1. Introduction

Nowadays, because of the limited reserves and quick depletion of hematite-rich iron ores, many iron and steel companies are attempting to exploit refractory and low-grade iron ores as alternative resources for ironmaking. Oolitic hematite ores are widely distributed in USA, France, Germany, Russia and China. In China, the total proven reserves of such resources are over 4.0 billion tons and are therefore receiving considerable attention. The oolitic hematite ore, which has a special concentric and layered oolitic texture as well as high phosphorus content (0.4 mass%–1.8 mass%), is regarded as one of the most refractory iron ore resources. The phosphorus is mainly in the form of apatite, where the particle size of the crystal is very fine and not easy to separate. Therefore, the high-phosphorus oolitic hematite is difficult to utilize as a raw material in ironmaking. For instance, low efficiency of dephosphorization, relatively high cost and environmental pollution represent drawbacks that make these techniques impractical.

In order to efficiently utilize high-phosphorus oolitic iron ores, many pyrometallurgical processes were also proposed. The processes of reduction roasting (magnetic roasting and direct reduction roasting) followed by magnetic separation have been applied to recover iron from oolitic hematite ore. For magnetic roasting, the magnetic separation of magnetite from gangues is unsatisfied because the typical structure of oolitic hematite remains approximately

Recently, additives such as CaCO₃ and Na₂CO₃ have been proven to be capable of enhancing the reduction of high-phosphorus oolitic hematite ore and obtaining high-grade direct reduction iron (DRI) with low-P by magnetic separation. In this study, the mechanisms of adding CaCO₃ during direct reduction were further studied by gas analyzer, X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was found that CaCO₃ can inhibit the reduction of fluorapatite, and more P still remained as fluorapatite which can be removed by magnetic separation. In addition, CaO and CO₂ decomposed from CaCO₃ promoted reduction atmosphere, but the contribution of CO₂ to reduction of iron oxides was very limited, in fact CaO played a central role. An appropriate CaCO₃ dosage can enhance reduction of oolitic hematite. Addition of CaCO₃ led to the consumption of quartz and inhibited the generation of fayalite, which reduced FeO content in the slag, and as a result more FeO was reduced to metallic iron. A DRI with 94.51 mass% Fe, and 0.17 mass% P can be obtained at an iron recovery of 89.3 mass% by adding 10 mass% CaCO₃. However, excessive CaCO₃ reacted with gangue and FeO to generate a large amount of slag (such as kirschsteinite), which hindered the diffusion and growth of the iron grain and increased the iron loss.

KEY WORDS: high-phosphorus oolitic hematite; direct reduction; CaCO₃; Fe-P; magnetic separation.
unchanged. Direct reduction roasting is performed at a relatively higher temperature compared with magnetic roasting, which is found to be an effective way to recover iron from oolitic hematite ore. However, the dephosphorization is poor without the aid of additives during reduction roasting, thereby there is an excessive content of phosphorus in the magnetic concentrate, which is then not suitable as a steelmaking feedstock. Yu et al. reported that without any additive, a direct reduction iron (DRI) with 91.14 mass% Fe and 0.3 mass% P was produced from an oolitic hematite ore containing 43.58 mass% Fe and 0.83 mass% P. It can be seen that the separation effect of Fe and P is not satisfied without the aid of additive. Therefore, additives such as CaCO3, Na2SO4, and Na2CO3 were used to enhance the dephosphorization. These alkali salts are proved to be capable of reducing the phosphorus content of DRI and facilitating the growth of metallic iron grains, which is favorable for the magnetic separation of iron from the gangue mineral. Cao et al. used the combination of CaCO3 and Na2CO3 as additives in the reduction of oolitic hematite ore at 1150°C; at last a magnetic concentrate with 91.88 mass% Fe, and 0.065 mass% P was obtained. Although CaCO3 has been proven to be capable of enhancing the reduction of phosphorus-rich oolitic hematite ore, the mechanisms of CaCO3 on iron oxides and dephosphorization have not yet been clearly elucidated. To further understand the effect of CaCO3 on the reduction atmosphere during reduction, the furnace was heated to 1150°C and held for 60 min in a nitrogen atmosphere at a N2 flow rate 4 L/min. Figure 1 shows schematic diagram of experimental apparatus. During heating, gases from furnace were allowed to enter the gas analyzer to measure the concentration changes of CO and CO2, and then resulting data was transferred to a computer’s data acquisition system in real-time. The gas analyzer detects gas error in the range of ±0.02%. After that, Metallization degree (Rm) of the reduced sample was calculated as follows:

\[ R_m = \frac{\omega_{Fe}}{\omega_{TFe}} \times 100\% \]

