	LOI*			
5.13	0.82	0.81			

\*Lost on Ignition.

The  $P_{80}$  value of the initial sample is 1560.89  $\mu$ m.  $P_{80}$  value indicates 80% of the total particles have a size of 1560.89  $\mu$ m.

Table 3. Feed sample particle distribution

Aperture ( $\mu$ m)	Average aperture size ( $\mu$ m)	Retained mass (gram)	% mass (%)	Cummulative oversize (%)	Cummulative undersize (%)
+600	600.000	340.000	73.325	73.325	26.675
+500-600	550.000	16.585	3.577	76.902	23.098
+425-500	462.500	13.642	2.942	79.844	20.156
+355-425	390.000	11.179	2.411	82.255	17.745
+212-355	283.500	35.469	7.649	89.904	10.096
+180-212	196.000	10.953	2.362	92.266	7.734
-180	180.000	35.861	7.734	100.000	0.000
total		463.689	100.000		

### 2.3 Design of Experiment and Statistical Analysis

CCD (central composite design) was used. The Minitab 19 software is also used for data processing. This method determined the effect of independent variables (number of balls, grinding duration, and rotational speed) on the particle

### 2.2 Grinding Process Using Ball mill

A lifter-equipped cylindrical ball mill with a diameter of 18.6 cm and a length of 21.5 cm is used for grinding. The ball mill is powered by a motor connected to a belt. The number of balls (5-15), the duration of the grinding (5-15 minutes), and the rotation speed of the ball mill were the experiment's independent variables (8-16 rpm). The mass and diameter of each steel ball used in this study are shown in Table 2. Each experiment required 200 grams of each sample.

Table 2. Steel ball characteristic

Balls	Mass (g)	Diameter (cm)
1	80.053	2.35
2	89.542	2.78
3	89.626	2.79
4	91.752	2.80
5	91.957	2.81
6	92.254	2.81
7	92.553	2.82
8	92.607	2.82
9	92.667	2.82
10	92.701	2.83
11	93.554	2.85
12	93.853	2.86
13	93.860	2.86
14	93.860	2.86
15	93.874	2.87
Total	1374.713	
Average	91.647	2.82

Following the grinding process, a sieve with a aperture size range of 180-600  $\mu$ m was performed using the Ristech Test Sieve (ASTM E 11-09). The mean diameter of solid particles was correlated with the average aperture size of each sieve. The mass fraction (percent) and cumulative mass passed are calculated using the mass of particles measured in each sieve (percent).

size value of 80% of the total particles ( $P_{80}$ ). CCD employs 2n (n=3) factorial experiments, 2n axial experiments, and nc center experiments. The goal of this experiment is to identify the optimal operating conditions for the grinding of laterite ore. The model uses a second-order polynomial to correlate the response ( $P_{80}$ ) to the

three independent variables, as shown in equation (1).

$$Y = b_0 + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij} x_i x_j \quad (1)$$

Where Y is the response ( $P_{80}$ ),  $b_0$  is the constant coefficient,  $b_i$  is the linear coefficient,  $b_{ij}$  is the interaction coefficient,  $b_{ii}$  is the quadratic coefficient, and  $x_i, x_j$  are independent variables. Table 4 shows the grinding process level used in this experiment.

Table 4. Variable level of grinding process

Variables	Label	Unit	Level		
			-1	0	1
Number of balls	A	-	5	10	15
Grinding duration	B	minutes	5	10	15
Rotating speed	C	rpm	8	12	16

### 3. RESULT AND DISCUSSION

#### 3.1 Mineral Characterization

Table 1 presents the concentration of each feed component. The nickel concentration in the feed was 1.9 percent, with Fe of 13.82 percent being the highest concentration in the laterite ore. Undesired elements such as Si, Mg, and Al were present at 9.36; 5.13; and 2.22 percent, respectively.

