Effect of different parameters on the direct reduction processes of natural iron ores from Uganda

Master of Science Thesis

by

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[Content]

### ACKNOWLEDGEMENT

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Abstract

Around 50 million tones hematite iron ores with high iron content at a grade of 68% were found in Muko area of Uganda. Currently, the iron and steel making industry in the country has not yet developed to meet the demand to process the natural ores. Based on the analysis of its composition and mechanical properties, I infer that the natural iron lump ores can charge directly into reduction furnace without any prior treatment, so that the cost of whole process without additional enrichment and sintering will be reduced quite much. Also direct reduction iron (DRI) process use natural gases instead of coals which can greatly reduce the environmental pollution and lower the processes cost. If the raw materials that fit for requirements of majority plants, the seller’s market can get greater control over prices, terms due to the easy and cheap processing. All lump ores were from Butare area which is one occurrence of Muko and named sample A in this work. This research aims to use the lowest cost method to reduce Butare area iron ores and find out the optimum parameters on reduction reactions of Butare iron lump ores, such as flow rate, temperature, size of samples. Base on experiments with different designed parameters, Butare iron lump ores successfully reduced by DR furnace and microstructure was found to be an important parameter that strongly influent reduction degree of Butare iron lump ores.

Keywords: Butare iron lump ores, direct reduction, temperature, flow rate, weight, microstructure.
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Lastly, I am very grateful to Ph.D student Mania Kazemi for doing experiments together with me.
1. Introduction

Worldwide, iron ore consumption for steelmaking was standing at 850 million tons at the end of the twentieth century and was estimated to reach more than 1.3 billion tones over the first quarter of the twenty-first century [1]. Uganda is a country that was famous by plenty of natural resources underground. The deposit of iron ores in southwest region named Muko, estimated exist 30-50 million tones with a grade of 68% iron content. To find out the main elements that could influence whole reduction process and the best reduction method, both internal factors, such as sample chemical composition, mechanical properties, thermal stability and external reducing environment were studied in this paper. All samples that tested by experiments were named sample A and came from Butare area of Uganda.

1.1 Chemical properties

Samples came from Butare area are high grade hematite iron ores which have around 65% total iron content. Hereby, I checked some high grade iron ores from references, which shown similar compositions with quite low gangue materials (\(\text{SiO}_2 + \text{Al}_2\text{O}_3\)) as sample A in table 1. Many researchers have studied that gangue materials strongly harmful effect on the iron ore reduction process. But for Butare mine, the percentage of \(\text{SiO}_2 + \text{Al}_2\text{O}_3\) were investigated less than 4% which means this influence would be neglected. Meanwhile, deterioration elements such as S, P are around 0.001% and 0.009% respectively. Consequently, iron lump ores from Butare area have a very high level of advantages on getting purer iron process at temperature lower than melting point.

<table>
<thead>
<tr>
<th>Iron Ore source</th>
<th>Fe (total)</th>
<th>(\text{Fe}_2\text{O}_3)</th>
<th>SiO(_2)</th>
<th>Al(_2\text{O}_3)</th>
<th>MnO</th>
<th>TiO(_2)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butare mine (Uganda)</td>
<td>64.82</td>
<td>92.6</td>
<td>2.28</td>
<td>1.35</td>
<td>0.01</td>
<td>0.07</td>
<td>2</td>
</tr>
<tr>
<td>L-A (Australia)</td>
<td>65.34</td>
<td>91.76</td>
<td>2.17</td>
<td>1.34</td>
<td>0.47</td>
<td>0.03</td>
<td>3</td>
</tr>
<tr>
<td>L-B (Australia)</td>
<td>64.22</td>
<td>90.22</td>
<td>2.30</td>
<td>0.57</td>
<td>0.14</td>
<td>0.02</td>
<td>3</td>
</tr>
<tr>
<td>L-C (Australia)</td>
<td>64.07</td>
<td>91.05</td>
<td>2.18</td>
<td>1.10</td>
<td>0.17</td>
<td>0.03</td>
<td>3</td>
</tr>
<tr>
<td>Zenith mine (India)</td>
<td>64.51</td>
<td>92.25</td>
<td>1.55</td>
<td>2.34</td>
<td>0.03</td>
<td>0.14</td>
<td>4</td>
</tr>
<tr>
<td>M.G.Mohanty mine (India)</td>
<td>64.52</td>
<td>92.27</td>
<td>1.81</td>
<td>2.11</td>
<td>0.02</td>
<td>0.08</td>
<td>4</td>
</tr>
<tr>
<td>D.R.Pattnaikmine (India)</td>
<td>65.22</td>
<td>93.26</td>
<td>0.92</td>
<td>1.62</td>
<td>0.06</td>
<td>0.07</td>
<td>4</td>
</tr>
</tbody>
</table>
1.2 Physical properties

Quality and delivery are obviously key issues and have to be strictly monitored and controlled on material sourcing and throughout the manufacturing phases. Tumble index (TI), abrasion index (AI) and shatter index are three indices to describe the mechanical properties of iron ores. The higher value of TI and lower values of AI, SI, the stronger strength of iron lump ores. Butare lump ores with high TI and low AI, SI compare with other reference mines indicated that Butare iron ores could keep its original shapes tightly during transportation, storage and charging to the reduction furnaces without disintegrating into small pieces. Also from table 2, Normally, iron ore lumps have much lower porosity than the corresponding fired pellets, but Butare lump iron ores have a better porosity than the India mines and shown the nearly value as India fired pellets. Therefore, Butare iron lump ores have very good strength and porosity that shown high possibility for charging into direct reduction (DR) furnace to get high reduction degree.

