Reducibility of Low Nickel Lateritic ores with Presence of Calcium Sulfate

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Abstract— The thermal upgrading of processing of nickel lateritic ore to produce ferronickel is energy intensive, and need source of high grade coal as reductor especially when low nickel content ores are processed. The beneficiation via selective reduction of laterite ore to produce high nickel content concentrate and abandon gangue minerals before smelting potentially offers an effective pre-treatment to this process. In this work, the authors conducted selective reduction of nickel laterite ores at 900 °C for 60 minute with addition of 10% calcium sulfate and 6% reductant coal lignite type and carbonation coal. The presence of calcium sulfate will increase of recovery of nickel to 92.1%. According to the microscopic study, nickel oxide had been reduced and nickel was mainly enriched into a Fe–Ni phase. The presence of sulfur significantly promoted Fe–Ni particle growth, from 15 μm to 30 μm, and improved the Ni enrichment in metallic phases.

Index Term— Lateritic ore, reducibility, coal, microstructure, selective reduction

1. INTRODUCTION
Research on nickel recovery continues on each stage of the process nickel pyrometallurgy extractive standards in order to obtain a high recovery for a variety of nickel laterite ore characteristics, create a new process that allows applied to nickel levels that are not economically viable at this time the levels low nickel <1.8% (Dalvi, 2004). Traditionally, about 60% of the nickel product is produced from sulfide ores (Norgate and Jahanshahi 2010; Kim et al., 2010). However, miners of nickel sulfide ores are now confronted with increasing challenges due to much deeper drilling requirements, higher production costs and depleting reserves. Moreover, only 30% of the land nickel reserves now exist as sulfide ores with the balance comprised of nickel laterite ores.

Research studies of nickel extraction be categorized into several sections: the way pretreatment of raw materials, the physical conditions that affect the reduction process as bauxite pellets or briquettes shape, hardness, porosity, etc.; to control the growth of ferrous metals for the reduction is the addition of elements sulfur and chloride in various types both as elements and compounds; against type and composition of reducing agents that allow such gas CO, CO\textsubscript{2}, coal, methane, hydrogen gas; reduction of silica and magnesia compounds making up the total nickel content, and with the study of the mechanisms to understand the mechanism of the reduction reaction that occurs obtain the optimum reduction conditions.

The reduction in elevated levels of nickel laterite ore Jungah Kim et.al. (2010), has explained that the initial preparation of the initial ore by roasting at some temperature and followed by magnetic separation, may increase levels of nickel from 1.5% to 2.9% or about 48% so that the nickel laterite with low levels can further upgrading. Preparation for initial processing is also done by MU Wen-ning (2010) by reducing Si and Mg in the laterite ore so there is increased levels of nickel significantly from 1.41% Ni ore baseline to 2.96% at the end of the process.

Effect of roasting the early (pre-roasting) on levels of nickel studied by Li Jinhui et.al (2009). Lateritic nickel ore roasting between 300 up to 800°C. Characteristically changes in the ore minerals each temperature elevated analyzed and conducted leaching with dilute HCl. Based on the results of the analysis can be seen that the solubility of nickel in dilute HCl after roasting between 300 up to 500°C temperature rise that high, above 90%, but at a temperature of 600°C solubility of nickel to fall back, it is suspected the formation phase forsterite (Mg\textsubscript{2}SiO\textsubscript{4}) and enstatite (MgSiO\textsubscript{3}). Forsterite this will trap nickel that is inert to external influences so that the solubility will drop. This confirmed the results of previous Valix and Cheung (2002) explaining that nickel will go and dissolved in forsterite phase at this temperature. While at temperatures up to 800°C specific surface area decreased suspected some fine particles combine into larger so that smaller area will cause a slow dissolution rate. Iron solubility will be low as the temperature rises, it happens for reasons similar to the formation of nickel (Mg, Fe)\textsubscript{2}SiO\textsubscript{4} followed forsterite crystallization and trapping Fe into it, so that Fe solubility decreases, the optimum pre-roasting to obtain a soluble nickel in an acid at a temperature of roasting 300°C.