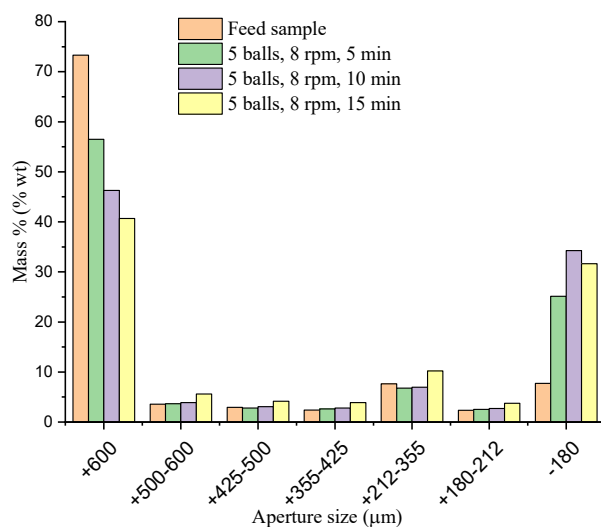


Figure 2. Particle size distribution with 5 balls and cumulative undersize

The mineral phase in the feed material is characterized via an XRD (x-ray diffraction) pattern (Fig. 1).

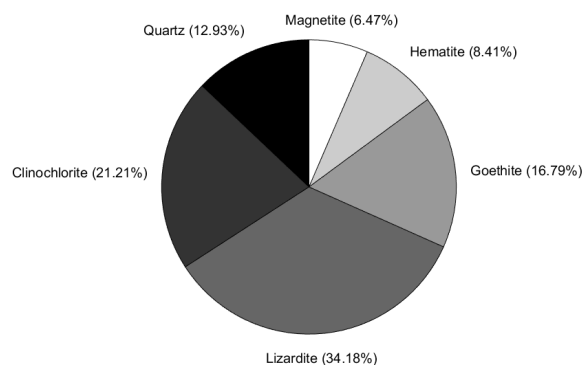
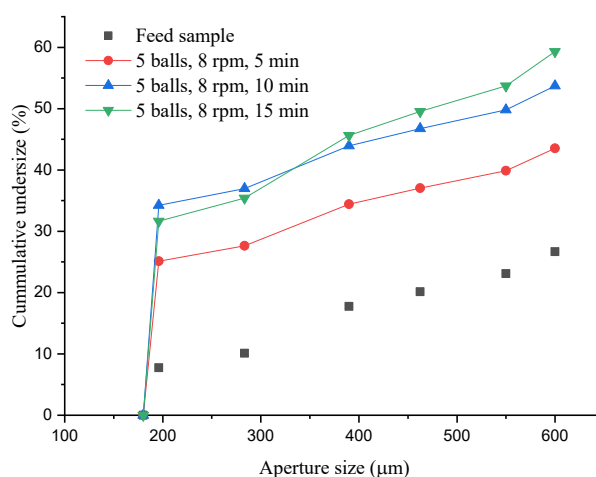


Figure 1. The mineral phase in the laterite ore

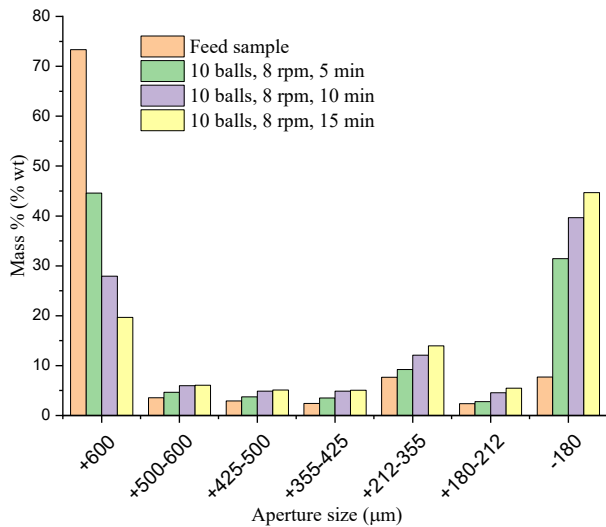
The material's XRD pattern analysis indicated that the most prominent phases were lizardite, clinoclhorite, goethite, and quartz, with hematite and magnetite appearing in minor amounts.

#### 3.2 Particle Size Distribution

Particle distribution analysis on the sieve was performed by obtaining retained mass data on each sieve. A wide range of aperture sizes ranged from 180 to 600 μm. The feed material and the grinding product from a ball mill were used to investigate the influence of the number of balls and residence time on the particle size distribution of laterite ore. The frequency distribution of the feed material is more frequent and homogenous at particle diameter sizes greater than 600 μm, as seen in Figs. 2-4.



The higher the number of balls used, the more particles grind to smaller particle sizes. The diameter of laterite ore particles was uniform at sizes smaller than 180  $\mu\text{m}$  at maximum operating



conditions (15 balls, 15 minutes, and 8 rpm). Meanwhile, the grinding process with many balls and a short duration produces a bimodal particle distribution pattern (which has two peaks).