Lignitous was used as reducing agent. The proximate analysis results (air dry) of the lignitous were composed of 15.72 mass% moisture, 9.46 mass% ash, 32.43 mass% volatiles and 42.39 mass% fixed carbon. CaCO3 and CaO used as additives were analytical reagent (AR) grade.

### 2. Experimental

#### 2.1. Materials

The oolitic iron ore used in this study was collected from Hubei Province, China. Chemical analysis of raw ore showed that the phosphorus content was 0.83 mass% and the iron content was 43.58 mass%. The detail information of the ore sample has been described in a previous paper.

| Rotameter |
|------------|
| N2         |
| Corundum crucible |
| Roasting under N2 |

Fig. 1. Schematic diagram of experimental apparatus during gas test.
where: $α_{Fe}$ is mass percent of total iron in the reduced sample, mass%; and $α_{MFe}$ is that of metallic iron in the reduced sample, mass%.

Meanwhile, in order to investigate the effect of CO$_2$ decomposed from CaCO$_3$ on reduction of iron oxides, CaO was used as additive in this study. Dosages of CaO used are equal to amounts of CaO contained in CaCO$_3$, so the dosage of CaO was calculated as follows:

$$w_{CaO} = \frac{56}{100} \times w_{CaCO_3},$$

where: $w_{CaO}$ is the dosage of CaO, mass%; $w_{CaCO_3}$ is the dosage of CaCO$_3$, mass%; 56 is the molecular weight of CaO and 100 is the molecular weight of CaCO$_3$.

### 2.3. Analysis and Characterization

The chemical analyses of the DRI were determined by ICP-AES (Optima 5300DV, Perkin Elmer, USA). The determination of metallic iron (MFe) content was performed using the potassium dichromate volumetric method. The phase compositions of the samples were performed by Rigaku DMAX-RB X-ray Diffract Meter (Cu target). Scanning electron microscopy (SEM) with energy dispersive spectrum (EDS) (Carl Zeiss EVO18) analysis was performed on reduced briquettes mounted on epoxy resin and subsequently polished and carbon sprayed. The concentrations of CO and CO$_2$ in the reaction process were detected with an infrared flue gas analyzer (MRU Messgeräte für Rauchgase und Umweltschutz GmbH, Neckarsulm, Germany).

### 3. Results and Discussion

#### 3.1. Effect of CaCO$_3$ Dosage on the Reduction Followed by Magnetic Separation

The effect of CaCO$_3$ dosage on the reduction followed by magnetic separation was investigated. The experiments were conducted at conditions of temperature 1150°C, roasted time 60 min and lignitous dosage 20 mass%. The dosages of lignitous and CaCO$_3$ are expressed by a percentage that refers to their mass ratio to the raw ore.

As shown in Fig. 2, the phosphorus content of DRI decreases gradually with increasing dosage of CaCO$_3$. A DRI with 0.32 mass% phosphorus can be obtained without CaCO$_3$. When the dosage of CaCO$_3$ increases to 10 mass%, the phosphorus content decreases to 0.17 mass%, and then the dephosphorization effect is gradually weakened with further increasing CaCO$_3$ dosage. The iron recovery of DRI increases from 86.7 mass% to 91.1 mass% when CaCO$_3$ dosage increases from 0 mass% to 15 mass%, but when CaCO$_3$ dosage is more than 15 mass%, a big drop in iron recovery is observed. The iron content of DRI increases 92.07 mass% to 94.51 mass% when CaCO$_3$ dosage increases from 0 mass% to 10 mass%, but it decreases gradually as CaCO$_3$ dosage exceed 10 mass%. These results reveal that an appropriate CaCO$_3$ dosage can improve the recovery of iron and the iron content of DRI, and decrease the phosphorus content. A DRI with 0.17 mass% of phosphorus is obtained by adding 10 mass% CaCO$_3$, while the iron content is 94.51 mass% with a recovery of 89.3 mass%. However, with further increasing CaCO$_3$ dosage, the dephosphorization effect was gradually weakened, and excessive CaCO$_3$ led to a low iron recovery and iron content of DRI. To fully explain the experimental results and to further explore the mechanism, roasting products obtained with different CaCO$_3$ dosage were studied by XRD.

