

Kinetics of direct reduction of El-Baharia Iron ore (El-Gedida) Oasis Egypt, briquette via Hydrogen

N. A. El-Husseiny¹, Atef El-Amir², F.M.Mohamed^{1,3}, S. Th. Abdel-Rahim⁴, M. E. H. Shalabi^{1*}

1- Central Metallurgical Research and Development Institute (CMRDI), El-Tabbin, Helwan, Cairo, Egypt

2--Egyptian iron and Steel Company, Helwan ,Cairo, Egypt

3- King Khalid University, Faculty of science and Arts For Girls. Sarat Abida . Saudi Arabia

4-Chemistry Department, Faculty of Science, Ain Shams University, Cairo, Egypt

* Corresponding authors. Email: shalabimeh@hotmail.com

Abstract: El-Baharia iron ore is a source of iron in iron and steel Co. in Egypt. This iron ore will be used for producing iron by direct reduction using hydrogen as a reducing agent. Briquettes were the form to be reduced under several conditions of changing hydrogen flow rate, and temperature.

Keyword: Iron ore, Reduction by hydrogen, Kinetic reduction model, briquette of iron ore

Introduction

Roohollah et al (2011) [1], indicated that in the nature, it is infeasible to find pure iron itself because it has a high tendency to react with oxygen. In general, it can find iron as iron oxide with other materials like: silicon, sulfur, phosphor, and manganese. Iron oxide with all other materials which exists in the nature calls iron ore or iron stone and is usually found in the form of magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite ($\text{FeO}(\text{OH})$), limonite ($\text{FeO}(\text{OH}) \cdot n(\text{H}_2\text{O})$) or siderite (FeCO_3).

Damien et al (2006)[2], indicated that the reduction of iron ores by hydrogen is a gas-solid reaction which occurs in two or three stages. For temperatures higher than 570°C , hematite (Fe_2O_3) is first transformed into magnetite (Fe_3O_4), then into wustite (FeO), and finally into metallic iron whereas at temperatures below 570°C , magnetite is directly transformed into iron since wustite is not thermodynamically stable.

Pineau et al (2006) [3], studied the reduction of Fe_2O_3 by H_2 in the temperature range of 220 – 680°C

to examine the rate controlling processes of this reaction in the widest and lowest possible temperature range. This is to be related with efforts to decrease the emission of CO_2 in the atmosphere thus decreasing its green house effect.

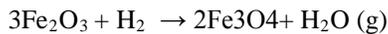
Reduction of hematite to magnetite with H_2 is characterized by an apparent activation energy ' E_a ' of 76 kJ/mol. E_a of the reduction of magnetite to iron is 88 and 39 kJ/mol for temperatures lower and higher than 420°C , respectively. They found that the mathematical modeling of experimental data suggests that the reaction rate is controlled by two- and three-dimensional growth of nuclei and by phase boundary reaction at temperatures lower and higher than 420°C , respectively.

Morphological study confirms the formation of compact iron layer generated during the reduction of Fe_2O_3 by H_2 at temperatures higher than 420°C .

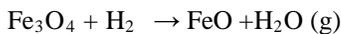
Pineau et al, (2007)[4] studied the reduction of Fe_3O_4 by H_2 in the temperature range of 210 – 950°C . In this work two samples of Fe_3O_4 produced at 600 and 1200°C , designated as $\text{Fe}_3\text{O}_4(600)$ and $\text{Fe}_3\text{O}_4(1200)$, have been used as starting material. Reduction of $\text{Fe}_3\text{O}_4(600)$ by H_2 is characterized by an apparent activation energy ' E_a ' of 200 , 71 and 44 kJ/mol at $T < 250^\circ\text{C}$, $250^\circ\text{C} < T < 390^\circ\text{C}$ and $T > 390^\circ\text{C}$, respectively. The important change of E_a at 250°C could be attributed to the removal of hydroxyl group and/or point defects of magnetite. This is confirmed during the reduction of $\text{Fe}_3\text{O}_4(1200)$, While transition at $T \approx 390^\circ\text{C}$ is probably

due to sintering of the reaction products as revealed by SEM. In situ X-rays diffraction reduction experiments confirm the formation of stoichiometric FeO between 390 and 570°C. At higher temperatures, non-stoichiometric wüstite is the intermediate product of the reduction of Fe₃O₄ to Fe. The physical and chemical modifications of the reduction products at about 400 °C, had been confirmed by the reduction of Fe₃O₄ (600) by CO and that of Fe₃O₄ (1200) by H₂. A minimum reaction rate had been observed during the reduction of Fe₃O₄ (1200) at about 760 °C. Mathematical modeling of experimental data suggests that the reaction rate is controlled by diffusion and SEM observations confirm the sintering of the reaction products.

