Research Article

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Dephosphorization kinetics of high-P-containing reduced iron produced from oolitic hematite ore

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Abstract: To comprehensively utilize Fe and P in oolitic hematite ore, an innovative method was proposed to enhance P enrichment in the reduced iron during the reduction process. The reduced iron was then converted to low-P-containing molten iron and high-P-containing slag in the presence of CaO–SiO₂–FeO–Al₂O₃ slag. In this study, the P content of the final iron after 0–1,800 s dephosphorization was investigated at different slag composition conditions, and the dephosphorization kinetics of the reduced iron was analyzed. The results showed that the P content of the final iron sample decreased rapidly within 600 s of dephosphorization and became almost constant with increasing dephosphorization time to 1,800 s. The basicity, FeO content, and Al₂O₃ content also affected the dephosphorization rate of the reduced iron. The apparent dephosphorization rate constant ranged from 1.141×10^{-3} to $2.363 \times 10^{-3} \text{ g} \cdot (\text{cm}^2 \cdot \text{s})^{-1}$, and the overall mass transfer coefficient ranged from 2.47×10^{-3} to $3.38 \times 10^{-3} \text{ cm} \cdot \text{s}^{-1}$. The rate-controlling step of the dephosphorization process was the mass transfer of P in both the slag and iron phases. The findings of this study provide a theoretical basis for the utilization of refractory oolitic hematite ore.

Keywords: reduced iron, dephosphorization, apparent dephosphorization rate constant, mass transfer coefficient

1 Introduction

Oolitic hematite ore is one of the most refractory iron ores. In China, approximately 3.72 billion tons of oolitic hematite ore has not been exploited because of its concentric and layered oolitic textures, fine-grained iron minerals, and high P content [1,2]. Several methods, such as fine grinding–magnetic separation, fine grinding–flotation, acid leaching, bioleaching, and magnetic roasting–magnetic separation, have been adopted to extract oolitic hematite ore [3–7]. However, efficiently obtaining a qualified iron concentrate using these technologies is difficult.

In the past decade, the coal-based reduction followed by magnetic separation was proposed to treat oolitic hematite ore and regarded as a promising method [8,9]. Iron oxides are directly reduced to iron and aggregate to iron particles during the reduction process, and the iron particles are easily recovered from the reduction products by magnetic separation after grinding. According to previous experimental results, reduced iron that has values of Fe content, Fe recovery, and metallization degree of more than 90% is obtained from oolitic hematite ore. In addition, many basic studies have been conducted on this subject. Iron oxides in oolitic hematite ore are reduced in the sequence $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO}$ (FeAl_2O_4 , Fe_2SiO_4) $\rightarrow \text{Fe}$ [10,11]. The reduction kinetics of oolitic hematite ore were investigated based on the nonisothermal method, and the results show that the reduction process is described by a three-dimensional diffusion reaction model, and the corresponding apparent activation energy is in the range of 159.2–169.6 kJ·mol^{−1} [11]. Moreover, the entire reduction process includes the chemical reaction, nucleation, and crystal growth stages. The mean size of the iron particles increases with increasing reduction temperature or by extending the reduction time [10,12].

With further research on the coal-based reduction technology focused on oolitic hematite ore, the existential state of P during the reduction process has received considerable attention. Li et al. [13] and Yu et al. [14] found a nonnegligible content of P in the reduced iron, and the P content increases with increasing coal dosage

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or decreasing size of ore particles. Given that P is a harmful impurity to many steel grades, research has focused on reducing the P content of the reduced iron. Rao et al. [15] reported that the P content of the reduced iron decreases from 0.88 to 0.02% when 20% sodium sulfate is used as dephosphorization agent. A study by Li et al. [16] shows that the reduced iron containing 0.09% P is produced from an oolitic hematite ore with the addition of 7.5% sodium sulfate and 1.5% borax. Yu et al. [17] found that the P content of the reduced iron decreases to 0.07% when the oolitic hematite ore is reduced in the presence of 15% Ca(OH)_2 and 3% Na_2CO_3 .