Other researchers who conduct roasting to enhance the acquisition of high nickel et.al. Guo Qiang (2011). Besides roasting with NaOH, roasting with Na\textsubscript{2}CO\textsubscript{3} on laterite ore has been carried out with the aim of improved its solubility of Ni and Co. Several type of reductor can be used to reduce the carbon nickel there are coal (Kawahara 1988), a combination...
of CO-CO\(_2\) (Purwanto, 2001), H\(_2\)-CO\(_2\) (Valix 2002), Combination of CH\(_4\)-methane (Sunati Mohanty 2008).

Carbon is the type most widely used reducing agent for the reduction of nickel ore because its abundance is very large. One of the most popular process is the production of ferronickel Krupp-Renn process. Nickel extraction process that occurs namely crushing ore by mixing with carbonaceous material which anthracite coal, coke, coal and limestone as a flux fuels then made briquettes. The next stage is reduced by hot gas flowed from the burning of coal.

In addition to coal, gas H\(_2\)-CO\(_2\), CH\(_4\) methane gas can be used to reduce the nickel from lateritic ores as practiced by Sunati Mohanty (2008). The survey results revealed that the maximum Ni can be reduced by CH\(_4\) at between 773 to 1073K.

In addition to using a carbon as reductor, nickel lateritic ore can be extracted by reduction using hydrogen gas. M. Kawahara (1988) reported that reducibility nickel from ore containing high iron has higher reducibility degree than the ore that contains low levels of iron.

Because the pre-reduction of nickel lateritic ore is also an energy intensive metal extraction process, several investigations have been performed to study the effect of different reluctant such as carbon or CO, on the reduction of lateritic ore. From an economic perspective, the continuing increase in the market price of steam coal, as well as its importance as a primary energy source in other fields, disadvantages carbon thermal reduction processes with high costs and relatively low energy efficiency. One option for improving the energy efficiency of ferronickel production is to utilize renewable energy sources, such as waste heat emitted from a smelter or from the carbonization of coal Kim et al. (2010).

Many researchers have carried out the reduction process with different types of reductor and various nickel ores. But only a few are using low grade nickel lateritic and microstructure investigate areas of reduction nickel lateritic which has the highest levels of metallization and growth the metal particle with selective reduction using any type coal.

This study has investigated observations degree of recovery nickel using additive sulfur substance in the form CaSO\(_4\) to increase the recovery and particle size. The type of reductor also investigate to know the effect two type source of coal on degree of recovery.

2. EXPERIMENTAL

2.1. Materials

Materials used in the study are the low grade lateritic nickel ore from Sulawesi Island and Calsium Sulfat anhydrous grade analytical from Merck Germany. Ore characterized using AAS (Atomic Absorption Spectrophotometer) GBC AAS listed in table 1. The main minerals contained in the sample that Antigorite with JCPDS No: 44-1447. Nickel content in the sample is considered low, with a ratio of SiO\(_2\)/MgO ratio is 1.84. The character can be said for the less well economically processed into a source of nickel metal. XRD peak intensity chart is dominated by mineral Antigorite serpentine types. Other minerals contained are listed in Table 2.

<table>
<thead>
<tr>
<th>Ni</th>
<th>Co</th>
<th>Fe</th>
<th>Mg</th>
<th>Cr</th>
<th>Mn</th>
<th>SiO(_2)</th>
<th>Al(_2)O(_3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.7</td>
<td>0.05</td>
<td>18.9</td>
<td>13.4</td>
<td>0.12</td>
<td>0.4</td>
<td>24.6</td>
<td>1.93</td>
</tr>
</tbody>
</table>

Sample then characterization using a Shimadzu XRD and analyzed with match 2 software in fig 1.
Fig. 1. XRD pattern of original ore analyzed with Match 2 Software

### Table II

<table>
<thead>
<tr>
<th>No</th>
<th>Mineral Name</th>
<th>JCPDS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Antigorite</td>
<td>44-1447</td>
</tr>
<tr>
<td>2</td>
<td>Enstatite</td>
<td>84-1925</td>
</tr>
<tr>
<td>3</td>
<td>Goethite</td>
<td>08-97</td>
</tr>
<tr>
<td>4</td>
<td>Montmorillonite</td>
<td>03-0009</td>
</tr>
<tr>
<td>5</td>
<td>Quartz</td>
<td>83-2473</td>
</tr>
<tr>
<td>6</td>
<td>halloysite</td>
<td>10-382</td>
</tr>
</tbody>
</table>

before the samples were made for the purposes of initial characterization and yield reduction, saprolite first heated for 2 hours at temperatures of 1000°C to remove water vapor.

