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The effect of sulfur, temperature, the duration of the process and reductant on the selective reduction of limonite ore

Introduction

Approximately 70% of the world reserves are lateritic nickel ore, and 30% are sulfidic ore. Increased demand for nickel and the rapid depletion of sulfidic ore have forced the industry to consider lateritic rocks as one of the alternative sources of nickel in the future.

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Recently, more nickel laterite processing in Indonesia has been carried out in high-grade ore using pyrometallurgy, which generates ferronickel and nickel matte (Dalvi et al. 2004).

Lateritic nickel ore is used in the manufacture of ferronickel. The nickel content in ferronickel ranges from 20–40%. Ferronickel is commonly used for producing stainless steel (Prasetyo and Puguh 2011). However, using the high-temperature process in ferronickel production, i.e. 1400°C, generates high energy consumption and can degrade the lining furnace, resulting in high-production costs (Cao et al. 2010). A new method to increase the nickel grade in lateritic nickel ore is selective reduction at temperatures lower than 1300°C. The objective of selective reduction in nickel laterite is to reduce the metal oxide content of the metal using a reducing agent at a specific temperature. It also adds additives to increase the selective reduction of iron. The selective reduction has been carried out at a temperature of 800–1200°C to obtain ferronickel concentrate after the process of removing impurities using magnetic separation (Elliot et al. 2017). The increase of the iron and nickel grade in concentrates can be generated by increasing the amount of reductant and the temperature and time reduction (Prasetyo and Firdiyono 2014). The addition of a sulfur-based additive leads to iron recovery being low but increases the nickel grade in the concentrate due to the formation of nonmagnetic iron sulfide (Cao et al. 2010). In this work, the selective reduction of low-grade nickel laterite used elemental sulfur as an additive with anthracite coal as a reductant.

1. Experimental method

Laterite ore comes from Konawe, Southeast Sulawesi, Indonesia. Atomic absorption spectroscopy (AAS) was conducted to determine the chemical composition of the nickel

Table 1. The chemical composition of limonitic nickel ore

Tabela 1. Skład chemiczny rudy limonitowej niklu

Element	Ni	Fe	Si	Mg	Al	Ca	Cr	Mn	Co
Amount (wt%)	1.4	50.5	16.5	1.81	4.86	0.177	2,68	0.847	.0662

Table 2. Rietveld refinement analysis of limonitic nickel ore

Tabela 2. Udoskonalona analiza Rietvelda rudy limonitowej niklu

Name	Chemical structure	Total (%)
Goethite	FeHO ₂	49.7
Olivine	MgNiO ₄ Si	37.5
Lizardite	H ₄ Mg ₃ O ₉ Si ₂	7.6
Quartz	SiO ₂	5.3

laterite, as listed in Table 1. The elemental analysis is 1.4 wt% of nickel and 50.5 wt% of iron, which indicates that the nickel laterite type is limonitic ore. Additionally, the impurities contained in the nickel limonite are 16.5 wt% Si, 1.81 wt% Mg, 0.177 wt% Ca, 4.86 wt% Al, 2.68 wt% Cr, 0.847 wt% Mn, and 0.0662 wt% Co.

The XRD results of limonitic nickel ore are shown in Figure 1. Limonite is dominated by goethite (FeO.OH), olivine (MgNiSiO₄), lizardite (Mg₃(Si₂O₅)(OH)₄) and quartz (SiO₂). Nickel is associated with oxide and iron in the goethite structure, which shows the highest intensity. In this study, coal as a reductant is anthracite and its additions were 5%, 10%, and 15%. The anthracite was analyzed proximate to determine the moisture, volatile, ash and fixed carbon contents, as shown in Table 3. The fixed carbon was used to determine the amount of coal used as a reductant in the reduction process of limonitic nickel ore.

