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Study on Structure and Reducibility of Iron Ore Sinter Containing Basic Oxygen Furnace Slag

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Abstract

Basic Oxygen Furnace (BOF) slag is a byproduct of the steelmaking process. The amount of slag produced in steel production lines is substantial, not only in Vietnam but also worldwide. Reusing this large amount of slag is an essential requirement, and this study assesses a solution for reusing BOF slag by incorporating it into the sintering process. Experiments were conducted using iron ore, coal, and slag as raw materials for the sintering process using a suction fan apparatus. The sintered product was analyzed for its microstructure, reduction degree, and softening properties. The results indicate that the reducibility of the sinter containing the slag is equivalent to that without slag. The yield of sintered ore decreases by approximately 1% compared to when the slag from steelmaking is used as a flux. The results show that the yield of sintered products meeting standards (size and strength) has values ranging from 59.5 to 71.9%. Besides, the highest reduction degree was achieved with a 24% slag at a basicity of 1.6 and a temperature of 1100 °C. In contrast, the reduction degree of the sintered ore was lower at 1000 °C. The initial softening temperature was determined to be 1200 °C. The microstructure of the sintered ore revealed the formation of a liquid bonding phase. It can be concluded that the main bonding phase is calcium ferrite. The liquid phase showed an even distribution of calcium, silicon, oxygen, and iron elements.

Keyword: BOF slag, iron sinter, reducibility, microstructure

1. Introduction

Basic oxygen furnace (BOF) slag is a byproduct of the steelmaking process in general. The slag formation process aims to remove impurities such as S, P, etc., to meet the required mechanical properties of the steel product. The amount of slag generated during this process accounts for about 15% of the weight of the steel product, typically, to obtain 1 ton of steel from the blast furnace, about 100 - 150 kg of slag is generated [1]. The main chemical components of blast furnace slag are CaO, FeO, and SiO_2, along with small amounts of other oxides. Indeed, recycling of large amount of BOF slag in ironmaking needs to be considered due to the suitability and value of slag components.

During the steel production process, some of the iron (Fe) in the molten iron is oxidized; the FeO amount in the slag is relatively high [2-4]. The BOF slag has a high amount of CaO, along with many other elements like Fe, Si, Mn, ... Using slag in the sintering process for iron ore in a blast furnace can make use of the CaO present in the slag to create basicity for the sintering process. This, in turn, reduces the amount of flux needed to be added to the sinter mix. Additionally, SiO_2 in the slag helps in controlling basicity and increasing the silicon amount in the iron ore to produce high-silicon pig iron. Moreover, introducing slag into the sintering process can help recover some metal elements like Fe, Mn, and others. On the other hand, this method indirectly transforms blast furnace slag into a reusable and economically efficient material for the iron-making process.

The research on using BOF slag as a fluxing agent in the sintering process for iron ore has not received much attention. Particularly in Vietnam, there has been no study to evaluate the potential reusability of steel slag when introduced into the iron ore sintering process. This research focuses on assessing the feasibility of using steel slag as part of the fluxing material for the iron ore sintering process. Specifically, sinter mixes containing slag are produced in laboratory equipment with properties similar to sinter mixes produced in steel plants in Vietnam today. Important properties such as sinter quality, reducibility, blending ratios, etc., need to be evaluated. Consequently, sintered product containing steel slag is examined for its potential use in the high blast furnace for iron smelting.

