

Sintering and Reduction via Hydrogen of Egyptian iron ore briquettes with Dolomite

N.A. El-Hussiny⁽¹⁾, I.A. Nafeaa⁽²⁾, M.G. Khalifa⁽³⁾, S.Th. Abdel-Rahim⁽²⁾, M.E.H. Shalabi^{(1)*}

- 1- Central Metallurgical Research and Development Institute, Cairo, Egypt.
- 2- Chemistry Department, Faculty of Science, Ain Shams University, Cairo, Egypt.
- 3- El-Tabbin Metallurgical Institute, Cairo, Egypt.

*Corresponding author : shalabimeh@hotmail.com

Abstract: This investigation studied the effect of dolomite addition to iron oxide raw material on the physicochemical properties of its briquette in a green form and indurate form. Also the effect of this addition on the degree of reduction was studied. The results indicated that the addition of (2% to 8%) dolomite improve the mechanical strength of the green briquettes while the mechanical strength of indurate form decreased. Also the reduction of these briquettes via hydrogen was studied and the model of reduction was put in this paper.

Keywords:- Indurate briquettes, dolomite addition to iron ore, models of reduction.

1. Introduction

Dolomite is an anhydrous carbonate mineral composed of Calcium Magnesium Carbonate, ideally $\text{CaMg}(\text{CO}_3)_2$. The word dolomite is also used to describe the sedimentary rock, which composed predominantly of the mineral dolomite (also known as dolostone).

El Kassabgy and Lu [1], found that the presence of MgO and CaO (2% wt. and 5% wt.) promote the metallization of wustite.

Sarat [2] was studied the influence of MgO addition on sinter mineralogy on sinter produced with a wide range of MgO/CaO ratios at several basicity indices $[B = (\text{CaO} + \text{MgO})/(\text{SiO}_2 + \text{Al}_2\text{O}_3)]$ between 0.7 to 1.9. The most striking influence of MgO is the suppression of hematite and Ca-ferrite phases and the increase in magnetite phase. In general, MgO favors the formation

of glass and suppresses the precipitation of dicalcium silicates in favor of Ca-Fe-Mg olivines and pyroxenes. Microprobe studies revealed that most of the Mg was picked up by the magnetite phase to form mixed spinels of type $(\text{Fe},\text{Mg})\text{O}$, Fe_2O_3 . At a constant basicity index, increased replacement of CaO by MgO also lead to increased participation of FeO in the slag formation process, thus increasing the overall FeO content of sinter. A mechanism for the formation of mixed spinels has been proposed. The effect on various sinter properties resulting due to change in sinter mineralogy has been outlined.

Timo [3] found that addition high amount of MgO and CaO to iron ore did not form a homogenous solid solution with magnetite but also magnesio-ferrite and calcium ferrite phases occurred in the prepared

samples. MgO and especially CaO have a strongly accelerative effect on the reduction of magnetite.

Bin et al [4] studied the effect of various dosages of dolomite on the reduction swelling property of iron ore pellets. The experimental results show that with increasing the dolomite dosage from 0 to 10.5% the reduction swelling index (RSI) decreases from 13.35% to 4.0%, while the porosity of roasted pellets increases from 35% to 40%. Meanwhile, the content of magnesium ferrite with high melting temperature, as well as the stability of magnetite (Fe_3O_4) in the roasted pellets increases with increasing the magnesium oxide (MgO) content from dolomite. The reason of the decrease of RSI really on the absence of crystal transformation from Fe_2O_3 to Fe_3O_4 , the increased porosity of roasted pellet, and the suppression of phase transition of $2CaO \cdot SiO_2$ resulted from the incorporation of magnesium into calcium silicate.

Dwarapudi et al [5] indicated that during indurations at a high temperature, a considerable amount of slag/melt phase forms inside the iron ore pellets, comprising SiO_2 , Al_2O_3 , CaO, MgO and FeO. After cooling, the slag phase solidifies and acts as an important bonding phase in the finished pellets and influences their room temperature as well as high temperature properties, especially reduction degradation. Fluxing agents play an important role in forming these bonding phases*

depending on the type and amount of flux. In the present study, the effect of different fluxing agents, namely, limestone, dolomite, magnesite and pyroxenite, on melt formation and microstructure during indurations and on reduction degradation behavior during reduction was examined. From the results, it was understood that to reduce the disintegration during reduction it is essential to increase the amount and distribution of bonding phases like silicates, which are more stable as compared to oxide phases like hematite. Acid pellets exhibited highest reduction degradation due to the presence of more hematite bonds and less silicate bonds. In limestone fluxed pellets, reduction degradation index dropped considerably with increasing CaO content due to the formation of more amount of bonding phase. Dolomite-pyroxenite pellets, on the other hand, showed lower reduction degradation index up to 0.4 basicity, and beyond that, higher degradation was observed due to the increased pore size, which resulted in poor strength of the reduced pellet matrix and hence more degradation. Low reduction degradation observed in pyroxenite and magnesite fluxed pellets could be due to the formation of magnesioferrite and silicate melt, which are more stable phases compared to hematite.

