

## Article

# Behavior of Carbothermal Dephosphorization of Phosphorus-Containing Converter Slag and Its Resource Utilization

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**Abstract:** Phosphorus-containing converter slag is a common waste in the iron and steel industry, and has the characteristics of high generation and low secondary-utilization values; however, the high-phosphorus content in converter slag limits its ability to be recycled during the steelmaking process. In this study, the dephosphorization behavior of converter slag by carbothermal reduction was studied through experiments and thermodynamic calculations. The results showed that the gas product of the converter slag produced by carbothermal reduction was mainly  $P_2$ , and that part of  $P_2$  entered the iron phase to generate iron phosphate compounds. With the increase in Fe content, the amount of  $P_2$  also increased, which may provide a suitable new direction for the production of ferrophosphorus. Based on the carbothermal reduction theory, a new “circulating steelmaking process of converter steel slag gasification” was proposed and applied to Chengde Iron and Steel Group Co., Ltd. (Chengde, China). The industrial production practice showed that the process did not affect the dephosphorization effect of the next furnace, and that the average iron, steel, and slagging-material consumption per furnace was reduced by 4.74 kg and 608 kg, respectively.  $CO_2$  emissions were reduced by 4.86 kg, thus achieving the goals of energy saving, environmental protection, and efficient dephosphorization.

**Keywords:** converter slag; dephosphorization; carbothermal reduction;  $CO_2$  emission; ferrophosphorus



**Citation:** Tong, S.; Li, C.; Ai, L.; Wang, S.; Zhang, S. Behavior of Carbothermal Dephosphorization of Phosphorus-Containing Converter Slag and Its Resource Utilization. *Processes* **2023**, *11*, 1943. <https://doi.org/10.3390/pr11071943>

Academic Editor: Blaž Likozar

Received: 8 June 2023

Revised: 20 June 2023

Accepted: 22 June 2023

Published: 27 June 2023



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## 1. Introduction

The iron and steel industry is a high-consumption and high-emission industry, in which the main emissions are gas wastes (such as  $CO_2$ ,  $SO_2$ , and  $NO_x$ ) and solid wastes (such as converter slag, granulated slag, dust and sludge) [1]. At present, the output of solid waste in China’s iron and steel industry accounts for about 15% of the national industrial solid waste output, and the accumulated storage of converter slag in China is nearly one billion tons [2]. The utilization rate of converter slag is less than 30% because of its high alkalinity, high free calcium oxide content and its many metastable phases. A large amount of converter slag is left idle, which not only consumes land resources, but also pollutes the environment and harms human health [3]. Therefore, how to realize the recycling of converter slag in the furnace has become an important research direction. Bao et al. [4] studied the leaching law of phosphorus in different acids from high-phosphorus converter slag using a selective leaching process. Wang et al. [5] studied the kinetics of microwave carbothermal dephosphorization of converter slag, indicating that the carbothermal reduction reaction is a second-order reaction, and that the limiting link may be an interfacial chemical reaction. Zhang et al. [6], based on a micro-sintering experiment, analyzed the influence of alkalinity, temperature, and carbon content on the dephosphorization rate of high-phosphorus steel-slag gasification. Ai et al. [7] studied the carbothermal reduction behavior of converter steel slag via microwave heating and found that the maximum reduction dephosphorization rate of steel slag can reach 91.5% at low temperatures. Zhao’s

research indicated that when reducing converter slag using a carbothermic method, coke powder was used as both a reducing agent and heating agent, and that the exothermic reaction between coke powder and oxygen could ensure the dephosphorization reaction at a higher temperature [8].

China's crude steel output is huge, and high-quality iron ore is constantly consumed, which has led to greater interest in high-phosphorus iron ore with large reserves. However, the phosphorus content in high-phosphorus iron ore ranges from 0.3% to 1.8%, and the ore structure is compact, which will inevitably increase the difficulty in treating high-phosphorus converter slag during smelting. If the phosphorus in high-phosphorus converter slag can be separated and utilized, and the remaining residue used for internal circulation in the steelmaking process, it will reduce environmental pressures and smelting costs [9,10].

At present, there are many basic research studies on the carbothermal reduction process and reaction thermodynamic conditions of converter slag, that have mainly been conducted in the laboratory. However, there are few reports on the dephosphorization behavior of converter slag by carbothermal reduction and the actual smelting effect of furnace recovery. This experiment mainly studied the dephosphorization behavior of converter slag by carbothermal reduction, which provided a new idea for the resource utilization of phosphorus-containing converter slag. The recovery and utilization of phosphorus resources after carbothermal reduction of phosphorus-containing converter slag were investigated.

