

Investigating the Effect of Water Gas Shift Reaction and Other Parameters on the Direct Reduction of Iron Ore Pellets

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Abstract - The enormous production cost associated with the use of the conventional route for steel making has necessitated the need to explore more economically viable routes. In this study we examined how the water gas shift reaction, reducing temperature, iron ore pellet type and gas composition (CO-H₂) influences the direct reduction of the iron ore pellets. Different iron ore pellets were reduced in a thermo gravimetric analysis (TGA) equipment using a reducing gas with different composition of CO and H₂ and at different temperatures. As the H₂ content in the gas increased, the reduction process became rapid while a much slower reduction process was observed for increased CO content in the reducing gas. The reducing temperature affected the reduction process such that higher reduction degrees were observed at higher temperatures. Two different pellet types; KPRS and CVRD were used in this study. By comparison, the KPRS pellets gave the highest degree of reduction at all the experimental conditions examined and this observation can be related to the relatively smaller size of the KPRS pellets. In general the water gas shift reaction impacted the reduction process in a positive manner as a result of the additional reducing gasses from the water gas shift reaction contributing to the kinetics of the reduction process.

Keywords-Water Gas Shift Reaction, Iron Ore Pellets, Degree of Reduction, Steel

I. INTRODUCTION

The iron and steel manufacturing industry has undergone a lot of technological revolution in the past decades since the industry is faced with a wide range of environmental concerns that are related to the high energy requirements, material usage and its associated by-products [1]. The conventional route for making steel consists of sintering or pelletization plants, coke ovens, blast furnaces, and basic oxygen furnaces. Such plants require huge capital expenses and raw materials of stringent specifications. Integrated steel plants of less than one million tons annual capacity are generally not economically viable. The coke ovens and sintering plants in an integrated steel plant are highly polluting and expensive units. Installation of highly complex and expensive pollution-control systems make this route highly capital intensive [2-6].

In the last three decades, there has been a rapid increase in the production of iron via direct reduction processes [1]. The largest advantage of the direct reduction iron making relies

on the fact that it does not require coke making and sintering. Coke making and sintering are costly and also results in enormous amount of environmental pollution [7]. Direct-reduced iron (DRI) is produced from direct reduction of iron ore (mainly hematite-Fe₂O₃) which is in the form of lumps, pellets or fines by a reducing gas produced from natural gas or coal. The reducing gas is a mixture of Hydrogen (H₂) and Carbon Monoxide (CO) which acts as reducing agent. This process of directly reducing the iron ore in solid form by reducing gases is called direct reduction. Direct reduction, an alternative route for iron making, has been developed to overcome some of these difficulties. This process is mostly carried out in a shaft furnace with the reduction occurring in stages. That is at temperatures above 570°C: the Hematite is reduced to Magnetite (Fe₃O₄), the magnetite is further reduced to wustite (FeO), the wustite is then reduced to Iron (Fe). Due to the instability of the wustite phase at temperatures below 570°C, the magnetite is reduced directly to Iron at those temperatures. DRI is successfully manufactured in various parts of the world through either gas or coal-based technology. Iron ore is reduced in solid state at 950—1050°C either by reducing gas (H₂+CO) or coal. The specific investment and operating costs of direct reduction plants are low compared to integrated steel plants; however they are relatively energy inefficient [2].

The evolution of direct reduction (DR) technology to its present status has included more than a hundred different DR process concepts, many of which have been operated experimentally. Most were found to be economically or technically unfavorable and abandoned. The two most common DR processes are the MIDREX and HYL processes. In the MIDREX process, the iron oxide feed descends in a cylindrical shaft furnace and is heated and reduced by rising hot reducing gas. The reducing gas used in this case is nearly 95 % H₂ and CO with H₂ to CO ratio of 1.5-1.6. The basic HYL process employs a conventional natural gas-steam reformer for reducing gas generation. In this process, oxygen is removed from the iron ore by chemical reactions based on H₂ and CO from the gas-steam reformer. The reformed gas is typically 72% H₂ and 17% CO [6-10].

