

Thermodynamic analysis of the carbothermic reduction of a high-phosphorus oolitic iron ore by FactSage

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Abstract: A thermodynamic analysis of the carbothermic reduction of high-phosphorus oolitic iron ore (HPOIO) was conducted by the FactSage thermochemical software. The effects of temperature, C/O ratio, additive types, and dosages both on the reduction of fluorapatite and the formation of liquid slag were studied. The results show that the minimum thermodynamic reduction temperature of fluorapatite by carbon decreases to about 850°C, which is mainly ascribed to the presence of SiO₂, Al₂O₃, and Fe. The reduction rate of fluorapatite increases and the amount of liquid slag decreases with the rise of C/O ratio. The reduction of fluorapatite is hindered by the addition of CaO and Na₂CO₃, thereby allowing the selective reduction of iron oxides upon controlled C/O ratio. The thermodynamic results obtain in the present work are in good agreement with the experimental results available in the literatures.

Keywords: thermodynamic analysis; oolitic iron ore; carbothermal reduction

1. Introduction

High-phosphorus oolitic iron ore (HPOIO), one of the most important iron ore resources, can be widely found in France, Germany, America, Canada, and China, among other countries [1–2]. However, HPOIO is currently unexploited worldwide because of its unique oolitic structure and high levels of phosphorus that is resistant to the conventional mineral processing methods. The fast depletion of easy-to-process iron ores has made the exploitation of HPOIO increasingly important. The coal-based direct reduction process has been applied to HPOIO by several researchers with the aim to produce the direct reduction iron (DRI) [3–6]. In this process, iron oxides are primarily reduced to metallic iron with grain growth, and the roasted product is then ground to the liberation size followed by magnetic separation. The as-obtained iron product, containing more than 90wt% Fe and less than 0.1wt% P, is expected to be a good substitute for steel scrap during the steelmaking process in electric arc furnaces.

The effects of the temperature and time of roasting, the

type and dosage of additives, and the type and dosage of coal on the reduction of HPOIO have been largely investigated [3–4,6–9]. The selective reduction of iron oxides over fluorapatite and the growth of resulting iron grains are the key in producing DRI with high iron content and maintaining low P levels. With this aim, the roasting temperature and the coal dosage must be strictly controlled. Excessively high roasting temperatures can result in the reduction of fluorapatite while lower temperature is not conducive to the reduction of iron oxides and limited the growth of iron grains. An excess of reductant will facilitate the reduction of fluorapatite, hindering the growth of iron grains. Insufficient amounts of reductant are undesirable for the reduction of iron oxides. The addition of Na₂CO₃, CaCO₃, CaO, and Ca(OH)₂ can suppress the reduction of fluorapatite, promote the reduction of iron oxides, and facilitate the growth of iron grains in some cases [3,10–12]. Most of these works deal with the reaction kinetics of iron ore, the DRI quality, and the phase and microstructure changes of the roasted products.

Thermodynamic studies on the standard Gibbs free en-

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ergy changes (ΔG^\ominus) of some key reactions taken place during the carbothermic reduction of HPOIO (e.g., carbothermic reduction of fluorapatite with different additives) are scarce in the literatures [11,13]. However, the reactions involved in the reduction process are complicated owing to the complex composition of HPOIO. Thus, the thermodynamic results obtained by simply calculating the ΔG^\ominus of certain reactions may not be representative of the real process. In addition, the formation of liquid slag, which significantly affects the growth of iron grains, is difficult to investigate by simply considering the key reactions.

Consequently, to better understand the carbothermic reduction behavior of HPOIO, the main components of HPOIO were taken into account and a detailed thermodynamic study was performed using the FactSage thermochemical software. The effects of the temperature, the carbon dosage, and the additions of CaO and Na₂CO₃ on both the reduction of fluorapatite and the formation of liquid slag

were investigated. The theoretical predictions were also compared to the experimental results available in the literature.

2. Materials and methods

The HPOIO used herein was collected from the Hubei Province. The chemical composition of the iron ore is shown in Table 1.

X-Ray diffraction (XRD) analysis of the HPOIO revealed Fe to be mainly as hematite, while P mainly existed as fluorapatite [3]. Therefore, for the sake of calculations, Fe and P elements were expressed as Fe₂O₃ and Ca₁₀(PO₄)₆F₂, respectively. The content of S, TiO₂, V₂O₅, SrO, MnO, and As₂O₃ components were considered negligible. The simplified chemical composition of the HPOIO ore is shown in Table 2. The HPOIO-carbon system was thus simplified as Fe₂O₃-Ca₁₀(PO₄)₆F₂-SiO₂-Al₂O₃-CaO-MgO-K₂O-C.

