

## THE EFFECT OF ADDING EL-DEKHILLA WASTE PELLETS UPON THE QUALITY AND REDUCTION KINETICS OF EL-BAHARIA IRON ORE BRIQUETTES USING HYDROGEN GAS

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### ABSTRACT

The effect of the addition of El-Dekhilla waste pellets fines (E) upon the quality of El-Baharia iron ore (O) briquettes was investigated in this study. The kinetics of the reduction of the briquettes formed having different composition using hydrogen gas was studied at different temperatures (700°C-950°C). The results indicated that best ore reduction was achieved at 950°C. The kinetic of the reduction process was studied using different models and the best-fit models determined was  $(Kt = 1-(1-R)^{1/2})$  and  $kt = R + (1-R) \ln(1-R)$ . XRD analysis was used to identify the main crystalline phases present within the reduced briquettes at the optimal temperature and it was found that the metallic iron (syn. Fe) was predominantly formed.

**Keywords:** El-Baharia iron ore (O), El-Dekhilla waste pellets fine (E), Iron ore reduction kinetics.

### 1. INTRODUCTION

The steel industry in Egypt represents an important sector of the economy that provides over 19.2% of the industrial GDP in 2002 [1]. During the early nineties, there has been a surge of investment in this industry's private sector in order to end the government control of steel production capacities and to cope with the increase in demand for steel rebars [1]. Although Egypt has established large iron ore resources, the local ore has a lower concentration ratio than the industrial requirements, a fact that made most of the private steel companies import raw materials to satisfy their production needs [1].

El-Dekhilla Iron Co. is one of the leading private steel producers in the Middle East Region [1]. The company controls around 54 % of the total market potential capacity and 61 % of the market share. In 2003, the plant produced 3.5 million tons and exported 0.85 million tons and is considered the largest steel exporter among the Arab nations [1].

The company has a production capacity for long steel stands of 1.8 million tons per annum and an annual capacity of one million tons for flat steel. Production in the company depends mainly upon importing iron ore pellets and high-grade scrap (15%) as the main feedstock; with the latter becoming, the main feed stock [2].

The Egyptian Iron and Steel Company, on the other hand, is the largest and only public owned firm that produced iron and steel from local resources since 1959[3-4]. The

company has been exploiting Egypt's largest iron ore resources located at El-Baharia Oasis (Western Desert) since 1973. However, a survey of the composition of the ore indicated that this iron ore has a low iron content (reaching 44.10% as iron oxide) and silica (36.81%) with iron being concentrated in the fine fraction (to more than 38% Fe). Furthermore, the chemical composition of this ore was highly variable going from one mine location to another in El-Baharia oasis [4].

Nonetheless, with a strategic reserve of over 144 million tons, conditioning of this ore to increase the fine content of Fe to over 46% prior to industrial processing is crucial for the up keeping of the company's production [3-4].

From another perspective, the demand for iron ore has increased significantly in the past decades, a matter that calls for the urgent recovery of all viable and available resources from mining wastes or upgrade lean-grade iron ore through beneficiation [5]. This is because millions of tons of these wastes contain useful minerals that are being discarded in the form of fines and slimes, which may have serious waste related environmental impacts. As well, recycling of iron bearing materials have great economical importance as it allows for the utilization of the iron present and preventing of mineral loss as well as it helps in the fuel rate reduction during the process [6-7].

Jouhari et al. (2000)[8] indicated that large amounts of fines were generated in the mechanized mining as well as low grade ores grinding during beneficiation before preparation of concentrates. Prior to the introduction into the blast furnace, this ore requires enlargement of its particle size for iron making which is achieved through sintering. In this case, fine wastes from sponge iron fines and ferruginous materials may be incorporated with the charge mix during the preparation of sinters.

In the case of the steel industry in Egypt, it was reported that the waste loss due to transportation of iron pellets raw materials used at El-Dekhilla Iron Co was estimated to be between 3-10%. These iron ore pellets waste fines are characterized by a high iron oxide and low silica content [9].

Previous studies have indicated that the addition of iron oxide wastes from 2.5 to 5% can improve upon the technical parameters of the ore sintering process [4,9]. As well, it was reported that the use of iron oxide wastes in steel production together with El-Baharia ore leads to a decrease in both the amount of limestone and the amount of crystalline water (which is found in the iron ore replaced) [9]. Moreover, iron oxide pellets fine replacement improves the granulation process (performed on the raw mixture before sintering process) which leads to improving of the sinter charge permeability during the sintering process as well it increases the amount of melt formation [9]. Overall, they indicated that replacement of iron ore concentrate with 10% iron oxide pellets fine increased the amount of readymade sinter, sinter strength and productivity of both sinter machine and blast furnace yard.

Wagner et al. (2006)[10] studied the reduction of iron ores using hydrogen gas instead of carbon dioxide in an attempt to reduce the greenhouse emissions related with the steel industry. They indicated that the reduction of iron ores using hydrogen was a gas-solid reaction, which occurs in two or three stages. At temperatures above than 570°C,

hematite ( $\text{Fe}_2\text{O}_3$ ) was first transformed into magnetite ( $\text{Fe}_3\text{O}_4$ ) then into wüstite ( $\text{Fe}_{1-y}\text{O}$ ), and finally into metallic iron. However, at temperatures below  $570^\circ\text{C}$ , the magnetite was directly transformed into iron since wüstite was not thermodynamically stable. Compared to ore reduction with carbon monoxide, the reduction with hydrogen was reported to be endothermic while it was exothermic using carbon monoxide, with the former being thermodynamically more favorable above  $800^\circ\text{C}$ . In addition to that, the kinetics using hydrogen gas was reported to be faster than that utilizing carbon monoxide.

Longbottom and Kolbeinsen (2008) [11] also studied the reduction of iron ore using hydrogen and carbon dioxide. They indicated that the reduction using both hydrogen and carbon monoxides was similar and it did proceed in three steps when temperatures were over  $570^\circ\text{C}$  while at temperatures below  $570^\circ\text{C}$ , it was a two steps process. In addition to that, they concluded that utilization of hydrogen achieved the best reduction at higher temperatures and that the process was generally endothermic. Comparatively, the use of carbon monoxide was preferred at lower temperatures and the process was mildly exothermic.

The aim of the current study is to investigate the process of reduction of briquettes formed from iron El-Baharia iron ore incorporating El-Dekhilla Iron Co iron oxide fine waste using molasses as binder. The reduction of the formed briquettes was done in a static bed using hydrogen gas at different flow rates. The thermodynamics of the process was explained based upon the experimental results and the kinetics was derived thereafter.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Materials and Methods

#### 2.1.1. Raw materials characterization

El-Baharia iron ore (O) was supplied by the Egyptian Iron and Steel Company, Helwan, Egypt. The chemical composition of this ore is indicated in table 1 [4].

XRD analysis of the obtained El-Baharia iron ore is also shown in figure 1. From this figure, it is clear that El-Baharia iron ore mainly consists of hematite and quartz.