Furthermore, another alternative method was proposed to comprehensively utilize Fe and P in oolitic hematite ore, namely promoting P into the iron phase during the reduction process. The reduced iron with a high content of P is converted to molten iron and high-P-containing slag, which can be used as a phosphate fertilizer, by a dephosphorization process [18,19]. Han et al. [20] revealed that the reduction behavior of apatite to P is a first-order reaction, and the reduction degree of apatite increases with increasing temperature or reduction time. Gao et al. [21] reported that P is easily enriched in the reduced iron during the reduction process of oolitic hematite ore. The P content of the reduced iron reaches approximately 2%. According to the author's earlier investigation, when the dephosphorization of the reduced iron with 1.74% P is carried out in the presence of a suitable $\text{CaO-SiO}_2\text{-FeO-Al}_2\text{O}_3$ slag, the final iron with 0.2% P and the high-P-containing slag with 14.41% P_2O_5 are obtained. The high-P-containing slag can be used as a phosphate fertilizer [22]. However, theories concerning the dephosphorization behavior of the reduced iron still need to be improved. This research focuses on the dephosphorization kinetics of the reduced iron. The apparent dephosphorization rate constant and overall mass transfer coefficient of the dephosphorization process were investigated in detail. The rate-controlling step was also analyzed.

2 Materials and methods

2.1 Materials

The reduced iron was produced from an oolitic hematite ore, which contained 42.21% Fe and 1.31% P. The oolitic hematite ore was crushed to -2 mm and then reduced by coal in a heating furnace. The temperature, reduction time, and C/O molar ratio (the molar ratio of fixed C in

coal to O in iron oxides) were, respectively, 1,548 K, 60 min, and 2.0. Then, the reduction products were ground using a $\Phi 460$ mm $\times$ 500 mm ball mill, and a two-stage magnetic separation was conducted using a $\Phi 240$ mm $\times$ 120 mm low-intensity magnetic separator. The magnetic field intensities of the magnetic separation were 79.62 and 47.77 $\text{kA}\cdot\text{m}^{-1}$. The chemical composition of the reduced iron is listed in Table 1.

As shown in Table 1, Fe existed in the form of metallic iron, apart from 10.19% FeO. The metallic iron content reached 84.34%. The P content of the reduced iron was 1.74%, and the other impurities included 2.12% SiO_2 , 1.19% Al_2O_3 , and 0.19% C.

2.2 Dephosphorization experiments

The reduced iron dephosphorization experiments were performed at 1,873 K using a MXGL1700-80 vertical tube-type furnace. $\text{CaO-SiO}_2\text{-FeO-Al}_2\text{O}_3$ slag was used for dephosphorization, which was formed by impurities in the reduced iron and analytically pure CaO , SiO_2 , Al_2O_3 , and FeO reagents. The mass ratio of the added reagents and reduced iron was 0.2.

A total of 40 g of the reduced iron and 8 g of reagents according to the predetermined chemical composition of slag were placed in a corundum crucible with a diameter of 50 mm. The crucible was placed in a furnace tube when the temperature reached 1,873 K. The dephosphorization process started when the slag and the reduced iron were melted under the protection of nitrogen. After 120–1,800 s of dephosphorization, the crucible was removed from the furnace and cooled to room temperature under the protection of nitrogen. The final iron sample and slag were then obtained, and the P content of the final iron sample was determined by chemical analysis.

2.3 Kinetics analysis

The two-film theory and kinetic modeling were used to analyze the dephosphorization process of the reduced

Table 1: Chemical compositions of reduced iron (wt%)

Constituents	MFe	FeO	P	C	SiO_2	Al_2O_3
Content	84.34	10.19	1.74	0.19	2.12	1.19

iron [23,24]. The dephosphorization reaction can be described by equation (1), namely (1) [P] in the iron phase and (FeO) in the slag phase diffuse to the slag–iron interface; (2) the chemical reaction occurs at the interface and produces (P_2O_5) and [Fe]; and (3) (P_2O_5) and [Fe] are transferred into the slag and iron phases, respectively.

