

Reduction Kinetics Behavior of Goethite Iron Ore in CO/CO₂ Atmosphere from Wood Charcoal

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ABSTRACT

The reduction of iron from its ores is a major sector in metallurgical industry worldwide. Suitable methods of extraction of iron from its ores have been a major concern since the nineteenth century due to the high level of impurity of its ore as those impurities can best be removed by chemical reduction. This work has sought to determine the reduction kinetics of a goethite ore using reduction gases from wood charcoal. The reduction kinetics of the particle sizes 5, 10, 15 and 20 mm at 570, 700, 800, 900 and 1000°C were studied. Scanning Electron Microscopy (SEM) and Energy Dispersion X-ray (EDX) analysis were done to investigate the spectrometric phase change and the metallic components of the ore sample after reduction respectively. From the result generated, the maximum degree of reduction of 43.8, 21.0 and 8.7% with swelling index values of 13.6, 38.4 and 67.5% were achieved at furnace temperatures of 570, 900 and 1000°C respectively after reduction time of 40mins. The result also showed degree of metallization of 91.62, 75.96 and 93.6% were achieved at reduction temperatures of 570, 800 and 1000°C respectively at residence time of 40mins. This indicated that the overall metallization degree of the reduced ore sample does improve with increasing reduction temperature. A sharp fall in the degree of metallization of the reduced ore sample was observed at the intermediate temperature of 800°C due to high ash layer deposit on the ore.

Keywords: Direct Reduced Iron (DRI); Degree of Metallization; Swelling Index; Scanning Electron Microscope (SEM); Energy Dispersion X-ray (EDX)

INTRODUCTION

Background of the Study

In the blast furnace process, temperatures about 1650°C are used in iron-ore reduction [1]. A mixture of ore with coke and limestone are fed from the top of the furnace, while blast of heated air is forced into the furnace from the bottom where, the coke reacts with oxygen in the air blast to produce CO [2]. Direct reduced iron (DRI) or sponge iron is a good substitute of scrap for making steel in electric arc furnace, basic oxygen furnace etc. which resulted in rapid worldwide growth in DRI production [3]. DRI is a solid state product of direct reduction processes which is produced either in the form of lump or pellets. Availability of huge amount of non-coking coal, scarcity of coking coal deposits and industrial significance of DRI led to many efforts for the development of many direct reduction and subsequently smelter-reduction processes [4].

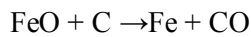
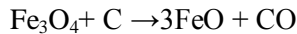
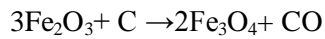
The reducer-smelter process is advantageous over the BF route since a variety of reductants

like lower grade coals, natural gas, charcoal etc. can be used in the reduction phase and the smelter phase the slagging off of gangues can be done [5-8]. This reduction kinetics using released gases from non-contact wood charcoal is the focus of study in this work.

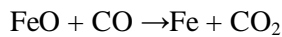
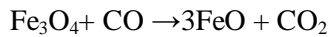
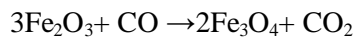
Reduction Processes of Iron Ore

Generally, the kinetic of reduction of iron ore deals with the rate of iron oxide conversion to metallic iron by the removal of oxygen as the rate of chemical reaction increases with temperature [9-11]. For direct reduction of iron ore, the mechanisms are complex because the oxide must go through a series of step wise changes before the conversions are complete. Only a negligible amount of reduction will occur by direct contact of carbon particles with iron oxide particles since such solid-solid reactions are very slow [12]. The actual reduction process occurs through the intermediary of CO. [13] asserted that the reduction of hematite iron ore by carbon in direct reduction process do occurs at three

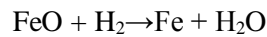
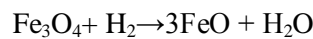
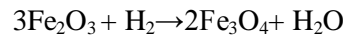
reduction reactions stages which can be written as:



Consequently, the Indirect Reduction of Iron Ore by carbon monoxide (CO) and hydrogen (H₂) also involves three stages when heat treated at firing temperature of 570°C while the second stage can be bypassed at temperature below 570°C due to disproportionation reaction as magnetite (Fe₃O₄) is directly reduced to Fe bypassing the wustite (FeO) stage. Also Liu et al [14] have shown that at reduction temperature below 570°C, rate of chemical reaction are not fast enough to cause significant errors except devolatilization as shown below.



Below the stated above temperature, FeO may likely undergoes a disproportionate reaction, $4\text{FeO} \rightarrow \text{Fe}_3\text{O}_4 + \text{Fe}$ is shifted toward products, whereas above that temperature the reverse reaction $\text{Fe}_3\text{O}_4 + \text{Fe} \rightarrow 4\text{FeO}$ dominates [15-17]. The disproportionate reaction does occur in neutral atmosphere and it does not involve hydrogen but it can influence the mechanism of the reduction of iron oxides in hydrogen in reductant such as wood charcoal [18-19]. Thus, the reduction of hematite ore by Hydrogen occurs in three stages as follows:



Depending on the reduction temperature the proposed paths of hematite iron oxides reduction can be assigned to the following scheme such as $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{Fe}$ (below 570°C) and $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$ (above 570°C).

