International Journal of Minerals, Metallurgy and Materials
Accepted manuscript, https://doi.org/10.1007/s12613-020-2025-0

The Evolution of the Ferronickel Particles During the Reduction of Low Grade Saprolitic Laterite Nickel Ore by Coal in the Temperature Range of 900 – 1250 °C with the Addition of CaO - CaF_2 - H_3BO_3

Zulfiadi Zulhan, and Windu Shalat

Metallurgical Engineering Department, Faculty of Mining and Petroleum Engineering,
Bandung Institute of Technology, INDONESIA
E-mail: zulfiadi.zulhan@gmail.com

*Both authors contributed equally to this work:
Dr.-Ing. Zulfiadi Zulhan, email: zulfiadi.zulhan@gmail.com
Windu Shalat, B.Sc., email: wshalat@gmail.com

The Evolution of the Ferronickel Particles During the Reduction of Low Grade Saprolitic Laterite Nickel Ore by Coal in the Temperature Range of 900 – 1250 °C with the Addition of CaO - CaF_2 - H_3BO_3

Zulfiadi Zulhan, Windu Shalat

Metallurgical Engineering Department, Faculty of Mining and Petroleum Engineering,

Bandung Institute of Technology, INDONESIA

E-mail: zulfiadi.zulhan@gmail.com

Abstract

The method to produce ferronickel at lower temperature (1250 – 1400 °C) was applied since 1950s at Nippon Yakin Oheyama, Japan. Limestone was used as an additive to adjust the slag composition for lowering slag melting point. The ferronickel product was recovered by means of magnetic separator from semi-molten slag and metal after water quenching. In order to increase the efficiency of magnetic separation, bigger particle size of ferronickel is desired. Therefore, in this study, the influences of CaO, CaF_2 and H_3BO_3 additives on the evolution of ferronickel particle at temperature $\leq$ 1250 °C were investigated. The experiments were conducted at 900 - 1250 °C with the addition of CaO, CaF_2, and H_3BO_3. The reduction processes were carried out in a horizontal tube furnace for 2 h under argon atmosphere. The results showed that at 1250 °C with the CaO addition of 10% of the ore weight, the size of ferronickel particles of 20 µm was obtained. The ferronickel particle size can be increased to 165 µm by adding 10% CaO and 10% CaF_2. The addition of boric acid further increased the ferronickel particle size to 376 µm as shown by the experiments with the addition of 10% CaO, 10% CaF_2 and 10% H_3BO_3.

Keywords: saprolitic laterite nickel ore, ferronickel particles, CaO, CaF_2, H_3BO_3

1. Introduction

Nickel is one of the important metals which is widely used in daily life. Nickel has relatively low thermal and electrical conductivity, high corrosion resistance, high strength and excellent toughness. Most of the nickel has been used in the form of ferroalloy for the manufacture of stainless steel which is around 65%, 20% for nickel based alloys applications, about 9% nickel for plating, and 6% for other applications including batteries, coins, nickel chemicals, etc. [1]. In 2018, global nickel metal resources in the nickel ore were estimated at least 130 million tons with an average nickel content of 1%. This resource consists of 60% ore with laterite type and 40% with sulphide. Currently, almost 60% of nickel production comes from sulfide type nickel ore [2].

Lateritic nickel ore can be processed via hydrometallurgy and / or pyrometallurgy. In general, saprolitic laterite nickel ore is processed by pyrometallurgy and limonitic by hydrometallurgy.

Using pyrometallurgical route, there are three proven technologies for processing of saprolitic laterite nickel ore, namely RKEF (rotary kiln – electric furnace), blast furnace and Krupp-Renn. Among these technologies, RKEF is the most popular due to its capability to melt the refractory type of ferronickel slag consisting mainly of silica and magnesia. The RKEF technology is mature technology and applied across the world for processing of saprolite type lateritic nickel ore for producing nickel metal in the form of ferronickel, nickel matte or nickel pig iron. High energy consumption and high capital expenditure are the limitations of ferronickel production via RKEF. Therefore, it is desired to find alternative lateritic nickel ore processing route which has low energy consumption, low operational cost and low capital cost. One method to address this challenge is to reduce laterite nickel ore at moderate temperatures to form ferronickel particles [3]. These ferronickel particles can be then physically separated using a magnetic separator. The effectiveness of this process is highly dependent on the ease of separation of ferronickel particles from the unreduced oxides in the ore. The ease of separation of particles is very dependent on the particle size of the ferronickel particles. Larger particles will be separated more easily from surrounding impurities.

Harris et al. [3] and Dong et al. [4] have summarized the thermal upgrading of nickeliferous laterites by using additives. Most of the researches have added sulfur-containing additives to increase the size of ferronickel [5-14]. Li et al. added 20% sodium sulfate and coal into saprolite type of lateritic nickel ore and reported that the ferronickel particle size increased from 5 – 10 µm to approximately 50 µm at 1100 °C due to the presence of troilite (FeS) as an activating agent to facilitate aggregation of ferronickel particles [5]. Similar experiments were conducted by Zhu et al. where 6% calcium sulfate was added into nickel ore together with coal at 1100 °C to produce ferronickel particle with the size of 16.1 µm [6]. The effect of sulphur elemental and pyrite addition into limonite nickel ore using coal as reductant on the ferronickel particle formation was investigated by Elliot et al. [8]. In general, the addition of sulfur-containing additives increases sulphur content in the ferronickel product which later requires high amount of desulfurizing agents during stainless steel making. On the other hand, high sulphur content in the ferronickel has benefit of producing pure nickel, where iron and nickel in iron - nickel sulphide can be easily separated. The other additives are chloride based, e.g. sodium chloride and calcium chloride. Zhou et al. used sodium chloride together with coal into high magnesium low nickel lateritic ore and found that the nickel recovery increased to 98.31% at 1200 °C [15]. However, the utilization of chloridizing agent (Cl_2, NaCl, and $CaCl_2$) for processing of lateritic nickel ore leads to heavy environmental pollution [16].

Lime in the form limestone was used as additive for Krupp-Renn rotary kiln process at Oheyama Works, Nippon Yakin Kogyo, Japan since 1952 to produce ferronickel luppen at 1250 – 1400 °C [17,18]. The particle size of ferronickel luppen is between 0.5 – 20 mm, with the average size around 1 mm [19,20]. Due to confidentiality, the amount of limestone addition for ferronickel luppen production was not reported. Li et al. has studied the effect of CaO addition for reduction of low-grade saprolite type nickel laterite ore and found that the optimum CaO addition was around 10% [21]. Besides lowering the melting point of slag, CaO had an important role for replacing NiO that is bound by SiO_2 in olivine. This phenomenon was proposed as alkaline oxide effect ($CaO + 2NiO.SiO_2 = CaO.SiO_2 + 2NiO$) [21]. Lv et al. adjusted the quaternary basicity $[(m_{CaO} + m_{MgO})/(m_{SiO2} + m_{Al2O3})]$ by addition of CaO into nickel laterite ore and found that the nickel content in the ferronickel of 11.53% and nickel recovery of 98.59% can be obtained [22]. Similar results of the influence of CaO addition on nickel grade in ferronickel and nickel recovery were investigated by the other researches [16, 23].