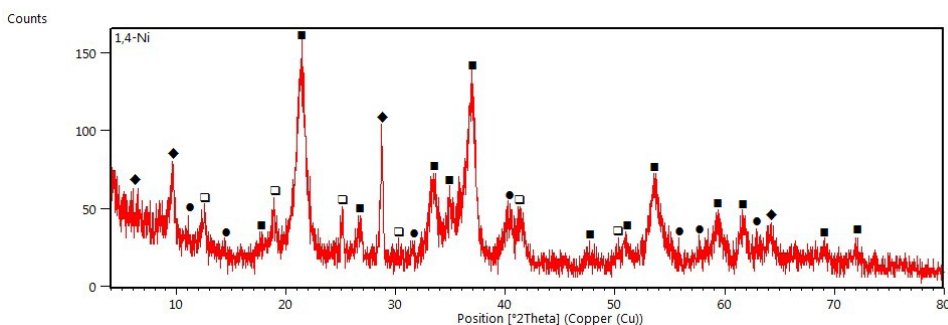


Fig. 1. XRD of limonitic nickel ore
 symbol: ■ = goethite, ● = olivine, □ = lizardite, and ◆ = quartz

Rys. 1. Dyfrakcja rentgenograficzna XRD rudy limonitowej niklu
 symbol: ■ = getyt, ● = oliwin, □ = lizardyt i ◆ = kwarc

Table 3. Results of the approximate analysis of anthracite

Tabela 3. Wyniki przybliżonej analizy antracytu

Element	Content (wt%)
Moisture	3.14
Volatile	18.25
Ash	18.25
Fixed carbon	60.35

In the first step, limonitic nickel ore, anthracite, and elemental sulfur were mixed and then homogenized and manually pelletized. The pellets were dried in an oven at a temperature of 120°C for 4 hours. Then, the pellet was heated in the furnace with air pressure of 1 atm to perform a reduction process at temperature variations of 950°C, 1050°C, and

1150°C with duration time variations of 60, 90, and 120 minutes. After the reduction process, the pellets were rapidly cooled in water. The dried pellets were crushed using a shaker mill to obtain a size of less than 200 mesh. Then, it was separated through the application of a wet magnetic separation process using a permanent magnet with a 500 Gauss magnetic field to obtain concentrate and tailing. AAS Shimadzu A-6800 analyzed the concentrates and tailings in the aqueous form. The SEM-EDS EVO® MA 10 was performed to investigate the microstructure of the reduced pellet. The XRD PANalytical X'Pert3 Powder (in the 200 mesh sample size, the two thetas are in the range 10–80° with a step size of 0.05 and analysis data by High Score Plus) was used to investigate the phase transformation of the reduction process of nickel laterite.

2. Result and discussion

2.1. The effect of temperature reduction on metal grade and recovery in concentrate

The fixed parameter is 5 wt% anthracite, 10 wt% sulfur and the duration time was 60 minutes at variable temperatures of 950°C, 1050°C and 1150°C. According to the AAS data in Figure 2, the increase of reduction temperature could decrease the nickel grade. This is due to the more CO gas produced in the reduction process and reacting with metal oxides in the ore by increasing the reduction temperature (Prasetyo and Firdiyono 2014). The recovery of iron and nickel also increases with the increasing temperature reduction, which agrees with Li et al. (Li et al. 2012). Sulfur as an additive could react with iron to form

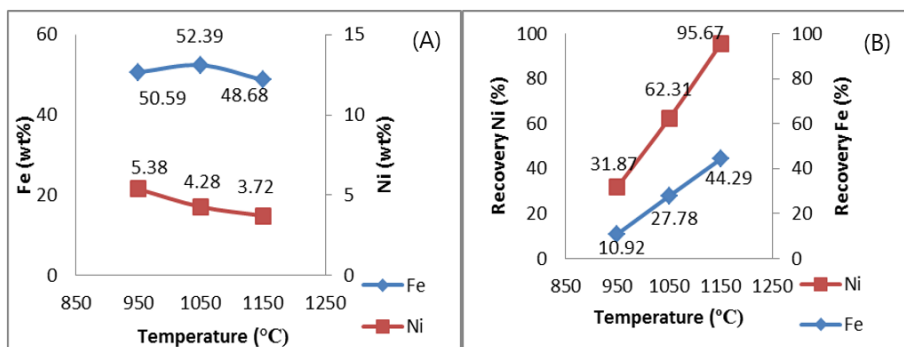


Fig. 2. The effect of temperature reduction on a – the content of nickel and iron and, b – the recovery of nickel and iron

