

## Article

# Experimental Study on Desulfurization and Removal of Alkali Behavior of BF Slag System in Low-Slag Ironmaking

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**Abstract:** The increased utilization of pellets in blast furnaces is one of the directions for low-carbon ironmaking. As a result, the low slag rate may affect the desulfurization of the hot metal and the removal of alkali in the blast furnace. Effective desulfurization and the removal of alkali in the low slag ironmaking process have become the focus of the steel industry. In this paper, the effects of slag quantity, temperature, reaction time and slag composition on the desulfurization and removal of alkali were studied using the slag-metal reaction method. It was found that the slag quantity had the same influence trend on the desulfurization and the removal of alkali. The greater the slag quantity, the more effective the desulfurization and the removal of alkali. The slag composition, temperature and reaction time had the opposite effect on the desulfurization and the removal of alkali. High temperature, long reaction time, high MgO concentration, high CaO/SiO<sub>2</sub> ratio and low Al<sub>2</sub>O<sub>3</sub> concentration increased the desulfurization of hot metal but reduced the removal rate of alkali from the blast furnace. Applications of the experimental results on high-proportion pellet blast furnace operation are discussed.

**Keywords:** blast furnace; high-proportion pellet; desulphurization; removal of alkali; ironmaking



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## 1. Introduction

The steel industry accounts for approximately 11% of the global carbon dioxide (CO<sub>2</sub>) emissions in the world [1]. Under the current policy and technology regime, the energy use and global greenhouse gas emissions of the steel industry are likely to continue increasing because of the increased demand for steel, particularly in developing countries [2]. A blast furnace (BF) is consistently the major technology for ironmaking in which sinter, pellet and lump are the iron-containing feeds. Increasing the proportion of high-grade pellets in the blast furnace is an effective method for carbon emission reduction [3,4]. Compared with sinter production, pellets can be produced with lower energy consumption and lower environmental pollution. In addition, a pellet contains a high concentration of iron which reduces the carbon consumption and CO<sub>2</sub> emission in a blast furnace due to the low slag rate [5–8]. Iron-containing and carbon-containing raw materials are composed of sulfur and alkalis that can affect the hot metal quality and the blast furnace operation. The hot metal from a blast furnace is mainly used for steelmaking where sulfur is a harmful element that affects the quality of the steel products. Alkali oxides are reduced to the metals by the hot carbon and vaporized to the upper of the blast furnace where they are oxidized and move down with the fresh feeds. Accumulation of the alkalis in a blast furnace can damage the coke and the refractories. Most of the sulfur and alkalis in a blast furnace can only be removed by the slag. The desulfurization of hot metal and the removal of alkali from the blast furnace are important roles of the slag. Reduced slag volume could affect the effective desulfurization and the removal of alkali in the blast furnace operation.

The desulfurization and the removal of alkali in the blast furnace have been investigated extensively [9–16]. However, the previous studies have certain limitations: (1) the

desulfurization and the removal of alkali experiments were conducted separately which is different from a real blast furnace operation; (2) the slag system studied was focused on high- $\text{Al}_2\text{O}_3$  [9–13] or low- $\text{MgO}$  [14,15]; (3) none of these studies considered the effect of the slag volume on the desulfurization and the removal of alkali [16,17]. As such, there is no direct theoretical guidance for high pellet and low slag BF operation.

In this study, the effects of the slag quantity, the temperature, the reaction time,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$  and  $\text{CaO/SiO}_2$  on the desulfurization and the removal of alkali were studied using the slag-metal reaction method. This study aims to provide systematic guidance to support high pellet ratio blast furnace operations.

## 2. Experimental Section

### 2.1. Preparation of Sulfur-Containing Iron

The sulfur-containing iron was prepared in a vacuum induction melting furnace. Five Kg pig iron from a blast furnace and a certain amount of  $\text{FeS}$  (analytically pure) were used as starting materials and placed in a magnesia-alumina spinel crucible. The furnace was flashed three times with high-purity argon (99.999%). The furnace was vacuumized to  $10^{-2}$  Pa before each flashing. The argon was filled to make the furnace slightly negative pressure and subsequently the power was turned on to start heating. The sample was cast after melting for 10 min. The S and C contents in the sulfur-containing iron from different locations were analyzed by a carbon-sulfur analyzer.

### 2.2. Preparation of Alkali-Containing Slag

The preparation of alkali-containing slag included three setups: sample preparation, high-temperature melting and sample examination.

