

Study on the effects of temperature in the carbothermic reduction laterite ore using palm kernel shell as reducing agent

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Abstract. Indonesia has a rich deposit of nickel. However, laterite in Indonesia has not been treated to its full potential. This happens because the refining process of lateritic nickel ore has a high cost, triggered by the amount of energy required and the complexity of the separation process. It needs pre-reduction to make the ore more easily to be reduced and increased the metal content so that it can maximize the nickel refining process and minimize the energy usage. One method of pre-reduction is carbothermic reduction. This research study the effect of temperature variation on the results of the carbothermic reduction of laterite ores using palm kernel shells as the reducing agent. The reduction process was done by heating the # 270 mesh lateritic ore and palm kernel shells with a mass ratio of 1: 4 for 60 minutes in the melting furnace at the temperature variations of 700, 800, 900 and 1000°C. The result of the reduction was then tested using XRF and XRD. Based on the calculation of % recovery, the optimum temperature for reducing the laterite ore with palm kernel shells for 60 minutes was 800°C which the content of NiO as much as 2.68%.

1. Introduction

Indonesia has many laterite ores. Based on the Information from Energy and Mineral Resources in 2010, resources of nickel ore in Indonesia is as much as 263,350,043,000 tonne. But, Indonesia has not processed the nickel ore optimally because the process of purifying laterite is costly. It is triggered by the required energy and complexity in the process of the recovery process [1].

General nickel laterite purification process is categorized into three processes, namely hydrometallurgy, pyrometallurgy, and caron which is a combination of pyrometallurgy and hydrometallurgy [2]. The pyrometallurgical process is still dominantly used in the processing of laterite ore in Indonesia because of its easy-to-use technology and economical production cost. On the other hand, the pyrometallurgical process requires high fuel consumption and high reducing agent, while also produces air pollution [3].

Indonesia has abundant biomass potentials. Biomass is a waste of agricultural or forest residue and other organic materials that can be regulated into fuel and environmentally friendly reducing agents (low emission) through pyrolysis and gasification process.



Reducing gases from pyrolysis and gasification processes includes hydrogen (H_2), carbon monoxide (CO), carbon dioxide (CO_2), nitrogen (N_2), methane (CH_4), and other hydrocarbons is known as syngas. The relative proportion of each component in syngas depends on the operating conditions of gasification, i.e. biomass type, temperature, pressure, catalyst and others [4,5].

One of the natural potentials that can be utilized as reducing agent is palm kernel shell [6] because the reductor gases can be generated in the palm kernel shell gasification process. The application of biomass in metallurgy industry has been widely utilized, including direct reduction of iron ore by biomass char, sintering with wood chips, and production of pig iron by utilizing fine wood as reducing agent [7-9]. However, nickel processing industry in Indonesia currently has not used biomass as their alternative reducing agent. According to the previous study [10] which aimed to optimize the using of potential Indonesian ore as a raw material for producing iron nugget by utilizing palm kernel shell as a reducing agent, this study will analyze the reduction process of laterite ore by using palm kernel shell as a reducing agent.

2. Materials and method

2.1. Materials

The nickel ore sample used in this study was delivered from Halmahera, North Maluku, while palm kernel shell was delivered from Palangkaraya, Central Kalimantan. The experimental tools were ball mill, digital scale, thermocouple, ceramic crucible and sieving machine.

2.2. Experiment

This process took place in the muffle furnace. The laterite ore sample which has been formed to fine powder (# 270) and mixed with palm kernel shell was put into the melting furnace. The process of nickel ore reduction was done in 4 various increasing temperature range, respectively 700°C, 800°C, 900°C, and 1000°C for 1 hour with a mass ratio of 1: 4 (nickel ore: palm kernel shell). After the direct reduction process, XRF and XRD tests were performed to analyze the compounds and nickel content contained in the laterite ore. XRF testing was done at BATAN and XRD testing was done at Department of Metallurgy and Materials Engineering UI Depok. The machine used was XRD-7000 Maxima-X Shimadzu engine.

3. Result and discussion

The palm kernel shell used as a reducing agent had proximate and ultimate composition as seen in table 1.

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

Parameter	Content (% adb)
Proximate Analysis	
Moisture in air dried sample	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

Based on result of XRD (figure 1) and XRF analysis (table 2), initial nickel ore could be categorized as laterite-saprolite nickel ore.

