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# Effect of concentration, agitation, and temperature of Pomalaa limonitic nickel ore leaching using hydrochloric acid

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**Abstract.** Pomalaa limonite nickel ore has nickel content of 1.29% and it has not been processed in Indonesia until now. In addition, The Minister of Energy and Mineral Resources Regulation No.5/2017 on adding the value of minerals. Therefore, the limonite nickel ore should be processed to produce nickel metal with the content 99.93%. The limonite nickel ore generally has low nickel content (less than 1.5%) therefore hydrometallurgy such as leaching is the suitable process. In this research is done 2 series of leaching. First, determination of leaching controlling rate by varying the temperature and time of leaching. Second, determination the optimum condition of the leaching variables (concentration of leaching agent, agitation and temperature) based on the leaching controlling rate. The amount of activation energy obtained from this research is 16.23 and 38.47 kcal/mol then it could be said that the reaction control of leaching limonite nickel ore from Pomalaa is a chemical reaction. In addition, a maximum extraction of 88.9% was achieved at 400 rpm, 6M hydrochloric acid concentration and a temperature of leaching about 90 °C.

## 1. Introduction

This research was motivated by the regulation of minister of energy and mineral resources on added value of local resources that mentioned in abstract section. There are two type resource of nickel such as nickel laterite and nickel oxide ores. Nickel laterite ores can be divided into two type i.e. saprolite and limonite. Saprolite nickel ore is high in nickel and magnesium (Ni > 1.6% and Mg > 20%), but low iron content (Fe < 20%). In Indonesia, saprolite nickel ore has been commercially processed through pyrometallurgy to produce nickel matte and ferronickel (FeNi).

It is recorded that in 2016, Indonesia is the world's sixth nickel producer with production of 160,000 tons. Indonesia has an abundant potential of nickel resources (5.8 billion tons), spread over areas such as South Sulawesi, Southeast Sulawesi, Maluku and Papua. The limonite nickel ore is a type of laterite with characteristics having a low nickel and magnesium content (Ni < 1.5% and Mg < 2%), but high iron content (Fe > 50%) [1] therefore the limonite nickel is not suitable to be processed through pyrometallurgy. It could be said that, hydrometallurgy is the suitable process for limonite nickel ore.

In nature, saprolite ore lies in the innermost layer, while limonite ore is present in the upper layer. To mine the saprolite ores must first mine the limonite layer above it. One of the areas in Indonesia that has limonite nickel ore reserves is in Pomalaa, Southeast Sulawesi with an average nickel content of 1.3% and



iron 28.63% [2]. Nowadays, the limonite layer is not processed because of its low nickel content ( $Ni < 1.5$ ) but in the presence of Ministry of ESDM regulation, it is need to do a research to process limonite nickel ore.

Leaching is a selective dissolution process that aims to dissolve precious metals from a concentrate without dissolving the impurity metal. The most influential factors in the leaching process include the concentration of leaching agent and the speed of agitation. If the reaction is controlled by a chemical reaction, the concentration of leaching agent affects the reaction whereas the speed of agitation will affect the reaction if the reaction is controlled by diffusion.

Leaching is the main stage of the hydrometallurgical process using leaching agents. the leaching technique chosen is atmospheric leaching. this process has advantages in terms of simple equipment procurement and done at atmospheric pressure [3]. In the leaching process, leaching agent plays an important role and in this research hydrochloric acid is used as leaching agents because of its ability to dissolve limonite nickel ore. Another important that should be considered in the leaching process is the reaction controlled such as diffusion or chemical controlled reaction that can be known from the amount of activation energy. Therefore, the first stage of leaching process with variations in temperature is done.

## 2. Methodology/experimental

In this research, the preparation processes include ore beneficiation (drying, crushing, and sizing) and atmospheric leaching method was used. Then, limonite nickel ore analyzed by using XRF to measure the chemical composition of the ore.

First, the leaching stage I is done with the variation of temperature (ambient, 50 and 90°C) and leaching time (3, 15, 30, 60, 90, 120, 180, 240, 360 and 480 minutes). Then, by collecting data from variation of leaching temperature and using Arrhenius equation it could be known the controlling leaching reaction steps.