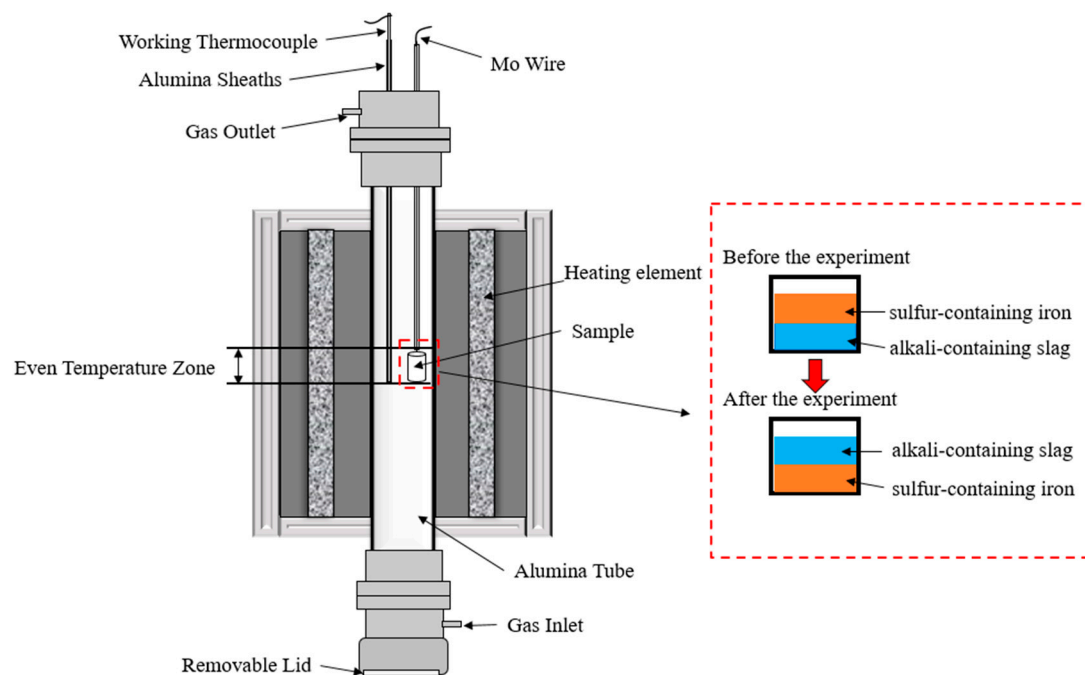
High-purity reagent powders of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{MgO}$ ,  $\text{CaCO}_3$ ,  $\text{Na}_2\text{CO}_3$  and  $\text{K}_2\text{CO}_3$  were used as starting materials. The oxide powders were mixed at the required ratios in an agate mortar to prepare the samples. Each mixture, approximately 15 g, was pelletized and placed in a graphite crucible.

The mixture in a graphite crucible was heated in a vertical tube furnace as shown in Figure 1. The detailed experiment procedure is given below. Firstly, the sample was introduced and kept at the bottom of the reaction tube and the furnace was properly sealed. Secondly, the reaction tube was flashed with argon gas for 30 min to remove the air. Thirdly, the sample was raised and kept in the hot zone of the furnace. The distance between the graphite crucible and the thermocouple was controlled within 5 mm to ensure the accurate temperature measurement. Fourthly, the furnace was heated to  $1500^\circ\text{C}$  to melt the sample. Fifthly, after 30 min at  $1500^\circ\text{C}$ , the Mo wire was pulled up and the sample was quenched directly into the cooling water. The rapid quenching of the silicate slags to room temperature converted the liquid phase to homogenous glass.

The quenched slag sample was dried on a hot plate and ground in an agate mortar to prepare the samples. The composition of the slag was analyzed by inductively coupled plasma spectrometry (ICP).

### 2.3. Experiments for Desulfurization and Removal of Alkali

The slag-metal reaction technique was employed to study the desulfurization and removal of alkali by the slag. The schematic diagram of the experimental set-up is shown in Figure 1. The prepared sulfur-containing iron and alkali-containing slag were put into a graphite crucible according to the required weight ratios. The procedure for slag-metal experiments was the same as the preparation of alkali-containing slag. After quenching, the sample was dried on a hot plate, and the slag and the hot metal were separated and analyzed by ICP and carbon-sulfur analysis, respectively. Following the discussions with the blast furnace operators, the slag to metal ratio, the temperature, the reaction time and the slag composition were the variables in the BF operation. The conditions of all slag-metal reaction experiments to cover the range of the industrial variables are summarized in Table 1.