Umadevi et al [6] said that flux materials such as dolomite and other MgO bearing materials

influence the microstructure and chemical properties of the resultant sinter. Improvement in raw material quality and use of alternative raw materials play an important role for improving the sinter quality and overall economics of iron and steel making. The physical and metallurgical properties of sinter mainly depend on the mineralogy of the sinter. Dolomite is composed of calcium magnesium carbonate and dunite consists of magnesium silicate. In this work laboratory sintering experiments have been carried out with different MgO level (1.40 to 2.60 mass %) the results indicated that the microstructural examinations of the produced sinter revealed that dunite sinter is bounded with higher amounts of hematite and less calcium ferrites when compared to dolomite sinter. Hematite and calcium ferrite phases decreased whereas magnetite phase increased with increase of magnetite content in sinter using both dolomite and dunite. From the test results it was found that dunite sinter showed better metallurgical properties whereas dolomite sinter showed better physical properties.

Umadevi, Nelson, et al [7] indicated that Dolomite and other MgO bearing materials are being increasingly used as basic flux constituents for production of fluxed sinters. Addition of flux materials in sinter influences the resultant sinter microstructure and chemical properties. The physical and

metallurgical properties of sinter mainly depend on mineralogy of the sinter. Dolomite is the source of double carbonate of calcium and magnesium. Recent studies reveal that, apart from the additional fuel needed, the addition of dolomite and MgO bearing material greatly influences the magnetite content and the properties of the sinter produced. The increasing use of MgO bearing fluxes in the blast furnace burden, and the trend to incorporate a major part of fluxes in the sinter mix led to an investigation of the influence of MgO on sinter properties and productivity. In this study, the systematic investigation has been made on the influence of MgO% (1.4 to 2.6) on sinter mineralogy and sinter properties with dolomite. Microstructural examination of dolomite sinter revealed that hematite and calcium ferrite phases decreased whereas magnetite phase increased with increase in MgO percentage in sinter. From the laboratory pot grate sintering results it was found that sinter reduction degradation index improved whereas tumbler index and reducibility decreased with increase in MgO%.

Min et al [8] illustrated that six additives, i.e., limestone, lime, magnesite, magnesia, dolomite and light-burned-dolomite, were added to iron ore for investigating their influences on the pellet quality. For green balls, adding lime and light-burned-dolomite makes the wet

drop strength decrease firstly, and then increase with further increase of additive dosage. $\text{Ca}(\text{OH})_2$ affects the bentonite properties at the beginning, but the binding property of $\text{Ca}(\text{OH})_2$ will be main when the dosage is higher. The other four additives decrease the drop strength for their disadvantageous physical properties. For preheated pellets, no matter what kind of additive is added, the compressive strength will be decreased because of unmineralized additives. For roasted pellets, calcium additives can form binding phase of calcium-ferrite, and suitable liquid phase will improve recrystallization of hematite, but excessive liquid will destroy the structure of pellets, so the compressive strength of pellet increases firstly and then drops. When adding magnesium additives, the strength will be decreased because of the oxidation of magnetite retarded by MgO.

Umadevi et al [9] indicated that the quality of iron ore sinter mainly depends on sinter mineralogy, which in turn depends on the chemical composition of the sinter mix. The reduction properties of the mineral phases formed in the sinter influences the sinter reducibility. MgO has a varying effect on sinter reducibility at different silica contents. A recent trend in blast furnace operation shows that there is a considerable increase in usage of dolomite as a basic flux either directly or through sinter. Recently the silica levels in the sinter product

of Sinter plant 1(SP1) of JSW Steel Limited have been fluctuating in the range of 5.5–9.6% due to variation in silica content of iron ore fines. At the same time, as per blast furnace requirement, the addition of dolomite has been changed from 2.4 to greater than 3.0% at SP1, and the reducibility of the sinter decreased (<60.0%). Laboratory pot grate sintering experiments have been carried out to determine the influence of MgO addition on microstructure and reducibility of low and high silica sinter. MgO additions have been varied from 1.4 to 3.2% for low silica (4.5%), and high silica (6.3%) iron ore fines.