<table>
<thead>
<tr>
<th>Lump ore name</th>
<th>Tumble index (wt% of+6.3mm)</th>
<th>Abrasion index (wt% of-0.5mm)</th>
<th>Shatter index (wt% of -5.0mm)</th>
<th>Apparent Porosity (%)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butare mine</td>
<td>92.96</td>
<td>3.41</td>
<td>2.01</td>
<td>14.0</td>
<td>----</td>
</tr>
<tr>
<td>Zenith mine</td>
<td>85.3</td>
<td>8.9</td>
<td>0.87</td>
<td>2.0</td>
<td>22.8</td>
</tr>
<tr>
<td>M.G.Mohanty mine</td>
<td>91.0</td>
<td>3.24</td>
<td>1.00</td>
<td>2.1</td>
<td>11.8</td>
</tr>
<tr>
<td>D.R.Pattnaik mine</td>
<td>86.7</td>
<td>5.01</td>
<td>0.98</td>
<td>2.5</td>
<td>19.5</td>
</tr>
</tbody>
</table>

From table 2, high porosity plus strong strength gives higher possibility to use lump ores instead of pellets in DRI furnace at high temperature environment. Meanwhile, as the low disintegration tendency related to the mechanical strength of the ore, TI greater than 95% and AI, SI lower than 5% are considerate to be accepted for great reduction behaviors.

1.3 Thermal properties

During the high temperature reduction, samples could be swelling, sticking or thermal disintegration into small pieces due to volume instability. Butare iron lump ore that named Ug 5 was tested by Thermogravimetry (TG) and Differential Thermal Analyzer (DTA) experiments. Figure 1 told that in temperature interval 365-632°C, γ-hematite transformed to α-hematite in these iron ore samples and in the temperature range from 1267°C to 1361°C, it exhibited an exothermic peak as the melting point [2]. So at temperature below 1000°C, Butare iron lump ore should be stable to keep its own shape and composition.
Figure 1: Thermo-grams results of Butare lump iron ores. Curve 1 is the mass change of samples by TG, Curve 2 is ex/endothermic reactions by DTA [2]

1.4 Main reduction routes for Butare iron lump ores

Currently, Metallurgy method should meet the feasible technological and economical keys to reach the industrial scale level and fulfill the production of green steelmaking process. The main routes for lump ores to produce iron productions are sintering, direct reduction (DRI) and blast furnace (BF) three categories. Butare lump iron ore was found to meet most of the feed raw material requirements (physical, chemical and metallurgical) for both blast furnace and the major direct reduction processes [2].

DR process generally defined as the reactions occurred below the melting point but blast furnace normally worked in temperature higher than iron ores melt points which means BF method consume more energy than DR route. On the other hand, due to the utilization of coal coke as the reducing agent, much carbon and sulfur pollutant generated from the process. But for known natural gases, which cost cheaper, produce less pollutant and easy to capture the generated gases. Considering many factors such as lower energy consumption, lower investment costs, high production value a higher flexible and using cheaper raw materials and reducing gases, less pollutant from natural reducing gases and increasing cost of coke, DR technology presently has demonstrated its flexibility and reliability through the operation of different industrial plants worldwide. Furthermore, The Midrex and HYL process contributes the biggest share of the DRI produced, which corresponds to about 60% of the world DR production. From the supply chain view, raw materials could boost its value if fits with many manufacture plant and the supplier could less depend on fluctuations from the purchasing market.
1.5 Aim and objective

The main purpose of this study is to use the cheapest reduction method, find out the main influence factors on reduction process of Butare iron lump ores and determine the best parameters of overall reduction process. Based on the analysis of its composition and properties, it is possible to determine whether Butare area iron lump ores can appropriately serve as a raw material for iron production by using the directly reduction (DR) method.

- Design ideal reduction system for the processing of Uganda lump ore reduction to get high reduction degree within short reduction time.
- Find out the main influence factors on the reduction process
- Determine the optimum parameters of reduction reactions.
- Establish the possible prospects to use the lowest cost and pollutants green process.
2. Experimental work

All samples were tested in a lab direct reduction (DR) furnace. As investigated by many researchers, four main experimental factors should be designed first for experiments.

2.1 Experimental parameters design

- Reducing agent and flow rate
- Temperature
- Sample size
- Microstructure

2.1.1 Reducing agent and flow rate

Argon was used to protect the resistor of furnaces and clean up air and pollutant gases inside the reactor chamber. Generally, whole gas system of reduction process was performed by reactant hydrogen and carbon monoxide and productions of CO₂ and H₂O, CH₄ for suitable steel industry. Carbon could be generated during the overall process as high content of CO in syngas. High percentage of reducing gas CO also indicated the higher possibility to get carbon deposition and carbides formation.

As investigated by researchers and steelmaking plants, the fastest reduction reaction obtained when using as much as H₂ content with lowest CO% composition of mixture gases. CO gas is cheap and can easily find at natural gases, mixture gases with CO and H₂ mainly used in steelmaking plants. Table 3 listed the gas system of different DR process. Also Midrex gas gives intermediate result and worked as the biggest DR steelmaking industry. It was recommend that the ratio of mixture gases of hydrogen and carbon monoxide (H₂: CO) should be 1.6 to 1.8 for steel industry [5]. In addition, the experimental setup was checked that it works stable if using reducing gas ratio of H₂: CO = 1.5.

Table 3: Reducing Gas system used by various DR processes [5]

<table>
<thead>
<tr>
<th>S No</th>
<th>Component</th>
<th>MIDREX</th>
<th>HYL Reformer</th>
<th>COREX</th>
<th>Syngas</th>
<th>ZR Scheme</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>H₂</td>
<td>55</td>
<td>73</td>
<td>19</td>
<td>51</td>
<td>50</td>
</tr>
<tr>
<td>2</td>
<td>CO</td>
<td>33.5</td>
<td>15</td>
<td>38</td>
<td>31</td>
<td>15</td>
</tr>
<tr>
<td>3</td>
<td>CH₄</td>
<td>3.1</td>
<td>5</td>
<td>1</td>
<td>9</td>
<td>25</td>
</tr>
<tr>
<td>4</td>
<td>CO₂</td>
<td>2.6</td>
<td>1</td>
<td>35</td>
<td>6</td>
<td>3</td>
</tr>
<tr>
<td>5</td>
<td>H₂O</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>1.5</td>
<td>4</td>
</tr>
<tr>
<td>6</td>
<td>N₂</td>
<td>0.8</td>
<td>1</td>
<td>4</td>
<td>1.5</td>
<td>3</td>
</tr>
<tr>
<td>7</td>
<td>H₂/CO</td>
<td>1.64</td>
<td>5</td>
<td>0.5</td>
<td>1.65</td>
<td>3.33</td>
</tr>
</tbody>
</table>
As many researcher studied, H₂ have higher diffusion rate than CO in higher temperature environment so that the reduction velocity of H₂ is higher than CO. Figure 2 shown, at temperature greater than 812°C, hydrogen is the primary reducing gas. But while the reducing gases contain both H₂O and CO₂, the effective concentration of CO and H₂ could be reduced by the water gas shift reaction (WGSR): CO (g) + H₂O (g) ↔ CO₂ (g) + H₂ (g), which shown the method to converting CO gas to more usable H₂, so that WGSR strongly influenced the speed of reduction reactions. The “dash-dot” line in figure 2 indicates the WGSR: equilibrium line for a gas mixture (CO + H₂) [6]. Temperature change will shift the equilibrium position along this line depending on temperature, higher temperature T₁ line in figure indicated more H₂O and less CO₂ content environment compare with lower temperature line T₂. In this case, WGSR turns to right side and preferred to generate more hydrogen gas. Limited by WGSR, temperature was suggested to be higher than 812°C to get faster process.