**Figure 1.** The schematic diagram of the experimental method desulfurization and removal of alkali.

**Table 1.** Experimental conditions for slag-metal reaction experiments.

Exp No	Metal (g)	Slag (g)	Al <sub>2</sub> O <sub>3</sub> (%)	MgO (%)	CaO/SiO <sub>2</sub>	Temp (°C)	Time (min)
1	20	4.4	15.6	8.3	1.2	1500	30
2	20	4.0	15.6	8.3	1.2	1500	30
3	20	3.8	15.6	8.3	1.2	1500	30
4	20	3.6	15.6	8.3	1.2	1500	30
5	20	3.2	15.6	8.3	1.2	1500	30
6	20	3.0	15.6	8.3	1.2	1500	30
7	20	4.0	15.6	8.3	1.2	1475	30
8	20	4.0	15.6	8.3	1.2	1525	30
9	20	4.0	15.6	8.3	1.2	1500	15
10	20	4.0	15.6	8.3	1.2	1500	60
11	20	4.0	13.0	8.3	1.2	1500	30
12	20	4.0	18.0	8.3	1.2	1500	30
13	20	4.0	15.6	6.0	1.2	1500	30
14	20	4.0	15.6	11.0	1.2	1500	30
15	20	4.0	15.6	8.3	1.0	1500	30
16	20	4.0	15.6	8.3	1.1	1500	30
17	20	4.0	15.6	8.3	1.3	1500	30

In this study, the desulfurization is directly expressed as sulfur in hot metal. The removal rate of alkali is defined as  $R = M_1/M_2$ , where  $M_1$  is the weight of Na<sub>2</sub>O or K<sub>2</sub>O remaining in the slag after high temperature experiments,  $M_2$  is the weight of Na<sub>2</sub>O or K<sub>2</sub>O in original alkali-containing slag.

#### 2.4. Thermodynamic Calculation

The CALPHAD (CALculation of PHase Diagram) approach has been developed to simulate high-temperature processes [18,19]. FactSage is one of the most successful thermodynamic models in predicting [20] the equilibria of slag and metal systems. Experimental data are compared with the predictions of FactSage 8.2. “FactPS”, “FToxid” and “FTmisc” databases were selected under the “Equilib” module. The solution species selected in the calculations include “FToxid-SLAGA”, and “FTmisc-FeLQ”.

### 3. Results and Discussion

High-sulfur iron and high-alkali slag were used as the starting materials for high-temperature experiments of desulfurization and the removal of alkali. The compositions of the sulfur-containing iron and alkali-containing slag are shown in Tables 2 and 3, respectively. It can be seen from the tables that the prepared iron had uniform composition, with a C content of 4.25 wt% and an S content of 0.4 wt%. The slag contained 2.33 wt% alkali ( $K_2O + Na_2O$ ).

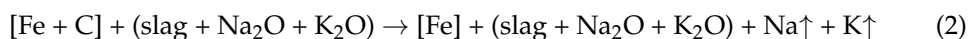
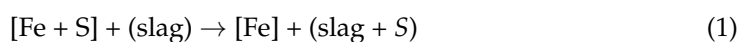
**Table 2.** C and S contents in the sulfur-containing iron analyzed by a carbon-sulfur analyzer.

Positions	C (wt%)	S (wt%)
Upper part of ingot	4.20	0.39
Lower part of ingot	4.27	0.40

**Table 3.** Composition of the alkali-containing slag analyzed by ICP.

$Na_2O$	$K_2O$	$SiO_2$	$CaO$	$MgO$	$Al_2O_3$
1.14 wt%	1.19 wt%	34.3 wt%	40.28 wt%	8.16 wt%	15.76 wt%

The metal and slag shown in Tables 1 and 3 were used as a base to evaluate the effects of the slag quantity, the temperature and the reaction time on the desulfurization and the removal of alkali. When the effect of slag composition was discussed, the alkali-containing slag was prepared separately to vary  $Al_2O_3$ ,  $MgO$  and  $CaO/SiO_2$  in the slag. The advantage of the research technique used in the present study is that better simulation conditions occurred in a BF hearth where the hot metal reacted with the slag for the desulfurization and the removal of alkali. The reactions can be expressed as

