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Pelletization of El-Baharia iron ore (O) with different amount of mill scale (M) and reduction kinetics of these pellets via hydrogen

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Abstract— The effect of the Addition of fine mill scale (M) upon the quality and reduction kinetics of El-Baharia iron ore (O) pellets via hydrogen was done at different temperatures ranging from 700˚C to 950˚C and. the best reduction properties were found at 950˚C.The kinetic of the reduction process was studied using the best fit model determined was kt = - ln (1-R). The main crystalline phases of reduced pellets at 950˚C were metallic iron (syn. Fe).

Index Terms— Iron ore, mill scale hydrogen, Reduction kinetics, pelletization . Metallic iron (syn. Fe)

1 Introduction

Mill scale is a steelmaking by-product formed by oxygen reacting with the iron in ferrous metals during the rolling mill in the steel hot rolling process. Mill scale contains both iron in elemental form and three types of iron oxides: Wustite (FeO), Hematite (α-Fe_2O_3) and Magnetite (Fe_3O_4). The chemical composition of mill scale varies according to the type of steel produced and the process used. The iron content is normally around 70 %, with traces of non-ferrous metals and alkaline compounds. The reduction of rolling mill scale to sponge iron powder is a new way to take advantage of a cheap by-product of the steelmaking industry, yielding sponge iron that can be reused to the electric furnace as metallic charge for steelmaking to obtain a product with a lower residual content and improved properties [1-4].

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About 500 kg/ton of solid wastes of different nature are generatedIn several iron and steel making processes,; one of these wastes is the mill scale which represents about 2% of steel produced [5]. Mill scale is a very attractive waste because its contain about = 72 % Fe [6].

About 13.5 million tons of mill scales are generated annually in the whole world [7[. Mill scale is suitable for direct recycling to the blast furnace via sintering plant [8]. Approximately, 90% of mill scale is directly recycled within steelmaking industry and small amounts are used for ferroalloys, in the petrochemicals industry and in cement plants [9-13].

Mill scale used also in manufacturing iron oxide pigments from mill scale. The mill scale is mixed with alien iron oxide and the resultant mixture is heated to a temperature of 200° C. to 900° C. in an oxidizing atmosphere to produce iron oxide pigment which can be black, brown or red.

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The alien iron oxide used and the iron oxide pigment produced are predominantly Fe_2O_3 or Fe_3O_4 or a mixture of both. The iron oxide pigment produced as above or from any other sources can be turn into black by mixing it with mill scale and the resultant mixture is heated to a temperature of 200° C. to 900° C. in a non-oxidizing or reducing atmosphere to produced iron oxide black pigment [14]

In the manufacture of glasses requiring the presence of iron to develop a colorant therein. It has been usual to add small amounts of mill scale.[14] .

El-Hussiny et al [15] found that, replacement of el-Baharia hig barite iron ore concentrate by mill scale waste increased the amount of ready made sinter, sinter strength and productivity of both sinter machine and at blast furnace yard.

The aim of this work is to study the pelletization the El-Baharia Oasis iron ore (O) with mill scale waste (M) . and reduce its in static bed by hydrogen .

2-Experimental Work

2-1- Material used and its characteristics

El-Baharia iron ore was supplied by the Egyptian Iron and Steel Company, The chemical composition of this ore is as follows:- [16-17] : 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 %

The X- Ray analysis of El-Baharia iron ore(O) is illustrated in figures 1 . From which it is clear that El-Baharia iron ore mainly consists of hematite and quartz [16-17].

Mill scale (M) which used in this work was delivered from Egyptian Iron and Steel Co. The chemical analysis of mill scale shows that it contains Fe total 69.33 % in the form (Fe_2O_3 70 %, Fe_3O_4 17.26 % and FeO 7.83 %). Sulphur 0.33 %, Phosphorus 0.22 %, MnO 0.66 %, SiO_2 1.92 % and carbon 0.04 %.[4, 18 , 19]

The X-ray of the mill (M) illustrated in Fig.2, from which it is clear that the main compound of mill scale is hematite.[4 ,18, 19].