2.3.1 Apparent dephosphorization rate constant

According to Fick's first law, the mass transfer rates of P in the iron and slag phases are given by equations (2) and (3):

$$J_{[P]} = k_M(C_{[P]}^i - C_{[P]}), \quad (2)$$

$$J_{(P)} = k_S(C_{(P)}^i - C_{(P)}), \quad (3)$$

where $J_{[P]}$ and $J_{(P)}$ ($\text{mol} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) are the mass transfer rates of P in the iron and slag phases, respectively; k_M and k_S ($\text{m} \cdot \text{s}^{-1}$) are the mass transfer coefficients of P in the iron and slag phases, respectively; $C_{[P]}^i$ and $C_{(P)}^i$ ($\text{mol} \cdot \text{m}^{-3}$) are the molarities of P in the iron and slag sides of the reaction interface, respectively; and $C_{[P]}$ and $C_{(P)}$ ($\text{mol} \cdot \text{m}^{-3}$) are the molarities of P inner the iron and slag phases, respectively.

The molarities in equations (2) and (3) are then converted to mass concentrations. The mass transfer equations can be expressed as follows:

$$\frac{d[\%w_P]}{dt} = -\frac{A}{W_M} \rho_M k_M \{[\%w_P]^i - [\%w_P]\}, \quad (4)$$

$$\frac{d(\%w_P)}{dt} = -\frac{A}{W_S} \rho_S k_S \{(\%w_P) - (\%w_P)^i\}, \quad (5)$$

where $[\%w_P]$ and $(\%w_P)$ are the mass percentage concentrations of P in the iron and slag phases, respectively; $[\%w_P]^i$ and $(\%w_P)^i$ are the mass percentage concentrations of P in the iron and slag sides of the reaction interface, respectively; W_M and W_S are the masses of the iron phase and slag phases, respectively; ρ_M and ρ_S are the densities of the iron and slag phases, respectively.

If the decreased P content of the iron phase is transferred into the slag phase, the following relationships should exist:

$$\frac{d(\%w_P)}{dt} W_S = -\frac{d[\%w_P]}{dt} W_M, \quad (6)$$

$$\rho_S k_S \{(\%w_P) - (\%w_P)^i\} = \rho_M k_M \{[\%w_P] - [\%w_P]^i\}. \quad (7)$$

The value of $[\%w_P]^i$ can be calculated by submitting the equilibrium distribution ratio of P (equation (8)) in equation (7).

$$L_P = \frac{(\%w_P)^i}{[\%w_P]^i}, \quad (8)$$

$$[\%w_P]^i = \frac{\rho_M k_M [\%w_P] + \rho_S k_S (\%w_P)}{\rho_M k_M + \rho_S k_S L_P}. \quad (9)$$

According to equations (9) and (4), the dephosphorization rate is given by equation (10):

$$\frac{d[\%w_P]}{dt} = -\frac{A}{W_M} \frac{1}{\frac{1}{\rho_S k_S} + \frac{L_P}{\rho_M k_M}} \{L_P [\%w_P] - (\%w_P)\}. \quad (10)$$

The apparent dephosphorization rate constant is defined as equation (11):

$$k_P = \frac{1}{\frac{1}{\rho_S k_S} + \frac{L_P}{\rho_M k_M}}. \quad (11)$$

Therefore, equation (10) can be rewritten as follows:

$$\frac{d[\%w_P]}{dt} = -\frac{A}{W_M} k_P \{L_P [\%w_P] - (\%w_P)\}. \quad (12)$$

According to mass conservation, the content of P in the iron phase at a given time, t , can be expressed as equation (13):

$$W_M [\%w_P] = W_M [\%w_P]^0 - W_S (\%w_P), \quad (13)$$

where $[\%w_P]^0$ is the initial P content of the iron phase.