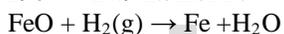
Raymond and Leiv (2008)[5] indicted that reduction of iron oxide with H₂ occurs in three steps when temperatures are over 570°C and two steps when the temperature is below 570°C:



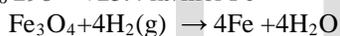
$$\Delta H_{0, 298} = 1.0 \text{ kJ/mol Fe}$$



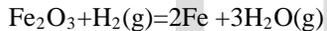
$$\Delta H_{0, 298} = +24.9 \text{ kJ/mol Fe}$$



$$\Delta H_{0, 298} = +25.4 \text{ kJ/mol Fe}$$



$$\Delta H_{0, 298} = +50.4 \text{ kJ/mol Fe}$$



$$\Delta H_{0, 298} = +49.4 \text{ kJ/mol Fe}$$

Asima and Itisree(2008) [6] indicated that reduction kinetics of iron ore reduction deals with the rate at which iron oxide is converted to metallic iron by removal of oxygen. This influences production rate of the process, which ultimately determines the economic feasibility and competitiveness of the technologies involved. The reduction of the iron oxides takes place in a series of sequential steps. The overall rate will be determined by the slowest of the process or processes in the series. The possible consecutive steps are:-

- i. Transport of gaseous reductant from the bulk gas phase to the particle surface through a boundary gas film;
- ii. Molecular diffusion of the gaseous reductant through the product layer to the reaction interface.
- iii. Adsorption of the gaseous reductant at the interface.
- iv. Reaction at the interface (reaction between adsorbed reductant and oxygen of the lattice).
- v. Desorption of the gaseous products from the interface.
- vi. Mass transport of iron and oxygen ions and transformations in the solid phase; formation and

growth of the reaction products, viz. magnetite, wüstite and iron.

vii. Molecular diffusion of gaseous products through the product layer to the particle surface.

viii. Transport of the gaseous products from the particle surface through the boundary gas film to the bulk gas phase.

The rate limiting cases are chemical control (steps iii to vi) and diffusion control (steps I & viii ; ii ; vi & vii).

More than 90% of iron is currently produced via the blast furnace (BF) process, while the balance is produced by the direct reduction (DR) processes [7]

Moo (2010) [8] , showed that the reduction rate of iron oxide was fast enough to obtain 90-99% reduction within 1-7 seconds at 1200-1500°C, depending on the amount of excess hydrogen supplied with iron oxide and this indicates that a high metallization degree within a few seconds of residence time is feasible for a suspension process.

Haitao (2011)[9] said that the kinetics feasibility tests showed that 90 - 99% reduction of iron ore concentrate by hydrogen was obtained within 1 - 7 seconds at 1200 - 1400 °C, depending on the amount of excess hydrogen supplied with iron oxide. This reduction rate is fast enough for a flash reduction process. And also the reduction rate has 1/2-order dependence on the partial pressure of hydrogen, which suggests that an increase in partial pressure of reducing gas has a more moderate effect on reduction rate than a typical 1st-order reaction. He also indicated that the reduction rate increased with the particle size when reduction temperature is lower than 1300 °C. The effect of particle size is negligible at temperatures; also the activation energy of hydrogen reduction of iron ore concentrate was determined to be 463kJ/mol demonstrate that this process has greater temperature effect on the reduction rate than most reactions.

The aim of this work is study the reduction of The El-Baharia Egypt iron ore *briquette* by hydrogen.