Procedure for Reduction and Swelling Extent in DRI Process

The degrees of reduction and swelling extent were calculated using the following relations as expressed in [20].

$$\text{Degree of Reduciton (\%)} = \left(\frac{\text{weight loss of the ore sample}}{\text{Total weight of oxygen present in ore sample}} \right) \times 100$$

$$\text{Degree of Reduciton (\%)} = \left(\frac{\Delta W_t}{W_\infty} \right) \times 100 \quad (1)$$

where, ΔW_t is the weight loss in the pellet/lump sample for a given time and W_∞ is the maximum possible weight loss due to removal of oxygen from the reduced ore sample.

The reaction extents as sourced from [21] can also be calculated as follows:

$$\text{Reaction extent } R = \left(\frac{W_o - W_t}{W_\infty} \right) \times 100\% = \left(\frac{W_o - W_t}{\%O_2 W_o} \right) \times 100\% \quad (2)$$

where W_0 is the initial weight of sample, W_t is the weight of sample at time t , and ∞ , W_∞ is the theoretical weight of oxygen present in the samples for complete reduction while $\%O_2$ is the percentage weight of the total oxygen content in the hematite lump samples which is equal to 30 percent for the as received hematite lump samples.

$$\text{Swelling index (\%)} = \left(\frac{V_f - V_i}{V_i} \right) \times 100\% \quad (3)$$

Where, V_i and V_f are the initial volume and final volume of the ore lump after reduction respectively.

EXPERIMENTAL PROCEDURE

Selection of Materials

The proposed raw materials as used in this present work are hematite concentrate and commercially acquired wood charcoal whose chemical compositions are derived by XRD analysis as shown in Table 1 below. The composition of the commercial pure goethite

hematite ore lumps were analyzed using X-ray diffraction (XRD) measurement with chemical empirical formula- $\text{Fe}_2\text{H}_4\text{O}_5$, $\text{Fe}_{1.698}\text{O}_3\text{Sn}_{0.228}$ and chemical formula- $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} \cdot x\text{H}_2\text{O}$, $\text{Fe}_{1.698}\text{Sn}_{0.228}\text{O}_3$.

Iron ore lumps of spherical shapes of different sizes where oven dried for several hours and experimentation was carried out by non-contact direct reduction through heat treatment under

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isothermal condition using wood charcoal fines 570, 700, 800, 900 and 1000°C at different time intervals at heating temperatures of

Table1. Chemical Analysis of Commercial Hematite ore Sample

Element	Hematite (Fe ₂ O ₃)	Wustite (FeO)	SiO ₂	TiO ₂	MnO
%wt	82.65	0.94	1.31	0.05	0.69

The weight of pellet/lumps were categorized in three different size ranges i.e. Set I (5-9.99mm), Set II (10-14.99mm) and Set III(15-20mm) respectively where placed in hollow crucibles inside the furnace as shown in Fig. 4 below. The irregular iron ore lumps were then surrounded with preheated charcoal fines. The furnace was tightly closed with an air-tight insulated cover with an outlet for the release of gases. The iron ore samples already placed in crucibles with different size ranges for identification were placed in the reactor at the preset reduction temperature. The final reduction temperature was controlled within + or – 5°C/mins.

Percentage calculation of the mass or weight of oxygen present in the iron ore samples indicated the theoretical weight of oxygen expected to be removed from the process under complete reduction. Using the data extracted from the X-ray diffraction analysis of the samples, the chemical composition of each metallic compound based on the molecular weight of the constituent element as shown in Table 1 above. We have

$$\%O_2 = \left(\frac{82.65 \times \frac{(16 \times 3)}{(16 \times 3) + (56 \times 2)} + 0.94 \times \left(\frac{16}{(16 + 56)} \right) + 1.31 \left(\frac{16 \times 2}{28 + (16 \times 2)} \right)}{(82.65 + 0.94 + 1.31)} \right) \times 100\%$$

$$\%O_2 = 30.2\% \text{ or } 0.302$$



Figure1. Iron Ore samples prepared at different size ranges



Figure2. Sample measuring instruments: (a) Electrical weighing balance (b) Vennier Calipers

The Reduction Furnace

In this present work, a charcoal bed was prepared in a Mild Steel Reactor or Activated Carbon Furnace as shown in Fig. 3 below with size 60 cm length x 40 cm width x 30 cm height). The crucibles containing the ores were immediately removed after attaining the required reduction time inside the reactor, one after another for the stipulated time and then

allowed to be cooled in air. The reduced iron lumps were weighed on the digital weighing balance as shown in Fig. 2 above and the losses in their weights were calculated.

