

THE ROLE OF MIXTURE ADDITIVES ON THE SELECTIVE REDUCTION OF SAPROLITIC NICKEL LATERITE: SODIUM SULFATE - CALCIUM SULFATE

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ABSTRACT

High energy consumption and production costs during the metal-slag smelting in the pyrometallurgical process caused many alternative methods to be developed. Selective reduction technology emerges as a potential solution, enabling the processing of nickel laterite into ferronickel concentrate at lower operational temperatures ranging from 1000°C to 1250°C with the presence of some additives. This work investigated the effect of a combination of two additives, i.e. sodium sulfate and calcium sulfate, on the reduction process of saprolitic nickel laterite on nickel and iron grade and recovery in ferronickel concentrate, phase transformation, and the morphology of the resulting ferronickel particles. The saprolitic nickel laterite, coal as a reductant, sodium sulfate and calcium sulfate as additives were mixed and then pelletized into a 10 - 15 mm diameter. The reduction process of pellets was carried out in a muffle furnace from 1050°C to 1250°C for 60 min, and a magnetic separation process was continued to segregate the ferronickel and impurities. Techniques such as XRF, XRD, and SEM-EDS are employed to ascertain the content of nickel and iron, phase changes, and the microstructure of the ferronickel. The study found that the addition of 10 wt. % of the mixture additives, with the composition ratio of 75 : 25 for sodium sulfate and calcium sulfate, at a reduction temperature of 1250°C, generated the optimum of the nickel content and recovery of 17.06 % and 57.01 %, respectively. It also enlarges the particle size of ferronickel up to 26.94 µm. The combination of sodium sulfate and calcium sulfate as additives could enhance the nickel content and recovery in ferronickel by the mechanism of segregating the nickel and iron from the magnesium silicates phase in saprolite and providing the formation of low-melting point phase of troilite promotes the aggregation of ferronickel particle, respectively.

Keywords: nickel laterite; sodium sulfate; calcium sulfate; selective reduction; ferronickel.

INTRODUCTION

Nickel is commonly used to manufacture stainless steel, superalloys, non - ferrous mixtures, castings, electric batteries, and other products [1 - 6]. The development of technology for processing lateritic nickel ore as a raw material for nickel metal continues to develop due to the depletion of sulfidic ore [7 - 9]. In addition, it is known that the activity of mining nickel laterite is also cheaper than nickel sulfide [10]. There are several pyrometallurgical technologies in the

processing of lateritic nickel ore, such as Rotary Kiln-Electric Furnace (RKEF), Falconbridge Nickel Smelting (FNS), Blast Furnace (BF), Submerged Arc Furnace (SAF) [11 - 13]. Using pyrometallurgical process technology can produce a higher nickel recovery than hydrometallurgical [11]. However, the pyrometallurgical process involves melting at high temperatures, increasing energy consumption, production costs, and exhaust gas emissions.

The selective reduction technology becomes an alternative in processing lateritic nickel ore into

ferronickel [14, 15]. Using selective reduction followed by magnetic separation, lateritic nickel ore can be processed into ferronickel concentrate. This process is carried out at low process temperatures of 1000°C - 1300°C, and the limited use of additives and reductants makes the process effective and efficient in terms of energy consumption and production costs [14]. The use of sulfur/sulfate-type additives in the selective reduction process of nickel ore aims to significantly increase the nickel recovery in the concentrate by forming low melting point compounds [16 - 18]. Generally, other additives are often used in the selective reduction process, including carbonate and chloride compounds [19 - 24].

Previous studies state that the type and amount of additives determine the product of the selective reduction process. From the previous research, adding 5 wt. % calcium sulfate (CaSO_4) resulted in 5.01 % Ni with a recovery rate of 98.70 %. It was noted that there is a difference compared to the experimental results without using calcium sulfate additive, where it produced 2.9 % Ni with a recovery rate of 88.3 % [25]. Calcium sulfate plays a crucial role in the selective reduction process by inhibiting the reduction of iron oxide and promoting the formation of compounds with low melting points, thus increasing the recovery results of ferronickel. Sodium sulfate (Na_2SO_4) could enhance the metallization ratio of nickel and decrease the metallization of iron through sulfidation reactions, leading to the formation of troilite (FeS), which inhibits the metallization of iron [13]. It was also mentioned that the presence of sodium sulfate leads to the formation of nepheline ($\text{Na}_2\text{Si}_2\text{O}_5$), which has low melting points. It resulted in the increasing volume of the liquid phase, thereby enhancing the rate of ion mass transfer. Concurrently, the reducing atmosphere decreases, reducing the reductant's diffusive gas transfer and inhibiting the metallization of iron oxide. Therefore, the selective reduction process of metallic nickel becomes more effective.

Low nickel grade in the selective reduction process of saprolitic nickel has been a concern due to the presence

of a complex magnesium silicate structure, which has low reducibility for metal oxide. Increasing nickel grade can be carried out by inhibiting the metallization of iron. Nevertheless, it will be lowering its recovery. Therefore, optimizing the use of additives in the selective reduction process is necessary to increase the nickel grade and recovery in the ferronickel concentrate. The studies on the additive mixture of sodium sulfate and calcium sulfate have yet to be extensively explored. Therefore, this research will investigate the effect of adding a mixture of sodium sulfate and calcium sulfate additives to saprolite ore. This mixed additive of sodium sulfate and calcium sulfate is expected to promote a more liquid phase and disrupt the structural bonds of iron-nickel-silicate-magnesium found in saprolite nickel ore.