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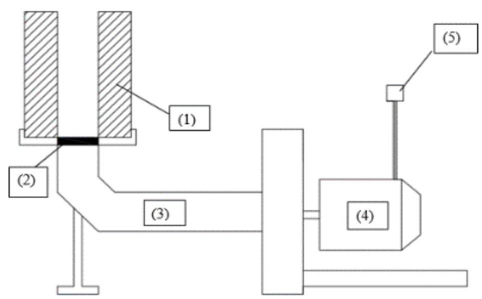
2. Experimental Procedure

The sintered product from iron ore is created using a suction pipe-type sintering apparatus (Fig. 1). The primary raw material for sintering is Cao Bang iron ore, and its composition is listed in Table 1. Blast furnace slag is introduced into the sintering process as an initial raw material, where it serves as part of the fluxing material and helps in regulating the appropriate basicity. Before being incorporated into the sintering process, the slag is finely ground and mixed with fluxes and iron ore. The particle size of the slag is less than 3 mm, and its composition is detailed in Table 2. Limestone, with a CaO amount exceeding 90%, is mixed with slag in various proportions to adjust the desired basicity. The particle size of limestone powder also meets the requirement of being less than 3 mm. Finally, coal is the last component in the blend of initial raw materials. Coal is added to the mix at 10%. The composition of coal is as described in Table 3, and the particle size of coal is less than 3 mm.



1.Furnace 2. Hearth layer 3. Pipe 4. Draught Fan 5. Control system

Fig. 1. Sintering process system apparatus.

Table 1. Iron ore mineral composition (%).

S	P	Mn	Pb	SiO_2	CaO
0.130	0.001	0.077	0.065	3.440	0.444
P_2O_5	**ZnO**	**MnO**	**CuO**	**Fe_2O_3**	**Moiture**
0.002	0.019	0.099	0.103	79.467	15.000

Table 2. BOF slag mineral composition (%).

SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	P_2O_5	MnO
9.560	1.180	55.480	5.320	14.970	1.260	5.660

Table 3. Coke composition (%).

C_{cd}	W	V	S	Ash	Al_2O_3	SiO_2
81.440	4.740	1.320	0.570	11.93	3.814	6.472

Table 4. Weight of initial raw material.

Sample	B	Slag amount (%)	Weight of initial raw material (g)
M1	1.2	24	2035
M2		43	2031
M3		55	2027
M4		64	2032
M5	1.4	24	2034
M6		43	2029
M7		55	2025
M8		64	2021
M9	1.6	24	2035
M10		43	2035
M11		55	2040
M12		64	2030
M13		0	2050

In the experimental process, the blending of slag and limestone is given at varying ratios. The amount of slag introduced is meant to replace a portion of the fluxing material at corresponding basicity values of 1.2, 1.4, and 1.6. Calculations for the mass of initial raw material with different basicity values are presented in Table 4.

The sintered product, obtained through the suction pipe-type sintering system, is subjected to morphological and elemental analysis using SEM equipment (JEOL JSM-IT200) and Energy Dispersive X-ray spectroscopy (Xplore 30), respectively. Additionally, the product, placed in a reduction environment, is introduced into a resistance furnace. The reduction degree of the sintered product is determined at temperatures of 1000 °C and 1100 °C. The holding time at these temperatures is 30 minutes. The heating process is carried out with a heating rate of 15 °C per minute. Subsequently, the sintered product is cooled along with the furnace and weighed to measure the mass after the reduction process in the crucible. The reduction degree is calculated using following equation, where H (%) represents the reducibility of the sinter.

$$\mathrm{H} = \frac{\mathrm{m}\ (\text{amount of reduced oxygen})}{m_0\ (\text{total oxygen amount of the sinter})} \times 100\ \% \tag{1}$$

The softening characteristics of sintered ore are particularly important. Softening behavior determines the position and shape of the softening zone when introducing the sintered product into the blast furnace. This study evaluates the softening properties of sintered ore containing steel slag. First, the sintered sample is placed in a heat-resistant crucible and positioned in a resistance furnace. Then, a 2 kg load is applied from above. The crucible containing the sample is heated, with the temperature increasing from room temperature to 1200 °C at a rate of 15 °C per minute. At a given temperature, the holding time is 15 minutes.