Based on the carbothermal reduction theory, our team put forward the process of "circulating steelmaking by gasification dephosphorization of converter slag", which was applied by the Chengde Iron and Steel Group Co., Ltd. (Cheng steel) and achieved considerable economic indicators. Gasification dephosphorization is a novel method that could make it easier to reuse convert slag. In this process, carbon is added to the converter before slag splashing. The slag and carbon are then fully mixed and reacted under dynamic conditions during the slag-splashing process. The reduction products of  $P_2O_5$  in converter slag are removed from the slag as gas, which makes it possible to reuse the treated slag in the next heat. This process provides a better direction for the comprehensive utilization of converter slag from carbothermal reduction.

## 2. Materials and Methods

### 2.1. Materials

The components of converter slag and coke are shown in Tables 1 and 2, respectively. The converter slag and coke were crushed to less than 0.063 mm by an electromagnetic crusher to obtain a larger specific surface area, and 100 g of converter slag was thoroughly mixed with coke powder with 2 carbon equivalent for each heat (100 g of converter slag for every 10.26 g of coke powder). The unit carbon equivalent was defined as the carbon content used for the total reduction of FeO,  $P_2O_5$ , and MnO in the slag.

**Table 1.** Chemical composition of converter slag (wt.%).

FeO	CaO	SiO <sub>2</sub>	MgO	MnO	P <sub>2</sub> O <sub>5</sub>	Basicity (R)
15.82	42.85	12.55	7.15	3.35	3.08	3.41

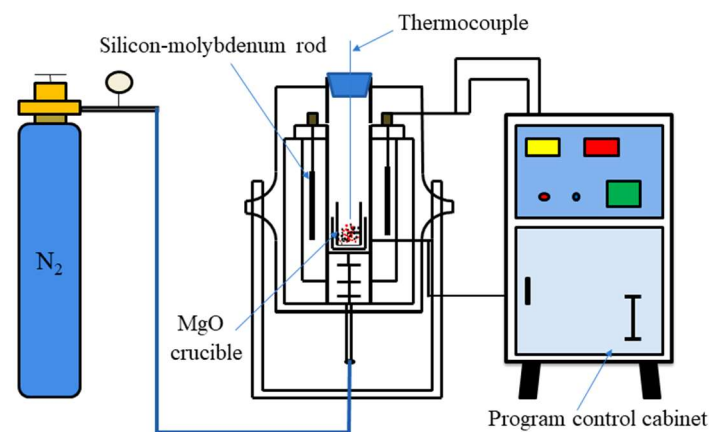
**Table 2.** Chemical composition of coke (wt.%).

Fixed Carbon	CaO	SiO <sub>2</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	S
86.55	1.37	6.42	0.33	0.36	0.98

### 2.2. Methods

The converter-slag-dephosphorization experiment by carbothermal reduction was conducted in a high-temperature heating furnace (Figure 1) comprising a gas control system

and temperature control system, and the temperature was measured with a dual platinum rhodium thermocouple. Before the experiment, the mixture was placed in an MgO crucible, covered with a graphite crucible, and placed in the heating furnace, which was heated to 1813 K and kept warm for 1 h. During the experiments, N<sub>2</sub> was circulated in the furnace with a flow rate of 0.7 L/min, and the gas flow rate was detected by a precise rotameter; cooling circulating water was continuously used. Finally, the slag was cooled to room temperature with the furnace temperature, and the chemical composition was determined by X-ray fluorescence (XRF). In addition, the component content of the slag was adjusted by analyzing pure reagents, and univariate adjustment was adopted. In this experiment, only the basicity and FeO content of the final slag were changed.



**Figure 1.** Carbon thermal reduction experimental apparatus.

The dephosphorization rate of the converter slag was calculated by comparing the mass fractions of P<sub>2</sub>O<sub>5</sub> in the slag before and after the reaction, as shown in Equation (1). The chemical composition of the carbothermal reduction slag is listed in Table 3. It can be seen from Table 3 that the average dephosphorization rate under the experimental conditions was 32.57%.

$$\varphi = \frac{w_0 - wt}{w_0} \times 100\% \quad (1)$$

where  $\varphi$  is the dephosphorization rate of converter slag,  $w_0$  is the initial P<sub>2</sub>O<sub>5</sub> mass fraction of converter slag, and  $wt$  is the P<sub>2</sub>O<sub>5</sub> mass fraction of converter slag reduced by the coke.