The effect of borax was studied by Morcali et al. [24] for extraction of Ni and Co from limonitic laterite ore. It was reported that Ni-Co-Fe matte can be produced at 1000 °C by addition of borax up to 12.7% together with sodium carbonate. The presence of sulphur enhanced further the nickel and cobalt recoveries. The nickel and cobalt recoveries of 98% and 95% can be achieved by the addition of 108% flux mixture (50% Na_2CO_3 + 35% $Na_2B_4O_7.10H_2O$ + 15% SiO_2), 3.2% coke, and 20% elemental sulphur into roasted limonitic laterite ore.

In this paper, we investigated the influences of lime (CaO), fluorite (CaF_2), and boric acid (H_3BO_3) as source of borax in the slag on the evolution of ferronickel particle. The aim of these additions is to find out alternative combinations of fluxes in order to increase the size of ferronickel particles, hence enhancing its separation from the unreduced oxides and lowering the operating temperature for the ferronickel production.

2. Experiments

The lateritic nickel ore was low grade saprolite ore (LGSO) originating from Sulawesi Island, Indonesia. Before being used in the experiment, the saprolitic nickel laterite ore was dried at 135 °C for 24 h, ground using a ball mill, and then sieved to obtain the grain size of less than 65# (-210 µm). The chemical composition of nickel ore which was determined by XRF is given in Table 1 and the XRD analysis is shown in Fig. 1 which indicates that the nickel laterite ore consisted mainly of lizardite/antigorite and quartz.

Table 1. Chemical composition of saprolitic nickel ore.

%Ni	%Fe	%Si	%Mg	%Al	%Cr	%Ca	%Mn	%LOI
1.31	8.3	20.79	15.04	1.69	0.414	0.395	0.178	10.14

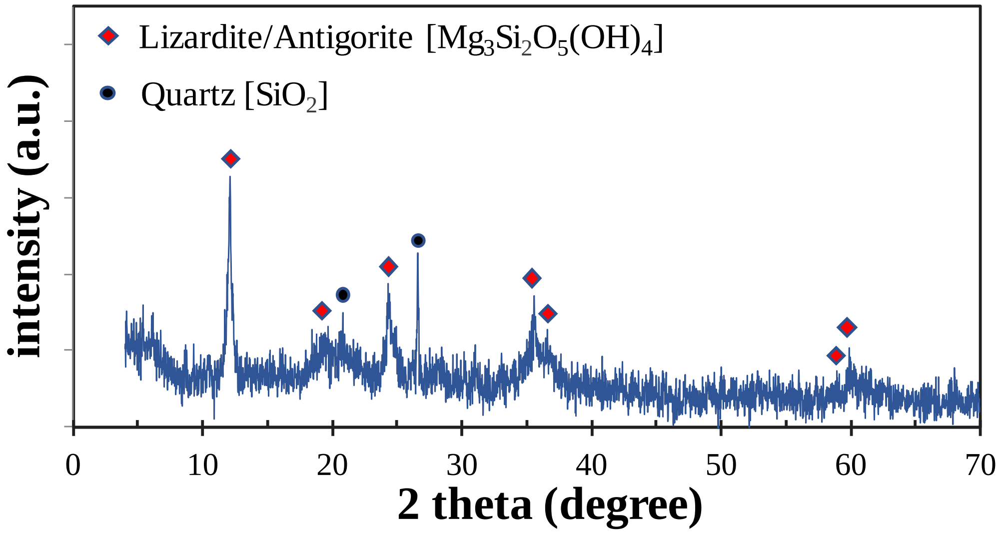


Fig. 1. XRD spectra of saprolitic nickel ore.

The coal for reductant was crushed using a roll mill, ground in a ball mill, and sieved to obtain a grain size of less than 65# (-210 µm). The coal was then dried at 135 °C for 24 h to remove the moisture contained in the coal. The results of proximate and ultimate analysis of coal are shown in Table 2 and Table 3, respectively. The CaO (p.a), H_3BO_3 (p.a) and CaF_2 (technical grade) additives were ground and sieved so that a size fraction of less than -80# (-177 µm) was obtained. The chemical composition of CaF_2 which was analyzed by XRF is listed in Table 4. These additives / fluxes were dried in an oven at 135 °C for 24 h.

Table 2. Coal proximate analysis (air dry basis).

Inherent moisture	*Ash*	*Volatile Matter*	*Fix Carbon*
1.39%	17.47%	35.21%	45.39%

Table 3. Coal ultimate analysis (air dry basis).

C	S	H	O
64.13%	0.92%	3.94%	12.3%

Table 4. Chemical composition of CaF_2 technical grade.

CaF_2	Fe_2O_3	Al_2O_3	SiO_2
97.21%	0.93%	0.66%	1.2%

The saprolitic nickel laterite ore was mixed with coal and additives and formed into briquettes using hydraulic press machine at a load of 5 tons. Three mixtures of briquette were prepared: briquettes A, B and C. Briquette A consisted of 3-gram nickel ore as basis with the addition of 10% coal and 10% CaO of the ore weight. Briquette B was 3-gram nickel ore as basis mixed with 10% coal, 10% CaO, and 10% CaF_2. Briquette C was prepared by mixing 3-gram nickel ore with the addition of 10% coal, 10% CaO, 10% CaF_2, and 10% H_3BO_3. Details of the mixture between nickel ore, coal, and additives are provided in Table 5.

Table 5. Different nickel ore, coal, and additives mixtures used in the present study.

Briquette	Ore (g)	Coal (g)	CaO (g)	CaF_2 (g)	H_3BO_3 (g)
A	3	0.3	0.3	-	-
B	3	0.3	0.3	0.3	-
C	3	0.3	0.3	0.3	0.3

The diameter of the briquettes was 15 mm and the height was in the range of 1.04 – 1.27 mm. The experiments for the reduction of nickel ore by coal with the addition of fluxes were conducted in a horizontal tube furnace under argon gas atmosphere with the flowrate of 1 L/min at 900, 1000, 1100, 1150, 1200 and 1250 °C for 2 h. Three briquettes from each mixture were placed into an alumina crucible. After the target temperature of the tube furnace had been achieved, the argon was flown for 3 minutes prior to the insertion of the briquettes into the furnace in order to remove the air inside the tube so that the inert conditions can be achieved. After the reduction process was completed, the briquettes were removed from the tube furnace and cooled to room temperature in a desiccator to avoid the re-oxidation of the formed ferronickel. The reduced briquette was then weighed, documented, and analyzed by optical microscope and SEM-EDS. The particle sizes were analyzed by ImageJ software, i.e. an open source image processing program [25].

3. Results and Discussion

3.1 Thermodynamics analysis

The influences of CaO, CaF_2 and H_3BO_3 additives on the reduction of saprolitic nickel laterite ore with chemical composition given in Table 1, was evaluated using the Equilib module in the FactSage 7.1 thermodynamic software using FToxid and FTmisc databases. The amount of carbon addition was 5% and the amount of additives were based on the mixtures in briquettes A, B and C. The output of the calculations were the compositions and the amounts of the gas, slag, solid oxides, and metal phases at equilibrium. At temperature ≤ 1250 °C, the ferronickel smelting slag is in solid state, therefore, it is interesting to observe the influence of additive on the formation of the liquid phase in the slag. During the reduction of the briquette, the formed ferronickel particles will be easier to migrate, merge, agglomerate, and grow in the molten phase compared to in the solid phase.