Table 1. Chemical composition of the HPOIO used in the present study

												wt%
Fe	SiO ₂	Al ₂ O ₃	MgO	CaO	S	P	K ₂ O	TiO ₂	V ₂ O ₅	SrO	MnO	As ₂ O ₃
43.65	17.10	9.28	0.59	3.58	0.048	0.830	0.65	0.20	0.075	0.020	0.20	0.023

Table 2. Simplified chemical composition of the HPOIO used in the present study

								wt%
Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Ca ₁₀ (PO ₄) ₆ F ₂	CaO	MgO	K ₂ O	Total	
65.26	17.89	9.71	4.72	1.12	0.62	0.68	100.00	

The thermodynamic properties of Fe₂O₃-Ca₁₀(PO₄)₆F₂-SiO₂-Al₂O₃-CaO-MgO-K₂O-C system were calculated by the Equilib Module of FactSage 6.4 based on the minimization of the total Gibbs free energy. FactSage software could calculate the multiphase equilibria, the liquidus temperatures, and the proportions of liquid and solid phases in a multiphase system at specified conditions [14]. The FToxid and FactPS databases were selected for all the calculations (100 g iron ores at atmospheric pressure). The phases with low contents or outside the scope of this work were not shown in the output results.

3. Results and discussion

3.1. Effect of temperature on the equilibrium compositions of the phases

The effect of temperature on the equilibrium compositions of the phases at a C/O mole ratio of 1.0 (i.e., the stoichiometric amount of carbon required for the complete reduction of iron oxides to iron metal) was studied from 800°C to 1300°C, and the results are present in Fig. 1.

As can be seen in Fig. 1(a), the fluorapatite phase is transformed into Fe₃P at ~850°C, which is significantly lower than the minimum thermodynamic reduction temperature of fluorapatite by carbon [15]. Thus, the carbothermic reduction of fluorapatite is facilitated by other components present in the raw ore or formed during the reduction process. Fe₃P can be recovered along with the metallic iron by magnetic separation, thereby increasing the P content of DRI.

The liquid slag appears at ~1140°C, and its amount increases sharply with temperature in Fig. 1(b). The presence of liquid slag facilitates the growth of iron grains, thereby improving the separation of the metallic iron from slag. These results are in good agreement with previous experimental results, which showed the increase of iron contents in DRI with the roasting temperature [9].

Although the calculated values revealed a reduction temperature of fluorapatite by carbon well below the minimum thermodynamic reduction value, the detailed information on the contents or reactions leading to this result was not provided.

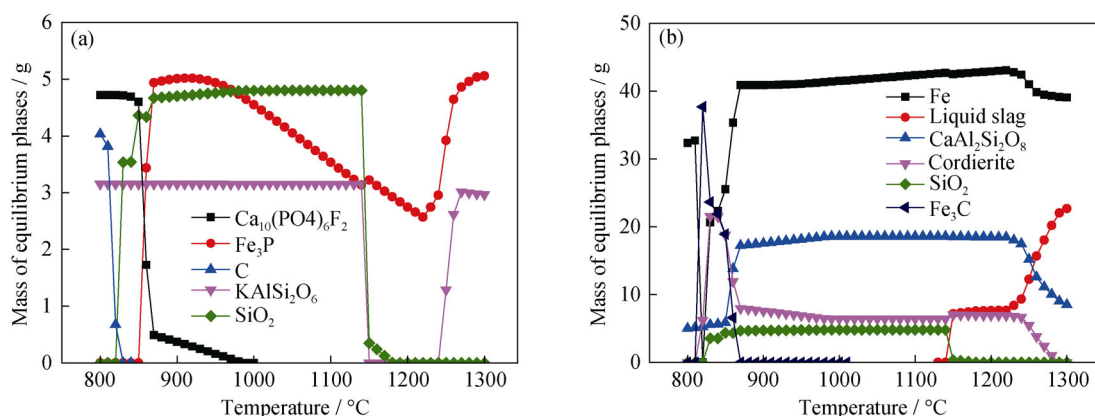


Fig. 1. Effect of temperature on the equilibrium compositions of the different phases.