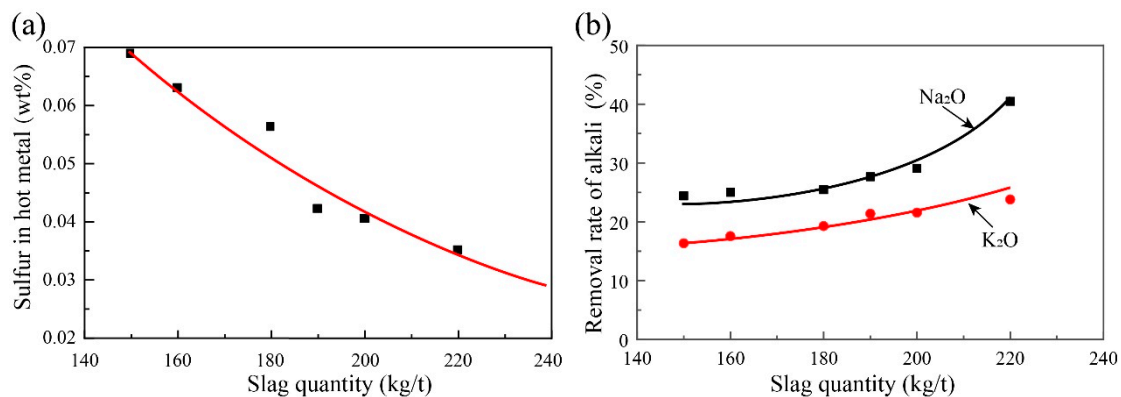


As a result of the metal-slag reaction, sulfur was transferred into the slag and part of the alkali in the slag was reduced by the carbon in the hot metal and vaporized before the metal and slag were tapped out of the furnace. The effects of different parameters on the desulfurization and the removal of alkali are discussed in the following sections.

#### 3.1. Effect of Slag Quantity

The slag quantity in a blast furnace decreased with an increased proportion of high-grade pellets. Figure 2 shows the effect of the slag quantity on the desulfurization and the removal of alkali at 1500 °C. It can be seen from Figure 2a that sulfur in the hot metal decreased with increasing slag quantity. The sulfur in the hot metal mainly reacted with  $Ca^{2+}$  and  $Mg^{2+}$  to enter the slag. With the increase of the slag-to-metal ratio, a greater amount of  $Ca^{2+}$  and  $Mg^{2+}$  in the slag were available to react with S in the iron. Under the experimental conditions, when the slag-to-metal ratio was more than 170 kg/ton, the content of the S in the iron was less than 0.05 wt% which is the limit of sulfur for steelmaking.

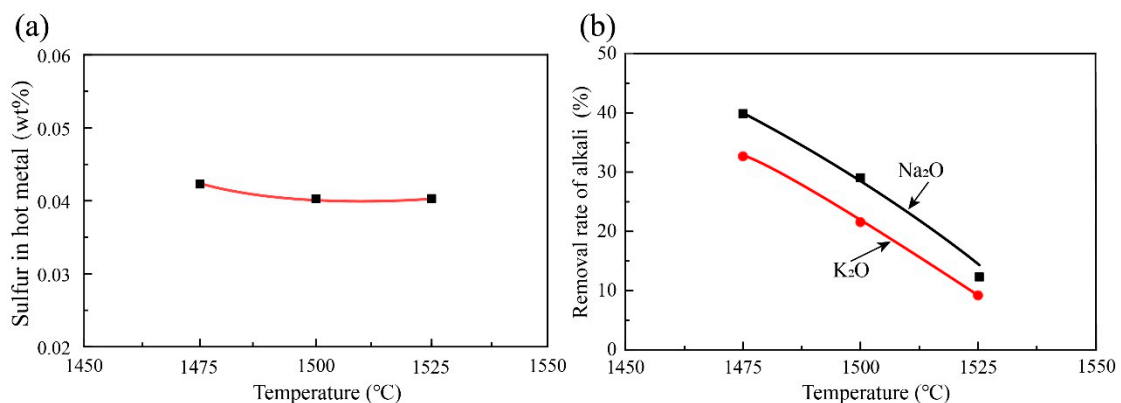
It can be seen from Figure 2b that with the decrease in slag-to-metal ratio, the contents of alkali in the slag also decreased. The effect of slag quantity seemed more sensitive to Na removal from the slag. On the other hand, it was easier to remove K from the slag because it is easier to reduce  $K_2O$  by carbon to form gaseous K. It was therefore more difficult to remove potassium from the blast furnace through slag. The recycling of potassium in the blast furnace was more severe than sodium. Both the desulfurization and the removal of alkali relied on slag. It was expected that low slag volume would influence the desulfurization of hot metal and the removal of alkali from a BF. Figure 2 gives a quantitative estimation of the effect for the industry to adjust the operating parameters accordingly.



**Figure 2.** The effect of the slag quantity on the desulfurization and the removal of alkali behavior. (a) Desulfurization; (b) the removal of alkali.