**Table (1): Chemical analysis of El-Baharia iron ore (%wt/wt).**

Chemical composition	% wt /wt	Chemical composition	% wt /wt
Fe total	52.35 %,	TiO <sub>2</sub>	0.16% ,
MnO	2.92%,	BaO	1.17%,
SiO <sub>2</sub>	10.84%,	ZnO	0.15%,
CaO	0.39%,	K <sub>2</sub> O	0.27%,
MgO	0.18%,	Na <sub>2</sub> O	0.25%,
Al <sub>2</sub> O <sub>3</sub>	1.44% ,	P <sub>2</sub> O <sub>5</sub>	0.5 %
S	0.74%,		

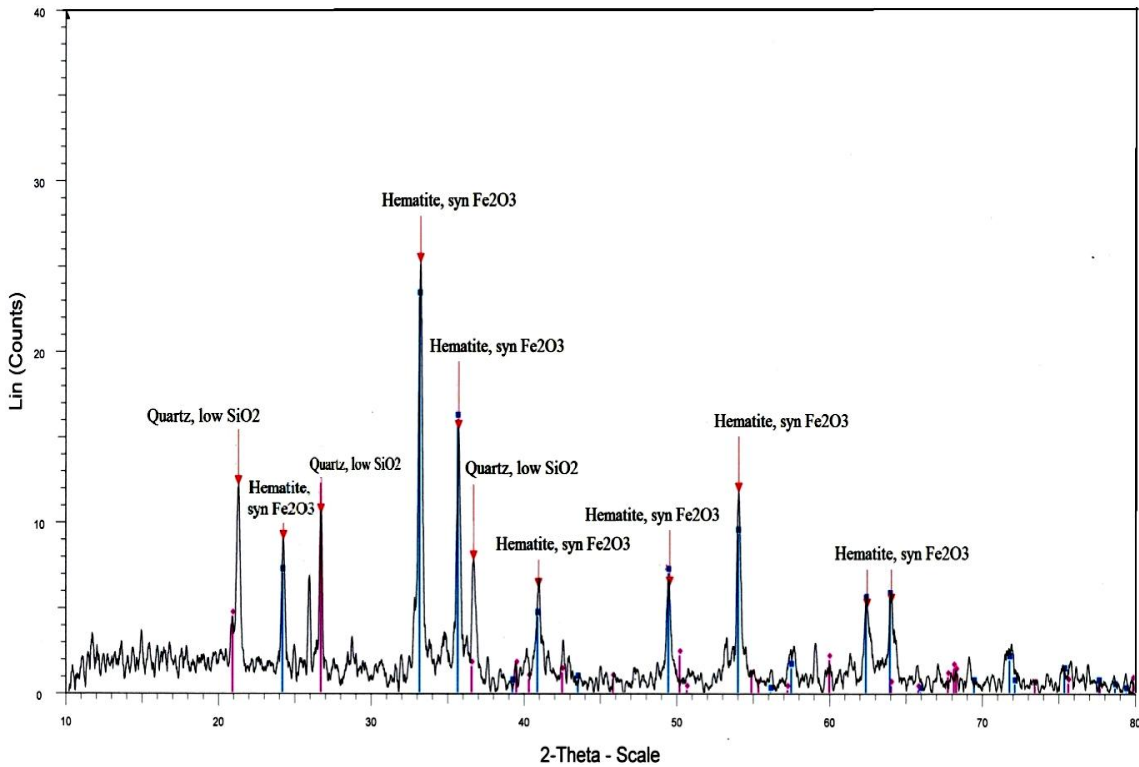


Fig. 1: XRD analysis El-Baharia iron ore.

El-Dekhillah waste pellets (E) used in this work was obtained from El-Dekhillah Steel Company, Alexandria, Egypt. The chemical analysis of these fines is as follows: Fe total = 66.5%,  $\text{Fe}_2\text{O}_3$  = 95%,  $\text{SiO}_2$  = 1.5% and  $\text{CaO}$  = 0.7%. XRD of the El-Dekhillah pellets waste is illustrated in figure 2 and it shows that the main compound of this waste was hematite.

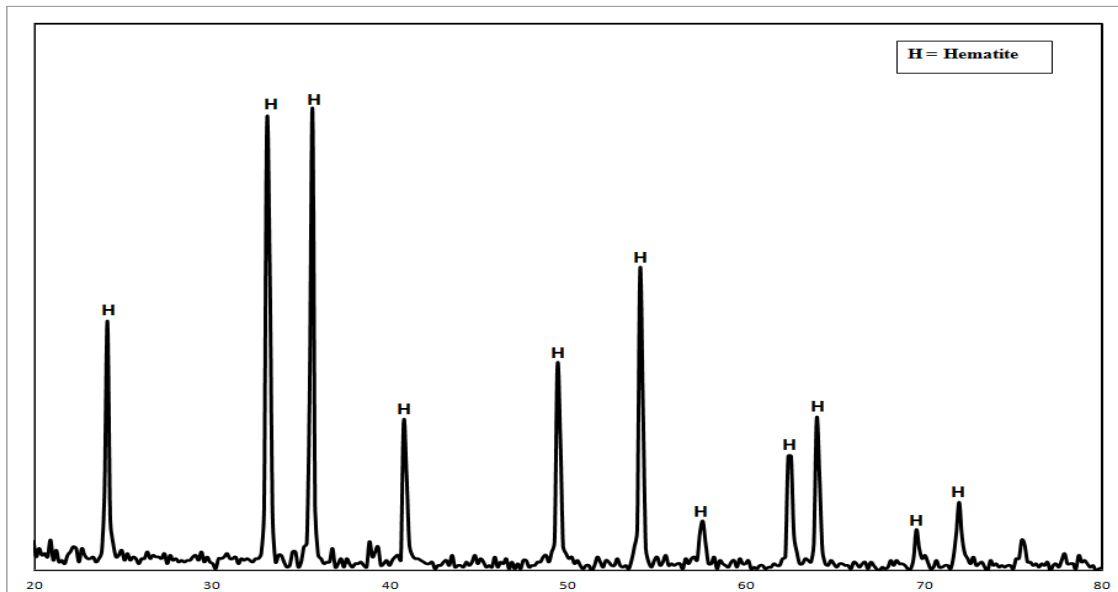


Fig. 2: XRD of El-Dekhillah pellets waste.

### **2.1.2. Preparation of iron ore briquettes and the characterization of its physical properties**

El-Baharia iron ore and El-Dekhilla waste pellets were ground separately in a vibrating mill to powder to achieve particle size less than 75  $\mu\text{m}$ . 10 g of each fine were mixed together with 3% molasses as binder and then pressed in a mould (12 mm diameter and height 22 mm) using MEGA.KSC-10 hydraulic press under different pressure (from 75 MPa up to 275 MPa). The briquettes were subjected to drop number test and crushing strength tests. The drop number indicates how often the prepared green briquette can be dropped from a height 46 cm before they show perceptible cracks or crumble.

Ten green briquettes were individually dropped onto a steel plate. The number of drops was determined for each briquette and the arithmetical mean values of the crumbling behavior of the briquettes yield the drop number. The average crushing strength was done by compressing the 10 briquettes individually between parallel steel plates up to their breaking point [12].

### **2.1.3. Briquette reduction procedures**

Reduction of the prepared briquettes under hydrogen gas was done using TGA apparatus [13-19]. The sample was placed in a Ni-Cr basket, which was suspended below the electronic balance by Ni-Cr wire. During the experimental run, the furnace temperature was raised to the required temperature (700-950  $^{\circ}\text{C}$ ) and was maintained constant ( $\pm 5$   $^{\circ}\text{C}$ ) and the nitrogen flow rate was kept at 0.5 L/min during all the experiments.

Prior to the introduction of the samples into the hot zone, the samples weights were initially recorded. During the experimental run, the sample weight was continuously monitored. The final weight of the sample was obtained at the end of the run as the samples were withdrawn from the furnace and kept in a desiccator to cool down to room temperature. The amount of removable oxygen was determined by the weight loss in the sample ( $W_o - W$ ) during the reduction with  $\text{H}_2$  in the furnace. The percentage of reduction was calculated according to the following equation:

$$\% \text{ reduction} = \frac{(W_o - W_t)}{W_o} \times 100 \dots\dots\dots (1)$$

Where  $W_o$  is the initial mass of sample in g and  $W_t$  is the mass of the sample after time,  $t$  min. The oxygen (mass) indicates the mass of oxygen present in the briquette in the form  $\text{Fe}_2\text{O}_3$ .

