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Kinetics of Direct Reduction El-Baharia (Egypt) iron ore pellets in static bed via Hydrogen

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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 of iron ore pellets using hydrogen as a reducing agent. Pellets reduction was examined under different conditions of changing hydrogen flow rate, and temperature ranging from (600^{o}C to 950^{o}C). The results showed that increasing both flow rate and temperature affect positively on reduction rate. The reduction kinetics was studied and it proved that the reduction controlling step was diffusion through thin ash layer with activation energy of 60.55 KJ/mole.

Key words —Iron ore, Reduction by hydrogen, Kinetic reduction model, pelletization of iron ore.

1 Introduction

Iron is believed to be the tenth most abundant element in the uiverse, and the fourth most abundant in the earth's crust. Iron is the most used of all the metals, comprising 95% of all the metal tonnage produced worldwide. Iron is extracted from its ore, and is almost never found in the free elemental state. In order to obtain elemental iron, the impurities must be removed by chemical reduction [1, 2].

The Egyptian iron ores of El-Baharia Oasis is the main feedstock for the blast furnace of Egyptian iron and steel Co.

Hydrogen is bestreductant and/or fuel from the environmental and reductionkinetics points of view, but it is currently expensive.

The pelletization of fine ores as well as the quality of produced pellets depend on many factors such as feed particle size, amount of water added during pelletization, disc rotating speed, inclination angle of the disc bottom and residence time of materials in the disc. The effect of these parameters on the mechanical properties of green, dried and fired iron ore pellets has been the subject of much research [3-5].

Pelletization is one of the agglomeration processes which converts the fines into pellets ofsuitable size. Binders are important for holding the fine particles together during thepelletization process. Either organic or inorganic binders can be used. Organic binders burn orvolatilize during movement of the flame front. Owing to the comparatively high prices of binders the only interest becomes feasible is that of using waste products such as molassesthat is both cheap and locally available [6-7].

Damien et al (2006)[8], 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 (Fe O), and finally into metallic iron, whereasat temperatures below 570°C magnetite is directly transformed into iron since wustite is not thermodynamically stable

Moo Eob Choi [9] and Haitao Wang [10]indicated that the kinetics feasibility tests showed that 90 - 99% reduction of iron oreconcentrate by hydrogen was obtained within 1 - 7 seconds at 1200 – 1400^o C, dependingon the amount of excess hydrogen supplied with iron oxide. This reduction rate is fast enough for a flash reduction process. The activation energy of hydrogen reduction of iron ore concentrate was determined to be 463 kJ/mol, which demonstrates that this process has greatertemperature effect on the reduction rate than most reactions.

Also it was found thatusing pure hydrogen as reducing agent gave a higher extent ofreduction than a mixture of CO-H2. Sulphur and phosphorus are partially removed in gaseous formfrom the ore; within the temperature range examined, sulphur removalincreased with increase in temperature, whereas phosphorus removalwas favoured at lower temperature [10].

Ezz and Wild [11] indicated that an increase in temperature exerts a major influenceon increasing reduction rate, while ore characteristics, such as porosity, shape factor, andsurface condition also affected reduction rate also the ore/gas ratio has a major influence on the reductionrate.

Rajnish Kumar [12] illustrated that 1-The Strength of hematite iron ore pellets vary with the variation of binder content. 2) Degree of reduction increases with increase in reduction temperature due to more diffusion of reducing gases in the pellet matrix. 3) The degree of reduction increases with increase in time at a particular reduction temperature.

Asima and Itishree[13]indicated that thatthe 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;

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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, wustite 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)

El-Husseiny et al [14] found that: - 1- The reduction of El-Baharia 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.