![Fig. 2. Low grade saprolite (a) and after crushing <74μm (b)](image)

Based on the analysis by DTA on Fig 3, goethite dehydration starts at temperatures of 240°C and ends around 288°C and then transformed into haematite. Dehydration Antigorite at temperatures of 582°C to 623°C. High peak seen at temperatures of 812°C and ended at approximately 840°C. This is the recrystallization of Forsterite.

![Fig. 3. DTA curve of lateritic ore](image)

Others additive that are bentonite with the chemical composition as in Table III.

### Table III

<table>
<thead>
<tr>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>Na₂O</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>60.0</td>
<td>13.8</td>
<td>0.60</td>
<td>3.75</td>
<td>0.10</td>
<td>1.97</td>
<td>0.20</td>
</tr>
<tr>
<td>5</td>
<td>8</td>
<td>2</td>
<td>9</td>
<td>1</td>
<td>3</td>
<td>4</td>
</tr>
</tbody>
</table>
Basic on DTA curve at fig 4, endothermic peak of montmorillonite as the main mineral that occurs at temperatures 105°C. endothermic dehydration of the-OH group at 600-700°C but less clearly visible as well as recrystallization peak at temperatures of 900-1000°C.

Fig. 4. DTA curve of bentonite

2.1. Coal

Coal used comes from Kalimantan Indonesia by category lignite and sub-bituminous coal that has pretreatment in closed furnace 1 hour at 500°C called ‘carbonation coal’. Both coal is used to determine the effect of volatile matter on reducibility metals nickel and iron. The chemical composition of coal are listed in Table IV.

<table>
<thead>
<tr>
<th>Specification</th>
<th>VM %</th>
<th>Ash %</th>
<th>Fixed Carbon %</th>
<th>S %</th>
<th>Moisture %</th>
<th>CV cal/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lignite</td>
<td>45.6</td>
<td>17.2</td>
<td>24.25</td>
<td>1.9</td>
<td>10.5</td>
<td>451</td>
</tr>
<tr>
<td>Carbonation Coal</td>
<td>4.6</td>
<td>24.1</td>
<td>65.00</td>
<td>0.4</td>
<td>6.26</td>
<td>686</td>
</tr>
</tbody>
</table>

Fig. 5. DTA curve of lignite coal

Fig. 6. DTA curve of carbonation coal

2.2. Sample Preparation

Briquettes made by mixing powder size <74 μm (-200 mesh) of saprolite with the variation of coal weight. Saprolite and mixed coal crushed in a mortar to 30 minutes for sample homogenization. About 2 gr of the sample was then subjected were produce a briquettes by the mechanical compression machine at 200 MPa. The size of briquettes a formed was 10 mm in diameter and about 8-12 mm in height. In the reduction process, briquettes are placed in a tube furnace use nitrogen as carrier gas. Schematic of experiment reduction in tube furnace as fig 7. Figure 2 and Figure 3 as a briquettes after and before reduction.

Fig. 7. Schematic of experiment in tube
2.3 Procedure

Briquetted sample is heated in a tube furnace at temperatures of 873K to 1273K, respectively for 1 h with nitrogen gas as carrier. Briquettes results in a reduction on of partially cooled desicator for testing purposes SEM (Scanning Electron Microscopy) JEOL SEM in this case, for the XRD (X-ray diffractometer) (Shimadzu) and chemical composition of total Fe and Ni using AAS (Atomic Absorption Spectrophotometer) analytical Jena. Some of sample direct input into bromine methanol solution for metallization test. Bromine methanol solution used is mixing methanol with 5% (v / v) acetic acid and 5% (v / v) bromine.

