

Reduction Kinetics of Fluxed Iron Ore Pellets Made of Coarse Iron Ore Particles

Amit Kumar Singh,* Biswajit Mishra, and Om Prakash Sinha

The present work demonstrates a sustainable approach of using relatively coarser iron ore particles for ironmaking. The motivation is to reduce the energy consumption in the milling of the iron ore by utilizing coarser iron ore particles (+0.05 mm) and to select a suitable binder for improving pellet properties. Iron ore fines in the range of 0.05–0.25 mm was selected and classified into three size ranges. Fluxed iron ore pellets were prepared using lime as a binder for the basicity of 0, 1, and 2. Reduction of these pellets with a packed bed of coal fines was performed in the temperature range of 900–1200 °C for a duration of 30–120 min. The direct reduction kinetics of the iron ore pellets were studied by employing diffusion and chemical reaction control models to the experimental data. The results show that pellets made with coarser iron ore particles have improved reduction behavior and kinetics. The reduction reaction is found to be a mixed control. The activation energy for the reduction reaction varies from 44.3 to 74.76 kJ mol⁻¹ as iron ore particle size decreases from 0.25 to 0.05 mm and basicity increases from 0 to 2.

amounts of waste fines. To utilize low-quality raw materials like iron ore fines and non-coking coal several alternative processes were developed for steelmaking. These processes were mainly of two types 1) direct reduction followed by electric arc furnace melting and 2) smelting reduction. These processes do not have stringent raw material quality requirements. These processes are also flexible in terms of the shape and size of the charge material. Waste iron ore fines can be agglomerated by pelletizing to form spherical pellets.

Iron ore pellets with or without additives are utilized as an alternate charge material in both conventional and alternative ironmaking processes. Agglomerates in the form of hardened/sintered pellets can partially replace the iron ore lumps in blast furnaces. These pellets are also suitable for use in direct reduction processes such as

Midrex, SL/RN, HyL, and rotary hearth furnaces to produce direct reduced iron (DRI). The performance of these pellets under actual reduction conditions is generally a function of their resistance to sticking, degree of reduction, and strength after reduction.^[2] The above properties are majorly influenced by the type and quantity of additives and the induration/hardening characteristics of the pellets. The thermodynamics and reaction mechanisms of the reduction processes vary with the reaction conditions, reductants, additives, etc.^[3] The surface morphology of the reduced pellets and the reduction reaction mechanism are majorly influenced by the reduction and sintering properties of iron ore pellets. To analyze these reduction and sintering phenomena two broad categories of models are available in literature, i.e., empirical models and structural or morphological models. Empirical models are based on the kinetics of the reaction, and structural or morphological models are based on morphological changes during the process. Empirical models and kinetic equations can be used to model different experimental processes and they are important for theoretical understanding of the process. It can be utilized in explicating the mechanism of the reduction reaction of iron ore pellets.^[3]


The reaction kinetics of the fluxed iron ore pellets are not studied extensively. There are some studies reported in the literature, however, much of it is not in agreement. It is generally accepted that neither there is a fixed mechanism nor a particular value of activation energy for these reduction reactions. Depending on the experimental condition and physical nature of the system, the rate of the reaction is controlled by one or more than one possible mechanism.^[4] In the case where solid carbon is used

1. Introduction

Blast furnace-basic oxygen furnace (BF-BOF) is currently the dominant route of steel production. High productivity and uninterrupted operation of the blast furnace depends on the quality of raw materials provided. The highest quality of iron ore and metallurgical grade coal (converted to coke) is a stringent requirement for blast furnace ironmaking. With the depletion of high-quality reserves of iron ore and coking coal, it is becoming increasingly difficult to utilize the full potential of blast furnaces. Blast furnace uses iron ore lumps of size $-45 + 15$ mm as charge material.^[1] Mining and crushing of iron ore generate huge

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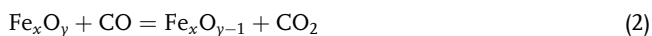
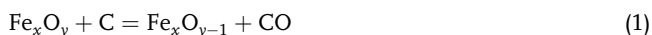
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to reduce iron ore, e.g., in composite pellets, direct reduction initiates the reduction process. The reaction between carbon and iron oxide begins at points of contact as shown in Equation (1). Iron oxide is reduced to Fe and as soon as the Fe is formed, contact is disrupted. Now, the reduction can proceed only by diffusion of carbon atoms through the newly formed iron layer. In the process, carbon monoxide is generated which indirectly reduces the iron ore (Equation (2)). The carbon-di-oxide formed as a product of the reduction reaction combines with the available carbon to form carbon monoxide again (Equation (3)). This cycle continues till either the reductant or the iron ore is exhausted.



The rate of dissolution of carbon to form CO (Equation (3)) is much slower than the iron ore reduction step at higher temperatures and is believed to be the rate-controlling step. This view was supported by several researchers.^[2–5]

In the cases (such as the present study) where reducing gas (carbon monoxide) is provided from outside, e.g., reduction of iron ore pellets without carbon embedded in it, reduction proceeds indirectly by the formation of carbon monoxide as per Equation (2). There have been several attempts at characterizing the reaction mechanism and rate-controlling steps by mathematical expressions. It is established that temperature, carbon-to-oxygen ratio, carbon particle size, addition or presence of catalysts, binders, etc. all affect the reaction kinetics.