## **3. RESULTS AND DISCUSSION**

### **3.1. The Effect of Pressure on the Physical Properties of the Produced in Green and Dried Forms**

Figures 3 to 10 show the relationship between the change of pressure load vs. the drop damage resistance and compressive strength of the wet and dried briquettes with E/O ratio of 0.0526 and 0.25, respectively, at a constant amount of molasses (3%) as binder.

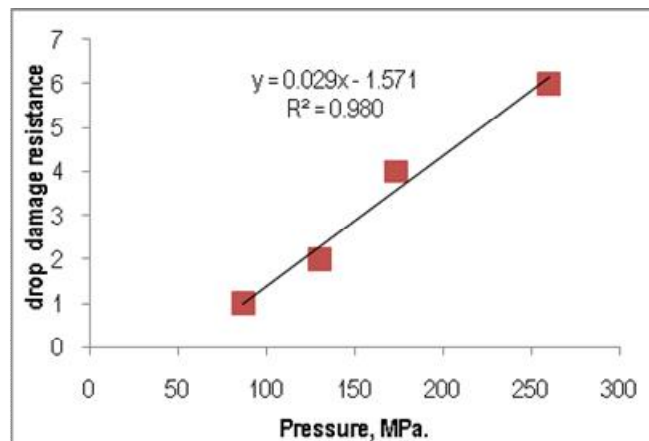


Fig. 3: The relationship between the change in pressure load vs. the drop damage resistance of the wet briquettes having E/O ratio = 0.0526 at constant amount of molasses (3%) binder.

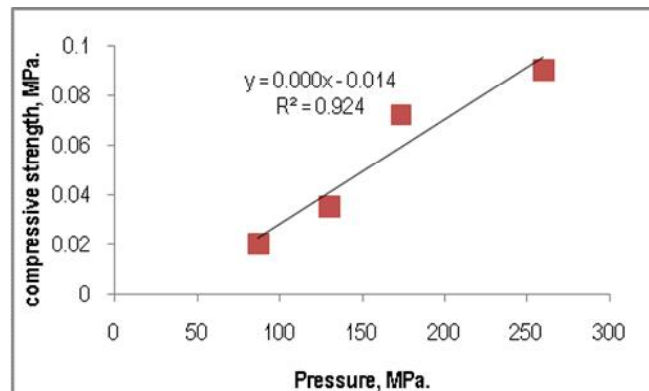


Fig. 4: The relationship between the change in pressure load vs. the compressive strength of the wet briquettes having E/O ratio 0.0526 at constant amount of molasses (3%) as binder.

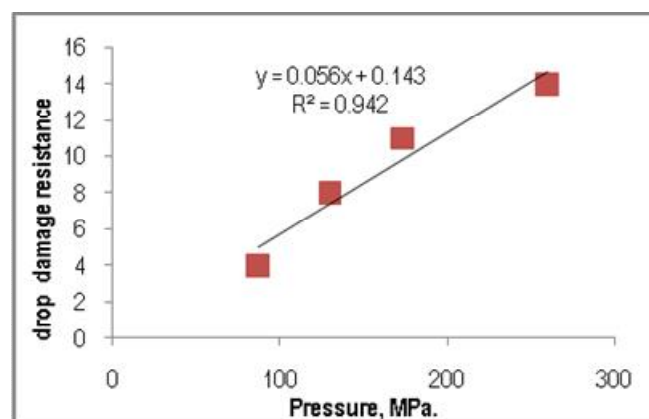
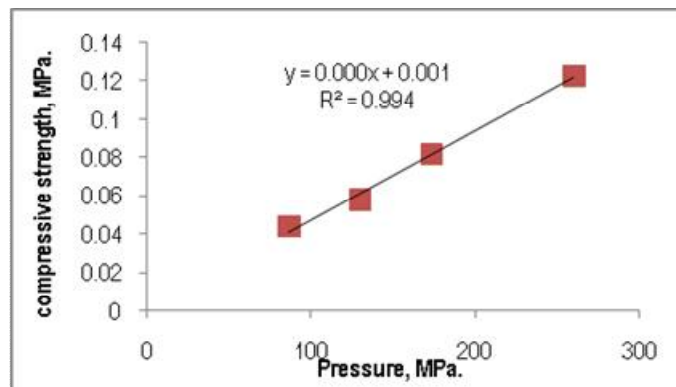
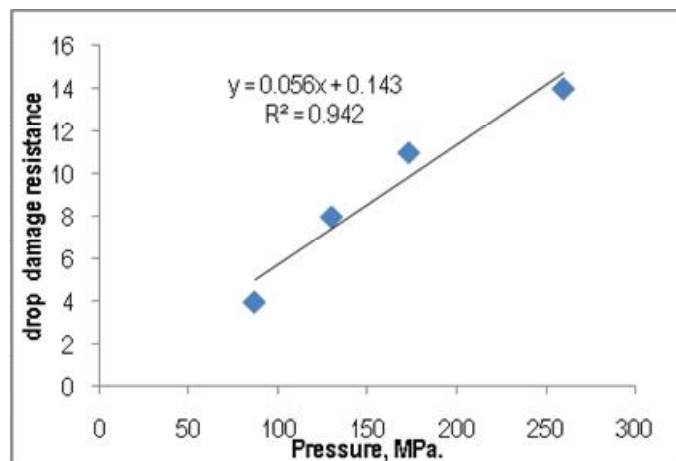


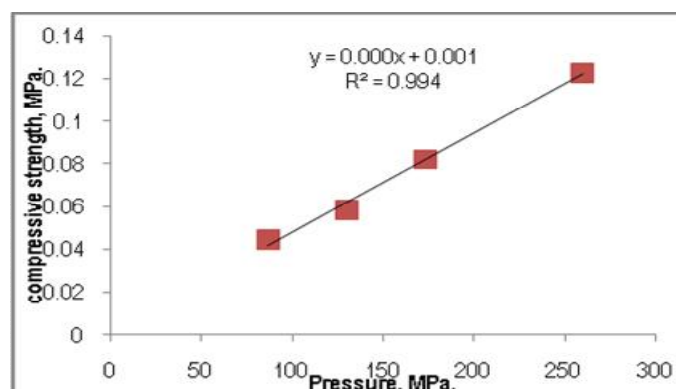
Fig. 5: The relationship between the change in pressure load vs. the drop damage resistance of the dried briquettes having E/O ratio = 0.0526 at constant amount of molasses (3%) binder.



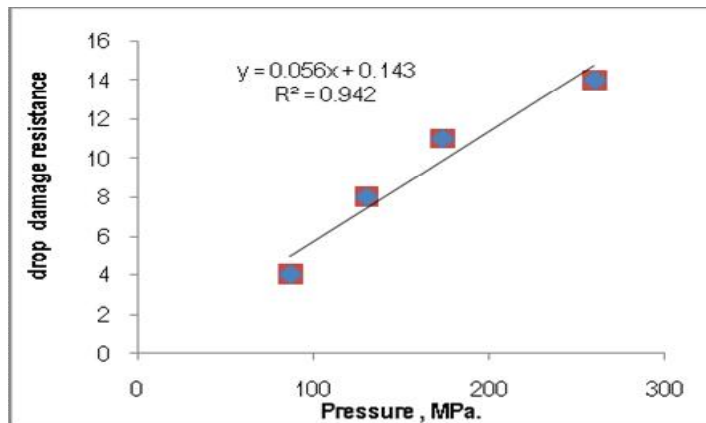
**Fig. 6:** The relationship between the change in pressure load vs. the compressive strength of the dried briquettes having E/O ratio 0.0526 at constant amount of molasses (3%) as binder.