From the studies it was found that the reducibility of both sinters decreased with increase in MgO addition due to an increase in magnetite/magnesian spinel phase and silicate/slag phase. Reducibility of low silica sinter was greater than high silica sinter. High silica with high MgO sinter had lower reducibility compared to low silica with low MgO/high MgO and high silica with low MgO sinter.

Umadevi et al [10] found that the dolomite is the critical additive for fluxing the sinter and controlling the sinter MgO. Recently, the silica level in the sinter product of the sinter plant 1 has been varying in the range of 5.51 to 9.58% owing to variation in silica content of iron ore fines. At the same time, as per the blast furnace requirement, the rate of addition of dolomite has been

changed from 2.40 to greater than 3.00% at the sinter plant 1. In iron ore sinter, MgO plays different roles on sinter properties at different silica ranges. Because of wide range of fluctuation in iron ore silica it is essential to optimize the MgO addition in sinter mix to get desired properties. Laboratory pot grate sintering experiments have been carried out to know the influence of MgO addition on microstructure and properties of low silica and high silica sinter. MgO addition has been varied from 1.40 to 3.20% for low silica (4.47%), and high silica (6.25%) iron ore fines. High silica with high MgO sinter showed lower strength, higher reduction degradation index, and lower reducibility compared to low silica with low and high MgO and high silica with low MgO sinter. To achieve better sinter properties MgO content in the sinter should not be more than 2.80% when silica is more than 6.0%.

In this paper, the effect of addition of dolomite as a fluxing material on both physical and reduction properties of briquettes produced from Egyptian iron ores with using molasses as binding materials will discuss.

2. Materials and Method

Iron ore and dolomite samples were supplied by the Egyptian Iron and Steel Company, The chemical composition of this raw material is as the following:- Chemical analysis of dolomite: CaO= %,

SiO₂= % and MgO= %. While the chemical analysis of El-Baharia iron ore: 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% [11&12].

The X-ray analysis of El-Baharia iron ore and dolomite are illustrated in figures (1) and (2) respectively. From which it is clear that El-Baharia iron ore mainly consists of hematite and quartz. While the X-ray analysis of dolomite mainly consists of CaMg(CO₃).

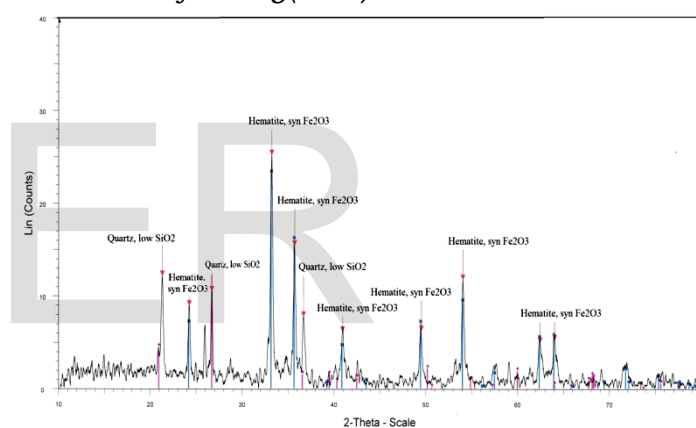


Fig.(1) X-ray of iron ore.

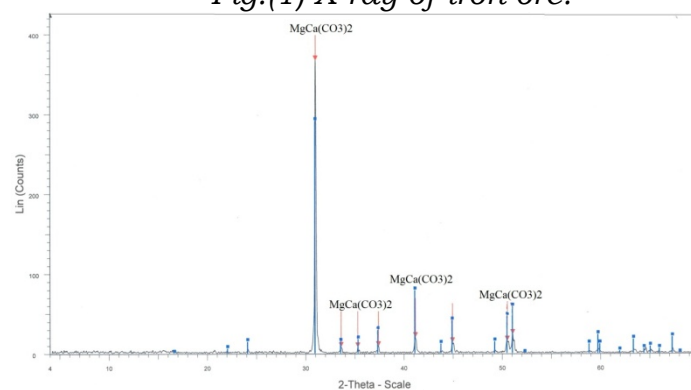


Fig.(2) X-ray of dolomite ore.