**Table 3.** Chemical composition of carbothermal reduction slag (wt.%).

Reduction Condition	TFe	CaO	SiO <sub>2</sub>	MgO	MnO	P <sub>2</sub> O <sub>5</sub>	Dephosphorization Rate/%
Final slag	19.69	44.22	16.32	8.30	3.09	2.22	27.92
Final slag of R = 2.81	20.43	42.05	19.03	7.38	3.47	1.90	38.31
Final slag of w(FeO) = 24%	23.66	42.01	15.96	7.17	3.27	2.11	31.49

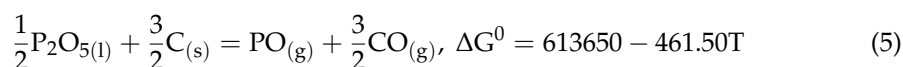
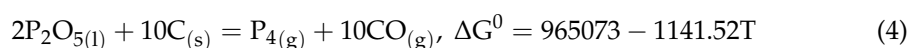
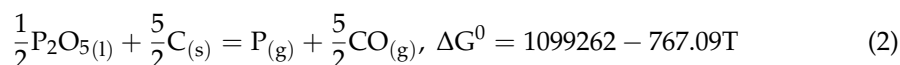
### 2.3. Characterization Methods

The compositions of the slag and coke were detected by X-ray fluorescence (XRF). The microscopic morphology of the slag was examined using a scanning electron microscope (SEM), and the element content was analyzed by energy-dispersive X-ray (EDS) spectroscopy attached to the SEM. The composition of molten iron was determined by inductively coupled plasma (ICP).

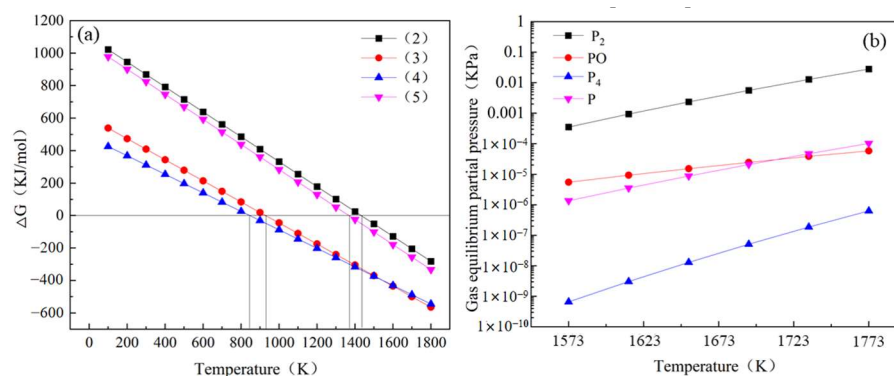
### 3. Experimental Results and Discussion

#### 3.1. Thermodynamic Analysis

The P in converter slag mainly consists of  $P_2O_5$ , which has been confirmed by most scholars. The gas products containing the P element formed from the reduction of pure  $P_2O_5$  under standard conditions are given by Equations (2)–(5) [11–15].



Equations (2)–(5) show that four products, P,  $P_2$ ,  $P_4$ , and PO are formed. According to the above Eqs, the relationships between the standard Gibbs free energy ( $\Delta G^0$ ) and temperature were established, as shown in Figure 2a. It can be seen from Figure 2a that with the increase of temperature, the standard Gibbs free energy decreased. When the reaction was  $\Delta G^0 = 0$ , the corresponding temperatures of Equations (2)–(5) were 1433 K, 930 K, 845 K, and 1330 K, respectively. Thus, as long as the temperature was higher than 1433 K, the carbothermal reaction could occur, and also at 930 K, 845 K, and 1330 K, respectively, the  $\Delta G^0 < 0$ .



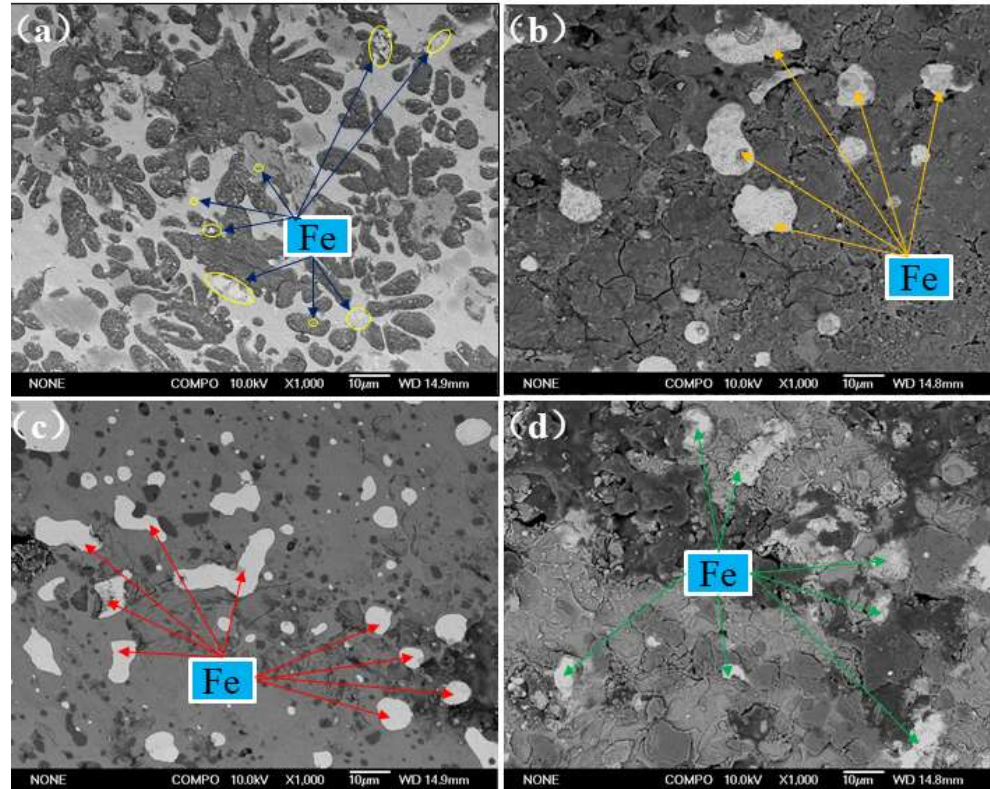
**Figure 2.** Thermodynamic analysis: (a) the relationship between Gibbs of carbothermal reduction and temperature; and (b) the relationship between temperature and partial pressure of gas products.