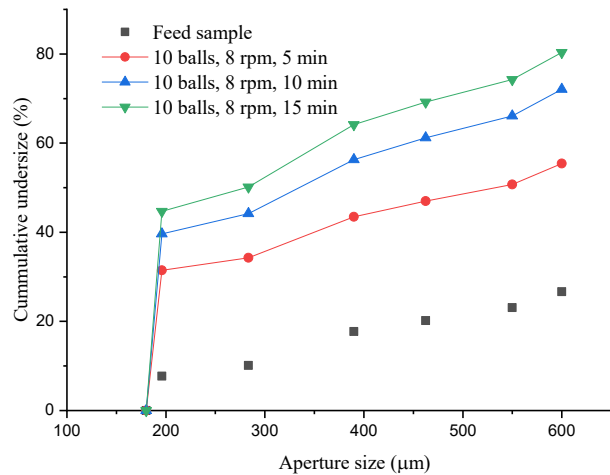


Figure 3. Particle size distribution with 10 balls and cumulative undersize

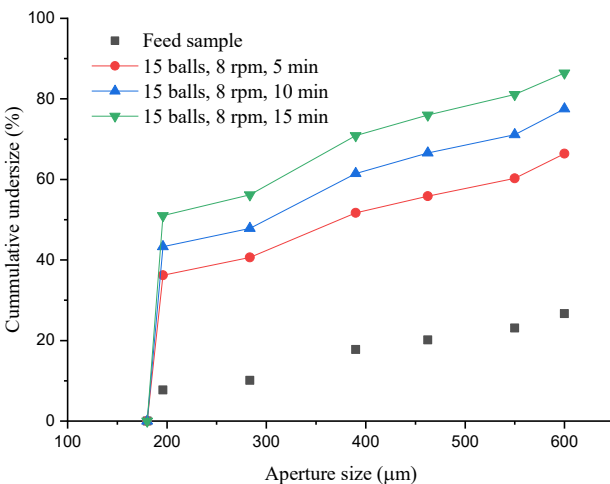
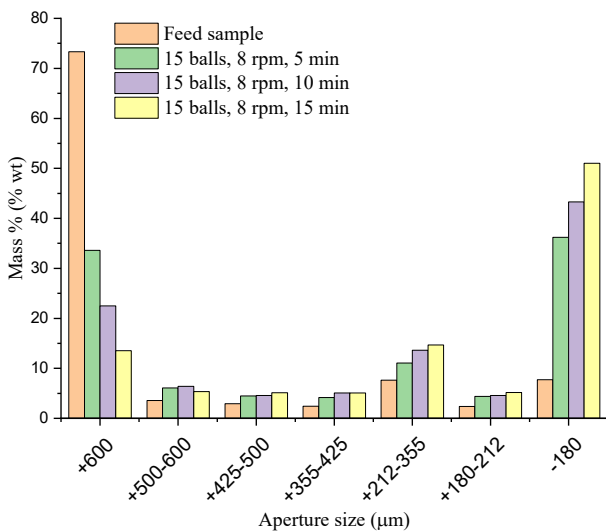


Figure 4. Particle size distribution with 15 balls and cumulative undersize

### 3.3 Grinding Process Optimization Using the RSM Approach

Data from three levels of the CCD (central composite design) were analyzed to find the best model to represent the relationship between the three independent variables (number of balls, grinding duration, and rotating speed) and the response ( $P_{80}$ ). Table 5 shows the actual  $P_{80}$  observed in the experiment and the predicted  $P_{80}$  from the model.

A quadratic model was developed to describe the relationship between the operating conditions and the obtained  $P_{80}$ . As seen in equation (2), the model is a polynomial of order 2:

$$Y = 1810 - 102.8A - 59.2B - 11.5C + 2.48A^2 + 0.31B^2 - 2.04C^2 + 0.683AB + 1.849AC + 2.698BC \quad (2)$$

Where Y represents the  $P_{80}$  value ( $\mu\text{m}$ ), A represents the number of balls, B represents the grinding duration (minutes), and C represents the rotating speed (rpm). The obtained model has an excellent correlation coefficient ( $R^2 = 0.9769$ ), which describes the model utilized quite well.

Table 5 shows the sum of squares and mean squares for each factor and the F-value and P-value. F-value describes the distribution of the mean of the test sample or the mean square divided by the error. While the P-value shows the significance of the test variable (P-value < 5%).

