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Study on reducing briquettes of El-Dekhaila iron oxide waste by hydrogen gas

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Abstract: Reduction of El-Dekhaila iron oxide waste briquettes was carried out in the temperature range 700 to 950 oC. In reduction kinetic study the most satisfactory model was to take the slope of the initial linear region of fractional reduction vs. time curve as a measure of rate constant (k). In k vs. 1/T plots were straight line from which Activation Energy was calculated.

Key words: El-Dekhaila iron oxide waste briquette, Reduction by hydrogen, kinetic reduction model, energy of activation

1- Introduction

El-Dekhaila iron Co. imported the pellets from the outside of Egypt, during the transportation from outside to Company, a lot of fine (waste pellets fine) formed, this fine was not suitable for the reduction inside the furnace of reduction.

Asima and Itishree [1] indicated that the blast furnace is used mainly for pig iron production all over the world. Thus because it has very high production rate and also greater degree of heat utilization to a remarkable extent as here counter current heat exchange principle is utilized. The reduction of iron oxides via gaseous and solid reductant has already been extensively studied [2].

Damien et al [3] concluded 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 ($Fe_{1-y}O$), and finally into metallic iron whereas at temperatures below 570°C, magnetite is directly transformed into iron since wustite is not thermodynamically stable.

Jouhart et al [4] indicated that all the iron bearing wastes generated at the plant site can be recovered in sinter charge mix. Fines of iron oxide pellets are characterized by high iron oxide and low silica content.

Mohamed et al [5] concluded that some iron oxide waste characterized by high iron oxide content such as El-Dekhaila iron oxide pellets waste can be recycled during the sintering of iron concentrate. The results show that, replacement of iron ore concentrate with 10% iron oxide pellets fine increases the amount of readymade sinter, sinter strength and productivity of both sinter machine and blast furnace yard.

This work aimed briquetting the iron oxide pellets waste which present in El-Dekhaila iron Co. and reduced it in static bed by hydrogen (hydrogen is best as a reductant and/or fuel from the environmental and reduction kinetics points of view) .

2-Experimental Work

2.1. Raw material

El-Dekhaila waste pellets which used in this work was delivered from El-Dekhaila steel Company (Alexandria, Egypt) the chemical analyses of this fine are, Fe total = 66.5%, Fe_2O_3 = 95%, SiO_2 = 1.5% and CaO = 0.7%. X-ray of the El-Dekhaila pellets waste illustrated in Fig.1, from which it is clear that the main compound of this waste is hematite.

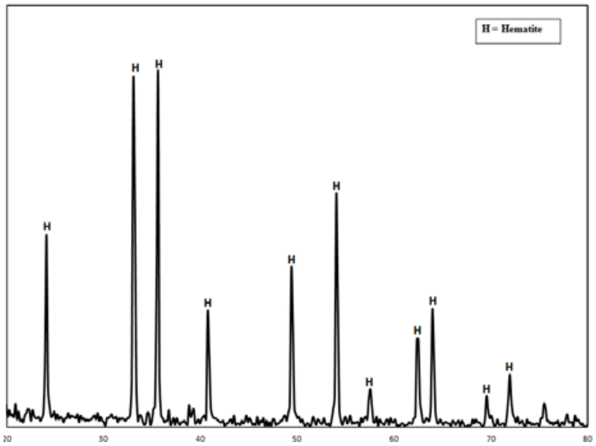

Fig.1. X-ray of El-Dekhaila Pellets waste

Fig.2 MEGA.KSC-10 hydraulic press

2.2. Preparation of the Briquetting and Its Physical Properties

El-Dekhaila waste pellets were grinding in vibrating mill to powder with size less than 75 micrometers. The fine of waste powder (10 g) are mixed with 3% molasses and then pressed in the mould (12 mm diameter and height 22 mm using MEGA.KSC-10 hydraulic press) as in Fig. 2 [6] under different pressure (the pressure range from 75 MPa up to 275 MPa).The briquette subjected to drop number test and crushing strength tests. The drop number 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 crumbing behavior of the ten briquettes yield the drop number .The average crushing strength is done by compressed 10 briquettes between parallel steel plates up to their breaking [7].

