



# Silica, Alkali Carbonate and Alkali Rich Metal Ore as Additive Effect on the Carbothermic Reduction Process of Phosphorus Ore

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## Abstract

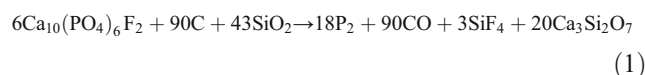
The phosphorus production industry is energy-intensive, which is one of the major reasons phosphorus has lower yields through furnace production. In this study, phosphorus conversion rate from phosphorus ore was investigated using four different fluxing agents: silica, potassium shale, potassium feldspar, and nepheline. Different holding times (10, 20, 30, and 40 min), acidity values (0.68, 0.88, 1.02, 1.42, and 2.02), coal surplus coefficients (1.05, 1.25, 1.5, 2, and 2.5), and calcination temperatures (1250 °C, 1300 °C, 1350 °C, 1400 °C, and 1450 °C) were studied. The results demonstrated that potassium shale, potassium feldspar, and nepheline as new fluxing agents improved phosphorus conversion rate under the same experimental conditions. To further ensure the significance of the experiment, the conversion rate of phosphorus from phosphorus ore was also investigated without an additive and with the addition of Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub>. The slag viscosity of different fluxing agents and different additives at high temperatures was analyzed via the spread area method. To investigate the mechanism of phosphorus conversion, silica and nepheline as fluxing slag at different calcination temperatures were analyzed using X-ray diffraction.

**Keywords** Phosphorus production · Alkali carbonate · Potassium shale · Potassium feldspar · Nepheline · Silica

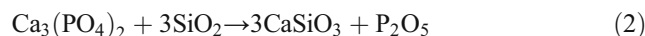
## 1 Introduction

Phosphorus is an important element and is considered at risk of depletion [1]. It is an essential raw material with wide applications in chemicals, food, cellular metabolism, pharmaceuticals, and military affairs [2–5]. It cannot be replaced in biochemical processes by any other element; humans ultimately rely on its availability [6–8]. For traditional mass production, the production of phosphorus requires a mixture of phosphorus ore, silica, and carbonaceous reductant through a carbothermal reduction reaction in an electric arc furnace

[9–11], where temperatures exceeding 1500 °C are required to complete yellow phosphorus production [3]. The total main reaction can be expressed as follows [12]:



In this, SiO<sub>2</sub> (silica) is used as a fluxing agent to promote the decomposition of phosphate ore and to reduce the reactants' melting temperature. The reactions can be expressed concisely as follows [13]:



The energy-intensive process of yellow phosphorus production is highly challenging due to the consumption of 13,000–15,000 kWh electric energy per ton of yellow phosphorus [14, 15]. A lower phosphorus yield is one reason why higher-power electricity consumption occurs in industrial phosphorus furnaces. Further, because the phosphorus production process results in serious environmental pollution and high energy consumption, productivity has decreased substantially in many developed countries for

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over a decade [3]. However, more than 1300 k tons per year of phosphorous are produced in China, more than 50% of which is yielded in Yunnan [13]. Therefore, it is extremely important to investigate energy-saving and cost-reducing technologies for the sustainable development of phosphorus production in electric arc furnaces.

There have been many studies devoted to recycled phosphorus, given the importance of phosphorus to various fields. Phosphorus recovery techniques include a wide spectrum of technologies [16–18], such as phosphorus precipitation as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) [19], chemical dosing methods [20], seawater-driven forward osmosis [21], acid-treated concrete sludge [6], sludge incineration with phosphorus recovery from ash [22–24], and others [25, 26]. In terms of technical feasibility, these approaches tend to be chemically, energetically, and operationally intensive [25], while the value of phosphorus products is too low to compete with the relatively low cost of mined phosphorus [17]. However, there is a paucity of research into replacing the fluxing agent in yellow phosphorus production to improve phosphorus yield using the relatively traditional process of phosphorus production in industrial furnaces. Yet it is important to examine the effects of fluxing agents as additives in phosphorus production, especially considering the amount of energy required in the process.

