The influence of palm kernel shell mass ratio as a reducing agent in the lateritic nickel ore carbothermic reduction process

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Abstract. Indonesia has many valuable mineral resources, such as lateritic nickel ore. Today, the world demand of lateritic nickel continues to increase. This is an opportunity for Indonesia to develop its potentials in the nickel processing industry. To perform nickel reduction process, reducing agents such as natural gas and coal are needed. In this study, the use of a reductant from palm kernel shell waste as a coal alternative energy in order to reduce the use of fossil fuel which limited availability and cause environmental pollution, being a focus of this research. The purpose of this study is to determine the effect of palm kernel shell as a reductant in lateritic nickel reduction process, using mass ratio variable between mass of nickel ore and reductant. The mass ratio between nickel ore and reducing agent used in this study are 1:1, 1:2, 1:3, and 1:4, with the temperature of reduction in 800°C for 60 minutes. To observe the results of this experiment, the sample characterization was carried out using XRD and XRF. XRD data showed the presence of silica (SiO₂), iron oxide compounds such as maghemite (Fe₂O₃) and magnetite (Fe₃O₄), also compounds from reduction of lizardite such as forsterite (Mg₂SiO₄) and liebenbergite (Ni₂SiO₄). The results of XRF analysis showed improvement of Ni recovery in line with the addition of the mass of reducing agents.

1. Introduction

Today, Nickel is one of the most important elements that are widely used in many industries. Indonesia has a large potential of nickel laterite minerals. Those nickel content is estimated in number up to 1.6 billion tons with an average nickel content of 1.57% which spread around Indonesia such as Sulawesi, Maluku and Papua [1].

Lateritic nickel ore processing is one of the processes that require high energy and high cost production. It is because there are several numbers of processes that should be passed to extract the laterite ore into concentrates, such as rotary-kilns and high-pressure acid leaching (HPAL). In general, nickel laterite ore purification process is categorized into three processes, which are hydrometallurgy, pyrometallurgy and caron process [2,3]. The conventional pyrometallurgy process is still dominantly used in the processing of laterite ore in Indonesia since it is using easy technology and more economical production costs. In other way, conventional pyrometallurgical process usually used coal as a reducing agent which can cause air pollutions [4].
In order to consider environmental issue, the mining and industrial sectors associated with the metal processing are expected to reduce fossil fuel consumption to reduce non-renewable energy consumption and greenhouse gas emissions. Therefore, a process that can reduce the use of coal is needed. The use of biomass energy or waste from natural resources is one alternative that can be done to replace the role of coal as a reducing agent. Palm kernel shell is one of biomass that can be developed as a reducing agent in laterite nickel ore carbothermic process because it has high caloric content, low ash content and low emissions gas production, also the availability of palm plantations in Indonesia is increasing. It is widely expected to produce 7.3 million tons/year of palm oil production’s wastes per year. This study was conducted to determine the effect of palm kernel shell as a reducing agent in laterite nickel ore reduction process.

2. Materials and method

1.1. Materials
Several equipments were in this study including melting furnace, ball mill, ceramic crucible, digital scales, thermocouple, sieving machine, Spectro Xepos METEK XRF machine at BATAN and 7000 Maxima-X Shimadzu XRD machine at Department of Metallurgy and Materials Engineering UI Depok. While materials that were used are lateritic nickel ore from ANTAM field at Pomalaa and palm kernel shell from Kalimantan. First sample were prepared, the lateritic ore were crushed up to # 270 and the particle size of palm kernel shell is in range 1-2 cm.

1.2. Experiment
After sample preparation finished, nickel ore and palm kernel shell were homogenized and mixed into 4 different mass of reducing agent with ratio: 1:1; 1:2; 1:3; 1:4 (nickel ore : palm kernel shell). Then they were placed into ceramic crucible. Roasting process takes place in the muffle furnace at temperature 800°C for 60 minutes. After the reduction process, XRF and XRD test were conducted to analyze the compound and nickel contained in the lateritic ore.

3. Result and discussion

![Figure 1. XRD pattern of initial lateritic nickel ore.](image-url)
To identify the mineral composition of raw materials, proximate analysis were performed for palm kernel shell, while XRD and XRF examination were performed for raw lateritic nickel ore. As the results, figure 1 presents XRD pattern of nickel ore that shows the peaks of goethite, fayalite, lizardite, and quartz which appeared in raw ore.

While the proximate analysis of palm kernel shell as can be seen in table 1. Palm kernel shells are used as reducing agents because of the existing of fixed carbon and CH₄ content in volatile matter which can also use as reducing agents.

Figure 2 presents a comparison of XRD examination result from all of sample with different mass ratio. Semi-qualitative data of laterite ore samples obtained from the examination using X-Ray Diffraction (XRD). Characterization of the initial sample of nickel ore needs to be done in order to know the changes in the compounds form that occurs after the carbothermic reduction at constant temperature 800°C with reduction time in 1 hour. The effect of using different amounts of reducing agents becomes observed in this study.