3. Result and Discussion

3.1 The Yield of Sinter with a BOF Slag-Containing Flux

The product of the iron ore sintering process must meet requirements for strength, sinter yield, reducibility, softening characteristics, etc., in order to be used as a raw material for the blast furnace and achieve high performance in liquid iron production [5]. Sintered ore is the product formed through the sintering process from raw materials that include iron ore, fluxes, and coal. Evaluating the yield of the sintered ore is crucial to assessing the usability of the raw materials (iron ore, fluxes, and coal). If the yield is low, it can adversely affect the ability to supply sufficient iron ore burden to the blast furnace. On the other hand, if the return fine ratio is too high, it can lead to low yield in the use of raw materials and energy during the sintering process. This, in turn, results in low overall productivity in the ironmaking process from iron ore. Therefore, evaluating the yield of sintered ore is of paramount importance.

In this study, the yield of sintered ore is determined by the ratio of the mass of the sintered product (meeting size and strength) to the total mass of the initial raw materials (including fluxes, slag, iron ore, and coal as listed in Table 4). The slag is introduced to replace a portion of the fluxing material in the sintering process at varying ratios. This study evaluates the influence of basicity and the proportion of the slag on the yield of sintered ore. The amount of sinter and standard sinter are listed in Table 5, B is the basicity value of the sinter. The values of yield at different basicity levels and steel slag proportions are plotted in Fig. 2. The results show that the sintered product meeting standards (size and strength) have values ranging from 59.5 to 71.9%, with the remainder being return fines; this means that the yield ranges from 59.5 to 71.9%. The sintering yield varies depending on basicity and the slag amount in the initial raw materials. The results indicate that the highest sinter yield is 71.9% at a basicity of 1.6 and a slag amount in the flux of 63%. When the flux contains 24% BOF slag and the basicity is 1.2, the yield is lowest at 59.5%, where the sintered product meets strength and size requirements.

Table 5. Yield of the sintering product.

Sample		Mass Sinter (g)	Standard mass sinter (g)
B = 1.2	M1	1607	1211
	M2	1495	1269
	M3	1518	1289
	M4	1499	1298
B = 1.4	M5	1530	1312
	M6	1560	1319
	M7	1530	1361
	M8	1590	1390
B= 1.6	M9	1928	1449
	M10	1904	1481
	M11	1933	1467
	M12	1877	1484

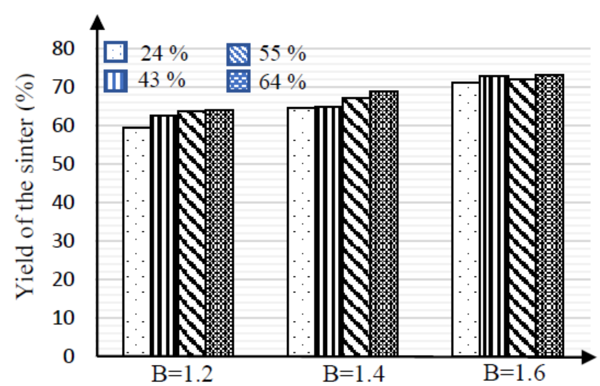


Fig. 2. Effect of basicity on yield of the sinter.

Basicity significantly affects the yield and strength of the sintered ore. The results show that increasing basicity leads to an increase in yield. Many authors have demonstrated the reasons why basicity affects the strength of sintered ore. Higuchi [6] showed that increasing basicity increases the formation of the calcium ferrite phase, which plays a role in binding iron ore particles during sintering. Increased basicity leads to more liquid phase formation due to a lower melting temperature [7]. A higher liquid phase formation creates favorable conditions for bonding between iron ore particles. This study indicates that increasing basicity enhances strength, which aligns with the result of increased strength. Additionally, the calcium ferrite phase is known for producing sintered ore with good reducibility. Thus, these results suggest that sintered ore with an appropriate slag amount at high basicity values is suitable for use as a raw material for the blast furnace.