Initially, the major reducing gas used in the reduction of the iron pellets is CO, which leads to the production of enormous amount of CO₂. It is well known that the iron and

steel industry is one of the largest producers of CO₂ in the metallurgical processing field, with a 3Mt blast furnace emitting as much as 4Mt of CO₂ per year, this is comparable to CO₂ emitted by a 500 MW coal fired power plant. In 1996, the iron and steel industry was responsible for 4.6 % of the total global CO₂ emission of 23.9Gt. With the increasing worldwide awareness of the effects of CO₂ emission on the global climate, the use of alternative 'cleaner' fuels in the iron and steel industry can be seen as an inevitable step [11]. Hydrogen gas can be used as an alternative, but this leads to a generation of a reduction process, which is thermally disadvantageous though it has a higher reduction degree. A combination of CO-H₂ otherwise known as syngas (synthetic gas) is used. This is obtained from a natural gas CH₄ after it has been reacted with CO₂ in the reform reaction. The combination of CO-H₂ in the reduction process leads to the production of CO₂ and H₂O. These reactant gases (CO-H₂) and product gases (CO₂-H₂O) reacts among themselves in the so called water gas shift reaction (WGSR), which is believed to have a dramatic effect on the reduction process [12].

From the previous paragraphs it can be deduced that, the reduction of the iron oxide pellets by gases is a thoroughly studied phenomenon that has found a wide technological application. However, many poorly understood factors still exist, such as the effect of the water gas shift reaction on the reduction process and whether the reduction process is diffusion controlled, interfacial chemical reaction controlled or mixed controlled. The reduction of the iron oxide is carried out with single (H₂ or CO) or mixed (H₂+CO) gasses. Reduction with single gasses has been studied extensively on laboratory scale while very few academic investigations have been carried out in the field involving mixed gasses.

The main objective of this study is therefore to carry out laboratory investigations into the effect of the water gas shift reaction on the production of DRI using mixed gases and to examine the effect of some process parameters such as mixed-gas composition and temperature on the rate of reduction. The reduction degree or degree of oxidation of the different type of pellets (KPRS and CVRD) was also compared.

II. MATERIALS AND METHODS

A. Iron Ores Investigated

The two ores investigated in this study were KPRS pellets from Luossavaara-Kiirunavaara AB (LKAB) and CVRD pellets from Arcelor-Mittal. The KPRS pellets are quite smaller than the CVRD pellets and this result in a uniform heat distribution in the former than the later. The composition of the KPRS pellet is shown in Table 1.

TABLE 1; Composition (wt. %) of KPRS iron ore pellets (Adopted from [13]).

S	P	Fe	MgO	MnO	CaO	Al ₂ O ₃	SiO ₂
0.002	0.02	67.9	0.58	0.07	0.9	0.16	0.75

B. Reduction of the Iron Ore Pellets in TGA Equipment

The reduction of the iron ore pellets were carried out in a thermogravimetric analysis (TGA) furnace, shown in Fig. 1. With the TGA furnace, we had the capability to change both the gas composition (in-terms of gas flow rate) and temperature during the experiment.

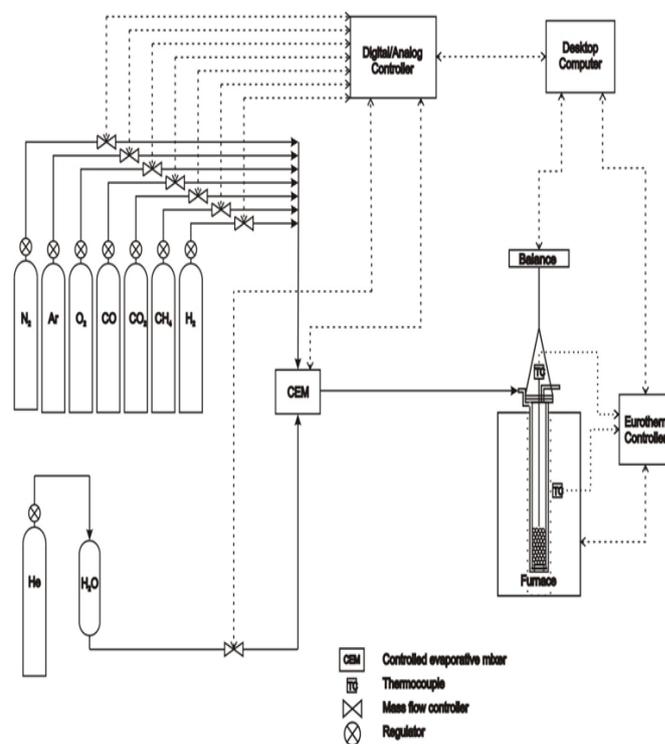


Fig 1: Schematic diagram showing the TGA equipment [14]

The TGA consisted of a retort (crucible, which is double walled) used to hold the sample of pellets. The retort was suspended from an electronic laboratory scales in an electric furnace as seen from Fig 1. Gas mixtures were supplied from a gas mixing unit (shown on the left side of Fig 1) via the double wall of the retort (for preheating the gas) and entered the sample from below. Spent gas exited through the retort lid at the top and was discharged to the atmosphere via ventilation [14]. The gas mixture consisted of Ar, H₂ and CO. Changes in gas mixture and temperature was programmed as a function of time through the LabVIEW computer interface which also recorded the sample weight as function of time [14].