Rys. 2. Wpływ obniżenia temperatury na: a – zawartość niklu i żelaza, b – odzysk niklu i żelaza

iron-sulfur (FeS), lowering the iron recovery (Jiang et al. 2013). However, the high-temperature reduction enhances the reduction rate of metallic oxide, thus increasing the iron and nickel recovery. Increasing iron recovery suppresses the nickel grade in the concentrate, as illustrated in Figure 2(a). The optimal temperature was found at 1150°C due to there being the highest nickel recovery in the concentrate.

The XRD analysis was performed on samples to observe the chemical compounds formed after the reduction process at various temperatures (950°C, 1050°C, and 1150°C) at a duration time of 60 minutes. Based on Figure 3, the phases are fayalite (Fe_2SiO_4), quartz (SiO_2), magnesioferrite (Fe_2MgO_4), wustite (FeO), iron-nickel (FeNi) and pyrrhotite (FeS), which were found in the XRD results. At 950°C, pyrrhotite (FeS) is not found, while at 1050°C and 1150°C, pyrrhotite appears. Mostly, iron-sulfur reacted at 950–1050°C which could inhibit the metallization of iron (Li et al. 2012). At 950°C, the iron-nickel (FeNi) phase begins to form, but the intensity is low according to the Rietveld calculation (Table 4). This is due to the iron and nickel being entrapped in the impurities compound. As the reduction temperature increased, the ferronickel content increased, as shown in Table 4. Therefore, it could enhance metal recovery (Valix and Cheung 2002). The wustite phase is only found at a temperature of 950°C, while at temperatures of 1050°C and 1150°C, there is no wustite phase, which means that more of wustite was transformed into metallic iron during the increase of the reduction temperature.

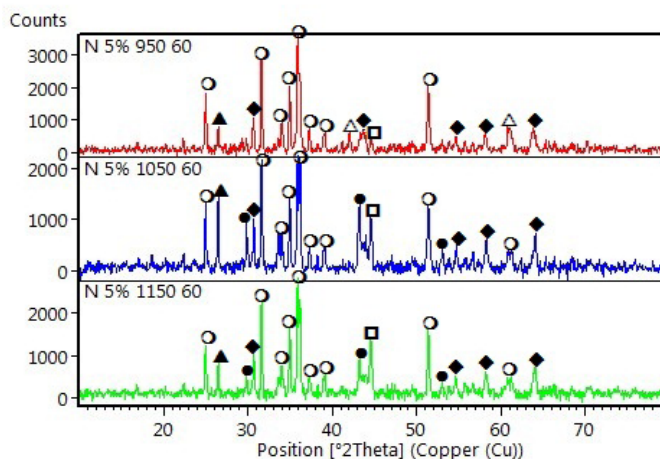


Fig. 3. XRD pattern of the effect of temperature of
 a – 950°C (red line), b – 1050°C (blue line), c – 1150°C (green line)
 and the changing phase with the duration time for 60 minutes
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi, ● = FeS

Rys. 3. Wzór XRD wpływu temperatury
 a – 950°C (linia czerwona), b – 1050°C (linia niebieska), c – 1150°C (linia zielona)
 oraz zmieniającej się fazy wraz z czasem trwania przez 60 minut
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi, ● = FeS

Table 4. Rietveld refinement of calculation of the reduction with the additive compounds 10% sulfur and variation of temperature.

