Effect of the first thermal stage temperature and retention time in two-stage thermal upgrading of low-grade nickel lateritic ore

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Abstract. Selective reduction process and magnetic separation of nickel lateritic ore were conducted through the two-stage thermal upgrading process with the addition of sodium sulphate and sodium chloride as the additives agent. Effect of the first thermal stage temperature has been investigated at various of temperature of 500, 600 and 700°C for 60 minutes and continued with the reduction process at temperature of 1050°C for 30 minutes. The mineralogical composition of the reduced samples was performed by XRD analysis. The optimum temperature was 500°C, which produced a concentrate with nickel grade of 2.98% and iron recovery of 40.17%. Effect of the first thermal stage retention time also has been investigated at temperature of 500°C for 30, 60, and 90 minutes, and continued with the reduction process at temperature of 1150°C for 60 minutes. The optimum temperature was 90 minutes, which produced a concentrate with 3.69% nickel grade, and 90.61% nickel recovery.

1. Introduction

Nickel is being a precious metal in infrastructure and technology field, especially in making industries of stainless steel, Ni-based alloy, alloy steels, electroplating, batteries, etc [1]. Nickel was used either in the form of metallic nickel or form of NPI/ferronickel with various of iron content [2]. More than 65% of the nickel consumption is used in stainless steel-making industry [11]. The extraction process of nickel lateritic process required high intensive energy and usually nickel is melted to produce low-grade ferronickel with the high amount of slag. Furthermore, this pyrometallurgy process merely is used for low-grade nickel lateritic ore containing nickel grade above 1.5%. Nevertheless, the grade of nickel lateritic ore around the world including Indonesia is about 1.45%, making this conventional process is ineffective [3]. Recently many researchers are focussing on the method for upgrading the effectiveness and efficiency of pyrometallurgy process to generate ferronickel by using selective reduction process followed by a magnetic separation process [4,5].

Thermal upgrading is a method to promote the value of nickel in the ore by applying physical beneficiation. This technique refers to the reduction process of nickel contained in limonite ore to
reduce the metallic and oxide producing ferronickel that can be separated from the gangue/non-metallic oxide by using magnetic separation process[6].

In this present work, the nickel oxide extraction of low-grade nickel ore from Desa Morombo, Konawe Utara District, Sulawesi Tenggara Province through selective reduction process followed by magnetic separation with the addition of the combination of sodium sulphate (Na$_2$SO$_4$) and sodium chloride (NaCl) as additive and palm kernel shell as reductant was investigated clearly.

2. Experimental

The raw materials used in this research are low-grade nickel limonitic ore, palm kernel shell as reductant, sodium sulphate and sodium chloride as the additives. The XRF analysis of nickel lateritic ore and proximate analysis of the reductant are listed in Table 1 and Table 2, respectively.

| Element | Ni | Fe | Si | Mg | Al | Ca | Cr | Mn | Co |
|---------|----|----|----|----|----|----|----|----|----|
| MassFraction (%) | 1.4 | 50.5 | 16.5 | 1.81 | 4.86 | 0.18 | 2.68 | 0.85 | 0.07 |

| Reductor Type | Volatile Matter | Ash | Fixed Carbon | Moisture |
|---------------|----------------|-----|--------------|---------|
| Palm ShellCharcoal | 22.57 | 21 | 77 | 0.43 |

The nickel ore was dried in an oven at temperature of 105°C for 24 hours to reduce the water content until less than 5 wt.%. The dried limonitic ore and the reductant were crushed and sieved to gain the particle less than 147 µm. Afterwards, 50 g of the ores, 5.5 g of reductants, and 5 g of each additive were mixed and pelletized. The pellets, which were 10-15 mm in size, were dried in an oven at the temperature of 100°C for 2 hours.

The two-stage thermal upgrading of selective reduction process was performed in this research. The first stage of reduction was done in various of temperature, which were 500°C, 600°C, and 700°C for 60 minutes and the second stage was 1050°C for 30 minutes. To investigate the effect of the first thermal stage retention time, another samples were added with the first stage of reduction temperature 500°C for 30 minutes, 60 minutes, and 90 minutes and the second stage was 1150°C for 60 minutes. After the selective reduction was done, the pellets were cooled rapidly in water. Then the pellets were dried and crushed to achieve particle size of 74 µm. The phase transformation was determined by XRD (X-ray Diffraction) analysis to observe the transformation of mineral composition and phase change after the selective reduction process. Afterwards, the wet magnetic separation process was conducted by dissolving the reduced ore into water and separating the concentrate and tailing product using 500 gauss magnet. The ratio of the reduced ore and water in this magnetic separation process was 1:10.