2.3. Reduction Procedures

The reduction of El-Dekhaila by hydrogen were done on thermo gravimetric apparatus (A schematic diagram of thermo gravimetric apparatus is shown in Fig. 3 [6, 8 -13]. 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 Ni-Cr basket which was suspended under the electronic balance by Ni-Cr wire. The furnace temperature was raised to the required temperature (700-950 ^{o}C) and maintained constant to ± 5 ºC. Then samples were placed in hot zone. The nitrogen flow rate was 0.5 l/min on all the experiments. At initial time and after the end of reduction only the weight of the sample was continuously recorded at the end of the run, the samples were withdrawn from the furnace and putted in the desiccators. The amount of removable oxygen was determined by the weight loss in the sample (Wo-W) during the experiment of reduction with H_2 in the furnace .The percentage of reduction was calculated according to the following equations [14-15]:-

Percentage of reduction = [(Wo-Wt) / Wo] *100 ------------------------- (1)

Where Wo the initial mass of sample.

Wt mass of sample after each time, t.

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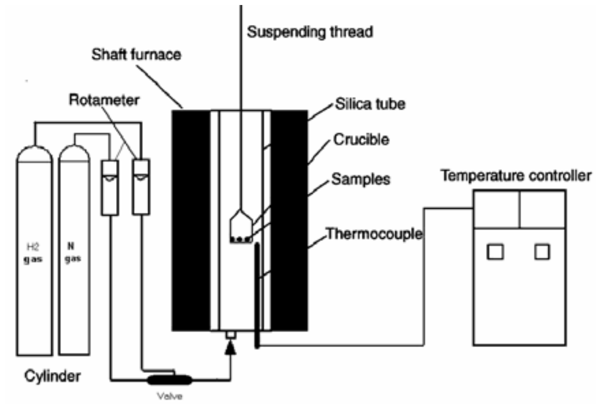

Fig.3. Schematic diagram of the apparatus

3. Results and discussion

3.1. Effect of pressing load on the quality of the produced briquettes

The drop damage resistance and compressive strength of the produced briquettes with respect to different pressing load and at constant amount of molasses (3%) are shown in Figures 4-7. From these figures, it was found that as the pressing load increased from 87 to 261 MPa. The drop damage resistance and the compressive strength for both green and dried briquettes (drying time 3 days) increased and reached to its maximum values at 261 MPa. This could be attributed to the fact that increasing pressing load leads to increase the number of contact points between particles and subsequently the Vander Waals force increased [9, 16-18].

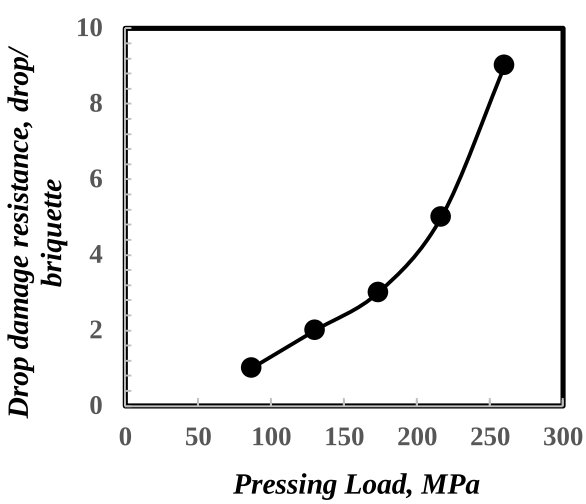

Fig.4 Relation between drop number of the green briquette and pressing load.