Tabela 4. Udoskonalenie Rietvelda dla obliczenia redukcji z dodatkami 10% siarki i zmiany temperatury

Compound	The percentage of reduction products (%)		
	950°C	1050°C	1150°C
Fayalite (Fe ₂ SiO ₄)	62.4	47.3	52.7
Quartz (SiO ₂)	3.5	9.0	3.2
Magnesioferrite (Fe ₂ MgO ₄)	28.4	24.0	25.8
Iron oxide (FeO)	4.9	–	–
Iron-nickel (FeNi)	0.9	3.2	7.2
Pyrrhotite (FeS)	–	16.4	11.1

2.2. The effect of process duration time reduction on metal grade and recovery in concentrate

The fixed parameters are 5 wt% reductant, 10 wt% sulfur and a reduction temperature of 1050°C with a duration times of 60 minutes, 90 minutes, and 120 minutes. In Figure 4, the AAS analysis shows that the nickel and iron grades decrease with increasing duration time. According to Elliot et al. (Elliot et al. 2017), prolonging the duration time in the reduction process could result in more CO production, resulting in more metallic iron. Thus, the iron recovery increases and suppresses the nickel grade in the concentrate. Nevertheless, at 120 minutes, the nickel and iron recovery decreases due to the exhausted reductant, which

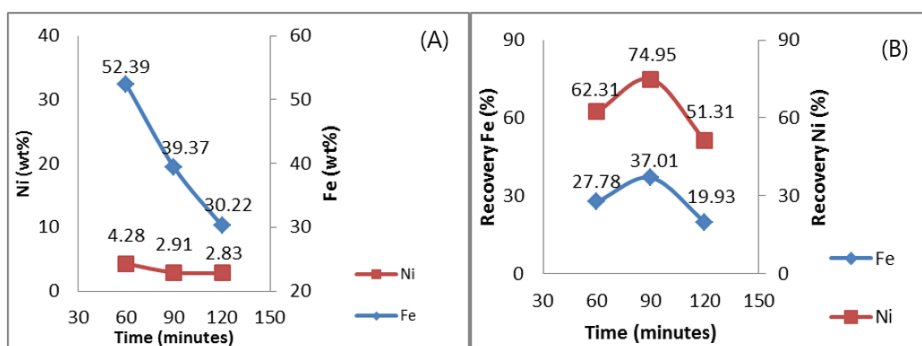


Fig. 4. The effect of the duration of process variation on a – the weight percent of nickel and iron and, b – the recovery of nickel and iron

Rys. 4. Wpływ czasu trwania zmienności procesu a – procent wagowy niklu i żelaza, b – odzysk niklu i żelaza

results in the re-oxidation of the metallic phase. The highest nickel recovery is found with the reduction process with a duration of 90 minutes.

According to Figure 5, the fayalite (Fe_2SiO_4), quartz low (SiO_2), magnesioferrite (Fe_2MgO_4), wustite (FeO), iron-nickel (FeNi) and pyrrhotite (FeS), which is found from the XRD analysis. Table 5 presents more detailed percentages for the phases. The longer duration time at 1050°C resulted in a low iron-nickel (FeNi) peak, which indicates that the

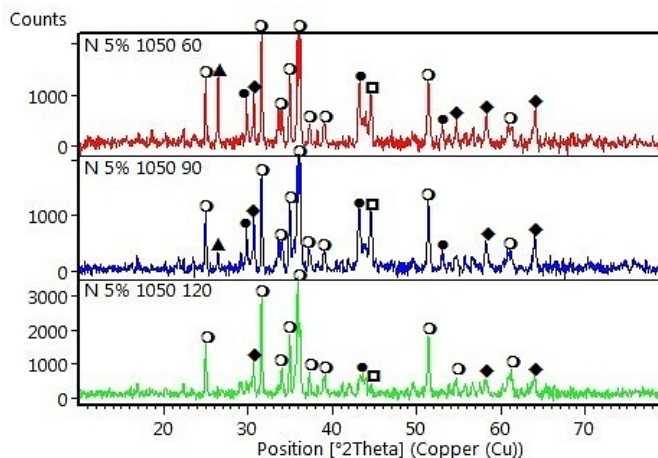


Fig. 5. XRD pattern of the effect of duration time 60 minutes (green), 90 minutes (blue), and 120 minutes (red) on the changing phase with 10% sulfur and temperature of 1050°C
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi , ● = FeS

Rys. 5. Wzór XRD wpływu czasu trwania 60 minut (zielony), 90 minut (niebieski) i 120 minut (czerwony) na zmianę fazy z 10% siarką i temperaturą 1050°C
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi , ● = FeS

Table 5. Rietveld refinement of calculation of the reduction with the additive compounds 10% sulfur and the duration time variation.