Fig. 3 illustrates the impact of the steel slag amount introduced into the sintering process on the strength of sintered ore. For samples with the same basicity and varying steel slag amount, the results confirm that increasing the steel slag amount leads to a higher yield of the sintered ore. For a basicity level of B = 1.2, in the four samples, M1, M2, M3, and M4 with increasing slag amount, it is evident that higher steel slag amount in the flux results in greater yield; M4 has a yield of 63.9%, while M1 has a yield of 59.5%. With a basicity of B = 1.4, samples M5, M6, M7, and M8 exhibit yields ranging from 64.5 to 68.8%. For samples with basicity of B = 1.6, introducing different steel slag amounts results in slight increases in yield value, with an average yield of about 71.9%. These results are in agreement with those of Umadevi *et. al* [8]. When basicity is high, steel slag is considered to have less impact on the yield of the sintered ore. To assess the role of steel slag when introduced into sintering, the study evaluated the yield of sintered ore both with and without steel slag in the flux at a basicity level of 1.6. The yield of sintered ore without steel slag (100% limestone) at a basicity of 1.6 is 73.1%. Therefore, when introducing steel slag, the yield of sintered ore decreases by approximately 1% compared to when steel slag from steelmaking is used as a flux.

Considering the impact of steel slag amount on strength, the results show that increasing the amount of steel slag does not significantly affect drop strength. The yield is evaluated based on the ratio between the mass of the sintered ore after sintering (before drop strength testing) and the mass of the sintered ore after the strength test. The mass of sintered ore before and after the strength test with different steel slag amounts and basicity levels is also listed in Table 5. The influence of basicity and steel slag amount on drop strength yield is quite apparent. This yield is calculated from the results in Table 5 and described in Fig. 4. The results show that basicity of the sinter has a significant impact on the strength of the sinter, with increasing basicity leading to a decrease in strength. However, the decrease is not substantial. Meanwhile, the amount of slag does not have a significant impact on strength at each given basicity level. Therefore, these results suggest that recycling steel slag in the sintered ore is a feasible solution when considering strength. Based on the analysis results, introducing steel slag as a replacement meets the requirements due to achieving high yield and strength.

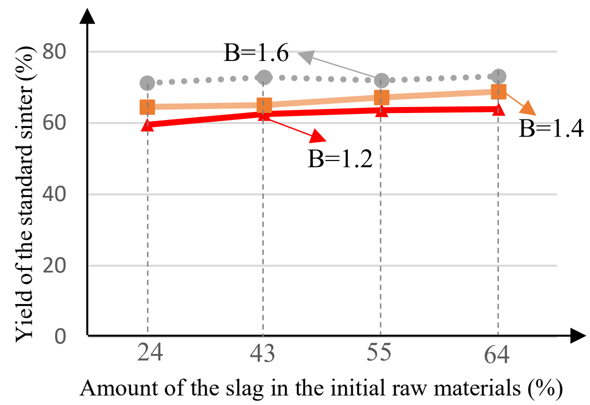


Fig. 3. Effect of slag amount on the strength of sinter.

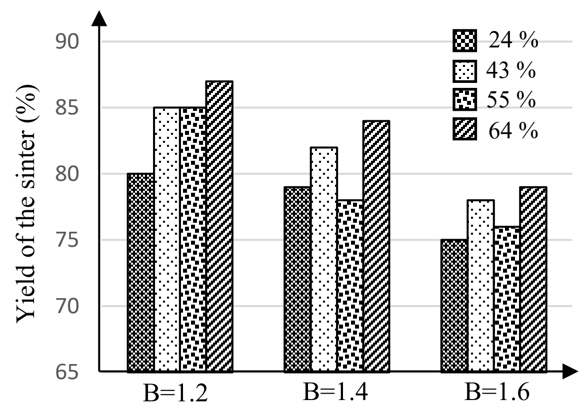


Fig. 4. Effect of basicity on yield.

