Effect of Agitation Speed and Leaching Time for Nickel Recovery of Morowali Limonite Ore in Atmospheric Citric Acid Leaching

Regna Tri Jayanti[1*], Yusdianto[2]

[12]Department of Mineral Chemical Engineering, Politeknik Industri Logam Morowali (PILM), Morowali – INDONESIA

* Corresponding author e-mail: regnatj@gmail.com

Abstract

This research work deals with atmospheric citric acid leaching of limonite ore from Morowali, Central Sulawesi by applying agitation speed and leaching time parameters. The composition of limonite ore treatment was characterized by X-Ray Fluorescence (XRF) method. Its containt dominated by iron 84.2 % and enrichment nickel content up to 5.15 %. The microstructure image of limonite ore was observed by Scanning Electron Microscope (SEM) whereas nickel recovery was confirmed by Atomic Absorption Spectroscopy (AAS). Leaching process was conducted by using citric acid in a flask on a magnetic heated stirrer at constant temperature. The effect of agitation speed on atmospheric leaching in citric acid indicated enhancement of nickel recovery. The agitation speed ranges employed were 100 to 1000 rpm with leaching times of 10 to 120 minutes. This study revealed that atmospheric citric acid leaching of limonite ore from Morowali under the condition temperature 70 ^oC, acid concentration 1 M, agitator speed 1000 rpm could recover nickel of 2824 ppm at 120 minutes of leaching time.

Keywords: Atmospheric leaching, Limonite ore, Agitation speed, Citric Acid, Leaching time

1. Introduction

Indonesia has approximately 16 % of total global nickel resources in the form of lateritic ores. They are widely distributed and spread among the islands of Kalimantan, Sulawesi, Halmahera, Gag, and Papua (Astuti *et al.*, 2016a). Central Sulawesi has big potential for mineral resources of nickel laterite deposition especially in Morowali (Hardyanto, Widodo and Nurwaskito, 2015). Indonesia applies pyro-metallurgy method to process nickel laterite rather than hydrometallurgy in order to produces nickel alloy and nickel matte (Mubarok and Hapid, 2014). Solihin and Firdiyono (2014) explained that alternative way to process nickel laterite in particular limonite is hydrometallurgy which has low operation temperature. The more applicable Hydrometallurgical processes to the limonite (high iron laterite) are ammonia-ammonium carbonate leaching (Caron process), atmospheric leaching, and high pressure acid leaching (HPAL) (Guo *et al.*, 2011).

Atmospheric pressure acid leaching has been recently receiving more attention due to higher capital cost and materials of construction problems of high-pressure acid leaching at commercial level. It involves direct leaching of nickel laterite ores either both inorganic and/or organic acids by either agitation or heap leaching (Mohammadreza, Mohammad and Ziaeddin, 2019). Acid leaching has become the primary technology for processing nickel laterite ores because of its advantage on recovers the elements such as nickel, cobalt, iron, and magnesium comprehensively (Liu, Chen and Hu, 2009). Inorganic acid utilization of leaching process ensued harmful effect to the environment that require acid waste handling (Gustiana, 2018). Citric acid is one of organic acid that shows good mechanism as a bonding agent in metal separation from the minerals. The hydronium ion of citric acid attack acid and metal ions to form metal-ligand compound and dissolved the metal (McDonald and Whittington, 2008; Astuti *et al.*, 2016b). Agitation has a pronounced effect on the leaching copper because of the increase in the rate of copper leaching with increasing the degree of stirring suggested that the reaction is either fully diffusion controlled or under mixed diffusion and chemical control (El-okazy, Zewail and Farag, 2018).

In this atmospheric citric acid leaching experiment, the effect of different parameters on agitation speed and leaching time was examined. Therefore, the aim of this study is to investigate recovery of nickel trough agitation leaching and leaching time of limonite ore at atmospheric citric acid leaching.