Compared to the study of other industrial and energy-intensive processes, fluxing agents are already widely used to improve the reactive process. In the iron and steel industry, which should be most familiar to readers of this field, the effects of pyroxenite, limestone, and magnesite as fluxing agents on swelling behavior has been studied [27]. Yang et al. [28] studied the effects of  $\text{CaF}_2$  as a fluxing agent on surface tension and density of  $\text{CaO-SiO}_2\text{-B}_2\text{O}_3$  ternary slag systems and found that adding  $\text{CaF}_2$  as a fluxing agent reduced slag systems, density of slag, and the temperature of surface tension. The effect of  $\text{CaF}_2$  as a fluxing agent was investigated in the silicothermal process of magnesium production, and  $\text{CaF}_2$  was found to improve the diffusion process [29]. In the ceramics industry, compared with a traditional ceramic fluxing agent (sodium-potassium feldspar), natural zeolite was used as a fluxing agent instead of the white wares used in the sintered process of ceramics. The sintering temperature was reduced by about 100 °C [30]. Despite these results, very few reports have considered replacing the fluxing

agent in the yellow phosphorus production process. Until now, only the Gibbs free energy and temperature of potassium feldspar-CaO as fluxing agents in place of silica-CaO have been investigated via thermodynamic analysis [13, 31]. Scholars have also examined the effects of  $\text{SiO}_2$ , silica, K-feldspar, and nepheline as fluxing agents in an idealized system of phosphorus production on the characteristic temperature and flow properties of materials during reduction [14]. Thus, although research into the fluxing agents used in phosphorus production has just begun, researchers continue to focus on the theories or ideal conditions for evaluation. Therefore, this study investigated the effects of different fluxing agents (silica, potassium shale, potassium feldspar, and nepheline) on phosphorus separation efficiency from phosphorus ore. The respective influences of temperature, holding time, acidity value, and the surplus coefficient of anthracite coal on phosphorus separation efficiency were also examined in detail.

## 2 Materials and Methods

### 2.1 Materials

All raw materials (phosphorus ore, silica, potassium shale, potassium feldspar, and nepheline) in this investigation were obtained from Guizhou, China. The chemical compositions of raw materials were analyzed using ICP-AES and are presented in Table 1. All samples were dried for 2 h at 105 °C before being crushed and sieved to obtain particle sizes of 75–100  $\mu\text{m}$ .

Coal was used as a carbonaceous reductant in this study; the proximate analysis of coal appears in Table 2.

### 2.2 Methods

#### 2.2.1 Carbothermic Reduction Experiment of Phosphate Ore

First, all raw materials (phosphorus ore, coal, silica, potassium shale, potassium feldspar, and nepheline) were weighed and blended in accordance with a proposed experimental system in an agate mortar; next, the samples were placed in a constant temperature zone in a high-temperature pipe boiler at a predetermined temperature; third, after the reactions, the

**Table 1** The chemical composition of raw materials (wt.%)

Raw materials	$\text{SiO}_2$	$\text{CaO}$	$\text{Al}_2\text{O}_3$	$\text{MgO}$	$\text{Fe}_2\text{O}_3$	$\text{P}_2\text{O}_5$	$\text{K}_2\text{O}$	$\text{Na}_2\text{O}$
Phosphorus ore	15.51	41.87	1.45	0.99	0.86	29.49	–	–
Silica	96.3	1.04	0.94	0.57	0.78	–	–	–
Potassium shale	58.63	1.46	17.62	1.14	5.66	–	8.05	0.5
Potassium feldspar	63.93	1.57	18.45	1.60	1.30	–	10.14	2.87
Nepheline	56.9	3.17	21.25	0.17	4.26	–	4.77	8.57

**Table 2** Proximate analysis of coal (wt.%)

	M <sub>ad</sub>	A <sub>ad</sub>	V <sub>ad</sub>	FC <sub>ad</sub>
Content	7.24	14.26	14.26	79.11

roasting slag was cooled naturally to room temperature in a dryer; and finally, the calcined sample was milled to an average particle size of less than 80 μm before conducting phosphorus analysis.