2.2. Preparation of the pellets and Its Physical Properties

The iron ore and mill scale were grinding separate in vibrating mill to powder with size less than 75 micrometers. After which the pellitization of (O) with the predetermine of (M) were done in a disc pelletizer of diameter 400 mm, collar height 100 mm Fig. 3 [17] , angle of inclination 60 °C, disc rotating speed 17 rpm and residence time 30 min. The materials were feed to the pelletizer. The predetermined moisture amount (12% water) and different percentage of the charge molasses) was then sprayed onto the rolling bed of material in the disc pelletizer. The green pellets in the size range 5-7 mm diameter were screened out to dried in the air for 3 days, to ensure the evaporation of all water used during the granulation process

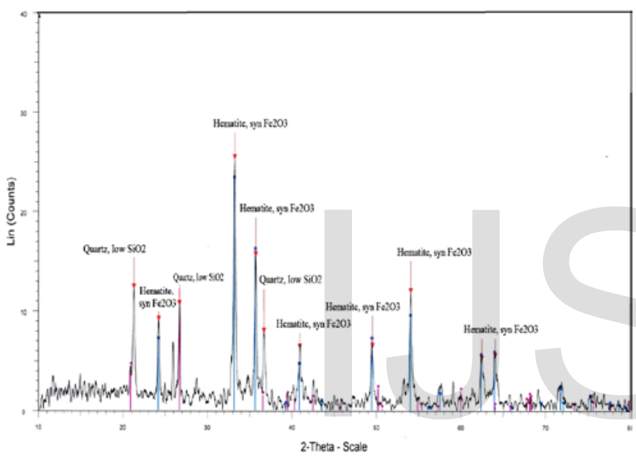

Fig.1. X-ray analysis of El-Baharia iron ore

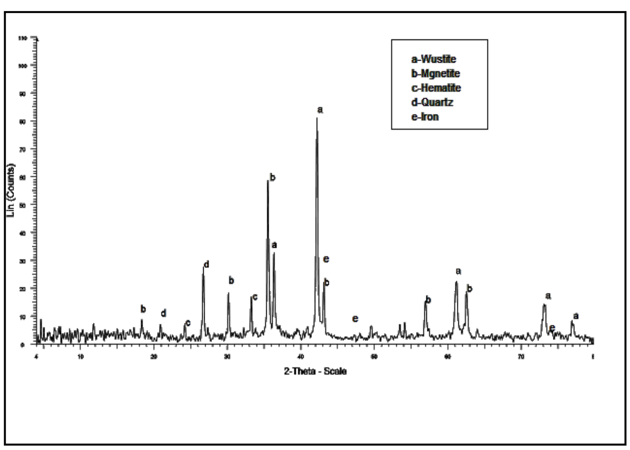

Fig.2. X-ray of mill scale

The green and dry pellets subjected to drop number test and crushing strength tests. The MEGA.KSC-10 hydraulic press used for crushing strength test Fig.4 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 pellets . The arithmetical average values of the crumbing behavior of the ten pellets yield the drop number[20-23] .The average compressive strength tests of at least 10 pellets; between parallel steel plates of MEGA.KSC up their breaking then the mean value of the tested pellets gives their compressive strength. [20-23]

Fig. 3 Disc pelletizer equipment

2.3. firing of pellets in muffle furnace

The dried pellets was fired in muffle furnace at temperature 800 – 1150 °C

2.4 Reduction Procedures

The reduction of pellets was performed in thermogravimetric apparatus. This scheme is similar to that present elsewhere [13,24-31] (Figure 5). 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 (700°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 during initial and final time of reduction. The weight of the sample was continuously recorde, 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 the sample, g.

Wt: mass of sample after each time, t.,g.

Oxygen mass: indicates the total mass of oxygen percent in the sample in form FeO, Fe_2O_3 and manganese oxide. g.

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Fig.4 MEGA.KSC-10 hydraulic press

3-Resulte and Dissication

3.1 Effect of binding material added on the pellet charcterstic (pellet consists of 5% M and 95 % O)

Tables 1 and 2 illustrated the effect of the percentage of molasses added (+12 % water) on the drop damge number and crushing strength of the wet (green) and dried pellets after 3 day in air From Table 1 it is clear that the increase of molasses added up to 2 % the drop number and crushing strength increase then more addition of molasses the drop damage resistance and crushing strength decreased While Table 2 shows that after 3 day both drop number and crushing strength of the pellets increases as the amount of molasses added increased.