In addition, analysis of variance (ANOVA) was used to explain the model obtained, as shown in Table 6. It is clear that factors A, B, C, AC, and BC are significant in this experiment. However, factors  $A^2$ ,  $B^2$ ,  $C^2$ , and AB are not.

Table 5. Comparison of the experimental  $P_{80}$  value and predictions from the obtained model

Number of balls	Time (min)	Rotating speed (rpm)	$P_{80}$ ( $\mu\text{m}$ )	
			Experiment	Prediction
15	5	16	545.072	545.548
5	5	16	723.489	746.229
5	15	16	687.492	681.624
15	15	16	532.864	549.260
15	5	8	688.908	698.712
5	5	8	1059.760	1047.30
5	15	8	771.299	766.887
15	15	8	513.294	486.618
10	5	12	734.766	729.950
10	15	12	571.040	591.600
10	10	16	594.558	560.814
10	10	8	630.540	680.028
10	5	8	826.643	810.899
10	10	12	660.804	653.030
10	10	12	661.000	653.030

The smaller the P-Value, the greater the significance of the variable to the change in response. According to previous studies, the significance value is found in factors with a P-value of  $\leq 0.05$  [9]-[12].

Table 6. Analysis of variance (ANOVA)

Source	DF	Sum of squares	Mean square	F-Value	P-Value
Model	9	262869	29208	23.52	0.0014
A	1	115657	115657	93.14	0.0002
B	1	49608	49608	39.95	0.0015
C	1	36834	36834	29.66	0.0028
$A^2$	1	4655	4655	3.75	0.1106
$B^2$	1	93	93	0.07	0.795
$C^2$	1	1646	1646	1.33	0.3017
AB	1	2334	2334	1.88	0.2287
AC	1	10938	10938	8.81	0.0312
BC	1	24364	24364	19.62	0.0068
Error	5	6209	1242		
Lack-of-Fit	4	6208	1552	80897.9	0.026
Pure Error	1	0	0		
Total	14	269077			

A=Number of ball, B=grinding time (min), C=Rotating speed (rpm)

Figure 5 depicts the effect of the number of balls, grinding duration, and rotating speed on the  $P_{80}$  value. In comparison, the feed material's  $P_{80}$  value is  $1560.89 \mu\text{m}$ . It can be seen that the

number of balls and the grinding time significantly impact the value of  $P_{80}$ . The larger

This is due to the grinding process using the dry method so that the ore will quickly move in the direction of the ball being lifted by the lifters. As a result, the probability of a collision between the ball and the laterite ore is reduced. Previous research has found similar results [5], [13]-[15].

This investigation obtained the optimum process conditions for the grinding of laterite ore in a ball mill. The optimum processing conditions are 15 balls, 15 minutes of grinding time, and an 8 rpm rotational speed (Figs. 5(a)-(c)).

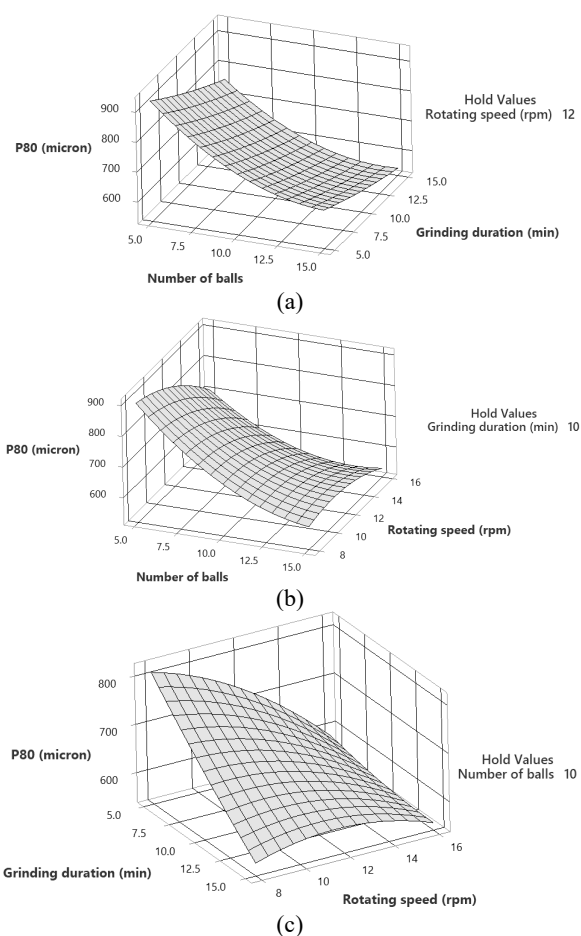


Figure 5. The effect of (a) number of balls and grinding duration, (b) number of balls and rotating speed, and (c) grinding duration and rotating speed on the  $P_{80}$  value