Fig. 2 shows the calculated liquid proportion in the slag in temperature range of 900 - 1250 °C for briquettes A, B and C. By adding 10% CaO as additive in dry saprolitic nickel laterite ore (briquette A), liquid slag was formed at 1250 °C with the proportion of 15%. No liquid slag was present at 900 – 1200 °C. The solid slag consisted of clinopyroxene and olivine. In briquette B, on the addition of 10% CaO and 10% CaF_2, liquid slag started to exist at 1000 °C with the amount of 4.5%. The proportion of liquid slag increased as temperature raised and achieved 66.7% at 1250 °C. The solid mineral at 1250 °C was olivine. The influence of boric acid (H_3BO_3) was studied in the briquette C where the additives consisted of 10% CaO, 10% CaF_2 and 10% H_3BO_3. The tendency of briquette C was similar with briquette B where the liquid slag was formed at 1000 °C. In term of the amount of liquid slag, briquette C had higher proportion of liquid slag than briquette B in temperature range of 1000 - 1150 °C but with lower proportion of liquid slag than briquette B at $T \geq 1200$ °C. In general, the additions of CaF_2 and H_3BO_3 into the slag increased the amount of liquid phase in the slag than the addition of CaO alone. The high proportion of the liquid phase in the slag will assist the agglomeration of the metal particles.

The thermodynamics calculation revealed that the nickel recoveries in briquettes A, B and C were 100 % due to the excess of carbon for reductant. However, the iron recovery and the content of nickel in ferronickel in each briquette were different as shown in Fig. 3. The figure shows that the ratio of Ni/Fe in the ferronickel was higher at lower temperature since the nickel oxide was easier to be reduced than iron oxide at lower temperature. The addition of H_3BO_3 increased the Ni/Fe ratio in the ferronickel which means that higher nickel content in ferronickel can be achieved and the reduction of iron oxide can be suppressed.

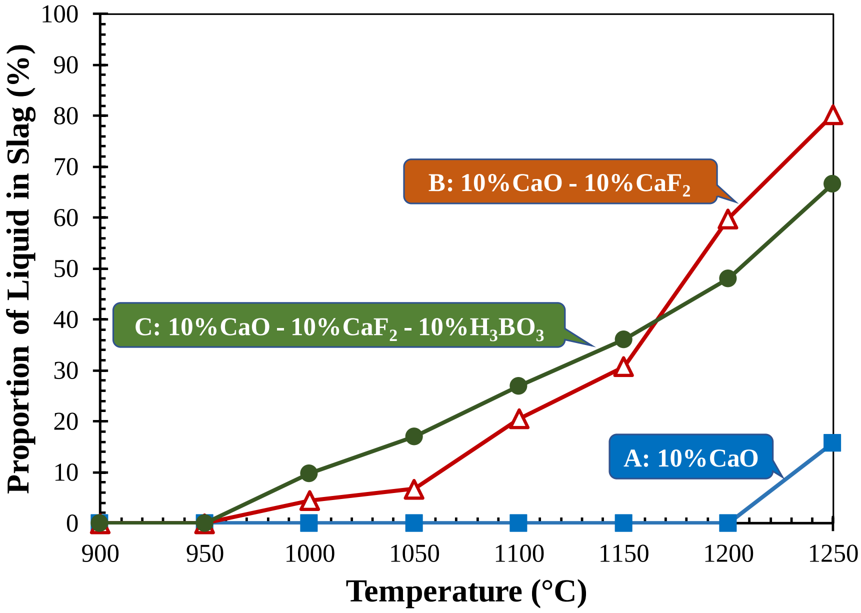


Fig. 2. Proportion of liquid in the slag as a function of temperature and flux at 5% carbon addition based on FactSage calculation.

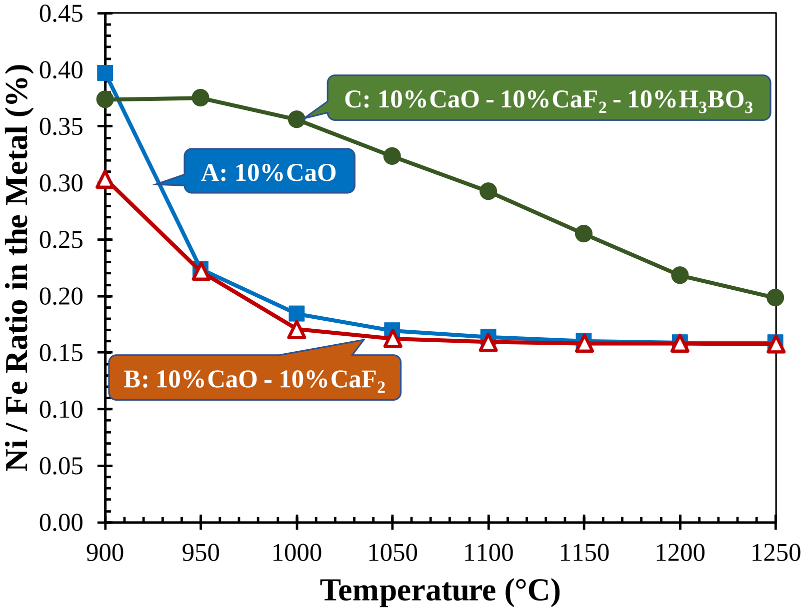


Fig. 3. Ratio of Ni/Fe in the ferronickel as a function of temperature and flux at 5% carbon addition based on FactSage calculation.

The stability of boric acid as function of temperature based on FactSage calculation is depicted in Fig. 4. The boric acid (H_3BO_3) was dissociated into $HBO_{3(s)}$ and $H_2O_{(g)}$ at temperature higher than 100 °C. In the range of 300 and 450 °C, the $B_2O_{3(s)}$, $(HBO_2)_{3(g)}$ and $H_3BO_{3(g)}$ were formed. At T > 450 °C, $B_2O_{3(s)}$ solid was converted into $B_2O_{3(l)}$ since the melting point of B_2O_3 is 450 °C. The present of B_2O_3 liquid in the slag lower the melting point and viscosity of the slag which facilitates the agglomeration and the growth of metal particles.

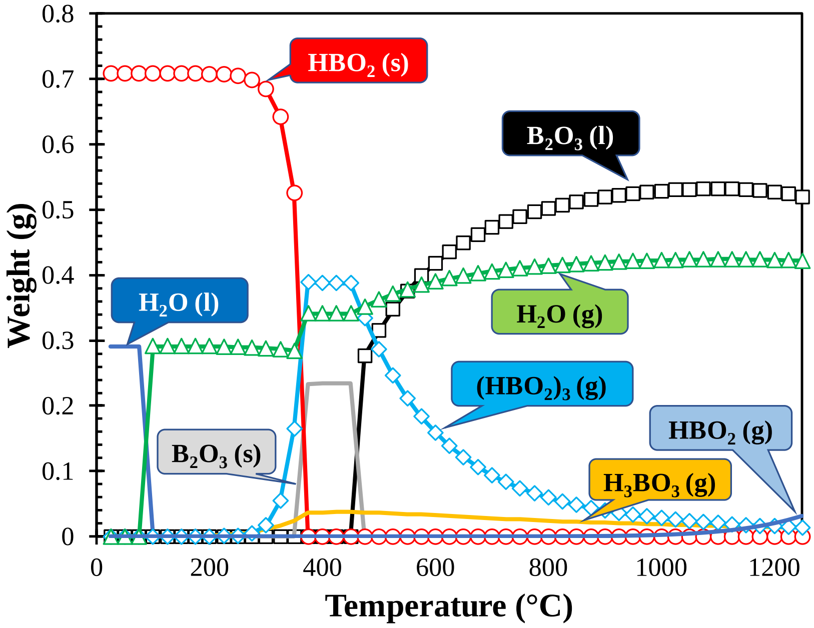


Fig. 4. The stability of H_3BO_3.