There have been several attempts at characterizing the reduction reaction mechanism and rate-controlling steps by mathematical expressions for fluxed iron ore pellets.^[3,5–13] Prakash et al.^[8] reported that the reduction kinetics in the case of fluxed iron ore pellets follows first-order reaction kinetics with significant dependence on reduction temperature. The addition of lime as a flux was reported to be important for the green strength of the iron ore pellets, however, beyond a certain value it does not have much influence on the reduction behavior and formed iron morphology. The authors fitted the experimental data to a diffusion-controlled model and reported activation energies in the range of 49–50 kJ mol⁻¹ and 47–52 kJ mol⁻¹ for 0.83 and 1.33 basicity, respectively. The authors further reported that chemical reaction is the rate-limiting step for fluxed iron ore pellets and diffusion of reducing gas is rate-controlling in the case of fluxed iron ore pellets. Kumar et al.^[9] studied the reduction kinetics of hematite iron ore with different particle sizes and non-coking coal as a reductant in a temperature range of 850–1000 °C. Authors reported that the reduction reaction switches from carbon gasification control to mixed control (carbon gasification as well as iron oxide reduction) as the basicity and coarseness of iron ore particles increase. It was also reported that activation energy decreased, and reduction kinetics improved with the addition of coarser particles. Singh et al.^[10] have studied reduction behavior and kinetics using hematite fines and charcoal as a reductant in the temperature range of 850–1000 °C. Authors have reported that the overall reduction reaction was controlled by

diffusion of the reducing gas followed by gasification of the charcoal. The activation energy values were reported to be in the range of 170–213 kJ mol⁻¹. Chowdhury et al.^[11] have studied the reduction kinetics of iron ore and graphite composite pellets in a packed bed reactor. It was reported that the reduction kinetics is controlled by the diffusion of CO gas through the pellets not the heat transfer. Sah and Dutta^[12] have studied the reduction kinetics of iron ore and coal composite pellets. It was reported that the reduction reaction was controlled by reductant gas diffusion initially and was controlled combinedly by gas diffusion and chemical reaction at the later stage. Mishra et al.^[14] have studied the reduction behavior and kinetics of low-grade multi-metallic magnetite ore. In the study, colemanite was used as a binder, and a non-coking coal bed was used as a reductant. It was reported that the chemical reaction $((1 - \alpha)^{-1/3} - 1)^2 = kt$ controls the overall kinetics with an activation energy value of 165 kJ mol⁻¹. In these low-grade multimetallic ores, a large amount of hard and non-reducible phases are present which makes the chemical reaction difficult. That could be possibly the reason for the chemical reaction acting as a rate-limiting step. Dishwar and Sinha^[13] studied the reduction kinetics of highly fluxed hematite pellets (i.e., 2, 4, 6, and 8 basicity) pellets in the packed coal bed. The authors reported that an increase in reduction temperature and basicity contributed positively toward the degree of reduction. The gas diffusion through the product layer was reported to control the reduction reaction and the activation energy values were reported as 32, 31, 26, and 21 kJ mol⁻¹ for 2, 4, 6, and 8 basicity pellets, respectively.

Iron ore particles coarser than 72 mesh (0.05 mm) are not used for pelletization industrially as it could not be agglomerated. However, utilizing coarser particles directly in iron making will save energy and time by avoiding the grinding stage. Studies carried out so far (except for Kumar et al.^[9]) have used iron ore particle sizes lower than 72 mesh. The present work explores a novel approach to utilize coarse iron ore particles with lime as a feed material for iron making. Lime will act as a binder and a flux for iron ore particles. DRI produced from these fluxed iron ore pellets decreases the demand and the loss of lime during steel-making step. Fluxed DRI pellets also have improved weather resistance. The direct reduction of waste iron ore fines in the range of 0.05–0.25 mm with a lime binder for application in iron-making was studied previously by our team.^[15] The present work aims to investigate the reduction kinetics, identify reaction barriers, and evaluate the activation energies of pellets made from waste iron ore fines in the particle size range of 0.05–0.25 mm with basicity in the range of 0–2.

2. Experimental Section

Raw material (i.e., iron ore, coal, and lime powder) procurement, processing, and composition are the same as reported in our previous work.^[15] Iron ore contains Fe₂O₃- 92.5%, Al₂O₃- 3.7%, SiO₂- 2.94%, and others- 0.86%. Coal contains 62.5% of fixed carbon. Lime contains 95% CaO. Iron ore, coal lumps, and lime powder were stored in desiccators. Iron ore lumps were crushed to generate fines in the range of -0.25 + 0.05 mm. These fines were further classified into three different size ranges, i.e., SR1(-0.25 + 0.1), SR2(-0.1 + 0.05), and SR3(-0.05) mm.