3.2. Softening Property of Sinter

During the operation of a blast furnace, the cohesive zone of the raw materials in the furnace should be positioned low in the bosh zone. When the cohesive zone is appropriately located, the result is good passing through the zone and improved indirect reduction reactions of the raw materials in the blast furnace. As a result, coke consumption in the operation of the furnace is reduced. Additionally, the shape of the cohesive zone is one of the crucial factors in blast furnace operation. The gas stream moves through the zone by passing through layers of coke. The pressure difference of the gas flow before and after passing through the softening zone plays a significant role. A lower pressure drop leads to more efficient and favorable blast furnace operation because the gas flow easily moves through this layer. Therefore, it's essential to maintain the lowest possible pressure drop in the gas when introducing raw materials into the blast furnace with a narrow cohesive zone [6,9].

The cohesive zone should be located low in the blast furnace, and the initial softening temperature of the raw materials needs to reach a high value. This is one of the factors shaping the formation of the cohesive zone. As the initial softening temperature increases, the position of the softening zone will be lower in the furnace. Therefore, one of the primary parameters to evaluate is the initial softening temperature of iron ore when introduced into the blast furnace. This parameter is used as one of the values to determine the furnace's operational capability when introducing materials into the furnace. Sinter is one of the widely used materials when introduced into the blast furnace. Currently, in Vietnam, most blast furnaces use sinter with a high ratio that can be as high as 90%. For the sinter, the softening temperature parameter needs to be checked and evaluated.

In this study, the softening characteristics were determined to evaluate the feasibility of introducing steelmaking slag-containing sinter into the blast furnace for iron smelting. The research identified the initial softening temperature of sinter when heated at various temperatures under specific loads. The results showed that at a temperature of 1100 °C, the influence of steelmaking slag on the formation of the liquid phase was not observed. The ore particles did not bond together; this indicates that at 1100 °C, the sinter containing steelmaking slag had not yet begun to soften, meaning there was no presence of a liquid phase at 1100 °C. Comparing this to the softening temperature of raw ore, it was found that the initial softening temperature of sinter containing steelmaking slag was higher, while the initial softening temperature of raw iron ore is typically less than 1000 °C [7]. Therefore, sinter containing slag can be more efficiently utilized in the blast furnace compared to raw ore. Additionally, Shatokha's research [10] identified that the initial softening temperature for sinter with a basicity value less than 1.6 was higher than 1100 °C. Hence, sinter with flux-containing steelmaking slag has the potential to be effectively used in the blast furnace in terms of the initial softening temperature.

The studies indicate that when the initial softening temperature is high, the yield of introducing materials into the blast furnace increases [8, 9]. The results show that the initial softening temperature and softening range of sintered ore are influenced by the MgO amount in the raw materials. The initial softening temperature gradually increases from 1128 to 1142 °C, and the softening temperature increases from 1255 to 1288 °C as the MgO amount increases. Furthermore, the softening range increases from 127 to over 145 °C when the MgO amount increases from 1.3 to 2.0% [11]. Various sinter samples containing different levels of slag were heated to temperatures exceeding 1100 °C to determine the temperature at which the liquid phase forms. The samples were heated up to a temperature of 1200 °C. Fig. 5 shows the agglomeration of the sinter. This can confirm that the sinter has started to soften. The temperature of 1200 °C is considered to be the temperature at which the appearance of the liquid phase begins. This temperature is deemed suitable when compared to sinter without slag. Comparing the softening temperature of the sinter with and without slag reveals that the initial softening temperature of the sinter meets the requirements for materials introduced into the blast furnace.

Fig. 5. Sinter sample after softening measurement at the temperature of 1200 °C.

3.3. Reducibility of Sinter Including the Slag

The primary purpose of producing sintered ore is for its effective use as a raw material in the blast furnace. To be efficiently utilized in the blast furnace ironmaking process, the ore needs to be evaluated in terms of its reducibility. Ore with high reducibility, when introduced into the blast furnace, results in highly efficient furnace operation. In this study, the reducibility characteristics of sinter were determined at various slag amounts. Sinter samples meeting the standards were prepared, with each sinter sample weighing 100 grams, and placed in a carbon crucible. To ensure a non-oxidizing environment, aluminum oxide was used to cover the surface, preventing the intrusion of air.