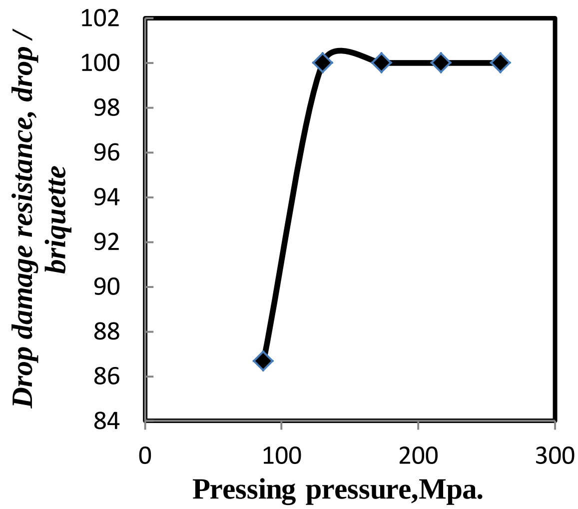

Fig.5 Relation between drop number of the dried briquette after 3 day and pressing load

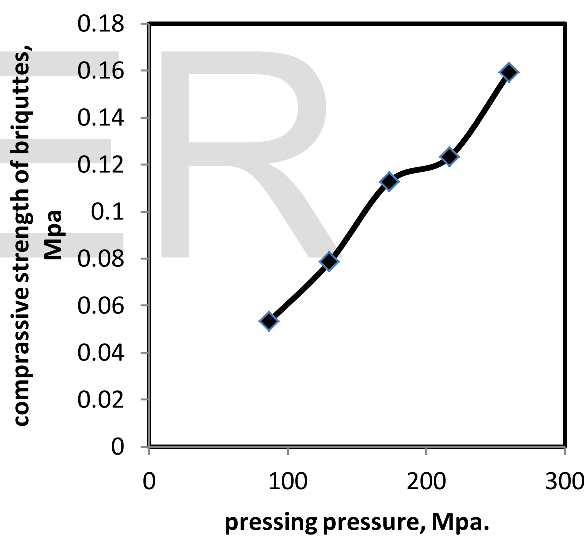

Fig.6 Relation between the strength of the green briquette and pressing load

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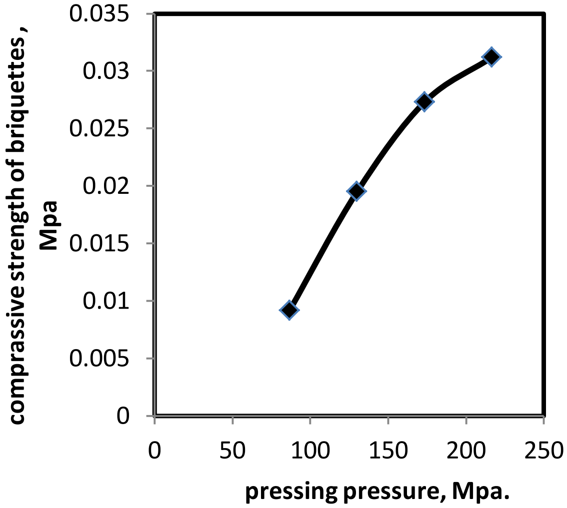

Fig.7 Relation between the strength of the dry briquette after 3 day and pressing load

3.2 Effect of hydrogen flow rate on the reducibility of the El-Dekhaila waste briquettes at 900°C

Figure 8 shows the effect of different flow rate on the percentage of reduction of El-dekhaila iron oxide waste when the reduction were done at constant temperature (900°C) , the weight of the sample was constant and this sample was pressed at 261 MPa. . It is clear that as the flow rate of hydrogen increased the reduction percentage increased. This may be due to the fact that increase of flow rate leads to increasing the number of hydrogen moles in the bulk phase, which in turn leads to the raise of hydrogen adsorption and the rate of reaction increased (13, 18-19) or the increase of flow rate increased the gas diffusion across the boundary layer (13, 18 -20). 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(18, 21) .

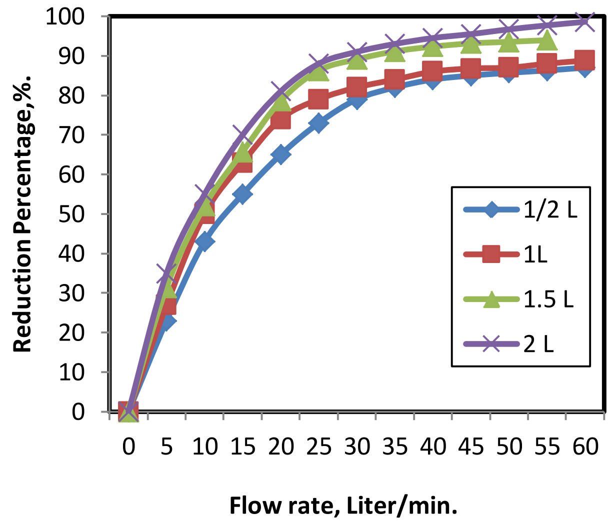

Fig. 8 - Effect of hydrogen flow rate on the reduction of El-Dekhaila waste briquette