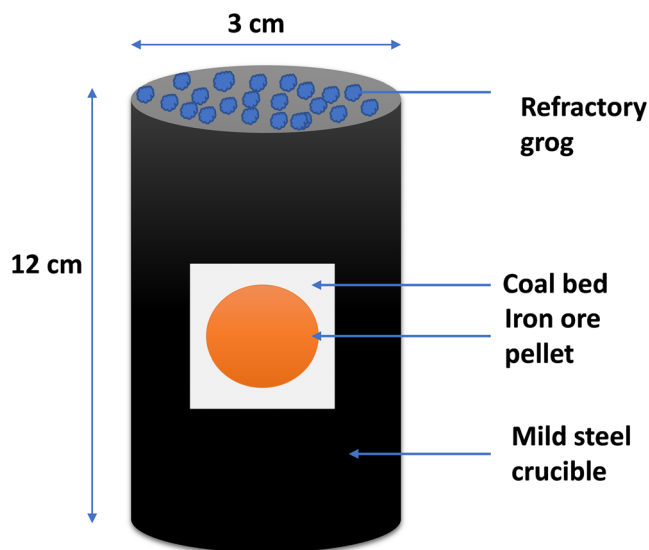


Figure 1. Mild steel crucible used for reduction of fluxed iron ore pellets.

Spherical fluxed iron ore pellets were prepared using lime as a binder with a target basicity of 0, 1, and 2. The ratio of CaO (from lime) to SiO₂ (from hematite fines) was used to calculate pellet basicity. There are no other significant acidic and basic constituents present in the hematite ore. We tried to keep the weight of

iron ore constant in each case. We tried to avoid weight change due to lime addition by selecting green pellets weighing 3% higher for 1 basicity and 6% higher for 2 basicity than 0 basicity pellets. Green pellets were hardened at 1200 °C for 1 h in a muffle furnace in an ambient atmosphere. Hardened flux pellets used in the study have a weight of 7.5 ± 1 g and a diameter of 16 ± 2.4 mm. Reduction of iron ore pellets with coal fines was performed at the temperatures of 900, 1000, 1100, and 1200 °C for the durations of 30, 45, 60, 90, and 120 min. A coal bed (outside the pellet) was used as a reductant, as shown in **Figure 1**. The crucible assembly was put inside the muffle-type resistance heating furnace at a preset reduction temperature for a predetermined reduction duration. After the fixed duration, the crucible was pulled out of the muffle furnace and placed in a vacuum oven to avoid contact with air. After cooling, the reduced pellet was cleaned and weighed to access extent of oxygen removal.

3. Results and Discussion

The degree of reduction was calculated as a ratio of weight loss during the reduction reaction to the total weight loss possible after the complete removal of oxygen from the ore. The ratio is termed as a fraction reduced (*f*). To express it in percentage terms the ratio value was multiplied by 100. All the experiments were carried out thrice and the variation among the values was found to be within 5%. The average values for the degree of

Table 1. The table presents the degree of reduction fractions for the various combinations of basicity, particle size, reduction temperature, and time. Here in the abbreviation of pellet types, the first three letters represent the size range i.e., SR1, SR2, SR3, and later two letters represent the basicity of the pellet i.e., basicity 0, 1, and 2. The table shows reduction trends with variations of the parameters.

Reduction Temperature [°C]	Reduction time [min]	SR1B0	SR1B1	SR1B2	SR2B0	SR2B1	SR2B2	SR3B0	SR3B1	SR3B2
900	30	34.8	33	32.2	33	31.5	30.3	31.1	30.2	28.8
	45	44.2	41.9	39.9	42	37.9	37.2	39.7	35.9	34.2
	60	52	51.4	51.1	47.3	44.3	43	42.4	39.7	37.2
	90	70.3	57.6	55.4	58.6	54.2	54	53.6	52.9	46.2
	120	94.5	68.1	63.9	78.2	64.3	57.7	57.3	56.2	55.9
1000	30	96	93	89.4	92.8	90.3	84.9	85.1	79.5	77.8
	45	98	93.1	90.2	95.5	92.1	88.3	90.1	88.3	78.3
	60	98.9	94.4	90.4	97.3	92.2	89.3	90.3	89.9	84.2
	90	99.2	96.4	90.9	98.7	95	89.9	94.1	93.1	88.5
	120	99.9	96.5	94.8	99.3	95	90	95.7	93.5	89.1
1100	30	98.6	93.9	90.1	93.5	90.7	87	87	85.1	78
	45	98.9	94.2	90.4	96.1	92.2	89.1	91.2	90	84.2
	60	99	95.2	91.2	98.5	93.1	89.5	91.5	90.9	85
	90	99.3	96.8	93.6	98.9	95.4	90.2	98.7	95.1	89
	120	99.9	96.9	94.9	99.3	96.1	90.7	99.2	95.7	90.1
1200	30	99.1	94.2	90.6	94	91.2	89.4	89.2	85.8	82.5
	45	99.5	95.2	91.3	98.3	93	89.6	91.9	90.8	88.6
	60	99.7	95.3	94.9	99.1	93.3	90.5	91.9	91.1	89
	90	99.7	96.9	95.3	99.3	95.8	90.5	99	95.7	89.9
	120	99.9	97.9	96	99.5	96.3	91.6	99.4	96	91.1

reduction (or %Reduction) are presented in **Table 1** to compare the results and analyze the trends with the variation of different parameters like reduction time, reduction temperature, basicity, and particle size. The average values of “f (fraction reduced)” were used to fit the pre-existing models.