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

2 Experimental Work

2.1 Raw materials

Iron ore sample was obtained fromthe Egyptian Iron and Steel Company, The chemical analysis of such materials was performed by XRF
Phase identification illustrated in figures 1 was performed using Philips type 1373 x-ray diffractometer; it is clear that El-Baharia iron oreis rich in hematite and quartz. [15]

2.2 Preparation of the pellets and Its Physical Properties

Iron ore was grinding in vibrating mill to powder with size less than 75 micrometers. After which thepellitization of iron ore were done in a disc pelletizer of diameter 400 mm, collar height 100 mm Fig. 2[16], angle of inclination 60oC, disc rotating speed 17 rpm and residence time 30 min. The materials were feed to the pelletizer. The predetermined moisture amount (8.5% water + different amount of molasses%) was then sprayed onto the rolling bed of material in the pelletizer. The green pellets in the size range 5-7 mm diameter were screened out to be dried in a drying oven at 110oC for 2h, to ensure the evaporation of all water used during the granulation process.

TABLE 1
THE CHEMICAL ANALYSIS OF EL-BAHARIA IRON ORE

Component	%
Fe total	52.35
MnO	2.92
SiO_2	10.84
CaO	0.39
MgO	0.18
Al_2O_3	1.44
S	0.74
TiO_2	0.16
BaO	1.17
ZnO	0.15
K_2O	0.27
Na_2O	0.25
P_2O_5	0.5

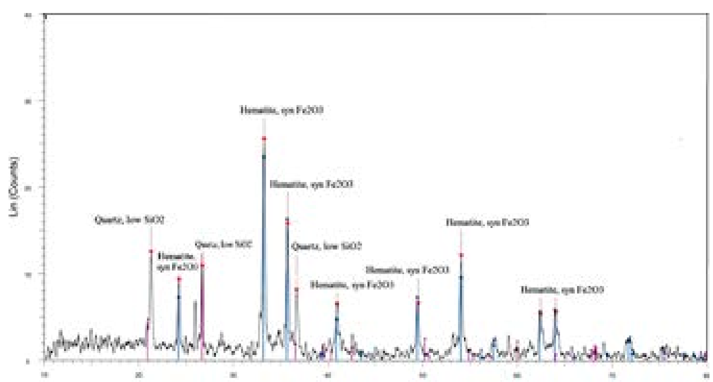

Fig.1. X-ray analysis of El-Baharia iron ore

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Fig. 2. Disc pelletizer equipment

The green and dried iron ore pellets subjected to drop number test and crushing strength tests. MEGA.KSC-10 hydraulic press Fig.3 was used to determine the crushing strength The drop number indicates how often green and dry pellets can be dropped from a height 46 cm before they show perceptible cracks or crumble. Ten green and dry pellets are individually dropped on to a steel plate. The number of drops is determined for each pellet. The arithmetical average values of the crumbing behavior of the ten pellets yield the drop number [4,15,17-21] .

The average compressive strength tests of at least 10 pellets were examined; between parallel steel plates of MEGA.KSC Fig. 3 up their breaking. The mean value of the tested briquettes or pellets gives their compressive strength. [22, 23].

2.3 Reduction Procedures

The reduction of ironore pelletswith hydrogen was performed in thermogravimetric apparatus. This scheme is similar to that present elsewhere (19, 24-25) (Figure 4).Typically,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 crucible 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 ±5°C. Then samples were placed in hot zone

The nitrogen flow rate was 0.5 l/min pass through furnace in all the experiments. At initial time air should be removed before each experiment and also after the end of reduction. 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:

Percent of reduction = [(Wo –Wt) x100/ Oxygen mass]

Where:

Wo: the initial mass of iron ore sample after removal of moisture content

Wt: mass of sample after each time, t.

Oxygen mass: indicates the total mass of oxygen percent in iron ore in form FeO, Fe_2O3 and manganese oxide.