![Figure 2: Water Gas Shift Reaction (WGSR) equilibrium diagram in general; definition of variables: Temperature axis is normal to the paper and T₂ is lower than T₁][6]

Flow rate defined as the gas velocity and strongly connect with the pressure in the reactor of furnace. Theoretically, flow rate should be as high as possible to supply heat for reactions and give the highest reduction until reaction equilibrium situation. But too high flow rate could obtain dust carryout and the greater reducing gases than equilibrium state generate waste. According to the overall reduction system and experiment equipment system, flow rate that higher than equilibrium requirement was not suggested because of wasting. So hereby, flow rate was tested from 0.25 to 1 L/min by experiment setup limitations.

2.1.2 Temperature

Reduction reaction of hematite will produce higher metallization degree as increase in reducing gas inlet temperature. However, this increasing temperature is limited by physical properties of iron. Based on the plant data, ring reduction temperature over than 880°C (1155K) will initiate formation of sticky iron. It implies that reduction gas inlet temperature should be below than 973°C (1246 K), although metallization degree will still increase as shown on figure 3 [7]. Under different temperature ranges, different reactions would happen, it means temperature setting used
in experiments was bounded by three reactions, sample thermal properties and limitation of experimental setups.

Figure 3: Metallization degree as function of gas inlet temperature [7]

- Reverse boudouard reaction: $2\text{CO} \rightarrow \text{CO}_2 + \text{C}$. Samples should heat up greater than 725°C according to Ellingham diagram on figure 4. Otherwise, the carbon would be deposited severely and formatted carbonization simultaneously during iron reduction process then hinder the reduction reactions ongoing. Figure 4 also shown that the reduction of hematite to wustite is favored at high temperatures. However, the capture of exhaust gas CO$_2$ via lime (CaO) is generally favored at temperatures less than 900°C. Nonetheless, the temperature window of 725–900°C affords thermodynamically favorable reaction conditions for both the oxidation of CO to CO$_2$ using iron oxide and the subsequent sequestration of CO$_2$ by lime [8]. Hence, in this investigation, the temperature range of 725–900°C was studied for the reduction process by mixture gases hydrogen and carbon monoxide.

Figure 4: Gibbs free energy with temperature [8]
• Water-gas-shift (WGS) reaction: \( CO \text{ (g)} + H_2O \text{ (g)} \leftrightarrow CO_2 \text{ (g)} + H_2 \text{ (g)} \). As mixture gases of hydrogen and carbon monoxide worked as the reducing agent, the performance of Hydrogen restricted by temperature that should higher than 812ºC.

• Reduction reactions on figure 5 indicated that temperature should be greater than 570ºC to generate less magnetite. As all samples reduced by hydrogen and carbon monoxide, the reduction process follow the stepwise as hematite to magnetite, magnetite to wustite, wustite to iron finally. \( Fe_3O_4 \) transformation to \( Fe_2O_3 \) leaded to a volume change during the reduction process. Crystallographic changes occur in the molecular arrangement, consequently, high thermal strain increased volumetric strain and cracking tendency.

![Figure 5: Mechanism of hematite reduction process [9]](image)

Reduction gas temperature over than 973ºC (1246 K) is not recommended because the formation of sticky iron. It implies that reduction gas inlet temperature should be lower than 973º C (1246 K). It also mentioned that the pure iron ore could be swelling at 800-950ºC in DR process which might reduce porosity of iron lump ores then decrease the reduction efficiency [10]. Meanwhile, the controller temperature of experiment equipment must be no higher than 1100ºC which has around 100ºC difference with reaction zone so that the reaction temperature should lower than 950ºC. Hence, reduction temperature at 800-900ºC proved to show good performance during DRI process.

2.1.3 Sample size

The sizes of particles linked with the gas-solid contact surface area. The larger area it is, the higher reduction degree could be obtained. But the reactor tube of experimental equipment has an inner diameter of 28 mm. To enable reducing gases passing through the reaction tube smoothly together with the sample piece could be soaked in the reducing gas atmosphere completely, all tested samples were made with the largest length around 20mm.

Sishen iron ores from South Africa had the similar composition and mechanical properties as our samples which already studied in HYL methods. It suggested the oversize of iron lump ore was 20mm in table 4. Because the shapes of sample lump ores were difficult to control by operators. Also if we respect the microstructure of samples which could cause different shapes after crushing steps, all iron lump ores shown irregularly shapes. So that it is difficult to set the size as main parameter. Since density of sample was fixed to be 5.7 g/cm³, the weight of sample instead of sample size was set as the main parameter of reduction degree. According to estimate value of sample volume, the weights of samples were checked ranging from 1 to 7 g.
Table 4: Comparisons of sizes between Sishen ores and sample A

<table>
<thead>
<tr>
<th>Description</th>
<th>Fe(%)</th>
<th>SiO2(%)</th>
<th>P(%)</th>
<th>Oversize (max)</th>
<th>Undersize (max)</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Sishen)20mm lump ore</td>
<td>66.00</td>
<td>3.70</td>
<td>0.057</td>
<td>20.0%-20mm</td>
<td>9.0%-8mm</td>
<td>11</td>
</tr>
<tr>
<td>Sample A</td>
<td>65.1</td>
<td>2.28</td>
<td>0.0087</td>
<td>23.27mm</td>
<td>11.31mm</td>
<td>2</td>
</tr>
</tbody>
</table>

2.1.4 Microstructure

Microstructure of sample gave information on grain size and shape, porosity and shapes of sample after crushing, which is a very important parameter for DRI process. As we know, larger sizes of grains are more difficult to react and have high resistance on corrosion. The reduction normally occurred from out layer of grains then gradually went inside. So samples with smaller size grains and high porosity microstructure could be reduced more completely under the same reduction conditions.