It is apparent from Table 1 that the degree of reduction increases with an increase in temperature and time from 30% to 99.9%. The same trend has already been reported by several researchers.^[8,16–18] As a general trend, the degree of reduction should increase with the increase in fineness of the iron ore particles as finer particles provide more surface area. Yi man et al.^[19] have reported the same as they found the highest degree of reduction with the finest iron ore particles in the case of iron ore-coal composite pellets. However, in the present study, the degree of reduction was found to increase with an increase in

the coarseness of iron ore particles. It seems counter-intuitive, as pellets made of finer particles will provide more surface area and hence faster kinetics and a greater degree of reduction. Here, it is worth mentioning that the degree of reduction will be combinedly influenced by the available oxide surface area and access to that surface by the reducing gas. For simplicity, we will combine the two and term it as exposed surface area. So, if the exposed surface area is higher degree of reduction will be higher. Yi man et al.^[19] used iron ore and coal composite pellets and got the highest reduction extent with the finest particles as the coal volatilizes leaving a more exposed surface area. In the present study, we have used iron ore only with small quantities of lime. An increase in the degree of reduction with an increase in the coarseness of the iron ore particles was also reported by Kumar et al.^[9] Due to a decrease in exposed surface area in

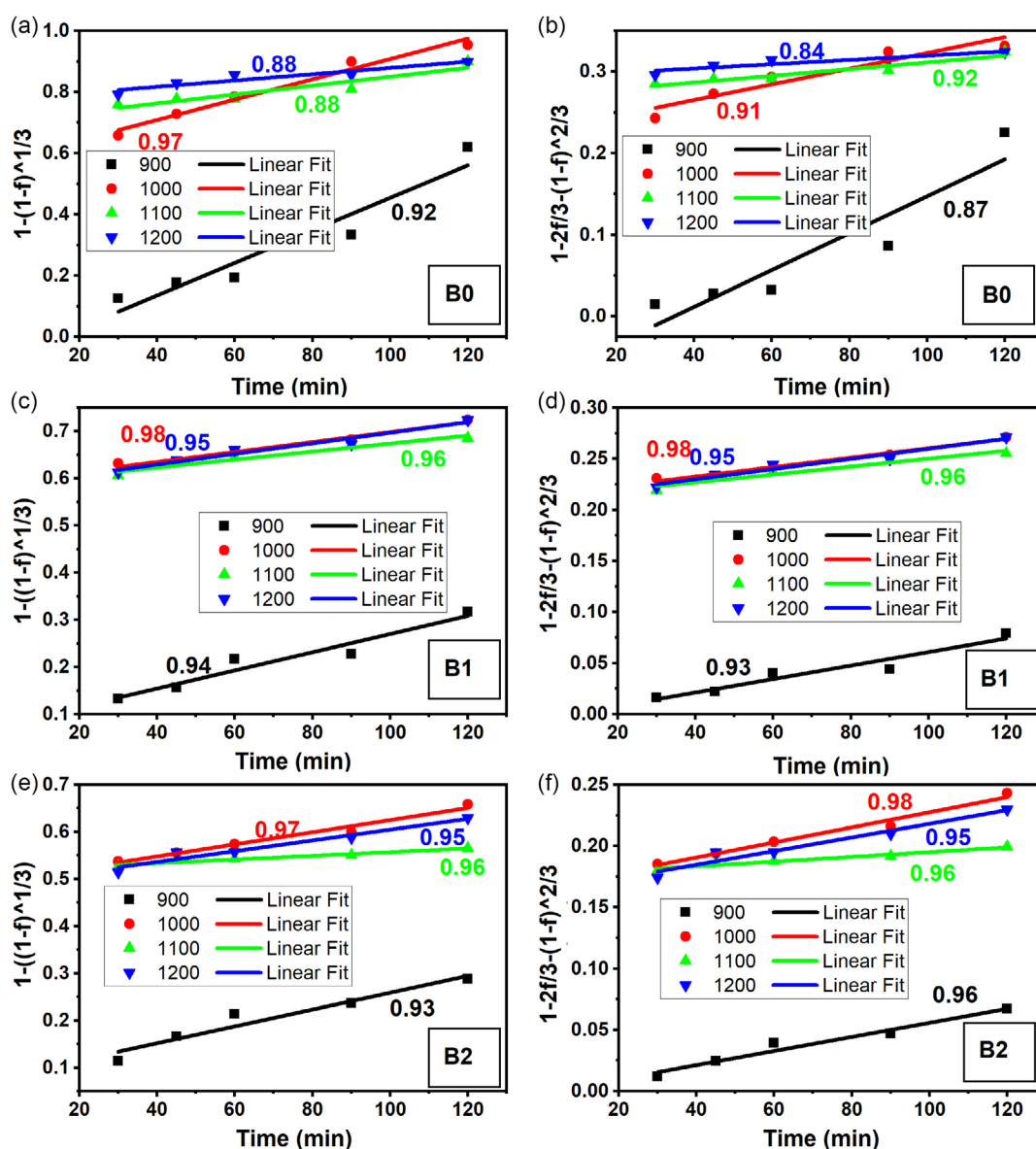


Figure 2. Fitting of the selected models to the experimental data for fluxed iron ore pellets with SR1 for a,b) 0 basicity, c,d) 1 basicity, and e,f) 2 basicity at different reduction temperatures.