The kinetic model for the change in the P content in the iron phase as a function of time can be given by equation (14) after submitting equation (13) into equation (12):

$$[\%w_P] = \frac{[\%w_P]^0 \cdot L_P}{L_P + \frac{W_M}{W_S}} e^{\frac{A}{W_M} k_P (L_P + \frac{W_M}{W_S}) t} + \frac{[\%w_P]^0 \cdot \frac{W_M}{W_S}}{L_P + \frac{W_M}{W_S}}. \quad (14)$$

To facilitate the processing of experimental data, equation (14) can be expressed as follows:

$$y = A_1 e^{-x/a} + y_0, \quad (15)$$

$$y_0 = \frac{[\%w_P]^0 \cdot \frac{W_M}{W_S}}{L_P + \frac{W_M}{W_S}}, \quad (16)$$

$$A_1 = \frac{[\%w_P]^0 \cdot L_P}{L_P + \frac{W_M}{W_S}}, \quad (17)$$

$$a = \left[\frac{A}{W_M} k_P (L_P + \frac{W_M}{W_S}) \right]^{-1}. \quad (18)$$

2.3.2 Overall mass transfer coefficient

The overall mass transfer coefficient of the dephosphorization of the reduced iron is defined as equation (19):

$$k_0 = \frac{1}{\frac{\rho_M}{\rho_S k_S L_P} + \frac{1}{k_M}}. \quad (19)$$

Combining equations (10), (11), (13), and (19), the overall mass transfer coefficient can be calculated as follows:

$$\frac{A\rho_M}{W_M} k_0 t = \frac{1}{1 + \frac{W_M}{W_S L_P}} \ln \left\{ \left(1 + \frac{W_M}{W_S L_P} \right) \frac{[\%w_P]}{[\%w_P]^0} - \frac{W_M}{W_S L_P} \right\} \quad (20)$$

3 Results and discussion

3.1 Apparent dephosphorization rate constant

The apparent dephosphorization rate constant, k_p , is an important parameter of dephosphorization kinetics, and the reciprocal, $\frac{1}{k_p}$, represents the mass transfer resistance of the dephosphorization process. The k_p value of the reduced iron can be determined by submitting the P content of the final iron sample into equations (15)–(18).

3.1.1 Effect of basicity

The FeO and Al_2O_3 contents of the slag were fixed at 55 and 6%, respectively. The effect of the slag basicity ($w_{\text{CaO}}/w_{\text{SiO}_2}$) on the P content of the final iron sample and the fitting curves of the P content as a function of dephosphorization time are shown in Figure 1.

As shown in Figure 1, the P content of the final iron sample decreased rapidly within 600 s and was almost constant after 600–1,800 s of dephosphorization. The basicity of the slag affected the P content of the final iron sample, L_p , and k_p , and the P content decreased to 0.20%. The value of L_p was in the range of 12.24–23.11, and the value of k_p ranged from 1.141×10^{-3} to $1.769 \times 10^{-3} \text{ g} \cdot (\text{cm}^2 \cdot \text{s})^{-1}$. The values of k_p were affected by L_p , which was also found in other dephosphorization studies [25]. The meanings of the relationship between k_p and L_p are discussed further in the Section 3.3. In addition, a basicity of 3.5 provided a better dephosphorization effect compared with those of a basicity of 3.0 or 4.5. This can be attributed to the higher basicity of slag, which increased the content of CaO and improved the P_2O_5 storage capacity of the molten slag. However, an excessively high basicity resulted in the incomplete melting of CaO, which was not conducive to dephosphorization.

Figure 1: Fitting curves of the P content of the final iron sample as a function of time with the basicity ranging from 3.0 to 4.5.

3.1.2 Effect of FeO content

The effect of the FeO content of the slag on the dephosphorization behavior was analyzed at a basicity of 3.5 and an Al_2O_3 content of 6%. The P content of the final iron sample and the fitting curves are shown in Figure 2.