The diameters of the DRI before and after the reduction were measured in three places by using vernier caliper and averaged weight was taken for each ore sample where the reduced ore lumps do maintained their original spherical

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shape. A reasonable amount of charcoal is placed in the hollow chamber where heat is generated before the prescribed reduction time can be

measure under an air tight condition at a specified temperature as shown in Fig. 3 below.

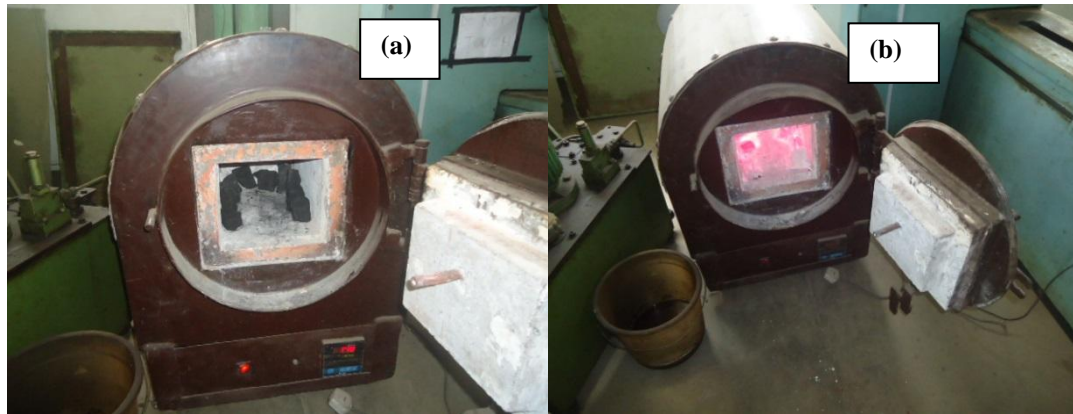


Figure3. Activated Carbon Furnace fired at specified temperature: (a) before heating (b) after heating

In this paper, the kinetics of reduction of iron ore using wood charcoal was extensively investigated showing all the relationship between the chemical and micro structural properties of direct reduced iron. The microstructures and chemical compositions of the DRI samples were investigated using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Combustion tests were applied for the analysis of carbon contents in a number of samples reduced by CO/CO₂ gas mixtures from the charcoal at specified preheated temperature. X-

ray diffraction (XRD) was used for identification of different phases in samples for its chemical composition as shown in table 1 above. Also, the method of data collection and analysis as used in this research work were derived by taking visual inspection of the reduced ore lump which entails the measuring of the initial, final weight and particle sizes. Other reduction parameters such as volume change, swelling index and percentage of degree of reduction in the process are achieved using Microsoft Excel 2010 version software.



Figure4. Reduction furnace crucibles containing iron ore samples of different sizes (a) Cup-Shaped (b) Hollow-shaped Crucibles

RESULTS AND DISCUSSION

Crack Formation and Iron Grain Disintegration

Disintegration of iron due to high swelling index was experienced at firing temperature at 700°C-800°C as shown in Fig. 5 below. Excessive swelling was observed at temperatures up to 900°C. This can be attributed to high content of carbon deposition in the metallic iron which results into subsequent evolution with large

amount of CO/CO₂ gases. High CO/CO₂ content in the system caused more frequent integration of iron grains and expansion. At 1000°C and above, disintegration of large particles of iron grains decreased appreciably and this may be due to recrystallization of iron grains which enhance sintering at such high temperature. It is also worthy of note that formation of large amount of iron grains at high furnace temperature where greater degree of metallization was achieved is likely dependent

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on the contact surface area as bigger particles, with great masses, have higher momentum and

lower tendency of sticking as shown in the Fig 6 below.

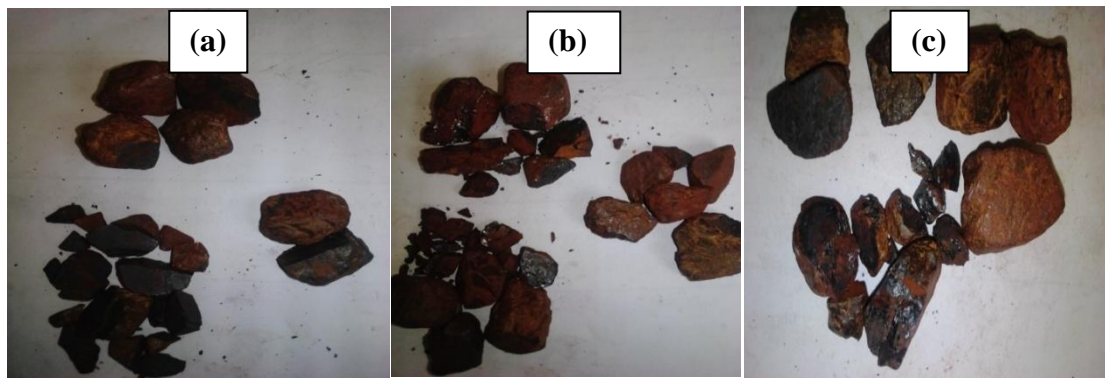


Figure 5. Crack formation and iron grain disintegration of fired hematite ore samples of different sizes as reduced for 40mins (a) 700°C (b) 800°C (c) 1000°C