SiO₂ and Al₂O₃ have been proven to be effective in promoting the carbothermic reduction of fluorapatite by providing a thermodynamic driving force [15]. The ΔG^\ominus values of the related reactions in Eqs. (1)–(8) were calculated by the FactSage reaction model. The ΔG^\ominus values were represented as a function of temperature, as shown in Fig. 2.

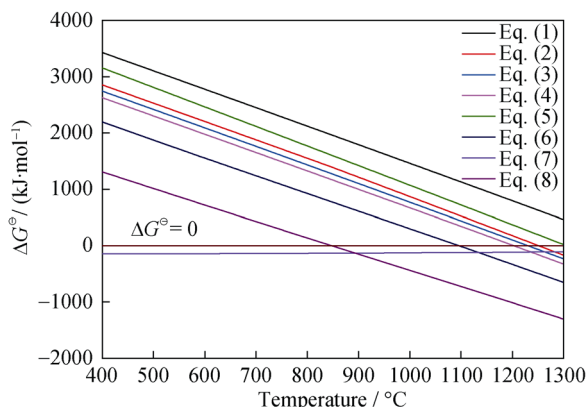
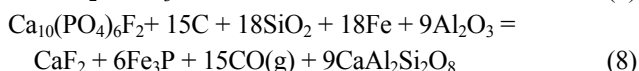
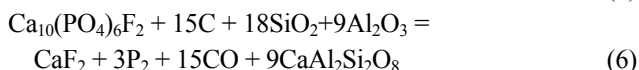
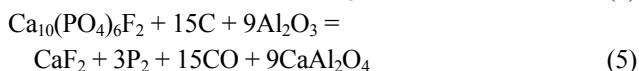
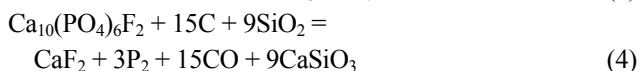
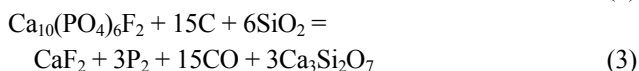
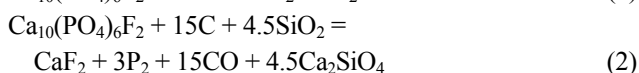
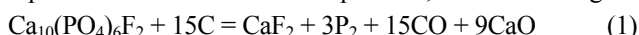


Fig. 2. ΔG^\ominus values of the processes described in Eqs. (1)–(8) as a function of temperature.

As shown in Fig. 2, SiO₂ significantly promotes the reduction of fluorapatite by carbon, and this effect increases

with the SiO₂/CaO mole ratio. Al₂O₃ also promotes the reduction of fluorapatite to a lower extent. The combined effect of SiO₂ and Al₂O₃ is higher than that of the single components.

Besides the components of the raw ore, the phases formed during the reduction of HPOIO may also affect the reduction of fluorapatite. The formation of Fe₃P in Eq. (7) was identified as the main source of P contamination in DRI. As shown in Fig. 2, the formation of Fe₃P shows the negative ΔG^\ominus values over the entire temperature range of the present study. Since P appears as a product in Eq. (6) and as a reactant in Eq. (7), these two reactions can be coupled as Eq. (8).

The process in Eq. (8) significantly shows the lower ΔG^\ominus values than other processes defined by Eqs. (1)–(6), as present in Fig. 2. These results indicate that Fe greatly facilitates the reduction of fluorapatite by carbon. Additionally, the minimum thermodynamic reduction temperature for the process in Eq. (8) is ~850°C, which is in agreement with the results in Fig. 1(a). Therefore, it can be concluded that the carbothermic reduction of fluorapatite by carbon is facilitated during the reduction of HPOIO via the processes described in Eqs. (1)–(8).

3.2. Effect of C/O ratio on the equilibrium compositions of the phases

The effect of the C/O ratio on the equilibrium compositions of the phases at 1200°C was performed by FactSage, and the results are shown in Fig. 3.

As can be seen in Fig. 3(a), no signs of fluorapatite are observed for the entire C/O ratio range. Instead, a Ca₃(PO₄)₂ phase is observed as a result of the decomposition of fluorapatite to Ca₃(PO₄)₂ and CaF₂. The amount of Ca₃(PO₄)₂ decreases with the C/O ratio. Fe₃P forms at a C/O ratio of 0.95, and the amount of this phase at equilibrium increases sharply for C/O ratios ranging from 0.95 to 1.1.