### 2.2.2 Analysis of Flow Temperature

The flow temperature of the slag was an important factor to evaluate in this study. The slag was ground and sieved to less than 2 μm, and the mixture was shaped into triangular cones with dextrin on a gray cone plate. These triangular pyramidal samples were dried for 24 h at room temperature. Finally, the samples were placed in an HR-4A tester to measure the flow temperature of the slag. The above process was repeated three times to obtain accurate data, which were then averaged.

### 2.2.3 Analysis of Slag Viscosity

The viscosity of the residue was determined by the spread area after calcination at certain temperatures. To compare and analyze the influence of different fluxing agents on residue viscosity, all samples were placed on corundum discs and calcined in a muffle furnace at preset temperatures for 2 h before being withdrawn from the corundum discs and cooled naturally to room temperature in a desiccator. Then, the spread area was measured with a ruler to evaluate the slag viscosity.

### 2.2.4 Conversion Rate of Phosphorus

The amounts of P<sub>2</sub>O<sub>5</sub> in the phosphorus ore and slag were measured according to the national standard (GB/T 1871.1–1995). The phosphorus conversion rate η (%) can be calculated using Eq. (4):

$$\eta = \frac{m_0 - m}{m_0} \times 100\% \quad (4)$$

where  $m_0$  is the phosphorus content (P<sub>2</sub>O<sub>5</sub> in phosphorus ore) of the initial sample (%), and  $m$  is the phosphorus content (P<sub>2</sub>O<sub>5</sub> in slag) of the sample after the carbothermic reduction experiment (%).

The slag samples were analyzed using a Rigaku X-ray diffractometer with CuKα radiation ( $\lambda = 0.154056$  nm, 40 kV, 200 mA). The diffractometer was then employed to scan an angular range of 10–80°. Steps of 0.02° were operated at 1 s per step.