CO/CO₂ analyser was connected to the TGA equipment to help analyse the CO/CO₂ in the exit gas stream. This would help in analysing the water gas shift reaction which is of outmost importance in this project.

In this particular project, different iron ore pellets (KPRS and CVRS) samples weighing about ~ 250g were placed in the crucible. Half of the samples were first placed in the crucible, the thermocouple detecting the temperature of the samples was placed in the middle and the remaining pellets were added. This was done to improve the accuracy of the temperature taken by the thermocouple. The crucible was then placed in the furnace. Care was taken to ensure that, the crucible fits well into the furnace without touching the furnace wall. The various process parameters were programmed into the LabVIEW software so that, the degree of reduction of the samples, set point temperature, crucible and sample temperatures could be monitored. Also the gas flow rates as well as the composition of the exit gas stream were monitored.

C. CO-H₂ Gas Mixture Composition

The sample (KPRS) pellets were heated to a temperature of ~812 °C in the furnace within a time interval of 45 minutes

in Ar. The flow rate of Argon was 1 l/min. It was then held at the said temperature for two hours during which the sample was subjected to different reducing gas composition. Due to the control system in the furnace, the heating procedure experienced some sort of overshoot which differs with the various gas compositions. The degree of reduction was then monitored using the LabVIEW computer software. At this temperature, different gas compositions were used: 100 % CO, 100 %, 20 % CO and 80 % H₂, 40 % CO and 60 % H₂, 60% CO and 40 % H₂ and 80 % CO and 20 % H₂. The various reducing gas compositions used in this study are summarized in Table 2. Before and after the experiment, Ar (1 l/min) was allowed to flow through the furnace for about 15 minutes. This was done to ensure that, no traces of the other gases remained in the gas tubes. The first experiment was carried out at the said temperature (812°C) because of the fact that, at this temperature, the equilibrium constant for the water gas shift reaction (WGSR) is Approximately 1. The same experimental procedure was repeated using CVRD pellets.

D. Temperature Variation

The effect of temperature was investigated by carrying out the reduction process for a fixed composition of the KPRS pellets at different temperatures; that is 812 and 822 °C. The summary of the various compositions and temperatures used for this analysis are also shown in Table 2.

E. Effect of Pellet Type

The TGA procedure described above was repeated with CVRD pellets at some specified reducing gas composition as summarised in Table 2. The results were compared with that of KPRS pellets

TABLE 2: Experimental conditions

Type of pellet	Gas composition in (l/min)		Sample weight (g)	W _{o,start} (g)	Stabilization temperature (°C)	
	CO	H ₂				
KPRS		5	250	75.14	822	
	5		250	75.14	822	
	4	1	250	75.14	822	
	3	2	250	75.14	822	
	2	3	250	75.14	822	
	1	4	250	75.14	822	
	5		250	75.14	812	
		5	250	75.14	812	
	1	4	250	75.14	812	
	2	3	250	75.14	812	
	3	2	250	75.14	812	
	4	1	250	75.14	812	
	CVRD	5		250	75.14	812
			5	250	75.14	812
5		2	250	75.14	812	

III. RESULTS AND DISCUSSION

The weight loss recorded by the TGA equipment was used to calculate the degree of oxidation at the different experimental conditions (temperature, different gas flow rates and different pellets). A plot of degree of oxidation against time was made for each of the different experimental conditions. The various plots were compared based on the different gas ratios, temperature and pellet type.

A. Degree of Oxidation

The degree of oxidation, O_x was calculated from the formula,

$$O_x = \frac{W_{o,start} - \Delta W}{W_{o,start}}$$

(1)

Where;

$W_{o,start}$ is the initial amount of oxygen by weight present in the pellet

ΔW is the weight loss recorded by the TGA equipment
Therefore,

$$\text{the reduction degree} = 1 - \frac{W_{o,start} - \Delta W}{W_{o,start}} \quad (2)$$