2- Experimental Work

2.1. Materials and sample characterization

El-Baharia Egypt iron ore used in this study was obtained from iron and Steel Co. Helwan Egypt. . After drying and grinding, the particle size of concentrate was 5.03-64.79 μm. The chemical composition of ore is as follows:- Fe total = 52.35 %, MnO= 2.92%, SiO₂= 10.84%, CaO= 0.39%, MgO= 0.18%, Al₂O₃= 1.44% , S= 0.74%, TiO₂= 0.16% , BaO= 1.17%, ZnO= 0.15%, K₂O= 0.27%, Na₂O= 0.25%, P₂O₅ = 0.5 %.[10]

The X- Ray analysis of El-Baharia iron ore is illustrated in figures 1. From which it is clear that El-Baharia iron ore mainly consists of hematite and quartz. [10]

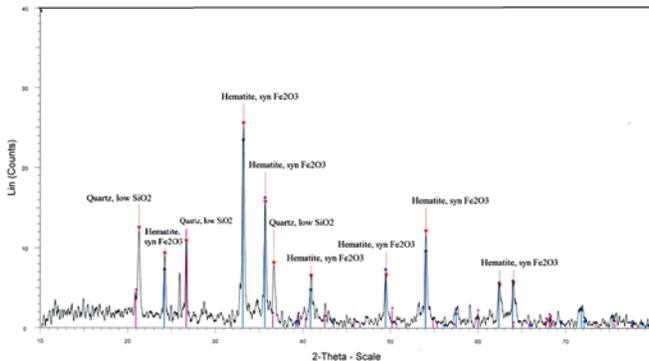


Fig.1. X-ray analysis of El-Baharia iron ore

2.2 Preparation of the briquettes and its physical properties

The iron ore was ground in a vibrating mill to a size less than 75 micro- meters. The iron ore powder (10 gm) are mixed with 2% molasses and then pressed in the mould (12 mm diameter and a height 22 mm using MEGA.KSC-10 hydraulic press Figure 2 [10], under different pressure (the pressure range from 75 MPa up to 275 MPa). The produced briquettes were subjected to drop damage resistance test and compressive strength tests. The drop damage resistance indicates how often green briquette can be dropped from a height 46 cm before they show perceptible cracks or crumble. Ten green briquettes are individually dropped on to a steel plate. The number of drops is determined for each briquette. The arithmetical average values of the crumbling behavior of the ten briquettes yield the drop number [10-14]. The average compressive strength is done by compressed 10 briquettes between parallel steel plates up to their breaking [15]



Fig.2. MEGA.KSC-10 hydraulic press

2.3. Reduction process

Reduction of the produced iron ore briquettes was performed in a thermo balance apparatus shown in Figure 3 [11-19]. It consisted of a vertical furnace, electronic balance for monitoring the weight change of reacting sample and temperature controller. The sample was placed in a nickel chrome basket which was suspended under the electronic balance by Ni-Cr wire. The furnace temperature was raised to the required temperature (600°C– 950°C) and maintained constant to $\pm 5^\circ\text{C}$. Then samples were placed in hot zone.

At initial time of experiments air should be removed from the reduction tube before each experiment and also after the end of reduction by nitrogen (0.5 l/min) during all the experiments. During the experiments the weight of the sample was continuously recorded at the end of the run; the samples were withdrawn from the furnace and put in the desiccators.

The percentage of reduction was calculated according to the following equations:

$$\text{Percent of reduction} = \frac{[(W_o - W_t) / \text{Oxygen (mass)}] \times 100}{100}$$

Where:

W_o: the initial mass of iron ore sample after removal of moisture.

W_t: mass of sample after each time of the reduction, t.

Oxygen (mass): indicates the mass of oxygen percent in the sample in form FeO & Fe₂O₃

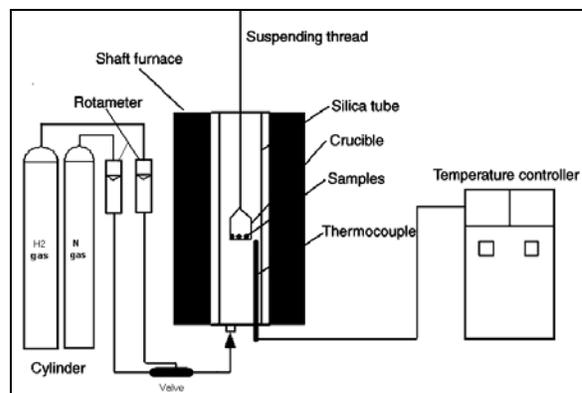


Figure 3. Schematic diagram of the apparatus

3-Results and discussions

3.1. Effect of pressing pressure on physical properties of the produced briquettes (green and dry samples).

Figs 4-7 show the relation between the change of pressing pressure load at constant amount of molasses (2%) on the drop damage resistance and compressive strength of the wet and dry briquette. These figures are evident that as the pressing pressure load increased both the drop damage resistance and compressive strength increased. This may be due to the fact that increase pressure leads to increase the compaction of briquette and subsequently the Vander Waals forces increased [15, 17-18], also this may be due to the increase of briquetting pressure leads to progressive crushing of the macro pores [15, 19].