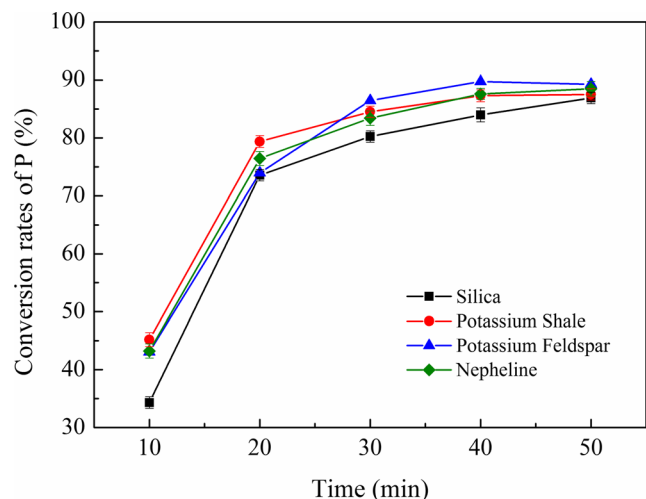
## 3 Results and Discussion

### 3.1 The Effect of Holding Time on the Conversion Rate of Phosphorus

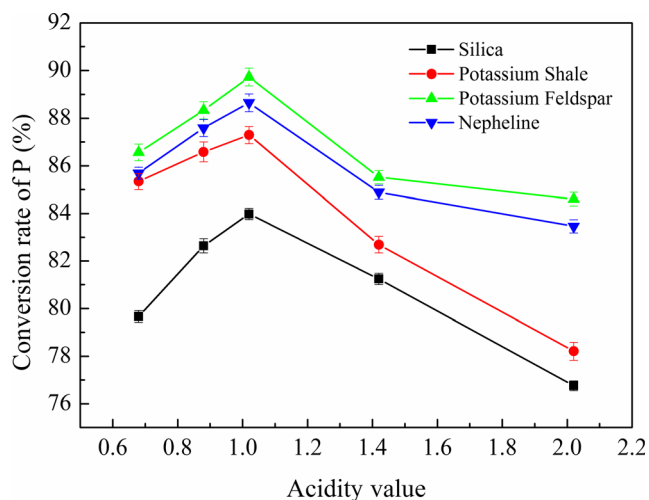
The effect of holding time on the conversion rate of phosphorus from phosphorus ore was measured at holding times of 10, 20, 30, and 40 min at a calcination temperature of 1400 °C, acidity value of 1.02, and coal surplus coefficient of 1.05. Figure 1 shows the effect of holding time on the conversion rate of phosphorus from phosphorus ore. As indicated, the holding time clearly influenced the conversion rate of phosphorus in phosphorus ore. For all samples, the conversion rate of phosphorus increased markedly over a 20-min holding time, after which the conversion rate increased less rapidly between 20 and 40 min holding time. After reaching a holding time of 40 min, there was no significant change in conversion efficiency with a change in holding temperature. This is likely because high temperature liquid substance was produced with increasing temperature, which will accelerate the transfer of matters and increase the conversion rate of phosphorus [32]. Therefore, considering the energy and economic costs and the efficiency of phosphorus, the optimal holding time was determined to be 40 min.

### 3.2 The Effect of Acidity Value on the Conversion Rate of Phosphorus

As shown in Fig. 2, the influence of the acidity value on the conversion rate of phosphorus from phosphorus ore was measured at acidity values of 0.68, 0.88, 1.02, 1.42, and 2.02 at 1400 °C calcination, a holding time of 40 min, and a coal surplus coefficient of 1.05. The acidity value holding time



**Fig. 1** Effect of holding time on the conversion rate of phosphorus from phosphorus ore (1400 °C calcination temperature, acidity value of 1.02 and coal surplus coefficient of 1.05)

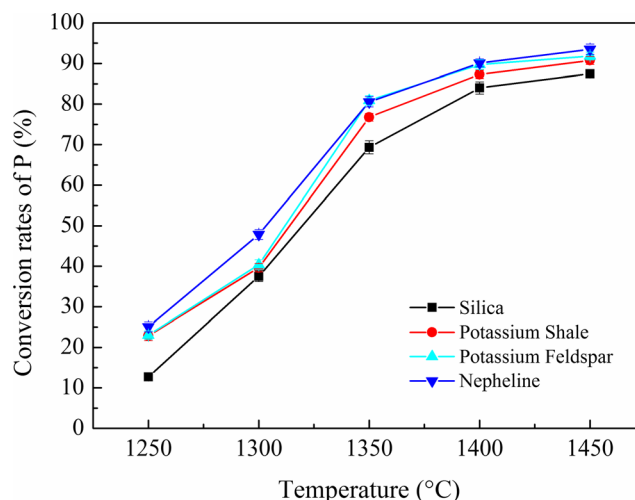


**Fig. 2** Effect of acidity value on the conversion rate of phosphorus from phosphorus ore (1400 °C calcination temperature, holding time of 40 min, coal surplus coefficient of 1.05)

clearly influenced the conversion rate of phosphorus in phosphorus ore. For all samples, the conversion rate of phosphorus increased with an increasing acidity value to 1.02. This phenomenon occurred due to increase the contact area between reactants after the acidity value was enhanced. But, after reaching an acidity value of 1.02, the conversion efficiency decreased with changes in acidity value, which mainly due to the phosphate concentration of system decline as the amount of fluxing agent increase [33]. Therefore, the optimal acidity value was determined to be 1.02 for all fluxing agents.