EXPERIMENTAL

Materials

Saprolitic nickel ore was obtained from Southeast Sulawesi, Indonesia. The chemical composition of the nickel ore was determined through the XRF testing, as shown in Table 1. The XRD and the Rietveld analysis in Fig. 1 and Table 2 identified the presence of compounds such as lizardite, quartz, forsterite, enstatite, goethite, and talc. It is observed that most of the nickel and iron elements are associated with hydroxides, magnesium silicate hydroxides, and magnesium silicates.

Coal was added to the reduction process as a reductant. The analysis of coal is listed in Table 3. Using coal as a reductant can generate reductant gas, such as carbon monoxide. The reductant dosages are determined to enhance the reduction efficiency, thus enabling more metal oxides to be reduced, as demonstrated by the following Eq. (1 - 3) [26].

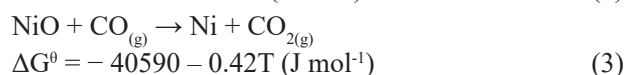
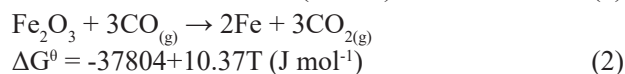
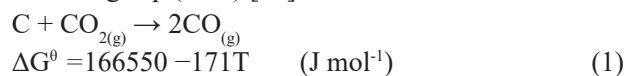


Table 1 Chemical composition of nickel laterite (wt. %).

Elements	Fe	Ni	MgO	CaO	Al_2O_3	SiO_2	Cr_2O_3
wt %	15.27	1.99	14.09	0.79	2.02	32.27	1.41

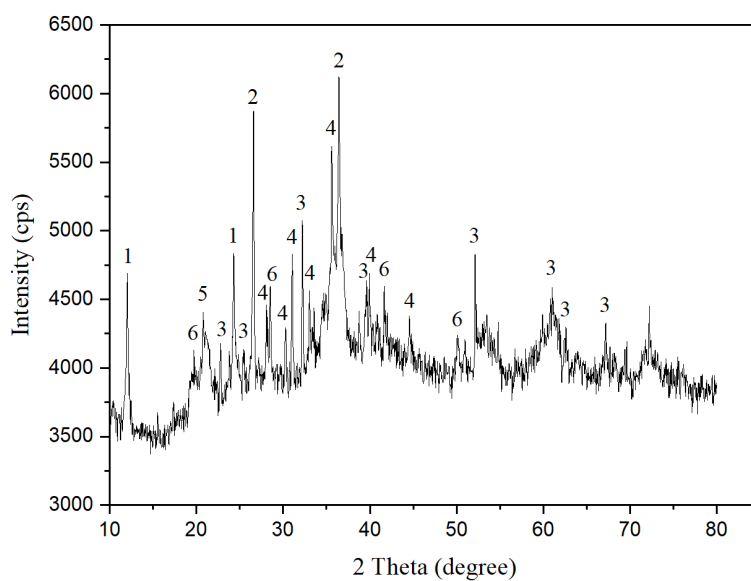


Fig. 1. XRD analysis of nickel laterite (1 - lizardite, 2 - quartz, 3 - forsterite, 4 - enstatite, 5 - gohetite, 6 - talc).

Table 2. Rietveld analysis of nickel laterite.

Compounds	wt. %
Lizardite - $(\text{Ni,Fe,Mg})_3\text{Si}_2\text{O}_5(\text{OH})_4$	16.4
Quartz - SiO_2	9.0
Forsterite - $(\text{Fe,Ni,Mg})\text{SiO}_4$	13.9
Enstatite - $((\text{Fe,Ni,Mg})_2\text{Si}_2\text{O}_6)$	19.0
Ghoetite - $(\text{Fe,Ni})\text{OOH}$	28.5
Talc - $\text{H}_2\text{Mg}_3(\text{SiO}_3)_4$	13.1

Reduction and Magnetic Separation Process

The saprolitic nickel ore and coal were crushed and ground less than 149 μm . The finely ground saprolitic nickel and reductant were thoroughly mixed with additives, i.e., calcium sulfate, sodium sulfate, and a mixture of both. The effect of coal and additive dosage was investigated on this reduction of saprolitic nickel ore. This mixture was then pelletized into 10 - 15 mm diameter pellets and dried in an oven at 120°C for 4 h.

The dry pellet samples were placed in a graphite crucible and reduced in a muffle furnace at various reduction temperatures, specifically 950°C, 1050°C, and 1150°C, for 60 min. After the reduction process, the reduced pellets were quenched in water and dried at 120°C for 4 h, then ground into a mesh size of less than 74 μm using a vibratory disk mill. Subsequently, a wet magnetic separation process was performed on the finely reduced ore using a 500 Gauss permanent magnet. The magnetic particles of the reduced ore or concentrate were rich in ferronickel, while the non-magnetic particles, or tailings, were rich in impurities.

Characterization

During the reduction process, phase transformations in saprolitic nickel ore were analysed using XRD. The XRF analysis was carried out to determine the nickel and iron content in ferronickel concentrates, while the SEM - EDS analysis was used to observe the ferronickel particle morphology.

Table 3. Proximate, sulfur, and calorie analysis of coke.