Table 6 shows the extent of oxygen reduction (corresponding to the reduction in sample weight) in sintered ore at different temperature levels and for samples with varying steelmaking slag amounts. The research results indicate that at 1000 °C, the sample's weight decreases only slightly. This means that the ability to reduce oxygen in the sintered ore is low at temperatures below 1000 °C. In contrast, the weight of oxygen lost in the sintered ore is higher as the temperature increases, specifically at 1100 °C. The ability to remove oxygen in sintered ore containing slag at 1000 °C is greater than at 1100 °C.

Table 6. Weight of sinter after reducing process.

Sample	Initial mass sample (g)	Weight after reduction (g)	
		1000 °C	1100 °C
M9	100	97.90	94.14
M10	100	97.78	95.78
M11	100	97.92	97.12
M12	100	97.53	94.53
M13	100	97.84	95.84

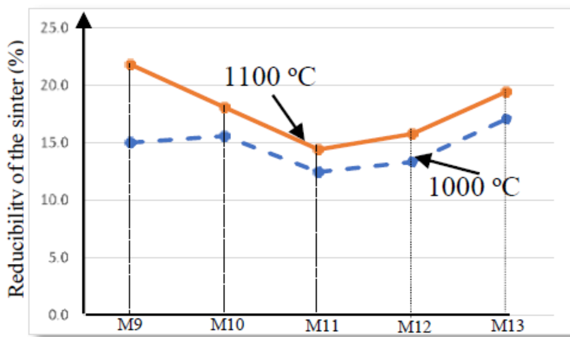


Fig. 6. Reducibility of the sinter.

The results of the reducibility are calculated using (1). Fig. 6 illustrates the relationship between the reducibility of sintered ore at different basicity values at temperatures of 1000 and 1100 °C. The results show that at lower temperatures, the reducibility characteristics of sintered ore are low. Meanwhile, basicity has a significant impact on the reducibility characteristics of sintered ore at both 1000 and 1100 °C. At both temperatures, the results indicate that M3 has the lowest reducibility with 12.5% at 1000°C and 14.4% at 1100 °C. M1 and M4 samples exhibit better reducibility. With sample M1, the best reducibility performance at 1100 °C was achieved, reaching 22.1 % compared to the sample without slag, which had a reducibility of 19.9 %. The results indicate that the ore's mineralogical phase has a significant impact on its reducibility. Additionally, it can be seen that using 21 % slag still ensures the reducibility of sintered ore when compared to using limestone as the flux in the sintering process. The reducibility results show the optimal value when slag is used as a fluxing agent in the sintering process. The reducibility levels in samples with a basicity of 1.6 show that using 24 % slag as a substitute for fluxing agent yields the best results. Therefore, it can be concluded that sintered ore with a basicity of 1.6 and a slag amount of 24 % exhibits good reducibility and meets the required recovery performance. This is a recommended outcome when using slag from the blast furnace in slag-containing sinter.

3.4. Microstructure of the Sinter

The initial materials consist of iron ore and slag from BOF steelmaking. The major mineral components in both the iron ore and fluxing agent mainly include Fe_2O_3, CaO, SiO_2, and Al_2O_3. The product formed after the sintering process results from the diffusion of mineral phases within the initial materials to create a liquid phase that acts as a binding material. Consequently, the sintered ore has three main phases, including Iron Oxides (Fe_2O_3, Fe_3O_4, FeO), Calcium Ferrites (Calcium Ferrite Silicate Aluminate, SFCA), and Calcium Silicate. SFCA is formed to serve as the bonding phase, playing a crucial role in strength and reducibility. The stability of the SFCA phase is highly important, and it is typically formed within the temperature range of 1050 to 1200 °C [12].