### 3.3 The Effect of Temperature on the Conversion Rate of Phosphorus

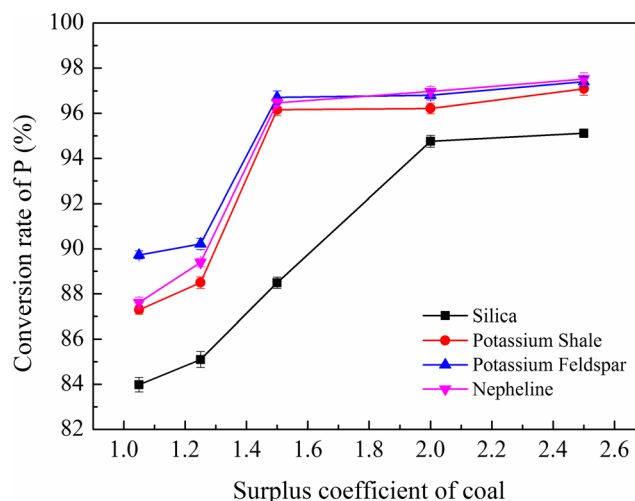
The influence of calcination temperature on the conversion rate of phosphorus was studied at calcination temperatures of 1250 °C, 1300 °C, 1350 °C, 1400 °C, and 1450 °C at a holding time of 40 min, acidity value of 1.02, and a coal surplus coefficient of 1.05. Figure 3 displays the effect of calcination temperature on the conversion rate of phosphorus from phosphorus ore, indicating that phosphorous conversion increased with increasing temperatures below 1400 °C. At temperatures at or above 1400 °C, the increase in the phosphorus conversion rate slowed as the temperature increased, potentially reaching a reactive equilibrium under this condition. This is because an increase in temperature intensifies both the reaction and diffusion rate of the oxidation-reduction reactions [34], while the final equilibrium concentrations achieved for a given set of reaction parameters depend on an equilibrium constant in the reactions. Therefore, the optimal calcination temperature was determined to be 1400 °C; a more detailed explanation is presented in Section 3.7.



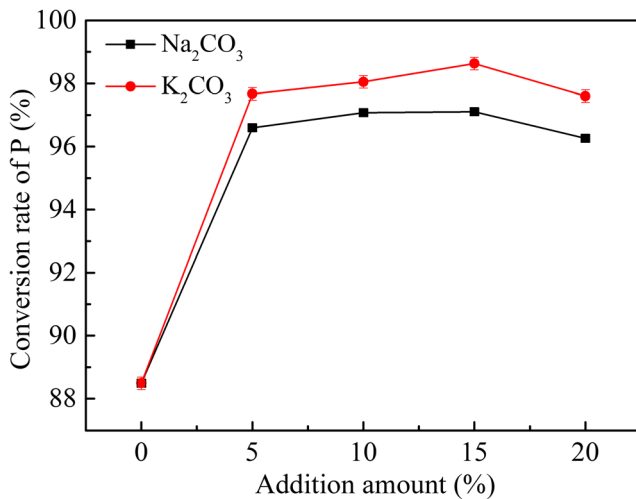
**Fig. 3** Effect of holding time on the conversion rate of phosphorus from phosphorus ore (holding time of 40 min, acidity value of 1.02, coal surplus coefficient of 1.05)

### 3.4 The Effect of Surplus Coefficient of Coal on the Conversion Rate of Phosphorus

The influence of the coal surplus coefficient on the conversion rate of phosphorus was investigated for coal surplus coefficients of 1.05, 1.25, 1.5, 2, and 2.5 at a holding time of 40 min, acidity value of 1.02 and 1400 °C calcination temperature. Figure 4 shows the effect of the coal surplus coefficient on the conversion rate of phosphorus from phosphorus ore. For the three fluxing agents (potassium shale, potassium feldspar, and nepheline), Fig. 4 shows that the conversions of phosphorus increased with increasing coal surplus coefficients below 1.5. At a coal surplus coefficient of 1.5 or higher, the phosphorus conversion rate was basically unchanged as the coal surplus coefficient increased. For silica as the fluxing agent,



**Fig. 4** Effect of coal surplus coefficient on the conversion rate of phosphorus from phosphorus ore (holding time of 40 min, acidity value of 1.02, 1400 °C calcination temperature)



**Fig. 5** Effect of alkali metal salts on the conversion rate of phosphorus from phosphorus ore (holding time of 40 min, acidity value of 1.02, calcination temperature of 1400 °C, coal surplus coefficient of 2)

the conversions of phosphorus appeared to increase with an increasing coal surplus coefficient below 2. At a coal surplus coefficient of 2 or higher, the phosphorus conversion rate was basically unchanged with an increasing coal surplus coefficient. Therefore, the optimal coal surplus coefficient was determined to be 1.5 for all fluxing agents in this study.