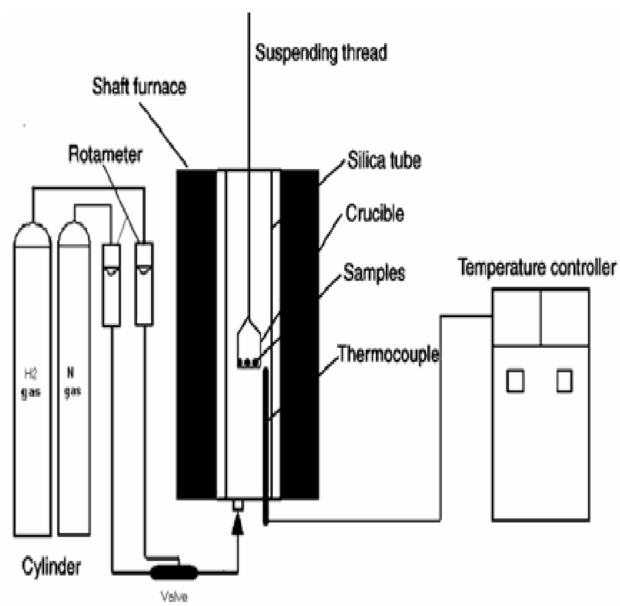

Fig. 5. Schematic diagram of the apparatus

Table 1 Effect of molass added on the drop number and strength of green pellets

Percentage of molasses added, %.	Drop No.	Strength, MPa.
1.5	**7**	**0.02**
2	**13**	**0.028**
2,5	**9**	**0.017**
3	**6**	**0.014**

Table 2 Effect of molasses added on the drop number and strength of dried pellets in air after 3 day.

Percentage of molasses added, %.	Drop No.	Strength, MPa.
1.5	**11**	**0.123**
2	**12**	**0.137**
2,5	**18**	**0.181**
3	**23**	**0.194**

3.2 Effect of change the amount of M added to O on the produced pellets charcterstic

Tables 3 and 4 illustrated the effect of the change M added to O (amount of molasses =2% + 12 % water) on the drop number and crushing strength of the wet and dried pellets after 3 day in air From Tables 3 and 4 it is clear that the increase of M added to O the drop number and crushing strength increases .

Table 3 Effect of M percentage added to O on the drop number and strength of green pellets.

Percentage of Mill scale (M) added to iron ore (O).	Drop No.	Strength, MPa.
5	**7**	**0.028**
10	**9**	**0.0291**
15	**10**	**0.0301**
20	**12**	**0.031**

Table 4 Effect of M percenage added to O on the drop number and strength of dried pellets in air 3 day .

Percentage of M added to O.	Drop No.	Strength, MPa.
5	**12**	**0.126**
10	**14**	**0.137**
15	**17**	**0.166**
20	**20**	**0.196**

3.3 Effect of change the amount of M added to O on the fireng produced pellets charcterstic

Table 5 illustrate the effect of the change M added to O (amount of water 12 % and molasses =2%) on the crushing strength of the fired pellets at different temperature . From Table 5 , it is clear that the increase of percentage of M in the mixure at any constant temperature of firing the crushing strength increase also at any constant amount of M in the pellets increase of firing temperature leades to an increase the crushing strength of fired pellets

Table 5 Effect of percenage of M added to O on the strength of fired pellets at different temperature

Percentage of Mill scale (M) added to iron ore, (O) .	Strength , MPa , when firing temperature 800 ^{O}C.	Strength, MPa. when firing temperature 900 ^{O}C.	Strength, MPa. when firing temperature 1000 ^{O}C.	Strength, MPa. when firing temperature 1150 ^{O}C
5	**0.171**	**0.48**	**0.95**	**7.89**
10	**0.218**	**0.527**	**1.127**	**8.32**
15	**0.227**	**0.618**	**1.17**	**9.104**
20	**0.25**	**o.66**	**1.3**	**12.14**

3.4. Effect of the change of the percentage of M which mixed with O on the reduction percentage

Figure.6 Illustrate the effect of change of percentage of M which mixed with O on the reduction of the pellets which fired at 1150 ᵒC (reduction temperature = 900 ᵒC , flow rate of hydrogen gas1.5 L/ min.) , from which it is clear that as M in the pellets increased the reduction percentage decreased this may be due to the fact that the low porosity of the mill scale .