Reduction Behavior of Fired Hematite Ore Lump Samples

Experimental data on the degree of reduction versus time for fired Goethite hematite iron ore lump been reduced in the activated carbon furnace, using wood charcoal as reduction at temperatures of 570, 700, 900 and 1000°C, have been presented graphically in Fig. 9. Similar plots were obtained for the reduction of lump sample under the similar firing temperatures at different reaction control time and conversion

factor for different size ranges of the iron ore lumps whose graphical illustrations are shown below. The reproducibility of the reduction results were thoroughly investigated for all the used samples as the physical changes on the heat treated samples are shown in Fig. 5 above and Fig. 6 below for different size range of iron ore samples as the dark reddish surfaces of the reduced iron oxide indicated the presence of ash deposit (presence of calcium and aluminum) on the surface of the ore sample.

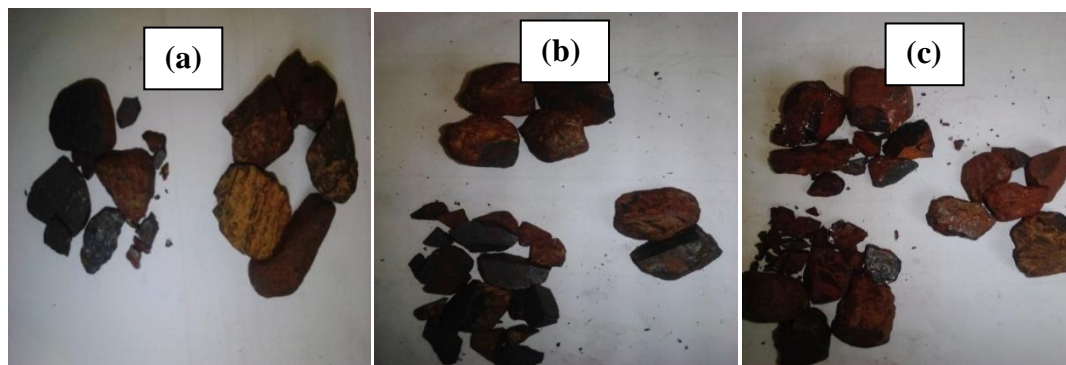


Figure 6. Effect of structural changes on reduction behavior of fired hematite lump at different temperatures in charcoal at reduction time of 40mins. (a) 570°C (b) 700°C (c) 900°C

The result as shown in Fig. 5 and Fig. 6 below all shows the presence of ash layer on the physical structure of the reduced sample which is depicted by their physical changes as a result carbon deposition from the preheated iron oxide samples. The swelling extents as noticed from the reduced sample are due to high percolation of oxygen with progressive acceleration of diffusion of the released CO-H₂ from heated charcoal.

This generate high level of disintegration of the reduced sample at very low rate of reaction and less degree of reduction as depicted in Fig. 7(b) and Fig. 8(b) which in-turn do affect the overall degree of metallization of the reduced hematite

sample regardless of sample size range under consideration for firing temperatures 700, 800 and 1000°C. The case is contrary to what was obtainable above in Fig. 7(a) at 570°C where the ash layer has little or no effect on the degree of reduction and metallization due to the absence of the wustite stage on iron oxide reduction process with less swelling extent.

Effect of Particle Reduction time on Degree of Reduction

Data presented graphically in Fig. 7 to Fig. 8 below illustrate the effect of time for different degree of reduction at temperature of 570, 700, 800, 900 and 1000°C for Goethite iron ore

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reduced by locally acquired wood charcoal under isothermal condition. It can be observed from the Fig. 7 below that the reduction time has an identical effect on the reduction behavior of almost all the studied iron ore-charcoal combination. With increase in reduction time, the degree of reduction increased at every temperature under consideration. The rate of reduction in general was observed to be high up to about 15 to 45% at 570°C reduction and then it decreased as firing reduction temperature increases from 700°C to 1000°C. The reduction of iron ore lump with solid reductant starts at the point of contact on the surface area of the sample which produces CO/CO₂ gas. The CO₂

produced combines with solid carbon and gets converted into CO gas. The CO gas diffuses into the lump ore and takes part in the reduction. The higher reduction rate in initial conditions may be attributed to the combined effect of less resistance offered to the flow of reducing gas into the ore sample and significance contribution of volatile matter release initially, as sourced from [22]. The released volatile matters of charcoal get almost completely reformed into H₂, H₂O and CO during the initial stage of reduction. It might be expected that the pressure of H₂ and CO in reducing gas gives boost to the reduction rate.