3.3- Effect of temperature change on the reduction percentage

In order to examine the effect of temperature on the reduction of El-Dekhaila waste briquette by 2 L/min hydrogen flow rate, experiments were carried out at 700 – 950°C. Plots of the reduction percentage as function of time are shown in Fig. 9. From this figure it is observed that the reduction temperature influences significantly the reduction percentage.

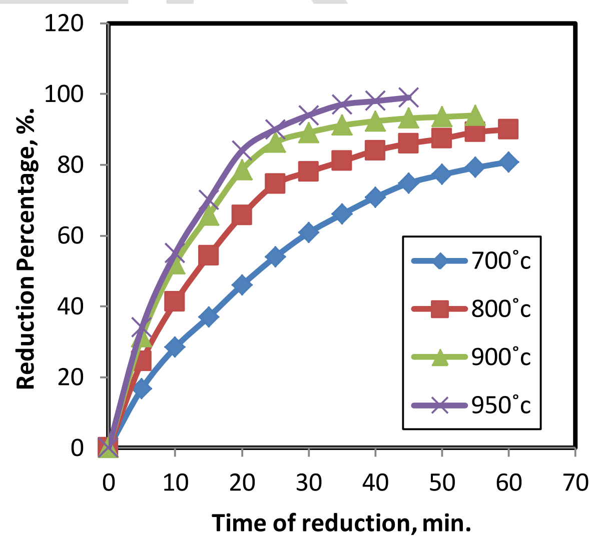

Fig.9.Effect of reduction temperature on the reduction of El-Dekaila waste briquette

3.4. Kinetics reduction of briquette

Kinetic studies for estimation the apparent activation energies were carried out for the briquettes at different temperatures range from 700˚C up to 950˚C for different time intervals in the range of 0 - 60 min. Using diffusion process control equation (Jander and Anorg Equation)[22-24]

$$[1 - (1-R)^{1/3}]^2 = kt \qquad \text{-----------------------------(2)}$$

Where R is fractional reduction, t is time of reduction, k is the rate constant.

Fig.10 illustrate the relation between $[1 - (1-R)^{1/3}]^2$ against time of reduction for different reduction temperature. From which it is clear that the straight line was observed. The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of reduction reaction. The results illustrate in Fig.11, from which it is clear that the activation energy= 36.12 kJ/ mole.