B. The Effect of Time

The effect of time on the degree of reduction was investigated with KPRS pellets at 5 l/min H_2 and 5 l/min CO and the plots are shown in Fig. 2. The effect of time on the reduction process was carried out at a temperature of 812 °C which is the temperature at which the water gas shift reaction has an equilibrium constant of 1. But due to poor temperature control system in the TGA equipment, the temperature was about 812 ± 8 °C. From Fig. 2, as the processing time increased, the degree of oxidation decreased, signifying an increase in the reduction degree. As an example, it can be deduced that using H_2 as the reducing gas resulted in a reduction degree of 0.3, 30, 61 and 98 % at 60, 600, 1800 and 7200 seconds respectively. However when CO was used as the reducing gas, 0.5, 19, 41 and 74 % reduction degrees at 60, 600, 1800 and 6650 seconds respectively were observed. The increase in the reduction degree with time is as a result of the increase in the amount of reducing gases in the pellet with time. The time dependency of diffusion is explained by Fick's 2nd law of diffusion, which is stated in the Eq. (3) below [15]:

$$\frac{\partial c}{\partial t} = D \nabla^2 c \quad (3)$$

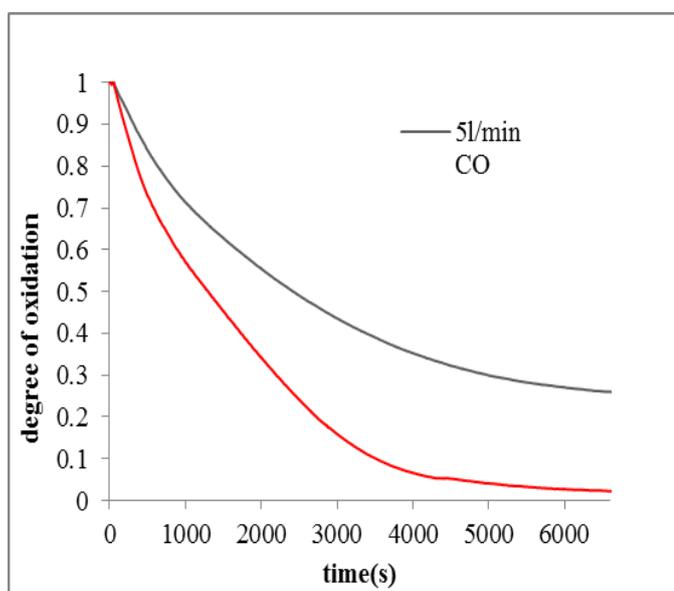


Fig. 2: Degree of Oxidation for KPRS pellets with CO and H_2 at different reaction time

That is as processing time increases, more of the CO and or H_2 diffuses into the pellet resulting in increase in reaction between the gases and the iron ore. The initial reduction degrees increased significantly within a short time due to increase in concentration gradient of the reducing gases on surface and in the inside of the pellets. The reduction degree levelled off at higher reaction times due to decrease in the driving force of the diffusing reducing gases which can be attributed to the reduction in concentration gradient. However, comparing the different plots in Fig. 2, it can be deduced that, though the reduction temperature was the same, the reduction degrees were different at the same reduction time. This can be attributed to the different reducing gases used to obtain the different plots in Fig. 2.

From the plots in Fig. 2, it was observed that the rate of reduction of the KPRS in 5 l/min CO was gradual and did not level off even after 2 hours while that of the KPRS in 5 l/min H_2 was very rapid and levelled off after 2 hours. The reduction degrees were 74 and 98 % for KPRS in 5 l/min CO and KPRS in 5 l/min H_2 respectively at the end of the reaction time. The reduction rate is controlled by the diffusion rate of the reducing gasses through the product layers, the interfacial chemical reaction or both. From Ficks first law of diffusion [16], the diffusion flux is inversely proportional to the molecular weight of the molecules being transported that is the diffusion rate of larger molecular weight gases will be lower than that of lower molecular weight gases. When this concept is related to the gasses used in the reduction process, it can be deduced that, the rate of diffusion of CO through the reactant and product layers will be relatively slower than that of H_2 . As a result, the rate of diffusion of CO through the various layers was gradual, and this is responsible for the gradual degree of reduction observed with CO as the reducing gas. However, the lower molecular weight of H_2 allowed for a rapid diffusion of H_2 through the product layer and hence the trend observed in Fig. 2. Examining the pellets under an optical microscope after the reduction process revealed that the product from the H_2 has a lot of cracks than that of the CO. This further buttress the rapid nature of the diffusion of H_2 through the product layer. The oxygen removal rate from the iron ore pellets were also higher for H_2 than for CO hence the reduction was favored by the use of H_2 compared to CO [17]. Above 812 °C, H_2 has higher reduction potential than CO and this is also responsible for the higher reduction degree obtained by using H_2 . Though reduction with CO gives a higher number of active sites for reaction compared to H_2 [6], reactions involving CO are generally slower and that impacted negatively on the whole reduction process. Fig. 3 revealed much clearer iron ore reduction rings for the iron ore reduced with H_2 than CO. The leveling off of the reduction process with 5 l/min H_2 is due to the formation of a dense product layer at the surface of the pellets which hinders further diffusion of the reducing gas (H_2) through the pellets surface. It can also be attributed to the decrease in concentration difference of the reducing gasses with time which decreased the diffusion rate of the reducing gasses.