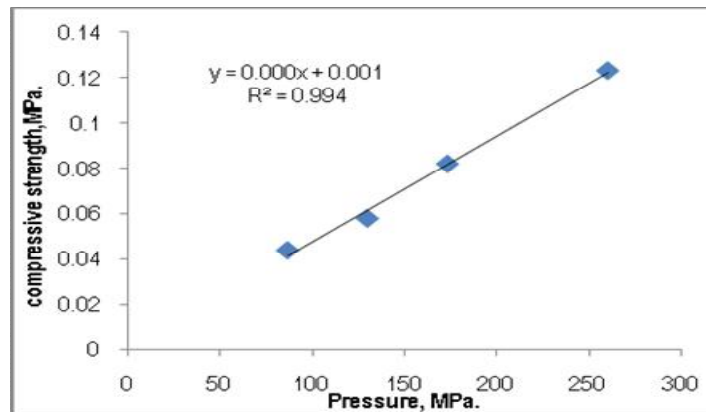
**Fig. 7:** The relationship between the change in pressure load vs. the drop damage resistance of the wet briquettes having E/O ratio= 0.25 at constant amount of molasses (3%) binder.



**Fig. 8:** The relationship between the change in pressure load vs. the compressive strength of the wet briquettes having E/O ratio = 0.25 at constant amount of molasses (3%) as binder.



**Fig. 9: The relationship between the change in pressure load vs. the drop damage resistance of the dried briquettes having E/O ratio = 0.25 at constant amount of molasses (3%) binder.**



**Fig. 10: The relationship between the change in pressure load vs. the compressive strength of the dried briquettes having E/O ratio = 0.25 at constant amount of molasses (3%) as binder.**

From these figures, it is evident that as the pressure load increases, both the drop damage resistance and compressive strength increased linearly. This may be due to the fact that increasing the pressure leads to an increase in the briquette compaction which leads to an increase in the inherent Van der Waals forces present [20-21]. As well, increasing the pressure load may have progressively crushed and decrease the macro pores present within the briquettes [20- 22].

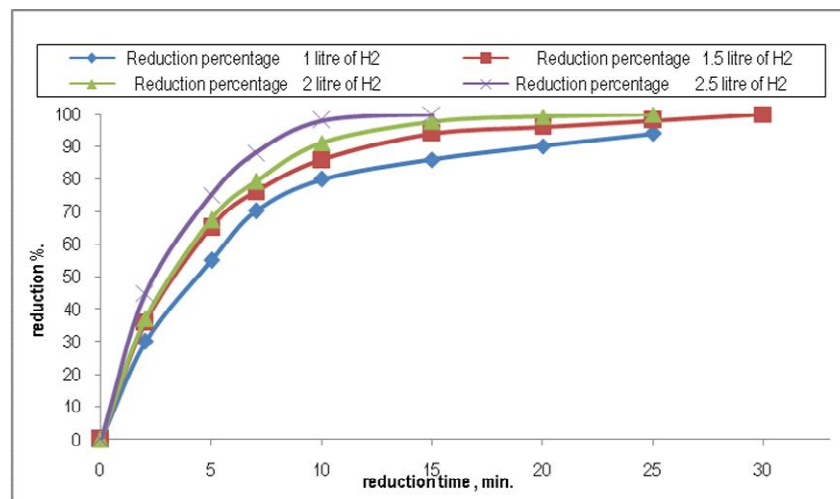
### **3.2. The Effect of Hydrogen Flow Rate upon the Reduction of E/O Mixture**

Figures 11 and 12 show the effect of changing the hydrogen flow rate upon the reduction of the briquettes pressed at 261 MPa at 900 °C for both E/O = 0.0526 and 0.25, respectively. From these figures, it is evident that as the flow rate increased the reduction percentage increased. Zuo et al. (2015)[23] indicated that at low hydrogen content, increasing the temperature takes the transition point of the rate-control step to a high reduction degree while at high hydrogen content, the effect of temperature on the transition point weakens.

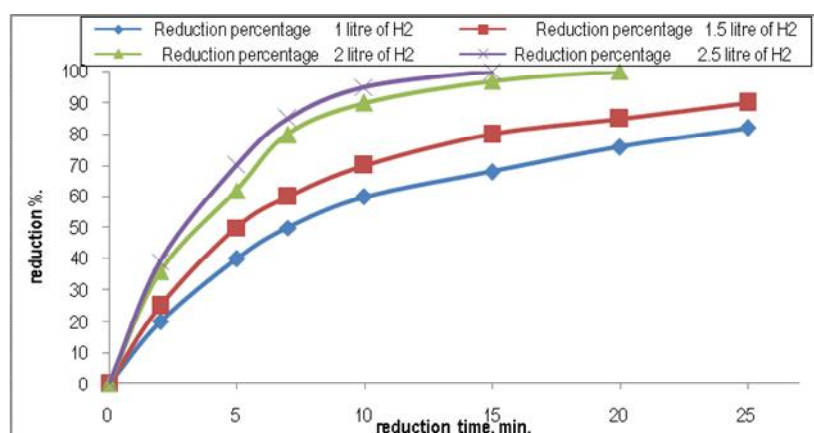


The increase in flow rate may increase the gas diffusion across the boundary layer increasing the rate of the reaction [21,22,24]. This may attributed to the fact that increasing the flow rate leads to the increase in the number of hydrogen moles passing through the bulk phase, which in turn leads to an increase in hydrogen adsorption onto the briquette material.

Zuo et al. (2015)[23] also indicated that the reaction rate increases gradually with the increase of H<sub>2</sub> content which may be attributed to the higher penetration capacity of H<sub>2</sub>. As well, there was a decline in diffusion resistance, as H<sub>2</sub> becomes a dominating factor. On the other hand, the prevailing increase in flow rate in the reaction zone enhances the rate of hydrogen absorption and subsequently the rate of chemical reaction increases [21,22,24,25]. Moreover, it was indicated that hydrogen had a role in improving the increasing of the rate constant that may be attributed to the endothermic reaction of hydrogen reduction [23].



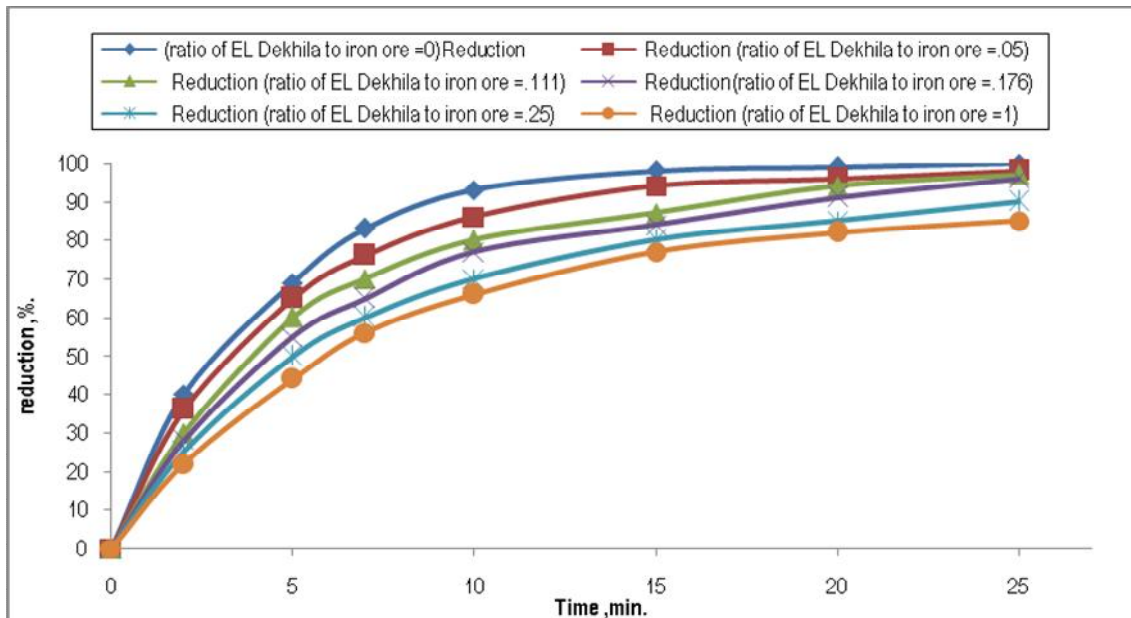
**Fig. 11:** The effect of changing the hydrogen flow rate upon the reduction of the briquette pressed at 261 MPa at 900 °C containing E/O ratio= 0.0526.