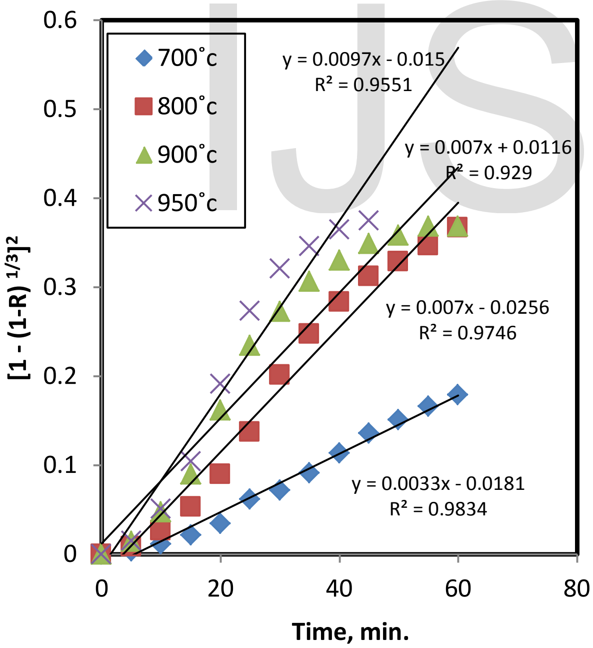

Fig. 10 The relation between $[1 - (1-R)^{1/3}]^2$ and time of reduction

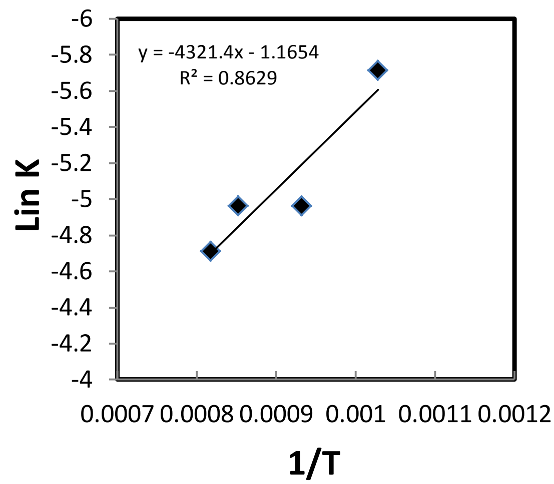

Fig.11 Relation between ln k and 1/T

3.5 X-Ray analyses of the reduced briquettes

Figs. 12 and 13 show the phases produced after reduction by hydrogen at 700 and 950 ^{o}C. From these figures it is clear that at temperature 950 ^{o}C the reduction is nearly completed and all hematite nearly converted to metallic iron, the present of magnetite in x- ray may be due to the secondary oxidation of the sample, while the reduction at 700 ^{o}C did not complete and the ore converted to metallic iron, magnetite and wustite.

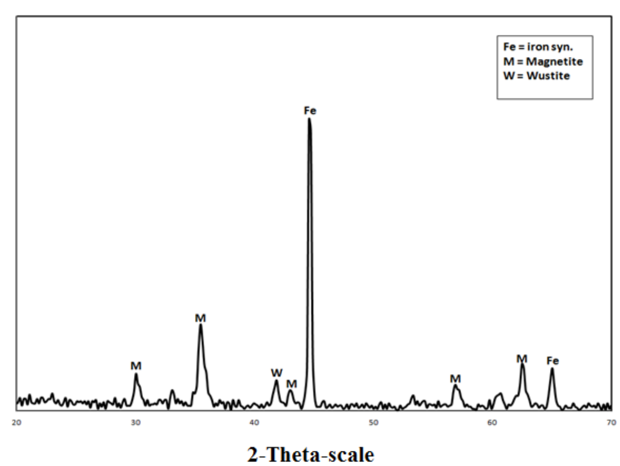

Fig.12 X ray analyses of the sample reduced by hydrogen at 700 ^{o}C

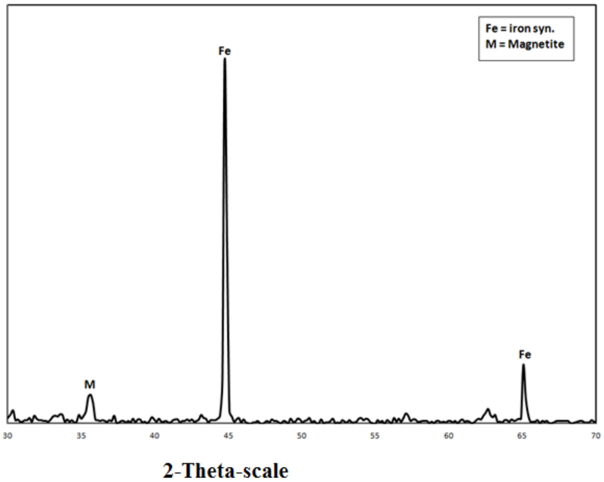

Fig.13 ray analyses of the sample reduced by hydrogen at 950 ^oC

4- Conclusions

1) The compressive strength and the drop damage resistances of briquettes increased with increasing the pressing pressure up to 260.09 MPa. at 3% molasses.
2) The reduction rates increased with increasing temperature of the reduction from 700 up to 950°C.
3) The reduction rate increased with increased of hydrogen flow rate at constant temperature.
5) The diffusion processes through the produced briquettes is the reduction control step and the briquettes have activation energy = 36.12 kJ/ mole.

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