In order to further determine the dephosphorization products, the equilibrium partial pressure of the gas products of the carbothermal reaction was calculated by Factsage, as shown in Figure 2b. It can be seen from Figure 2b that, with the increase in temperature, the equilibrium partial pressure of the gas products increased, and it was found that the equilibrium partial pressure of  $P_2$  gas was the largest. Therefore, it can be speculated that the main phosphorus gas product of the carbothermal reduction converter slag was  $P_2$ . This result was consistent with other scholars' research [16–18].

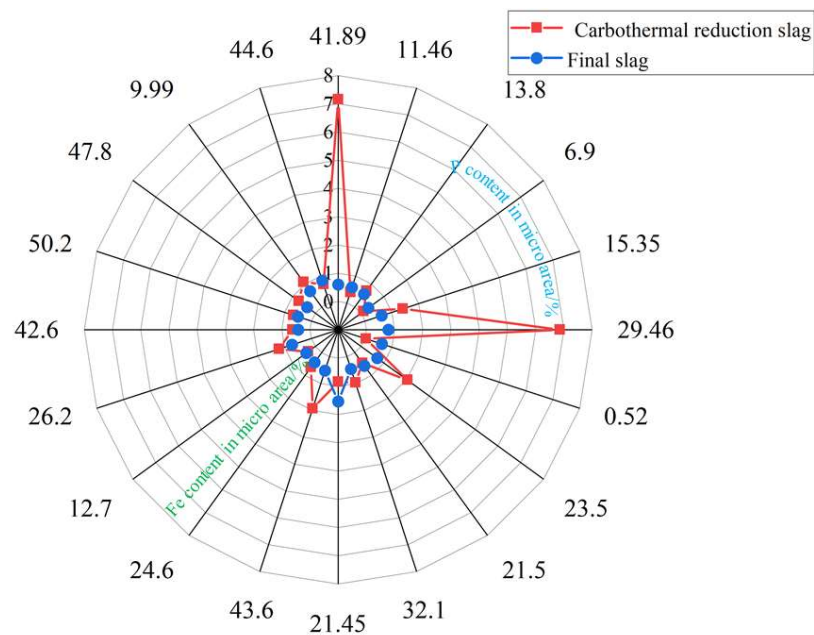
#### 3.2. Dephosphorization Behavior

Because it was impossible to capture the location of P during carbothermal reduction at high temperatures, the Fe phase micro-area of the slag before and after the reduction was analyzed. Figure 3 shows the microstructure before and after the carbothermal reduction of the converter slag. The SEM showed that the Fe phase of the final slag consisted of small particles, while the Fe phase in the carbothermal reduction slag consisted of large-diameter particles or strips. The EDS indicated that the Fe phase in the carbothermal reduction slag contained P element, as shown in Figure 4. It can be seen from Figure 4 that the Fe content

in the Fe phase micro-area of the final slag was between 10% and 50%, and the P content was between 0.4% and 0.8%, with few fluctuations. However, the content of P in the Fe phase micro-area of carbothermal reduction slag was between 0.15% and 7.5%, which was generally higher than that in the Fe phase micro-area of the final slag.



**Figure 3.** SEM images of carbothermally reduced slag in different conditions: (a) final slag; (b) the final slag was reduced; (c) final slag of  $w(\text{FeO}) = 24\%$  was reduced; and (d) final slag of  $R = 2.81$  was reduced.