3.2 Visual observation and weight loss

The photographs of the briquettes after the reduction are shown in Fig 5. For briquette A with the addition of 10% CaO, the deformation of briquette was observed at 1250 °C. At 900 to 1200 °C, the briquettes appeared to be in solid state. These results are in line with the thermodynamics calculation (Fig. 2), where the proportion of liquid phase in the slag was 15% at 1250 °C and no liquid slag was formed at 900 - 1200 °C. On the other hand, the briquettes B and C were already deformed at 1200 and 1150 °C, respectively. These phenomena are also in agreement with the thermodynamics calculations shown in Fig. 2 where the slag liquid was readily formed at 1000 °C.

After the reduction, weight loss of the briquette was observed as shown in Fig. 6. It was due to the evaporation of bound water, release of volatile matter and inherent moisture in the coal, and reduction of oxygen in metals oxide (iron oxide, nickel oxide, chromium oxide) by carbon in coal forming CO or CO_2 gas. The percentage of weight loss from each experiment is shown in Fig. 6. Percentage of weight loss increased as the temperature raised due to the reduction of oxygen in the nickel ore by carbon in coal to produce ferronickel. Accordingly, the amount of ferronickel formation increased as the temperature increased. The weight loss based on thermodynamic software FactSage 7.1 calculations were 17.83 – 19.54% for briquette A, 17.83 – 19.54% for briquette B, and 21.13 – 25.56% for briquette C. The highest weight loss was associated with briquette C which was due to the dissociation of H_3BO_3 into $H_2O_{(g)}$, $B_2O_{3(l)}$, $(HBO_2)_{3(g)}$ and $H_3BO_{3(g)}$. The experimental results had similar trends with the thermodynamic calculation results.

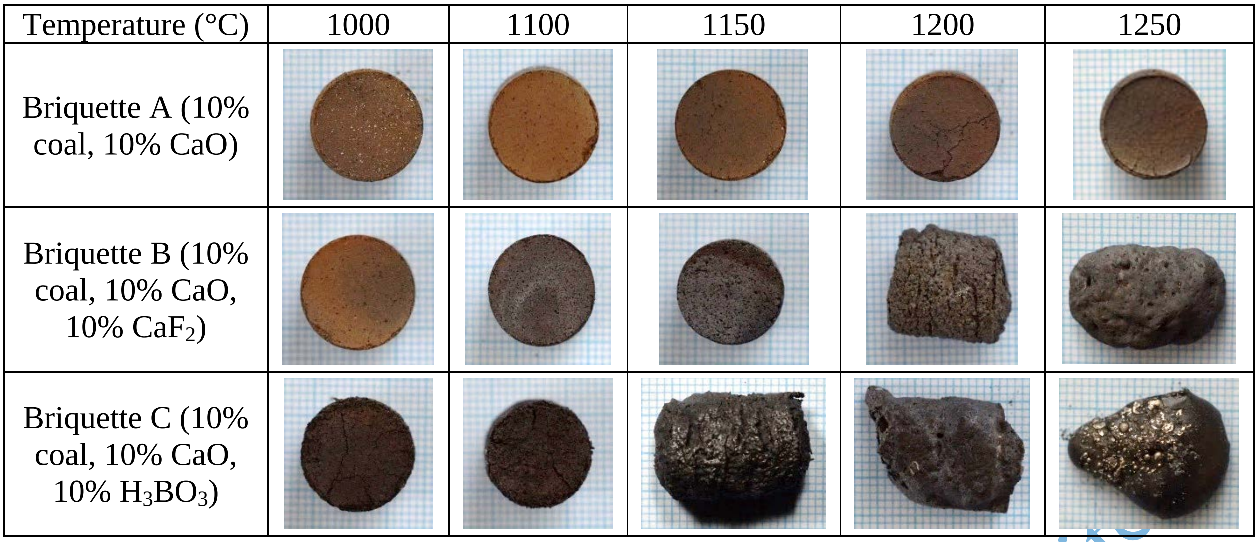


Fig. 5. Photograph of briquettes after the reduction process.

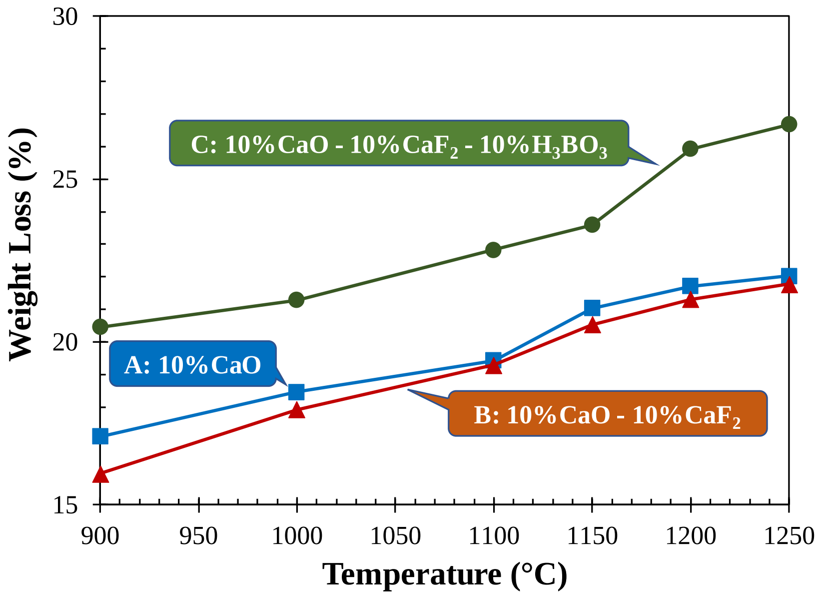


Fig. 6. Weight loss of briquettes after the reduction process.

3.3. Ferronickel Particles Size

Optical microscope observations on the surface of briquettes A, B, and C at 1100 - 1250 °C are shown in Fig 7. For briquette A, metallographic preparation technique can only be applied to the reduced briquette from experiments at 1250 °C. At temperature ≤ 1200 °C, the samples were weak and difficult to be metallographically prepared for optical microscope observation. In general, the ferronickel size in briquette C had larger particles, followed by briquette B and A. To determine the ferronickel particle sizes, five photographs from each briquette were taken and the number of particles sizes were quantified using ImageJ software. Because the particles have irregular shapes, the particle size was calculated based on the diameter of the spherical shape using the surface area data from ImageJ software. The result of the ferronickel particle size quantification at 1250 °C is depicted in Fig. 8 which shows the relationship between the number of particles and their sizes. All briquettes with large number of ferronickel particles had particle sizes of less than 10 µm. The maximum size of ferronickel particles in briquettes A, B and C at reduction temperature of 1250 °C were in the range of 20 – 30 µm, 100-200 µm and 300 - 400 µm, respectively.