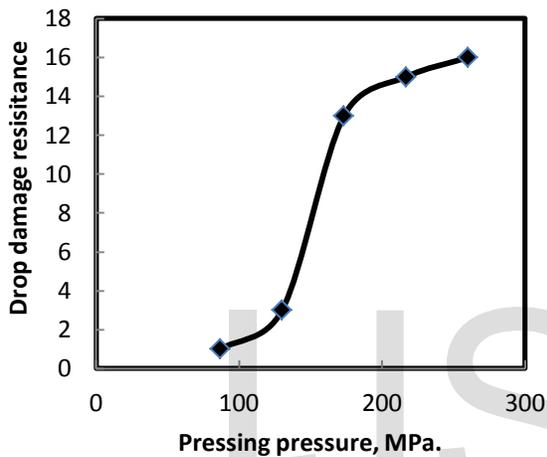


Figure 4. Effect of change of pressing pressure on the drop damage resistance of produced green briquettes.

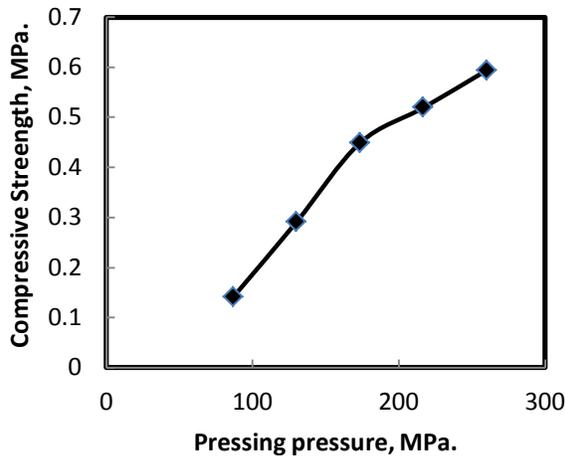


Figure 5 Effect of change of pressing pressure on the compressive strength of produced green briquettes.

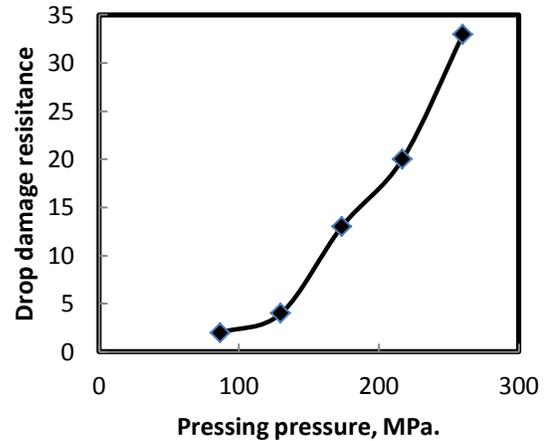


Figure 6 Effect of change of pressing pressure on the drop damage resistance of produced dry briquettes.

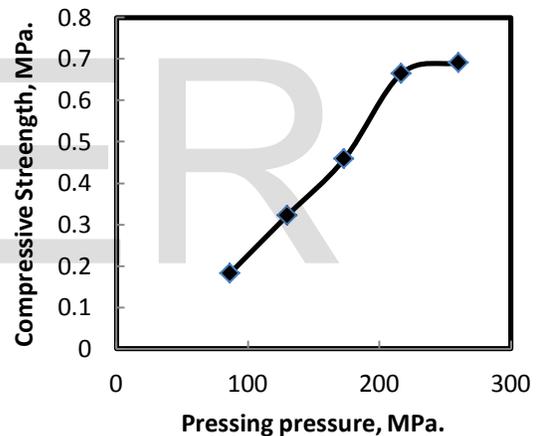


Figure7. Effect of change of pressing pressure on the compressive strength of produced of dry briquettes

3.2. Effect of pressing pressure on the reduction degree of produced briquettes.

The effect of pressing pressure load of the briquetting process on the reduction of the produced briquettes was studied at constant weight of the briquette sample, constant flow hydrogen rate 1.5 l/min and constant reduction temperature 900 °C, and the results are shown in Fig.8. From which it is evident that as the compression load increases up to 260.09 MPa. , the reduction percentage increases. This may be due to the increase of pressing load leads to decrease the micro pores subsequently the diffusion rates relatively increased.