Figures 6 and 7 show component concentrations at optimum conditions as well as a comparison of feed material XRD patterns at optimum conditions. The decrease in  $P_{80}$  potentially indicates mineral liberation in the laterite material and an increase in the material's surface area. The  $P_{80}$  value obtained at the optimum grinding conditions was  $51.29 \mu\text{m}$ .

Minerals with high hardness, such as quartz, are detected less than the feed material. This is

because the harder the mineral is to release, the stronger the bonds between the minerals.

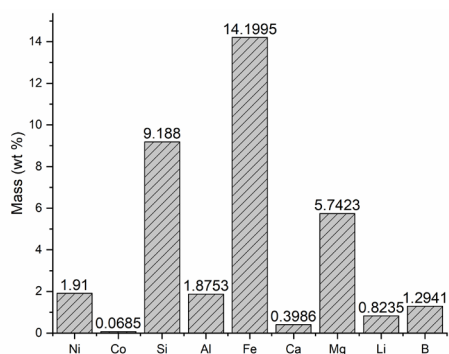


Figure 6. Elemental concentration of grinding product at optimum operating conditions

The hardness scale values of each mineral identified in laterite ore Morowali are shown in Table 7 [13]-[14].

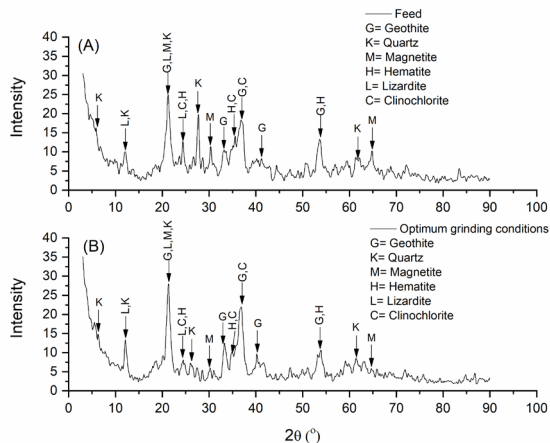


Figure 7. XRD pattern of laterite ore grinding products at optimum operating conditions

The hardness data for each of these minerals shows that the lowest to highest hardness scale values are clinoclortite, lizardite, goethite, magnetite, hematite, and quartz, respectively.

Table 7. The hardness scale (Mohs's scale) of the mineral in the laterite ore Morowali

Minerals	Mohs's	
	Hardness	Wt.%
Quartz	7	12.93
Clinoclortite	2.5	21.21
Lizardite	2.5	34.18
Goethite	3.8	16.79
Hematite	6.5	8.41
Magnetite	5.75	6.47

Lizardite was the most dominant phase in the sample, accounting for 34.18 percent of the total.

#### 4. CONCLUSION

Initial characterization data and grinding process optimization for Morowali laterite ore

have been obtained. In the Morowali laterite ore, it is evident that there are nickel carrier minerals such as goethite, which contain 1.9% nickel. The most common minerals are lizardite, clinoclortite, goethite, and quartz. In contrast, hematite and magnetite are only present in trace concentrations.

The size distribution of laterite ore in grinding products has a bimodal distribution. The response surface method was used to optimize the grinding process by determining the correlation between process conditions and the  $P_{80}$  value and determining the optimum grinding conditions. This optimization method applies CCD (central composite design). The analytical results show that statistical methods can assist in determining the optimum point of the laterite ore grinding process. The optimal grinding conditions were obtained using 15 balls, 15 minutes of grinding time, and 8 minutes of rotation speed. The material's  $P_{80}$  value was decreased from 1560.89  $\mu\text{m}$  to 513.29  $\mu\text{m}$  under these conditions. This is considered useful information for the Indonesian laterite mineral processing industry.

#### ACKNOWLEDGMENT

The authors would like to thank Makassar ATI Polytechnic for funding the study.