**Fig. 12:** The effect of changing the hydrogen flow rate upon the reduction of the briquette pressed at 261 MPa at 900 °C containing E/O ratio = 0.25.

### 3.3. The Changing of the E/O Ratio upon the Reduction Process at 900°C

Figure 13 shows the effect of changing the E/O ratio upon the reduction process within the formed briquette (pressed at 261 MPa) at 900°C. The figure indicates that as the E/O ratio increased the reduction percentage decreased which may be attributed to the loss of porosity in briquette due to the increase in melt within.



**Fig. 13: The effect of varying the E/O ratio upon the ore reduction process at 900°C.**

### 3.4. The Effect of Temperature upon the Reduction Percentage

In order to examine the effect of temperature upon the reduction of E/O briquettes formed, the experiments were carried out at 700 – 950°C and under 2 L/min hydrogen flow rate. The plots of the reduction percentage versus time are shown in figures 14 and 15.

From these figures, it was observed that the reduction temperature did significantly influence the reduction percentage. This may be due to the fact that as the reduction percentage increases with increase in temperature as the number of reacting moles having excess of energy increased. Moreover, the raise of temperature may lead to an increase of the rate of mass transfer through diffusion as well as the rate of desorption [26-29].

Zuo et al. (2015) [23] indicated that rate constant of the chemical reaction speeds up with the increase in the reaction temperature in the presence of hydrogen gas. Moreover, using pure H<sub>2</sub> as the reducing agent decreases both the resistance of diffusion and chemical reaction with the rise of temperature due to the endothermic reaction of the gas [23]. This leads to a higher reducing potential at high temperatures which enhances the reaction rate as well the increase in temperature contributed to a high mass transfer which increases the gas mobility and decreases the gas diffusion resistance.

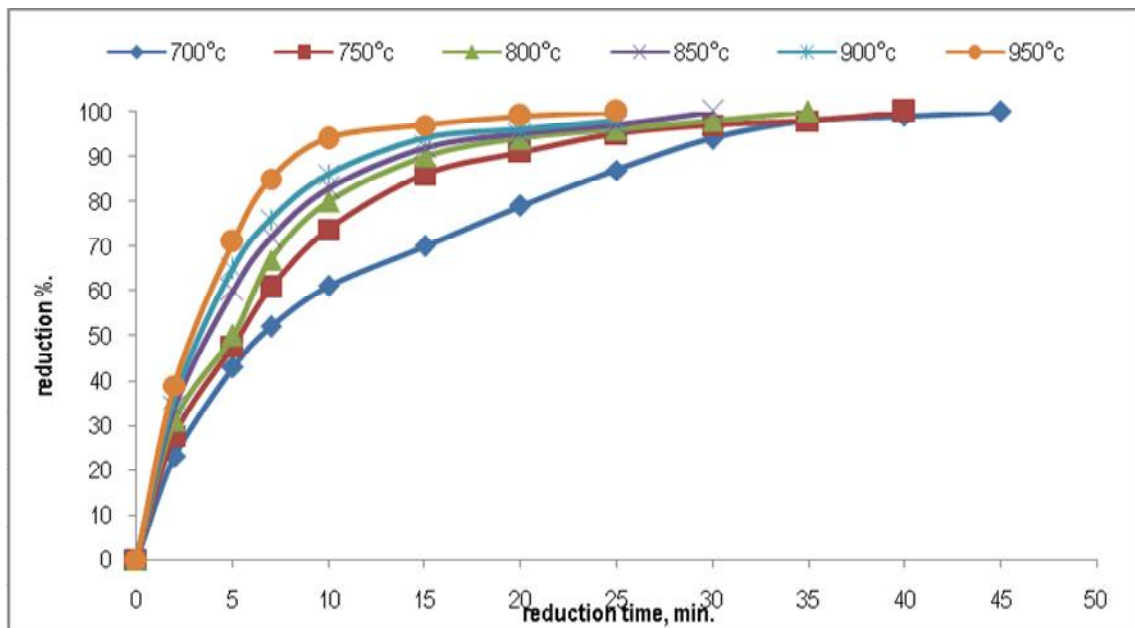


Fig. 14: The effect of temperature upon the reduction percentage of briquettes containing E/O ratio =0.0526.

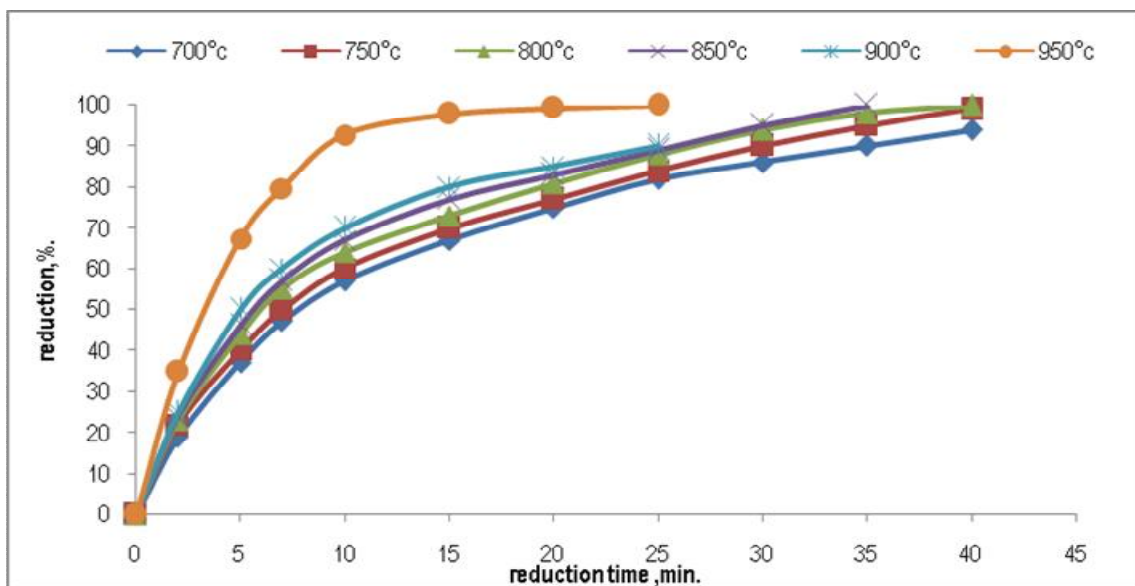


Fig. 15: The effect of temperature upon the reduction percentage of briquettes containing E/O ratio = 0.25.

### 3.5. Kinetics of the Reduction Process

Kinetic studies for estimation of the apparent activation energies of the process were carried out at different temperatures from 700°C - 950°C for briquettes formed at different time intervals (0 - 60 min). Using equation  $kt=1-(1-R)^{0.5}$  and equation  $kt= R+ (1-R) \ln (1-R)$  for both E/O ratio 0.0526 and 0.25 respectively for briquettes of E/O ratio 0.0526 and 0.25 [30-31].

Where R is the fractional reacted of solid reactant, t is the time and k is the rate constant.