the case of finer iron ore particles, the degree of reduction decreased. Exposed surface area can be characterized by porosity measurement of the hardened pellets. Porosity in the hardened pellets decreased from 43 to 31% with a decrease in particle size which resulted in a decrease in the degree of reduction from 99% to 89%.^[15]

It is apparent from Table 1 that as the basicity of the pellet increases, the degree of reduction decreases for every reduction temperature. This trend does not agree with the results already reported in the literature.^[18,20–22] Some other researchers also reported a drop in the degree of reduction after reaching a peak.^[23,24] Combining the effects of particle size and basicity, it can be noted from Table 1 that the degree of reduction decreases as basicity increases and particle size decreases. This effect can be attributed to the higher density (lower porosity)

of pellets made of finer particles and the formation of low melting slag in the case of basic pellets.^[15,24] Higher density decreases the space between iron oxide particles and restricts access to reducing gases. The low melting slag fills the space between iron oxide particles during reduction and hinders the flow of reducing gases which hampers the reduction process.

To study reduction kinetics various models have been developed which analyze the experimental data for gas-solid system. However, in the present study, only mathematical models are taken into consideration. Objective is to find out the rate controlling step. There are two popular first-order models for gas-solid systems, i.e., the progressive conversion model (PCM) and the shrinking core model (SCM). The PCM assumes that the reaction always occurs throughout the particle with different rates at different places. The SCM assumes that the reaction starts from