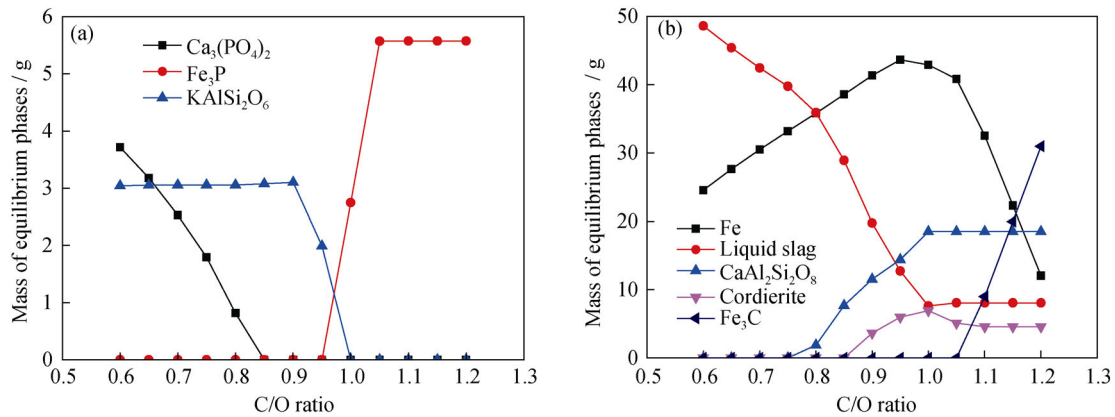


Fig. 3. Effect of the C/O ratio on the equilibrium compositions of the different phases.

The amount of metallic iron at equilibrium increases and the amount of liquid slag decreases for C/O ratios up to 1.0 (Fig. 3(b)). The amount of metallic iron reaches a maximum at a C/O ratio of ~1.0 and decreases thereafter as a result of the onset of Fe_3C and Fe_3P . The amount of liquid slag at equilibrium decreases to ~8 g at a C/O ratio of 1.0 and remains stable thereafter. The amount of liquid slag decreases as a result of the reduction of FeO in the slag to metallic Fe. This FeO plays a key role in decreasing the liquidus temperature of the slag by forming a fayalite-type slag with low melting point [16]. This trend is in agreement with the experimental results which show the lower melting degrees of the reduced products and the decrease of iron grain particle sizes with the reductant dosage [8,17].

Thus, it could be concluded that higher C/O ratios led to increasing P contents in DRI by facilitating the formation of

Fe_3P and hindering the separation of metallic iron from the P-containing slag. Several experimental studies showed the higher P and lower iron contents in DRI as the coal dosage increased [7–8,17]. Thus, to selectively reduce the iron oxides and promote the coarsening of iron grains, the C/O ratio should not be too high.

3.3. Effect of CaO content on the equilibrium compositions of the phases

The addition of CaO, CaCO_3 , and $\text{Ca}(\text{OH})_2$ has been reported to drastically affect the reduction of HPOIO [3,9,12,18]. Thus, it is of interest to investigate the effect of CaO on the equilibrium compositions of the phases. The calculations were performed at a C/O ratio of 1.0 and a reduction temperature of 1200°C, and the results are shown in Fig. 4.

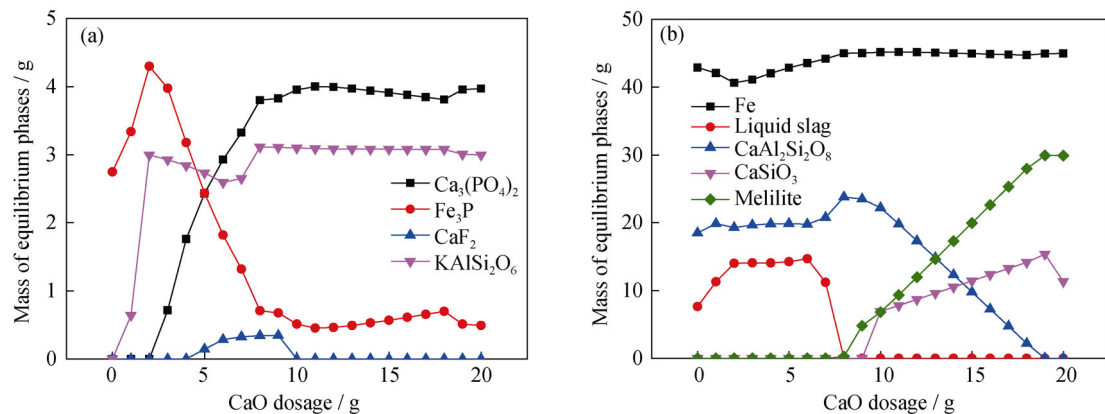


Fig. 4. Effect of the CaO dosage on the equilibrium compositions of the different phases.

