Research Article

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Dephosphorization behavior of reduced iron and the properties of high-P-containing slag

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Abstract: Reduced iron (1.74% P) is produced from oolitic hematite ore by coal-based reduction and magnetic separation. To realize the comprehensive utilization of Fe and P, the dephosphorization behavior of the reduced iron is investigated in the presence of CaO–SiO₂–FeO–Al₂O₃ slag. The P content of the final iron and the P₂O₅ content of the high-P-containing slag are determined, and the phase composition and P₂O₅ solubility of the slag are analyzed. The P content can be decreased to 0.29% when the initial slag has a basicity of 3.5 and contains 55% FeO and 6% Al₂O₃. The phases of the high-P-containing slag are mainly Ca₂Al₂SiO₇, Ca₂SiO₄, Ca₅(PO₄)₂SiO₄, and FeO, and P exists in the form of Ca₅(PO₄)₂SiO₄. Excessively high basicity or low content of FeO and Al₂O₃ results in free CaO, which affects the dephosphorization results. The change rule of the intensity of the Ca₅(PO₄)₂SiO₄ diffraction peak agrees well with the dephosphorization indexes, which further verify the accuracy of the dephosphorization experiments. Moreover, the P₂O₅ content and P₂O₅ solubility of the high-P-containing slag reached as high as 14.41 and 94.54%, respectively, indicating that it can be used as a phosphate fertilizer.

Keywords: high-P-containing reduced iron, dephosphorization, slag, phosphate fertilizer

1 Introduction

Iron ores are listed as strategic raw materials in the iron and steel industry by some countries. With the consumption of high-quality iron ores, the utilization of refractory iron ores has attracted increased interest [1]. Oolitic hematite ore is regarded as one of the most refractory iron ores because of its layered oolitic texture, fine-grained hematite size, and high P content [2]. It is difficult to obtain a qualified iron concentrate from oolitic hematite ore using traditional processing methods, including fine grinding and froth flotation, gravity separation, and magnetic separation [3–5].

Previous research has shown that coal-based reduction followed by magnetic separation is a promising method to utilize oolitic hematite ore [6,7]. In the reduction process, the hematite is reduced to iron which is then aggregated together to iron particles, which can then be easily recovered by magnetic separation [8,9]. Reduced iron is produced with iron recovery and a metallization ratio above 90%. However, parts of the apatite in oolitic hematite are also reduced to P and migrate into the iron phase. The P content of the reduced iron is considerably higher than that of direct reduced iron [10,11]. Therefore, the reasonable disposition of P is a challenge in this technology.

Some researchers have attempted to decrease the P content of reduced iron because P is deleterious to many steel grades. In normal practice, dephosphorization reagents, such as sodium carbonate, sodium sulfate, and borax, are added to oolitic hematite ore during the reduction process, and the reduction temperature is controlled to not exceed 1,473 K [12–15]. The reduction reaction of apatite is restricted, which effectively decreases the generation of P. Therefore, the P content of the reduced iron is low. For example, Li et al. [13] reported that the P content of reduced iron decreased to 0.09% when an oolitic hematite ore was reduced at 1,323 K and in the presence of 7.5% sodium sulfate and 1.5% borax.

Concurrently, another approach was proposed to enhance P migration into the reduced iron. The high-P-containing reduced iron could be converted to molten

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iron and high-P-containing slag by a dephosphorization process [16,17]. This method was expected to realize the comprehensive utilization of Fe and P in oolitic hematite ore because the high-P-containing slag could be used as a phosphate fertilizer. Li *et al.* [16] and Han *et al.* [18] found that increasing the temperature or C/O molar ratio (the molar ratio of fixed C in coal to O in the iron oxides of the ore) was beneficial for promoting the reduction of apatite and improving the P content of the reduced iron. When the oolitic hematite ore with a C/O molar ratio not lower than 2.0 was reduced at temperatures exceeding 1,523 K, approximately 80% apatite was reduced to P, and the P content of the reduced iron reached approximately 2%. However, the dephosphorization of reduced iron with a high P content has not been systematically studied.