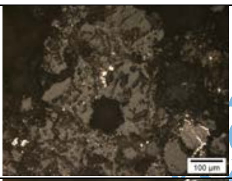
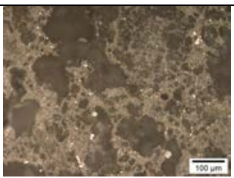
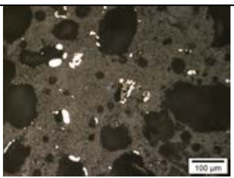
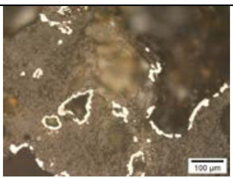
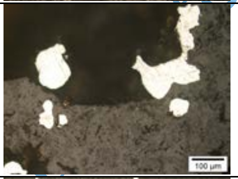
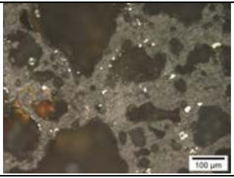
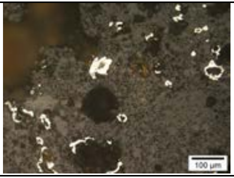
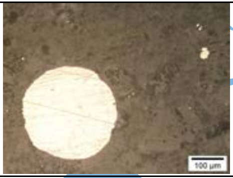
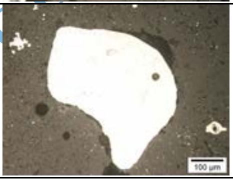
Temperature (°C)	1100	1150	1200	1250
Briquette A	n.a.	n.a.	n.a.	
Briquette B				
Briquette C				

Fig. 7. Optical microscope observation on the briquettes after the reduction process with 200X magnification.

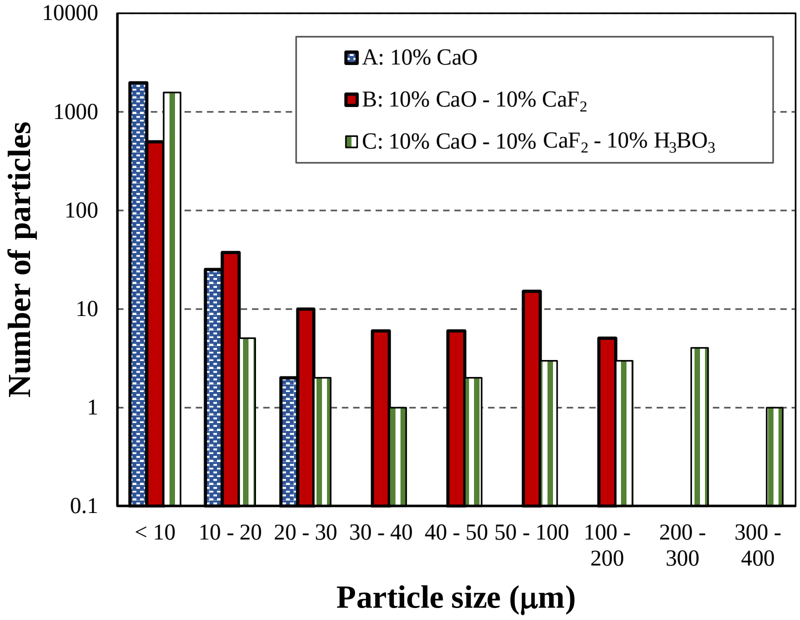


Fig. 8. Relationship between number of particles and particle size at 1250 °C.

The particle size distributions of ferronickel in briquette B and briquette C at various reduction temperatures are summarized in Fig 9 and Fig. 10, respectively. Increasing the temperature shifted the particle size distribution toward the larger sizes. The number of particles size of less than 10 µm tend to decrease with increasing temperature. At 1100 °C, the maximum particle size for briquettes B and C was 20 – 30 µm. At temperatures 1150 °C and higher, the ferronickel particle sizes continued to grow and in general the particle sizes in briquette C were larger than briquette B. In terms of thermodynamics, increasing the reduction temperature causes the Gibbs free energy value to be more negative. Gibbs free energy which has a more negative value indicates more spontaneous reduction reaction. In terms of reaction kinetics, rising the temperature cause the reaction to occur faster. Furthermore, higher temperature provided more liquid phase in the slag which makes the ferronickel particles move and agglomerate more easily to form larger particles.

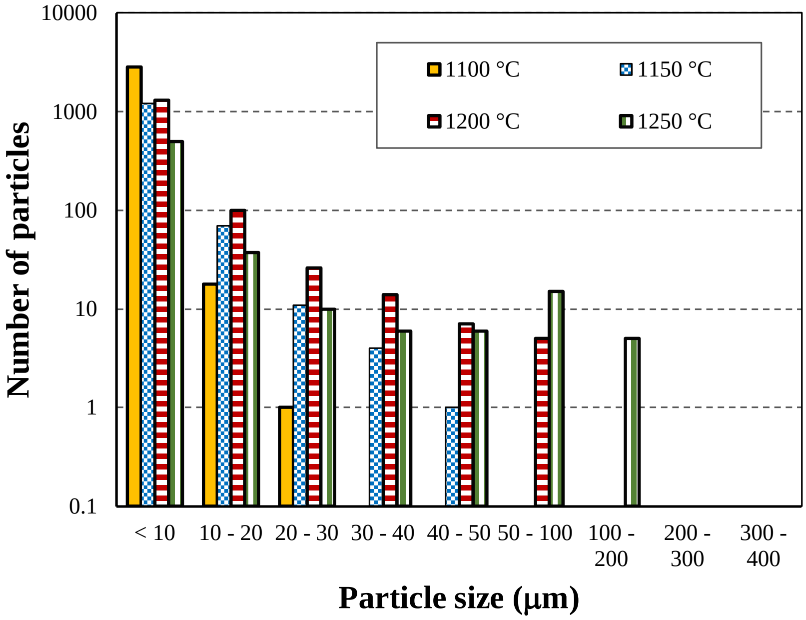


Fig. 9. Relationship between number of particles and particle size at 1250 °C for briquette B (10% CaO – 10% CaF_2).

The maximum sizes of ferronickel particles in briquettes A, B, and C at 900 – 1250 °C can be seen in Fig. 11. When the reduction temperature was raised from 1100 to 1250 °C, maximum particle size increased in briquettes B and C. Drastic increase in the maximum size of ferronickel occurred between 1200 and 1250 °C for briquette B, which is 115% growth (from 76 µm to 165 µm). On the other hand, drastic increase in maximum particle size in briquette C occurred between 1150 and 1200 °C, which is 426% growth (from 55 µm to 290 µm). From a temperature of 1200 to 1250 °C, ferronickel particle size in briquette C increased by 39% (from 290 µm to 376 µm).