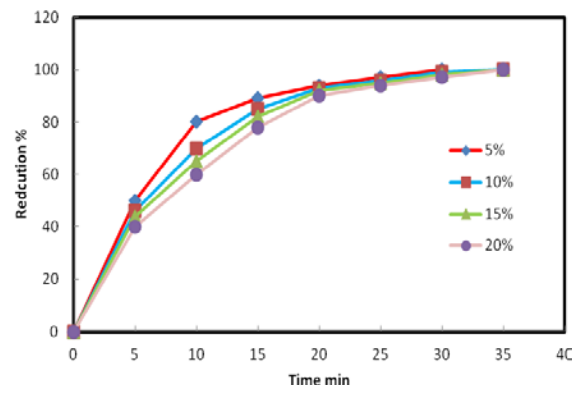

Figure 6 The effect of change of percentage of M which mixed with O on the reduction of the pellets which fired at 1150 ^{o}C

3.5 Effect of different hydrogen flow rate on the reduction percentage of pellets containing mixture of M and O

Figures 7 and 8 Show the effect of change of hydrogen flow rate on the reduction of the pellets produced using 2% molasses with 12 % water and dried in air 3 day and fired at 1150 °C. for both M/O ratio 0.0526 and 0.25 , from these figures it is clear that as the flow rate increased the reduction percentage increased , this results may be due to the increase of flow rate leads to increasing the number of hydrogen moles in the bulk phase, which leads to the raise of hydrogen adsorption , thus , the rate of reaction increased or due to the increase of flow rate of hydrogen increased the gas diffusion across the boundary layer of pellets (32 -34).

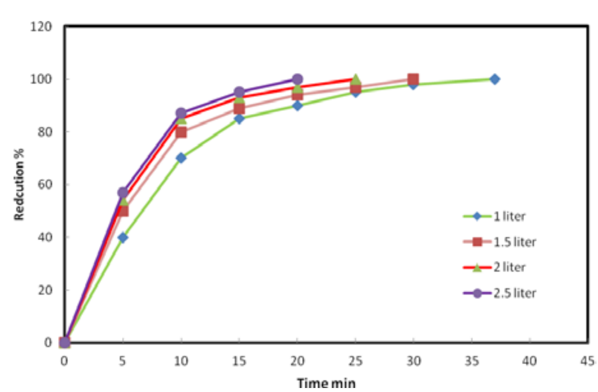

Figure 7 The effect of change of hydrogen flow rate on the reduction of the pellets which fired at 1150 °C. for M/O ratio 0.0526

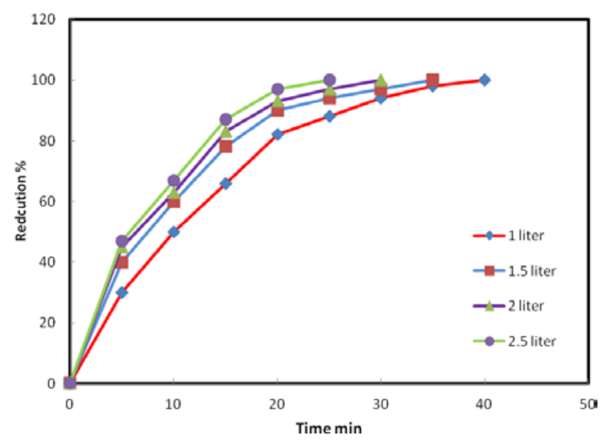

Figure 8 The effect of change of hydrogen5 flow rate on the reduction of the pellets which fired at 1150 oC M/O ratio 0.25

3.6 Effect of change of temperature on the reduction degree of the mixture of (O)with (M) by constant amount of hydrogen flow rate

The effect of temperature on the reduction of the mixture of iron ore with mill scale and fired at 1150ᵒC by 1.5 L/min hydrogen flow rate, were done at 700 – 950ᵒC. The plots of the reduction percentage as function of time are shown in Figs. 9and 10 .From these figures it is evident that the reduction percentage increased as the temperature increased 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. Also the raise of temperature leads to an increase of the rate of mass transfer of the diffusion and rate of desorption.