Fig. 3: Pictures of CVRD pellets reduced in different reducing gas composition: Top pellet: reduced in 5 l/min CO-2 l/min H₂, Bottom left pellet: reduced in 5 l/min H₂, Bottom right pellet: reduced in 5 l/min CO

C. Effect of Pellet Type

CVRD and KPRS pellets were used in this study to examine how they can influence the reduction process. Increase in the degree of reduction was gradual as observed from Figs. 4 and 5, and for all the reaction time examined, the degree of reduction of the KPRS was higher than that of CVRD. For instance in Fig. 4, after the first 33 minutes, the degree of reduction was 45 % and 33 % for KPRS and CVRD pellets respectively while that of KPRS and CVRD pellets were 66 and 55 % respectively in Fig. 5. That is the KPRS pellets resulted in a higher reduction degree than the CVRD pellets. The difference in the reduction degrees may be attributed to the different ways in which the pellets were synthesized since they were manufactured by two different manufacturing companies. Also, one notable difference between the two pellets is their sizes. The KPRS pellets were smaller than the CVRD pellets and the smaller size resulted in a uniform temperature distribution throughout the KPRS than the CVRD pellets. The smaller size pellets are characterized with high surface to volume ratio and as a result enhanced the degree of contact between the reducing gases and the iron ore pellets which subsequently improved the reduction degree. That is the physical properties of the pellets also influenced their degree of reduction.

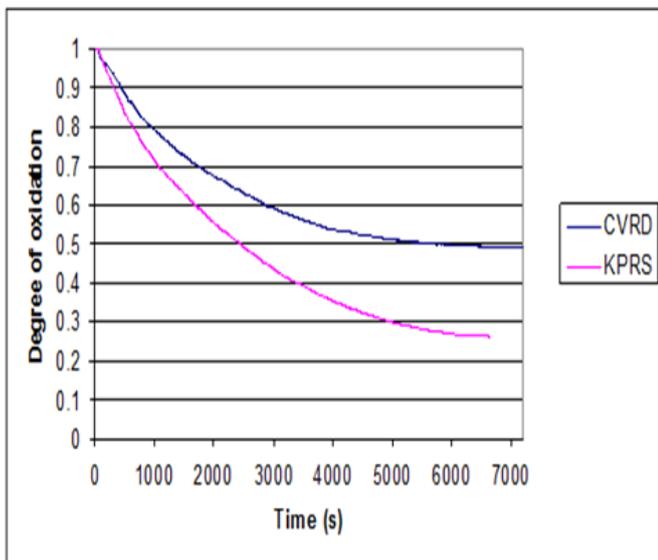


Fig. 4: A plot of degree of Oxidation against time comparing CVRD and KPRS pellets reduced with 5 l/min CO

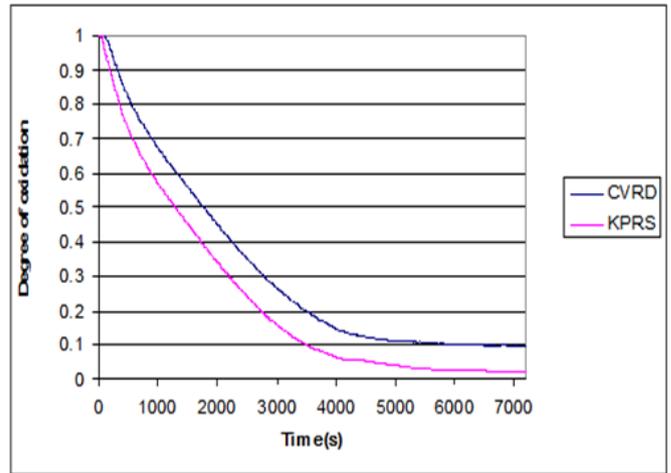


Fig. 5: A plot of degree of Oxidation against time comparing CVRD and KPRS pellets reduced with 5 l/min H₂