Proximate analysis (wt. %)				Sulfur (wt. %)	Calorie Value, Cal gr ⁻¹
Moisture	Volatile Matter	Ash	Fix Carbon		
3.14	18.25	18.25	60.35	1.90	5832

RESULTS AND DISCUSSION

Effect of Sodium Sulfate on Selective Reduction

Fig. 2 shows the effect of sodium sulfate additive on nickel and iron grade and recovery of ferronickel in a selective reduction process, which was carried out at a temperature of 1150°C for 60 min with 0.2 stoichiometry of reductant. Fig. 2a shows a significant increase in nickel grade, found by adding 10 wt. % of sodium sulfate, which is 17.29 % with the iron grade is 40.26 %. Conversely, Fig. 2b shows that the recovery of nickel and iron tends to decrease by increasing sodium sulfate. The nickel and iron recovery is only 12.74 % and 17.28 %, respectively, when 10 wt. % of sodium sulfate is added. Furthermore, adding sodium sulfate ranging from 10 to 20 wt. % decreases the nickel and iron grade and recovery due to the intensive sulfidation mechanism occurring for nickel and iron oxide. It is agreed with the experiment reported by Rao et al. that the sulfidation mechanism occurs in iron and nickel [27].

Fig. 3 shows the phases in saprolitic nickel ore that have been reduced with the addition of sodium sulfate, where the compounds such as ferronickel (FeNi), troilite (FeS), forsterite ((Mg,Fe,Ni) SiO₄), and enstatite ((Mg,Fe,Ni) SiO₃) are present. The presence of magnesium silicate compounds can be a cause for the low grade and recovery of nickel and iron produced in the selective reduction of saprolitic nickel ore, where nickel and iron can substitute the magnesium atom. The

reducibility of nickel and iron in magnesium silicate compounds in saprolitic nickel ore is significantly lower compared to iron - nickel hydroxide in limonitic nickel ore [28].

From the XRD analysis in Fig. 3, as the addition of sodium sulfate increases, the peak of troilite (FeS) also increases, formed due to the decomposition of the additive reacting with iron. The decrease in grade and recovery of nickel and iron can be attributed to form compounds such as FeS and NiS due to the excessive additive addition, both are non-magnetic. Consequently, the nickel and iron in the sulphide phase will enter the tailings, lowering the recovery rate of the reduction process [29].

Effect of calcium sulfate on selective reduction

Fig. 4 shows the effect of calcium sulfate additive on the nickel and iron grade and recovery in the selective reduction process of saprolitic nickel ore. The reduction was carried out at 1150°C for 60 min with the addition of 0.2 stoichiometry reductant. Fig. 4a shows an increase in nickel and iron grade with the increase of calcium sulfate addition, although not significantly. In Fig. 4b, the graph also shows the recovery of nickel and iron, which does not vary much but indicates a trend of decreasing recovery with adding more calcium sulfate. The presence of sulfur in calcium sulfate has a similar effect to sodium sulfate, which promotes a sulfidation mechanism for inhibiting the metalization of

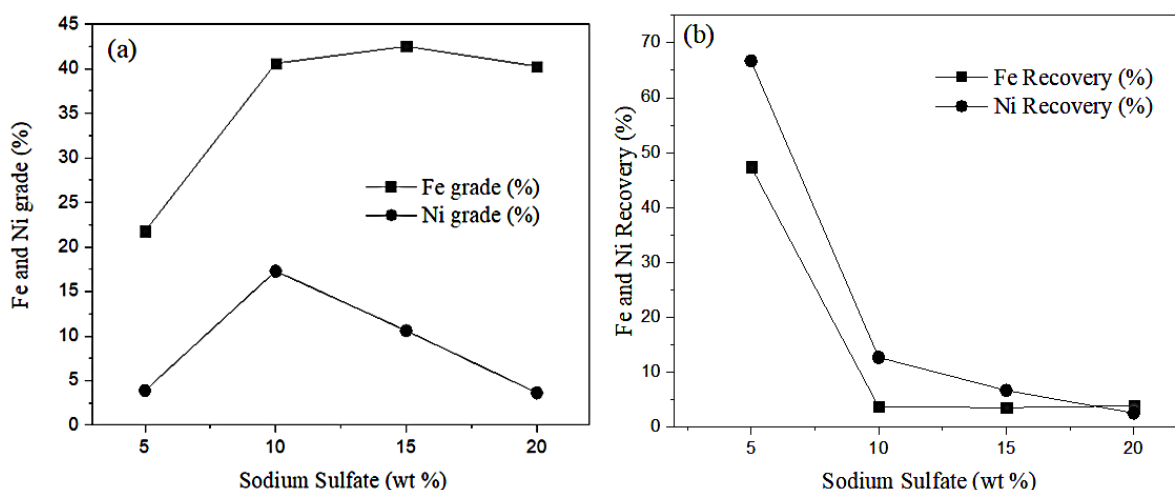


Fig. 2. Effect the increasing of sodium sulfate dosage on nickel and iron: (a) grade and (b) recovery.