As shown in Figure 2, the FeO content of the slag also affected the P content of the final iron sample, and the lowest P content of the final iron sample was obtained at an FeO content of 55%. The values of L_p and k_p at FeO contents of 45, 55, and 60% were 8.15×10^{-3} and 2.363×10^{-3} , 23.11×10^{-3} and 1.414×10^{-3} , and 12.65×10^{-3} and $1.803 \times 10^{-3} \text{ g} \cdot (\text{cm}^2 \cdot \text{s})^{-1}$, respectively. The higher FeO content was favorable for the oxidation of P in iron phase, and then this molecule migrated into the slag phase, as described by equation (1). However, the polarization force of Fe^{2+} was stronger than that of Ca^{2+} , which led to the polarization, deformation, and even destruction of PO_4^{3-} . Phosphate generated in the slag phase became unstable in the presence of excess FeO.

3.1.3 Effect of Al_2O_3 content

The basicity and FeO content of the slag were 3.5 and 55%, respectively. The P content of the final iron sample

Figure 2: Fitting curves of the P content of the final iron sample as a function of time with the FeO content ranging from 45 to 60%.

Figure 3: Fitting curves of the P content of the final iron sample as a function of time with Al_2O_3 content ranging from 4 to 10%.

as a function of dephosphorization time under different Al_2O_3 conditions is shown in Figure 3.

Figure 3 shows that the P content of the final iron sample decreased to 0.2% when the Al_2O_3 content increased from 4 to 6%, although the P content increased as the Al_2O_3 content increased to 10%. The L_p and k_p values were, respectively, 23.11×10^{-3} and $1.414 \times 10^{-3} \text{ g}/(\text{cm}^2 \cdot \text{s})^{-1}$ at a suitable slag composition, specifically a basicity of 3.5, an FeO content of 55%, and an Al_2O_3 content of 6%. This result occurred because Al_2O_3 reacted with other oxides and generated low melting point materials, which improved the rheological properties of the molten slag. The dephosphorization effect was then strengthened.

Overall, the fitting curves in Figures 1–3 exhibit good agreement with the test results. The k_p determined in the present study ranged from 1.141×10^{-3} to $2.363 \times 10^{-3} \text{ g}/(\text{cm}^2 \cdot \text{s})^{-1}$. A suitable initial slag resulted in a high value of k_p , which represented a low mass transfer resistance during the dephosphorization process.

3.2 Overall mass transfer coefficient

The mass transfer of P in the iron or slag phases is difficult to distinguish during the dephosphorization process. To

analyze the mass transfer behavior of P, some researchers assumed that the mass transfer process is completely controlled by one of the phases [26]. Furthermore, another method was proposed to regard the iron and slag phases as a liquid phase system, and the overall mass transfer coefficient, k_0 , was used to characterize the dephosphorization rate [24,27,28]. The k_0 value for the dephosphorization of the reduced iron was analyzed by submitting the P content and time values into equation (20).

3.2.1 Effect of basicity

Figure 4 shows the fitting curves of the P content as a function of time and the calculated k_0 at different basicities. Slag basicities of 3.0, 3.5, and 4.5 resulted in k_0 values of 2.91×10^{-3} , 3.38×10^{-3} , and $2.78 \times 10^{-3} \text{ cm} \cdot \text{s}^{-1}$, respectively. Furthermore, the effect of basicity on k_0 was the same as its effect on the amount of P removed from the iron phase.

3.2.2 Effect of FeO content

The values of k_0 were calculated when the slag contained different amounts of FeO. The results are shown in Figure 5.

Figure 4: Variation of the P content as a function of time with the basicity ranging from 3.0 to 4.5.

As shown in the figure, the k_0 value increased from 2.47×10^{-3} to $3.38 \times 10^{-3} \text{ cm}\cdot\text{s}^{-1}$ with an increase in the FeO content from 45 to 55% but this value decreased to $2.92 \times 10^{-3} \text{ cm}\cdot\text{s}^{-1}$ with a further increase in the FeO content to 60%. The results further showed that an excess content of FeO was unfavorable for the stable existence of phosphate in the slag phase.