D. Effect of Reducing Gas Composition

The highest reduction degree was obtained from the reducing gas with the highest H₂ content as can be seen in Figs. 6 and 7. For instance in Fig. 6, After 33 minutes, reduction degree of 62 %, 56 %, 51 % and 45 % were recorded for CO/H₂ ratios of 0.25, 0.67, 1.5 and 4 respectively. A similar trend was observed in Fig. 7, where reduction degrees of 44, 65 and 57 % were recorded for CO:H₂ ratios of 5:0, 0:5 and 2:3 respectively. Though hydrogen reduction is more thermally disadvantageous than CO reduction, it is generally advantageous in the sense that, the rate of reduction with H₂ is much greater than that with CO. The degree of oxidation at a particular time was increased by adding a small amount of H₂ and from previous studies, the rate enhancement effect of H₂ on the gaseous reduction remarkably appears between 812 °C and 820 °C [12]. The effect originates from the proceedings of the water gas shift reaction as shown by Eq. (4).

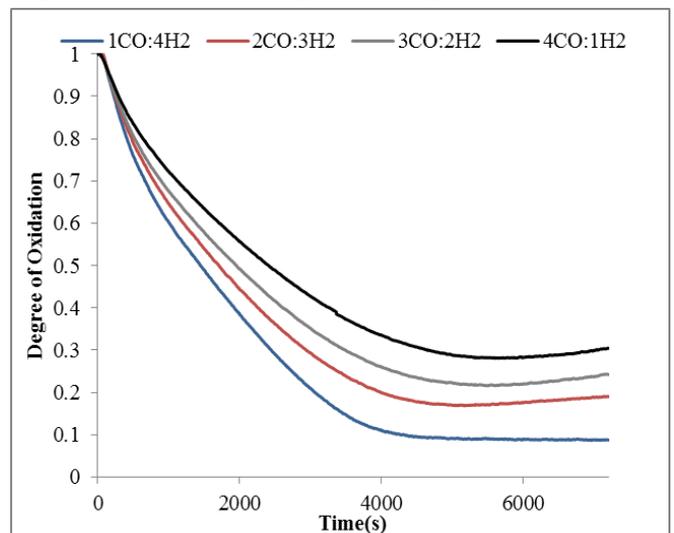
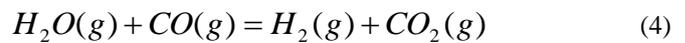


Fig. 6: Degree of oxidation against time for KPRS pellets at different gas compositions.

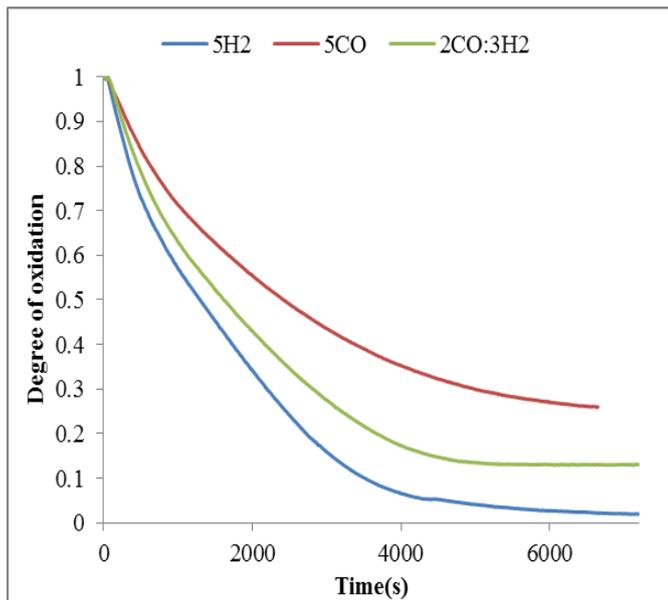
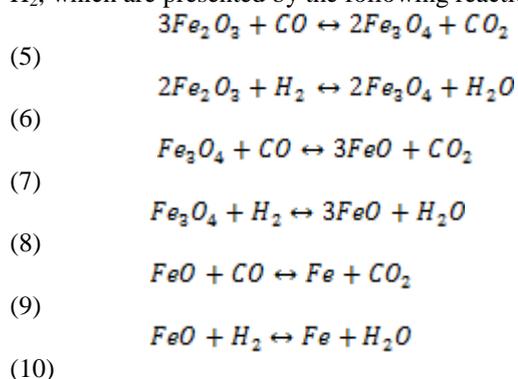


Fig. 7: Degree of oxidation against time for KPRS pellets at different gas compositions.