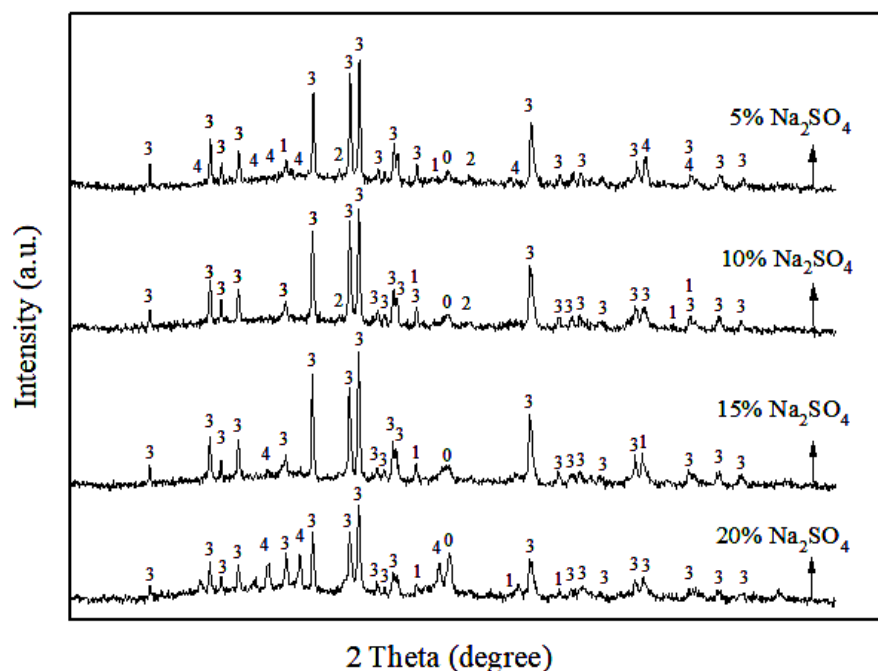


Fig. 3. Effect of increasing sodium sulfate dosage in phase transformation on the result of selective reduction at the temperature of 1150°C for 60 min with 5; 10; 15; 20 wt. % of sodium sulfate (0 - ferronickel, 1 - troilite, 2 - fayalite, 3 - forsterite, 4 - enstatite).

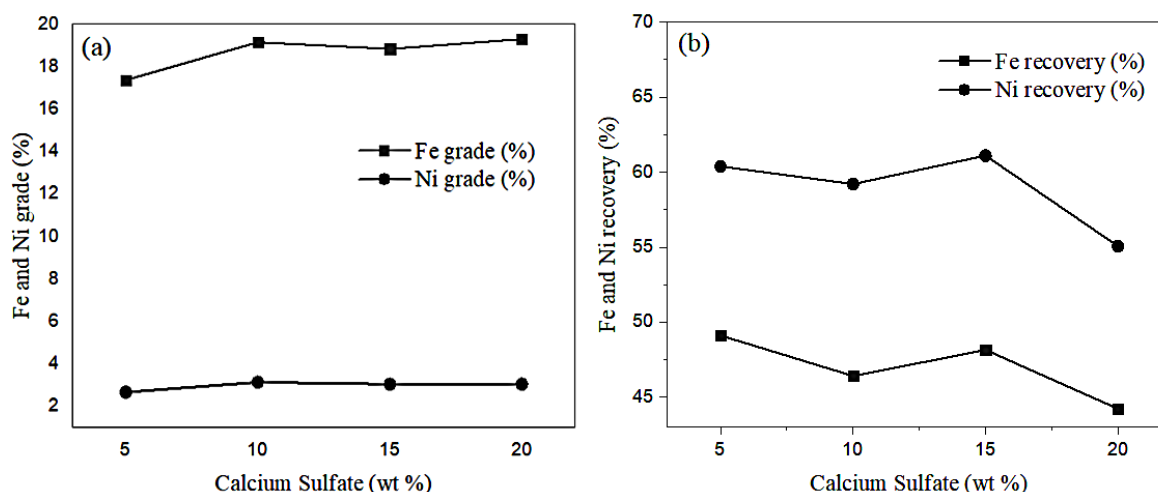


Fig. 4. Effect of increasing dosage of calcium sulfate with the temperature of 1150°C for 60 min and 0.2 of stoichiometry reductant on nickel and iron: (a) grade and (b) recovery.

iron. However, the nickel and iron grade of ferronickel resulting from the use of sodium sulfate is higher than calcium sulfate. Nevertheless, it is contrary to the nickel and iron recovery. This work found the highest recovery of nickel and iron at 15 wt. % of calcium sulfate addition, i.e. 61.11% and 48.16%. The nickel and iron grades at this point are 3.05 % and 18.83 %, respectively.

The XRD analysis was carried out to observe the effect of calcium sulfate in the reduction of saprolitic nickel ore using calcium sulfate on phase transformation. Fig. 5 shows the presence of clinoenstatite and forsterite compounds due to the dehydroxylation of lizardite - $(\text{Mg,Fe})_3\text{Si}_2\text{O}_5(\text{OH})_4$. The dominant pigeonite - $(\text{Ca}(\text{Mg,Fe})\text{Si}_2\text{O}_6)$ compound is also found. It indicates that calcium sulfate can

