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Optimal conditions for selective reduction process of Nickel Laterite Ore

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Abstract. A low-grade nickel laterite ore from Indonesia was selected to investigate the effect of temperature and time of reduction upon the extraction of nickel through the selective reduction-magnetic separation process to produce the ferronickel. The nickel ore was mixed with 5 wt.% of anthracite coal and 10 wt.% of sodium sulfate additive. The mixed ore was pelletized into 10-15 mm of diameter. A series of selective reduction processes were carried out into the pellet ores at various temperatures of 950-1150 °C for 60-120 minutes. The optimal selective reduction parameters of nickel laterite ore were observed clearly. The experimental results indicated that the reduction reactions proceed more completely at a higher temperature and longer reduction time. The optimum of nickel grade and recovery in the ferronickel concentrate was obtained at the reduction temperature of 1150 °C for 120 minutes, which was 8.45% and 72.1%, respectively. The increasing of temperatures and times of reduction process resulted in higher of troilite phase formation which suppressed the iron metallization and also increased the ferronickel particle size.

1. Introduction

Nickel is an important metal for stainless steel and alloy steel with extensive industrial applications, especially for modern infrastructure and technology [1]. Nickel is widely used in the form of pure metal or as a ferronickel alloy with varying iron content [2]. Due to the high demand for nickel in the future, there is an increasing interest in lateritic nickel ore as an alternative raw material to replace sulfide nickel ore in nickel production [3].

Nickel laterite ore accounts for up to 70% of total nickel resources in the world. Nevertheless, it was difficult for applying the physical beneficiation process in nickel laterite ore [4]. An effective process known as selective reduction-magnetic separation has been widely studied to produce a high-grade nickel concentrate from Indonesian low-grade nickel laterite ore [5]. Selective reduction of nickel laterite ore was accomplished by the addition of sodium sulfate then followed by the magnetic separation process to separate ferronickel from the gangue minerals [6]. It also has been studied the optimal condition of selective reduction process with variable temperature and reduction time.

The grade of metal in the concentrate increased with the higher reduction temperature due to the increasing degree of metallization [7]. The nickel grade also increased with the longer reduction time because many metallic phases are formed. In addition, the presence of sulfur-based additive increased

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the kinetic reduction rate and suppressed iron metallization by the formation of troilite (FeS) which increased the selectivity of nickel in magnetic separation process so the nickel grade and recovery were high [8]. Most of the reduction process used bituminous coal as reductant due to its high sulfur content which could improve the formation of troilite [3,9-10]. Nevertheless, the bituminous coal has moderate fix carbon.

In this present work, the reduction process of low-grade nickel ore was carried out by using anthracite containing high sulfur as reductant, which had higher fix carbon than bituminous coal. The effect of the temperature and time of reduction on nickel grade and recovery in ferronickel concentrate was investigated clearly. The size of the ferronickel particle was also observed in this selective reduction process.

2. Experimental

The ore used in this study was from Sulawesi, Indonesia. Table 1 provides the ore composition as determined by X-ray Fluorescence (XRF) analysis. Anthracite coal from Padang, Indonesia was used as the reductant, the composition of the coal used is given in Table 2. The sodium sulfate was used as additives in this selective reduction process.

Table 1. The chemical composition of the laterite ore samples (mass fraction, %)

Element	Ni	Fe	Si	Mg	Al	Ca	Cr	Mn	Co
wt. %	1.4	50.5	16.5	1.81	4.86	0.177	2.68	0.847	0.0662

Table 2. Proximate analysis of anthracite coal (wt.%)

Reductant	Volatile Matter	Ash	Fixed Carbon	Moisture
Anthracite coal	18.25	18.25	60.35	3.14

Both the ore and the reductant were ground to less than 149 µm. The 100 grams of limonitic ore, 5 wt.% of reductant and 10 wt.% of Na_2SO_4 were manually mixed and made into pellets with an approximate diameter of 10-15 mm. The pellets were dried in an oven at 100 °C for 4 h to remove the water, then it placed in a graphite crucible and inserted into a muffle furnace. The reduction temperature was varied in the range of 950-1150 °C and the reduction time in the range of 60-120 minutes.

The reduced pellets were removed from the furnace and quenched into the water, then dried in an oven at 100 °C for 4 h and ground to less than 74 µm in a shaker mill. The reduced pellets were separated in magnetic separation at a magnetic field intensity of 500 gauss magnet to obtain the magnetic product (ferronickel concentrate). The phase transformation of nickel laterite ore in the reduction process was investigated using XRD. The XRF was performed to investigate the nickel and iron grade and recovery in the ferronickel concentrate, while SEM equipped with an EDS was used to observe the phase and its morphological changes after the magnetic separation.