2.1 Preparation of the briquette and its physical properties

The iron ore and dolomite were grinding in vibrating mill to powder with size less than $75\mu\text{m}$. The 10g mixture of iron ore with certain amount of dolomite powder and 2% molasses are pressed in the mould (12mm diameter and a height 22mm using MEGA.KSC-10 hydraulic press fig.(3) [11]. Under the pressure= 196.133MPa. The produced briquette 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 46cm 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 compressive strength is done by compressed 10 briquettes between parallel steel plates up to their breaking [13]. The green briquette then burned in the computerize muffle furnace in different temperature from 900°C to 1200°C . Then 10 sinter briquette subjected to compression test.

Fig.(3) MEGA.KSC-10 hydraulic press.

2.2 Reduction process

The reduction of iron ore with dolomite briquette by hydrogen was done in a thermo balance apparatus. A schematic diagram of thermo balance apparatus is shown in Fig.(4) [11,12 & 14-18]. 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 (650°C - 950°C) and maintained constant to $\pm 5^{\circ}\text{C}$. Then samples were placed in hot zone. The nitrogen flow rate was 0.5 l/min in all the experiments (at initial time in order to remove air 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:- [19&20].



$$\text{Percent of reduction} = \frac{(W_0 - W_t) \times 100}{\text{Oxygen (mass)}}$$

Where:

W_0 : The initial mass of sample after removal of moisture.

W_t : Mass of sample after each time (t).

Oxygen (mass): indicates the mass of oxygen percent in the sample in form FeO & Fe_2O_3 .

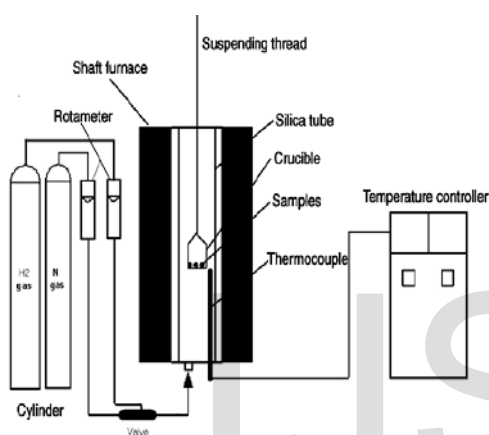


Fig.(4) A schematic diagram of the reduction apparatus.

3. Result and Discussion

3.1 Effect of dolomite addition on physical properties of green briquette without burning

The effect of addition varying percentage of dolomite on the green iron ore briquette properties (briquettes pressed under pressure = 196.133MPa.) is shown in Figures (5 & 6). From these figures, it is clear that increase dolomite addition leads to a decrease the drop damage resistance (drop number of the briquette) while the compressive

strength of green briquettes increases.

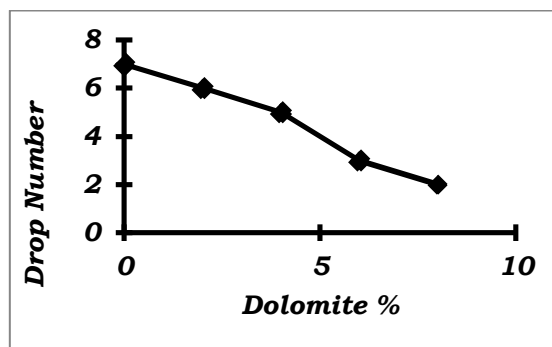


Fig.(5) The effect of adding varying percentage of dolomite on the drop number of green iron ore briquette.

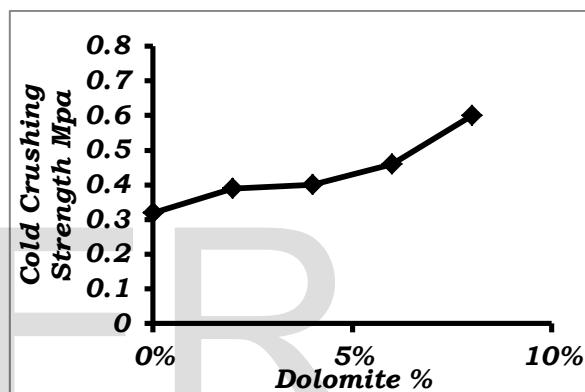


Fig.(6) The effect of adding varying percentage of dolomite on the crushing strength of green iron ore briquette.