This study focuses on the dephosphorization behavior of high-P-containing reduced iron in the presence of the CaO–SiO₂–FeO–Al₂O₃ slag. The effect of the chemical composition of the initial slag on the P content of the final metal and high-P-containing slag was investigated in detail. Meanwhile, the phase compositions of the high-P-containing slag and the solubility of P₂O₅ in the slag were analyzed, which will boost the comprehensive utilization of Fe and P in the oolitic hematite ore.

2 Experimental

2.1 Materials

The reduced iron was produced from an oolitic hematite ore, which contained 1.31% P and 42.21% Fe. First, the oolitic hematite ore was reduced in a unidirectional heating furnace at a 1,548 K reduction temperature, 2.0 C/O molar ratio, and 60 min reduction time. The ore sample used for each test was 3 kg. The reduction products were ground to 85% passing 74 μ m using a Φ 460 mm $\times$ 500 mm ball mill. Then, the grinding products underwent a two-stage magnetic separation by a Φ 240 mm $\times$ 120 mm low-intensity magnetic separator with the magnetic field intensities of 79.62 kA/m and 47.77 kA/m, respectively. The magnetic concentrates were the reduced iron. The chemical composition of the reduced iron is listed in Table 1.

As shown in Table 1, the reduced iron contained 92.27% total Fe, of which metallic Fe accounted for 84.34%. The P content of the reduced iron was 1.74%. The impurities were mainly 10.19% FeO, 2.12% SiO₂, and 1.19% Al₂O₃, which will become part of the CaO–SiO₂–FeO–Al₂O₃ slag in the dephosphorization process.

Table 1: Chemical compositions of the reduced iron (mass %)

TFe	MFe	FeO	P	C	SiO ₂	Al ₂ O ₃
92.27	84.34	10.19	1.74	0.19	2.12	1.19

Table 2 shows the particle sizes of the reduced iron. Most of the iron particles were <0.1 mm and accounted for 87.24% of the Fe content.

The materials used for slag-making included analytically pure CaO, SiO₂, Al₂O₃, and FeO reagents.

2.2 Experimental method

The dephosphorization experiments were conducted at 1,873 K, and the mass ratio of slag to reduced iron was fixed at 0.2. Based on the designed basicity ($w_{\text{CaO}}/w_{\text{SiO}_2}$) and the FeO and Al₂O₃ contents, the amounts of CaO, SiO₂, FeO, and Al₂O₃ to be added could be calculated. The oxides in the reduced iron were also considered in the calculation because the oxides were present in the slag phase after melting the reduced iron.

About 40 g of reduced iron and 8 g of initial slag were loaded in a 50 mm-diameter corundum crucible. The crucible was then placed in an MXGL1700-80 vertical tube-type furnace at 1,873 K. The dephosphorization reaction started when the reduced iron and slag were melted under the protection of an N atmosphere. After 2 to 30 min of dephosphorization, the corundum crucible was removed from the furnace. The final metal and high-P-containing slag were obtained after cooling the crucible to room temperature under an N atmosphere. The P content of the final metal and the P₂O₅ content of the high-P-containing slag were determined by chemical analysis. The dephosphorization ratio of the reduced iron was calculated using equation (1):

$$\eta_p = \frac{w_{i[\text{P}]} - w_{f[\text{P}]}}{w_{i[\text{P}]}} \times 100\%, \quad (1)$$

where η_p (%) is the dephosphorization ratio, $w_{i[\text{P}]}$ (%) is the initial P content of the reduced iron, and $w_{f[\text{P}]}$ (%) is the P content of the final iron.