2.2 Experiment apparatus

Experimental device worked on thermal gravimetric analysis (TGA) principles. Environment of high temperature and high reducing gases content resulted in high iron reduction degree according the Baur-Glaessner diagrams on figure 6. The graph of the apparatus described as below figure 7: Reduction temperature controlled by controller and reducing gases passed through downside of furnace which closed to the boating materials. It indicated that the whole reduction atmosphere was surrounded by CO and H₂ with very high ratios of H₂/H₂O and CO /CO₂ and production gases went away by the blowing reducing gases. Basing on the operator skills, two thermocouples generally had not the same position which given two different reading temperatures on the display panel. Both boating materials and reactor had no reaction with any input ingredients.

Figure 6: Combination of the Baur-Glaessner diagram for H₂-H₂O and CO-CO₂ atmosphere [12]
2.3 Experimental operations

- Measure sample size and sample weight.
- Clean the reaction container.
- Set the computer parameters and run COM2 software program.
- Put sample into the reaction zone and record the weight of sample from COM2 software.
- Open the tap of cold water and switch Argon gas to be 1L/min around 20 minutes to clean up reaction zone and balance zone.
- Move up the sample from reaction zone and heat reaction zone up to reduction temperature.
- Set the Argon flow rate to be 0.2L/min and wait the temperature to reach reduction temperature.
- Keep 10 minutes and check the conditions are all stable or not.
- Switch reduction gases and set reduction flow rate then close Argon gas inlet in the reaction tube and open the gas reaction tube.
- Check the leaking of reduction gas.
- Let the reduction gases inlet and wait around 5 to 10 minutes.
- Move down the sample to the reduction furnace chamber.
- Move up the sample from reduction zone while the reaction ended.
- Turn off the reduction gases and set the Argon flow rate to be 1 L/min.
- Close the gas reaction tube and open the Argon reaction tube.
- Cooling sample and pick sample out from reaction tube.
2.4 Experiment proceeding graph

Twenty samples were tested to check the reduction properties of Butare iron lump ores. As shown in figure 8, sample weight keep decreasing which caused by vapor water and dust blowing away, during period 0-A of sample heated from room temperature to around 170°C with a heating rate 6 degree/min.

As the sample was balanced after moving it down to the reduction furnace chamber at A point in figure 8, I point is sample initial weight before put it into equipment. During I to A period, water weight loss due to temperature higher than 100°C and dust remove by gas inlet lead to weight of sample keeping decrease firstly. But while the sample move down to furnace, the force added on the reaction chamber caused the increasing of sample weight in a short moment. Iron lump ore reacted with reducing gases so that the weight of sample keeping decrease until C point. Point C is the moment that weight did not decrease which means the reduction process end up. Point F is the moment that sample was picked out from experimental equipment. So reduction time (RT) = t_C – t_A and weight change caused by reduction process ∆W = W_I - W_F.

Meanwhile, the temperature of sample increased very fast from D point while it move down to the reaction furnace and took 3-5 minutes to reach the reaction zone temperature. Temperature of sample was obtained increase to 600°C within 1 min which could give very fast reduction reactions. Period D-E takes less than 5% of whole reduction process. So we can assume that samples were reduced at the setting constant reduction temperature.
3. Results and discussion

In this study, I try to find out the optimum parameters for the Butare iron lump ore to get purer iron (high reduction degree RD) and faster process (short reduction time RT). The samples A209 and A210 were reduced since room temperature until reduction temperature to check carbon deposition problems. During these periods, so much carbon deposited and retarded the proceeding of experiments, thus, data of sample A209 and A210 did not take into analyze the reduction behave results. Samples A201 to A215 came from big ores block 1 and 2 but sample A216 to A220 prepared from block 3. All data of sample reduction conditions listed below in table 5.