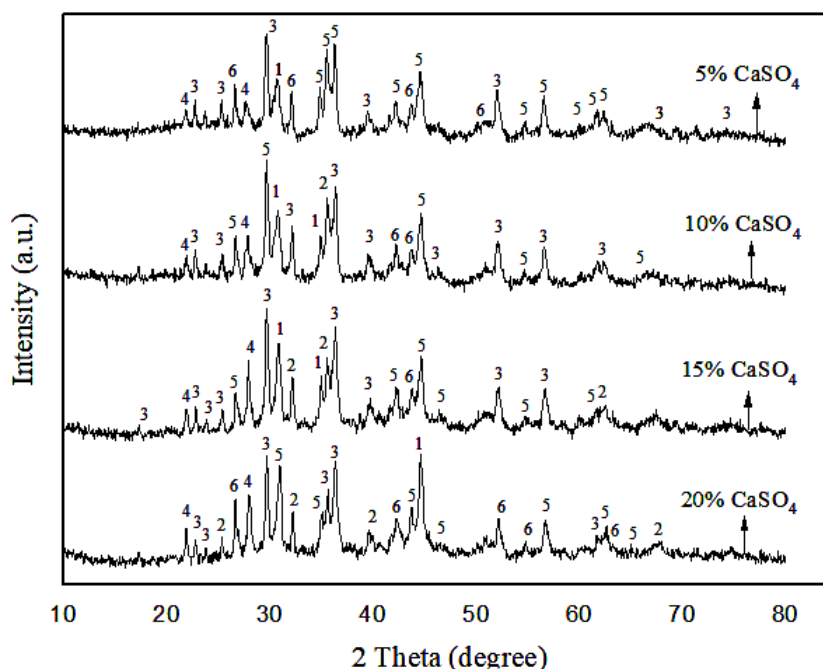


Fig. 5. Effect of increasing calcium sulfate dosage in phase transformation on the result of selective reduction at the temperature of 1150°C for 60 min with 5; 10; 15; 20 wt % of calcium sulfate (0 - ferronickel, 1 – troilite, 2 - fayalite, 3 - forsterite, 4 - enstatite, 5 - pigeonite, 6 - clinoenstatite).

enhance the reduction process by replacing the nickel and iron with calcium in the magnesium silicate structure of clinoenstatite and forsterite [17]. Thus, it explained the higher recovery of nickel and iron from calcium sulfate than sodium sulfate.

Additionally, a peak of troilite, which is effective in inhibiting the metallization of iron, is also observed. It is due to the sulfidation reaction of FeO into FeS, which hinders the metallization reaction of Fe. The decrease in grade and recovery of nickel and iron can be caused by excessive sulfidation reactions, where FeS and NiS are formed [17, 27].

Effect of mixture sodium sulfate and calcium sulfate on selective reduction

Fig. 6 shows the effect of the mixture of sodium sulfate with calcium sulfate as an additive on iron-nickel grade and recovery. The total additive was observed at 10 wt. % and 15 wt. % added on the nickel ore along with the 0.2 stoichiometry of reductant. The composition ratio of the mixture of the additives was 75:25, 50:50 and 25:75 in wt. % for sodium sulfate and calcium sulfate. The reduction was carried out at 1150°C for 60 min. Fig.

6a presents the iron-nickel grade and recovery by adding 10 wt. % of the mixture of additives. The iron-nickel grade decreases with the decreasing volume of sodium sulfate in the mixture, contrary to the recovery. Fig. 6b illustrates the effect of a mixture additive with 15 wt. % in this reduction process. It shows a trend line graph, similar to adding mixture additives with 10 wt. % in Fig. 6b. Nevertheless, a different trend line shows in nickel grade, which increases with 25 wt. % of calcium sulfate in the mixture additives. The nickel grade decreased with increased calcium sulfate in the mixture afterward. It confirms that using a mixture additive comprising sodium sulfate and calcium sulfate plays distinct roles in the iron-nickel grade and recovery in the selective reduction process of saprolitic nickel ore. The sodium sulfate plays a role in inhibiting the metallization of iron through the formation of troilite (FeS) phase. Thus, it increases the nickel grade in ferronickel. While calcium sulfate plays a role in releasing nickel and iron from the magnesium silicate structure, thus increasing the recovery of nickel and iron. A ferronickel product with more than 8 wt. % nickel should be important to consider due to the minimum nickel grade in stainless

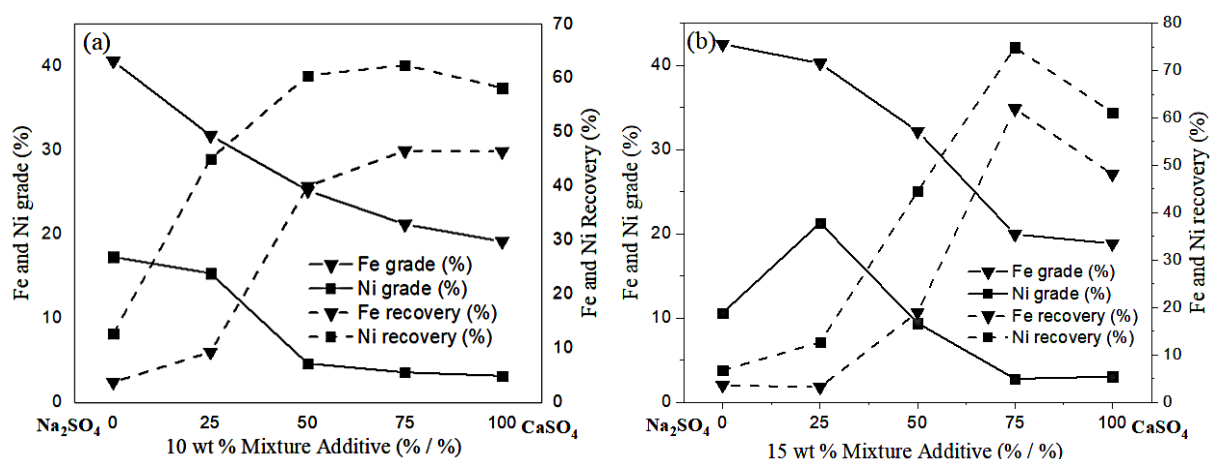


Fig. 6. Effect of the mixture sodium sulfate with calcium sulfate at the temperature of 1150°C for 60 min on iron-nickel grade and recovery: (a) 10 wt. % and (b) 15 wt. % of mixture additives.