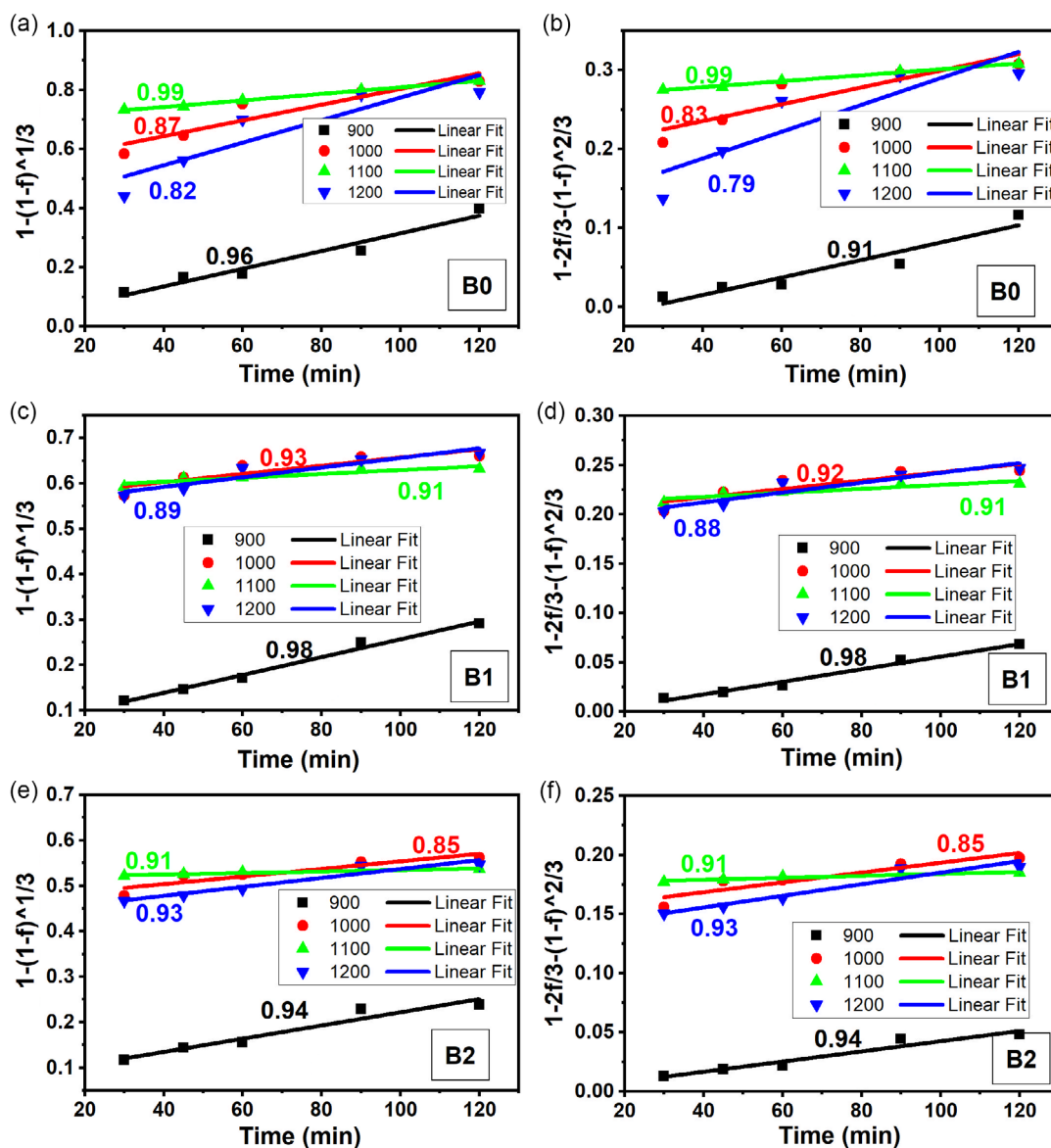


Figure 3. Fitting of the selected models to the experimental data for fluxed iron ore pellets with SR2 for a,b) 0 basicity, c,d) 1 basicity, and e,f) 2 basicity at different reduction temperatures.

the particle's outer surface and progresses toward the core, leaving the solid product layer behind. It was observed over time that SCM fits better to the experimental data. Various steps involved in the reduction of fluxed iron ore pellets are 1) generation of reducing gas CO, 2) chemical reaction of the CO at the gas and unreacted iron ore interface, and 3) diffusion of the CO through the reactant and product layer (Fe) to the unreacted iron ore. These resistances can solely control the overall gas–solid reaction, or they can act consecutively.^[4] Various equations were developed considering these resistances for gas–solid reactions. In the present work, the most popular model equations corresponding to each step are considered.

Equation (4)–(6) are model equations used to fit experimental data for the reduction of iron ore.^[25,26] Fraction reacted (f) and time (t) are used as two input parameters in these equations. Equation (4) assumes the carbon gasification step as the

rate-controlling step. Equation (5) assumes the chemical reaction step as rate controlling step. It further assumes that the reduction reaction takes place on the outer surface of iron ore particles and the porous product layer separates the unreacted core from the reducing gases. The rate-controlling step assumed in Equation (6) is the diffusion of reducing gases through the product layer.

$$-\ln(1 - f) = kt \tag{4}$$

$$1 - (1 - f)^{1/3} = kt \tag{5}$$

$$1 - \frac{2}{3}f - (1 - f)^{2/3} = kt \tag{6}$$

where,

f = fraction reacted at time t ; k = rate constant (s^{-1})