Table 5: Parameters of samples reaction processes

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Temperature (°C)</th>
<th>weight (g)</th>
<th>RT (min)</th>
<th>Flow Rate (L/min)</th>
<th>Width (mm)</th>
<th>Length (mm)</th>
<th>Reduction degree</th>
</tr>
</thead>
<tbody>
<tr>
<td>A201</td>
<td>800</td>
<td>3.78</td>
<td>59</td>
<td>0.5</td>
<td>10.18</td>
<td>15.15</td>
<td>0.909</td>
</tr>
<tr>
<td>A202</td>
<td>800</td>
<td>2.46</td>
<td>45</td>
<td>1</td>
<td>5.6</td>
<td>20.45</td>
<td>0.814</td>
</tr>
<tr>
<td>A203</td>
<td>800</td>
<td>2.23</td>
<td>48</td>
<td>0.25</td>
<td>6.73</td>
<td>20.14</td>
<td>0.808</td>
</tr>
<tr>
<td>A204</td>
<td>800</td>
<td>1.65</td>
<td>30</td>
<td>0.5</td>
<td>10.07</td>
<td>11.31</td>
<td>0.869</td>
</tr>
<tr>
<td>A205</td>
<td>800</td>
<td>6.89</td>
<td>71</td>
<td>0.5</td>
<td>13.12</td>
<td>23.27</td>
<td>0.862</td>
</tr>
<tr>
<td>A206</td>
<td>900</td>
<td>2.24</td>
<td>43</td>
<td>0.5</td>
<td>5.87</td>
<td>16.49</td>
<td>0.957</td>
</tr>
<tr>
<td>A207</td>
<td>900</td>
<td>1.28</td>
<td>60</td>
<td>0.5</td>
<td>4.08</td>
<td>16.33</td>
<td>0.886</td>
</tr>
<tr>
<td>A208</td>
<td>700</td>
<td>4.00</td>
<td>43</td>
<td>0.5</td>
<td>10.25</td>
<td>17.56</td>
<td>0.859</td>
</tr>
<tr>
<td>A211</td>
<td>800</td>
<td>3.58</td>
<td>41</td>
<td>1</td>
<td>9.18</td>
<td>17.23</td>
<td>0.811</td>
</tr>
<tr>
<td>A212</td>
<td>850</td>
<td>4.15</td>
<td>52</td>
<td>0.5</td>
<td>8.84</td>
<td>17.07</td>
<td>0.908</td>
</tr>
<tr>
<td>A213</td>
<td>800</td>
<td>4.08</td>
<td>49</td>
<td>0.75</td>
<td>10.09</td>
<td>20.89</td>
<td>0.838</td>
</tr>
<tr>
<td>A214</td>
<td>800</td>
<td>3.27</td>
<td>44</td>
<td>1</td>
<td>7.88</td>
<td>14.15</td>
<td>0.836</td>
</tr>
<tr>
<td>A215</td>
<td>900</td>
<td>3.10</td>
<td>34</td>
<td>0.5</td>
<td>10.04</td>
<td>15.39</td>
<td>0.947</td>
</tr>
<tr>
<td>A216</td>
<td>900</td>
<td>2.96</td>
<td>51</td>
<td>0.5</td>
<td>8.1</td>
<td>15.47</td>
<td>0.867</td>
</tr>
<tr>
<td>A217</td>
<td>800</td>
<td>4.21</td>
<td>34</td>
<td>0.6</td>
<td>8.44</td>
<td>17.28</td>
<td>0.776</td>
</tr>
<tr>
<td>A218</td>
<td>800</td>
<td>3.39</td>
<td>30</td>
<td>0.4</td>
<td>8.65</td>
<td>16.67</td>
<td>0.767</td>
</tr>
<tr>
<td>A219</td>
<td>900</td>
<td>2.95</td>
<td>61</td>
<td>0.5</td>
<td>7.93</td>
<td>17.82</td>
<td>0.881</td>
</tr>
<tr>
<td>A220</td>
<td>900</td>
<td>4.62</td>
<td>76</td>
<td>0.5</td>
<td>9.35</td>
<td>16.68</td>
<td>0.880</td>
</tr>
<tr>
<td>A209*</td>
<td>190-800</td>
<td>1.39</td>
<td>42</td>
<td>0.5</td>
<td>5.93</td>
<td>15.10</td>
<td>0.697</td>
</tr>
<tr>
<td>A210*</td>
<td>43-800</td>
<td>1.91</td>
<td>77</td>
<td>0.5</td>
<td>5.54</td>
<td>15.73</td>
<td>-1.064</td>
</tr>
</tbody>
</table>

3.1 Reduction degree

Reduction degree (RD) defined as the level of removed oxide amount that directly determined the extent of reduction processes. \[ \text{RD} = \frac{\Delta w}{m \times O\%} \] (O\% means the oxygen weight percent that can remove from oxides and \(\Delta w\) is the weight loss of sample after reduction, m is the initial weight of
sample, ΔW is weight loss during reduction process). As we know, carbon deposition and high temperature are two points would weak the strength of samples. To check accuracy of reduction degree of iron ore, it is very important to find out the whole loss weight caused by whole specimen broken down or not. So that samples reduced at high temperature with different flow rate were checked in table 6. It proved both samples shapes and sizes were quite similar between samples that before and after reduction process. So we can assume that the weight losses during reduction process were not caused by the thermal but by reducing reactions.

Table 6: Comparison of sample shapes and sizes

<table>
<thead>
<tr>
<th>Sample</th>
<th>Before reduction</th>
<th>After reduction</th>
<th>Flow rate (L/min)</th>
<th>Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A201</td>
<td><img src="image1" alt="Image" /></td>
<td><img src="image2" alt="Image" /></td>
<td>0.5</td>
<td>800</td>
</tr>
<tr>
<td>A202</td>
<td><img src="image3" alt="Image" /></td>
<td><img src="image4" alt="Image" /></td>
<td>1</td>
<td>800</td>
</tr>
<tr>
<td>A203</td>
<td><img src="image5" alt="Image" /></td>
<td><img src="image6" alt="Image" /></td>
<td>0.25</td>
<td>800</td>
</tr>
<tr>
<td>A204</td>
<td><img src="image7" alt="Image" /></td>
<td><img src="image8" alt="Image" /></td>
<td>0.5</td>
<td>800</td>
</tr>
<tr>
<td>A205</td>
<td><img src="image9" alt="Image" /></td>
<td><img src="image10" alt="Image" /></td>
<td>0.5</td>
<td>800</td>
</tr>
<tr>
<td>A206</td>
<td><img src="image11" alt="Image" /></td>
<td><img src="image12" alt="Image" /></td>
<td>0.5</td>
<td>900</td>
</tr>
<tr>
<td>A207</td>
<td><img src="image13" alt="Image" /></td>
<td><img src="image14" alt="Image" /></td>
<td>0.5</td>
<td>900</td>
</tr>
<tr>
<td>A212</td>
<td><img src="image15" alt="Image" /></td>
<td><img src="image16" alt="Image" /></td>
<td>0.5</td>
<td>850</td>
</tr>
</tbody>
</table>
The chemical composition of sample A is listed in table 7. Only oxides (FeO and Fe₂O₃) were calculated to be reduced during the overall reduction process due to below two points.

- At temperature range 700 – 900°C, according to Ellingham-diagram, FeO and Fe₂O₃ will be reduced together with the neglect content of ZnO and CuO before others oxides.
- The sulfur and phosphide would De-S and De-P after reduction process which happened in EAF at high temperature ranges greater than melting point.

### Table 7: Composition of sample A [2]

<table>
<thead>
<tr>
<th>Ore name</th>
<th>Chemical composition, mass%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe₂O₃</td>
</tr>
<tr>
<td>Sample A</td>
<td>92.6</td>
</tr>
</tbody>
</table>

Consequently, the totally removed oxygen content \( O\% = \frac{92.6 \cdot \frac{16+2}{16+56} + 0.44 \cdot \frac{16}{16+56}}{92.6 \cdot 0.44} \cdot 100\% = 30\%.