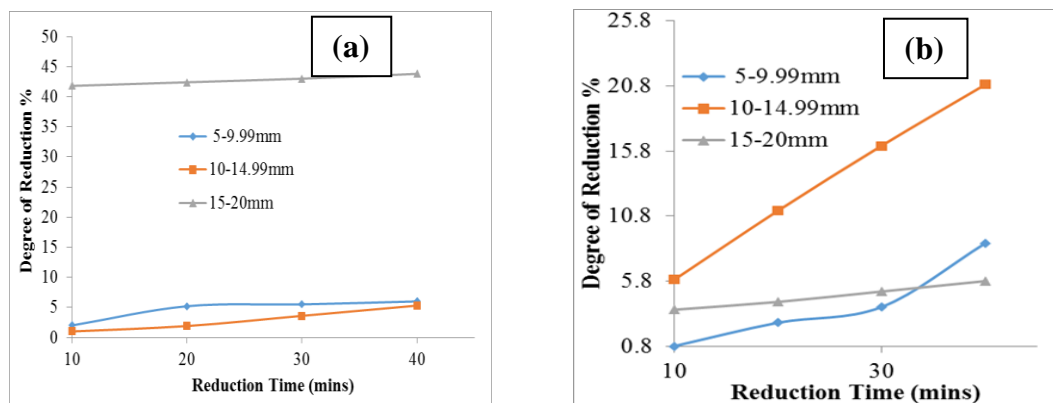


Figure7. Effects of reduction time and Iron lump sizes on the degree of reduction of fired hematite lumps reduced in commercially derived wood charcoal (a) 570°C (b) 700°C.

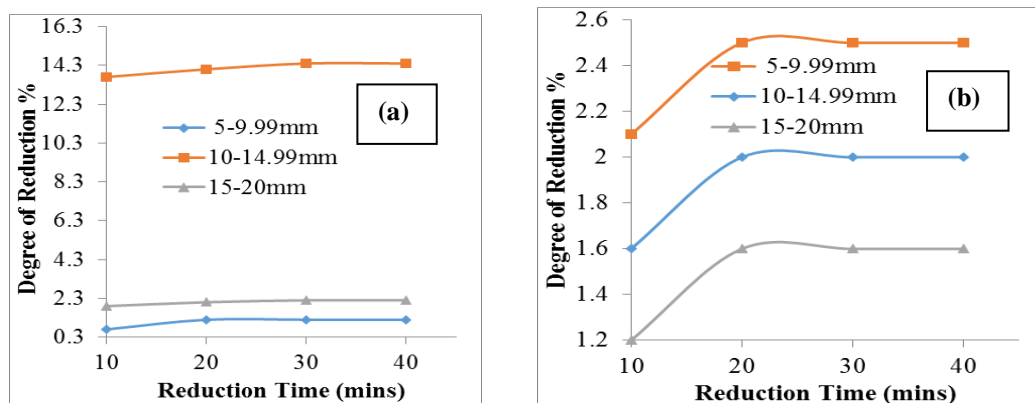


Figure8. Effects of reduction time and Iron lump sizes on the degree of reduction of fired hematite lumps reduced in commercially derived wood charcoal (a) 900°C (b) 1000°C.

As the reduction progress with time, the thickness of the product iron layer increases and offers greater resistance to the diffusion of carbon and reducing gas onto the surface of unreduced iron oxide. This is the reason for lower rate of reduction in the later stages as all the firing temperatures increases.

Effect of Residence Time on Particle Swelling of Iron Lump

It can be seen as shown in Fig. 9 and Fig. 10 below where reduction time increases with the

average extent of swelling at 700°C, 800°C, 900°C and 1000°C irrespective of the required samples size range at reduction time of 10, 20, 30 and 40 minutes respectively as swelling index of more than 30% was observed at 900°C and 1000°C. At highest temperature (1000°C), increase in volume change with not more than 60% swelling of reduced iron ore lumps were observed with increase in reduction time. The expansion of the reduced iron ore pellets is due to the nucleation and formation of disintegrated grains of iron with time at high temperatures [8,

21]. This may be due to an increase in carbothermic reaction where there are

tendencies for high carbon deposition in the iron lump at the wustite stage of the process.

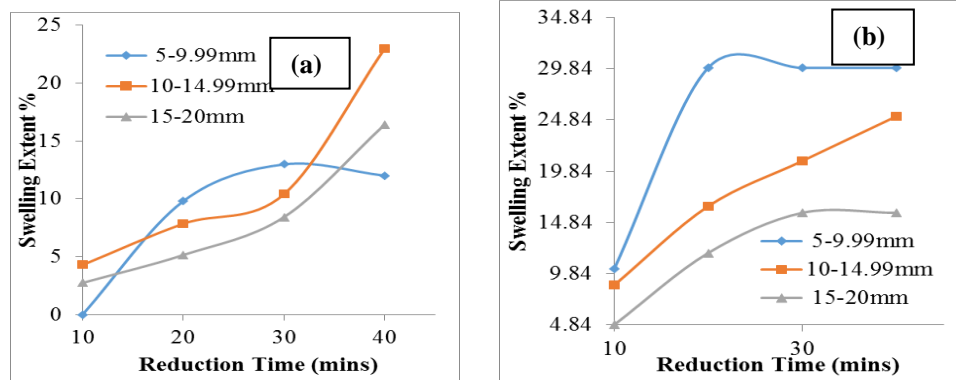


Figure 9: Effects of reduction time and Iron lump sizes on the swelling index of fired hematite lumps reduced in commercially derived wood charcoal (a) 700°C (b) 800°C.