Fig.3. MEGA.KSC-10 hydraulic press

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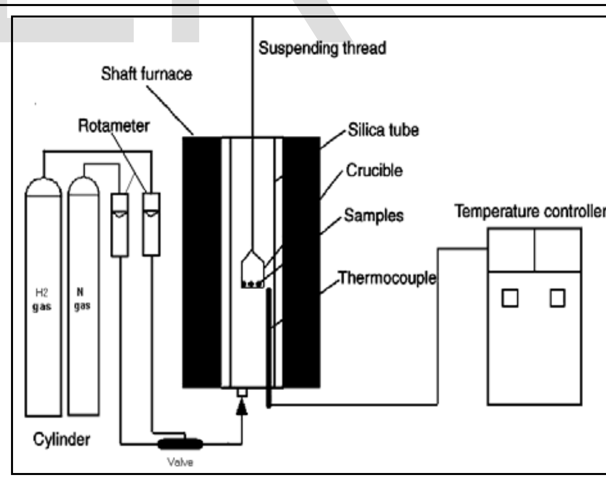

Fig. 4. Schematic diagram of the reduction apparatus

3 RESULTS AND DISCUSSIONS

3.1 Effect of adding molasses as binding materials on the quality of the produced green pellets

Figs.5 and 6.Illustrate the effect of percentage of molasses added on the drop number (drop damage resistance) and cold crushing strength of the green pellets of iron ore. It is clear that as the percent-

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age of molasses increased both the drop damage resistance and crushing strength increased; this may be due to the effect of binding materialof molasses.

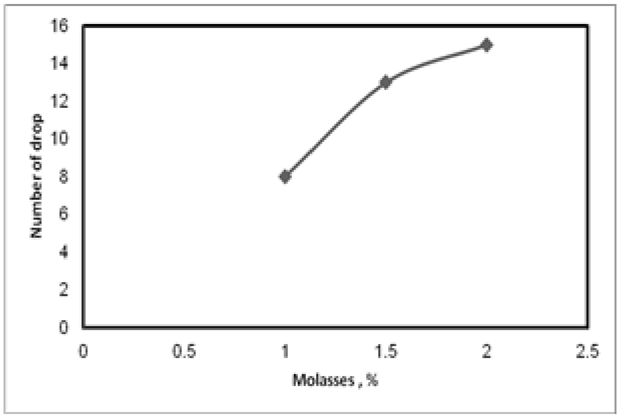

Fig. 5 Relationship between the dop number of green iron ore pellets and percentage of molasses added to the iron or during pelletization process

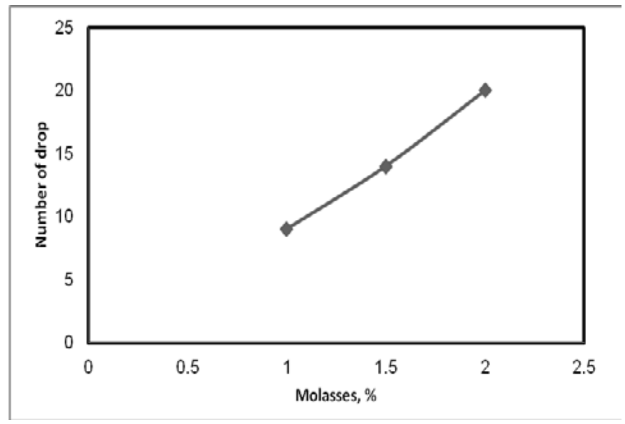

Fig. 7 Relationship between the drop number of dried iron ore pellets and percentage of molasses added to the iron ore during pelletization process

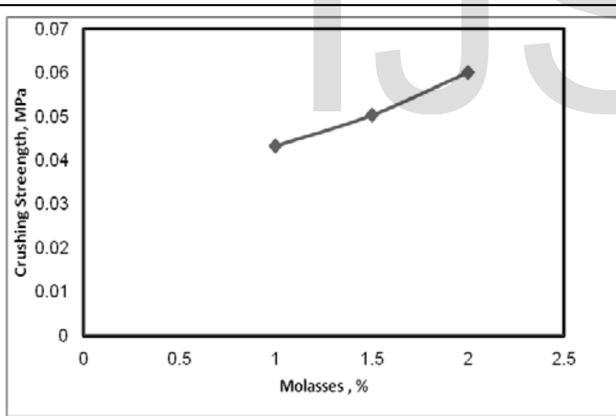

Fig. 6.Relationship between the crushing strength of green iron ore pellets and percentage of molasses added to the iron ore during pelletization process