#### 3.2. Effects of CaCO$_3$ on Mineral Composition of Roasting Products

In the presence of different dosage of CaCO$_3$ (0 mass%, 10 mass%, 20 mass%, and 30 mass%), the phase transformations of roasting products were identified by XRD. As shown in Fig. 3, metallic iron, quartz, fayalite and anorthite are observed in the roasting products without any additives; the appearance of fayalite indicates that reaction (1) occurred. When the CaCO$_3$ dosage increases from 0 mass% to 10 mass%, the diffraction peaks of gehlenite are observed, which suggest that CaCO$_3$ reacted with Al$_2$O$_3$ and SiO$_2$ to form Ca$_2$Al$_2$Si$_2$O$_7$ (gehlenite) by reaction (2) and (3), and the diffraction peaks of metallic iron become stronger, whereas that of quartz and fayalite become weaker. This indicated that addition of CaCO$_3$ led to the consumption of quartz and inhibited the generation of fayalite, that also means reactions (3) between were prior to the reduction (1). Thus more FeO was reduced to metallic iron. In addition, the diffraction peaks of fluorapatite is observed, which shows that the reduction (4) was inhibited and some phosphorus still remained in the gangue as fluorapatite.

\[
2\text{FeO} + \text{SiO}_2 = \text{Fe}_2\text{SiO}_4 \quad (1)
\]
\[
\text{CaCO}_3 = \text{CaO} + 2\text{CO} \quad (2)
\]
\[
2\text{CaO} + 2\text{SiO}_2 + \text{Al}_2\text{O}_3 = \text{Ca}_2\text{Al}_2\text{Si}_2\text{O}_7 \quad (3)
\]
\[
\text{Ca}_{10}(\text{PO}_4)_6 \text{F}_2 + 15\text{C} + 6\text{SiO}_2 = \text{CaF}_2 + 3\text{P}_2 + 15\text{CO} + 3\text{Ca}_3\text{Si}_2\text{O}_7 \quad (4)
\]
\[
\text{CaO} + \text{SiO}_2 + 2\text{FeO} = \text{CaFe}_2\text{SiO}_4 \quad (5)
\]

By increasing the CaCO$_3$ dosage from 10% to 20%, the peak intensity of fluorapatite increases, which indicated that the presence of 20 mass% CaCO$_3$ further inhibits the reduction of fluorapatite and more phosphorus remained as...
fluorapatite which can be removed by magnetic separation. These results explain why the P-content of DRI decreased as CaCO₃ dosage increased. Besides, the peak intensity of metallic iron decreases and peaks of kirschsteinite and wustite appear, suggesting that the oolitic hematite was inhibited; this is attributed to that the CaO dissociated from CaCO₃ reacted with SiO₂ and FeO in the raw ore to formed kirschsteinite by reaction (5), which inhibited the reduction of FeO to Fe. When CaCO₃ dosage increases from 20 mass% to 30 mass%, no new phase is observed, and the peak intensity of fluorapatite, kirschsteinite and wustite further increases, whereas that of metallic iron further decreases. This shows that more iron existed in the form of kirschsteinite and wustite. These results also explain the low iron recovery of DRI when adding excessive CaCO₃ dosage.

3.3. Effects of CaCO₃ or CaO on Gas Changes

It is well known that the coal-based reduction of hematite proceeds mainly through reaction (6) and reaction (7). In the presence of different dosage of CaCO₃ (0 mass%, 10 mass%, 20 mass%, and 30 mass%), the effect of CaCO₃ dosage on reduction atmosphere was investigated. Besides, in order to investigate the effect of CO₂ decomposed from CaCO₃, CaO was also used as additive in this study. The different dosages of CaO was 0 mass%, 5.6 mass%, 11.2 mass% and 16.8 mass%, respectively. Figure 4 shows concentration changes of CO₂ and CO by adding different dosages of additive. Solid lines represent CO₂ and dashed lines represent CO.

\[ C + CO_2 = 2CO \]  

Figure 3. XRD patterns of roasting products with different CaCO₃ dosages. A-iron (Fe); B-quartz (SiO₂); C-fayalite (Fe₂SiO₄); D-fluorapatite (Ca₅(PO₄)₃F); E-anorthite, (CaAl₂SiO₆); F-gehlenite (Ca₂Al₂SiO₇); G-kirschsteinite (CaFe₂SiO₄); H-wustite (FeO).