3.2 Effect of dolomite addition on physical properties of burned green briquette under different temperatures.

The effect of adding varying percentage of dolomite on the burned green iron ore briquette properties (briquettes pressed under pressure = 196.133 MPa, burning temperature from 900°C to 1200°C is shown in Figure (7). From this figure, it is clear that at constant amount of dolomite addition the increase of burning temperature leads to an increase in the compressive strength of

briquettes. While at any constant sintering temperature the increase in the amount of dolomite decreased the crushing strength this may be due to the remove of carbon dioxide of dolomite.

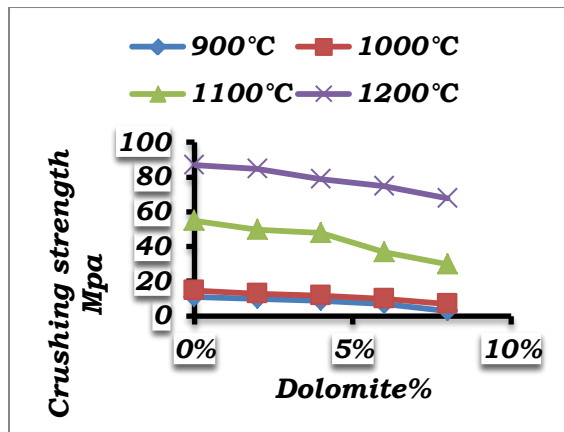


Fig.(7) Effect of different sintering temperatures on the cold crushing strength of the iron ore with different amount of dolomite briquette sample.

3.3 Effect of dolomite addition on reduction of iron ore with dolomite burned at 1200°C.

Fig.(8) illustrates the reduction percentage of iron ore by hydrogen at temperature 900°C for different briquette sample contains different amount of dolomite. (weight of samples was constant= 10g and hydrogen flow rate was constant= 1.5 l/min) sintered at 1200°C. From this figure, it is clear that the percentage of reduction of iron ore decreased as the percentage of dolomite increased for all samples sintered at temperature 1200°C. Also it is clear that at any constant amount of dolomite the reduction

increased as the time of reduction increased.

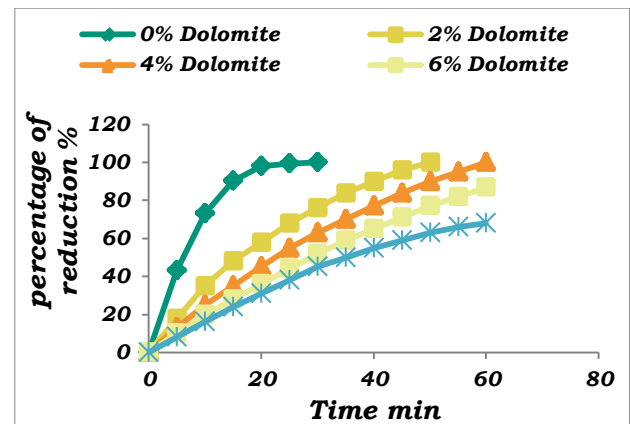


Fig.(8) Effect of dolomite addition on reduction of iron ore with dolomite (burned at 1200°C)

3.4 Effect of temperature variation on the reduction of iron ore which contains dolomite

The effect of temperature on the reduction process for briquette contain 4% and 8% dolomite was studied and the results shown in Figures (9) and (10) which Plots the reduction percentage by 1.5 l/min H₂. From these figures, it can be seen that at constant time of reduction with temperature increasing, the reduction percentage increase. 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 [21-25]. Also the raise of temperature leads to an increase of the rate of mass

transfer of the diffusion and rate of desorption [26-29].

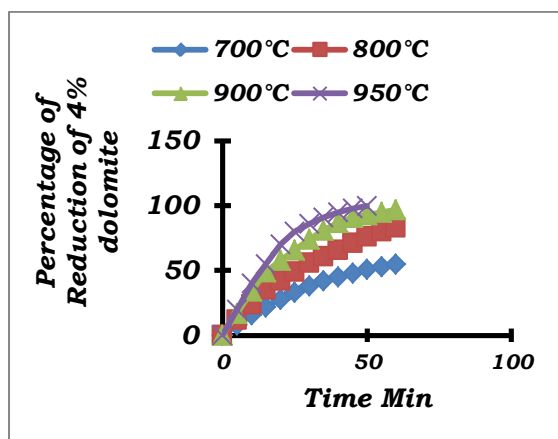


Fig.(9) The effect of different temperatures on the reduction process for briquette contain 4% dolomite.