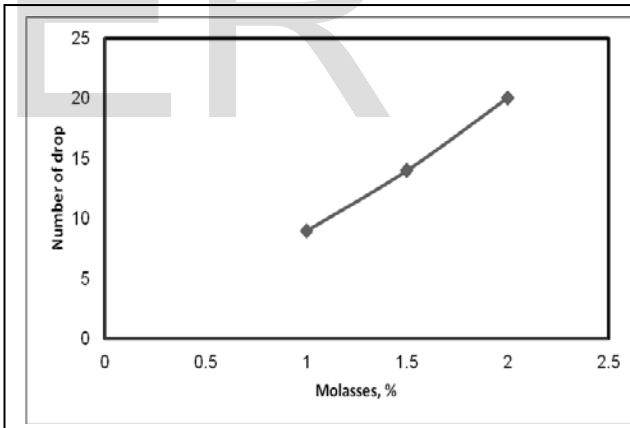

Fig 8 Relationship between the crushing strength of dried iron ore pellets and percentage of molasses added to the iron ore during pelletization process

3.2 Effect of adding molasses as binding materials on the quality of the produced dried pellets

Figures 7 and 8 illustrate the effect of molasses on the drop damage resistance and compressive strength of dried pellets (drying temperature 100 °C for 20 minutes). From this figure, it is clear that both the drop damage resistance and the compressive strength increased as the percentage of molasses increased. This may be attributed to the fact that increasing molasses addition leads to an increase in the contactpoints between the particles of iron ore fines and a decrease in the distance between them sothe compressive strength of dried granules increases [6,7].

3.3 Reduction of the pellets by hydrogen

3.3.1 Effect of hydrogen flow rate

Fig. 9 illustrates the relation between the reduction degree of iron ore pellets and time of reduction at different hydrogen flowrate when the reduction were done at constant temperature (900˚C), the weight of the sample was constant .It is clear that as the flow rate of hydrogen increased the reduction percentage increased. Many explanation

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for increasing reduction rate were introduced; first 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. So, the rate of reaction increased [20, 26,27]. Second the increase of flow rate increased and the gas diffusion across the boundary layer subsequently the ion reduction increased [27, 28]. Third 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 [20, 25, 30].

3.3.2 Effect of Reduction Temperatures on the Reducibility of Iron ore pellets

The reduction was carried out at different temperatures ranging from 600˚C to 950˚C, where the pellets weight is constant and the hydrogen flow rate was 2 liter/min. It is clear that increasing temperature of reduction is favour the reduction rate as shown in Fig.10.
The analysis of the curves relating the reduction percentage and time of reduction show that for each single reduction curve, the rate of reduction of iron ore pellets was rabidly increased with increasing time till 15 min at early stages after this time no appreciable changes occur, indicating after15 min.the time almost had no effect.This increase with temperature could be due to increase of number of reacting moles having excess energy which leads to the increase rate of reaction. Also increasing temperature leads to increase the rate of mass transfer of the diffusion and rate of chemical reaction [27] [30].

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 [31-32]. Also the raise of temperature leads to an increase of the rate of mass transfer of the diffusion and rate of desorption [23-24].

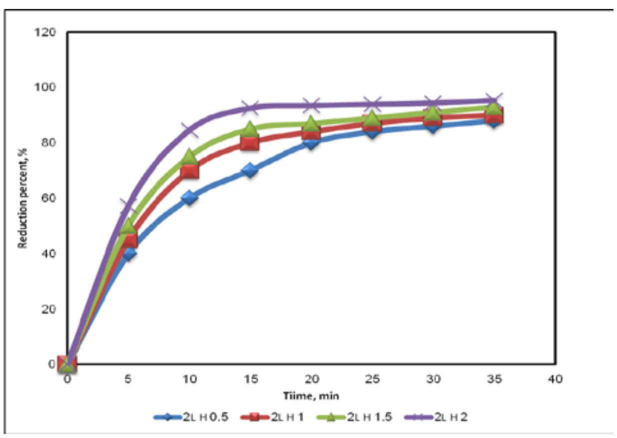

Fig. 9 Effect of hydrogen flow rate on the reduction percentage of iron ore pelletswhere the temperature of reduction is 900 oC