Second, the leaching stage II is carried out with the variation of concentrations of hydrochloric acid (1, 4, and 6 M), agitation speeds (200, 400, and 600 rpm) and time of leaching (3, 180 and 480 minutes). After leaching is done, the filtration stage is generated then the filtrate and residue are produced. The filtrate was tested its chemical composition with AAS and the residue tested with XRF. Figures 1, 2, 3 and Table 1 show the limonite nickel ore, the laboratory scale of leaching limonite nickel, filtration process after leaching and the composition of limonite nickel ore that used in this research, respectively.



**Figure 1.** Pomalaa limonite nickel ore



**Figure 2.** Laboratory scale of leaching limonite



**Figure 3.** Filtration process

**Table 1.** Chemical composition of limonite nickel ore pomalaa

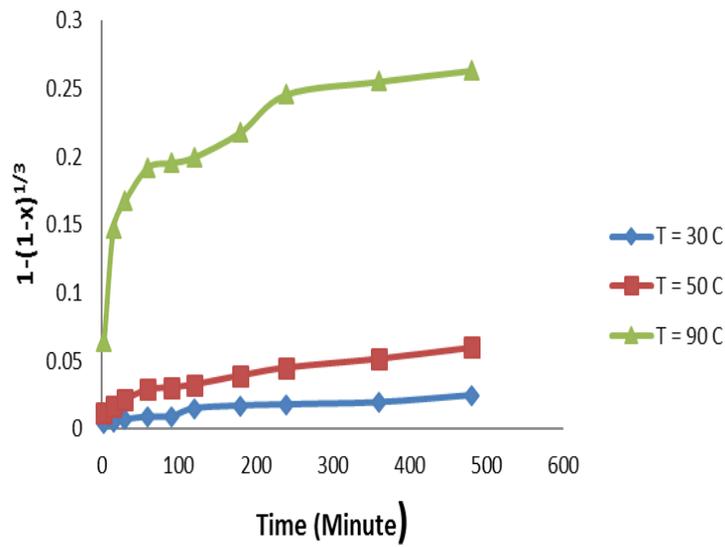
Substitute	%	Substitute	%	Substitute	%
SiO <sub>2</sub>	11.20	Na <sub>2</sub> O	0.035	Cr <sub>2</sub> O	2.18
Al <sub>2</sub> O <sub>3</sub>	6.23	K <sub>2</sub> O	0.22	NiO	1.64
Fe <sub>2</sub> O <sub>3</sub>	49.50	TiO <sub>2</sub>	0.10	Co <sub>2</sub> O <sub>3</sub>	0.10
MnO	0,61	P <sub>2</sub> O <sub>5</sub>	0.014	CuO	0.014
MgO	2.20	CaO	3.86		

### 3. Results and discussion

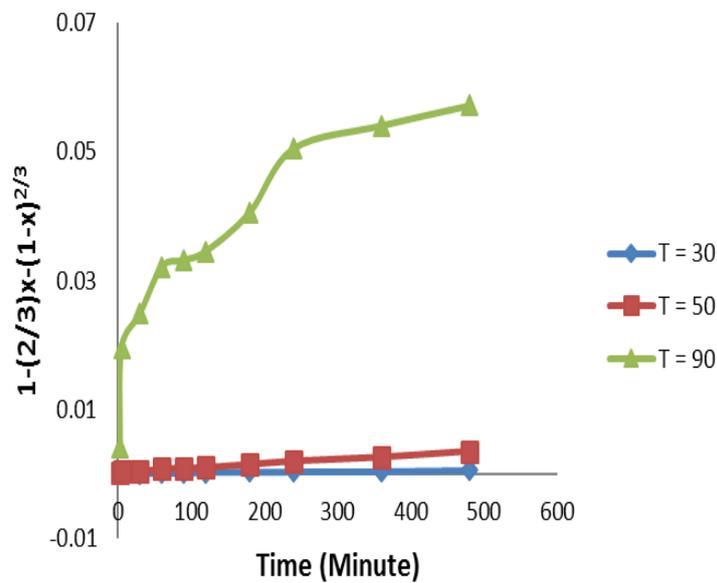
#### 3.1. Determination of rate controlling leaching