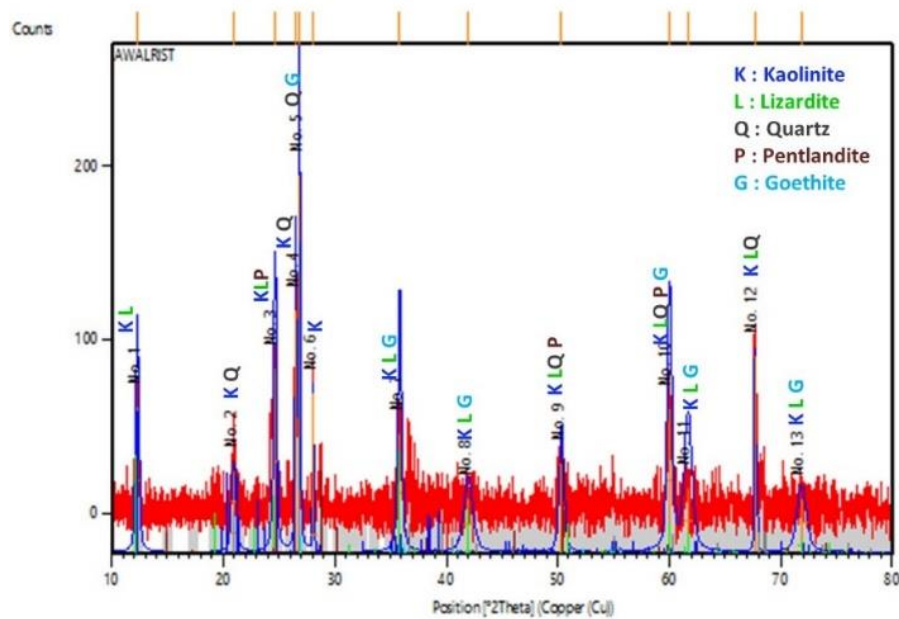


Figure 1. XRD pattern of initial lateritic nickel ore sample.

Table 2. XRF result of initial lateritic nickel ore sample.

Substance	Content (%)
Mg	14.28
Al	2.216
Si	20.12
Mn	0.196
Ni	2.092
Fe	11.06
MgO	23.68
Al ₂ O ₃	4.86
SiO ₂	43.05
MnO	0.253
NiO	2.662
Fe ₂ O ₃	14.93

The following graph represents the comparison of the sample XRD test results after carbothermic reduction at all temperatures in figure 2.

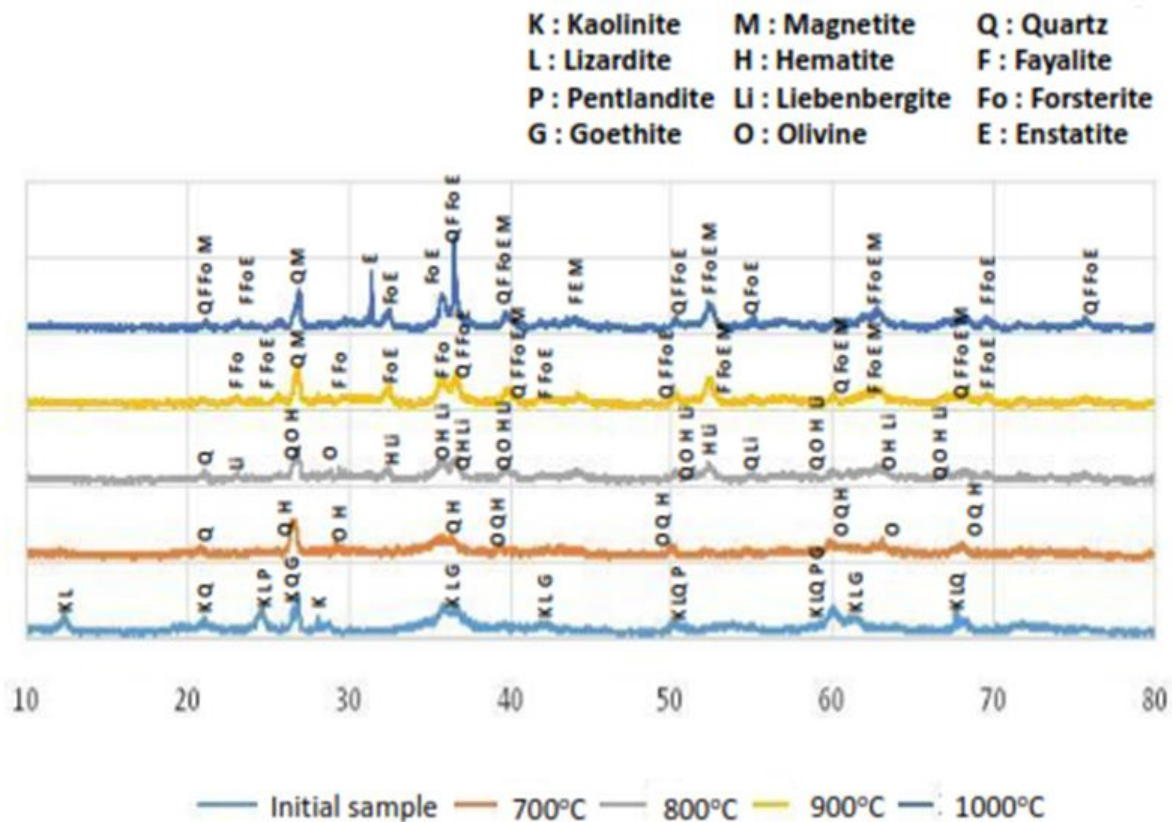


Figure 2. XRD patterns of carbothermic reduction from lateritic nickel ore at all reduction temperature variations.