3.3.3 Kinetic of reduction of iron ore pellets

Kinetic studies for estimation the apparent activation energies were carried out for the reduction of iron ore pellets samples at five different temperatures 600,700,800, 900 and 950ºC for different time intervals in the range of 0 – 60 minutes. By using diffusion through thin ash layer (jander equation):-

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

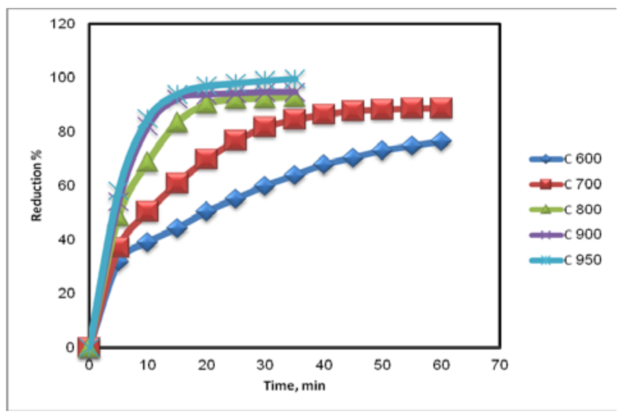

Fig. 10 Effect of reduction temperature on the reduction percentage of iron ore pellets where the hydrogen flow rate 2 liter/min

Where f is fractional reduction, t is time of reduction, k is the rate constant.
Figure 1 illustrates the relation between (1-(1-f)1/3)2 against time of reduction for different reduction temperature. From which it is clear that the relationship is represented by straight line. The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of reduction reaction. The results were illustrated on Figures 12 from which it is clear that briquette has activation energy = 60.55 kJ/ mole.

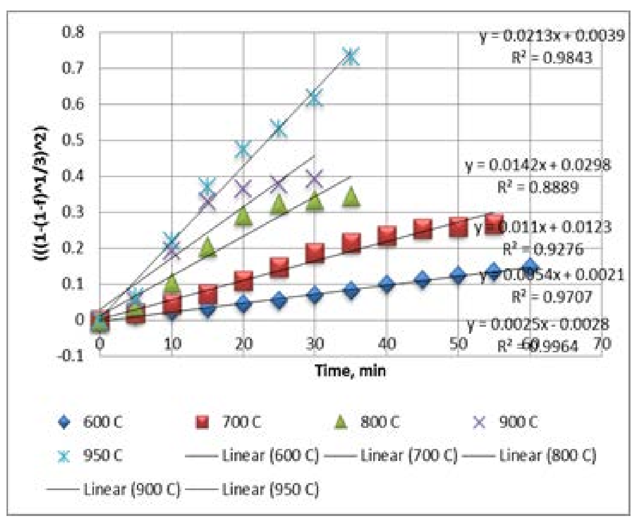

Figure 11 The relationship between $(1-(1-f)^{1/3})^2$ and time of reduction of iron ore pellets for different reduction temperature at

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constant hydrogen flow rate

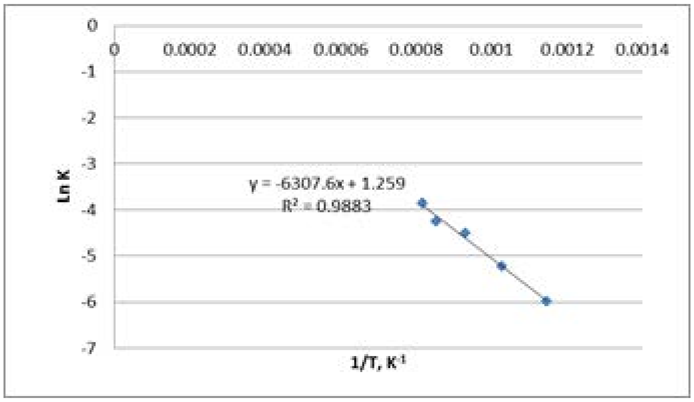

Figure 12. The relation between the reciprocal of absolute temperature $1/T$ and $\ln K$ (Arrhenius plot for reduction reaction) for model $(1-(1-f)^{1/3})^2$ =kt