According to some researchers that there are several kinetics models are known in the leaching process such as unreacted core models [4] and Shrinking Core Models [5,6]. There are three mechanisms that can be determined by leaching process such as mass transfer from the ambient fluid through the fluid film surrounding the particle; diffusion through the inert product layer left behind as the core shrinks; chemical reaction at the surface of unreacted core. The standard Shrinking Core model is able to account. The first mechanism relatively has little impact on the leaching process [5] therefore in this research diffusion and chemical mechanisms are discussed.

On the application of shrinking core is assumed that the shape of particles is spherical. Figure 4 shows the kinetic model of leaching limonite nickel rate controlling steps such as diffusion and chemical, respectively while Table 2 shows the R<sup>2</sup> values obtained from linear regression equation using shrinking core model on Figure 4. Based on the data shown on Table 2, the value of R<sup>2</sup> equals one is a diffusion model therefore it could be said that leaching limonite nickel Pomalaa is controlled by diffusion.



(a)



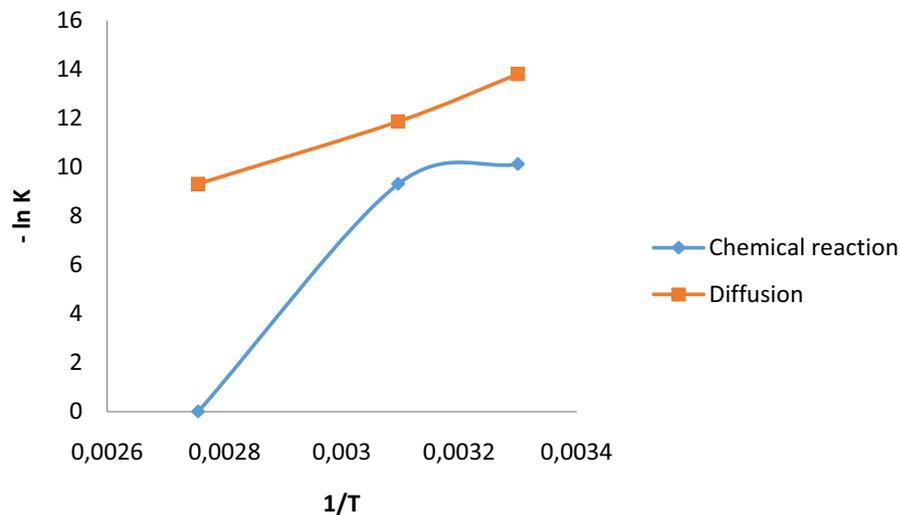
(b)

**Figure 4.** Shrinking Core Model for (a) Diffusion controlled reaction and (b) Chemically controlled reaction of leaching limonite nickel Pomalaa

**Table 2.** The Value of  $R^2$

Temperature (°C)	$R^2$	
	$1-(1-x)^{1/3}$	$1-(2/3)x-(1-x)^{2/3}$
30	0.913	0.956
50	0.932	0.993
90	0.672	0.801

In addition, the amount of activation energy can be used to determine the rate controlling step of limonite nickel leaching process. The value of the activation energy can be determined from the curve  $(-\ln K)$  vs  $(1/T)$  while the value of  $(-\ln K)$  is obtained from the slope of the curve shown in Figure 5. Figure 5 shows the curve  $(-\ln K)$  vs  $(1/T)$ .