### 3.2. Effect of Temperature

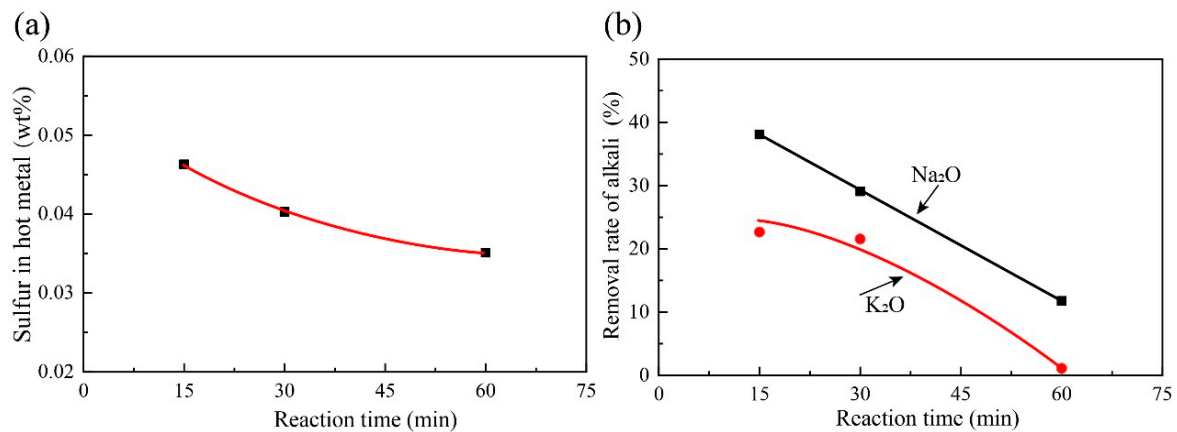
Figure 3 shows the effect of temperature on the desulfurization and the removal of alkali under a fixed slag-to-metal ratio of 200 kg/t. It can be seen from Figure 3a that the content of S in the hot metal decreased slightly when the temperature was increased from 1475 to 1525 °C which means that the desulfurization was not sensitive to temperature within the operating range. However, it can be seen from Figure 3b that the removal rate of alkali decreased significantly with increasing temperature. This can be explained by the fact that reaction (2) was much faster at a higher temperature. For a given BF, the slag and the metal temperature in the hearth was relatively stable which provided a hot metal product with stable sulfur. Removal of the alkali from the BF could be operated periodically. Figure 3 shows that a low temperature is beneficial to remove the alkali without a significant increase of sulfur in the hot metal.



**Figure 3.** Effect of temperature on the desulfurization and the removal of alkali at slag-to-metal ratio of 200 kg/t (a) Desulfurization; (b) the removal of alkali.

### 3.3. Effect of Reaction Time

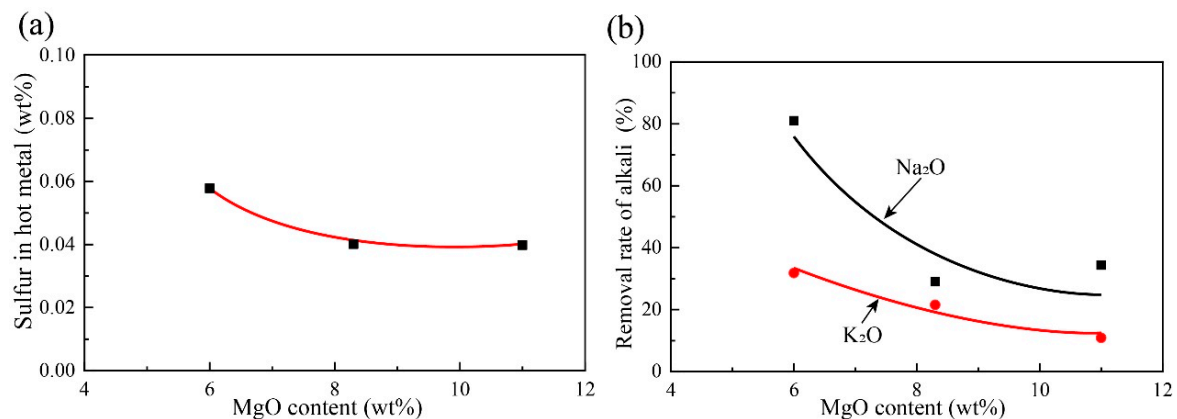
The effect of the reaction time on the desulfurization and the removal rate of alkali at a fixed slag quantity of 200 kg/t is shown in Figure 4. It can be seen that a longer reaction time between the slag and metal decreased the sulfur content in the hot metal and the alkali in the slag. Specifically, a better desulfurization was achieved by a longer reaction time. However, with the continuous reaction, the alkali in the slag was reduced and volatilized. It can be seen from the figure that the reaction between the slag and metal did not reach equilibrium in 60 min. The reaction time could be adjusted by the tapping period in a BF operation. A short tapping period of the slag and the metal is favorable for keeping the alkali in the slag.