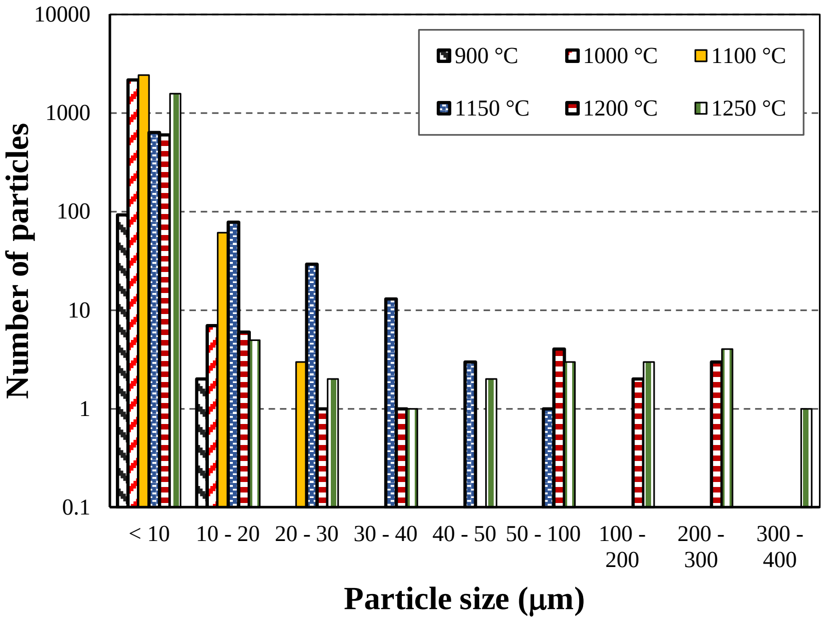


Fig. 10. Relationship between number of particles and particle size at 1250 °C for briquette C ($10\%\ CaO - 10\%\ CaF_2 - 10\%\ H_3BO_3$).

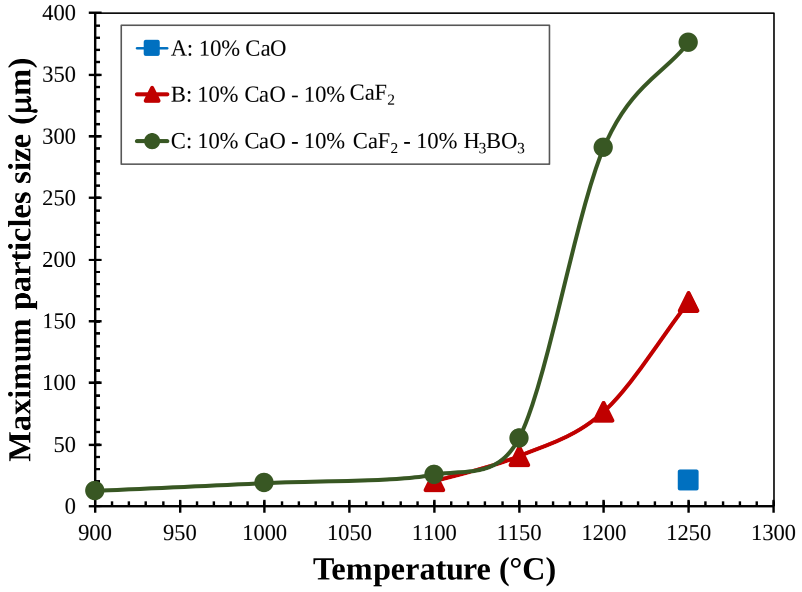


Fig. 11. Maximum particles size as function of temperature and flux.

Upon heating of saprolitic nickel laterite ore, the mineral serpentine in the ore decomposes. At 750 °C, serpentine dissociates into olivine and silica according to the following reaction [8]:

$$2\ (Mg, Fe, Ni)_3Si_2O_5(OH)_4\ (s) = 3\ (Mg, Fe, Ni)_2SiO_4\ (s) + SiO_2\ (s) + 4H_2O\ (g)$$

Olivine is a phase that is difficult to reduce and can inhibit the reduction of nickel and iron oxides. The addition of CaO to the saprolitic nickel laterite ore can reduce the melting point of slag. The addition of CaO can also exchange the NiO component in the olivine phase leading to easier reduction of NiO in the ore.

The CaF_2 can reduce the melting point of slag during the reduction of saprolitic nickel laterite ore. The SiO_2 / (SiO_2 + MgO) ratio of the ore used in the present study was 0.67 (see Table 1). At this composition, thermodynamic calculation indicated melting point of slag at around 1600 °C. When 10% CaF_2 was added, the thermodynamic calculation showed a decrease in slag melting point to around 1200 °C. The addition of 10% CaF_2 in briquette B allowed the formation of a liquid phase at lower temperatures making it possible to produce larger ferronickel particles.

Simultaneous addition of CaO, CaF_2, and H_3BO_3 to the saprolitic nickel laterite ore resulted in further lowering of the slag melting point. During heating, boric acid will undergo a gradual decomposition. This decomposition of H_3BO_3 will finally produce boron trioxide (B_2O_3). Boron trioxide is an oxide compound that has a low melting temperature, which is around 450 °C. The addition of boric acid to briquette C promoted the formation of a liquid phase at a lower temperature compared to those of other two briquettes. The higher the temperature, the more liquid phases formed.

3.4. Percentage of Metal Phase and Chemical Composition of Metals

The percentages of metal phases of ferronickel particles in briquettes A, B, and C at various temperatures are shown in Fig. 12. The determination of the metal phase percentage was carried out using ImageJ software [25]. The metal phase percentage was calculated by dividing the surface area of metal phase with total surface area of the sample from the optical microscope images. The metal phase percentages in briquette B and briquette C increased with increasing reduction temperatures. The highest metal phase percentage was produced from the reduction of briquette C at a temperature of 1250 °C, i.e. 21.46 area %. The trend of the percentage of metal phases are similar with the maximum particle sizes shown in Fig. 11.

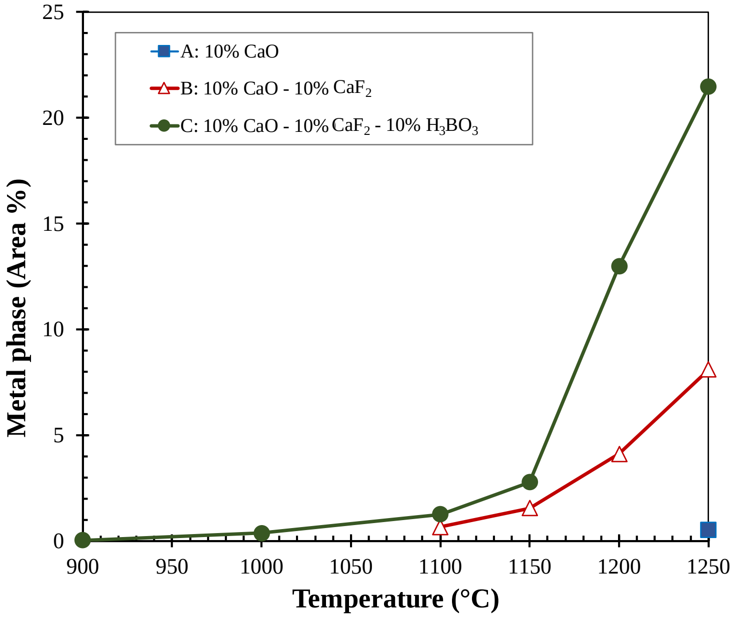
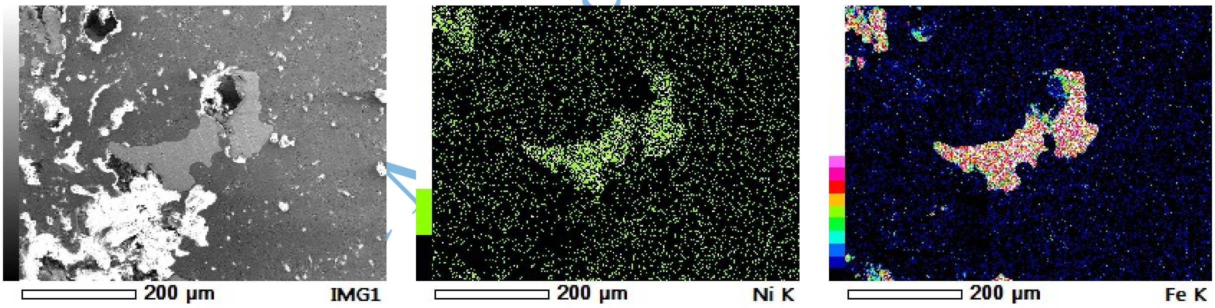