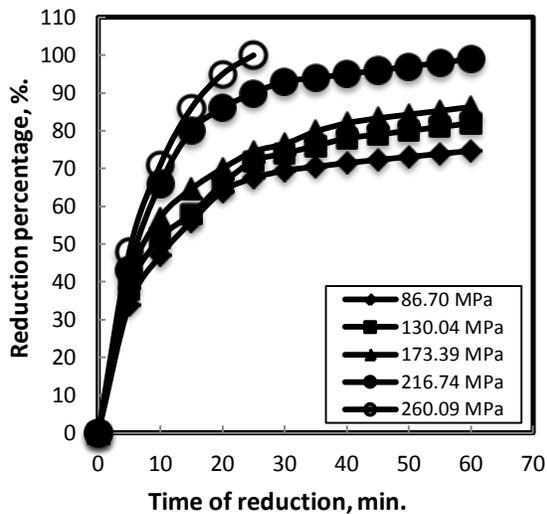


Figure 8 Relationship between time of reduction and the percentage of reduction under different pressing pressure load and at 1.5 l/min. flow hydrogen rate.

3.3. Effect of different hydrogen flow rate on the percentage of reduction of the pressed briquetted at constant reduction temperature 900 °C.

Fig. 9 illustrates the relation between the reduction percentage and hydrogen flow rate when the reduction was done at constant temperature (900°C) and constant weight of the sample. It is clear that as the flow rate of hydrogen increased the reduction percentage increased. This may be the increase of flow rate leads to an increase of number of hydrogen mole in the bulk phase, which in turn leads to the raise of hydrogen adsorption and subsequently the rate of reaction increased [15, 20] or the increase of flow rate increased the gas diffusion across the boundary layer subsequently the reduced ion increased [21]. Also may be the higher flow rate prevailing in the reaction zone which enhances the rate of hydrogen absorption and subsequently the rate of chemical reaction steps increased [22]. But from the economic view, it is preferred to use 1.5 l/min. hydrogen flow rate in the rest of this investigation.

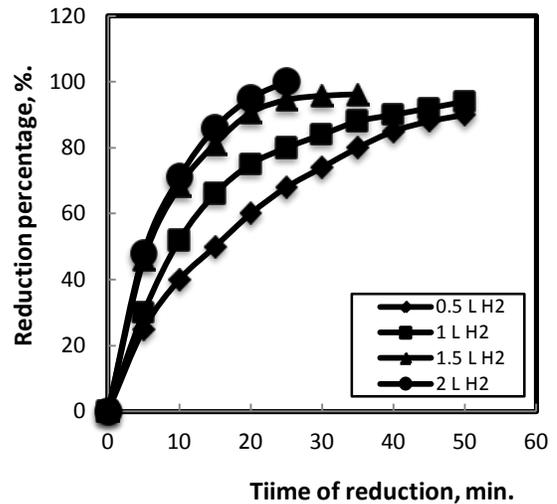


Figure 9 Relationship between time of reduction and the percentage of reduction at different hydrogen flow rate and at constant reduction temperature 900 °C.

3.4. Effect of different reduction temperature on the percentage of reduction of the pressed briquetted sample at 1.5 l/min hydrogen flow rate.

The effect of temperature on the reduction process was studied and the results shown in Fig. 10 plots of the reduction percentage of iron ore reduction by H₂. From this figure, it can be seen that temperature influences the reduction degree. With temperature increasing, the reduction degree increases. The increase of reduction percentage with rise of temperature may be due to the increase of number of reacting moles having excess of energy which leads to the increase of reduction rate (23-24). Also the raise of temperature leads to an increase of the rate of mass transfer of the diffusion and rate of desorption (15, 25).