3. Results and Discussion

3.1. The effect of reduction temperature

The grade of metal in the concentrate increased with the higher reduction temperature. This is due to the increasing nickel and iron metallization so metal oxides are reduced to the metal [7]. According to the Fig. 1, the highest nickel grade was obtained at a temperature of 1150 °C where it reached 6.1% with a recovery of 70.3%. The nickel grade increased with the increasing of reduction temperature [8].

Otherwise, the nickel and iron recovery decreased at the high temperature. The nickel recovery was 70.3% during the reduction process at 1150 °C, while the iron recovery was continued to decrease to 17.98%. The decreased of iron recovery was tend to be more significant due to the formation of troilite

(FeS) phases. The formation of non-magnetic troilite phases suppressed the iron metallization which contributes to the increase in the nickel grade in concentrate [2].
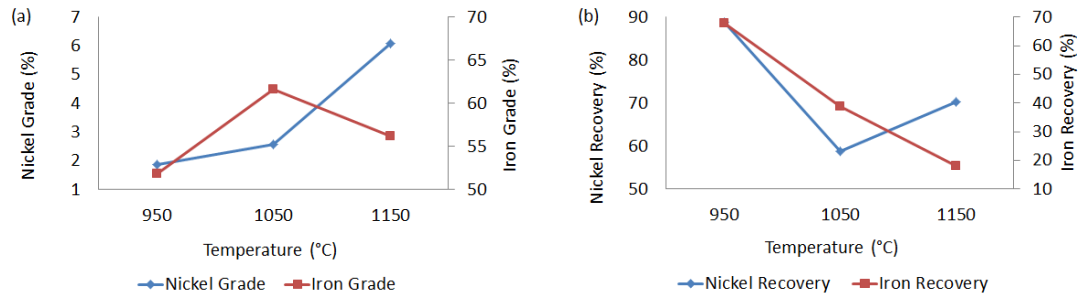
Figure 1. Effect of reduction temperature with the addition of 10 wt.%sodium sulfate on (a) nickel and iron grades and (b) nickel and iron recoveries

($\blacktriangledown$ = FeS, $\blacktriangle$ = Fe$_2$SiO$_4$, $\blacksquare$ = FeNi, $\bullet$ = FeO, $\blacklozenge$ = SiO$_2$)
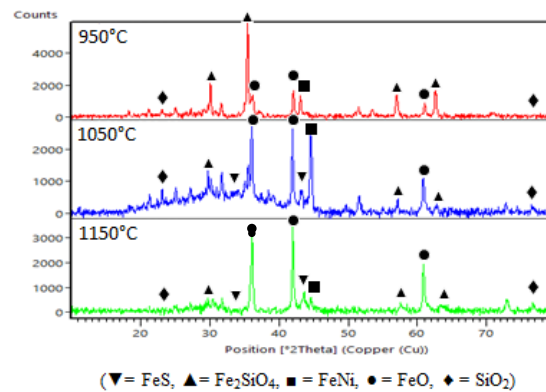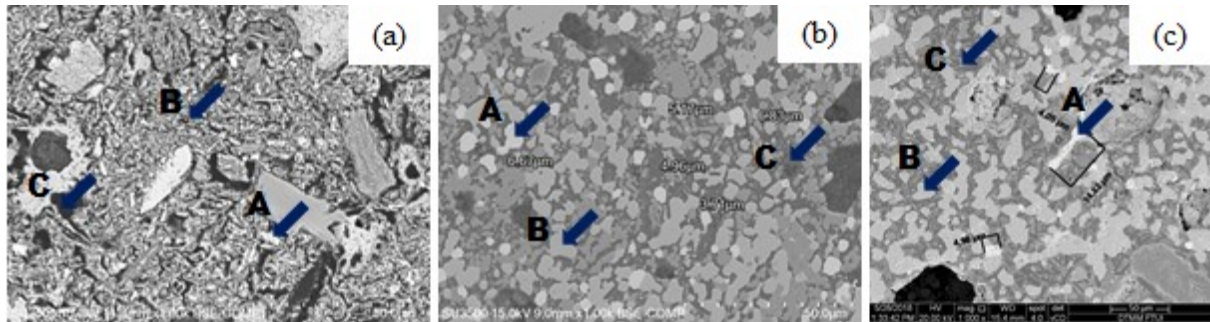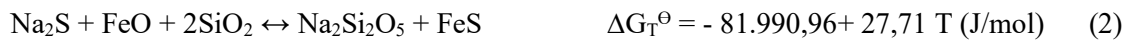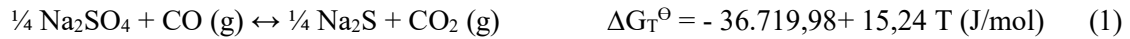
Figure 2. XRD pattern of reduced laterite pellets at various temperature for 60 minutes with the addition 10 wt.% of sodium sulfate.