**Figure 4.** The relationship between P and Fe content in micro-area.

On the whole, there was no obvious linear relationship between the Fe content and P content in the carbothermic reduction slag, but when the Fe content was less than 30%, the P content increased with the increase in Fe content. When the Fe content was greater than 30%, with an increase in Fe content, the fluctuations of the P content were few, but there still were large P micro-regions. Therefore, in the reduction process, part of the gas product  $P_2$  will persist in the metallic iron, and with the increase in Fe content generated by the reduction of FeO by coke, the content of P in metallic iron will also increase.

#### 4. Resource Utilization of Converter Slag after Carbothermal Dephosphorization

##### 4.1. Application of Carbon Thermal Reduction Slag Circulation Steelmaking

Our team has conducted a considerable amount of research on the carbothermic reduction of slag [19–24], and, based on the efficient resource utilization of the carbothermic reduction of converter slag, a new process of “circulating steelmaking by gasification dephosphorization of converter slag” was put forward. Coke was added during the slag-splashing and furnace-protection stages of the converter to realize gasification dephosphorization, and the slag after gasification dephosphorization was reserved for recycling in subsequent furnaces, which could then be used as hot slag to realize low-temperature dephosphorization.

##### 4.1.1. Industrial Test Conditions

The industrial test of this process was carried out in Cheng steel’s 100 t converter, and the smelting steel grade was HRB400E. The enterprise adopts a semi-steel process, and the composition of the molten steel is shown in Table 4. Taking the coke powder produced by Cheng steel as the reducing agent, part of the slag was poured out after tapping, and the reducing agent was added all at once from a high bunker before the slag-splashing operation. Then, the gun was lowered, and nitrogen was blown to carry out the slag-splashing operation.

**Table 4.** Composition of molten steel (wt.%).

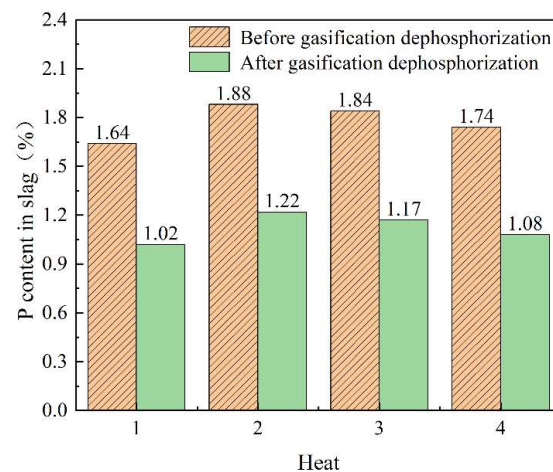
Type	C	P	S	V
Semi-molten steel	3.5~4.0	0.17~0.20	0.025~0.06	<0.04
Terminal molten steel	0.04~0.11	0.017~0.032	0.022~0.045	vestige

##### 4.1.2. Changes of Phosphorus Content in Slag

Figure 5 shows the change in P content of the slag before and after gasification dephosphorization. It can be seen in Figure 5 that the P content in the slag decreased after carbothermal reduction; the average P content in the slag before and after gasification dephosphorization was 1.78% and 1.12%, respectively, and the average gasification dephosphorization rate was about 37%. Because the P in the converter slag mainly resided in  $C_2S$  in the form of  $P_2O_5$ , the partial  $P_2O_5$  in converter slag could be effectively removed by gasification dephosphorization.

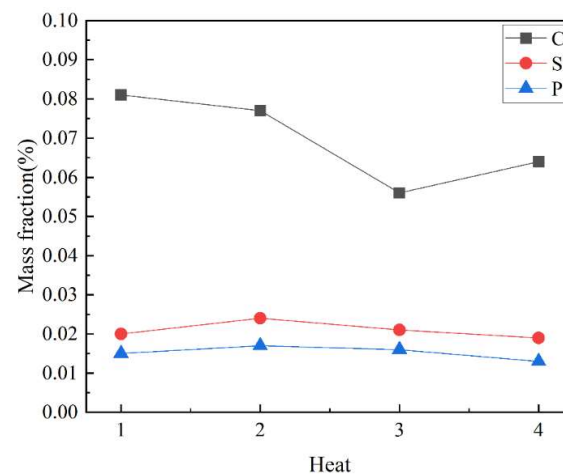
##### 4.1.3. Composition of Terminal Molten Steel

The phosphorus enrichment of converter slag limits its recycling in the furnace, and the high-phosphorus content of converter slag will affect the dephosphorization effect of the subsequent furnace. After gasification-dephosphorization treatment, the phosphorus capacity of converter slag is increased, and it can thus continue to participate in dephosphorization processes. In order to better study the influence of the recycling of gasification-dephosphorization slag on subsequent dephosphorization processes, the composition changes of the molten steel at the end of each smelting procedure in the test were analyzed.