Compare with iron ores from India and Australia as shown in table 8, Butare iron lump ores shown greater reduction degree than other lump ores from Australia and India, high thermal stability that did not break into fine fragments and gave the comparable reduction degree as India pellets as table 8.

### Table 8: Comparison of reduction degree with reference iron ores

<table>
<thead>
<tr>
<th>Iron lump Ore source</th>
<th>Average Reduction Degree at 900°C</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>lump</td>
<td>pellet</td>
</tr>
<tr>
<td>Butare mine (Australia)</td>
<td>0.905</td>
<td>------</td>
</tr>
<tr>
<td>L-A (Australia)</td>
<td>0.82</td>
<td>------</td>
</tr>
<tr>
<td>L-B (Australia)</td>
<td>0.75</td>
<td>------</td>
</tr>
<tr>
<td>L-C (Australia)</td>
<td>0.73</td>
<td>------</td>
</tr>
<tr>
<td>Zenith mine (India)</td>
<td>Broken into fine fragments</td>
<td>0.878</td>
</tr>
<tr>
<td>M.G.Mohanty mine (India)</td>
<td>0.707</td>
<td>0.910</td>
</tr>
<tr>
<td>D.R.Pattnaik mine (India)</td>
<td>Broken into fine fragments</td>
<td>0.881</td>
</tr>
</tbody>
</table>
3.2 Sample microstructure

All samples used in this research came from Butare area of Uganda. Different positions of mine might have different chemical compositions and microstructures then influence the reduction process. In this case, all samples tested, respectively, were from top side, middle part and bottom layer of mine and named block 1(B1), block 2(B2), and block 3(B3) here to check reduction characterizers of sample ores.

The microstructures were checked by scanning electron microscope (SEM) as figure 9, samples iron ores that that came from block 1 (B1) and block 2 (B2) almost have the same microstructure which were consist by the long and small strip grains, the clear grain boundary and small pores between each grains. But samples from block 3 shown it have much bigger grain size and fewer pores between each grain. As all tested samples were from different blocks, we can predict that samples from block 3 could obtain lower reduction degree compare with smaller grain size samples from block 1 and 2. Meanwhile, the whole reduction reactions started from outside layer then went inside. Bigger size grains were adjusted by researchers would more difficult to be reduced so block 3 could need longer reduction time compare with block 1 and 2 samples.

![Figure 9: Microstructures of three Butare iron lump ore Block](image-url)
3.3 Reduction tendency of all samples

The weight loss tendency of all samples during whole reduction processes was shown in figure 10, weight loss gradually decrease with reduction time. The red area in figure 10 is the period which samples took to reach the desired reduction temperature in reaction zone. As shown in figure 8 that A-B period is 3 to 5 minutes, which is a short time compared with the whole process. But the reduction degrees of samples in period are relatively large around 10-20%. Fe₃O₄ supposed to be generated more at temperature lower than 600°C. The drawback can be eliminated by improve operation steps and there are four factors to cause this undesired result:

- As we know, the porosity of out layer determined the rate of diffusion reducing gases, furthermore decrease the reduction degree and reduce quality of reduction processes. Transformation of hematite to magnetite leaded to crystallization swelling and sample volume change at out layer. In this case, the porosity of sample would reduce and caused a lower and slower reduction process.
- It was easier for reducing gas diffuse through outside layer and getting more and more difficult inside, thus the reduction process was very fast during A-B period.
- Hematite started to react by hydrogen and carbon monoxide since beginning at very low temperature and sample temperature reached to 600°C within one minute, then reaction would be very fast from outside layer went to inner part.
- The Gas-solid contact area was larger and outer reduced iron part had higher content of weight compare with inner part as shown in figure 11. So the total removed oxygen weight was high and the process finished fast.

Figure 10: Weight change (ΔW) within the reduction time
3.4 Effective parameters on reduction degree

3.4.1 Flow rate

Six samples reduced at 800°C with different flow rates were tested to check flow rate influence on reduction degree. The reducing gases flow rate totally were 0.25, 0.5, 0.75, 1 L/min with the fixed ratio of H₂ : CO = 1.5. The main tendency in figure 12 shown samples from block 1 and 2 have quite higher reduction degree than samples from block 3. In this case, microstructure more effective on reduction degree than flow rate. Furthermore, samples with same microstructure reduced shown 0.5 L/min is the best flow rate for Butare iron lump ores at 800°C reduction environment and it had more than 8% reduction degree gap with samples reduced at other flow rates. There are three points to explain this phenomena:

- The highest level of reduction degree should be the equilibrium situation.
- Lower flow rate can not supply enough reducing gas to reach reaction equilibrium so that get lower reduction degree.
- Every reduction reaction need enough soaking time for the reactants contact each other, high flow rate that higher than reaction requirement cause waste and high speed of reducing gases lead to less contact time between reactants agents. Hence, the reduction do not have enough time to finish reaction process. In addition, samples reduced at higher flow rate obtained more carbon deposited on sample surface which strongly block the reducing gases pass through inside part of samples then caused uncompletely reduction processes. The reason is high flow rate environment contain higher amount of carbon which shown higher possibility to get carbon deposition. Consequently, the reduction degree of samples was lower under high flow rate conditions.

In figure 13, no matter the different temperatures and different blocks that samples came from, all samples that above the red line were reduced at 0.5 L/min flow rate environment and shown the higher reduction degree more than 85% than other samples with higher or lower flow rates. So that, the best flow rate for the iron ores that came from Butare area should be 0.5 L/min from all experiments.
3.4.2 Temperature

As we know, Diffusion rate of reducing gases increased with reaction temperature from 700 to 900°C. Thus, reduction degree of samples increased with reaction temperature as many researchers have proved. Besides, samples reduced at high temperature 900°C shown small cracks on the surface which lead to free diffusion of reducing gases. As we know, free diffusion rate offer more completely reactions and faster processes so that the reduction degree could be higher than other samples that reduced by volume diffusion.

Under same flow rate conditions, all samples with same microstructure shown reduction degree
of sample increased with reaction temperature under the same flow rate 0.5 L/min. But samples came from block 1 and 2 had the reduction degree of around 8% higher than block 3, it furthermore proved the strong influence from microstructure.