3. RESULT AND DISCUSSION

3.1. Effect of reduction temperature and duration

A series of reduction tests were performed for various times to determine the effect of reduction time on nickel and iron concentration as shown in Fig. 8. The data shows that the nickel content was stable at times up to 60 min, but further increases in reduction time resulted in a decrease in the nickel content with a corresponding increase in iron content. The nickel recovery increased over time up to 60 min after which no significant improvements were observed. A reduction time of 60 min is recommended to obtain optimum nickel recovery of 91% to the final concentrate.

The laterite briquettes were reduced at temperatures between 700 °C and 1000 °C to determine the effect of temperature on nickel concentration. Fig. 9 shows the nickel and iron enrichment in the concentrate as a function of reduction temperature. The data shows that when the reduction temperature was increased from 700 °C to 900 °C, nickel recovery increased from 76% to 91% and decrease to 89% at 1000°C. Similarly, despite that the iron content gradually rose, there was a slightly decrease, in iron recovery at 1000°C. From a kinetic view point, the increasing temperature should improve the degree of metallization of both iron and nickel. However, the steady decrease in iron recovery suggests that kinetics were favorable for the formation of fayalite as opposed to further reduction of wustite to metallic iron in this range of temperatures. A reduction temperature of from 900 °C to 1000°C is recommended based on this data. Thus the nickel without CaSO₄ decrease due to the forsterite formation. Conversely existence of CaSO₄ made the nickel stable as metal.
The degree of reduction increased at 900°C temperature reduction but at a higher temperature will decrease significantly. It is possible without the addition of CaSO₄ formation occurs forsterite easier. CaSO₄ compound will undergo decomposition into CaO and SO₃, which although CaO has high decomposition temperatures > 1000°C but with the possibility of a reaction SiO₂:

$$2\text{CaSO}_4 + 2\text{SiO}_2 \rightarrow \text{S}_2 + \text{O}_2 + 2\text{CaSiO}_4$$

Sulfur formed will react with Ni to form nickel sulfide. Nickel sulfide is a compound that is not soluble in Forsterite and would surround the iron-nickel metal formed. So the possibility of merging some metal particles are formed into larger metal. Meanwhile, when not added CaSO₄ then NiO formed by heating and oxidation will be trapped in the lattice forsterite so would complicate subsequent reaction.

3.2. Effect of the type of reductant

A series of experiments were conducted at various type reductant to determine the impact on nickel concentration. Fig. 10 shows the nickel and iron concentration as a function type of reductant. The type reductant are lignite and carbonation coal with specification on table 4. There are two types of reducing agents is low quality coal is lignite that have a high volatile matter and ‘carbonation coal’ containing low volatile matter. Effect of the type of coal can be seen in the figure 10. Reducing conditions with additional additives as much as 10% CaSO₄ for each sample. The degree of reduction of nickel and iron higher using lignite reductant is likely this happens because lignite coal has a high volatile matter that still contains sufficient reducing gas reduction process running so fast that before forsterite formed more than 90% nickel is reduced. But it still appears that at higher temperatures will decrease the degree of reduction but nevertheless the degree of reduction in lignite and CaSO₄ conditions are always higher than those without both.

Lignite coal containing reducing gases and water vapor, with the possibility following reaction:

$$\text{CO} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{H}_2$$
$$\text{CH}_4 + 2\text{H}_2\text{O} \rightarrow \text{CO}_2 + 4\text{H}_2$$
However in some cases the use of coal has low calorie, and high volatile will cause it to overheat, causing a sudden reduction process and hot spots that will increase establishment phase of forsterite rapidly before nickel was reduced and this case is common in the reduction kiln which luppe as the main product. in this study controlled speed reduction of about 10 degrees per minute with no hope of going to the hotspot.

3.2. Peak analysis by XRD

Figure 11 shows the change of the crystal structures of saprolite reduction using coal at several temperatures. Tests carried out using a Shimadzu XRD-7000 XD. Of images can be seen that the diffraction peaks of F = FeNi appeared more dominant at 900°C reduction zone until 1000°C, while at temperatures about 1000°C the peak F = FeNi down. This occurs due to the formation of the peak Forsterite that is disturbing in a way to make the surface inert Fe and Ni so that hinder the process of further reduction of these metals. So on the surface of the samples contained more briquettes if the FeNi metal reduction process was conducted 1000°C under conditions of direct reduction using coal as reductor.