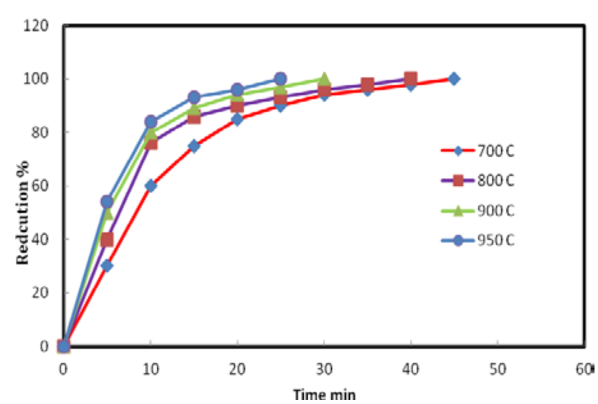

Figure 9 Effect of temperature on the reduction of of the mixture of 95 % O with 5 % M and fired at 1150°C by 1.5 L/min hydrogen flow rate

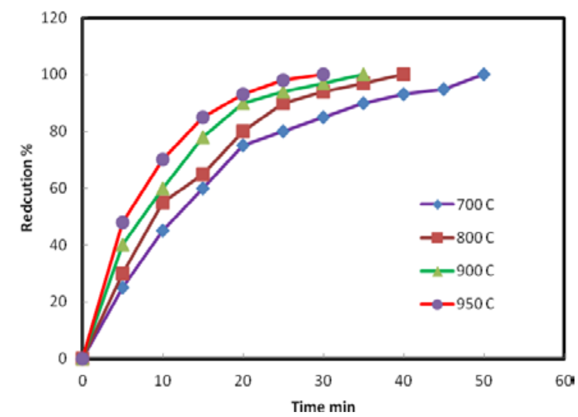

Figure.10 Effect of temperature on the reduction of of the mixture of 80 % O with 20% M and fired at 1150°C by 1.5 L/min hydrogen flow rate

3.7. Kinetics reduction of pellets

Kinetic studies for estimation of apparent activation energies were carried out for the pellets at different temperatures range from 700˚C up to 950˚C for different time intervals in the range of 0 - 60 min. Using equation unimolccuku decay law [35]

$$-\mathrm{Ln}\,(1-R) = kt$$

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

Figs.11 and 13 illustrate the relation between -ln (1-R) against time of reduction for different reduction temperature, 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 Figs.12, 14, from which it is clear that the activation energy= 20.94 kJ/ mole .29.13 kJ/mole for 5 % M and 20% M added to O respectively.

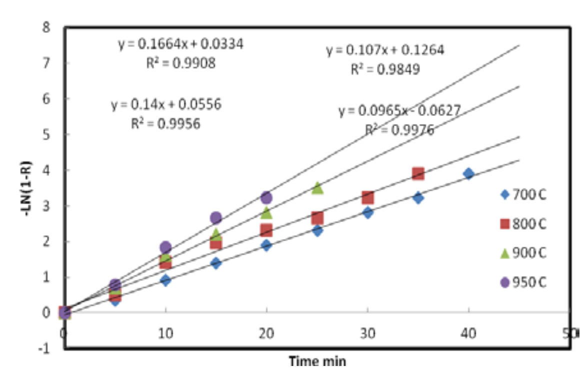

Fig.11 Relation between -ln (1-R) and time of reduction at different reduction temperature for mixture of 95 % O with 5 % M and fired at 1150oC reduced by 1.5 L/min hydrogen flow rate

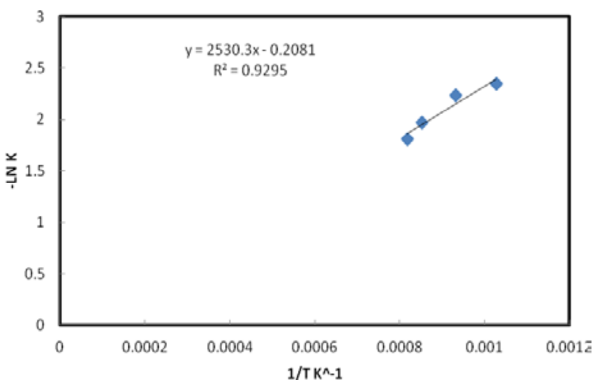

Fig.12 The relation between natural logarithms an 1/ T for reduction of M/O = 0.052