### 3.5 Flow Temperature Analyses of Samples

Superficially, analyses of images like those shown above indicate that silica as a traditional fluxing agent may not be best. Indeed, the conversions rate of phosphorus were greatly enhanced by the additions of potassium shale, potassium feldspar, and nepheline as fluxing agents; however, it is surprising that an alkali metal as an additive improved the carbothermic reduction of phosphorus ore based on the chemical composition of the raw materials presented in Table 1. This study confirmed the effects of alkali metals on the conversion rate of phosphorus. The influence of alkali carbonates (analytically pure Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub> as additives) on the conversion rate of phosphorus was investigated at a holding time of 40 min, acidity value of 1.02, coal surplus coefficient of 1.5, and

holding temperature of 1400 °C. As shown in Fig. 5, compared to the conversion rate without additives, the conversion rate of phosphorus alkali carbonates was significantly enhanced with the additions of Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub>. Figure 5 also demonstrates that the conversions of phosphorus apparently increased when increasing the additive proportion to below 15%. At an additive proportion of 15% or higher, the conversion efficiency decreased with a change in the additive amount. We also noted that the conversion rate of phosphorus by the different carbonates differed considerably. The catalytic activity of alkali (Na, K) carbonate followed the order K>Na. This could be due to the stronger alkalinity and the smaller ion potential of the alkali metals [35]. Therefore, the optimal additive proportion appears to be 15%. Considering coal as a carbonaceous reductant in the phosphorus production process, alkali carbonates reduce the apparent activation energy of the reaction, thereby further improving the carbothermic reduction of phosphorus ore [36–38].

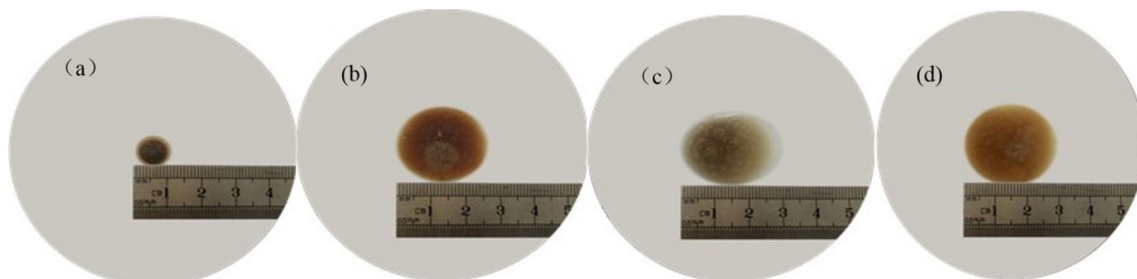
### 3.6 Slag Viscosity Analyses of Samples

The slag viscosity of samples was evaluated by the spread area after calcination at a high temperature. The residue was obtained at a holding time of 40 min, acidity value of 1.02, coal surplus coefficient of 1.5, and holding temperature of 1400 °C. The spread area of samples after calcination of the four different fluxing agents is shown in Fig. 6. Compared with silica as a fluxing agent, the samples with potassium shale, potassium feldspar, and nepheline as fluxing agents had a tremendous spread area, indicating a lower slag viscosity and higher conversion efficiency as described above.

As in the above case, the spread area of samples after calcination of two additives is shown in Fig. 7. The spread area of samples with additives is larger compared to those with silica as a fluxing agent, indicating higher conversion efficiency similar to earlier examples.