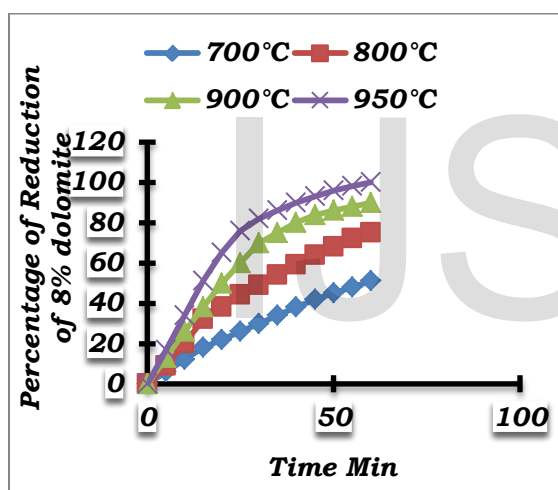


Fig.(10) The effect of different temperatures on the reduction process for briquette contain 8% dolomite.

3.5 Kinetics reduction of briquettes

1- Using diffusion models, Ginstling-Brounshtein equation [30]

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

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

Figs.(11) and (12), illustrate the relation between $1-2/3R-(1-R)^{2/3}$ against time of reduction for different reduction temperature. From which it is clear that the straight line was observed.

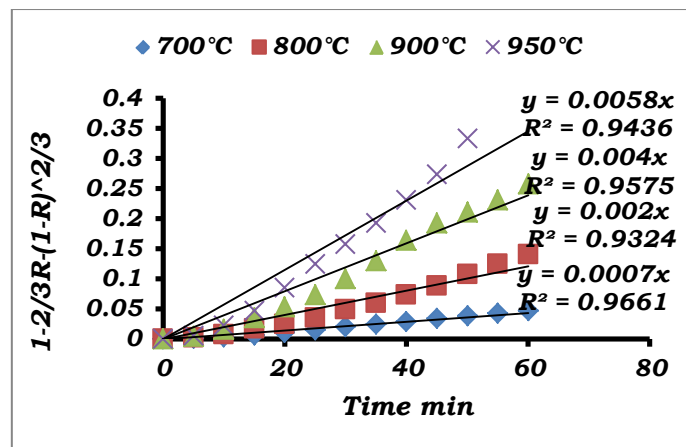


Fig.(11) The relation between $1-2/3R-(1-R)^{2/3}$ against time at different reduction temperature for 4% dolomite briquette.

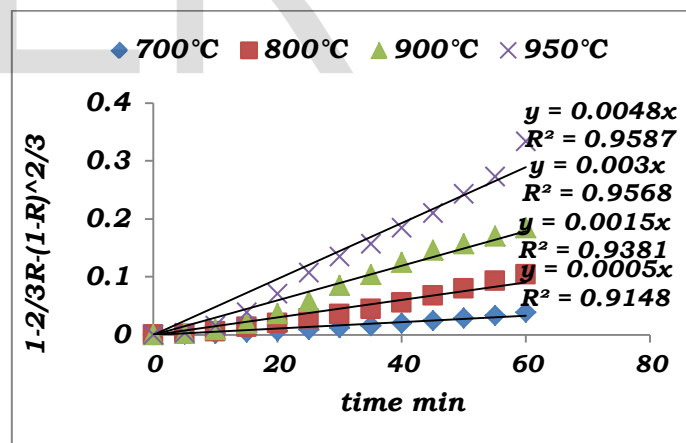


Fig.(12) The relation between $1-2/3R-(1-R)^{2/3}$ against time at different reduction temperature for 8% dolomite briquette.

The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of reduction reaction. The results illustrate in Figs.(13) and (14), from

which it is clear that the activation energy equal to 55.83 kJ/mole and 58.39 kJ/mole for briquette contain 4% and 8% dolomite respectively.

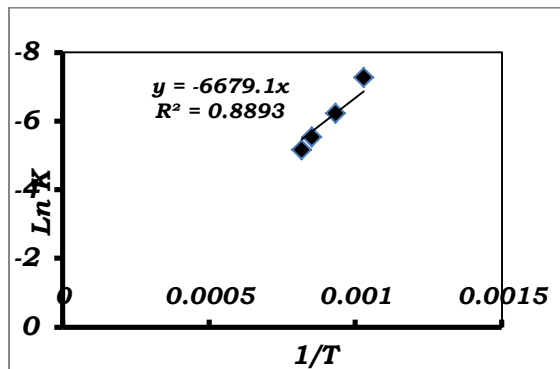


Fig.(13) Relation between the reciprocal of absolute temperature $1/T$ and $\ln K$ (Arrhenius plot for reduction reaction) for model $1-2/3R-(1-R)^{2/3}$ against time for 4% dolomite briquette.