XRD analysis of the sample results with the addition of CaSO₄ reduction at various temperatures were measured with an image as shown in figure 11. Forsterite phase is the dominant phase of the start temperature reduction of 700 to 1000°C so forsterite has began to emerge in which the temperature of 700°C was the beginning of dehydroxilation the magnesium silicate hydrate mineral . FeNi metal already reveal a peak at 2θ ; 43.8 , 51.1 and 75.07 with peaks clearly visible . But the elements Ni sulfide is not visible because very low content so be detected by XRD . Then CaSiO₄ phase also formed ( JCPDS No. 76-186 ) which appears on the main peak 2θ ; 26.8 and 28.9 . This is most likely the occurrence of a reaction between CaO with SiO₂ to CaSO₄ formed .

\[
\text{CaO + SiO}_2 \rightarrow \text{CaSiO}_3
\]

With the calcium content in the ore is small then most likely CaSiO₃ formed come from CaO from calcium sulfate be added . At temperature of 1000°C, FeNi peak appear to have decreased while the peaks begin to form crystalline Forsterite is seen with increasingly sharp peaks produced . There is a possibility FeNi formed dissolved in Forsterite thereby decreasing subsequent reducibility.

Fig. 10. Effect of reductor type on recovery of nickel and iron

(CaSO₄ 10% )
3.3 Thermodynamic and microstructure analysis

Free sulfur element that vaporizes at a temperature of about 450°C, is expected to react with carbon to form CS₂ gas. It is based on two reasons,
- Direct reaction between S₂ with NiO based thermodynamic calculations are not possible because the standard free energy positive.
- When C first reacted with NiO, although it can take place thermodynamically, but kinetically the reaction between a solid and more difficult compare dense gas solid reaction.

Based on the above, it is most probably the first S₂ reacts with C to form Ni₂S and CO₂ gas. His reaction is:

\[
\text{C + S}_2 \rightarrow \text{Ni}_2\text{S} + \text{CO}_2 \quad \text{ΔG}^0_{550} = -5.253 \text{ kcal}
\]

\[
2\text{NiO} + \text{CS}_2 \rightarrow 2\text{NiS} + \text{CO}_2 \quad \text{ΔG}^0_{550} = -58.83 \text{ kcal}
\]

\[
2\text{NiO} + \text{C + S}_2 \rightarrow 2\text{NiS} + \text{CO}_2 \quad \text{ΔG}^0_{550} = -64.083 \text{ kcal}
\]

Because of the exothermic reaction, the higher the reaction temperature the formation of nickel sulfide is not getting better. Another possibility is the formation of Ni₃S₂ by the reaction:

\[
2\text{NiO} + 3/2\text{C + S}_2 \rightarrow \text{Ni}_3\text{S}_2 + 3/2\text{CO}_2 \quad \text{ΔG}^0_{550} = -70 \text{ kcal}
\]

This supervised reactions that may occur when selective reduction of nickel laterite ore carried on:

\[
\begin{align*}
\text{2CaSO}_4 + 2\text{SiO}_2 & \rightarrow \text{S}_2 + \text{O}_2 + 2\text{CaSiO}_4 \\
\text{2Fe}_3\text{O}_4 + 2\text{CO} & \rightarrow 6\text{FeO} + 2\text{CO}_2 \\
\text{Fe}_3\text{O}_4 + \text{S}_2 & \rightarrow 6/5\text{FeS} + 4/5\text{SO}_2 \\
4/3\text{FeO} + \text{S}_2 & \rightarrow 4/3\text{FeS} + 2/3\text{SO}_2 \\
2\text{FeO} + 2\text{CO} & \rightarrow 2\text{Fe} + 2\text{CO}_2 \\
2\text{NiO} + 2\text{CO} & \rightarrow 2\text{Fe} + 2\text{CO}_2 \\
2\text{Fe} + \text{S}_2 & \rightarrow 2\text{FeS} \\
2\text{Ni} + \text{S}_2 & \rightarrow 2\text{NiS} \\
2\text{Fe} + 2\text{NiS} & \rightarrow 2\text{FeS} + 2\text{Ni}
\end{align*}
\]