#### REFERENCES

- [1] Kementerian Energi dan Sumber Daya Mineral, *Indonesian Minerals Year Book 2018*. Jakarta: Kementerian Energi dan Sumber Daya Mineral, 2018. Accessed: Nov. 12, 2021. [Online]. Available: <https://geologi.esdm.go.id/assets/media/content/content-indonesian-minerals-yearbook-2018.pdf>.
- [2] S. Stopic and B. Friedrich, "Hydrometallurgical processing of nickel lateritic ores," *Vojnotehnicki glasnik*, vol. 64, no. 4, pp. 1033-1047, 2016. Doi: 10.5937/vojtehg64-10592.
- [3] E. Petrakis and K. Komnitsas, "Improved modeling of the grinding process through the combined use of matrix and population balance models," *Minerals*, vol. 7, no. 5, pp. 67-84, 2017. Doi.org/10.3390/min7050067.
- [4] J. V. Nava, T. Llorens, and J. M. Menéndez-Aguado, "Kinetics of dry-batch grinding in a laboratory-scale ball mill of Sn-Ta-Nb minerals from the Penouta mine (Spain)," *Metals*, vol. 10, no. 12, pp. 1687-1706, 2020. Doi.org/10.3390/met10121687.
- [5] R. Zhao, Y. Han, M. He, and Y. Li, "Grinding kinetics of quartz and chlorite

- in wet ball milling,” *Powder Technology*, vol. 305, pp. 418-425, 2017. Doi: 10.1016/j.powtec.2016.07.050.
- [6] H.-I. Yoo, “Diffusion in continuum,” in *Lectures on Kinetic Processes in Materials*, Cham: Springer International Publishing, pp. 1-16, 2020. Doi: 10.1007/978-3-030-25950-1.
- [7] L. Tong, B. Klein, M. Zanin, K. Quast, W. Skinner, J. Addai-Mensah, and D. J. Robinson, “Stirred milling of nickel laterites for selective comminution,” in *SAG Conference*, pp. 1-20, 2015. Doi: 10.1007/978-3-030-25950-1.
- [8] E. Petrakis, V. Karmali, and K. Komnitsas, “Factors affecting nickel upgrade during selective grinding of low-grade limonitic laterites,” *Mineral Processing and Extractive Metallurgy*, vol. 130, no. 3, pp. 192-201, 2018. Doi: 10.1080/25726641.2018.1521578.
- [9] I. Ani, J. Okafor, M. Olutoye, and U. Akpan, “Optimization of base oil regeneration from spent engine oil via solvent extraction,” *Advances in Research*, vol. 4, no. 6, pp. 403-411, 2015. Doi: 10.9734/air/2015/16795.
- [10] Z. N. Garba, I. Bello, A. Galadima, and A. Y. Lawal, “Optimization of adsorption conditions using central composite design for the removal of copper (II) and lead (II) by defatted papaya seed,” *Karbala International Journal of Modern Science*, vol. 2, no. 1, pp. 20-28, 2016. Doi: 10.9734/air/2015/16795.
- [11] E. Sasikumar and T. Viruthagiri, “Optimization of process conditions using response surface methodology (RSM) for ethanol production from pretreated sugarcane bagasse: kinetics and modeling,” *BioEnergy Research*, vol. 1, no. 3-4, pp. 239-247, 2008. Doi: 10.9734/air/2015/16795.
- [12] J. Bayuo, M. A. Abukari, and K. B. Pelig-Ba, “Optimization using central composite design (CCD) of response surface methodology (RSM) for biosorption of hexavalent chromium from aqueous media,” *Applied Water Science*, vol. 10, no. 6, pp. 135-147, 2020. Doi: 10.9734/air/2015/16795.
- [13] C. S. Sridhar, P. S. Sankar, and R. K. Prasad, “Grinding kinetics, modeling, and subsieve morphology of ball mill grinding for cement industry ingredients,” *Particulate Science and Technology*, vol. 34, no. 1, pp. 1-8, 2016. Doi: 10.9734/air/2015/16795.
- [14] T. S. Yusupov, E. A. Kirillova, and L. G. Shumskaya, “Mineral hardness effect on the combined mineral grinding,” *Journal of Mining Science*, vol. 43, no. 4, pp. 450-454, 2007. Doi: 10.1007/s10913-007-0046-y.
- [15] T. P. Olejnik, “Selected mineral materials grinding rate and its effect on product granulometric composition,” *Physicochemical Problems of Mineral Processing*, vol. 49, no. 2, pp. 407-418, 2013. Doi: 10.5277/ppmp130203.