**Figure 5.** Content of P in slag before and after gasification dephosphorization.

The composition of the terminal molten steel in the test furnace is shown in Figure 6. It can be seen that the composition of the terminal molten steel was relatively stable. All the elements met the requirements of the steel grade; the P content was between 0.009% and 0.013%; the S content was between 0.020% and 0.026%; and the C content was between 0.053% and 0.082%. It was proven that the recycling of gasification-dephosphorization slag will not affect the dephosphorization effect of subsequent heats, and the gasification-dephosphorization slag could thus participate in the slagging of the next heat with the appropriate slag alkalinity and FeO content to ensure the dephosphorization rate.

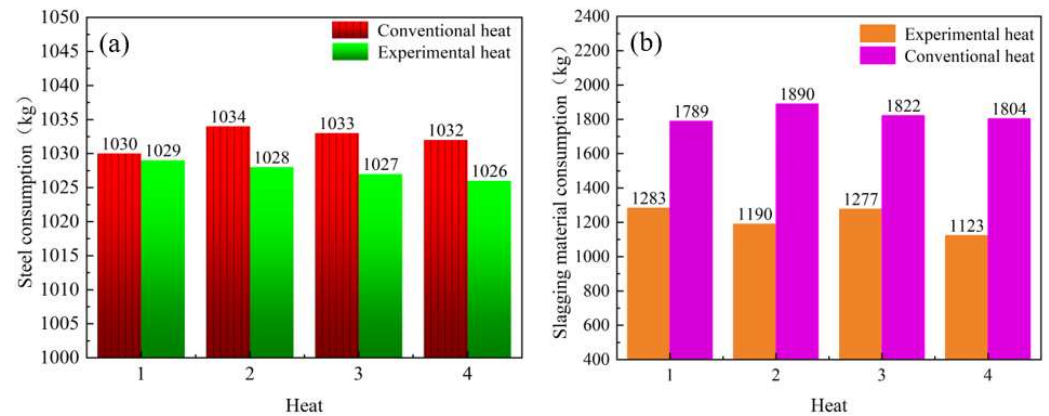


**Figure 6.** Composition of terminal molten steel after gasification dephosphorization.

#### 4.1.4. Comparison of Economic Indicators

Figure 7a,b shows a comparison between iron and steel consumption and slagging material consumption at conventional and experimental heats, respectively. Figure 7a shows that the circulating heat of the gasification-dephosphorization slag could effectively reduce steel consumption. The average iron and steel consumption in the conventional heat was 1032.25 kg, and that in the test heat was 1027.5 kg, with an average reduction of 4.74 kg. The slag contained a lot of metallic iron and iron oxide. A carbon-reducing agent was added at the slag-splashing stage to reduce some of the FeO in the slag to Fe at a high temperature, which reduced the iron loss and improved the steelmaking level. The recycling of gasification-dephosphorization slag can reduce slagging-material consumption. The average slagging-material consumption for the conventional furnace was 1826.25 kg, and that for the experimental furnace was 1218.25 kg, which was 608 kg lower than that for the conventional furnace, as shown in Figure 7b. Because the gasification-

dephosphorization slag had the characteristics of “high CaO and low FeO” [25], the amount of lime could be appropriately reduced when it stayed in the furnace to participate in subsequent dephosphorization procedures.



**Figure 7.** Comparison of economic indicators after gasification dephosphorization: (a) iron and steel consumption; and (b) slagging-material consumption.

The recycling of converter slag can not only reduce the generation of new slag, but can also reduce the consumption of lime because of the high content of CaO in the slag, and at the same time increase the steel output of the subsequent smelting process using the same amounts of iron and steel.

The production practice showed that the gasification-dephosphorization slag had a certain dephosphorization ability, and could directly participate in the early dephosphorization of the next heat if it stayed in the furnace. Gasification-dephosphorization slag itself belonged to the categories of “hot slag” and “cooked slag”, which have a micro-phase suitable for phosphorus occurrence without undergoing the melting and slagging stages. It could be quickly dephosphorized by making full use of the low-temperature conditions during the early stages. However, the gasification-dephosphorization slag was still phosphorus-containing slag, and its dephosphorization ability was limited. Therefore, the gasification-dephosphorization slag cannot be used as the leading dephosphorization agent, and needs to be mixed with fluxes such as lime and dolomite to achieve a better dephosphorization effect.