**Figure 4.** Effect of reaction time on the desulfurization and the removal of alkali at slag-to-metal ratio of 200 kg/t. (a) Desulfurization; (b) the removal of alkali.

### 3.4. Effect of MgO

Figure 5 shows the effect of the MgO content on the desulfurization and the removal of alkali at a fixed slag quantity of 200 kg/t. The experimental results showed that high MgO can decrease the sulfur content in the hot metal. When the MgO content was higher than 7 wt%, S in hot metal was lower than 0.05%. As a basic ion,  $\text{Mg}^{2+}$  tends to react with the acidic sulfur in the hot metal. Conversely, high  $\text{Mg}^{2+}$  concentration in the slag increased the activities of the alkali such that they could be easily reduced and removed from the slag. Figure 5b shows that alkali in the slag decreased with increasing MgO concentration in the slag. MgO is usually added as a flux in the BF operation. A lower addition of MgO can decrease the slag volume and also increase the removal of alkali from the BF.

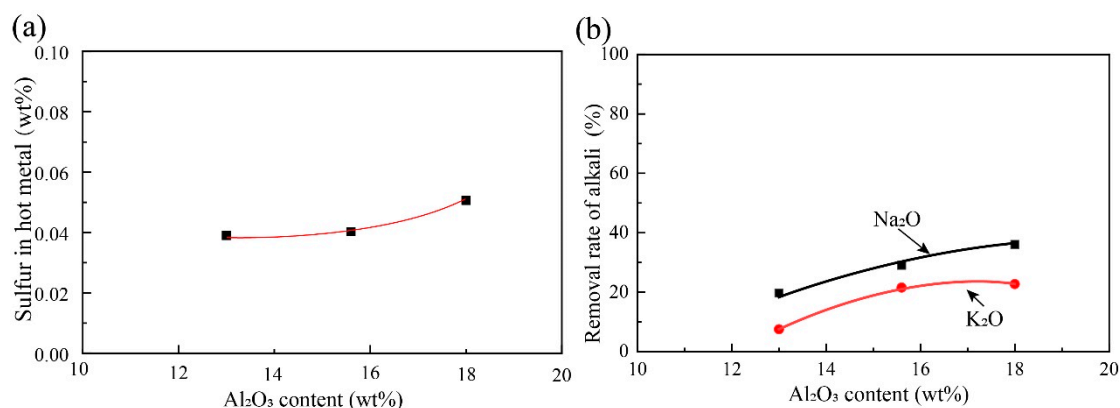


**Figure 5.** Effect of MgO content on the desulfurization and the removal of alkali at slag-to-metal ratio of 200 kg/t (a) Desulfurization; (b) the removal of alkali.

### 3.5. Effect of Al<sub>2</sub>O<sub>3</sub>

Figure 6 shows the effect of the Al<sub>2</sub>O<sub>3</sub> content in the slag on the desulfurization and the removal of alkali at a fixed slag quantity of 200 kg/t. Al<sub>2</sub>O<sub>3</sub> had an opposite influence compared to MgO on the desulfurization of the hot metal and the removal of alkali from the BF. High Al<sub>2</sub>O<sub>3</sub> in the slag increased the content of S in the hot metal and the alkali contents in the slag. This indicated that Al<sub>2</sub>O<sub>3</sub> behaved as a weak acid oxide in the BF slag which tended to keep K<sub>2</sub>O and Na<sub>2</sub>O in the slag. Al<sub>2</sub>O<sub>3</sub> usually comes from burden materials which increases the liquidus temperature and viscosity of the BF slag. The removal of alkali from the BF appeared to be the advantage of using high- Al<sub>2</sub>O<sub>3</sub> feeds.