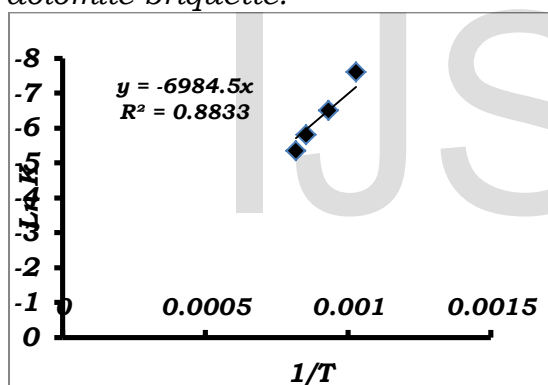


Fig.(14) Relation between the reciprocal of absolute temperature $1/T$ and $\ln K$ (Arrhenius plot for reduction reaction) for model $1-2/3R-(1-R)^{2/3}$ against time for 8% dolomite briquette.

2- Using geometrical contraction models (contracting volume) [30], or diffusion model [31&32]

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

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

Figs. (15) and (16) illustrate the relation between $1-(1-R)^{1/3}$ against time of reduction for different reduction temperature for briquettes containing 4% and 8% dolomite. From which it is clear that the straight line was observed.

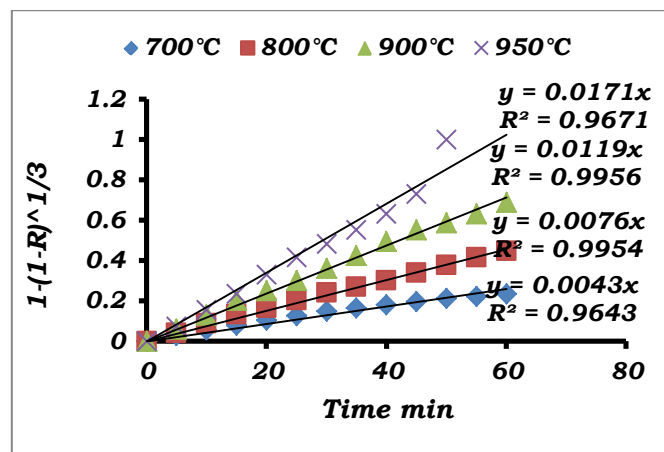


Fig.(15) The relation between $1-(1-R)^{1/3}$ against time of reduction at different reduction temperature for briquettes containing 4% dolomite.

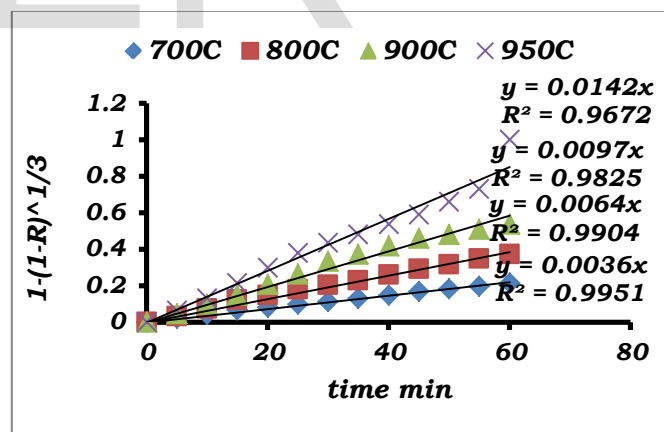


Fig.(16) The relation between $1-(1-R)^{1/3}$ against time of reduction at different reduction temperature for briquettes containing 8% dolomite.

The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of reduction reaction. The results

illustrate in Figs.(17) and (18), from which it is clear that the activation energy equal to 43.44 kJ/mole and 45.12 kJ/mole for briquette contain 4% and 8% dolomite respectively.

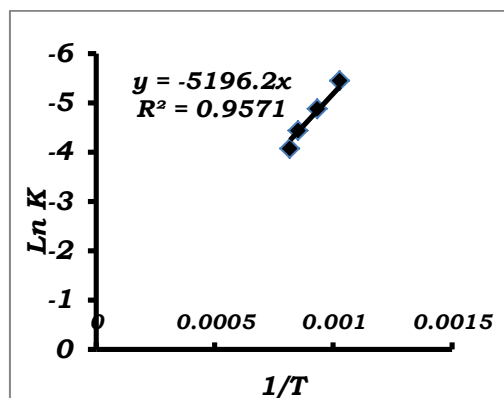


Fig.(17) Relation between the reciprocal of absolute temperature $1/T$ and $\ln K$ (Arrhenius plot for reduction reaction) for model $1-(1-R)^{1/3}$ against time for 4% dolomite briquette.