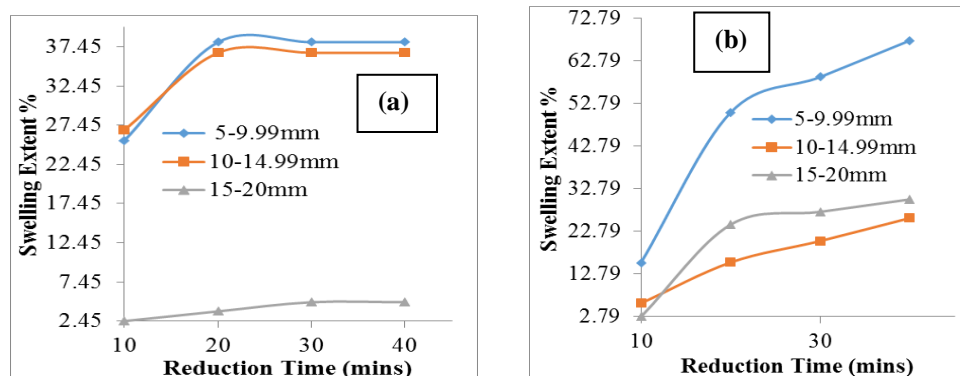


Figure 10. Effects of reduction time and Iron lump sizes on the swelling index of hematite fired lumps reduced in commercially derived wood charcoal (a) 900°C (b) 1000°C.

Analysis of Reduced Hematite Lump Sample by SEM/EDX

The SEM/EDX analysis of this present work was done to practically identify the micro structural segregation of the component of the hematite lump sample as well as the presence of silicon in the different shapes and sizes of the reduced iron ore lump. The SEM/EDX representation analysis of the reduced iron ore spectrum from a spherical lump shaped ore-silicon composites as reduced at 570, 800 and 1000°C as shown in Fig. 11 to Fig 13 below.

It is worthy of note that the micro structural spectrum of virtually all the tested samples shows a high percentage of silicon (39.20, 29.59 and 33.90) respectively. This indicates the presence of slag region where reduced iron oxide mixed with the silicate. The EDX spectrum also clearly shows very high amount of silicon at the micro level of the any composites ore as there may be possibility of silicate formation in the ore sample. Fig. 13 below also depicts a clear representative of the spot of a lump 5-9.99mm size range reduced ore-charcoal sample at 1000°C. The spectrum

analysis shows a strong indication of a region of wustite along the metalized iron. The silicon content of 33.9% was observed indicating that the silicates are mixed with already metalized iron and wustite. It was also observed that the silicon present in the spectrum analyzed at fired ore-charcoal mixture at 570°C and 800°C are as high as 39.2% and 29.56% respectively. This also indicates some significant unreduced oxide especially at 800°C due to slag zone regardless of the size range of the reduced ore sample as some microspots in the ore-charcoal lump shaped composite can also be identified. Such spot only contains metallic irons due to its devoid of slags after reduction residence time of 40minutes as shown in Fig. 12 above.

Thus, the above data indicate a clear cut slag metal separation at least at the micro-level of the reduced iron ore lump shaped ore-charcoal composites samples as shown in Fig 6 above.

It is also worthy of note that an incomplete reduction and carbon deposition during reduction resulted in low reduction extents. It may hamper diffusion of reducing and product gases through the reduced hematite ore lump.