3. Results and discussions

3.1. Effect of the first thermal stage temperature
Figure 1. Effect of the first thermal stage temperature on: (a) grade of nickel and iron, (b) recovery of nickel and iron in concentrate at reduction temperature of 1050°C for 30 minutes.

From Figure 1, it shows that the highest nickel grade and recovery produced at the first stage temperature of 500°C with 2.98% and 73.20%, respectively. The decreasing nickel grade followed by increasing iron grade in the temperature interval of 500°C - 700°C was due to the selective reduction mechanism of trevorite. At the temperature of 500°C, trevorite had been reduced into nickel oxide and partially metallic nickel, but the iron oxide had not been reduced yet. It was because iron oxide would be reduced into metallic iron at 557°C. Hence, the nickel grade was relatively high with less iron grade contained. Higher temperature would result in higher metallic iron being produced from iron oxide and causing the increase in iron recovery. The higher the first thermal stage temperature resulted in decreasing of nickel recovery to 70.25% and increasing of iron recovery.

Figure 2 shows that the observed phase at the temperature of 500°C was trevorite, magnetite, silicate, and natrium chloride. Goethite could not be observed due to its free-crystal water released and transformed to hematite phase. The peak of lizardite, which less amount in ore, also could not be observed due to the hydroxylated process, even though it was not completely transformed yet. The transformation of magnetite was intensified at the temperature of 600°C due to the more hematite phase being reduced. Hematite phase could not be observed at the temperature of 700°C. It shows that hematite had been completely transformed into magnetite. Silicate also could not be observed due to its reaction with sodium sulphate as follows:

$$\text{Na}_2\text{SO}_4 + \text{SiO}_2 + \text{CO} \rightarrow \text{Na}_2\text{SiO}_3 + \text{SO}_2 + \text{CO}_2 \quad T \geq 670 \degree \text{C}$$  

(1)
3.2. Effect of the first stage retention time

The aim of the holding process at the first stage is to let the selective reduction process effectively applied to nickel which were contained in goethite phase in the ore[8]. Dehydroxylation mechanism plays a crucial role in achieving optimum nickel recovery. In the limonitic ores, nickel is substituted into the crystal structure of goethite[9]. Goethite is a crystal containing hexagonal closed-packed oxygen with iron occupying the octahedral interstices[10].

![Figure 3](image)

**Figure 3.** Effect of the first thermal stage retention time on: (a) grade of nickel and iron, (b) recovery of nickel and iron in concentrate at the first thermal stage temperature of 500°C and reduction temperature of 1150°C for 60 minutes

Longer retention time of the first thermal stage process will increase the reduction of hematite to trevorite and finally becoming nickel oxide. Figures 3 shows that longer retention time of the first thermal stage will increase the nickel grade in the concentrate. This was due to reduction reaction of nickel oxide having lower free energy value than the reduction reaction of iron oxide, thus it makes the reduction of nickel oxide will takes place before the reduction of iron oxide. Longer retention time will facilitates more complete reduction of nickel oxide but not allowing iron oxide to occur. It means that there will be more nickel reduced from goethite phase thus increasing nickel grade. And it also reduce the amount of iron oxide to be reduced and thus lowering the iron recovery. Another effect of the longer retention time is the increasing carbon monoxide as the residue from the reduction process of hematite. It will promote the transformation of some phases, such as sodium silicate and nickel sulfide, which was being prepared for the reaction at higher temperature. Nickel sulfide will be reacted with wustite to form nickel oxide and FeS. Nickel oxide is more capable to be reduced at higher temperature (≥ 700°C) due to more formation of gaseous CO. The longer retention time also affected to the addition of sodium chloride which will promote the reaction process between nickel oxide and gaseous hydrochloric acid to form nickel chloride. Nickel chloride, afterwards, will be segregated and forming metallic nickel. Figures 3 shows that longer retention time of the first thermal stage will increase the nickel grade in the concentrate. It was due to the more nickel that had been completely reduced from goethite phase. The optimum time was 90 minutes for the first thermal stage with nickel grade of 3.69% and nickel recovery of 90.61%.

4. Conclusions

The first thermal stage at the two-stage thermal upgrading process in selective reduction of nickel ore was focused on the reduction process of nickel form goethite phase and hinder the reduction process of iron. It affects the decreasing iron recovery and the increasing nickel grade. The longer retention time at the first thermal stage promoted the increasing grade and recovery of nickel due to the more reduction process of nickel occurred.
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