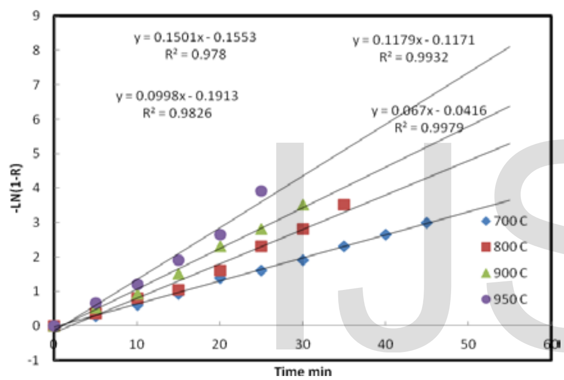

Fig. 13 Relation between -ln (1-R) and time of reduction at different reduction temperature for mixture of 80 % O with 20 % M and fired at 1150°C reduced by 1.5 L/min hydrogen flow rate

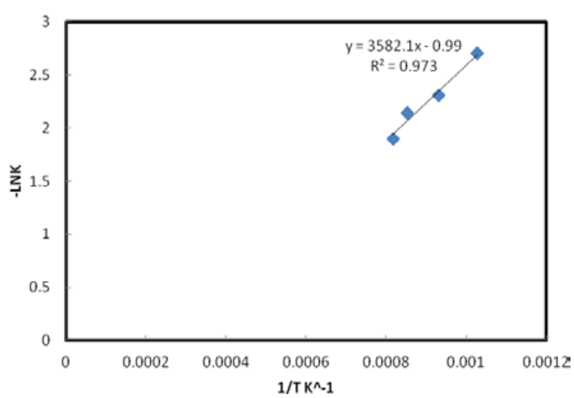

Fig.14 The relation between natural logarithms an 1/ T for reduction of M/O = 0.25

3.8. X-Ray analyses of the reduced pellets

X-ray analyses of the sample reduced at 950°C for M/O 0.0526 and 0.25 shows that the present phases are metallic iron (syn. Fe), and some traces of magnetite [M] (Fe_3O_4,) as shown in Figures 15- 16

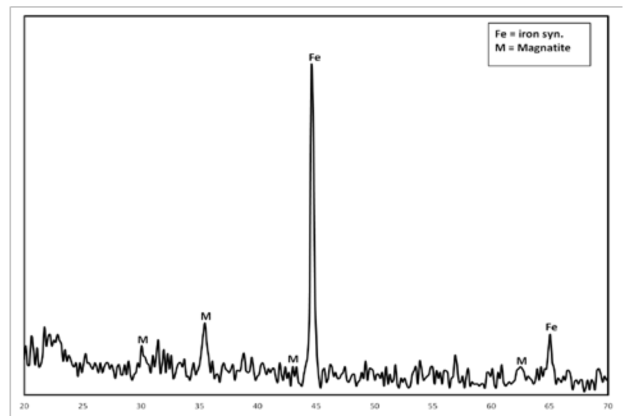

Fig. 15 X-ray analyses of the sample reduced at 950°C for M/O 0.0526

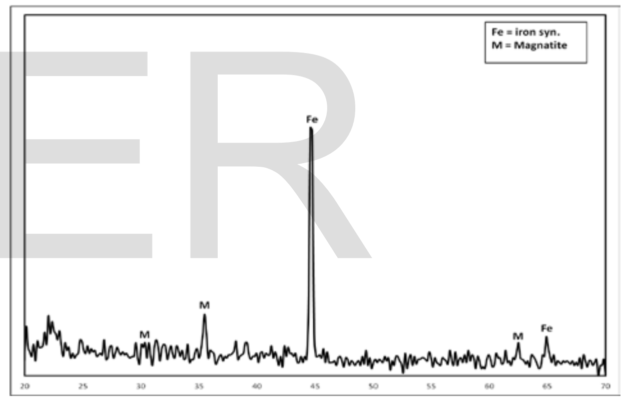

Fig. 16 X-ray analyses of the sample reduced at 950°C for M/O 0.25

4 CONCLUSION

1- When the M/O increases (2% molasses with 12 % water added during pelletization) both drop number and compression strength of green pellet increased.
2- The increase of percentage of M in the mixture at any constant temperature of firing the crushing strength increase also at any constant amount of M in the pellets increase of firing temperature leads to an increase the crushing strength of fired pellets
3- When the M/O increased the reduction of the pellets decreased.
4- The reduction percentage of the pellets increased when the temperature of reduction increased
5- AS the hydrogen flow rate increased the reduction of the pellets increased.

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