### 3.7 XRD Analyses of Samples

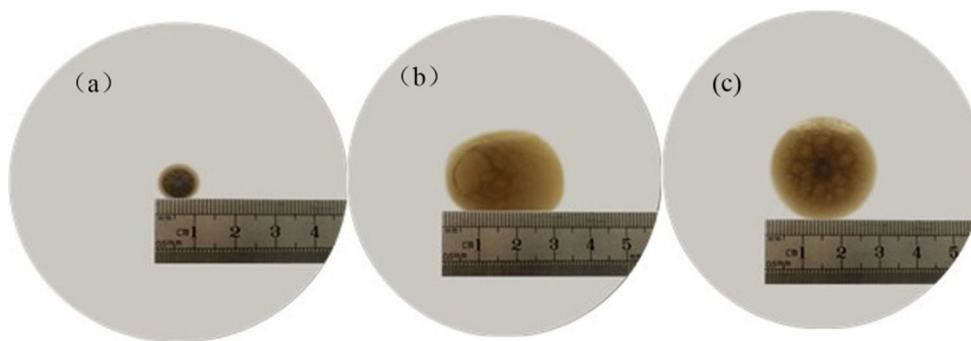
Silica and nepheline were selected as fluxing slag at different calcination temperatures and a holding time of 40 min, acidity



**Fig. 6** **a** phosphorus ore, coal, and silica as raw materials (1.1310 cm<sup>2</sup>); **b** phosphorus ore, coal, and potassium shale as raw materials (2.1382 cm<sup>2</sup>); **c** phosphorus ore, coal, and potassium feldspar as raw materials (2.4053 cm<sup>2</sup>); **d** phosphorus ore, coal, and nepheline as raw materials (2.5447 cm<sup>2</sup>)



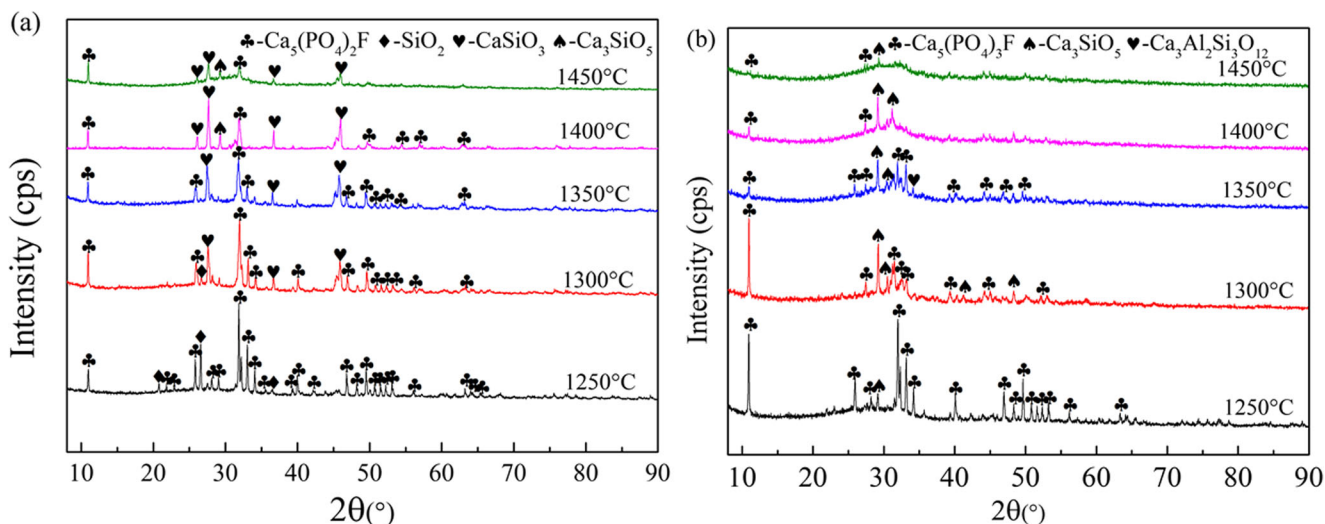
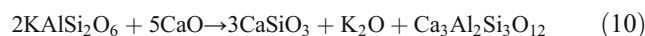
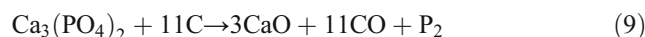
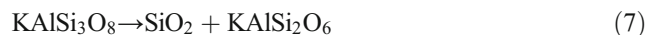
**Fig. 7** **a** phosphorus ore, coal, and silica as raw materials ( $2.4053\text{cm}^2$ ); **b** phosphorus ore, coal, silica, and  $\text{Na}_2\text{CO}_3$  of 10% as raw materials ( $2.4745\text{cm}^2$ ); **c** phosphorus ore, coal, silica, and  $\text{K}_2\text{CO}_3$  of 10% as raw materials ( $2.5447\text{cm}^2$ )