The composition of the BOF slag is analyzed before being introduced into the sinter, as shown in Table 2. The slag's mineral composition is considered suitable for inclusion in the sinter as a part of the fluxing agent. Notably, it contains 5.32% MgO and 1.18% Al_2O_3. The presence of Al2O3 and MgO is expected to enhance the characteristics of the sintered ore. The improvement of SFCA stability in the presence of Al_2O_3 amount has been confirmed [12]. Additionally, the Al_2O_3 amount in the initial materials reduces the formation temperature of ferrites. Furthermore, MgO can react with the silicate and oxide phases, producing a substantial amount of high-strength calcium ferrite [13]. Yin *et al.*, in their 2013 study [14], investigated the formation of calcium ferrite at temperatures below 1200 °C when using calcium hydroxide and calcium carbonate as sources of CaO. Moreover, MgO is highly beneficial for sintering and for the introduction of sintered ore into the blast furnace containing MgO [8]. Yin and colleagues [14] observed that the formation of calcium ferrite is related to the source of CaO, and they found that the formation rate of $CaFe_2O_4$ is higher when using $CaCO_3$ as the source to generate CaO.

To evaluate the microstructural properties of sintered ore, the microstructure of the sintered ore should be analyzed. Fig. 7 shows images of the microstructure of sintered ore samples containing varying slag amount. The results indicate that the sintered ore comprises two distinct regions: a white region and a gray region. The gray region is identified as the region that forms the bonding phase between the ore particles. The bonding phase plays a crucial role in creating the strength of the sintered ore.

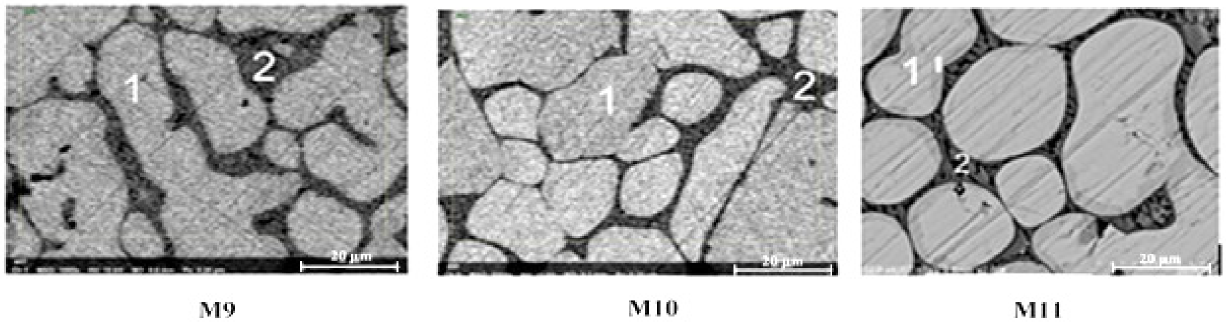


Fig. 7. Microstructure of the sinter.