The product gas H_2O (g) and the reducing gas CO (g) react to produce another reducing gas H_2 (g) and a product gas CO_2 (g). At equilibrium constant equal to 1, more reducing gases were produced which were used in further reducing the pellets. The blend of $CO-H_2$ also enhanced the diffusion process due to concentration build up in the pellet. This is expected to have a positive impact on the kinetics of the reduction process which will be studied into much detail our future work. The temperature at which the reduction was carried out also had a dramatic effect on the reduction process. This affected both the rate of diffusion of the reducing gas through the layers and also, the rate of reaction of the reduction process. Nevertheless, the reduction degree was still higher when only H_2 was used as a reducing gas as is depicted in Fig. 7. It has been shown from present studies that iron ore (Fe_2O_3) undergoes stepwise reduction by CO and H_2 , which are presented by the following reactions:



The above reactions are dependent on temperature. As an example, the Fe_2O_3 and Fe_3O_4 reaction are initiated at 580 and 670 °C respectively [7].

E. Effect of Temperature

The plots in Figs. 8, 9, 10 and 11 depict the effect of temperature on the degree of reduction. It can be deduced that, the highest degree of reduction was obtained at higher temperatures.

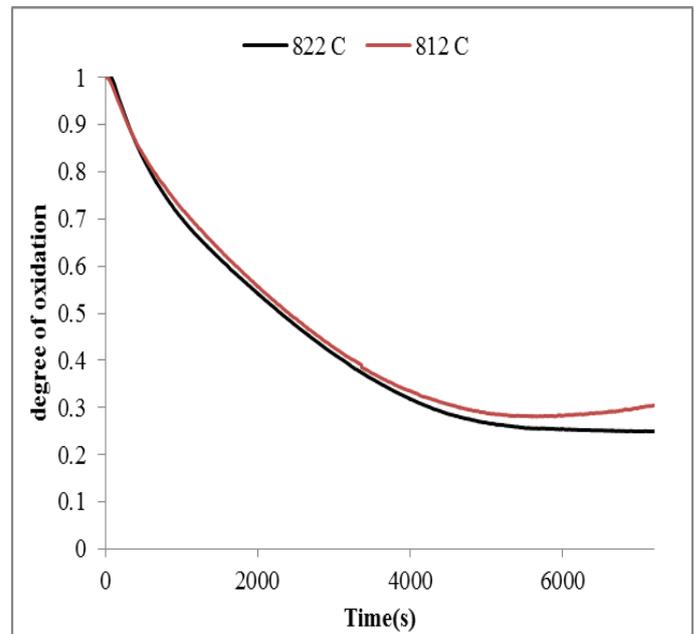


Fig. 8: Degree of oxidation against time for KPRS pellets at different temperatures using reducing gas composition of 4 l/min CO - 1 l/min H_2 .

The reduction degrees recorded at higher temperatures were higher than that of lower temperatures. Consider the effect of temperature on the rate of diffusion (diffusion coefficient) in Eq. (11).

$$(11) \quad D = D_0 \exp\left(-\frac{\Delta E}{RT}\right)$$

Where:

D is the diffusion coefficient, D_0 is the temperature dependent pre-exponential, R is the gas constant, T is the absolute temperature and ΔE is the activation energy for diffusion.

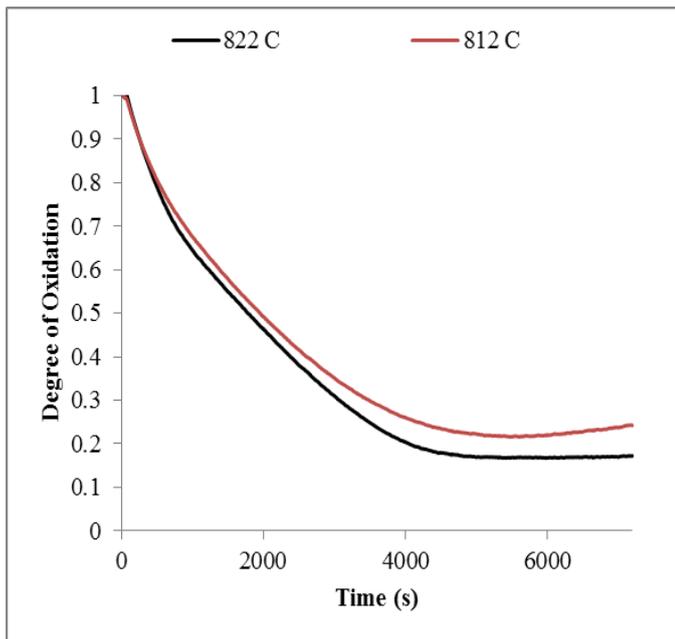


Fig. 9: Degree of oxidation against time for KPRS pellets at different temperatures using reducing gas composition of 3 l/min CO – 2 l/min H₂.