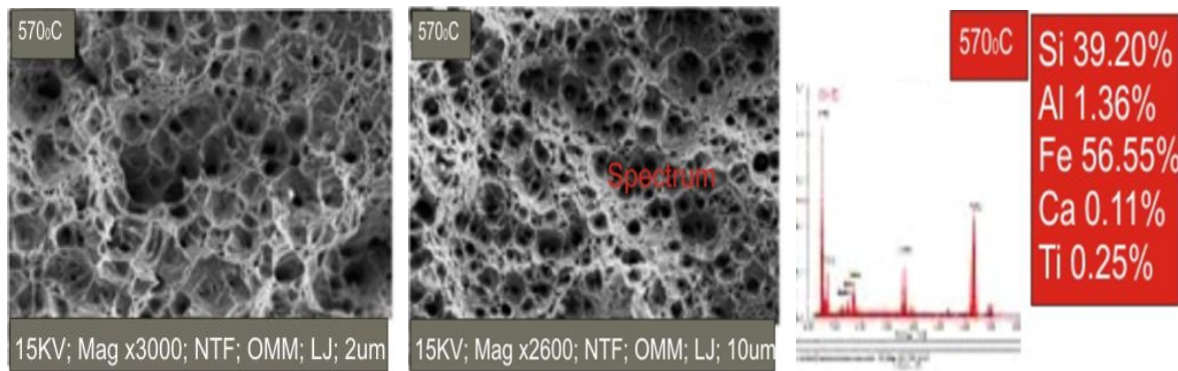


Figure11. SEM/EDX Micrograph and Spectrum view at 570°C with size range 5-9.99mm

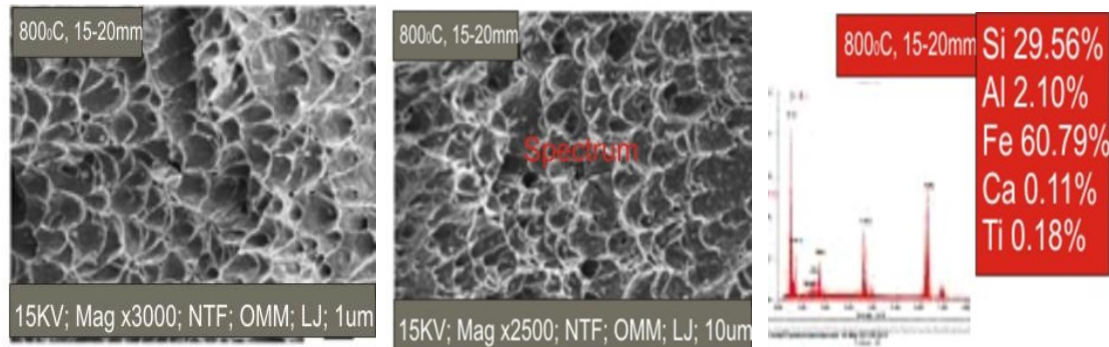


Figure12. SEM/EDX Micrograph and Spectrum view at 800°C with size range 15-20mm

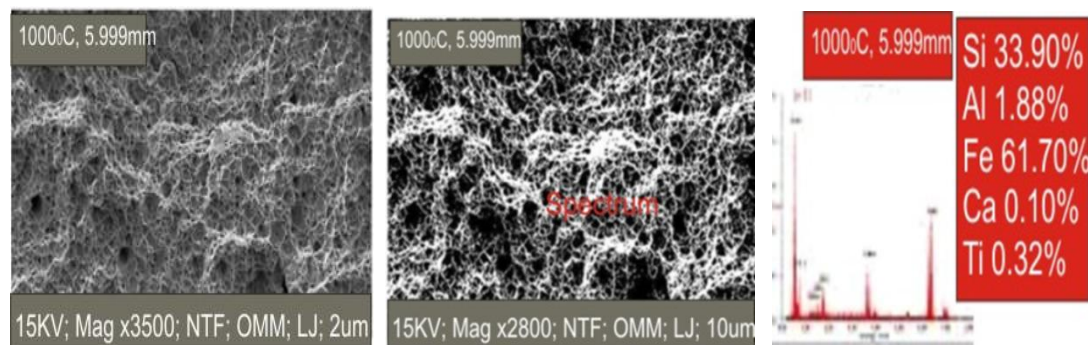


Figure13. SEM/EDX Micrograph and Spectrum view at 1000°C with size range 5-9.99mm

The SEM micrographs of Set-I and Set-III hematite lump sample reduced at 1000°C for 40minutes as shown whose spectrometric view are also shown in Fig. 11 to Fig. 13 above. In the images the light grey phases are metallic iron; the reddish part of the reduced ore contains other metallic components in the sample and the dark grey areas are iron oxide; while the black areas are pores. As seen in the figures, the iron content increases with furnace firing temperature regardless of the hematite lump size range been considered.

Effect of SEM/EDX Analysis on Degree of Metallization

The graph of degree of metallization based on changes observed on the surface on the surface of the iron ore-charcoal samples is depicted below with reference to the firing temperature of

the reactor. The total possible percentage of weight of oxygen in the ore sample has been calculated from the derived data from the X-ray Diffraction (XRD) analysis as shown in Table 1 above.

Thus, the metallic components or compositions in the reduced ore-carbon mixture at prescribed firing temperature 570°C, 800°C and 1000°C are shown in Table 2 below where the remainders of elemental components of the reduced ore samples are taken as the non-metallic component (i.e. oxygen).

The metallic summation of the constituent for each samples spectrum irrespective of the reduced ore sample size range are also depicted in Table 2 after the lump samples have undergone furnace treatment with reduction time of 40minutes.

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The results in Fig. 14 and Fig. 15 below indicate that the difference in degree of metallization and the percentage of iron content after reduction of

hematite lump sample using charcoal for equal residence time of 40 minutes.