As shown in Fig. 4(a), CaO dosages higher than 2 g make the amount of Fe_3P decrease and generate $\text{Ca}_3(\text{PO}_4)_2$, thereby indicating that the addition of CaO can inhibit the reduction of $\text{Ca}_3(\text{PO}_4)_2$. However, a small amount of Fe_3P remains at high CaO dosages. Thus, the amounts of $\text{Ca}_3(\text{PO}_4)_2$ and Fe_3P remain constant (4 and 0.5 g, respec-

tively) for CaO dosages higher than 10 g.

It was previously showed that the reduction of fluorapatite during the direct reduction of HPOIO at 1200°C could be inhibited by the addition of $\text{Ca}(\text{OH})_2$ [3]. However, a small amount of P was still detected in the metallic iron obtained at optimum $\text{Ca}(\text{OH})_2$ dosages (i.e., 15wt%, 11.35 g of

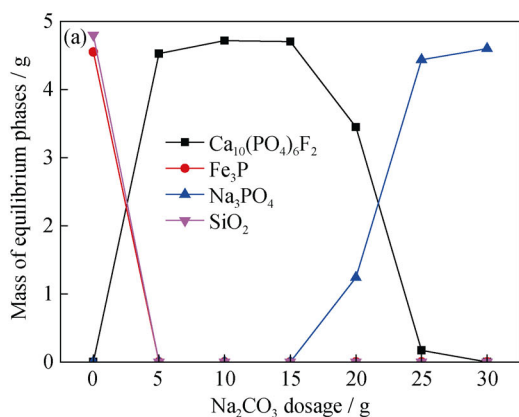
CaO in the present study). The calculated results were thus in agreement with the experimental results.

In Fig. 4(b), the amount of liquid slag increases from 7.73 to 14.01 g for CaO dosages up to 2 g, and remains stable for CaO dosages ranging from 2 to 6 g. A further increase of the CaO dosage sharply decreases the amount of liquid slag (e.g., the mass of liquid slag is zero for CaO dosages above 8 g). It was previously showed that the iron content of the DRI increased until a certain Ca(OH)₂ dosage and decreased thereafter [3]. The calculated results were thus in agreement with the experimental results.

3.4. Effect of Na₂CO₃ on the equilibrium compositions of the phases

Na₂CO₃ has been employed as an additive for the carbothermic reduction of HPOIO at temperatures ranging 1000–1100°C by several researchers, and its promoting effects on dephosphorization and iron grain growth have been generally recognized [4,6,19]. The effect of Na₂CO₃ on the equilibrium compositions of the different phases was investigated. The calculations were performed at a roasting temperature of 1000°C and a C/O ratio of 1.0. The results are shown in Fig. 5.

As can be observed in Fig. 5(a), the addition of Na₂CO₃ inhibits the reduction of fluorapatite, thereby avoiding the formation of Fe₃P. In the absence of Na₂CO₃,



Fe₃P is formed (4.55 g) at the expense of fluorapatite. No Fe₃P is detected and the amount of fluorapatite increases greatly at a Na₂CO₃ dosage of 5 g. At a Na₂CO₃ dosage of 10 g, the amount of fluorapatite reaches 4.72 g, indicating that the practical totality of fluorapatite is unreduced at these conditions. When Na₂CO₃ dosage exceeds 15 g, the amount of fluorapatite markedly drops while the amount of Na₂PO₄ increases to a large extent, thereby suggesting that the fluorapatite is converted into water-soluble Na₂PO₄. Yang *et al.* [6] carried out the carbothermic reduction of HPOIO at 1000°C by adding 30wt% sodium salt (i.e., a mixture of Na₂CO₃ and Na₂SO₄). As a result, 38.78wt% P present in the HPOIO was dissolved in water during the grinding and magnetic separation processes. The calculated results were thus in agreement with the experimental results.