Table 3. The rietveld refinement calculation of reduced laterite pellets at various temperature for 60 minutes with the addition 10 wt.% ofsodium sulfate.

Phase (wt. %)		FeNi	Fe$_2$SiO$_4$	FeO	FeS	SiO$_2$
Temperature (°C)	950	7.9	44.4	37.3	2	8.4
	1050	6.2	23.2	45.4	16.2	9
	1150	3.6	22	40.6	24.4	9.3

The mineralogical composition of the reduced ore with the addition of Na$_2$SO4 was wustite (FeO), troilite (FeS), fayalite (Fe$_2$SiO$_4$), quartz (SiO$_2$), and ferronickel (FeNi), is shown in Fig. 2. The peak and composition of wustite increased to a temperature of 1050°C where the reduction of hematite to wustite still occurs, then at a temperature of 1150 °C the peak decreased as some wustite has been reduced to iron and partly reacts with Na$_2$S to form FeS, as expressed in Eq. (2). The peak of FeS was found at a temperature of 1050 °C and the composition increased until the temperature of 1150 °C. The higher reduction temperature, the more FeS were formed, that suppressed iron metallization so the nickel grade was increased [2,12]. From Table 3, the amount of ferronickel was decreased during the increasing of the reduction temperature due to the transformation of some iron oxide into troilite, thus the small amount of ferronickel that resulted in higher temperature had a more nickel content in concentrate.

$$\tfrac{1}{4}\,Na_2SO_4 + CO\,(g) \leftrightarrow \tfrac{1}{4}\,Na_2S + CO_2\,(g) \qquad \Delta G_T^{\Theta} = -36.719,98 + 15,24\,T\ (J/mol) \qquad (1)$$

$$Na_2S + FeO + 2SiO_2 \leftrightarrow Na_2Si_2O_5 + FeS \qquad \Delta G_T^{\Theta} = -81.990,96 + 27,71\,T\ (J/mol) \qquad (2)$$

Figure 3. SEM picture of the sample at reduction timeof 60 minutes and the addition 10 wt.% of sodium sulphatewith various reduction temperature of (a) 950, (b) 1050, and (c) 1150°C

The point A, B, and C indicated the ferronickel (FeNi), troilite (FeS), and impurities dominated by Si and O elements, respectively. At a temperature of 950 °C, no measurements of ferronickel grains due to the very small size.The ferronickel particle was growth into 6.83 µm and 14.82 µm at a temperature of 1050 °C and 1150 °C, respectively. During the reduction process, there was a liquid phase caused by the Fe-FeS phase which decreased the eutectic point at 985 °C that caused the agglomeration of ferronickel. At the higher temperature, it accelerated the growth of ferronickel grains [11].

3.2. The effect of reduction time
The metallic phase increasing as the reduction time increased [12]. The more of metal oxide was reduced due to the longer contact time with the gas [13]. At 120 minutes, the nickel grade was 8.45% with the recovery of 72.09%, while the iron recovery was very low. This may occur due to the re-oxidation of the metallic iron when the reduction time was taking too long [12].
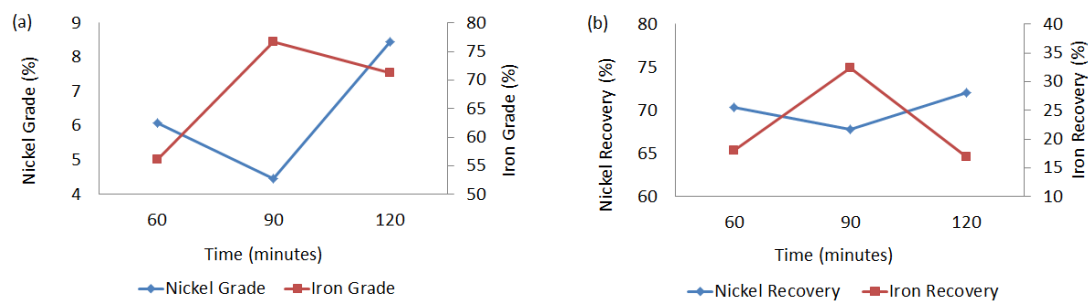
Figure 4. Effect of reduction time with the addition of 10 wt.%sodium sulfate on (a) nickel and iron grades and (b) nickel and iron recoveries