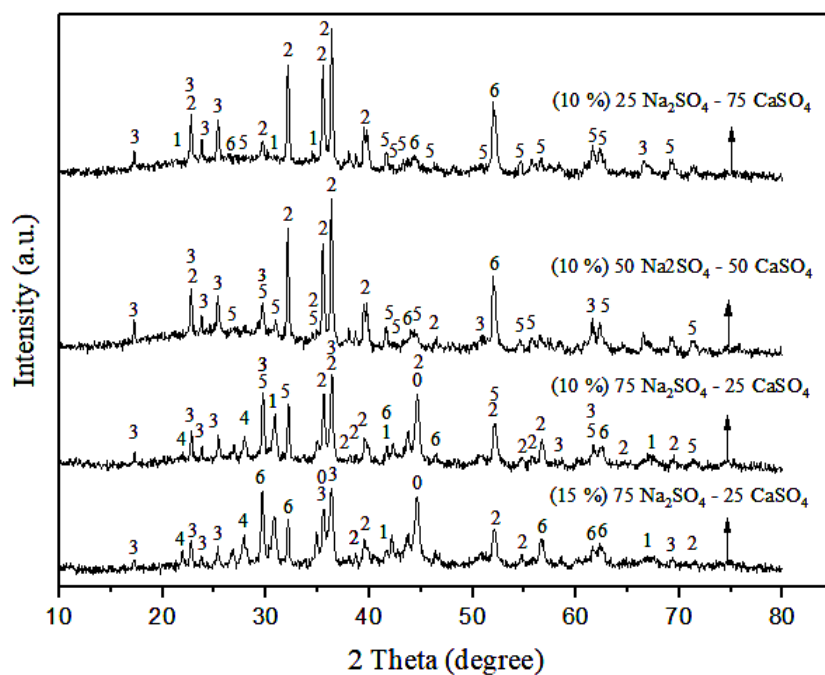


Fig. 7. Effect of sodium sulfate-calcium sulfate mixture dosage dosage in phase transformation on the result of selective reduction at the temperature of 1150°C for 60 min (0 - ferronickel, 1 - troilite, 2 - fayalite, 3 - forsterite, 4 - enstatite, 5 - pigeonite, 6 - clinoenstatite).

steel products. It is also important to consider having good nickel recovery. However, iron grade and recovery are not crucial in the product of ferronickel because it is not valued in the market. Therefore, the optimum of this mixture was selected using a 10 wt. % of mixture additives with the composition 75:25 of sodium sulfate-calcium sulfate generates 15.36 % and 45.07 % for nickel grade and recovery, respectively. Improvement of this work will be discussed later in the following sub - section

by investigating the effect of reductant amount and temperature reduction process.

Fig. 7 identifies the phases such as ferronickel (FeNi), troilite (FeS), fayalite (Fe_2SiO_4), clinoenstatite (MgSiO_3), pigeonite ($\text{Ca}(\text{MgFe})\text{Si}_2\text{O}_6$), and forsterite ($(\text{MgFe})_2\text{SiO}_4$). Forsterite and clinoenstatite, generated from the hydroxylation of lizardite, become the dominant phase formed when the composition of sodium sulfate additive is higher, such as in the 75:25

ratio for sodium sulfate and calcium sulfate. Meanwhile, the dominant phase is pigeonite when calcium sulfate dominates, as in the 25:75 ratio for sodium sulfate and calcium sulfate. Pigeonite formation resulted from the addition of calcium sulfate, releasing the bonds of nickel and iron in a magnesium silicate compound replaced with calcium [17].

Fig. 8 shows the ferronickel microstructure with a mixture of sodium sulfate and calcium sulfate additives. Iron is still associated with magnesia silicate compounds forming forsterite, as illustrated by points 3, 11, and 13, which indicates that the reduction of nickel and iron in saprolite is not entirely reduced [30]. Additionally, the presence of nickel in impurities, as seen at point 7, is negatively affected in the selective reduction process, such as the existence of nickel and iron associated with sulfur, as indicated by points 9, 10, 12, 15, and 18, can lead to low recovery of nickel and iron during the selective reduction process. It is observed that an increase in the volume of calcium sulfate in the mixture additive results

in the formation of ferronickel particles that are small and dispersed, causing low ferronickel content and high recovery in the magnetic separation process, implying the absence of a liquid phase in the sample, leading to the formation of small and dispersed ferronickel particles. The small size of these particles hinders the optimal release of ferronickel particles from impurities through the grinding process. Consequently, the nickel and iron content in the concentrate becomes low. However, it is known that using calcium sulfate additive can destroy the magnesium silicate structure, as indicated by points 5, 8, 14, and 17, and forming the pigeonite compounds.

The addition of sodium sulfate additive at 10 wt. % in the mixed additive, as shown in Fig. 8a - c, shows an increase in particle size from 8.80 μm to 16.16 μm due to the presence of troilite and fayalite, which has a low melting point. However, the ferronickel particles formed with a 15 wt. % mixture ratio of additives with a 75:25 of sodium sulfate and calcium sulfate appears larger, reaching 20.33 μm .