Tabela 5. Udoskonalenie Rietvelda dla obliczenia redukcji z dodatkami 10% siarki oraz zmiennym czasem trwania procesu

Compound	The total percentage of reduction products (%)		
	60 minutes	90 minutes	120 minutes
Fayalite (Fe_2SiO_4)	47.3	50.8	59.4
Quartz (SiO_2)	9.0	1.5	–
Magnesioferrite (Fe_2MgO_4)	24.0	26.4	24.6
Iron-nickel (FeNi)	3.2	3.1	0.1
Pyrrhotite (FeS)	16.4	16.8	13.3

re-oxidation of the metallic phase occurred due to the limit of reductant amount in pellets (Elliot et al. 2017; Valix and Cheung 2002). The SiO_2 peak decreases with increases of the process duration because it binds the FeO to form a fayalite phase (Fe_2SiO_4). The addition of sulfur could reduce the amount of magnetite to form FeS. In general, it has been proven that the addition of sulfur could increase the grade and recovery of nickel (Elliot et al. 2015).

2.3. Effect of anthracite coal reductant variations on metal grade and recovery in nickel concentrate

The fixed parameters are 10% sulfur, duration time for 60 minutes and the temperature of 1150°C with a variations in the amount of reductant – 5 wt%, 10 wt% and 15 wt%. The AAS results in Figure 6 show that increasing the reductant amount from 5 to 10% could decrease the nickel grade due to the increasing rate of iron's metallization (Foster et al. 2016). Nevertheless, the addition of more coal would lower the metal grade and the recovery. The impurities in the coal, such as phosphor and quartz, could inhibit the reduction of metallic oxide. The increasing percentage of reductant to the optimum amount of carbon might contribute to the reduction of iron and nickel so that the metal increases and the excess carbon decreases (Wang et al. 2017).

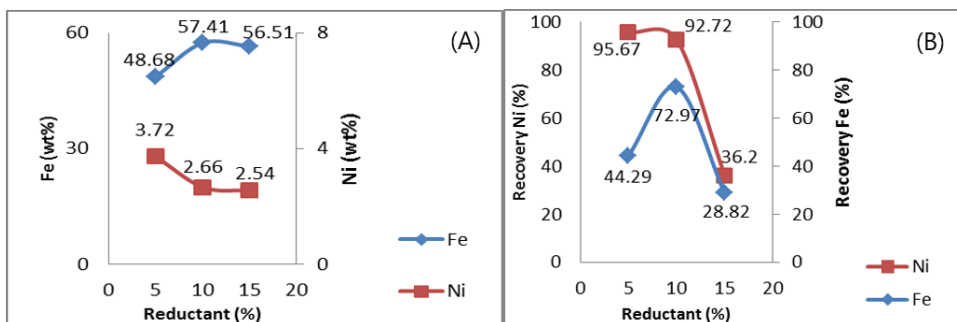


Fig. 6. The effect of percentage variation of reductant on a – the weight percent of nickel and iron and, b – the recovery of nickel and iron

Rys. 6. Wpływ procentowej zmienności reduktora a – procent wagowy niklu i żelaza, b – odzysk niklu i żelaza

In Figure 7, the fayalite (Fe_2SiO_4), quartz low (SiO_2), magnesioferrite (Fe_2MgO_4), wustite (FeO), iron-nickel (FeNi) and pyrrhotite (FeS), which is characterize observed by the XRD analysis. Table 6 presents more detailed percentages of the phases. The addition of more reductant increased ferronickel's intensity because it could enhance the reduction rate of metallic oxide into metallic phase (ferronickel). The FeS phase increased with the addition of the reductant and diminished the fayalite phase. This could be formed from the reaction between FeO and SiO_2 .