Furthermore, the high-P-containing slag produced under suitable dephosphorization conditions was ground to different particle sizes using an XPM- Φ 120 $\times$ 3 three-head grinder. The P₂O₅ in the ground product was insoluble in water but was soluble in a 2% citric acid solution. Therefore, the soluble P₂O₅ content was determined by chemical analysis according to the national standard GB

Table 2: Size distribution of the reduced iron (mass %)

Size fraction (mm)	−0.15 + 0.1	−0.1 + 0.074	−0.074 + 0.063	−0.063 + 0.045	−0.045
Content	12.76	30.41	18.95	19.84	18.04

20412-2006 [19]. The solubility of P_2O_5 was calculated using equation (2):

$$\alpha = \frac{\omega_{S(P)}}{\omega_{(P)}} \times 100\%, \quad (2)$$

where α (%) is the solubility of P_2O_5 , $\omega_{S(P)}$ (%) is the soluble P_2O_5 content in the ground product, and $\omega_{(P)}$ (%) is the P_2O_5 content in the ground product.

2.3 Characterization

The phase compositions of the high-P-containing slag were analyzed using a PANalytical X'pert PW3040 X-ray diffraction analyzer (XRD). The sample was scanned at a voltage of 40 kV and a current of 40 mA, while the diffraction angle ranged from 10 to 90°.

2.4 Dephosphorization process

The dephosphorization process can be described by the theoretical model of molten slag ions, given by equation (3) [20]:

If the $[O]$ is provided by the FeO in the slag, then

The overall process can be written as equation (5):

3 Results and discussion

3.1 Dephosphorization behavior of reduced iron

3.1.1 Determination of dephosphorization time

Before investigating the effect of the slag composition on the dephosphorization indexes, a suitable dephosphorization time needed to be determined. Figure 1 shows the P content of the final metal and the P_2O_5 content of the high-P-containing slag as a function of time where the initial slag composition was 55% FeO and 6% Al_2O_3 content with 3.5 basicity.

Figure 1: Effect of the dephosphorization time on the P content of the final metal and the P_2O_5 content of the high-P-containing slag: (a) P content and dephosphorization ratio and (b) P_2O_5 content.

Figure 1 shows that as the dephosphorization time increased from 2 to 5 min, the P content of the final metal decreased to 0.23% from 0.88% and the dephosphorization ratio increased from 49.43 to 86.78%, while the P_2O_5 content of the high-P-containing slag increased from 7.58 to 13.90%. With a further extension of the dephosphorization time from 10 to 30 min, the P content of the final metal slightly decreased to $0.20 \pm 0.01\%$, and the P_2O_5 content of the high-P-containing slag was approximately 14.4%. The dephosphorization reaction was considered to be almost complete after 10 to 30 min because the experiments were not thermodynamic equilibrium tests. Therefore, the dephosphorization time was determined to be 20 min in follow-up studies.

3.1.2 Effect of basicity

Figure 2 shows the effect of basicity on the dephosphorization results at a FeO content of 55% and an Al_2O_3 content of 6%.

Figure 2 shows that the basicity considerably affected the dephosphorization of the reduced iron. The P content of the final metal decreased from 0.29 to 0.20% and the dephosphorization ratio increased from 83.33 to 88.51% as the basicity increased from 3.0 to 3.5. The corresponding P_2O_5 content of the high-P-containing slag increased from 13.12 to 14.41%. However, a further increase in basicity was unfavorable for dephosphorization. The P content of the final metal and the P_2O_5 content of the high-P-containing slag were 0.34 and 12.37%, respectively, at a basicity of 5.0. This is because high basicity increases

the CaO content in the slag system, which reduces the activity of P_2O_5 in the molten slag and improves the phosphorus storage capacity of the molten slag. However, the excess CaO did not melt completely, which was not conducive to dephosphorization.

3.1.3 Effect of FeO content

Figure 3 plots the effect of the FeO content on the P content of the final metal and the P_2O_5 content of the high-P-containing slag at a basicity of 3.5 and an Al_2O_3 content of 6%.