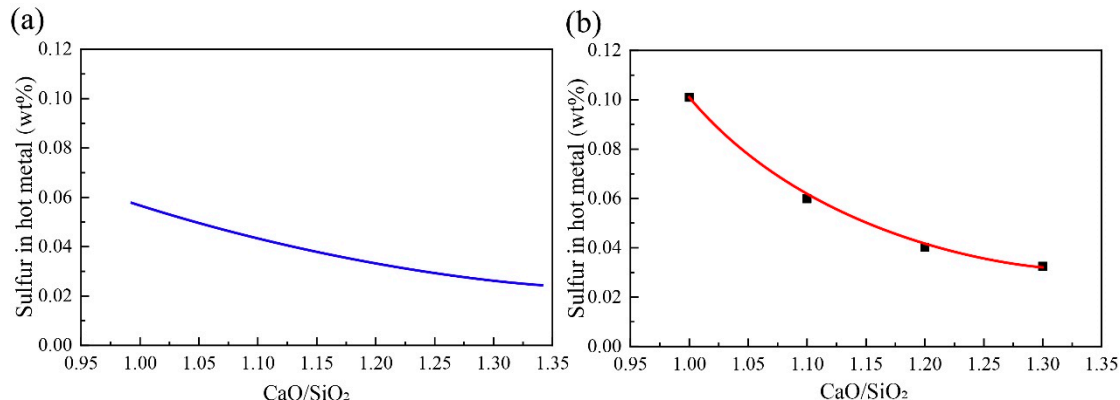




**Figure 6.** Effect of  $\text{Al}_2\text{O}_3$  content on the desulfurization and the removal of alkali at slag-to-metal ratio of 200 kg/t (a) Desulfurization; (b) the removal of alkali.

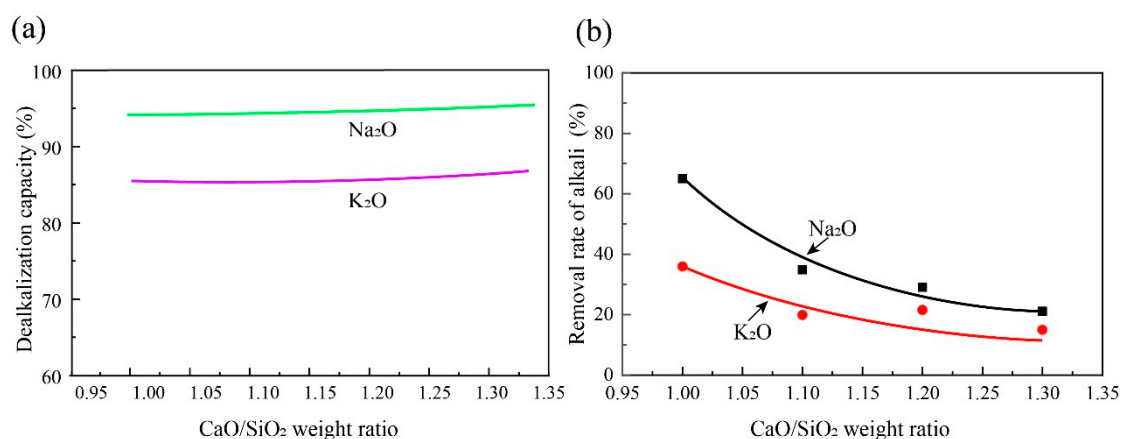
### 3.6. Effect of Binary Basicity

Figure 7 shows the effect of the  $\text{CaO}/\text{SiO}_2$  ratio on the desulfurization of the hot metal at a fixed slag quantity of 200 kg/t. Thermodynamic prediction by FactSage 8.2 is shown in Figure 7a for comparison. It can be seen that both the prediction and the experimental results showed the same trend that the sulfur in hot metal decreased with increasing  $\text{CaO}/\text{SiO}_2$  in the slag. However, the experimental results show a more significant effect of binary basicity on the desulfurization than the predictions. Figure 7b indicates that with the  $\text{CaO}/\text{SiO}_2$  ratio increased from 1.0 to 1.3, and the sulfur in the hot metal decreased from 0.1 to 0.038 wt%. However, the predicted sulfur in the hot metal only decreased from 0.06 to 0.025 wt% when the  $\text{CaO}/\text{SiO}_2$  ratio increased from 1.0 to 1.3.



**Figure 7.** The effect of the  $\text{CaO}/\text{SiO}_2$  on the desulfurization at slag-to-metal ratio of 200 kg/t, (a) FactSage calculation, (b) experimental results.

Figure 8 presents the effect of the  $\text{CaO}/\text{SiO}_2$  ratio on the removal rate of alkali from a blast furnace. The experimental results are compared with the predictions by FactSage 8.2. As can be seen from Figure 8b, the removal rate of alkali decreased significantly with increasing  $\text{CaO}/\text{SiO}_2$  ratio in the slag. This is understandable because  $\text{Ca}^{2+}$  is a strong basic ion which increased the activities of the alkali in the slag. As a result, the alkali could be easily reduced and removed from the slag. However, it can be seen from Figure 8a that the removal rate of alkali increased significantly with increasing  $\text{CaO}/\text{SiO}_2$  ratio in the slag according to the FactSage predictions. Although the FactSage software has been developed rapidly and successfully applied to the high-temperature process, there was still a difference between the experimental results and the predictions, indicating that the thermodynamic database needed to be further optimised.