Figures 16 and 17 illustrate the relationship between  $1-(1-R)^{0.5}$  versus reduction time (t) for the different reduction temperatures for briquettes from with E/O ratio of 0.0525 and 0.25, respectively. From both figures, a linear relationship was observed.

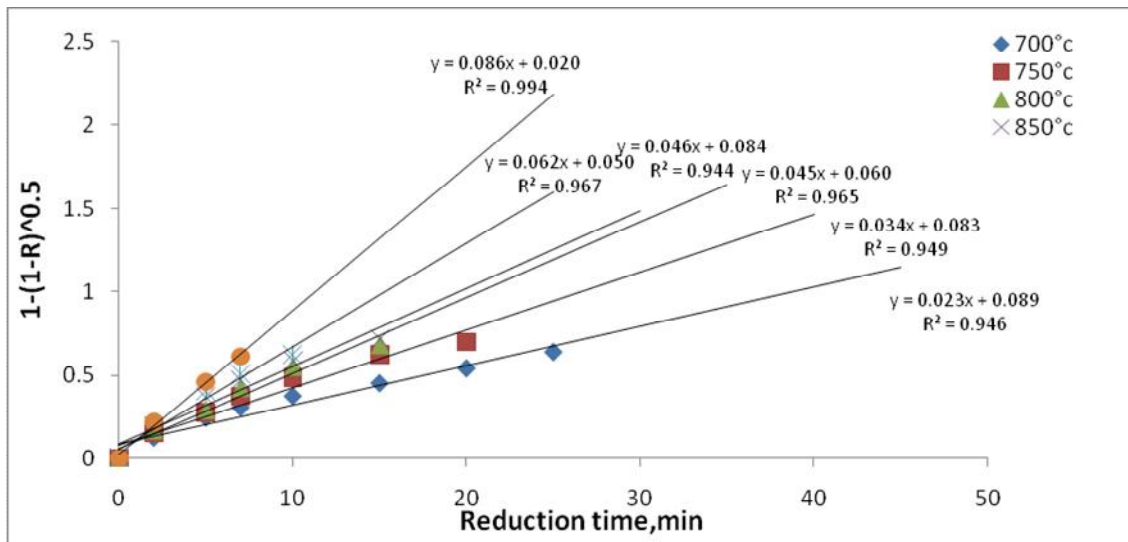


Fig. 16: The relationship between  $1-(1-R)^{0.5}$  versus reduction time (mins) at the different reduction temperatures for briquettes from with E/O ratio of 0.0526.

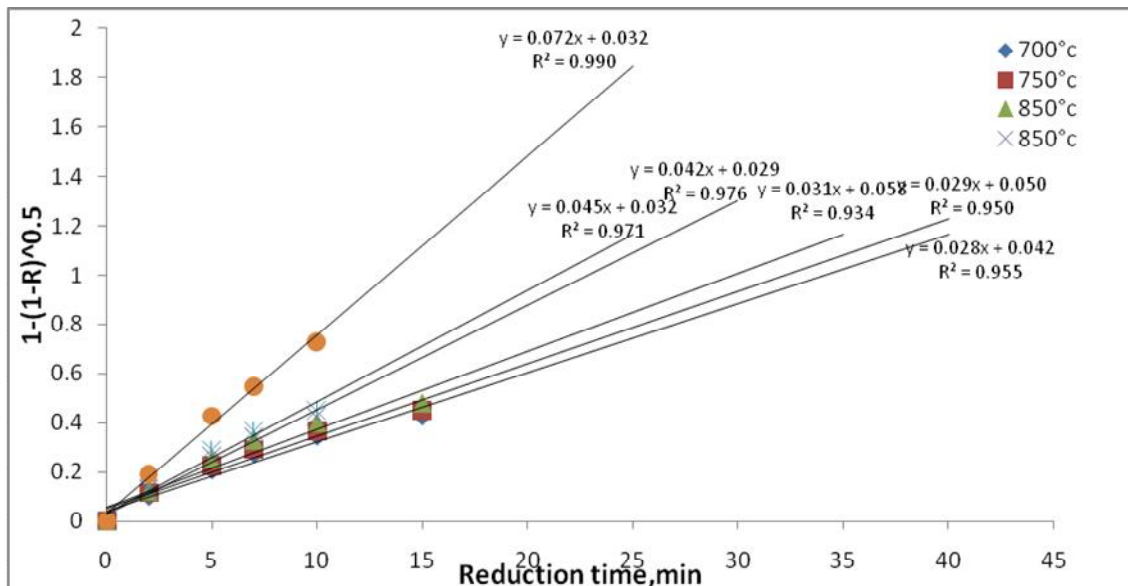
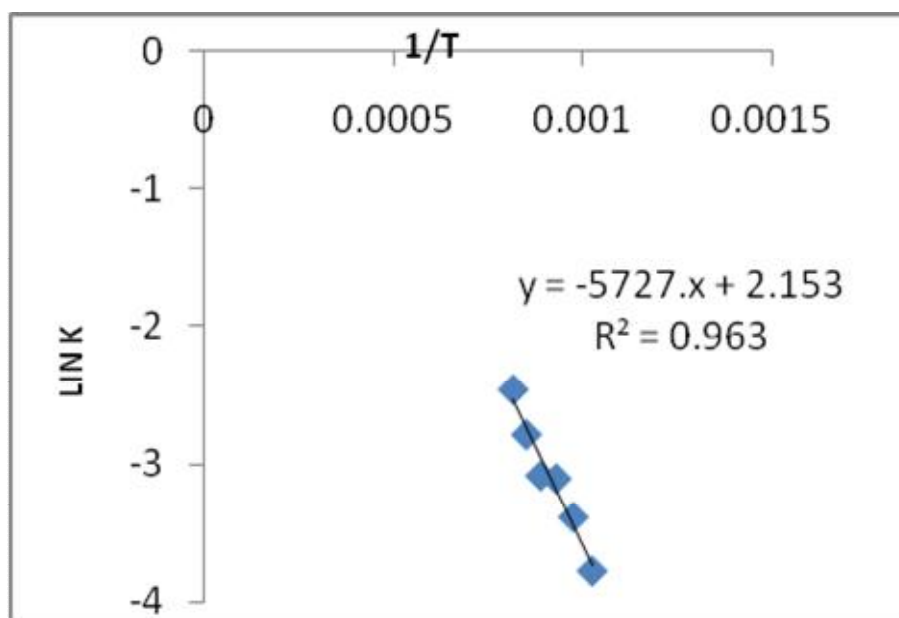
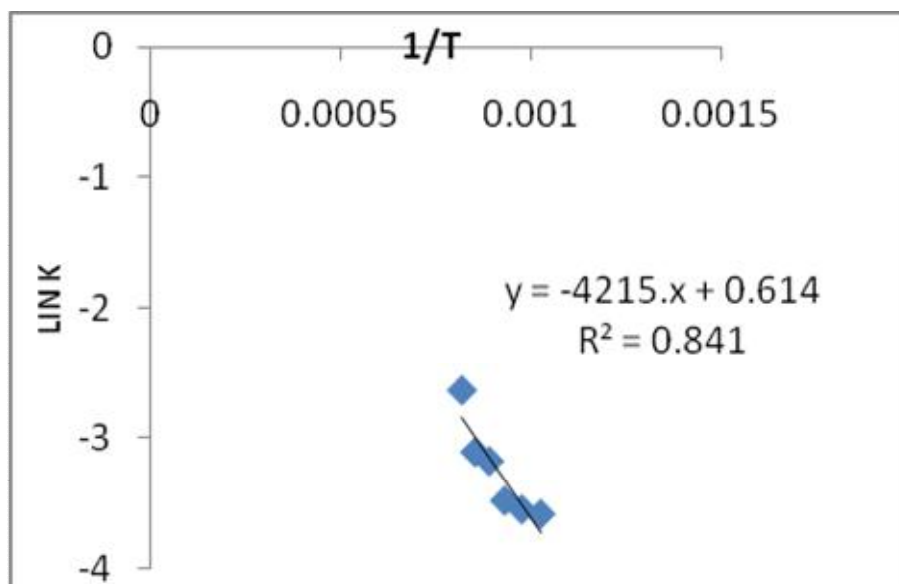


Fig. 17: The relationship between  $1-(1-R)^{0.5}$  versus reduction time (mins) at the different reduction temperatures for briquettes from with E/O ratio of 0.25.