Figure 3 shows that the P content of the final metal decreased from 0.45 to 0.20% as the FeO content increased from 40 to 55%, while the dephosphorization ratio increased from 74.14 to 88.51%. The corresponding P_2O_5 content of the high-P-containing slag increased from 7.88 to 14.41%. However, when the FeO content reached 60%, the P content of the final metal increased to 0.31%. This may be attributed to an excess FeO content that decreases the CaO content of the slag system, reducing the P storage capacity of the molten slag. Furthermore, excess FeO reduced the stability of phosphate and deteriorated the dephosphorization effect.

3.1.4 Effect of Al_2O_3 content

The basicity and FeO content were fixed at 3.5 and 55%, respectively. Figure 4 shows the effect of the Al_2O_3 content on the dephosphorization indexes.

Figure 2: Effect of basicity on the P content of the final metal and the P_2O_5 content of the high-P-containing slag: (a) P content and dephosphorization ratio and (b) P_2O_5 content.

Figure 3: Effect of the FeO content on the P content of the final metal and the P_2O_5 content of the high-P-containing slag: (a) P content and dephosphorization ratio and (b) P_2O_5 content.

Figure 4 shows that as the Al_2O_3 content increased from 4 to 6%, the P content of the final metal decreased from 0.26 to 0.20%, the dephosphorization ratio increased from 85.06 to 88.51%, and the P_2O_5 content of the high-P-containing slag increased from 11.66 to 14.41%. A further increase in the Al_2O_3 content resulted in a higher P content in the final metal and a lower P_2O_5 content in the high-P-containing slag. This phenomenon occurs because an appropriate amount of Al_2O_3 can generate low melting point materials with other oxides in the slag, which is favorable for reducing the melting point of the slag and improving the rheological properties of the molten slag.

However, the high-melting-point substances may be formed when the Al_2O_3 content exceeds 6% [21].

3.2 Phase compositions of high P-containing slag

3.2.1 Effect of basicity

Figure 5 shows the XRD patterns of the high-P-containing slag at different initial basicity. The basicity affected the

Figure 4: Effect of the Al_2O_3 content on the P content of the final metal and the P_2O_5 content of the high-P-containing slag: (a) P content and dephosphorization ratio and (b) P_2O_5 content.

Figure 5: XRD patterns of the high-P-containing slag at different basicities.

phase composition of the slag and the intensity of the diffraction peaks. The high-P-containing slag was mainly composed of $\text{Ca}_2\text{Al}_2\text{SiO}_7$, Ca_2SiO_4 , $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$, and FeO. Phosphorus existed in the form of $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$, and its diffraction peak intensity increased with a basicity increase from 3.0 to 3.5 but decreased when the basicity exceeded 4.0. The intensity of the Ca_2SiO_4 diffraction peaks decreased and the intensity of the $\text{Ca}_2\text{Al}_2\text{SiO}_7$ and FeO diffraction peaks increased with an increase in the basicity. Additionally, the diffraction peak of free CaO could not be detected when the basicity was below 3.5, but the peak appeared and became stronger when the basicity reached and exceeded 4.0.

Figure 6: XRD patterns of the high-P-containing slag at different FeO contents.

3.2.2 Effect of FeO content

The effect of the FeO content on the phase composition of the high-P-containing slag was analyzed at basicity of 3.5. The XRD patterns are shown in Figure 6.

Figure 6 shows that the diffraction peak of free CaO was visible at a 40% FeO content but decreased gradually and even disappeared with a further increase in FeO. The intensities of the Ca_2SiO_4 and FeO diffraction peaks exhibited an increasing trend, while the intensity of the $\text{Ca}_2\text{Al}_2\text{SiO}_7$ diffraction peaks decreased with an increase in the FeO content. Furthermore, the intensity of the $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$ diffraction peaks increased as the FeO content increased from 40 to 55% but decreased slightly at a 60% FeO content.