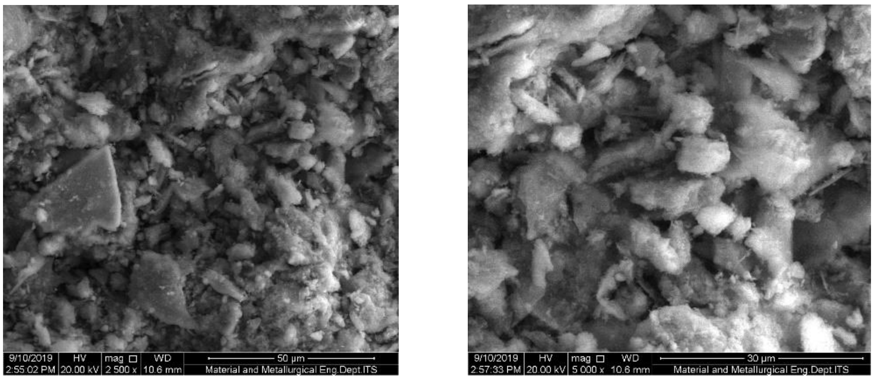
2. Materials and methods

2.1. Materials

The representative samples used in this research is limonite ore type from mine area of Morowali, Central Sulawesi. Preparation of sample started by reducing water content in an oven at temperature 110ºC along 4 hours, then sample was grinded by using a laboratory Jaw crusher and Hammer mill. At last, ore was sieved by sieve shaker until the particle size reach of 100 mesh. Recent study stated that the particle size of -100+200 mesh has good contribution in leaching limonite ore (Mubarok, Astuti and Chaerun, 2013). The chemical composition of sample was examined by X-Ray Fluorescence Zetium Panalytical and listed in table 1. Scanning Electron Microscope (SEM) analysis supported mineral formation, since the particles composed by metal elements. Samples consist of heterogeneous form and grain sizes are quite small (below -50 μm), it also has a flattened morphology (Fig.1). This is adequate a valuable increase in particle size since both the nickel and iron powders are originally <10 μm (Fischmann and Dixon, 2009).

Table 1: Composition of Limonite ore of Morowali

Sample	**Ore Composition (wt%)**									
	Si	P	Ca	Sc	V	Cr	Mn	Fe	Ni	Zn
Limonite ore	6.5	0.25	0.61	0.01	0.054	1.86	1.3	84.2	5.15	0.1



(a) (b)

Fig. 1: Microstructure of limonite ore of Morowali (a). Magnification 2500x and (b). Magnification 5000x

2.2. Methods

Atmospheric pressure acid leaching was performed in a flask equipped with magnetically heated stirrer and thermometer. The comparison of sample and citric acid was 8.3 % w/v. The parameters selected in this experiment were temperature (ºC), type of acid, and acid concentration (M). As counted in XRF data of mineral composition, predominant element of Morowali limonite ore was iron. Mcdonald and Whittington, (2008) claimed that iron extraction could be controlled if the total chloride concentration is kept below 230 g/L and the initial acidity is maintained between 1 M and 2 M. Therefore, acidity of citric acid used in this work is 1 M. Citric acid was added and heated until the temperature reached 70ºC. Then, limonite ore was poured gently to the opening flask and leaching time starts observed at the time of solid sample added. The effect of leaching time was determined as the sample taken after period of time 10, 20, 30, 60, 90, and 120 minutes. All the experiments were kept on for 2 h and final solutions were filtered using a paper filter. To calculate nickel recovery, filtered solution was analyzed by using Atomic Absorption Spectroscopy (AAS).

3. Results and discussion

3.1. Influence of agitation speed

The influence of agitation speed on variation in the nickel recovery responses was evaluated. The results indicated agitation speed had convincing effect. Figure 2 shown the effect of agitation speed in the range 100 – 750 rpm on the reductive leaching of limonite ore. Moreover, slight decrease in nickel recovery was detected when agitation speed at 750 rpm. The reason of the phenomena was probably due to instabilities of the contact area between limonite ore and citric acid. Since leaching kinetics increased as the intensive mixing among them, the enhancement of nickel recovery tends to rise in the long period of reaction time and the high agitation speed of leaching. Consequently, the agitation speed of 1000 rpm was quite promising to ensure the control of chemical reaction with the mineral grains (limonite ore) in atmospheric citric acid leaching.