#### 4.2. Assessment and Analysis of Carbon Emission Reduction

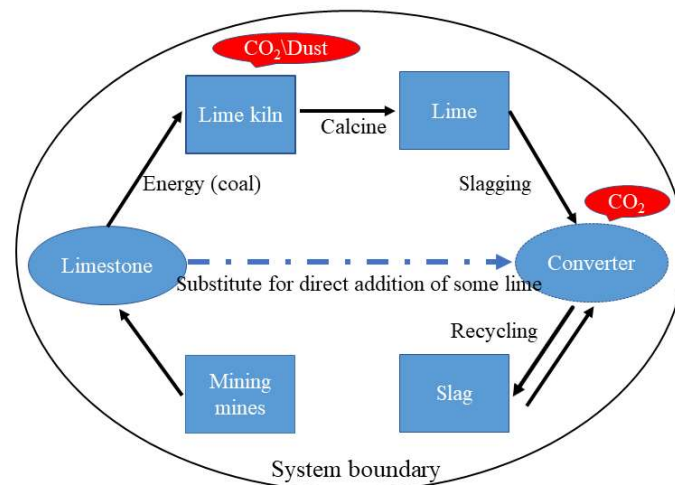
Under the background of the “double carbon” target strategy, carbon emissions reductions in iron and steel metallurgical processes play a leading role, so putting controlled lime into use is conducive to reducing CO<sub>2</sub> emissions. The lifecycle assessment method is used to quantify carbon emissions and carbon footprint data [26,27]. Taking the boundary of the traditional lime-slagging-mode system as the research object, the starting point of limestone mining and the ending point of converter slag are presented in Figure 8.

According to the emission factor method provided by China Energy Conversion Standard IPCC [28,29], CO<sub>2</sub> emissions are represented by the calorific value of unit standard coal. The calorific value of 1 kg standard coal was thus 29.308 MJ, which was equivalent to 2.5 kg of CO<sub>2</sub> emissions, and its calculation formula is shown in Equation (6). The lime kiln parameters and CO<sub>2</sub> emission factors are listed in Table 5 [30].

$$E_{\text{CO}_2} = \sum_i^n E_i \times F_i \quad (6)$$

where  $E_{\text{CO}_2}$  is CO<sub>2</sub> emitted per unit product, kg;  $E_i$  is the emission factor of fuel, kg/kg;  $F_i$  is the consumption of the  $i$  fuel, kg.





**Figure 8.** System boundary used in calculation.

**Table 5.** Parameters of lime kiln and CO<sub>2</sub> emission factor used in calculation.

Energy Type	Ton of Lime Consumes Energy	CO <sub>2</sub> Emission Factor
Anthracite	90 kg	2.68 kg/kg
Converter gas	770 m <sup>3</sup>	0.79 kg/m <sup>3</sup>
Electric power	60 kW·h	1.03 (kg/kW·h)

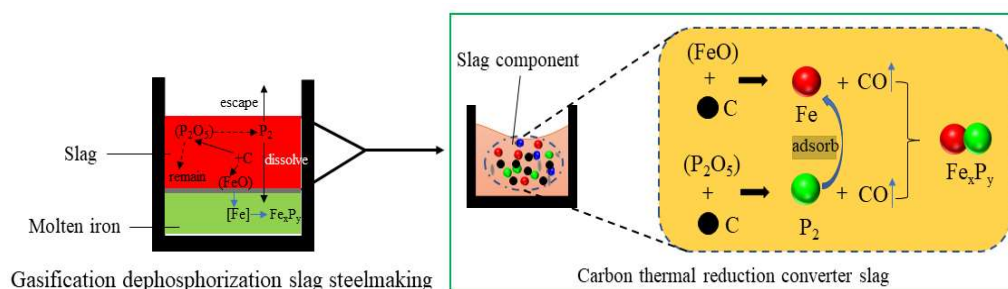
The lime kiln mainly used anthracite and converter gas as fuels, and the converter gas can be produced for self-use, so its energy consumption was not considered. The energy consumption and CO<sub>2</sub> emission of other links are shown in Table 6. It can be seen from Table 6 that CO<sub>2</sub> emissions were the highest during the process of limestone decomposition and lime calcification, followed by lime slagging. Therefore, from the start point to the end point of the system boundary, the estimated emissions per ton of lime was about 1034 kg CO<sub>2</sub>. Taking Cheng steel's 100 t converter as an example, the average slagging material was reduced by 4.74 kg, and CO<sub>2</sub> emissions were reduced by about 4.86 kg per heat.