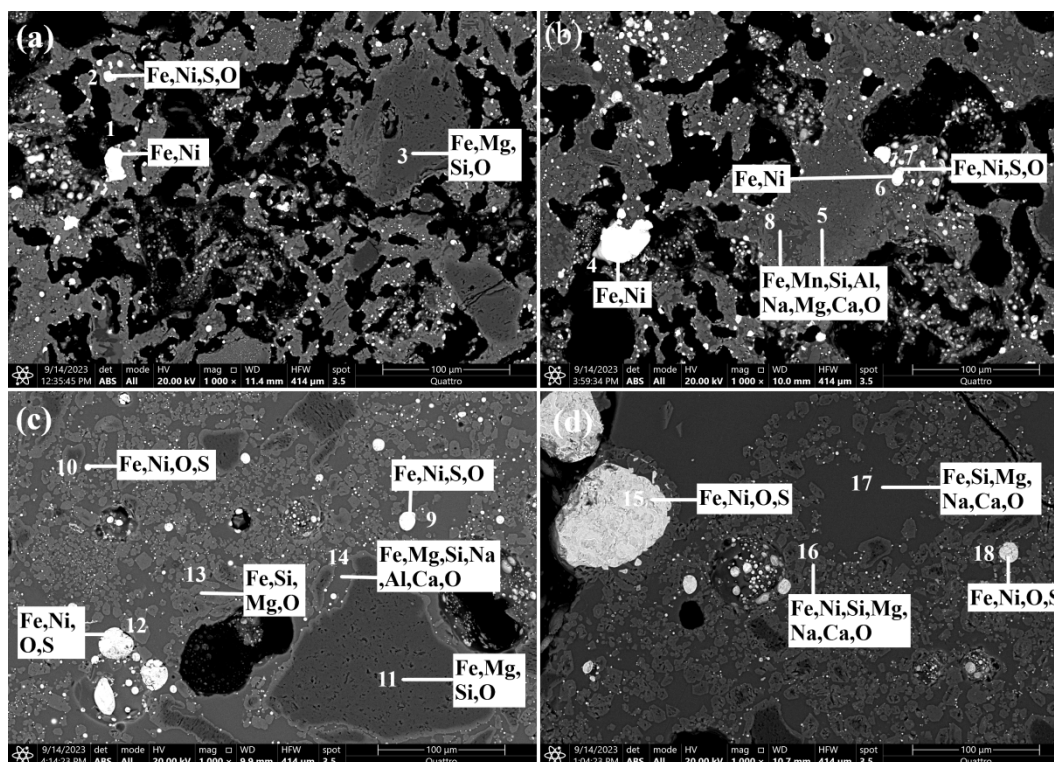


Fig. 8. SEM - EDS analysis on saprolitic nickel ore after reduction with a mixed additive of sodium sulfate and calcium sulfate at dosages and ratios of (a) 10 wt. % 25:75 Na_2SO_4 - CaSO_4 ; (b) 10 wt. % comprises of 50:50 Na_2SO_4 - CaSO_4 ; (c) 10 wt. % comprises of 75:25 Na_2SO_4 - CaSO_4 ; dan (d) 15 wt. % comprises of 75:25 Na_2SO_4 - CaSO_4 .

Effect of Reductant on the Selective Reduction with The Mixture Additive

This section investigated the effect of reductant dosage on the mixture additives (sodium sulfate - calcium sulfate) in selective reduction of saprolite. The composition ratio of mixture additives is 75:25 for sodium sulfate and calcium sulfate in 10 wt. % of total additive. The reduction process was carried out at 1150°C for 60 min. Fig. 9a shows that the nickel grade increases with the increase of reductant dosage until it reaches 0.2 stoichiometry, then decreases afterward. However, the recovery increases, as shown in Fig. 9b. The excessive supply of reductant or carbon will increase the reduction rate of metallic oxide. Thus, it increases the recovery of nickel and iron. Obtaining a high recovery of iron will suppress the nickel grade [31, 32]. As a result of 0.1 of stoichiometry carbon of reductant, insufficient reductant will make the reduction process incomplete, producing ferronickel concentrate with low nickel grade and recovery. Limited reductant should be considered for obtaining high nickel grade by inhibiting the reduction of iron oxide into metallic iron [22]. However, it will sacrifice the nickel recovery. From Fig. 9a - b, the reduction process with the 0.2 stoichiometry was selected as the optimal condition regarding the highest nickel content in the concentrate of 15.35 % with a nickel recovery of 45.07 %.

Effect of Temperature on the Selective Reduction with The Mixture Additive

Fig. 10 shows that nickel and iron grade and recovery in the concentrate increase with the rise in the reduction temperature of saprolitic nickel ore. There is a difference in the trend line increasing between nickel and iron grade, as described in Fig. 10a, where the iron grade increases significantly, unlike nickel, due to the more significant reduction of iron oxide compounds along with the reduction temperature increases, thereby suppressing the nickel content in the concentrate. Fig. 10b shows that the recovery of nickel and iron continues to increase with the rise in the reduction temperature to 1250°C. It is attributed to the increased metallization rate for nickel and iron. The ferronickel concentrate contains 17.06 % Ni and 45.48 % Fe, with recoveries reaching 57.01 % and 23.35 %, for nickel and iron respectively, was obtained from reduction of saprolite at 1250°C. According to the study reported by Dong et al., it has been reported that the reduction process of saprolitic nickel ore requires a high reduction temperature of up to 1300°C and the addition of proper and significant amounts of additives to release the bonds of nickel and iron from silicate compounds [19]. However, the high reduction temperature undoubtedly implies the high production cost required.