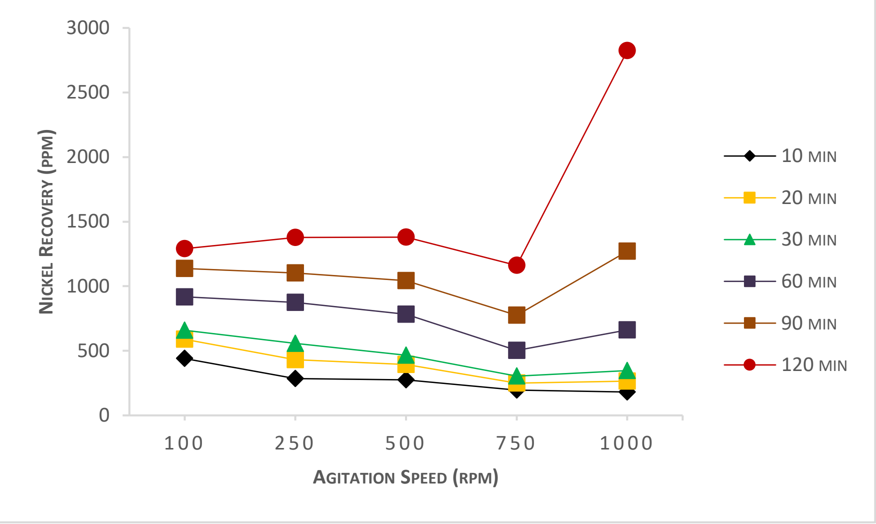


Fig. 2: Effect of agitation speed on nickel recovery

3.2. Influence of leaching time

The effect of reaction time on atmospheric citric acid leaching of limonite ore is given in figure 3. The leaching efficiency of nickel recovery obtained by varying reaction time 10, 20, 30, 60, 90, and 120 minutes. At the beginning of reaction time, nickel recovery tends to decreased along the higher agitation speed. The leaching efficiency curves shows that nickel recovery increases with a slight augment in reaction time of 10 minutes to 120 minutes for agitation speed from 100 rpm to 750 rpm. Furthermore, the recovery of nickel increases dramatically at agitation speed 1000 rpm in reaction time from 30 minutes to 120 minutes.

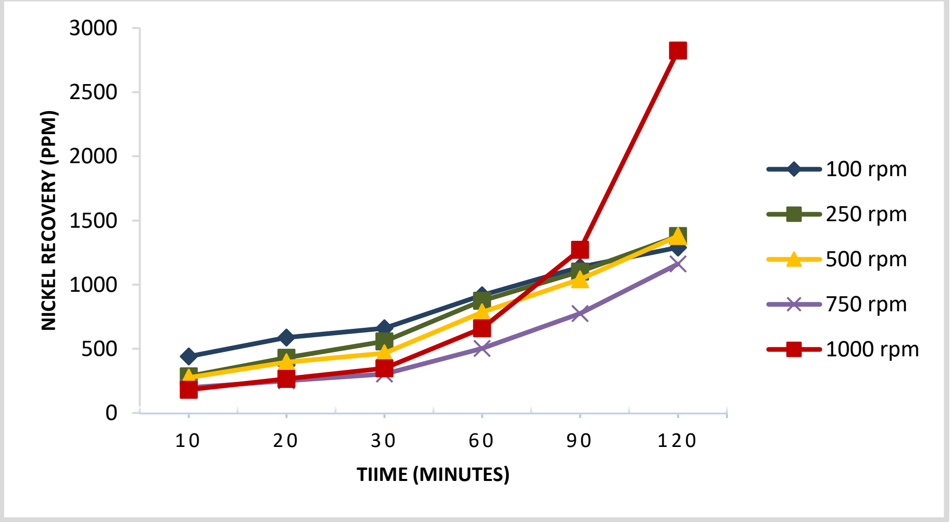


Fig. 3: Effect of leaching time on nickel recovery

4. Conclusion

Agitation speed and leaching time experiments showed that nickel from limonite ore could be recovered at atmospheric condition. Two factorial experimental were used to distinguish significant parameters on the process. The representative sample of nickel laterite ore assayed by XRF analysis principally consisted of Fe as a major element 84.2 %, Si 6.5 %, Ni 5.15%, Cr 1.86 %, Mn 1.3% and other metal elements below 1%.

Results analysis were done by Atomic Absorption Spectroscopy (AAS) and exhibited that atmospheric citric acid leaching could solubilize nickel. By increasing agitator speed from 100 to 1000 rpm and leaching time process from 10 to 120 minutes, the effect of agitation speed and leaching time showed the enhancement of nickel recovery indirectly.

Agitation speed enlarged the contact of limonite ore with citric acid that helped leaching reaction done well. The leaching time effected to nickel recovery by the period of interaction between limonite ore and citric acid. The results obtained from this research indicate that the agitation speed and leaching time parameters could be implemented to identify enhancement of nickel recovery on atmospheric citric acid leaching of limonite ore from Morowali under the condition temperature 70 ºC, acid concentration 1 M and agitation speed 1000 rpm. The amount of nickel recovery by this reseaach work reached 2824 ppm after reaction time of 120 minutes.

5. Nomenclature

h hour

M Molarity

ppm part per millions

rpm rotary per minutes

w/v weight per volume

6. Acknowledgements

This work was funded by Politeknik Industri Logam Morowali. The authors gratefully acknowledge the Professor Isa Setiasyah Toha for valuable comments, Mineral Chemical Department Colleague for supporting many of information sources, also Feby Aryani and Wirawan for the helpful efforts.