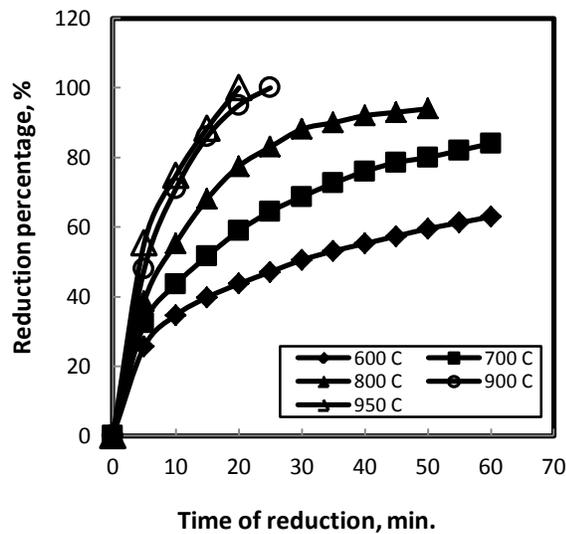


Figure 10 Relationship between time of reduction and the percentage of reduction of the pressed briquetted sample at 1.5 l/min hydrogen flow rate and at different reduction temperature.

3.5. Kinetics of the reduction process of iron ore briquette.

Kinetic studies for estimation the apparent activation energies were carried out for the reduction of samples at five different temperatures 600,700,800, 900 and 950°C for different time intervals in the range of 0 – 60 minutes.

A large number of reaction models have been formulated. These kinetic models include:-

1- Diffusion through thin ash layer (jander equation):-

$$(1-(1-f)^{1/3})^2 = kt$$

2-Diffusion controlled:-

$$1-2/3f-(1-f)^{2/3} = kt$$

3-Diffusion through ash layer (crank-cinslling-Broushten equation):-

$$Kt=(1-3(1-f)^{2/3} +2(1-f))^{1/3}$$

The reduction behavior of the pellets shown by graph was plotted between $\ln t$ and $1/T$. Arrhenius equation was used to calculate the activation energy. By applying several kinetic models on the reduction process the following results was obtained.

Figs. 11-16, illustrate the reaction kinetics of the reduction reaction controls models. From these figures it is clear that the graph nearly straight lines.

To calculate the activation energy, graph was plotted between $\ln t$ and $1/T$ to calculate according to Arrhenius equation as follows:-

$$K = Ae^{-E/RT}$$

Or

$$\ln K = \ln A - E/RT$$

Where;

K = Reaction rate constant

A = Frequency factor

E = Activation energy

T = Temperature (°K)

R= Universal gas constant

The plot of $\ln k$ versus $1/T$ results in a straight line.

The slope of the plot is $-E/R$. So activation energy can be obtained by multiplying slope $\times R$.

The value of E_a is -62.6KJ/mole for Diffusion through thin ash layer (jander equation (Fig. 12), 46 KJ/mole for Diffusion controlled Fig. 14 and 45.2 KJ/mole for the Diffusion through ash layer (crank-cinslling-Broushten equation) Fig.16.

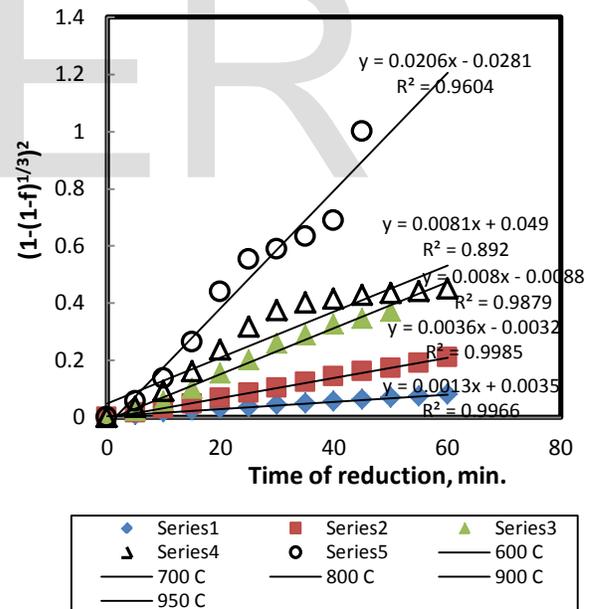


Fig.11 Relation between $[1-(1-f)^{1/3}]^2$ (jander equation) and time of iron ore reduction by hydrogen