($\blacktriangledown$= FeS, $\blacktriangle$= Fe$_2$SiO$_4$, $\blacksquare$ = FeNi, $\bullet$ = FeO, $\blacklozenge$ = SiO$_2$)
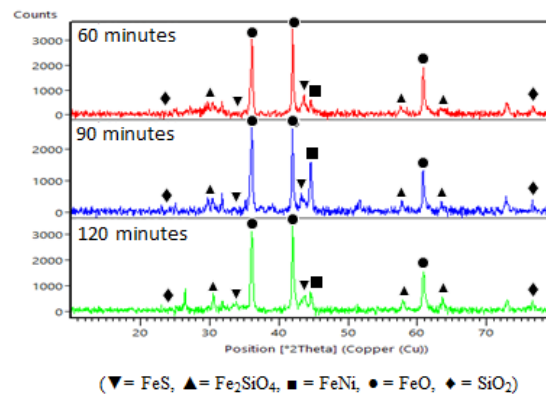
Figure 5.XRD patterns and the rietveld refinement calculation of reduced pellets with various reduction time at the temperature of 1150 ° C and the addition 10 wt.% of sodium sulfate.

Table 4. The rietveld refinement calculation of reduced laterite pellets at 1150 °C in various time with the addition 10 wt.% of sodium sulfate.

Phase (wt. %)		FeNi	Fe$_2$SiO$_4$	FeO	FeS	SiO$_2$
Time (minutes)	60	3.6	22	40.6	24.4	9.3
	90	11.5	30.2	30.1	13.8	14.4
	120	7.1	27	35.6	20.6	9.7

From Fig. 5 and Table 4, the wustite decreased after 60-90 minutes of reduction time due to the reduction of wustite to metallic iron. It is also indicated by the high peak and composition of ferronickelat 90 minutes. However, the wustite increased again after the reduction process at 120 minutes. At a very long reduction time, the reductant would run out and the metallic iron will re-oxidize to iron oxide [14]. It is indicated by the high peak of wustite and followed by the low peak of ferronickel after the reduction process at 120 minutes.
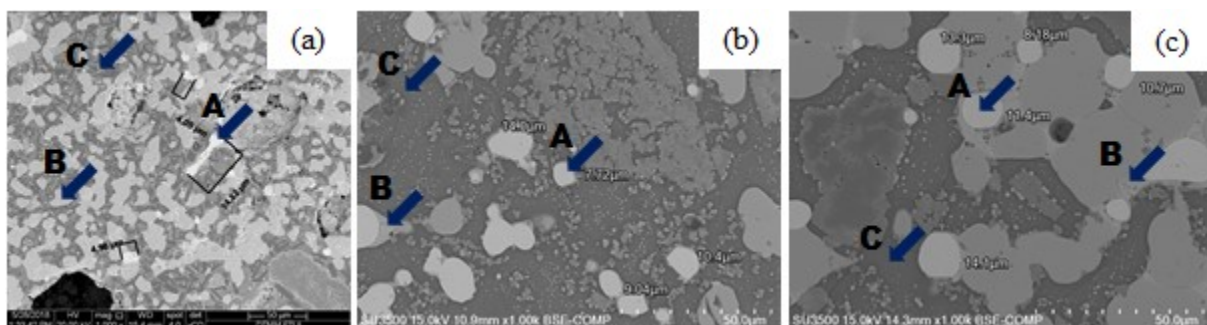
Figure 6. SEM picture of the sample at the temperature of 1150°C and the addition 10 wt.% of Na$_2$SO$_4$with various reduction time of (a) 60 minutes, (b) 90 minutes, and (c) 120 minutes.

The point A, B, and C indicated the ferronickel (FeNi), troilite (FeS), and impurities dominated by Si and O elements. The size of ferronickel grains of sample 60, 90 and 120 minutes were14.82μm, 15.39μm, and 21.12 μm, respectively. Increasing the reduction time caused the reaction occurred more considerable and led to high metallic phase formation [12].

4. Conclusions

An increase in the reduction temperature led to high nickel grade in ferronickel concentrate. The reduction process at a temperature of 1150 °C resulted in the nickel grade of 6.1% with a recovery of 70.3%. The Na$_2$SO$_4$ presence affected the greater formation of troilite which suppressed the iron metallization then resulting in high nickel grade. The reduction of nickel ore until 120 minutes

resulting in the highest nickel grade of 8.45%. The long reduction time caused the re-oxidation of metallic iron into iron oxide thus promoting the nickel grade in concentrate. In addition, the high temperature and the long reduction time promoted the growth of ferronickel particle due to the formation of troilite which increased the mass transfer of metals resulting in the agglomeration of ferronickel grains.

5. Acknowledgment

6. References

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