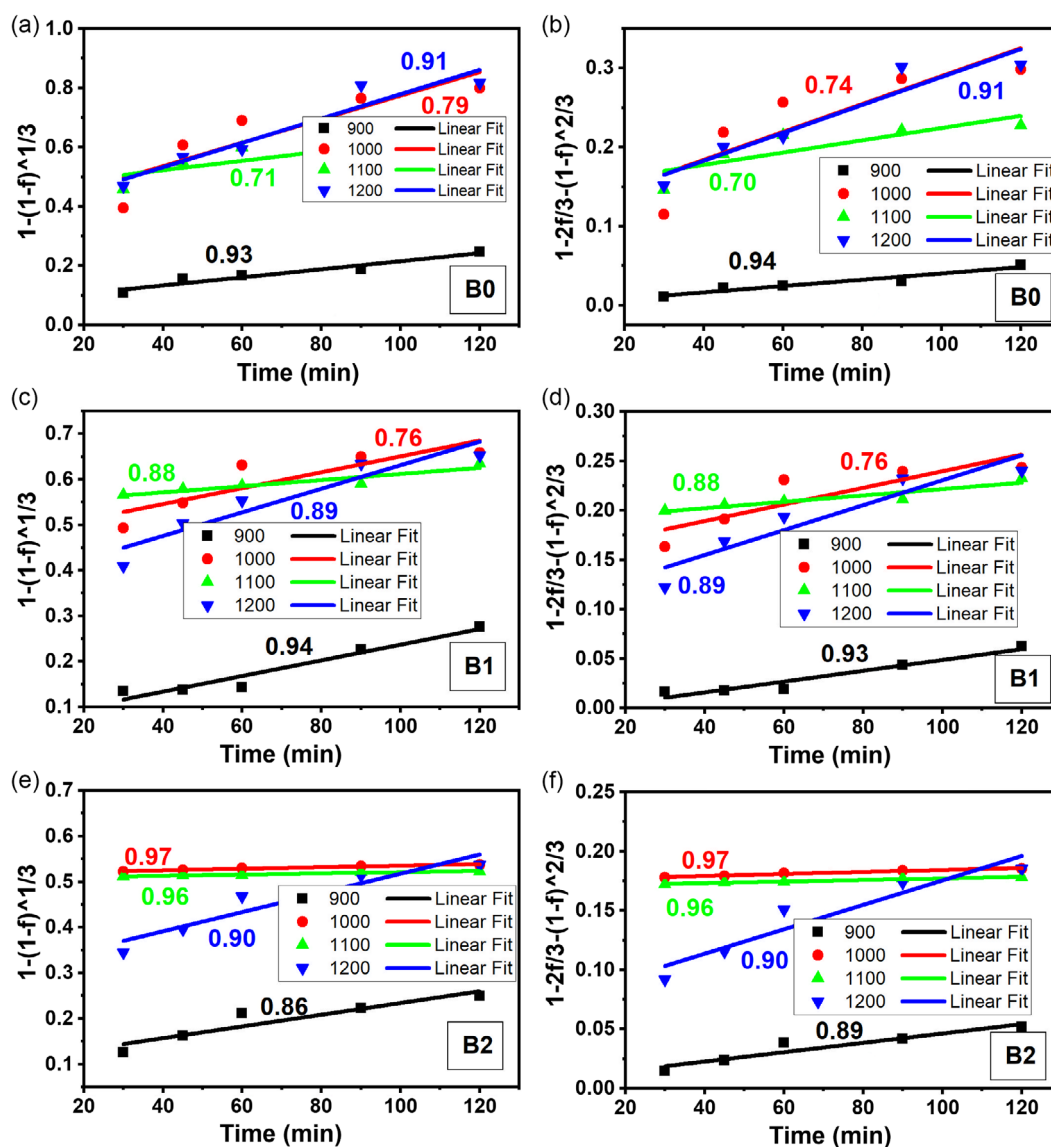


Figure 4. Fitting of the selected models to the experimental data for fluxed iron ore pellets with SR3 for a,b) 0 basicity, c,d) 1 basicity, and e,f) 2 basicity at different reduction temperatures.

CO generation via the carbon gasification step is the rate controlling in the case of iron ore-carbon composite pellets where carbon particles in the form of coal, graphite, or charcoal are present inside the pellet. In the present study, R^2 values for the fit corresponding to Equation (4) were in the range of 0.86–0.92. The poor fitting suggests that in the present case where a packed coal bed was used as a reductant, CO generation is not a rate-limiting step. Chemical reaction and diffusion control models were considered further to identify the rate-controlling step in the reduction reaction.

The fitting of the selected models to the experimental data for different types of fluxed iron ore pellets at different reduction temperatures is shown in Figure 2–4. Plots of the fit of experimental data corresponding to SR1, SR2, and SR3 are shown in Figure 2–4 respectively. In Figure 2, chemical reaction and diffusion control plots corresponding to 0 basicity, 1 basicity, and 2 basicity pellets are shown in a,b; c,d and e,f respectively. 0 basicity, 1 basicity, and 2 basicity plots are similarly arranged in Figure 3 and 4. Goodness of fit in terms of coefficient of determination (R^2) is presented in plots corresponding to different lines.

Both chemical reaction control (Equation (5)) and diffusion control (Equation (6)) models fit well to the data. The chemical reaction control model however fits slightly better to the experimental data. Close R^2 values for both models indicate that the overall reaction is mixed control i.e., controlled by both chemical reaction and diffusion steps. The same mixed type of control was

suggested in multiple studies reviewed by Prakash and Kumar et al.^[3,8,9]

Fitting of the plots with the diffusion control model improves while moving from 0 basicity to 2 basicity in each particle size. This improvement is noticeable concerning the relative fitting of the plots. It implies that diffusion of the reducing gases is gaining more control of the reaction kinetics. As basicity increases, low melting slag phases fill the available pores which leads to a decrease in porosity. Reduced porosity could be causing increased hindrance to reducing gas movement. The same could be the reason for the improved control of the gaseous diffusion step over the chemical reaction step in the overall kinetics of the reaction. Since, the diffusion and chemical reactions are rate limiting steps, increasing the temperature, and providing excess reactants (such as CO) can improve the reduction reaction. The rate of the reduction reaction follows the Arrhenius equation, which is used to calculate the activation energy for the reduction reaction. The slopes (termed as k) were used to plot $\ln(k)$ versus $10^4/T$ plot shown in Figure 5. The slope (m) of the plot in Figure 5 is equal to $(-E_a/R)$, where E_a is activation energy and R is gas constant. Calculated activation energies are listed in Table 2.