3.2.3 Effect of Al_2O_3 content

The effect of the Al_2O_3 content on the phase composition of the high-P-containing slag was investigated at a basicity of 3.5 and FeO content of 55%. The XRD patterns are shown in Figure 7.

Figure 7 shows that the diffraction peak of free CaO appeared at a 4% Al_2O_3 content but disappeared when the Al_2O_3 content reached 6%. Meanwhile, the presence of free CaO resulted in low-intensity Ca_2SiO_4 diffraction peaks. The intensity of the $\text{Ca}_2\text{Al}_2\text{SiO}_7$ diffraction peaks did not increase with increasing Al_2O_3 content, while the intensity of the FeO diffraction peaks decreased slightly. This phenomenon may be attributed to the formation of Fe aluminates, which were present in small quantities

Figure 7: XRD patterns of the high-P-containing slag at different Al_2O_3 contents.

Figure 8: Effect of particle size on the P_2O_5 solubility of the high-P-containing slag.

and were not detected by the XRD analyzer. Moreover, the intensity of the $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$ diffraction peaks increased with increasing Al_2O_3 content from 4 to 6% but decreased with an Al_2O_3 content above 6%.

In summary, the change rule of the intensity of the $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$ diffraction peaks with basicity, FeO content, and Al_2O_3 content was consistent with the P_2O_5 content of the high-P-containing slag in Section 3.2. This further verifies the accuracy of the dephosphorization tests.

3.3 P_2O_5 solubility of high-P-containing slag

The slag was ground to different particle sizes, and the effect of particle size on the specific surface area and P_2O_5 solubility of the high-P-containing slag is shown in Figure 8.

Figure 8 shows that the specific surface area and the P_2O_5 solubility were affected by the fineness of the high-P-containing slag. When the particle size decreased from $d_{90} = 92.4$ to $38.7 \mu\text{m}$, the specific surface area of the slag increased from 682.1 to 1,606 m^2/kg , but the P_2O_5 solubility changed slightly and ranged from 87.59 to 87.65%. Moreover, the specific surface area of the slag increased to 2,373–2,677 m^2/kg when the particle size further decreased to $d_{90} = 23.8$ – $20.7 \mu\text{m}$, while the P_2O_5 solubility reached 93.81–94.54%. This can be attributed to the lattice energy of the slag increasing during the grinding process, which resulted in the breakage of chemical bonds and a decrease in the degree of crystallinity

on the particle surface [22]. The solubility of P_2O_5 in the slag was then improved.

4 Conclusion

The dephosphorization of high-P-containing reduced iron was investigated in the presence of $\text{CaO-SiO}_2\text{-FeO-Al}_2\text{O}_3$ slag. The properties of the high-P-containing slag, including phase composition and P_2O_5 solubility, were also analyzed. The following conclusions were drawn from the experimental results:

- (1) The composition of the initial slag affected the dephosphorization of the reduced iron. Higher basicity, FeO content, and Al_2O_3 content favored dephosphorization to a certain extent. The P content of the final iron was decreased from 1.74 to 0.2% with a basicity of 3.5, FeO content of 55%, and Al_2O_3 content of 6%. The dephosphorization ratio reached 88.51%.
- (2) The high-P-containing slag was obtained apart from the final iron, which was composed of $\text{Ca}_2\text{Al}_2\text{SiO}_7$, Ca_2SiO_4 , $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$, and FeO, and P existed in the form of $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$. The intensity of the $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$ diffraction peaks changed with the initial slag composition, and the change rule was in accordance with the indexes of dephosphorization.
- (3) The P_2O_5 content of the high-P-containing slag was 14.41% under suitable dephosphorization conditions, while the P_2O_5 solubility of the slag reached approximately 94%, which indicates that the slag could be used as a phosphate fertilizer.

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