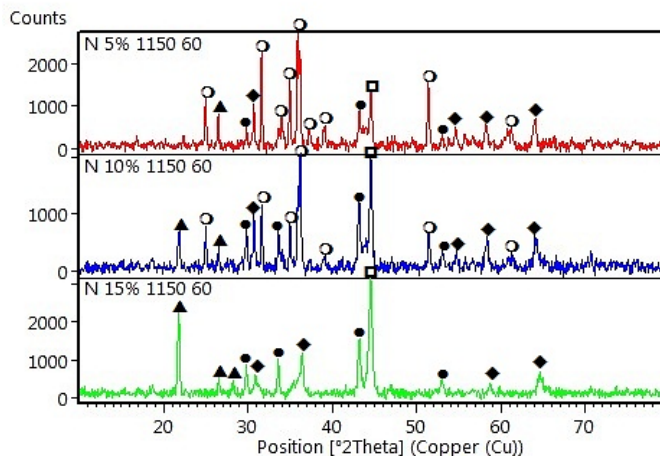


Fig. 7. XRD pattern of the effect of percentage reductant 5% (green), 10% (blue), and 15% (red) on the changing phase with temperature reduction of 1150°C and a duration time of 60 minutes
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi , ● = FeS

Rys. 7. Wzór XRD wpływu reduktora 5% (zielony), 10% (niebieski) i 15% (czerwony) na zmianę fazy ze spadkiem temperatury 1150°C i czasem trwania 60 minut
 symbol: ○ = Fe_2SiO_4 , ▲ = SiO_2 , ◆ = Fe_2MgO_4 , □ = FeNi , ● = FeS

Table 6. Rietveld refinement of calculation results of the reduction with the additive compounds 10% sulfur and the variation addition of reductant

Tabela 6. Udoskonalenie Rietvelda dla wyników obliczeń redukcji z dodatkami 10% siarki i zmienności dodatku reduktora

Compound	The total percentage of reduction products (%)		
	5%	10%	15%
Fayalite (Fe_2SiO_4)	52.7	29.3	–
Quartz (SiO_2)	3.2	7.3	22.1
Magnesioferrite (Fe_2MgO_4)	25.8	29.0	22.3
Iron-nickel (FeNi)	7.2	12.0	25.4
Pyrrhotite (FeS)	11.1	20.7	27.4

SEM-EDS analysis.

The fixed parameters are the variations in the reductant amount and additives of 10% at 1150°C with a duration time of 60 minutes. Figure 8 shows that the high reductant concentration caused a decrease of ferronickel grain size (white). In the reductant, 5% gained an average grain size of 24.69 μm , the 10% reductant gained an average grain size of 20.86 μm ,

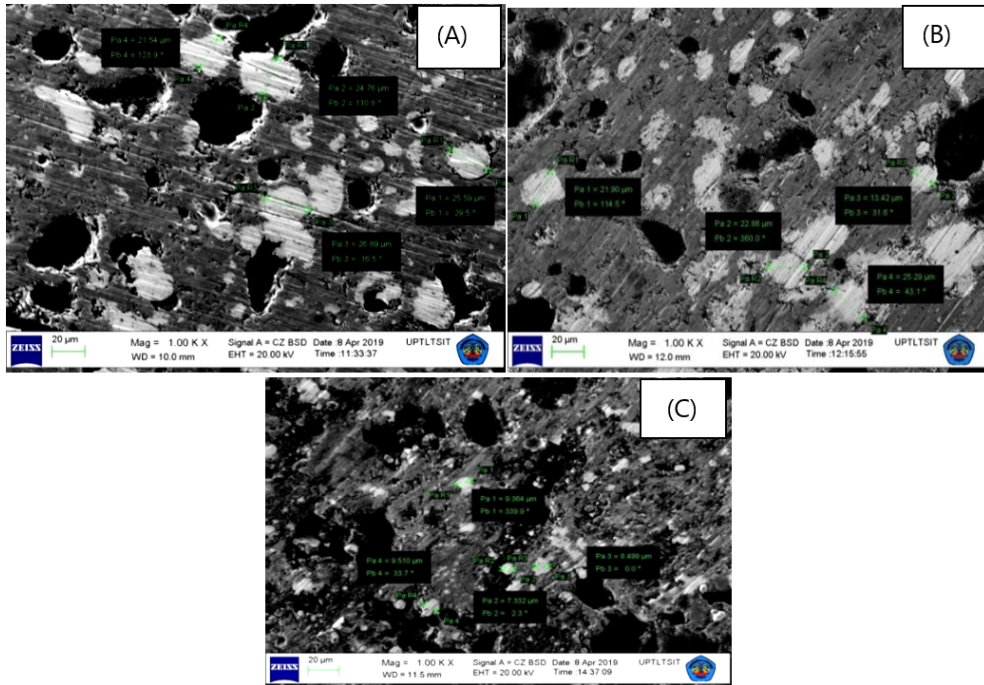
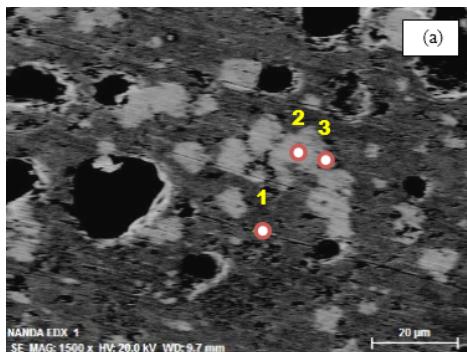