3.2.3 Effect of Al_2O_3 content

The effect of the Al_2O_3 content of the slag on the k_0 value is shown in Figure 6. The values of k_0 were 2.49×10^{-3} to $3.38 \times 10^{-3} \text{ cm}\cdot\text{s}^{-1}$ when the Al_2O_3 content of the slag ranged from 4 to 10%. The slag with 6% Al_2O_3 gave the maximum value of k_0 , which further proved that a suitable content of Al_2O_3 improved the dephosphorization capability of the molten slag.

Overall, the values of k_0 for the dephosphorization process of the reduced iron ranged from 2.47×10^{-3} to $3.38 \times 10^{-3} \text{ cm}\cdot\text{s}^{-1}$ at the present slag compositions. The effect of the slag composition on the k_0 was essentially the same as that of P removed from the iron phase. A suitable initial slag composition gave a higher value of k_0 , which also indicated a faster dephosphorization rate

Figure 5: Variation of the P content as a function of time when the FeO content ranged from 45 to 60%.

and better dephosphorization effect. Compared with the apparent dephosphorization rate constant, the overall mass transfer coefficient represents the mass transfer of P in the dephosphorization process more intuitively. In addition, the k_0 values in this study were slightly lower than those of the dephosphorization of iron containing 0.4% P [24]. This result was attributed to the P content of the reduced iron being as high as 1.74%, and the k_0 values of the investigation provided more reference for dephosphorization rate of the reduced iron with high P content.

3.3 Analysis of the rate-controlling step

Equation (11) can be transformed into the form of equation (21):

$$\frac{1}{k_P} = \frac{1}{\rho_S k_S} + \frac{L_P}{\rho_M k_M}. \quad (21)$$

As shown in equation (21), the mass transfer resistances of the dephosphorization process include the resistance of the slag phase, $\frac{1}{\rho_S k_S}$, and iron phase, $\frac{L_P}{\rho_M k_M}$. If the dephosphorization process is restricted only by the mass

Figure 6: Variation of the P content as a function of time when the Al_2O_3 content ranged from 4 to 10%.

transfer of P in the slag phase, L_P does not affect k_P . However, k_P exhibits an inversely proportional relationship with L_P when the dephosphorization process is restricted by the mass transfer of P in the iron phase.

Based on the values of k_P and L_P in Figures 1–3, the relationship between k_P and L_P is shown in Figure 7.

Figure 7: Relationship between k_P and L_P .

A correlation between k_P and L_P is shown in Figure 7. The values of k_P exhibited a decreasing trend as the values of L_P increased, which indicated that the mass transfer of P in the iron phase affected the dephosphorization rate. However, the inversely proportional relationship between k_P and L_P was not strict, which meant that the mass transfer of P in the slag phase also affected the dephosphorization rate. Thus, the dephosphorization of the reduced iron was controlled by the mixed mass transfer of P between the slag and iron phases.

4 Conclusion

The dephosphorization kinetics of a high-P-containing reduced iron in the presence of $\text{CaO-SiO}_2\text{-FeO-Al}_2\text{O}_3$ slag were analyzed in detail. The apparent dephosphorization rate constant and overall mass transfer coefficient were determined for different slag compositions. The rate-controlling step of the dephosphorization process is also discussed. The following conclusions were drawn from the present study.

- (1) The dephosphorization rate was rapid during the first 600 s, which was affected by the basicity, FeO content, and Al_2O_3 content of the slag. The apparent dephosphorization rate constant ranged from 1.141×10^{-3} to $2.363 \times 10^{-3} \text{ g} \cdot (\text{cm}^2 \cdot \text{s})^{-1}$.
- (2) The overall mass transfer coefficient was also affected by the slag composition, and the change rule of the overall mass transfer coefficient exhibited the same trend as that of P removed from the iron phase. The overall mass transfer coefficient was $3.38 \times 10^{-3} \text{ cm} \cdot \text{s}^{-1}$ at suitable slag compositions.
- (3) The rate-controlling step of the dephosphorization process was analyzed according to the relationship between the apparent dephosphorization rate constant and equilibrium distribution ratio. The dephosphorization process was restricted by mixed mass transfer in both the slag and iron phases.

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