**Figure 8.** Effect of the CaO/SiO<sub>2</sub> ratio on the removal of alkali at slag-to-metal ratio of 200 kg/t, (a) FactSage calculation, (b) experimental results.

### 3.7. Applications

The desulfurization of the hot metal and the removal of alkali from the blast furnace were related not only to the metal-slag reaction but also to the sulfur and alkali loads to the blast furnace. The experimental technique used in the present study closely simulated the blast furnace conditions by considering the reaction between the sulfur-containing metal and the alkali-containing slag. However, it was shown in the present study that the metal-slag reaction under the laboratory conditions did not reach equilibrium. The sulphide capacity or desulfurization ability was usually reported to evaluate the ability of a BF slag to absorb sulphur which was an equilibrium property [9–15]. Desulfurization ability is the ratio of the sulfur-in-slag to the sulfur-in-metal after the equilibrium reaction. It was reported that desulfurization ability of the synthetic slag was in the range of 15–30 at 1500 °C [9]. The desulfurization ability increased with increasing CaO/SiO<sub>2</sub> and MgO/Al<sub>2</sub>O<sub>3</sub>. Under the same conditions, the ratios of the sulfur-in-slag to the sulfur-in-metal were calculated to be 5–11 which was lower than that reported in the literature [9]. However, it can be seen from the above discussion that the same trends were observed in the present study for the effects of CaO/SiO<sub>2</sub> and MgO/Al<sub>2</sub>O<sub>3</sub> on desulfurization. Yang et al. [16] reported that Na<sub>2</sub>O or K<sub>2</sub>O concentration in the slag decreased with increasing temperature and CaO/SiO<sub>2</sub>. The same trends were reported in the present study but the rates of the decrement were different. These studies did not use the sulfur-containing iron and the alkali-containing slag simultaneously in the high-temperature experiments which is different from the blast furnace conditions. A large-scale reaction in the blast furnace is significantly different from that in the laboratory condition. It is more appropriate to refer to the trends reported in the present study in regard to blast furnace operation.

The effects of the variables on the desulfurization and the removal of alkali are summarized in Table 4. Only the slag quantity had the same effect on the desulfurization and the removal of alkali. Low slag quantity decreased both the desulfurization and the removal rate of alkali. Al<sub>2</sub>O<sub>3</sub> in the slag had a negative effect on the desulfurization and a positive effect on the removal of alkali. The other factors, including the temperature, the reaction time, MgO and CaO/SiO<sub>2</sub>, had a positive effect on the desulfurization and a negative effect on the removing of alkalis.

The results from the present study showed that it was impossible to increase the desulfurization of the hot metal and the removal of alkali with a simple variation of the operating parameters. Under the low slag operation of a blast furnace, good desulfurization could not be attained simultaneously with the high removal of alkali. The quality of the hot metal was a continuous control index. Desulfurization therefore required daily operation to keep the quality of the hot metal for steelmaking. A high temperature, long reaction time, and high MgO and CaO/SiO<sub>2</sub> were favorable for desulfurization. The damage of alkali to the blast furnace resulted from the accumulation by recirculation. The removal



of alkali from the blast furnace could be carried out at regular intervals. Within a certain period, low-temperature operation, short reaction time, and low MgO and CaO/SiO<sub>2</sub> slag could remove the alkali from the blast furnace. During the period of alkali removal, the sulfur content in the raw materials should be controlled to a low level to obtain satisfactory hot metal.

**Table 4.** Summary of the desulfurization and the removal of alkali by different parameters, + indicates positive effect, – indicates negative effect.

Variables	Desulfurization	Removal of Alkali
Slag quantity	+	+
Temperature	+	–
Reaction time	+	–
CaO/SiO <sub>2</sub>	+	–
Al <sub>2</sub> O <sub>3</sub>	–	+
MgO	+	–

#### 4. Conclusions

An improved slag-metal reaction technique was developed to investigate the desulfurization and the removal of alkali under iron blast furnace conditions. The experimental results showed that the desulfurization and the removal rate of alkali decreased with decreasing slag quantity. Al<sub>2</sub>O<sub>3</sub> in the slag was helpful for the removal of alkali but reduced desulfurization. The temperature, the reaction time, MgO and CaO/SiO<sub>2</sub> in the slag increased the desulfurization but influenced the removal of alkali. The change of a single operating parameter did not achieve high desulfurization and the removal rate of alkali simultaneously. It is proposed that the alkali should be removed from the blast furnace periodically with low loads of sulfur and alkali.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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