In Fig. 5(b), the addition of Na₂CO₃ facilitates the formation of the liquid slag. Thus, the amount of liquid slag increases with the Na₂CO₃ dosage (no liquid slag is formed in the absence of Na₂CO₃ at 1000°C). As mentioned above, the liquid slag plays an essential role in facilitating the coarsening of the iron grains, thus improving the separation of metallic iron from slag. The addition of Na₂CO₃ can thus increase the iron content, then reduce the amount of P in the DRI. This result was basically in agreement with Bai's experimental results [19].

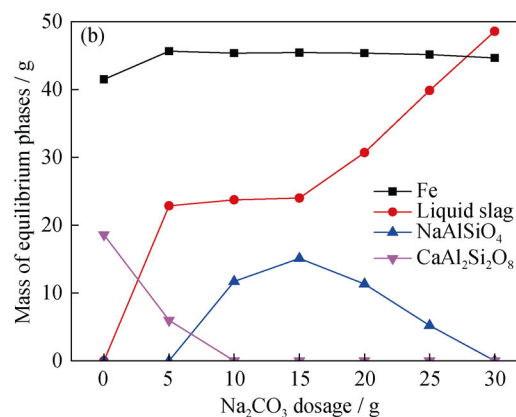


Fig. 5. Effect of Na₂CO₃ dosage on the equilibrium compositions of the different phases.

According to the thermodynamic analysis, the fluorapatite can be reduced by carbon at low temperatures and the addition of Na₂CO₃ can inhibit the reduction of fluorapatite. However, several experimental results showed that the fluorapatite was reduced to a very low extent and most of the fluorapatite phase remained unchanged during the reduction of HPOIO at temperatures below 1100°C. Moreover, the catalytic activity of Na₂CO₃ (i.e., gasification of carbon) during the carbothermic reduction of iron oxides has been

generally recognized [20]. Thus, the main functions of Na₂CO₃ during the reduction of HPOIO at low temperatures were to improve the reduction of iron oxides and facilitate the growth of the iron grains.

3.5. Effect of Na₂CO₃ on the equilibrium compositions of the phases in the presence of CaO

It was previously showed that the addition of Na₂CO₃ in the presence of 15wt% Ca(OH)₂ could further inhibit

the reduction of fluorapatite and then increase the iron content of the DRI [3]. The effect of Na_2CO_3 dosage on the equilibrium compositions of the phases in the presence of 11.35 g of CaO was calculated at a temperature of 1200°C and a C/O ratio of 1.0, the results are shown in Fig. 6.

As can be seen in Fig. 6(a), Fe_3P disappears upon the addition of 1 g Na_2CO_3 . The amount of $\text{Ca}_3(\text{PO}_4)_2$ increases after the addition of Na_2CO_3 and remains stable thereafter. Thus, the addition of Na_2CO_3 in the presence of 11.35 g CaO further hinders the reduction of fluorapatite. The amount of liquid slag increases sharply with the Na_2CO_3 dosage (Fig. 6(b)). Conversely, the amounts of CaSiO_3 , $\text{CaAl}_2\text{Si}_2\text{O}_8$, and Melilite are found to decrease with the Na_2CO_3 dosage. Thus, the addition of Na_2CO_3 promotes the

formation of the liquid slag.

In summary, the co-addition of Na_2CO_3 and CaO inhibited the reduction of fluorapatite, while also created the favorable conditions for the growth of iron grains. Under the optimal conditions, P remained as a compound in the slag and thus could be removed by ground and magnetic separation. These results were in good agreement with our previous experimental observations [3].

3.6. Effect of C/O ratio on the equilibrium compositions after addition of 11.5 g CaO and 3 g Na_2CO_3

The effect of C/O ratio on the equilibrium compositions after the addition of 11.5 g CaO and 3 g Na_2CO_3 was calculated at a temperature of 1200°C and a C/O ratio of 1.0, and the results are shown in Fig. 7.