**Table 6.** Calculation results of carbon emissions in production links.

Emission Link	Standard Coal Quantity Converted into Energy Consumption per Ton of Lime/Kg	CO <sub>2</sub> Emission/Kg
Electricity consumption (production and anthracite supply)	24.88	62.2
Limestone decomposition and lime calcination	339.8	849.5
Lime slagging	48.75	121.88

#### 4.3. Thoughts on Recovery and Utilization of Phosphorus Resources

The above research demonstrates that it is practical to reduce slag using the carbothermic method in the furnace. Under the conditions of high-temperature carbothermal reduction, the P<sub>2</sub>O<sub>5</sub> in slag can be effectively gasified. A part of the P<sub>2</sub> persists in the molten metal to generate Fe<sub>x</sub>P<sub>y</sub>, while the other part escapes in the form of gas. Figure 9 shows the schematic diagram of the reaction process of converter-slag gasification-dephosphorization steelmaking and the flow direction of the phosphorus.



**Figure 9.** Schematic diagram of the reaction process of converter-slag gasification-dephosphorization steelmaking.

For the recovery and resource utilization of phosphorus resources after the carbothermal reduction of phosphorus-containing slag, the appropriate carbothermal reduction conditions and the best gasification-dephosphorization parameters should be considered first to ensure the full reduction of  $P_2O_5$  in converter slag. Second, the efficient recovery and utilization of reduced phosphorus resources should be considered. We can consider the following two aspects [31] by:

1. Making more  $P_2$  overflow and collecting it, and then carrying out an oxidation–hydration reaction to prepare the phosphoric acid for use as phosphate fertilizer, thereby making it less difficult for entry into the industry. This idea can provide a new direction for a steel chemical co-production project, which is an innovative development model of the iron and steel industry combined with the chemical industry. At present, there have been successful cases reported in China, such as the project of Shandong Shiheng Special Steel, in which formic acid was produced through the use of converter gas, and the production of sodium bicarbonate raw materials by Handan Xinxing Cast Pipe, and so on [32,33];
2. After phosphorus-containing slag is processed by carbothermal reduction gasification dephosphorization, favorable conditions for dissolving the reduced phosphorus vapor into metallic iron phase are controlled, so that more phosphorus vapor can enter the molten iron to generate ferrophosphorus, which can be used as electrode raw materials or directly used as ferrophosphorus in steelmaking, with high added value [31,34].

Because the  $FeO$  in converter slag is reduced prior to  $P_2O_5$ , part of the  $P_2$  gas will be dissolved into molten iron to generate ferrophosphorus, which will reduce the overflow of  $P_2$ . At the same time, the process will seriously pollute the environment and the equipment investment is expensive for the preparation of phosphoric acid. Therefore, the recycling of phosphorus-containing slag is a suitable method for preparing ferrophosphorus.

## 5. Conclusions

The resource utilization of converter slag is of great significance to green and low-carbon production in the iron and steel industry. In this paper, the dephosphorization behavior of converter slag by carbothermal reduction and its industrial applications, were mainly studied, and ways of using the phosphorus in the dephosphorization process of converter slag by carbothermal reduction and gasification were analyzed. The main conclusions and prospects are as follows:

- (1) On the basis of our experiments and thermodynamic calculations, the feasibility of phosphorus removal from converter slag by carbothermal reduction was demonstrated. When the temperature is higher than 1433 K, the carbothermal reduction reaction can take place, and the main reduction product is  $P_2$ . The average dephosphorization rate was 32.57% under the experimental conditions;
- (2) The production practice of Cheng steel shows that the dephosphorization effect of the subsequent furnace procedure will not be affected by the carbothermic reduction of converter slag. The composition of the terminal molten steel was stable, and the average consumption of iron and steel per heat was reduced by 4.74 kg, the slagging

material was reduced by 608 kg, and CO<sub>2</sub> emissions were reduced by 4.86 kg, which will result in emissions reductions and energy savings;

- (3) Part of the P<sub>2</sub> produced by the carbothermal reduction enters the Fe phase, and the other part is discharged with the furnace gas. With an increase in Fe content, the amount of P<sub>2</sub> also increases. In order to efficiently recycle phosphorus resources, appropriate carbothermal reduction conditions and optimal gasification-dephosphorization parameters should be fully considered so that more P<sub>2</sub> can enter the molten iron to generate ferrophosphorus.

However, the preparation of ferrophosphorus by adding P<sub>2</sub> into metallic iron should also be comprehensively studied while ensuring appropriate carbothermal reduction conditions and the optimal parameters for gasification dephosphorization, which will be studied through thermodynamic calculations and thermal experiments in our follow-up work.

**Author Contributions:** S.T. and C.L. conceived and designed the study; S.T. and S.Z. performed the calculation; S.T. conducted the experiment; L.A. and S.W. analyzed the experimental data; and S.T. and C.L. wrote the paper. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the National Natural Science Foundation of China, grant number 52004097 and 52274334.

**Informed Consent Statement:** Written informed consent has been obtained from the patient(s) to publish this paper.

**Data Availability Statement:** Not applicable.

**Acknowledgments:** Thanks to S. Cui of Tangshan Normal University for checking and revising the manuscript.

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

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