![RD-temperature](image)

**Figure 14: Reduction degree Vs temperature**

3.4.3 Sample weight

Theoretically, finer particles have better reduction degree because the reactions happened more completely. For raw iron ore materials, the exact size range was bounded with crushing skills that indicated high fluctuation could obtained on sample size. As the density of hematite iron ore were stable at a vule of 5.7 g/cm³, the volume or size of samples changed together with the mass of samples then weight of sample turned to be the parameters which was easier to control.

Figure 15 shown every curve have a peak point 3.78g, 3.27g, 2.24g for different environment reduction processes. It means diverse conditions have each the best range of sample weight and the best weight range could be 2 to 4 g. Furthermore, under 800° C, samples tested at 1 L/min and 0.5 L/min shown a minimum 7.3% gap of reduction degree. Samples reduced at 900 °C in Fig. 15 b shown better reduction degree than samples reduced at 800 °C. So, Butare iron lump ores should choose temperature at 900 °C and 0.5 L/min flow rate conditions to get better reduction degree.

- High flow rate caused more carbon deposite on samples surface which blocked the reaction proceeding. Thus the reduction degree of samples were lower in high flow rate environment.
- High temperature lead to free diffusion of gases and higher diffusion rate of reducing gases that gave higher reduction degree.
- Different reduction environment with different flow rate and temperature shown different equilibrium situations which means sample weight that meet the requirment of equilibrim
could be different. The equilibrium state means highest reduction degree could obtained, in this case, the sample weight shown its best range under different reduction processes.

Figure 15: reduction degree and sample weight. a) all samples reduced at 800°C; b) all samples reduced at 900°C.

3.4.4 The composition and microstructure of samples after reduction process

How much volume of samples had reduced during the process, why microstructure shown its important influence on reduction degree compare with flow rate and temperatuer and which kind of reduction mechanisms for our relatively high porosity iron lump ores?

To understand well about this three points, microstructures and compositions in the middle part of each sample were checked by SEM to ensure the whole reduction reaction have done completely on total pieces or not.
Iron ore from different blocks shown quite different results although reduced at same conditions in table 5. Thus, microstructure could be an important parameter on effect reduction processing. Normally, the middle inner part of sample would reduce more difficult due to lower diffusion rate of reductant gases and lower porosity of sample in high temperature range. Hereby, I choosed samples which represented different parameters (temperature, flow rate, microstructure) and the highest reduction degree sample A206 to be tested. Also, the tested four samples had four different shapes respectively as triangle, rectangular, oval, and irregularity.

- Sample from different blocks such as A206 and A220.
- Sample A201 and A206 were from different temperature.
- Sample A201 and A206 had different flow rate.

3.4.4.1 Sample A201

Sample A201 came from block 1 was reduced at 800ºC with 0.5 L/min flow rate and seen as the normal situation. The reduction degree was 90.9%. Table 9 listed compositions of the different points that choosed as different phases in SEM photos.

White area represented the pure iron which means the part of iron ore totally be reduced as point 2. Gray area as point 1 was check to be FeO content in figure 16. So the middle part of sample A201 was reduced and have many pores inside. Thus, the whole reduction process determined by porosity and diffusion rate of reducing gases. Figure 16 shown the pure iron content area was larger than FeO content surface and it still have some smaller pores as point 5, so that the reduction degree was high and take up around 90.9%. Also, we can check each grain of sample was reduced from out layer went inside. So it proved that bigger size grains take longer time to be reduced and the reduced pure iron part took up less weight percentage of each grains.

<table>
<thead>
<tr>
<th>Spectrum (A201)</th>
<th>Element (wt%)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>O</td>
<td>Fe</td>
</tr>
<tr>
<td>1</td>
<td>24.11</td>
<td>75.58</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>99.90</td>
</tr>
<tr>
<td>3</td>
<td>14.03</td>
<td>85.33</td>
</tr>
<tr>
<td>5</td>
<td>2.86</td>
<td>96.92</td>
</tr>
<tr>
<td>6</td>
<td>10.82</td>
<td>88.67</td>
</tr>
</tbody>
</table>
Sample A206 tested at 900°C with 0.5L/min flow rate and was from block 1. White areas in the picture represent pure iron. The reduction degree of A206 was 96.7% which proved by the figure under SEM photo.

The middle part of sample still shown high reduced iron content which indicated the reduction time of process governed by gases diffusion rate into inner layer. But it still shown many small pores inside the materials which means reducing gases can pass through into middle part easier than other samples. Only a little dark area in the bigger grains of the middle part that surrounded by compacted iron was not reduced. As the reduction process went gradually from out layer inside inner part, at 900°C, high temperature have high efficiency diffusion rate, so that higher temperature caused completely reduction process.
### Table 10: Composition of sample A206

<table>
<thead>
<tr>
<th>Spectrum (A206)</th>
<th>Element (wt%)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>O</td>
<td>Fe</td>
</tr>
<tr>
<td>1</td>
<td>24.11</td>
<td>75.58</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>99.90</td>
</tr>
<tr>
<td>3</td>
<td>14.03</td>
<td>85.33</td>
</tr>
<tr>
<td>4</td>
<td>2.86</td>
<td>96.92</td>
</tr>
</tbody>
</table>

**Figure 17**: Microstructure of sample A206

3.4.4.3 Sample A214

Sample A214 from block 2 and reduced at 800ºC with high flow rate 1 L/min. Compare with normal situation, high flow rate gave high amount of CO content in whole reduction gas system which could cause high possibility of carbon deposition. Compare with sample A201, large area of FeO still did not be reduced and the white pure iron area was very small. It looked like that the reduction process was limited by the high flow rate. Carbon deposited more service on the surface of sample which blocked the reducing gases diffused inside the inner part. Thus, larger area of grey surfaces obtained in sample A214 grains.
### Table 11: Composition of sample A214

<table>
<thead>
<tr>
<th>Spectrum (A214)</th>
<th>Element (wt%)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>O</td>
<td>Fe</td>
</tr>
<tr>
<td>1</td>
<td>23.75</td>
<td>76.04</td>
</tr>
<tr>
<td>2</td>
<td>4.15</td>
<td>95.85</td>
</tr>
<tr>
<td>3</td>
<td>0.31</td>
<td>99.69</td>
</tr>
<tr>
<td>4</td>
<td>2.18</td>
<td>97.82</td>
</tr>
<tr>
<td>5</td>
<td>3.61</td>
<td>96.39</td>
</tr>
</tbody>
</table>