The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of the reduction process of briquettes from with E/O ratio of 0.0526 and 0.25, respectively. The results are illustrates in figures 18 and 19. The activation energies calculated for this process for the briquettes formed with E/O ratio of 0.0525 and 0.25 was = 47.39 kJ/ mole and 34.88 kJ/ mole, respectively.



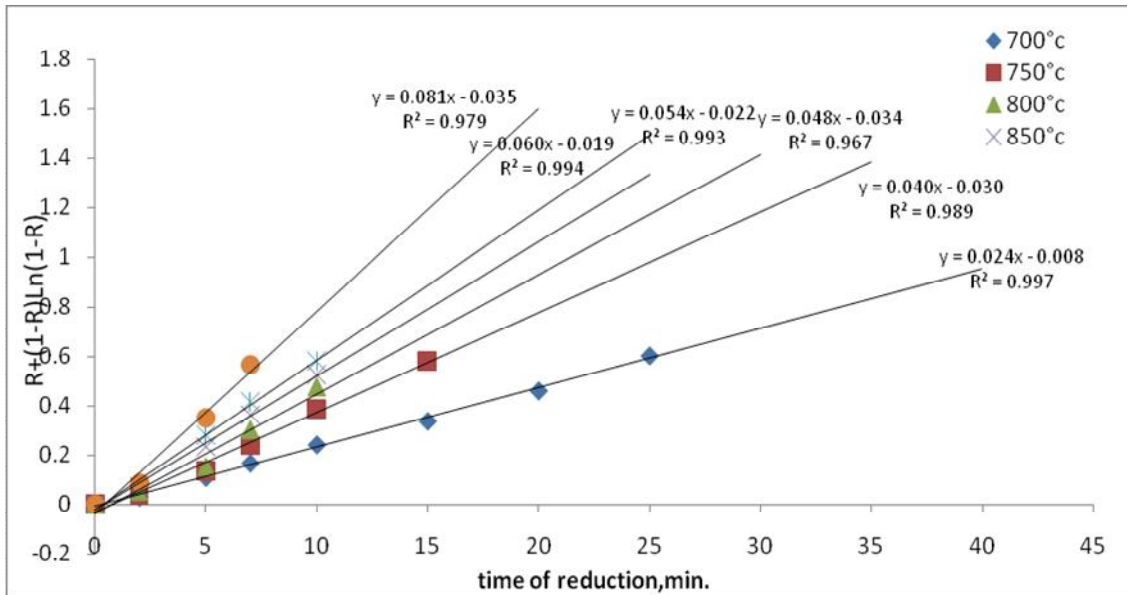
**Fig. 18: The Arrhenius plot for the reduction process of briquettes from with E/O ratio of 0.0526.**



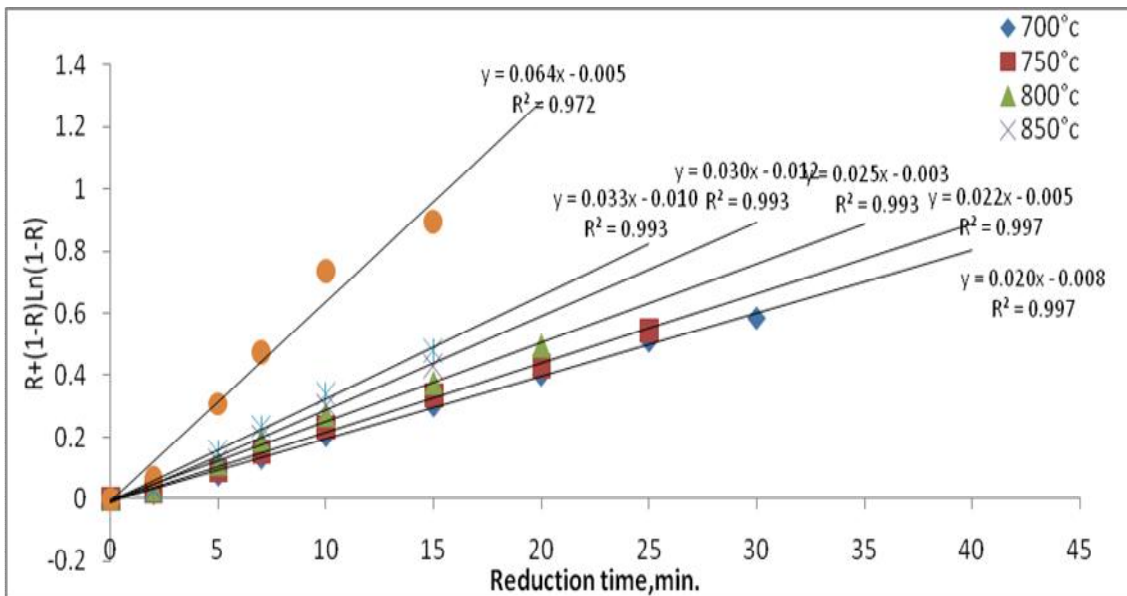
**Fig. 19: The Arrhenius plot for the reduction process of briquettes from with E/O ratio of 0.25.**

Figures 20 and 21 illustrate the relationship between  $R + (1-R) \ln(1-R)$  versus reduction time (t) for the different reduction temperatures for briquettes from with E/O

ratio of 0.0525 and 0.25, respectively. From both figures, a linear relationship was observed.



**Fig. 20:** The relationship between  $R+(1-R) \ln(1-R)$  versus reduction time (mins) at the different reduction temperatures for briquettes from with E/O ratio of 0.25.



**Fig. 21:** The relationship between  $R+(1-R) \ln(1-R)$  versus reduction time (mins) at the different reduction temperatures for briquettes from with E/O ratio of 0.25.

The natural logarithms were used according to the Arrhenius equation to calculate the activation energies of the reduction process of briquettes from with E/O ratio of 0.0525 and 0.25, respectively. The results are illustrated in figures 22 and 23. The activation

energies calculated for this process for the briquettes formed with E/O ratio of 0.0525 and 0.25 was = 42.15kJ/ mole and 39.62 kJ/ mole, respectively.

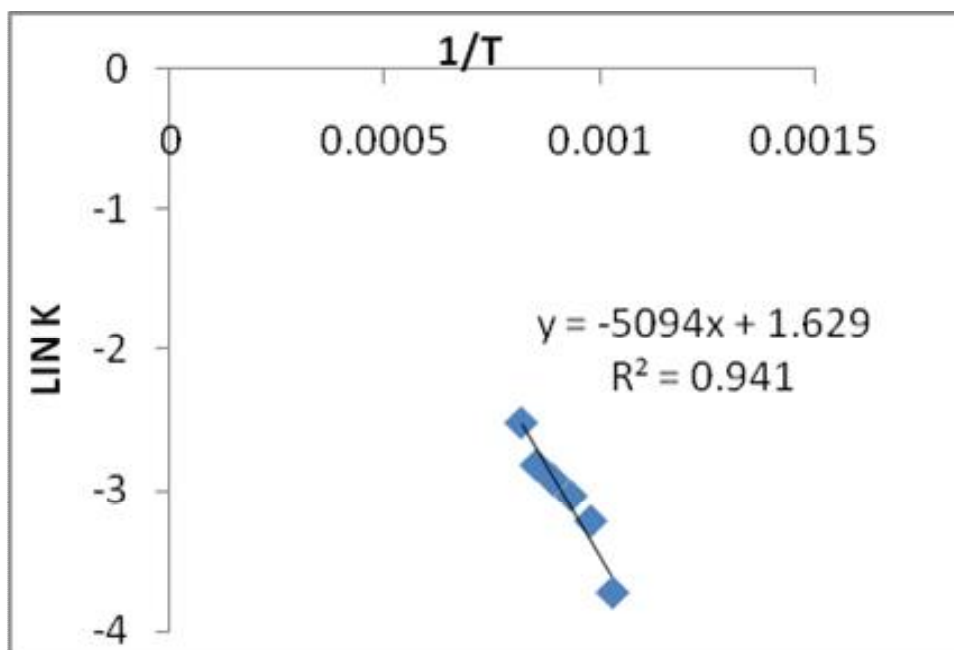


Fig. 22: The Arrhenius plot for the reduction process of briquettes from with E/O ratio of 0.0526.