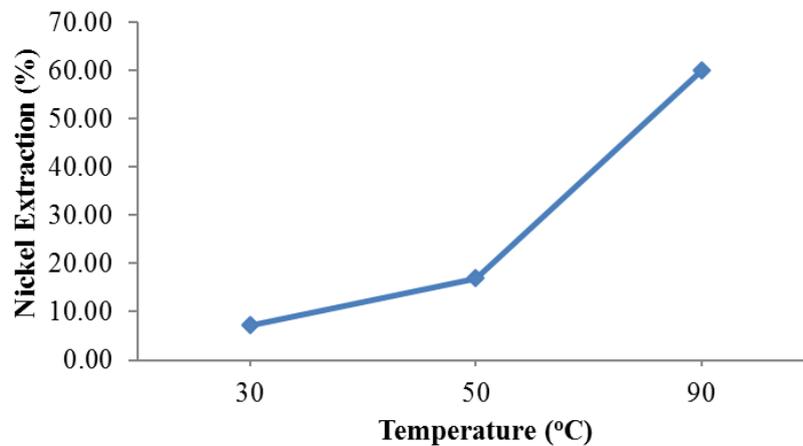


**Figure 5.** The Graph of  $(-\ln K)$  vs  $(1/T)$

Based on figure 5, the value of activation energy for chemical controlled reaction is 38.47 kcal / mol, while for diffusion controlled is 16.23 kcal / mol. According to Habashi, the activation energy with values greater than 10 Kcal / mol indicates that the process is controlled by chemical controlled reaction [7]. Therefore, it could be said that leaching limonite nickel ore from Pomalaa is chemically controlled reaction.

### 3.2. *Effect of leaching temperatures on percent extraction of nickel*

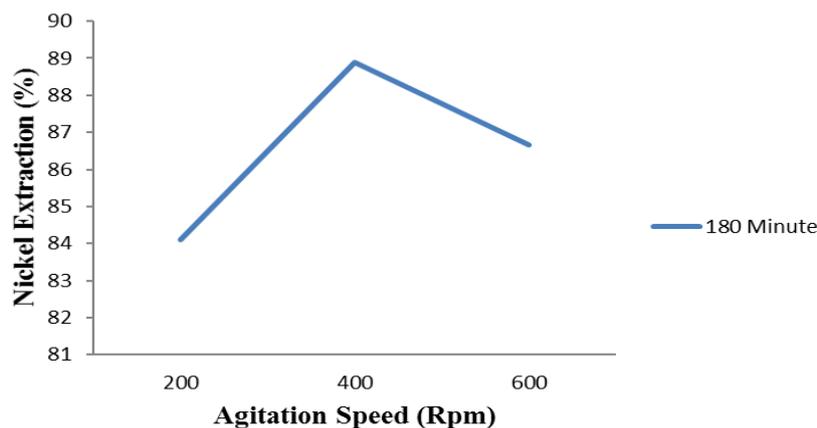
Figure 6 shows the effect of temperature on the percentage of nickel extraction. It could be seen from Based on Figure 6 it can be seen that the temperature significantly affects the percentage of nickel extraction, the higher the leaching temperature, the higher the percentage of nickel extraction. A maximum extraction percent value of 60.01% is achieved at 90 °C. Meanwhile, the percentage of extraction increased significantly from the temperature of 50 (16.91%) to 90 °C (60.01%). This is one of the indication that the leaching process of the limonite nickel ore of Pomalaa is chemically controlled reaction In addition, it supports the results of this research using the shrinking core model as described in point 3.1.



**Figure 6.** Effect of temperature leaching on nickel extraction

### 3.3. Effect of stirring rate on percent extraction of nickel

Figure 7 shows the effect of stirring rate on percentage of nickel extraction. The maximum percentage of extraction (88.90%) was obtained at a stirring rate of 400 rpm, while the minimum extraction percent was 84.10%. In addition, the percentage of nickel extraction did not change significantly with the increasing of stirring rate. According to Habashi on Kinetics of heterogeneous reactions, on increasing the rate of the rate of stirring in a solid - liquid reaction (leaching process), “an increase in the rate of dissolution sometimes takes place”. This is the case when the process is diffusion controlled because the thickness of the boundary layer decreases with increased speed of stirring. In addition, the rate of dissolution also increases. On the other hand, “a chemically controlled process independent of the speed of stirring” or is affected to a limited extent by a stirring rate [7]. It could be said that the leaching limonite nickel is controlled by chemical reaction.