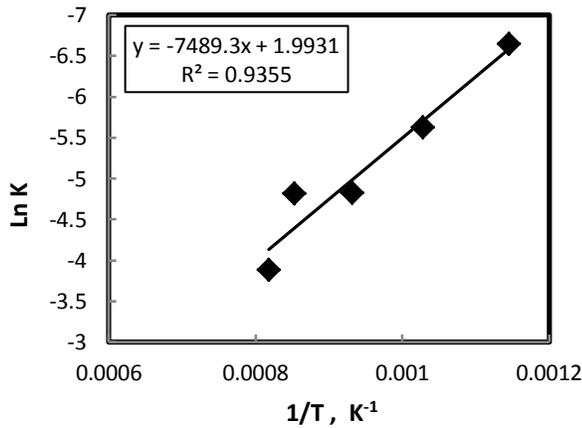


Fig.12 Relation between $\ln k$ and $1/T$ for the models of Diffusion through thin ash layer $[1-(1-f)^{1/3}]^2$ (jander equation):-

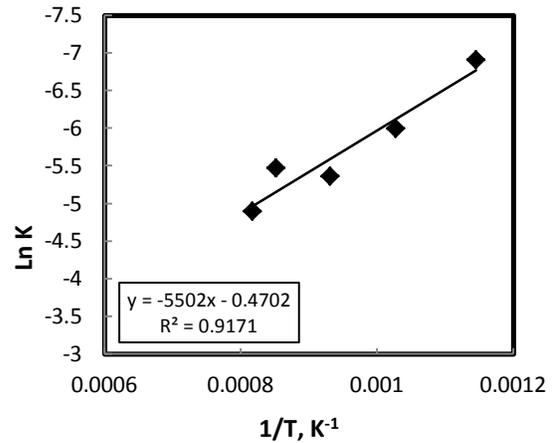


Fig.14 Relation between $\ln k$ and $1/T$ for the models of Diffusion controlled

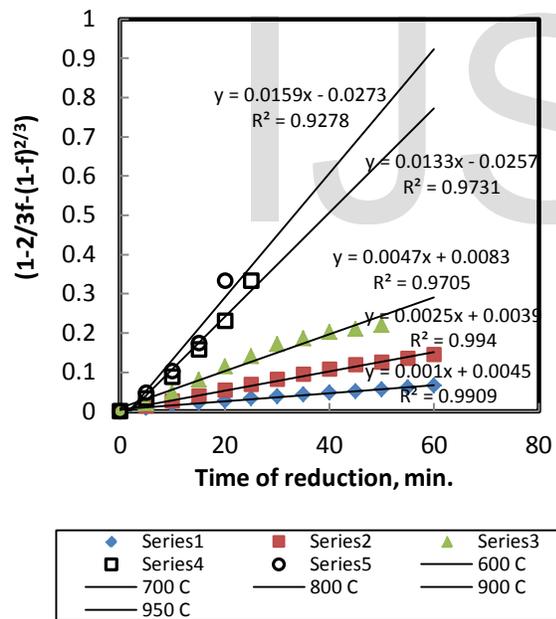


Fig. 13 Relation between $(1-2/3f-(1-f)^{2/3})$ Diffusion controlled and time of iron ore reduction by hydrogen

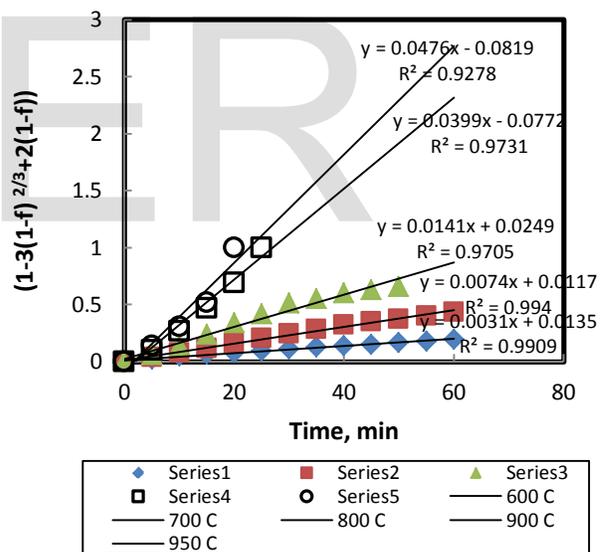


Fig.15. Relation between $(1-3(1-f)^{2/3}+2(1-f)^{2/3})$ (crank-insling-Broushten equation) and time of iron ore reduction by hydrogen