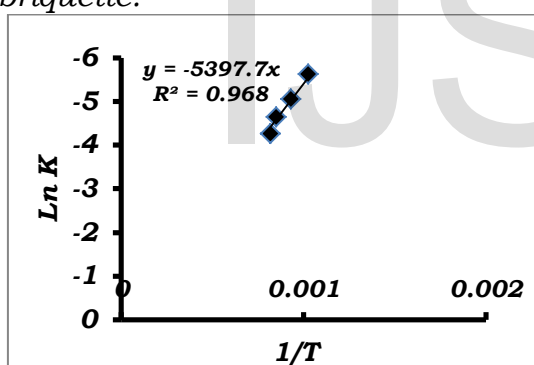


Fig.(18) Relation between the reciprocal of absolute temperature $1/T$ and $\ln K$ (Arrhenius plot for reduction reaction) for model $1-(1-R)^{1/3}$ against time for 8% dolomite briquette.

3.6 X-ray diffraction of the reduced iron ore

Figs.(19) and (20) illustrated the reduction of iron ore containing 4% and 8% dolomite by hydrogen at

950°C respectively from these figures, it is clear the Fe syncetic is mainly elements in both samples.

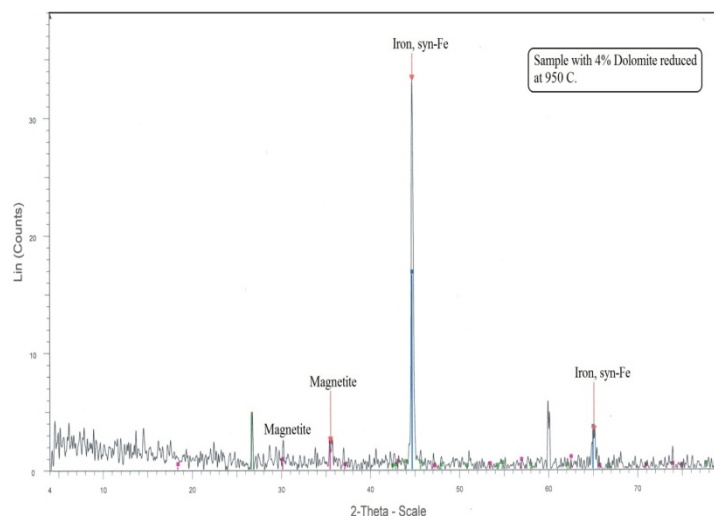


Fig.(19) X-ray of reduced iron ore sample containing 4% dolomite by hydrogen at 950°C.

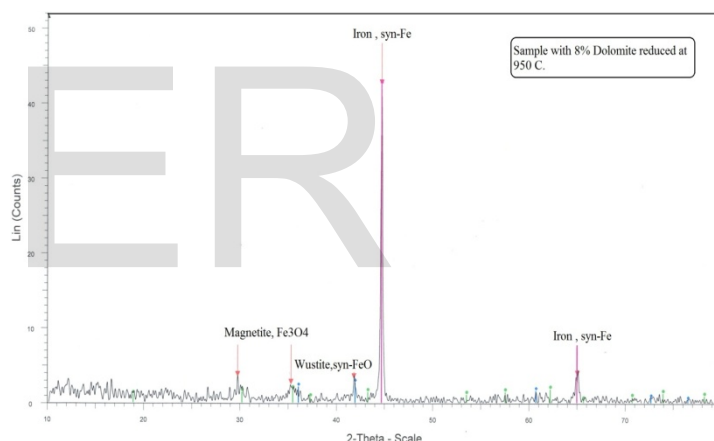


Fig.(20) X- ray of reduced iron ore sample containing 8% dolomite by hydrogen at 950°C.

4. Conclusions

- 1- The reduction of iron ore with dolomite briquette by hydrogen depend on the temperature of the reduction, as the temperature increased the reduction increased.
- 2- As the percentage of dolomite in the briquette increased the reduction rate decreased.

3- *The reduction of the iron ore with dolomite briquette control by one of the following models.*

a- *Diffusion models, Ginstling-Brounshtein equation*

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

b- *Geometrical contraction models (contracting volume) or diffusion model*

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

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