value of 1.02, and coal surplus coefficient of 1.5). The XRD of samples is shown in Fig. 8. The XRD samples with silica as a fluxing agent appear in Fig. 8a, which demonstrates that fluorapatite did not react obviously with a reducing agent or react less without a new feature peak at  $1250^\circ\text{C}$ . At a temperature of  $1300^\circ\text{C}$ , the carbothermic reduction of fluorapatite began, but there was lower conversion efficiency because excessive amounts of silicon oxide and a large amount of  $\text{CaSiO}_3$  exist in this system. The conversion rate of phosphorus clearly increased with an increasing temperature, and  $\text{Ca}_3\text{SiO}_5$  was obtained from the reaction between excessive amounts of calcium oxide and  $\text{CaSiO}_3$  at  $1400^\circ\text{C}$ ; however, there is no obvious liquid phase in this system. Notably, the increasing phosphorus conversion rate slowed at temperatures below  $1450^\circ\text{C}$  because most silicate salts form a low eutectic product. These results tend to support the phenomenon shown in Fig. 3. The specific reaction of the system can be expressed as follows:



In Fig. 8b, according to the experimental product derived from XRD, the vitreous is generated with the advent of the  $\text{Ca}_3\text{SiO}_5$  characteristic peak at  $1250^\circ\text{C}$ . Based on the appearance of the  $\text{Ca}_3\text{SiO}_5$  peaks, the carbothermic reduction of fluorapatite reached a high level; that is, nephrite, reaction products, and oxidized silicon in the raw materials blended together to form the low eutectic products resulting from the existence of amorphous form and the disappearance of the nephrite peak. The XRD analysis indicates that the alkali metal in nepheline was changed by calcium and generated more  $\text{Ca}_3\text{Al}_2\text{Si}_3\text{O}_{12}$ ; as such, the resulting phosphorus conversion rate slowed with increasing temperature. However, the carbothermic reduction of fluorapatite gradually ended as the temperature increased and a large amount of liquid was produced. The specific reaction of the system can be expressed as follows:



**Fig. 8** XRD of silica and nepheline as fluxing slag at different calcination temperatures (holding time of 40 min, acidity value of 1.02, coal surplus coefficient of 1.5); **a** silica as fluxing agents; **b** nepheline as fluxing agents

In contrast to the XRD analysis results, with fluorapatite as a main component of phosphorus ore, yellow phosphorus is generated through the carbothermic reduction of fluorapatite based on the mechanism of calcium oxide. The reduction products of fluorapatite and the fluxing agents generated high temperature stabilization and promoted the reduction of fluorapatite. Compared with silica as a fluxing agent, nepheline as a fluxing agent has a liquid phase at lower temperatures and provides conditions for diffusion that increase the phosphorus reduction rate.

## 4 Conclusion

In this study, compared with traditional fluxing agents, the phosphorus conversion rate from phosphorus ore was investigated with three different fluxing agents: potassium shale, potassium feldspar, and nepheline. Meanwhile, the addition of  $\text{Na}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$  to the traditional production process was also studied. Results indicated that the new fluxing agents and additive ( $\text{Na}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$ ) can improve phosphorus conversion efficiency under the same experimental conditions compared to silica as a fluxing agent. This is likely because the alkali metal in fluxing agents improve the slag viscosity at high temperatures and strengthen the carbothermic reduction process of phosphorus based on analysis of the spread area. The XRD analysis indicated that alkali metals in the mixtures have a liquid phase at lower temperatures, which provides conditions for diffusion and increases the phosphorus reduction rate. The optimal conditions identified here (a holding time of 40 min, acidity value of 1.02, coal surplus coefficient of 1.5, and calcination temperature of 1400 °C) should be used in future applications.

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