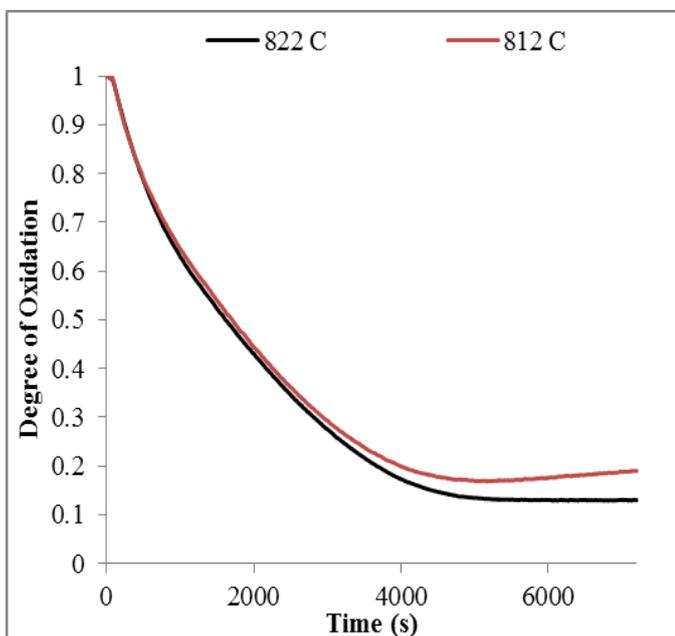


Fig. 10: Degree of oxidation against time for KPRS pellets at different temperatures using reducing gas composition of 2 l/min CO - 3 l/min H₂.

From Eq. (11), higher temperatures result in higher diffusion coefficients and as a result higher diffusion rates were observed at higher temperatures. This higher rate of diffusion impacted the reduction degree positively since at higher diffusion rate, more of the reducing gasses diffused into the pellets and subsequently contributed to higher reduction. From Fig. 8 to 11, it was observed that, after the reduction period of two hours, the maximum reduction degree was higher at high temperatures and lower and low temperatures.

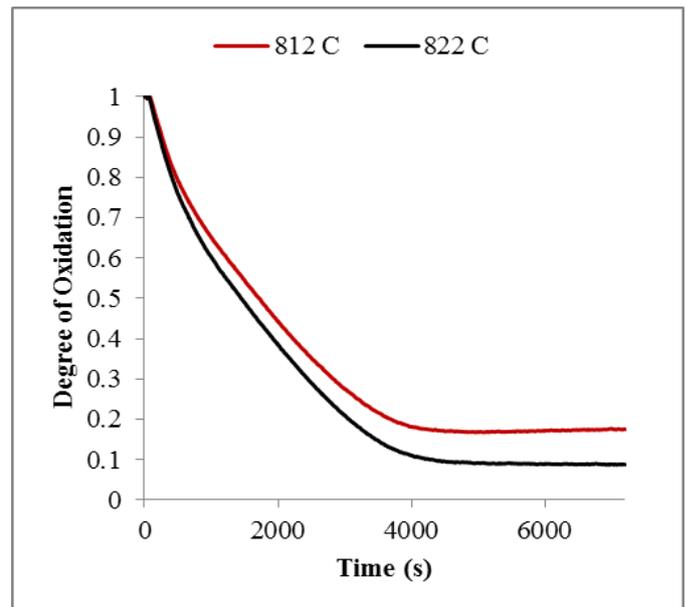


Fig. 11: Degree of oxidation against time for KPRS pellets, at different temperatures using reducing gas composition of 1 l/min CO - 4 l/min H₂.

IV. CONCLUSION

This paper focused on the effect of water gas shift reaction, reducing gas composition, reaction temperature and pellets type on the direct reduction of iron ore pellets. The studies showed that, the water gas shift reaction has a dramatic effect on the reduction process since it contributed positively to the kinetics of the reduction process by producing additional reducing gasses. The reduction degree obtained from KPRS pellets under the experimental conditions reported in this article was higher than CVRD pellets. It was observed that, the rate of reduction increases with increasing H₂ content in the reducing gas while increasing CO content decreased the reduction degree. Higher reaction temperatures resulted in higher reduction degree for each gas composition studied.

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