Activation energy increases from 44 to 75 kJ mol⁻¹ with different combinations of particle size and basicity. Calculated activation energies align well with the previously reported values by Dishwar & Sinha et al.^[8,13] With decreasing particle size, activation energy increases from 44 to 52 kJ mol⁻¹, 50 to

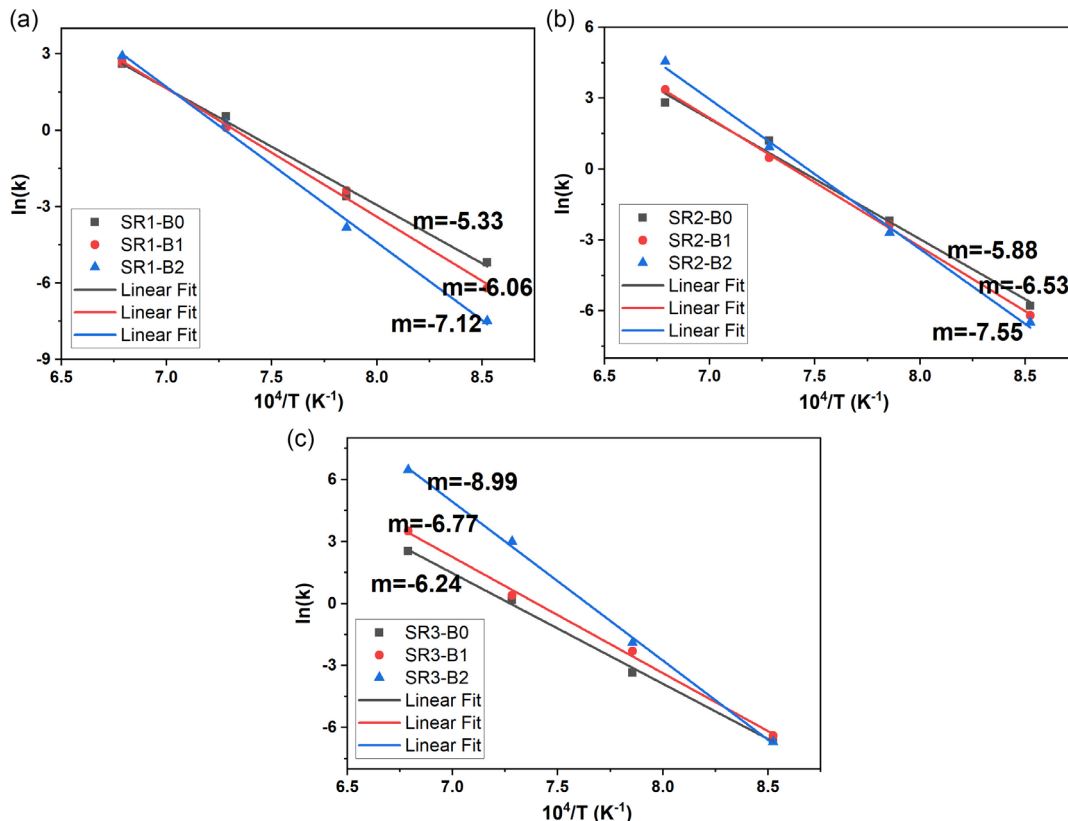


Figure 5. $\ln(k)$ versus $10^4/T$ plot for a) SR1, b) SR2 and c) SR3 pellets.

Table 2. Activation energy for different sizes and basicity.

Particle Size	SR1			SR2			SR3		
	0	1	2	0	1	2	0	1	2
Basicity									
Activation energy [kJ mol ⁻¹]	44.35	50.41	59.19	48.89	54.29	62.76	51.88	56.33	74.76

56 kJ mol⁻¹, and 59 to 75 kJ mol⁻¹ for 0, 1, and 2 basicity pellets, respectively. The activation energy for SR1 with 0 basicity is the lowest (44.35 kJ mol⁻¹) among all the size and basicity combinations. Activation energy values suggest that the reduction rate is increasing with an increase in the particle size and a decrease in the basicity. Kumar et al.^[9] also reported a decrease in activation energy and improvement in reduction kinetics with increasing extent of coarser particles.

4. Conclusion

An approach of using relatively coarser iron ore particles with a suitable binder was evaluated for ironmaking in the present work. Using coarser particles will decrease the energy needed in comminution, and selecting lime as a binder will decrease slag volume during steelmaking. Acidic and fluxed iron ore pellets made of different particle sizes were directly reduced at 900–1200 °C for 30–120 min. Reduction kinetics was studied using two models concerning the chemical reaction and diffusion control. The following conclusions were drawn from the study: 1) Pellets made of coarser particles have better reducibility than made of finer ones. Pellets with higher basicity have lower reducibility. 2) Fitting of the existing models to the experimental data indicates a mixed type of reduction reaction control, i.e., controlled by both chemical reaction and gas diffusion steps. 3) Activation energy increases from 44 to 75 kJ mol⁻¹ with different combinations of particle size and basicity. The activation energy for SR1 with 0 basicity is the lowest (44.35 kJ mol⁻¹) among all the size and basicity combinations. With a decrease in the particle size, activation energy increases from 44 to 52 kJ mol⁻¹ for 0 basicity, 50–56 kJ mol⁻¹ for 1 basicity, and from 59 to 75 kJ mol⁻¹ for 2 basicity pellets.

It is evident from the study that the rate of reduction is higher for iron ore pellets having bigger particles and lower basicity. Interestingly, in the present study, acidic pellets showed a higher rate of reduction than fluxed pellets. Based on the degree of reduction and kinetics, it is concluded that with more research on the right binders, coarser iron ore particles can be pelletized and effectively used as a feed material in direct reduction-based ironmaking processes.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

Keywords

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