Based on surface mapping elemental analysis of the sample is known that there are areas where content elements Mg, Si and O higher while Al has a tendency separate elements. In areas with high levels of Mg, Si, O high mean there places where the formation of MgSiO₃ Forsterite Fe and Ni will get stuck into that phase so as to inhibit the reduction as it has been
brief by Valix (2002). Nevertheless, the results further mapping the levels of Fe and Ni is relatively evenly across the sample surface in contact with the gas either reductant. So Forsterite phase will hinder the reduction rate when forsterite phase is higher than the rate of reduction of Fe and Ni. When the Fe and Ni metal is first reduced to the formation of phase Forsterite not significantly inhibit the rate of reduction. It appears that disribusi Fe and Ni on the sample surface is uneven both in the area Forsterite and aluminum.

The preparation of the laterite ore briquette was as described in section 2.2. One briquette was put into a alumina crucible with graphite breeze reductant surrounding it to provide the reducing atmosphere. Nitrogen gas was metered into the tube furnace during the calcinations at a flow rate of 2 L/min. The sample was heated to 900 °C and the temperature increase from 500 to 900°C maintained for 10°C per minute. After reduction at 900 °C for 60 min, nitrogen gas quenching at a flow rate of 60 L/min was applied to cool the sample. For the microscopic analyses, reduced briquettes were set in an epoxy resin mount and polished. All the polished resin mounts were examined under an optical microscope equipped with a digital image acquiring system and connected to a personal computer. The image acquisition software use pseudo analysis to estimate metal phase on surface. These images were then used for SEM (Scanning Electron Microscopy) The samples were coated with a 25 nm–30 nm thick carbon film prior to SEM analysis. The SEM analyses were carried out in the Research Centre for Metallurgy, using a JEOL JSM-6390A with energy-dispersive spectrometers.

![image](image)

![Mg](Mg)

![Si](Si)

![O](O)

![Ni](Ni)

![S](S)

Fig. 11. Elemental mapping with SEM (condition of reduction 900°C, lignite 6%, CaSO₄ 10%, 1 hour)
Basic on figure 11 it can be seen that the nickel particles collected on one side to form a large enough group of about 30µm. While elemental sulfur were surrounding the metal particles indicates that around the FeNi metal particles are the most likely compound is FeS and NiS or Ni₂S₃ which serves as a collector of metal with a metal reaction mechanisms such as those set forth above where nickel metal reacts with sulfur to form nikelsulfide and will surround the nickel metal formed into a bigger metals particle because it does not dissolve in Forsterite. Figure 12 is an image of a microscope with pseudo mode to estimate the surface of the metal phase. Figure 12 is an image of the sample does not use CaSO₄ on it’s reductions, the size of the metal particles are formed about 15 micrometers. This is a result opposite to that sample containing CaSO₄ wherein the particle size to twice the magnification to 30 µm. Figure 13 shows the particle size of approximately 30 µm FeNi and dark colored compounds from particles surrounding sulfide compounds. From SEM image distribution of sulfur element is not appears in particle metal particles but they are distributed around the metal particles so it is clear that the element’s generated CaSO₄ has a positive impact on the recovery of nickel metal.
4. CONCLUSION

Selective reduction was conducted on low nickel content laterite ore samples. The results indicate that a high nickel recovery with 91% nickel recovery could be produced from reduction nickel ore with sulfur addition in form CaSO₄. Nickel was mainly concentrated into a FeNi phase in the reduced ore and growth of this phase was improved. The formation nickel sulfide appropriate increase the growth metals particle with inhibit nickel dissolved of silicate minerals as forsterite phase during selective reduction was beneficial to nickel enrichment. The size of metal particle increase two times from 15 µm to 30 µm. Other hand the type reductor which high containing reductor gas i.e. lignite have the great potential to increase the metal recovery thus lowers cost during nickel laterite ore reduction.

REFERENCES

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