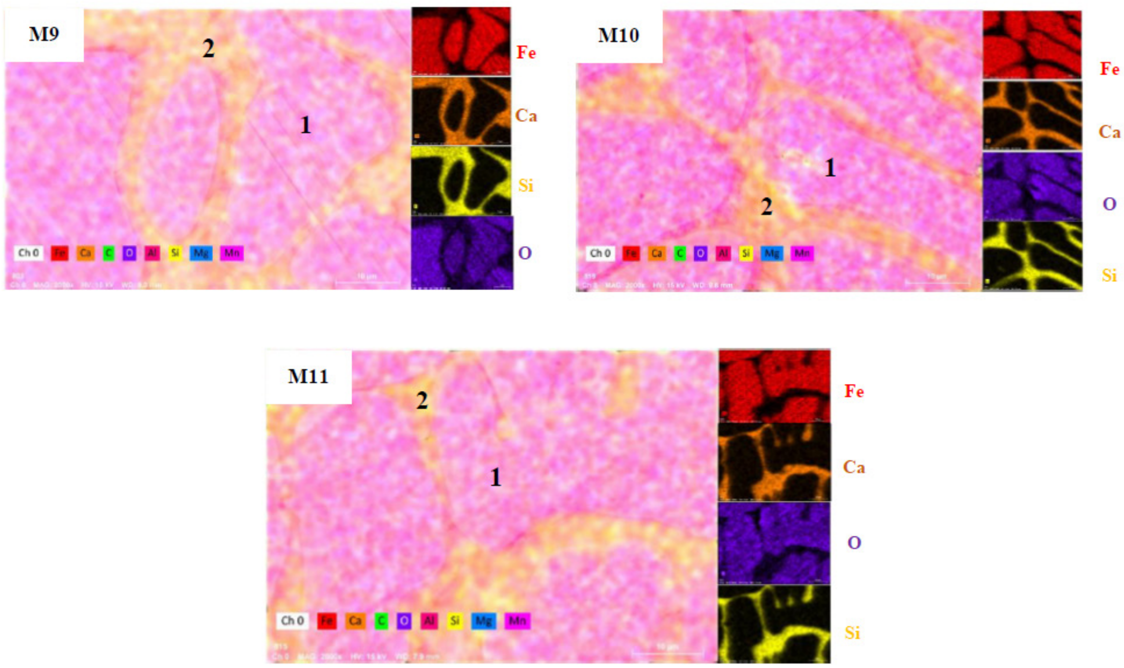


Fig. 8. Energy dispersive X-ray spectroscopy mapping results of the sinter.

Additionally, this phase contributes to determining the reducibility of the sintered product. To determine the composition of the bonding phase, the sintered ore samples are analyzed using a mapping analysis method. The results of the mapping analysis (Fig. 8) illustrate the distribution of elements in the sintered ore. The minerals present in the sintered ore depend on the composition of the initial materials. The formation of the bonding phase is determined by the sintering and cooling process. The composition of the bonding phase is formed through the diffusion of initial solid phases such as CaO, FeO, and Al_2O_3

The white region (Region 1) in the optical microscopy results contains the elements Fe and Oxygen (as indicated by the mapping results). The elemental distribution analysis confirms that the white region consists of iron oxides, namely hematite and magnetite. The white region was determined using the Jimage software and covers 75.6% of the surface area, while the remaining portion is the gray bonding region, covering 24.4% of the area. The results indicate that the bonding region is relatively small in comparison.

The elements Ca, Si, and O are primarily concentrated in the gray region (Fig. 7). The gray region is thought to contain calcium silicate and calcium ferrite. Calcium ferrite phases typically include some silica and alumina, forming the SFCA phase. This is the primary bonding phase in sintered ore. The gray region represents the bonding phases that hold the ore particles together as they cool from the liquid phase. The gray phase surrounds Region 1, which contains iron oxides. This is confirmed to be the formation of the liquid phase bonding the ore particles. Thus, with the addition of slag, the formation of the bonding phase can be affirmed similarly to the bonding phase when no fluxing slag is used. This condition is necessary to create sintered ore that meets the requirements for strength and reducibility. From the images, it is evident that sintered ore containing slag includes phases such as CaFe, SFCA, and more.

4. Conclusion

The addition of steelmaking slag into the sintering process has been demonstrated to be feasible. The results indicate that the reduction capacity of the ore containing slag is equivalent to that without slag. The yield of sintered ore decreases by approximately 1% in comparison with the steel slag used as a flux. The sintered product meeting standards (size and strength) has values ranging from 59.5 to 71.9%. The highest reduction degree was achieved with a 24% slag at a basicity of 1.6 and a temperature of 1100 °C. In contrast, the reduction degree of the sintered ore was lower at 1000 °C. The initial softening temperature was determined to be 1200 °C. The morphological features of the sintered ore indicate the formation of a liquid bonding phase. It can be concluded that the primary bonding phase is calcium ferrite. The liquid phase shows an even distribution of the elements Ca, Si, O, and Fe.

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