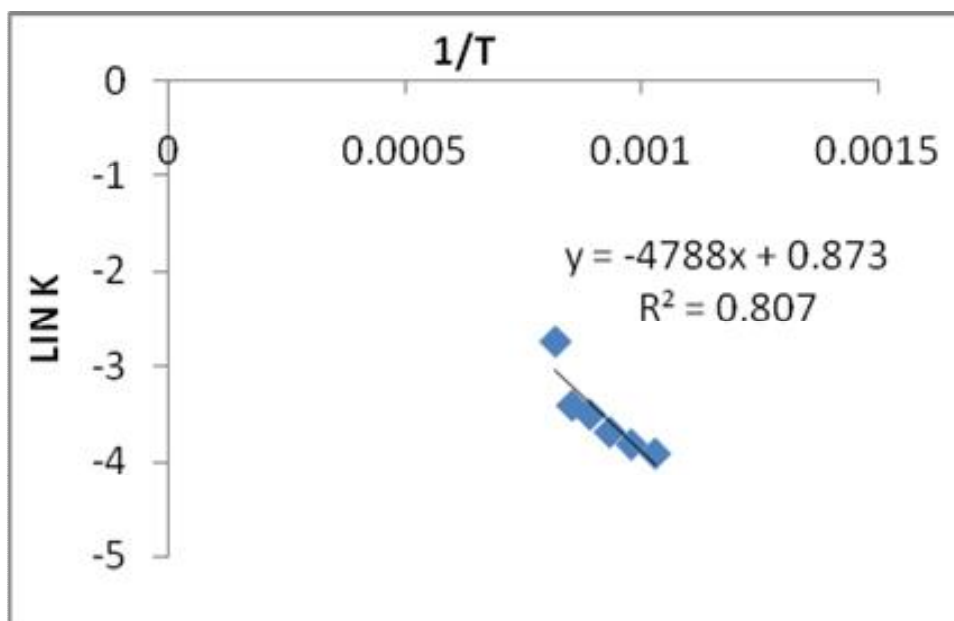
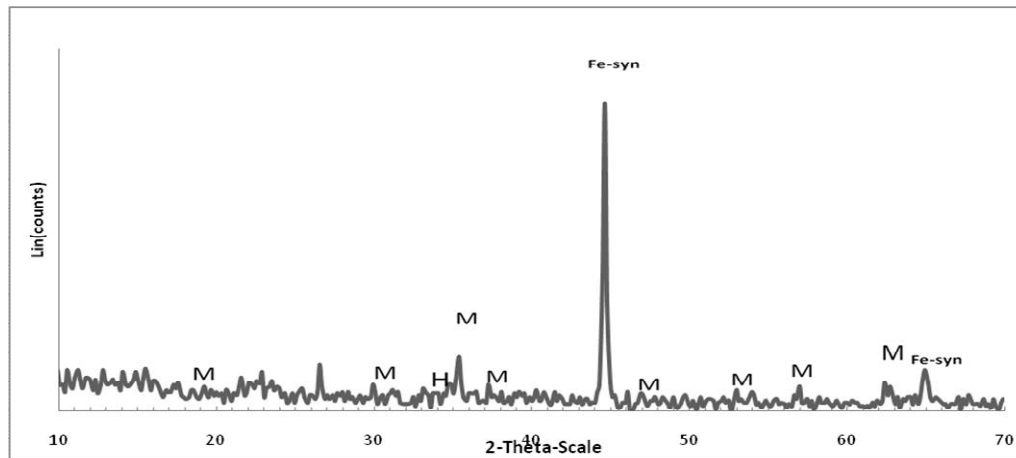


Fig. 23: The Arrhenius plot for the reduction process of briquettes from with E/O ratio of 0.25.

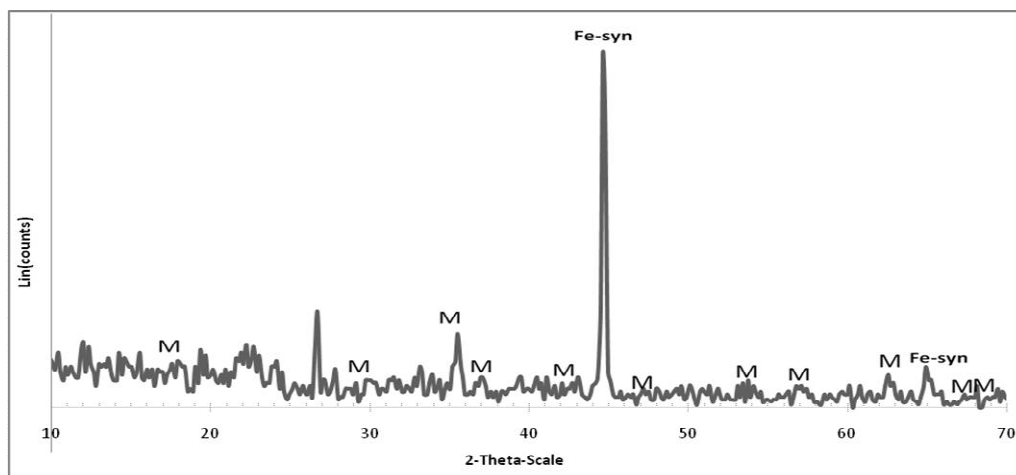
### 3.6. X-Ray Analyses of the Reduced Briquette

The results of the X-ray analyses of the samples reduced at 950°C for briquettes formed with E/O ratio of 0.0526 and 0.25 are shown in figures 24 and 25, respectively. The

results indicate the dominant phase was metallic iron (syn. Fe) with some traces of magnetite [M] ( $\text{Fe}_3\text{O}_4$ ) present.



**Fig. 24: X-ray analysis of the samples reduced at 950°C for briquettes formed with E/O ratio of 0.0526.**



**Fig. 25: X-ray analysis of the samples reduced at 950°C for briquettes formed with E/O ratio of 0.25.**

#### 4. CONCLUSIONS

From the obtained results, the following can be concluded:

- As E/O ratio increased, both the drop number and compression strength increased in the briquettes formed from El-Dekhillah waste pellets fines (E) and El-Baharia iron ore (O).
- Reduction of the briquettes formed increased applying a higher hydrogen flow rate.
- The activation energies calculated for this process for the briquettes formed with E/O ratio of 0.0526 and 0.25 Using equation  $kt = 1-(1-R)^{0.5}$  was = 47.39 kJ/mole and 34.88 kJ/ mole, respectively.
- The activation energies calculated for this process for the briquettes formed with E/O ratio of 0.0526 and 0.25 Using equation  $kt = R+ (1-R) \text{Ln} (1-R)$  was = 42.15 kJ/ mole and 39.62 kJ/ mole, respectively.



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