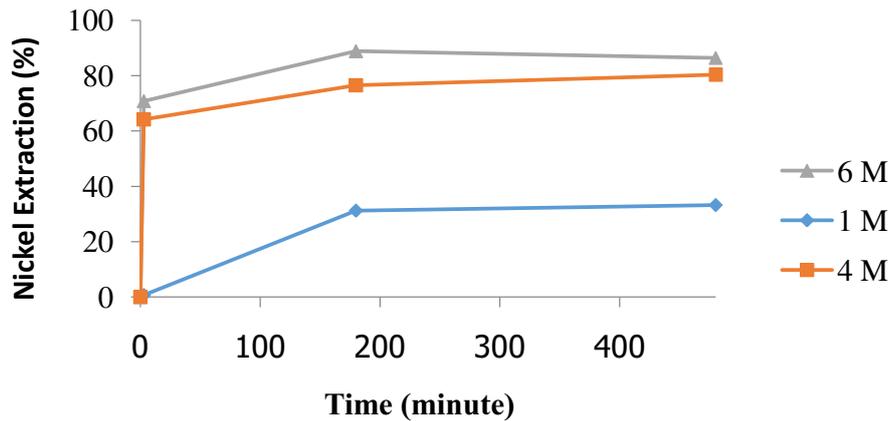
**Figure 7.** Effect of stirring rate (rpm) on nickel extraction

### 3.4. Effect of concentration of hydrochloric acid on percent extraction of nickel

Figure 8 shows the effect of concentration of hydrochloric acid on Nickel extraction. It could be said that the percentage of Nickel extraction is increasing with increasing HCl concentration. Maximum nickel extraction (86,49%) was obtained at HCl concentration 6 M, while minimum extraction percentage

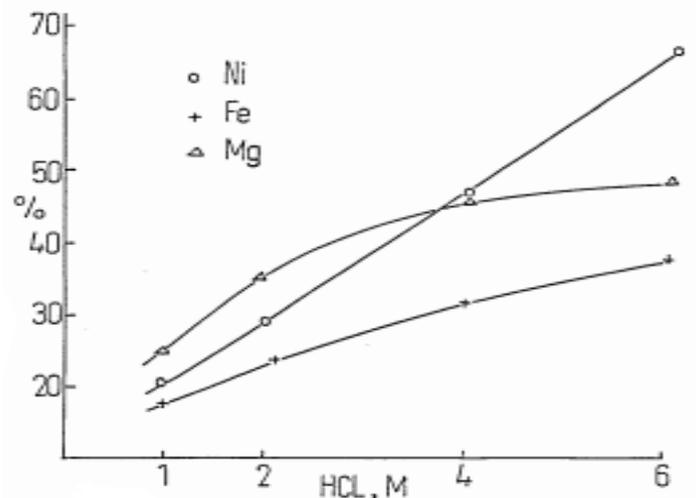
(31.22%) at 1M HCl concentration. The formula to calculate the percentage of nickel extraction is stated in the following equation:

$$\% \text{ extraction of nickel} = \frac{\left[ \frac{\text{ppm} \times \text{amount of dilution}}{1000} \right] \times [\text{volume of hydrochloric acid}]}{(\text{weight of limonite ore} \times \% \text{Ni on limonite ore})} \times 100\%$$



**Figure 8.** Effect of concentration of hydrochloric acid on nickel extraction

The increasing of percentage of nickel extraction due to the increasing of concentration of HCl is supported by the research conducted by N.M. Rice and L.W. Strong [8] which states that the increasing of hydrochloric acid concentration causes the increasing of percentage of nickel. Figure 9 shows the result of N.M. Rice and L.W. Strong [8] research.



**Figure 9.** Effect of acid strength on nickel, iron and magnesium extraction ore [8]

#### 4. Conclusion

Based on this research, it can be concluded that:

- According to the shrinking core kinetic model, it was found that the leaching process of nickel limonite ore from Pomalaa by using hydrochloric acid had an activation energy as much as 38.47 kcal/mole. It could be said that the leaching process was a chemically controlled reaction.
- The optimum percentage of nickel leaching process for limonite Pomalaa ore was 88.90%. This result was achieved under leaching process conditions such as 400 RPM of stirring rate, 90oC of leaching temperature and 180 minutes of leaching time.

### Acknowledgement

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