From the data above, we assumed that during the carbothermic process, there was a change of compounds occurred in the temperature range of 700-1000°C. This could be observed among others on the removal and addition of diffraction peaks in samples heated above the temperature of 700°C.

At the temperature of 700°C, the formation of olivine (Mg_2SiO_4) and hematite (Fe_2O_3) compounds could be seen. At the temperature of 800°C, a new diffraction peak was found when compared to the diffraction pattern at 700°C. The diffraction pattern showed the presence of liebenbergite compounds (Ni_2SiO_4). At the temperature of 900°C, a new diffraction peak was assumed showing the presence of forsterite (Mg_2SiO_4) and enstatite (MgSiO_3) compounds, in relation to the addition of diffraction peaks which might indicate the presence of magnetite compounds (Fe_3O_4). At the temperature of 1000°C, there were two peaks that could be indicated as the presence of enstatite compounds, in addition to an increase in the intensity of the diffraction peaks at a position value of 2θ 36.5° significantly when compared to the diffraction peaks at 900°C, indicating an increase in the number of forsterite compounds at 1000°C.

This study also conducted XRF testing to determine the amount of oxides and chemical elements contained in the ores that had been reduced. In the results of XRF testing of the lateritic ore used in this study, there were 2 dominant metal oxide compounds: nickel oxide and iron oxide. The amount of each oxide in the lateritic ore could be seen in table 3.

From the XRF test result data in table 3, it was found that the amount of NiO compound increased as the reduction temperature increased. It showed that the highest% NiO was obtained at temperature 900°C. At the temperature of 700°C, there was a decrease in nickel oxide and iron oxide grades when compared to the initial sample. It was occurred as the result of the decomposition process accompanied by oxidation of sulfide ores of the pentlandite ($\text{Fe.Ni}_9\text{S}_8$) compound encountered in the initial sample. At the temperature of 800°C, NiO and Fe_2O_3 grades increased again and reached the

maximum value at the temperature of 900°C. It was related to serpentine decomposition process and nickel sulphide decomposition process into nickel oxide. At the temperature of 1000°C, NiO and Fe₂O₃ grades decreased. It happened due to the formation of forsterite and fayalite compounds. When Ni entered and was trapped in the olivine lattice, the nickel would be very difficult to reduce.

Table 3. XRF test results of lateritic nickel ore after carbothermic reduction for 60 minutes at all temperature

Sample	NiO (%)	Fe ₂ O ₃ (%)
Initial Sample	2.662	14.93
700°C	2.166	14.79
800°C	2.680	15.90
900°C	2.695	16.77
1000°C	2.428	15.85

Table 4. Recovery of laterite ore after carbothermic reduction for 60 minutes at all temperature variations.

Reduction temperature	Initial weight F (gr)	Ni ore. f (%)	End weight C (gr)	Ni Concentrate c (%)	Ni recovery
700°C	20	2.092	15.586	1.702	63.401
800°C	20	2.092	14.835	2.106	74.674
900°C	20	2.092	13.378	2.118	67.719
1000°C	20	2.092	12.900	1.908	58.826

The calculation of nickel recovery in concentrate based on the XRF testing before and after carbothermic process could be seen in Table 4. The highest recovery value was obtained at the temperature of 800°C. It concluded that between the temperature range of 700-1000°C, the temperature of 800°C was the optimum temperature to obtain nickel concentrate.

In this study, there were no diffraction peaks of NiO compounds in the result of sample characterization using XRD. The reduction of nickel in saprolite ore, which was a silicate-bearing type mineral, was more complicated when compared to the limonite ore. This was related to the olivine formation, where higher reduction conditions were required to reduce olivine [6].

4. Conclusion

The major conclusions drawn from the study were as follow:

- XRD results of lateritic ores reduced at 700°C, 800°C, 900°C and 1000°C showed that there was no NiO reduction process to Ni. This was due to the mineral type was in the form of silicate-bearing which binded the Ni element so that Ni was difficult to be reduced, but the reduction of hematite (Fe₂O₃) to magnetite (Fe₃O₄) happened. The magnetite compound was detected on the XRD diffraction pattern of laterite ore reduction at the temperature of 800°C.
- The highest % recovery value of lateritic lateral was obtained on laterite ore which was reduced to the temperature of 800°C, equal to 74.67%.
- Based on the % recovery, the optimal temperature for reducing laterite ore with oil palm shells was 800°C for 60 minutes resulting in 2.68% NiO content.

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