Table2. Reduce Ore-Carbon mixture from SEM/EDX analysis for metallic components

Temp. (°C)	%W _o of O ₂ at XRD	%W _o of O ₂ at EDX	%W _i of Fe at EDX	Degree of Metallization (%)
570	30.2	2.53	56.55	91.62
800	30.2	7.26	60.79	75.96
1000	30.2	2.10	61.70	93.05

The respective percentages of iron in the metallic composition as derived from Scanning Electron Microscopy (SEM) and Energy Dispersion Spectrometry (EDX) indicates that the reaction control mechanism or steps for this reduction process may fully be ascertained based on these two factors: increase of weight due to carbon deposition and incomplete reduction [18, 21].

It is also observed in Fig. 5 and Fig. 6 above that at higher infiltration of the reducing gasses in the hematite sample, large amounts of oxide are entrapped in iron layers which indicate possibility for incomplete reduction of the iron oxide. This is essentially observed while

carrying out reduction at intermediate firing temperatures (i.e. 700, 800, 900°C). The sharp drop in degree of metallization at 800°C indicated the tendency for high deposit of carbon at this stage coupled by the possibility for high oxygen content remaining in the pores.

This may also indicate the tendency for incomplete reduction of the hematite lump sample regardless of the size range. Thus, generally, the degree of metallization of the reduced hematite lump sample increases with reaction temperature which in-turn lead to high reduction rate with appreciable iron content achieved in the overall reduction process as shown in Fig. 14 below.

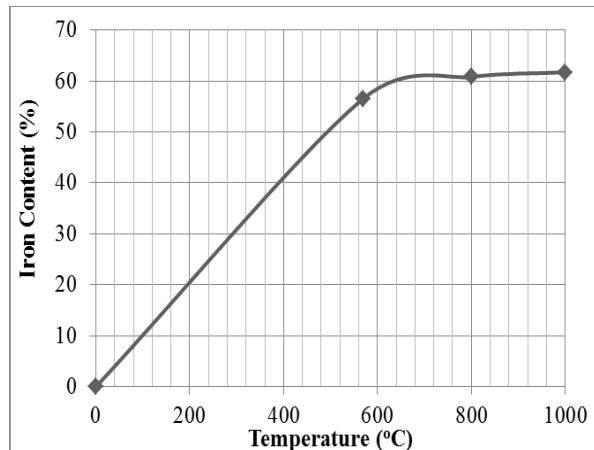
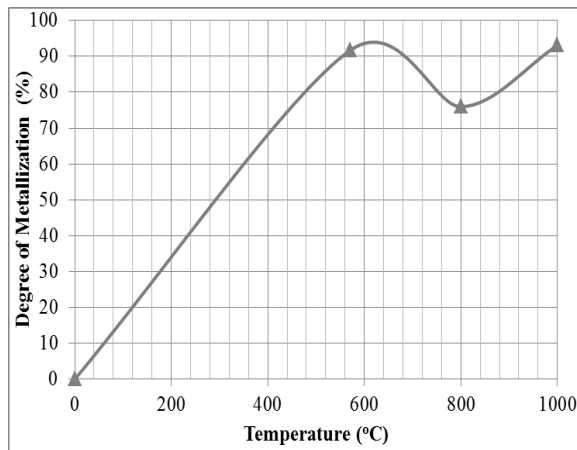


Figure14. (a) Degree of metallization as a function of reducing gas temperature; (b) Iron content (%) as a function of inlet temperature from SEM/EDX micrograph

CONCLUSION

The reduction of commercially obtained Goethite iron ore have been successfully achieved with thorough investigation of the its kinetics of reduction process using commercially acquired wood charcoal as carried out in the laboratory furnace (activated carbon furnace) at a preset firing temperature under isothermal conditions at various reduction time. The result achieved on successfully experimentation and micro structural analyses of the reduced ore samples have revealed the following:

- Further application of the experiment data as obtained on visual inspection by weight and size of reduced sample have shown that the degree of reduction of 40 percent and more for the least firing temperature of 570°C in Set III ore sample with size range 15-20mm. This indicates the influential tendency of large size iron ore samples to exhibit higher degree of reduction with low swelling index.
- Consequently, an abnormal swelling condition between 30 to 65 percent was observed at 700, 800 and 900°C. This validates the presence of high carbon deposition in the

reduced ore sample irrespective of reduction time or pellet size. This also indicates a sharp fall in metallization degree in respect to reduction temperature which is caused by the dominance of the wustite (FeO) stage at the said temperatures.

- SEM/EDX result of the reduced ore samples also validated the analysis given above by visual inspection with claim that degree of metallization increases with reduction temperature regardless of the iron ore pellet size. This implies that the rate of reaction is fastest at low firing temperature (less than 570°C) and slowest at the intermediate temperature (700-900°C) due the tendency of high carbon deposition on the sample which may slow down the diffusion of the reducing gases into the iron ore sample.
- Finally, degree of metallization of more than 90 percent was achieved in the overall reduction process at 570 and 1000°C with least value of metallization degree (75.6 percent) as obtained at 800°C. This is a clear reflection of high percentage iron content and high oxygen discharge from the reduced samples as achieved in the reduction reaction for total reduction time of 40minutes.

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