![Figure 18: microstructure of sample A214](image)

3.4.4.4 Sample A220

Sample A220 reduced under the same conditions as A206 but was from block 3. Much more dark area of samples than A206 in figure 19 which means the microstructure of samples strongly influence the production process. The reduced grains were bigger than samples from block 1 and 2. As the reduction reacted from out layer gradually, bigger size grain sample took longer time and needed more reducing gases contact with each grain. Combine with lower porosity between each grain, thus, all samples from block 3 have around 8 percentage of reduction degree lower gap with other samples.
Table 12: Composition of sample A220

<table>
<thead>
<tr>
<th>Spectrum (A220)</th>
<th>Element (wt%)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>O</td>
<td>Fe</td>
</tr>
<tr>
<td>1</td>
<td>24.04</td>
<td>75.96</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>3</td>
<td>0.89</td>
<td>99.11</td>
</tr>
</tbody>
</table>

Figure 19: Microstructure of sample A220

3.5 Reduction time of Butare iron lump ore

Not only reduction degree seems as the main feature to evaluate the reduction processes of iron ores, but also the reduction time. Nowadays, every factory for steelmaking wants the fast and great reduction process. Reduction time defined as the period that reduction of iron ore start until the weight of sample did not change during whole reduction process. Thus, the overall gas-solid reduction process is constituted by five steps:

- Transportation of Reducing gas into the surface of lump ore
- Chemical reaction of reducing gases and lump iron ore
- The reducing gases passed through iron and diffuse inward to the inner part.
- The product gases outwards from iron ores
- Reducing gas transport to outside
Consequently, the whole reduction process should be determined by two steps: the speed of chemical reaction and the diffusion process. If the temperature is not so high, the whole process could be controlled by chemical step, because of the high porosity and flow rate. Oppositely, high temperature cause bigger granularity then the diffusion step determined the total reduction periods. The slower step controls the whole process and generally, there are three reduction modes for iron ores:

- The first mode, while the iron ore is compacted structure that has low porosity, the reduced layer diffuse into the inner part gradually which indicated the middle unreduced part getting smaller and smaller.
- The second mode described the high porosity iron ores that reducing gas can diffuse inside easily and our samples are belongs to this reduction mode which already checked by SEM. Also in high temperature the diffusion step controlled whole process so that gas diffusion rate determined reduction time.
- If the soften temperature is low, the porosity getting very small while the sample reach the soften temperature. In this case, the reduction speed is decreased in the high temperature environment.

Normally, the main parameters effect on reduction time were carbon deposition and temperature which strongly influent the diffusion rate of reducing gases. Carbon deposited on the surface area strongly hindered the reducing gases diffusion.

Hereby, reduction reactions happened at 800 degree with 0.5 L/min seen as the normal situation. In figure 20 a), samples took longer time to complete reduction process with increasing sample weight. But figure 20 b) shown the opposite tendency as samples reduced at high flow rate. Checked by SEM, samples after reduction with high flow rate shown larger unreduced area and reduction degrees all were lower than normal situation. Also, samples reduced at high flow rate could see more carbon deposition on the surface layer while we picked out samples from reaction furnace. Relatively, samples at high flow rate reduction environments take shorter reduction time to finish whole processes since the surface carbon block the whole proceeding.

Why smaller size samples reduced at high flow rate takes longer time to finish reduction process in figure 20 b)?

- Specific surface area (SSA) is a very important parameters to check absorption and reaction happened on surface and it defines as surface area devide sample weight, in this case, smaller weight samples respectively have bigger value of SSA.
- As the high flow rate gave higher carbon depositon on surface which caused by high concentration of carbon monoxide gas, thus, smaller sample with high SSA will be blocked easier and took longer time to diffuse reducing gases inside. So, smaller samples took longer time to complete whole process.
Samples reduced at different temperature but with same flow rate 0.5 L/min shown opposite tendency again as normal situation in figure 21. Smaller pieces took longer time to complete reaction in 900°C environment.

- As the whole reduction process controlled by diffusion rate of gases, higher temperature caused lower porosity of sample, according to swelling of volume in samples then the porosity strongly reduced. So that reducing gases were difficult to diffusion into the middle part of materials.
- Smaller size samples with bigger value SSA were easier to get sinterskin on surface in high temperature (900°C) environment and sinterskin on the surface strongly blocked the reduction process.
- Samples reduced at 900°C shown many small cracks on the surface. While sample move down to reduction furnace, the uneven heat distribution of samples yielded cracks on bigger samples surfaces and leaded to gas free diffuse inside the samples which gave much higher reduction degree compare with smaller samples.

Figure 21: Temperature influence on reduction time
4. Conclusions

- Butare area iron lump ores can be reduced by direct reduction method and get relatively high reduction degree. The reduced sample kept its shape without breaking into small fragments at high temperature 900°C.

- Reduction degree of samples increases with reaction temperature. Samples reduced at high temperature 900 °C shown higher reduction degree in comparison with samples reduced at 700 - 850°C (the difference of reduction degree $\Delta RD$ is around 8 - 11%).

- At 900°C, H2 worked as the more effective reducing gas to give higher reduction degree and faster process.

- 0.5 L/min is the best flow rate for Butare iron lump ores compare with other flow rates ($\Delta RD \sim 8 - 10\%$).

- Sample weight influential reduction degree, under different reduction conditions, samples shown the best weight ranges. Although the reduction process occurred at its own best weight range, the different reduction processes still shown the difference $\Delta RD \sim 2.5 - 8\%$.

- Microstructure strongly influential reduction degree, smaller grain size samples reduced faster and more completely ($\Delta RD \sim 8\%$).

- Samples reduced at high flow rate 1L/min and high temperature 900°C took longer reduction time although the sample weight was smaller.

- Butare iron lump ores could get its best reduction degree at 900°C with 0.5 L/min flow rate.
5. **Future work**

- Improve experimental operations.
- Do more experiments on high temperature with high flow rate.
- Do more experiments on each parameter to ensure every tendency.
- Check the thermal degradation index of samples.
- Focus on microstructure influence.
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