7. References

Astuti, W. et al. (2016a) ‘Comparison of atmospheric citric acid leaching kinetics of nickel from different Indonesian saprolitic ores’, Hydrometallurgy. Elsevier B.V., 161, pp. 138–151. doi: 10.1016/j.hydromet.2015.12.015.

Astuti, W. et al. (2016b) ‘Comparison of effectiveness of citric acid and other acids in leaching of low-grade Indonesian saprolitic ores’, Minerals Engineering. Elsevier Ltd, 85, pp. 1–16. doi: 10.1016/j.mineng.2015.10.001.

El-okazy, M. A., Zewail, T. M. and Farag, H. A. (2018) ‘Recovery of copper from spent catalyst using acid leaching followed by electrodeposition on square rotating cylinder’, Alexandria Engineering Journal. Faculty of Engineering, Alexandria University, 57(4), pp. 3117–3126. doi: 10.1016/j.aej.2017.12.001.

Fischmann, A. J. and Dixon, D. G. (2009) ‘Awaruite (Ni 3 Fe) as a nickel resource — leaching with ammoniacal – ammonium solution containing citrate and thiosulfate’, Hydrometallurgy. Elsevier B.V., 99(3–4), pp. 214–224. doi: 10.1016/j.hydromet.2009.08.009.

Guo, X. Y. et al. (2011) ‘Leaching behavior of metals from limonitic laterite ore by high pressure acid leaching’, Transactions of Nonferrous Metals Society of China (English Edition), 21(1), pp. 191–195. doi: 10.1016/S1003-6326(11)60698-5.

Gustiana, H. S. E. A. (2018) ‘Pelindian Nikel dari Nikel Laterit Pomalaa Menggunakan Asam Asetat’, Tesis Sarjana S-2 Program Studi Teknik Kimia, Magister Teknik Proses Pengolahan Mineral, (April), pp. 1–7.

Hardyanto, Widodo, S. and Nurwaskito, A. (2015) 'Pemodelan endapan nikel laterit, kabupaten morowali, provinsi sulawesi tengah', Jurnal Geomine, 02, pp. 89–96.

Liu, K., Chen, Q. and Hu, H. (2009) 'Hydrometallurgy Comparative leaching of minerals by sulphuric acid in a Chinese ferruginous nickel laterite ore', Hydrometallurgy. Elsevier B.V., 98(3–4), pp. 281–286. doi: 10.1016/j.hydromet.2009.05.015.

Mcdonald, R. G. and Whittington, B. I. (2008) 'Atmospheric acid leaching of nickel laterites review . Part II . Chloride and bio-technologies', 91, pp. 56–69. doi: 10.1016/j.hydromet.2007.11.010.

McDonald, R. G. and Whittington, B. I. (2008) 'Atmospheric acid leaching of nickel laterites review. Part I. Sulphuric acid technologies', Hydrometallurgy, 91(1–4), pp. 35–55. doi: 10.1016/j.hydromet.2007.11.009.

Mohammadreza, F., Mohammad, N. and Ziaeddin, S. S. (2019) 'Nickel extraction from low grade laterite by agitation leaching at atmospheric pressure International Journal of Mining Science and Technology Nickel extraction from low grade laterite by agitation leaching at atmospheric pressure', International Journal of Mining Science and Technology. China University of Mining & Technology, 24(4), pp. 543–548. doi: 10.1016/j.ijmst.2014.05.019.

Mubarok, M. Z. and Hapid, A. (2014) 'PELINDIAN BIJIH NIKEL LATERIT SULAWESI TENGGARA', in Prosiding Pemaparan Hasil Penelitian Pusat Penelitian Geoteknologi LIPI Tahun 2014. Tanggerang, pp. 527–534.

Mubarok, Z., Astuti, W. and Chaerun, S. K. (2013) 'Effects of Individual Use , Mixed Culture and Sulfur Addition on the Effectiveness of Nickel Laterite Ore Bioelaching with Penicillium verruculosum and Galactomyces geotrichum Effects of Individual Use , Mixed Culture and Sulfur Addition on the Effectiven', (January). doi: 10.4028/www.scientific.net/AMR.825.380.

Solihin and Firdiyono, F. (2014) 'Perilaku pelarutan logam nikel dan besi dari bijih nikel kadar rendah sulawesi tenggara', Majalah Metalurgi, pp. 139–144.