3.4 X-ray analyses of the reduced pellets

Figs. 13 and 14 illustrates the X-ray analysis of the reduced pellets via hydrogen at 60oC and 950 oC respectively. From these diffraction patterns it can be observed that, the iron (Fe) , hematite and magnetite are the main minerals of the pellets reduced by hydrogen at 600 oC While the x-ray of the pellets reduced at 950 oC shows that the main phase is iron (Fe) .The result of x-ray give agreement with the reduction curves as in Fig.10

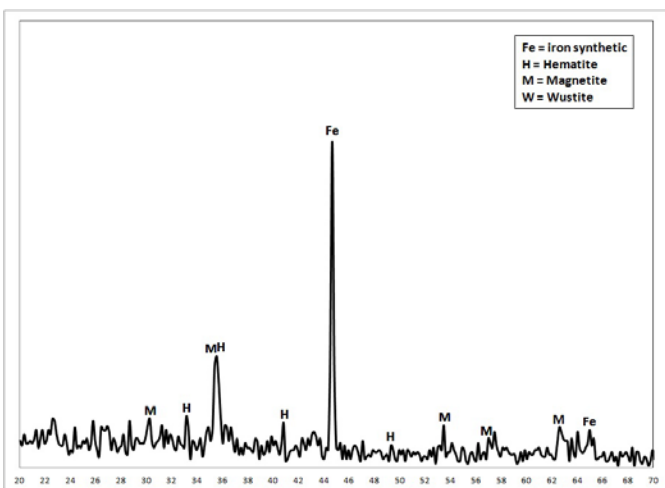

Figure13. XRD analysis of reduced iron ore pellets by hydrogen at 600°C

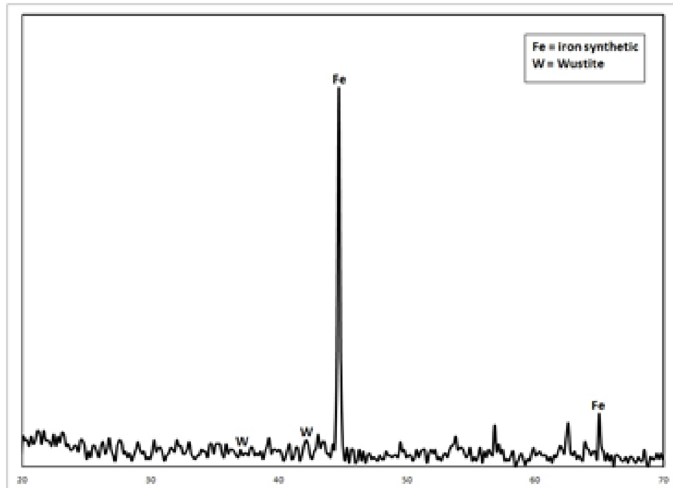

Figure14. XRD analysis of reduced iron ore pellets by hydrogen at 950°C

4. Conclusions

*Iron is the most used of all metals, it extracted from its ores as it is not found in the elemental form by reduction. In this work hydrogen was used in reduction of El-Baharia iron ore to remove oxygen and obtain iron.

**Binder material (molass) was used in this study, increasing molasses percentage (1% up to2%) shows increase both drop damage resistance and cold crushing strength for green and dried iron pellets.

***Increasing hydrogen flow rate (0.5L up to 2L) and temperature (600^{o}C up to 950^{o}C) have the effect to increase the reduction rate .

****Jander equation was applied for reduction of pellets by hydrogen . The correlation coefficient was about 0.988 indicating that diffusion through thin ash layer could describe well the experimental data.

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