![Figure 3](image1)

![Figure 4](image2)
FeO + CO $\rightarrow$ FeO$_{\text{tot}}$ + CO$_2$  \hfill (7)

As shown in Fig. 4(a) or 4(b), according to concentration changes of CO and CO$_2$, direct reduction can be divided into four stages. CO and CO$_2$ are not detected in the first stage, indicating that carbothermal reduction reaction had not yet started. In the second stage, CO concentration is below 0.1%; CO$_2$ concentration increases slowly below 1.0% with rising temperature. Under different dosages of CaCO$_3$ or CaO, curves of CO, CO$_2$ are similar in this stage, suggesting that the additives did not act at low temperatures. In the third stage which occurs in the temperature range of 845–1150°C, as shown in Fig. 4(a)–III, CO production increases with increasing dosages of CaO, which suggest that CaO had influence on reaction (6) and the reduction atmosphere was slightly enhanced by adding CaO. For CO$_2$, when CaO dosage increases from 0 mass% to 5.6 mass%, CO$_2$ production increased, which may indicate that the reaction (7) was also promoted to production of CO$_2$. However, when CaO dosage further exceeds 5.6 mass%, CO$_2$ production decreases; this phenomenon may be attributed to two factors. On the one hand reaction (6) was promoted, thus more C reacted with CO$_2$ with the generation of CO, leading to the decrease of CO$_2$ production. On the other hand the excessive CaO reacted between gangue and FeO, generating a large amount of slag (such as kirschsteinite), which inhibited reaction (7), and thereby CO$_2$ production was also decreased when CaO dosage further increased. As shown in Fig. 4(b)–III, with increasing dosages of CaCO$_3$, CO, CO$_2$ production all increased, and CO production by adding CaCO$_3$ was more than that by adding CaO at the same CaO content. These results show that CO$_2$ decomposed from CaCO$_3$ further promoted the reaction (6). In the final stage, as shown in Fig. 4–IV, CO concentration was less than 0.1%, CO$_2$ concentration decreased slowly below 0.5%, suggesting that carbothermal reduction proceeded toward the end and the additives did not much effect in the late stage. In order to further investigate the effect of CO$_2$ and CaO on reduction of iron oxides, the metallization degree was studied.

3.4. Effects of CaCO$_3$ or CaO on Metallization Degree of Roasted Products

As shown in Fig. 5, it is observed that CaCO$_3$ and CaO have significant influence on the degree of iron oxide reduction. When CaCO$_3$ dosage increases from 0 mass% to 10 mass%, an increase in the metallization degree is observed, and the metallization degree of roasting products rises from 79.63% to 85.08%. After the metallization degree reaches a maximum, it then decreases rapidly; when CaCO$_3$ dosage increases to 30 mass%, the metallization degree decreases gradually to 66.98 mass%. The above results show that an appropriate CaCO$_3$ dosage increased the metallization degree of roasting products, but excessive CaCO$_3$ inhibited reduction of oolitic hematite ore. The metallization degree at different CaO dosage has a similar trend with that at different CaCO$_3$ dosage. However, At the same CaO content, the metallization rate by adding CaO is slightly lower than that by adding CaCO$_3$ when CaO dosage is 5.6 mass%, and when CaO dosage is 11.2 mass%, 16.8 mass%, the metallization rate by adding CaO is slightly higher than that by adding CaCO$_3$. These results suggest that although CO$_2$ decomposed from CaCO$_3$ promoted reduction atmosphere, the contribution of CO$_2$ to reduction of iron oxides was very limited, in fact CaO played a central role.