Fig. 8. The results of the grain size reduction of the sample with additives of 10% sulfur at the temperature of 1150°C with the duration of process of 60 minutes and the variation of reductant: a – 5%, b – 10%, c – 15%

Rys. 8. Wyniki rozdrabniania próbki z dodatkami 10% siarki w temperaturze 1150°C z czasem trwania procesu 60 minut i zmienną ilością reduktora: a – 5%, b – 10%, c – 15%



b)

Element	Point 1 (wt%)	Point 2 (wt%)	Point 3 (wt%)
O	98.03	–	–
Fe	1.92	98.26	75.31
Si	0.03	–	–
Mg	0.02	–	–
Ni	–	1.74	–
S	–	–	24.69

Fig. 9. a – SEM images of 10% elemental sulfur additive at 1150°C with a duration time of 60 minutes and 10% reductant, b – EDS spectra of points 1, 2, and 3

Rys. 9. a – obrazy SEM 10% dodatku siarki elementarnej w 1150°C z czasem trwania 60 minut i 10% reduktorem, b – widma EDS punktów 1, 2 i 3

and the 15% reductant gained an average grain size of 17.35 μm . This indicates that the excessive coal addition could inhibit the reduction rate of metal oxide due to more impurities from coal, such as phosphor, quartz and aluminum oxide (Elliot et al. 2017). The ferronickel size is an important parameter in the selective reduction of nickel laterite for determining the recovery of ferronickel. The large size of ferronickel improves the degree of ferronickel release from impurities by crushing/grinding process continued with the magnetic separation process. Therefore, the addition of 5 wt% of reductant should be the optimum condition. From SEM-EDS analysis, the largest grain size (with 5 wt% of reductant) contains quartz at levels not higher than 5%, as shown in Table 6 (Rietveld analysis).

The optimum parameter is 10% sulfur at a temperature of 1150°C with a duration time of 60 minutes and 5% reductant according to SEM-EDS data. Point 1 could imply that the highest element is oxygen and another element, such as iron, silicon, and magnesium; thus, the possible phases formed are Fe_2SiO_4 and Fe_2MgO_4 . Point 2 indicates that the highest element is iron with another element, nickel. Therefore, it should form iron-nickel alloy or ferronickel (FeNi). The last point indicates that the highest element is iron and other sulfur elements that form FeS. The indication of sulfur can suppress the formation of iron and decrease the grain size of ferronickel.

Conclusion

The optimum grade and recovery of nickel appears at a temperature of 1150°C with a duration time of 60 minutes and 10% reductant, which obtains 3.72 wt% and 95.67 %. The increasing reduction temperature could enhance the reduction rate of metallic oxide, which transformed into a metallic phase. Nevertheless, high metallization of iron could suppress the nickel grade in concentrate. The very long process duration negatively affected the metal grade and recovery due to the possibility of re-oxidation of the metallic phase. The excessive reductant is affected to inhibit the reduction rate of metallic oxide due to its content of impurities such as phosphor, quartz and aluminum oxide, which could also lower the grain size of ferronickel.