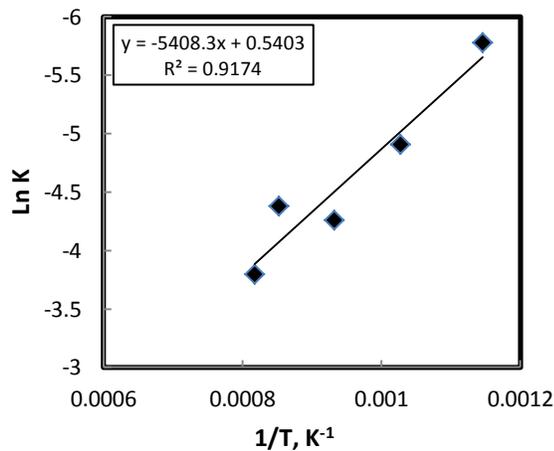


Fig.16 Relation between $\ln k$ and $1/T$ for the models of Diffusion through ash layer (crank-cinslling-Broushten equation)

3.6. X-ray diffraction of the reduced iron ore

Fig.17 and 18 illustrated the reduction of iron ore by hydrogen at 600 and 900°C respectively from these figures it is clear that the synthetic Fe increased as the temperature increased. Also it is clear that the magnetite is more in the sample reduced at 600°C than 900°C.

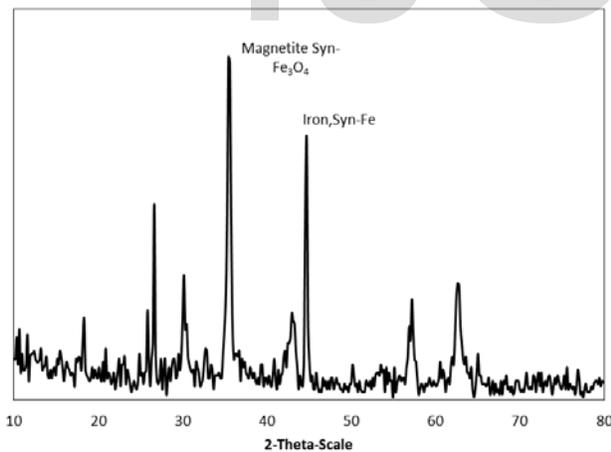


Fig.17. X-ray of the reduce sinter by hydrogen at 600°C.

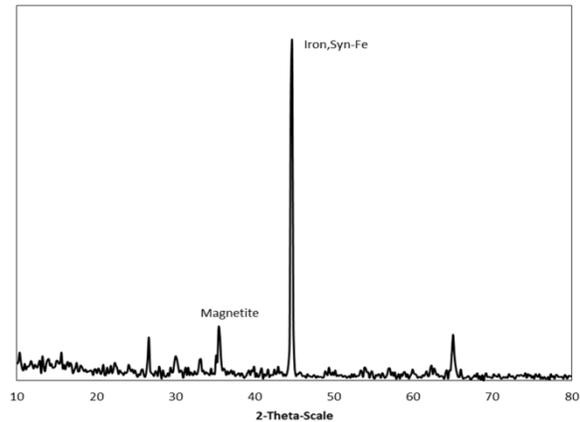


Fig.18. X-ray of the reduce sinter by hydrogen at 900°C.

4. Conclusions

- 1- The reduction of iron ore briquette by hydrogen depends on the flow rate of hydrogen and temperature of the reduction pressure of the briquetting.
- 2- As the temperature increased the reduction increased.
- 3- As the flow rate of hydrogen increased the reduction rate increased
- 4- The reduction of the iron ore briquette is controlled by one of the following models:-
 - a- Diffusion through thin ash layer (jander equation)
 - b- Diffusion controlled
 - c- Diffusion through ash layer (crank-cinslling-Broushten equation)

5. References

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