Fig. 12. Metals phase as function of temperature and flux.

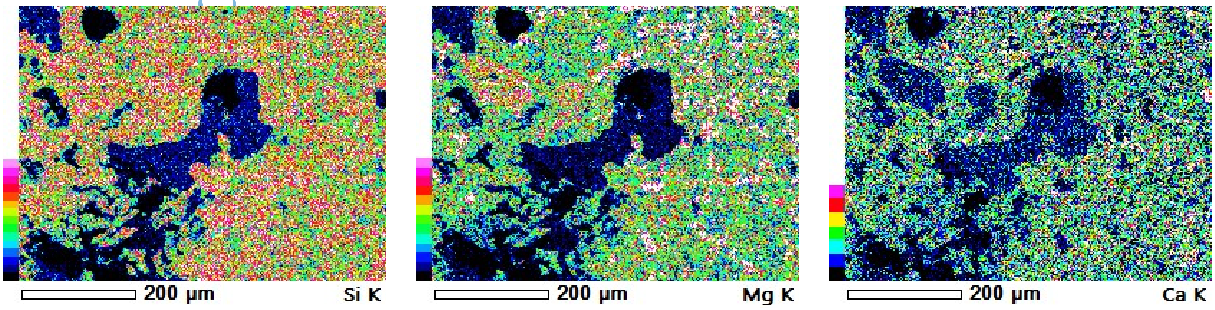
Liquid phase formation in slag at various temperatures and the mechanism of agglomeration of ferronickel particles have been described by Elliot et al. [8] and Rao et al. [12]. At the time of reduction, the presence of a liquid phase in slag causes sintering of ferronickel particles through capillary forces between adjacent particles, the liquid phase in slag becomes a facility for smaller ferronickel particles to agglomerate into larger particles. In general, increasing the reduction temperature can produce more liquid phase as indicated by the thermodynamic calculations. The more liquid phase will encourage the agglomeration of particles to larger sizes and can increase percentage of the metal phase.

The results of SEM-EDS observations on briquettes B and C after the reduction at 1250 °C are shown in Fig. 13 and Fig. 14, respectively. It can be observed that iron and nickel were accumulated in the ferronickel metals. On the other hand, silicon, magnesium, and calcium were contained in the slag. The chemical compositions of ferronickel are given in Table 6. The carbon content in the briquette is not reported due to the inaccurate carbon determination in the present measurement. In general, the nickel content in briquette C was higher than in briquette B. The experimental data appears to confirm the thermodynamic calculation result, i.e. the addition of H_3BO_3 increases the nickel content in the ferronickel and suppresses the reduction of iron oxide.

The experimental results show the feasibility of producing ferronickel particles with significant particle size at low temperatures through the modification of the slag by the addition of different additives. The large ferronickel particle size leads to better separation of metals from the unreduced components. The present study demonstrates the possibility of processing of lateritic nickel ore at lower temperatures, hence lower energy consumption and lower operational cost.

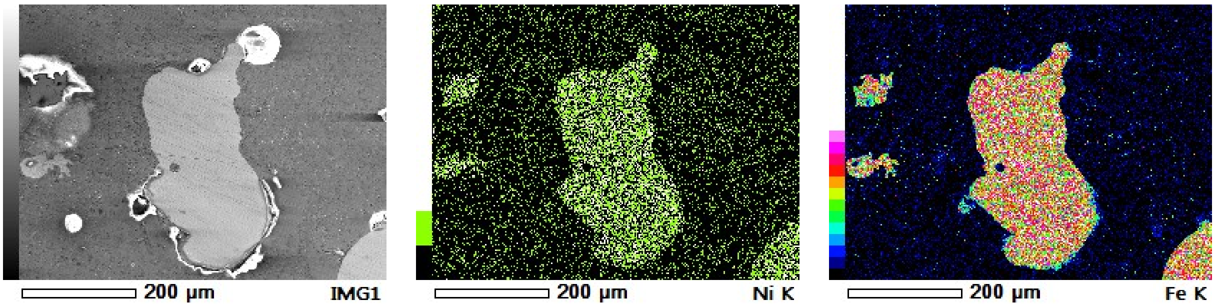


(a) SEM image (b) Nickel (c) Iron

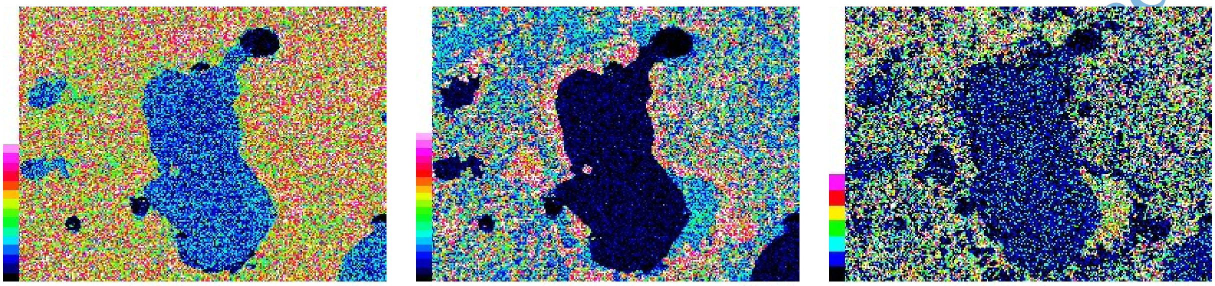


(d) Silicon (e) Magnesium (f) Calcium

Fig. 13. SEM image and EDS elemental mapping of briquette B after reduction at 1250 °C, at 200X magnification.



(a) SEM image (b) Nickel (c) Iron



(d) Silicon (e) Magnesium (f) Calcium

Fig. 14. SEM image and EDS elemental mapping of briquette C after reduction at 1250 °C, at 200X magnification

Table 6. Chemical composition of ferronickel determined by SEM-EDS.