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THE EFFECT OF SULFUR, TEMPERATURE, THE DURATION OF PROCESS AND REDUCTANT ON THE SELECTIVE REDUCTION OF LIMONITE ORE

Keywords

limonite, sulfur, selective reduction, temperature, the duration of process

Abstract

Lateritic nickel ore is used for producing of ferronickel. Nickel grade in ferronickel ranged from 20–40%. Ferronickel is commonly used to manufacture stainless steel. A new method that can increase the levels of nickel grade is selective reduction, which is a process to reduce the metal oxide to the metallic phase with the addition of additives. In this work, the selective reduction of limonitic nickel ore was carried out by add the 5 wt%, 10 wt%, and 15 wt% of reductant and the 10 wt% of sulfur as additive. The process of selective reduction is performed at temperatures of 950, 1050, and 1150°C

with the duration of process of 60, 90, and 120 minutes, followed by magnetic separation to separate between the concentrate and tailings. The characterization used AAS, XRD, and SEM-EDS for grade and recovery; phases transformation; and the microstructure analysis. The optimum of the grade and recovery of nickel was obtained at a temperature of 1050°C with the duration of process of 60 minutes and 5 wt% of reductant and 10 wt% of additive, which obtain 3.72 wt% and 95.67%. The metal grade and recovery was increase with the increasing of temperature reduction. Nevertheless, too long of the duration of process and too many reductant addition resulted in negative effect on selective reduction of lateritic nickel ore. Highest recovery could get more nickel in the process. And sulfur has the important rules when the selective reduction has been done on the increasing nickel content, the forming of FeS, and decreasing the grain size of ferronickel according to the microstructure in the SEM images around ~30 µm.

WPLYW SIARKI, TEMPERATURY, CZASU TRWANIA PROCESU I REDUKTORA NA SELEKTYWĄ REDUKCJĘ RUDY LIMONITU

Słowa kluczowe

limonit, siarka, redukcja selektywna, temperatura, czas trwania procesu

Streszczenie

Ruda laterytowa niklu jest wykorzystywana do produkcji żelazoniklu. Zawartość niklu w żelazoniklu wahała się w przedziale 20–40%. Żelazonikiel jest powszechnie stosowany do produkcji stali nierdzewnej. Nową metodą, która może zwiększyć poziom niklu, jest redukcja selektywna, czyli proces redukcji tlenku metalu do fazy metalicznej z dodatkami. W niniejszej pracy prowadzono selektywną redukcję rudy limonitowej niklu przez dodanie 5, 10 i 15% wag. reduktora oraz 10% wag. siarki jako dodatku. Proces selektywnej redukcji odbywa się w temperaturach 950, 1050 i 1150°C z czasem trwania 60, 90 i 120 minut, po czym następuje separacja magnetyczna w celu oddzielenia koncentratu od odpadów. Zastosowano: AAS, XRD i SEM-EDS do oceny procesu, transformację faz oraz analizę mikrostruktury. Optimum stopnia odzysku niklu uzyskano w temperaturze 1050°C przy czasie trwania procesu 60 minut i 5% mas. reduktora i 10% mas. dodatku, co daje 3,72% mas. i 95,67%. Gatunek i odzysk metalu wzrastały wraz z obniżeniem temperatury. Niemniej jednak zbyt długi czas trwania procesu i zbyt częste dodawanie reduktora skutkowało negatywnym wpływem na selektywną redukcję laterytowej rudy niklu. Przy najwyższym odzysku można w tym procesie uzyskać więcej niklu. A siarka spełnia ważną rolę, gdy selektywna redukcja została przeprowadzona w celu zwiększenia zawartości niklu, tworzenia FeS i zmniejszenia wielkości ziarn żelazoniklu, zgodnie z mikrostrukturą na obrazach SEM około ~30 µm.