3.5. Effect of CaCO$_3$ on Microstructure of Roasted Products

Figure 6(a) shows SEM image of roasting product in the absence of CaCO$_3$. The light areas are metallic iron, and the oolitic structure in raw ore is incompletely destroyed. Figure 6(b) show that the some Fe is overlaps with P in the observed area. The result shows that the reduced P$_2$ entered into metal iron during reduction process. In fact, once the reduction (4) occurs along with the reduction of FeO to Fe, some elemental phosphorus can migrate into the metallic iron phase, which leads to be difficult to remove P by wet magnetic separation and results in a high phosphorus content in DRI.\cite{29,31} When 10 mass% CaCO$_3$ is added, in the partially enlarged view (f) of (c), it can be seen that the metallic iron particles are more than 40 μm with a strip structure. The EDS mappings for Fe and P of Fig. 6(f) are shown in Figs. 6(i) and 6(j). There is almost no phosphorus in the metallic iron in the presence of 10% CaCO$_3$. It indicated that CaCO$_3$ inhibited the reduction of fluorapatite. Yu et al.\cite{12,26,29} reported that when the oolitic hematite ore is reduced in the presence of CaCO$_3$, reactions between CaCO$_3$, SiO$_2$, and Al$_2$O$_3$ are prior to the reduction of fluorapatite, and the decrease of quartz content may not be conducive to the reduction of fluorapatite. These findings of research are further confirmed in this study. When CaCO$_3$ dosage was 20 mass%, in the partially enlarged view (g) of (d), the crystal grain of iron were widely distributed in the slag, and tiny wustite were observed. These results resulted in the recovery of iron and the iron content of DRI decreased by magnetic separation. In addition, the kirschsteinite (point 1) were observed in slag. It indicates that not only iron oxides were reduced to metallic iron during the reduction roasting, but reactions between CaO, FeO and SiO$_2$ also occured, which prevented FeO from forming metallic iron, thus the iron metallization degree decreased. When 30 mass% CaCO$_3$ is added, more fine-grained wustite
are observed as shown in Fig. 6(h); and besides the iron particles are tiny and closely exist in the slag, which is difficult to recover iron. In summary, the appropriate CaCO₃ dosage which is 10 mass%, can not only enhance the reduction of oolitic hematite ore but also reduce the phosphorus content of DRI. However, excessive CaCO₃ can react with gangue to generate a large amount of slag during the reduction process, which will hinder the diffusion and growth of the iron grain and increase the iron loss.

4. Conclusions

(1) CaCO₃ enhances the Fe and P separation during coal-based reduction of high-phosphorus oolitic hematite. CaCO₃ can inhibit the reduction of fluorapatite, and more phosphorus still remains as fluorapatite which can be removed by grinding and magnetic separation. The P content of DRI decreased from 0.32 mass% to 0.17 mass% when CaCO₃ dosage increased from 0 mass% to 10 mass%,

Fig. 6. SEM images of roasting products with different CaCO₃ dosages. (a)-0 mass% CaCO₃; (b)-elemental maps for Fe, P of (a); (c)-10 mass% CaCO₃; (d)-20 mass% CaCO₃; (e)-30 mass% CaCO₃. (f) partially enlarged view of (c); (g) partially enlarged view of (d); (h) partially enlarged view of (e); (i), (j)-elemental maps for Fe, P of (f).
and then the dephosphorization effect was gradually weakened with further increasing CaCO₃ dosage.

(2) According to concentration changes of CO and CO₂, coal-based reduction was divided into four stages. CaO and CO₂ decomposed from CaCO₃ can promote reduction atmosphere in the third stage. However, the contribution of CO₂ decomposed from CaCO₃ to reduction of iron oxides is very limited; in fact CaO plays a central role.

(3) An appropriate CaCO₃ dosage can enhance reduction of oolitic hematite. Addition of CaCO₃ leads to the consumption of quartz and inhibits the generation of fayalite, and the contribution of CO₂ decomposed from CaCO₃ can promote reduction of FeO in the slag, and as a result more FeO is reduced to metallic iron. A DRI with 94.51 mass% FeO is reduced to metallic iron. A DRI with 94.51 mass% can be obtained at an iron recovery of 89.3 mass% by adding 10 mass% CaCO₃ and 0.17 mass% P can be obtained at an iron recovery of 94.51 mass%.

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