Briquette	%Ni	%Fe	%Si	%Cr
B	7.91	88.52	0.71	2.76
C	8.59	85.65	3.52	2.25

The CaF_2 was considered not environmentally friendly, therefore the slag containing CaF_2 shall be treated and handled carefully. Furthermore, the volume of produced slag will be higher due to the presence of B_2O_3 with has low density. Principally, this slag can be utilized as raw material for magnesium production. It was reported that magnesium metal can be recovered from ferronickel slag with a higher recovery degree by addition of calcium oxide [26]. The influence of CaF_2 and B_2O_3 on the recovery of magnesium from the ferronickel slag should be further studied.

Conclusions

Low grade saprolitic nickel laterite ore was reduced by 10% coal and additives. The addition of 10% CaO had little influence on the formation of ferronickel particles where the maximum particle size was 20 µm at 1250 °C. The addition of 10% CaO and 10% CaF_2 into low grade saprolitic nickel laterite ore increased the ferronickel particle size up to 165 µm at 1250 °C due to the increase of liquid slag phase which facilitated the metal migration and agglomeration into larger ferronickel particles. The addition of 10% CaO, 10% CaF_2, and 10%

of H_3BO_3 resulted ferronickel particle size of 376 µm at 1250 °C. The presence of boric trioxide (B_2O_3) reduced further the melting temperature of the slag. The presence of liquid phase in the slag and the larger ferronickel size lead to better separation of the ferronickel particles from the impurities.

Acknowledgments

The authors would like to thank to the Program of Research, Community Service, and Innovation of the Institut Teknologi Bandung (P3MI–ITB) for funding this research.

References

[1] http://www.insg.org/

[2] USGS, Mineral Commodity Summaries, February 2019 (https://www.usgs.gov)

[3] C.T. Harris, J.G. Peacey, and C.A. Pickles, Thermal upgrading of nickeliferous laterites – A review, *Proc. Pyromet. Nickel and Cobalt 2019*, Ontario, p. 51.

[4] J. Dong, Y. Wei, S. Zhou, B. Li, Y. Yang, and A. McLean, The effect of additives on Ni, Fe and Co from nickel laterite ores, *JOM*, 70 (2018), p. 2365.

[5] G. Li, T. Shi, M. Rao, T. Jiang, and Y. Zhang, Beneficiation of nickeliferous laterite by reduction roasting in the presence of sodium sulfate, *Minerals Engineering*, 32 (2012), p. 19.

[6] D. Q. Zhu, Y. Cui, K. Vining, S. Hapugoda, J. Douglas, J. Pan, and G.L. Zheng, Upgrading low nickel content laterite ores using selective reduction followed by magnetic separation, *International Journal of Mineral Processing*, 106 (2012), p. 1.

[7] D. Zhu, L. Pan, Z. Guo, J. Pan, F. Zhang, Utilization of limonitic nickel laterite to produce ferronickel concentrate by the selective reduction-magnetic separation process, *Adv. Powder Tech.*, 30 (2019), p. 451.

[8] R. Elliott, C.A. Pickles, and J. Peacey, Ferronickel particle formation during the carbothermic reduction of a limonitic laterite ore, *Miner. Eng.*, 100 (2017), p. 166.

[9] M. Jiang, T. Sun, Z. Liu, J. Kou, N. Liu, and S. Zhang, Mechanism of sodium sulfate in promoting selective reduction of nickel laterite ore during reduction roasting process, *Int. J. Miner. Process.*, 123 (2013), p. 32.

[10] J. Lu, S. Liu, J. Shangguan, W. Du, F. Pan and S. Yang, The effect of sodium sulphate on the hydrogen reduction process of nickel laterite ore, *Miner. Eng.*, 49 (2013), p. 154.

[11] C.T. Harris, J.G. Peacey, and C.A. Pickles, Selective sulphidation and flotation of nickel from a nickeliferous laterite ore, *Miner. Eng.*, 54 (2013), p. 21.

[12] M. Rao, G. Li, X. Zhang, J. Luo, Z. Peng, and T. Jiang, Reductive roasting of nickel laterite ore with sodium sulphate form Fe-Ni production. Part II: Phase transformation and grain growth, *Separat. Sci. Tech.*, (2016), p. 1520.

[13] X. Wang, T. Sun, C. Chen, and J. Kou, Effects of Na_2SO_4 on iron and nickel reduction in a high-iron and low-nickel laterite ore, *Int. J. Miner. Metall. Mater.*, 25 (2018), p. 383.

[14] G. Chen, J. Shiau, S. Liu, and W. Hwang, Optimal combination of calcination and reduction conditions as well as Na_2SO_4 additive for carbothermic reduction of limonite ore, *Mater. Trans.*, 57 (2016), p. 1560.

[15] S. Zhou, Y. Wei, B. Li, H. Wang, B. Ma, and C. Wang, Chloridization and reduction roasting of high-magnesium low-nickel oxide ore followed by magnetic separation to enrich ferronickel concentrate, *Metall. Mater. Trans. B*, 47B (2016), p. 145.

[16] Z. Wang, M. Chu, Z. Liu, H. Wang, W. Zhao, and L. Gao, Preparing ferro-nickel alloy from low grade laterite nickel ore based on metallized reduction – magnetic separation, *Metals*, 7 (2017), p. 1.

[17] T. Watanabe, S. Ono, H. Arai, and T. Matsumori, Direct reduction of garnierite ore for production of ferro-nickel with a rotary kiln at Nippon Yakin Kogyo Co., Ltd., Oheyama Works, *Int. J. Miner. Proc.*, 19 (1987), p. 173.

[18] Y. Kobayashi, H. Todoroki, and H. Tsuji, Melting behavior of siliceous nickel ore in a rotary kiln to produce ferronickel alloys, ISIJ Int., 51 (2011), p. 35.

[19] H. Tsuji, Behavior of reduction and growth of metal in smelting of saprolite Ni-ore in a rotary kiln for production of ferro-nickel alloy, ISIJ Int., 52 (2012), p. 1000.

[20] A.E.M. Warner, C.M. Diaz, A.D. Dalvi, P.C. Mackey, and A. V. Tarasov, JOM world nonferrous smelter survey, Part III: Nickel: Laterite, JOM, April (2006), p. 11.

[21] B. Li, H. Wang, and Y. Wei, The reduction of nickel from low-grade nickel laterite ore using a solid-state deoxidation method, *Miner. Eng.*, 24 (2011), p. 1556.

[22] X. Lv, L. Wang, Z. You, J. Dang, X. Lv, G. Qiu, and C. Bai, Preparation of ferronickel from nickel laterite ore via semi-molten reduction followed by magnetic separation, *Extraction 2018*, TMS (2018), p. 913.

[23] X. Ma, Z. Cui, and B. Zhao, Efficient utilization of nickel laterite to produce master alloy, *JOM*, 68 (2016), p. 3006.

[24] M.H. Morcali, L.T. Khajavi, and D.B. Dreisinger, Extraction of nickel and cobalt from nickeliferous limonitic laterite ore using borax containing slags, *Int. J. Miner. Proc.*, 167 (2017), p. 27.

[25] https://imagej.nih.gov/ij/

[26] X. Zhang, F. Gu, Z. Peng, L. Wang, H. Tang, M. Rao, Y. Zhang, G. Li, T. Jiang, and Y. Wang, Recovering magnesium from ferronickel slag by vacuum reduction: thermodynamic analysis and experimental verification, ACS Omega, 4 (2019), p. 16062.