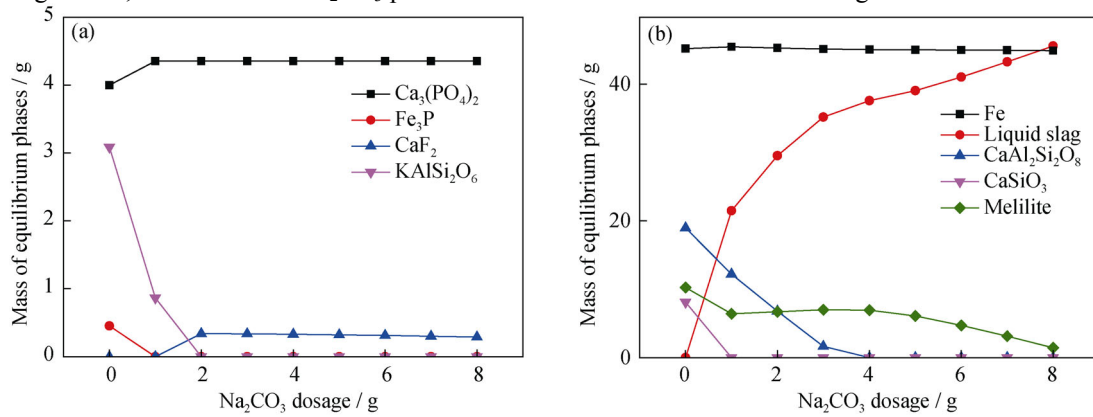


Fig. 6. Effect of Na_2CO_3 dosage on the equilibrium compositions of the different phases in the presence of 11.35 g of CaO.

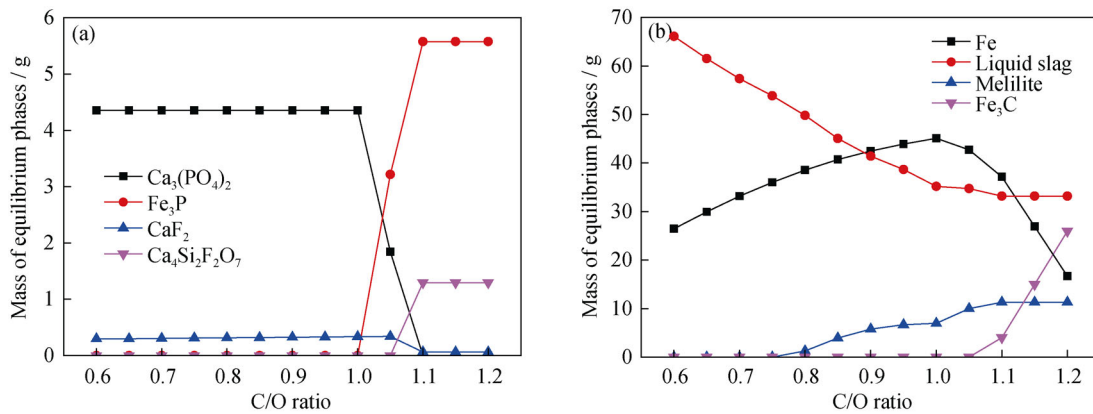


Fig. 7. Effect of C/O ratio on the equilibrium compositions after the addition of 11.5 g CaO and 3 g Na_2CO_3 .

For C/O ratios higher than 1.0 (11.35wt% CaO and 3wt% Na_2CO_3), the amount of Fe_3P sharply increases with the C/O ratio at the expense of $\text{Ca}_3(\text{PO}_4)_2$ (Fig. 7(a)). Thus, to selectively reduce the iron oxides over the fluorapatite, the C/O ratio should be controlled even after the addition of CaO and Na_2CO_3 . Additionally, the amount of liquid slag decreases with the C/O ratio (Fig. 7(b)), thereby hindering the coarsening of iron grains [17].

4. Conclusions

The carbothermic reduction of HPOIO was studied by thermodynamic modeling. The effects of the temperature, the C/O ratio, and the dosages of additives both on the fluorapatite reduction and the production of liquid slag were studied.

(1) The minimum thermodynamic reduction temperature of fluorapatite by carbon is decreased to ~850°C by the

presence of SiO₂, Al₂O₃, and Fe.

(2) An increase in the C/O ratio leads to higher reduction rates of fluorapatite and lower amounts of liquid slag. Therefore, to obtain a DRI powder with high iron and low P contents, the C/O ratio should not be too high.

(3) At a C/O ratio of 1.0 and a temperature of 1200°C, the reduction of fluorapatite can be hindered by the addition of CaO. However, a small amount of Fe₃P is formed even at optimum dosages of CaO. The co-addition of Na₂CO₃ along with CaO can further hinder the reduction of fluorapatite and facilitate the formation of liquid slag. Moreover, the C/O ratio should be controlled even after the addition of CaO and Na₂CO₃ to prevent the formation of Fe₃P.

(4) The thermodynamic results obtained in the present work are in general agreement with the experimental results available in the literatures.

Acknowledgements

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