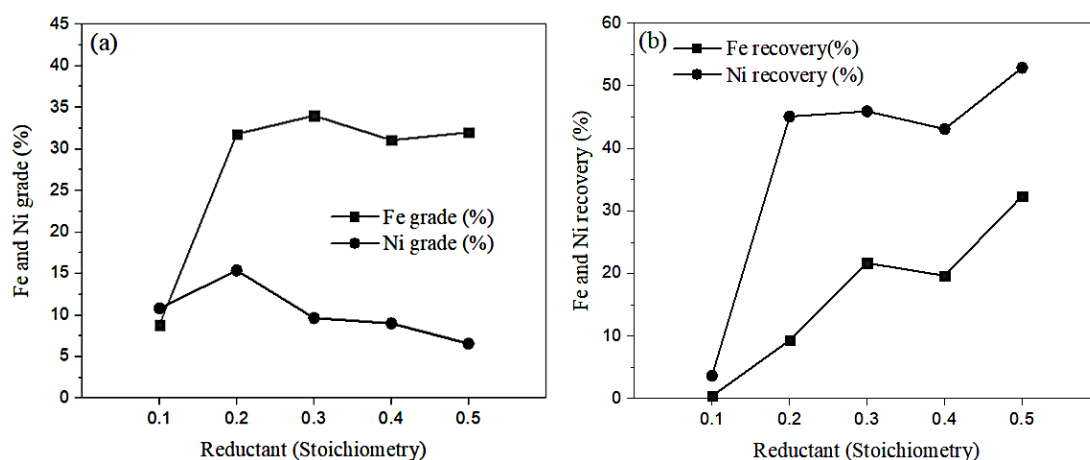


Fig. 9. Effect of reductant dosage on nickel and iron: (a) grades and (b) recovery.

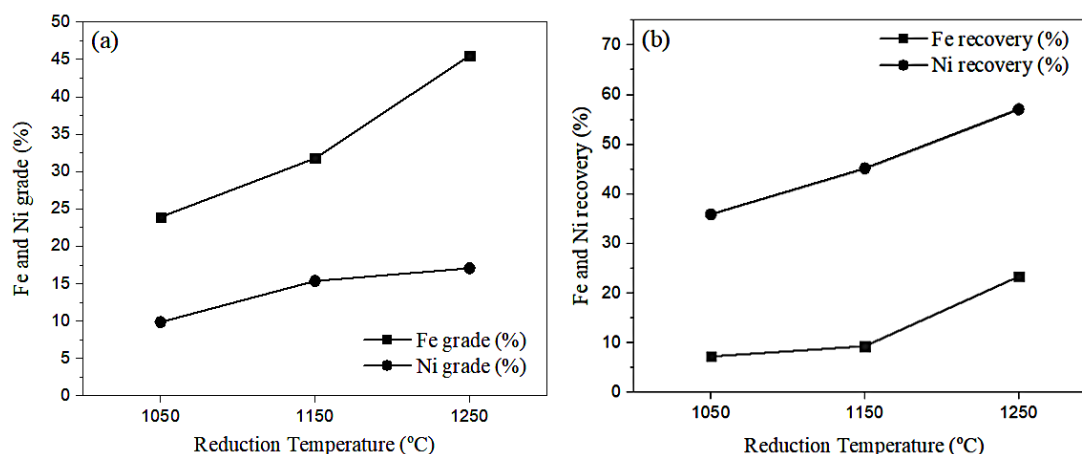


Fig. 10. Effect of various reduction temperature on nickel and iron: (a) grades and (b) recovery.

CONCLUSIONS

Using two additives, sodium sulfate and calcium sulfate in the selective reduction process of saprolitic nickel ore has been successfully producing high nickel grade of ferronickel. Mixing these two additives positively affected the selective reduction process by increasing the nickel grade and recovery. The optimal condition is observed for a 10 wt. % of mixed additive with a composition ratio of 75:25 for sodium sulfate and calcium sulfate. Calcium sulfate enhances the ferronickel recovery by destroying the magnesium silicate phase, where nickel and iron are trapped inside this structure. Calcium replaced the nickel and iron in this structure, thus releasing the nickel and iron and forming a calcium-magnesium silicate phase or pigeonite. Furthermore, the nickel and iron will be reduced and transformed into ferronickel. Meanwhile, sodium sulfate promotes the formation of a troilite, which has a low melting point phase. Forming a liquidus phase in the reduction process leads to increased particle size and higher nickel grade of ferronickel in the concentrate due to the inhibition metallization of iron by the sulfidation mechanism. Limited reductant should be considered for obtaining high nickel grade in concentrate by suppressing the carbothermic reduction of iron oxide into metallic iron. The selective reduction of saprolite with a 10 wt % of mixture additives (75:25 for sodium sulfate and calcium sulfate) with 0.3 of stoichiometry coal at 1250°C has successfully produced ferronickel concentrate containing

17.06 % Ni with 57.01 % recovery accompanied by an enlargement of particle size up to 26.94 μm .

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