Figure 2 indicates the different peaks of four samples which have different mass ratio. The variable of mass ratio would be affecting the form of compounds during the carbothermic reduction process determined by different peaks that occurred.

**Table 1.** Proximate and Ultimate Analysis of palm kernel shells.

| Parameter               | Content (% adb) |
|-------------------------|-----------------|
| Proximate Analysis      |                 |
| Moisture in air dried   | 3.70            |
| Ash                     | 2.09            |
| Volatile Matter         | 74.04           |
| Fixed Carbon            | 20.17           |
| Ultimate Analysis       |                 |
| Carbon                  | 49.90           |
| Hydrogen                | 6.00            |
| Nitrogen                | 0.28            |
| Oxygen                  | 41.66           |

**Figure 2.** XRD pattern of samples after reduction with different mass ratio (G=goethite, Q=quartz, Fy=fayalite, M=maghemite, L=lizardite, F=forsterite, W=wustite).
The change of compound was found differently in every sample with the mass addition of reducing agent from 1:1; 1:2; 1:3; and 1:4. At mass ratio 1:1, the phases of maghemite (Fe$_3$O$_4$), forsterite (Mg$_2$SiO$_4$), liebenbergite (Ni$_2$SiO$_4$), and quartz (SiO$_2$) compounds were shown. Furthermore, at mass ratio 1:2 the diffraction pattern shows the presence of diiron-silicate Fe$_2$(SiO$_4$) compounds. While at mass ratio 1:3 the presence of Magnetite (Fe$_3$O$_4$) compounds was appeared. The magnetite compound that appears in this variable is the result of further reduction of goethite and magnetite compounds. This transformation also occurred in the previous reduction research using other biomass [5,6]. The last variable in this study is the ratio of mass of nickel ore and palm kernel shell of 1:4. Changes occurred in the magnetite compound, where the compound was un-reidentified at results of XRD data above. The presence of new compound, wustite (FeO), at mass ratio 1:4 indicates the result of further reduction of magnetite compounds.

However, there were no peaks diffraction of NiO compounds on the result of sample characterization using XRD. The reduction of nickel in saprolite ore which is a silicate-bearing type mineral is more complicated when compare with limonite ore, this is related to olivine formation, where higher reduction conditions are require to reduce olivine [7].

In this study XRF examination to determine the quantitative data of oxides and chemical elements contained in the ores after reduction process were conducted. As the results of XRF examination, there are two dominant metal oxide compounds, namely nickel oxide and iron oxide, the XRF examination results of each variable can be seen in table 2.

| Sample           | NiO (%) | Fe$_3$O$_4$ (%) |
|------------------|---------|-----------------|
| Initial Sample   | 2.66    | 15.82           |
| Mass ratio 1:1   | 2.32    | 15.86           |
| Mass ratio 1:2   | 2.30    | 15.41           |
| Mass ratio 1:3   | 2.72    | 11.66           |
| Mass ratio 1:4   | 2.67    | 16.64           |

Figure 3. Comparison of % Recovery of samples after reduction with different mass ratio (laterite : palm kernel shell).
The purpose of XRF examination is to know the NiO and Fe$_2$O$_3$ content of carbothermic reduction result in each variable, so that we can know the most optimum reduction variable from the process. From the data above, it can be seen that the highest NiO content is obtained in the ratio of 1:3 and the lowest content is in sample 1:1. While for the Fe$_2$O$_3$, the highest content is obtained in the ratio 1:4 and the lowest content is in sample 1:3.

By calculating the sample weight and the element content after the reduction process compared with the weight and element content in the initial sample, data %recovery is obtained. The lowest value of nickel recovery was found in the sample 1:1 which is 66.20%, while the highest value of nickel recovery was obtained in the sample 1:4, with a recovery of 74.40%. The value of nickel recovery is projected into figure 3. It can be seen on the trend from the graph, there is an increasing number of recovery value along with the addition of mass of palm kernel shell as a reducing agent. The increase of the reduction degree obtained from the increasing number of reducing agents in this research is similar to the previous research results, both of which are coal [8] and sugarcane bagasse [9] as reducing agent.

4. Conclusion
a. XRD results showed that there is no Ni form appeared, this is due to the form of the mineral type in the form of silicate-bearing which binds the Ni element so that Ni were difficult to be reduced. But the reduction of hematite (Fe2O3) to wustite (FeO) was found. The wustite compound appears on the sample with variable 1:4.
b. The highest Ni % recovery was determined as 74.40% respectively with 80 grams palm kernel shell addition (sample 1:4) for 60 minutes reduction process.
c. The XRF results showed improvement of NiO and FeO in line with the addition of the mass of palm kernel shell. It was observed that in reduction process, the addition of palm kernel shell with various mass ratio does affecting metallization process of Fe and Ni in small amount.

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