STUDY ON THE KINETICS OF GAS-SOLID BASED SYNERGISTIC REDUCTION OF LIMONITE CARBON-CONTAINING PELLETS

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Abstract

The gas-solid-based synergistic reduction of pellets is an innovative and effective method for iron ore smelting. With the development and utilization of iron resources, ore reserves have been greatly depleted; therefore, as a scarce mineral resource, the comprehensive utilization of limonite has become increasingly important. To study the reduction kinetics of pellets in depth, this study used coke and reducing gases (CO and H_2) to study the reduction characteristics and changes occurring in carbon-containing limonite pellets. The results showed that the total weight loss percentage of pellets gradually increased with the temperature. The C/O molar ratio had a greater effect on the total weight loss percentage of pellets in a N_2 atmosphere, but it had no significant effect in CO or H_2 atmospheres. The maximum reaction rate increased with increasing temperature. The reduction reaction was the most difficult to proceed in the N_2 atmosphere, and the reaction was most likely to occur in the CO atmosphere. The analysis of pellets by EPMA and XRD found that the pellets reduced in the N_2 atmosphere had the lowest porosity, and the pellets reduced in the H_2 atmosphere had the highest porosity, which was more conducive to gas diffusion. Some of the unreduced Si, Al, Mn, Ca, and Fe in the pellets reduced in the N_2 atmosphere precipitated in the form of oxides, but when a reducing gas (CO, H_2) was introduced, precipitation did not occur.

Keywords: Limonite; Pellet; Reduction

1. Introduction

Blast furnaces have been extensively studied due to their prevalence in the ironmaking industry [1]. When selecting iron ore, most processes use magnetite or hematite for blast furnace smelting, but China has a large amount of limonite resources. Due to their low grade and high water of crystallization contents, these iron ores are not used in many pellets [2]. During the dehydration process of limonite, FeOOH is completely converted into Fe_2O_3, which introduces nanopores and increases the specific surface area of limonite [3]. As a result, its reduction performance is improved [4]. Blast furnace smelting produces serious pollution, consumes large amounts of energy, and has high requirements for ore strength. These drawbacks affect the application of limonite in the ironmaking process, which ultimately affects the development of steel enterprises. Direct reduced iron produces much less environmental pollution and has a wide application range of raw materials, which solves the shortcomings of blast furnace smelting.

Direct reduction methods can be divided into gas-based direct reduction and coal-based direct reduction. Gas-based direct reduction ironmaking methods use a reducing gas to reduce iron ore, most commonly H_2 and CO [5]. Under a H_2 atmosphere, because H_2 has a lower molecular weight and more easily diffuses, the reduction performance of pellets is significantly higher than that under a CO atmosphere, and the reduced pellets have a higher reduction rate [6].

Coal-based direct reduction methods use coal as a reducing agent and fuel (which can be solid or processed into a reducing gas) is added to the granular material layer of the reduction reaction device to reduce iron-containing raw materials to directly reduce iron at high temperatures. The most mature coal-based direct reduction method is the rotary kiln process of non-carbon-containing pellets, but it requires large and expensive equipment and has a slow reduction rate and low recovery rate. Carbon-containing pellets can be made from a wide range of raw materials, strong adaptability of raw material particle size, and produce low amounts of

environmental pollution [7]. Some researchers have found that the reduction degree of carbon-containing pellets increases upon increasing the C/O molar ratio and temperature of the pellets [8-9]. Interfacial reactions are the limiting step during the reduction of carbon-containing pellets [10].

Although carbon-containing pellets have certain advantages, they still have several disadvantages such as high energy consumption, poor quality, poor equipment stability, high product energy consumption, and easy adhesion of pellets. If a small volume of reducing gas is introduced during the reduction of carbon-containing pellets, some shortcomings of the reduction process of carbon-containing pellets can be improved. In order to study the influence of the atmosphere, temperature, and C/O molar ratio on the reduction characteristics of pure limonite pellets, this paper uses a thermogravimetric method to calculate the reduction kinetics under different temperatures and different atmospheres, and uses EPMA and XRD to analyze the reduced pellets.

2. Materials and methods
2.1. Materials

The limonite samples used in this study were obtained from Honghe Iron and Steel Co., Ltd. It had a high porosity, strong water absorption, large wet capacity, and it easily melted. Its chemical composition is shown in Table 1. The crystal phases in the sample were characterized by X-ray diffraction, and the XRD pattern of limonite is shown in Figure 1(a). The pattern showed that the main mineral form of limonite is FeOOH, which contained a large amount of crystal water. Coke was produced by Yunnan Fanya Electronic Commerce Co., Ltd.

(Shizong), and its chemical composition is shown in Table 2. The XRD pattern of coke is shown in Figure 1(b). It can be seen from the figure that in addition to fixing C, the ash was mainly composed of SiO_2 and contained a small amount of Fe_2O_3. The particle size of limonite powder and coke powder used in the experiment both ranged from 0.075 mm~0.15 mm.
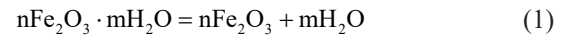
2.2. Preparation of limonite-coke composite pellets

A disc pelletizer was used to prepare the limonite-coke powder composite pellets with a diameter of 10 ~ 12.5 mm. The prepared pellets were dried in a drying oven at 110 ℃ for 2 h to remove free water.

2.3. Thermodynamic analysis

Under a N_2 atmosphere, the main reactions of limonite pellets occurring during direct carbon reduction were the direct reduction of iron oxides, indirect reduction, and coke gasification, as shown in equations (1) ~ (8).
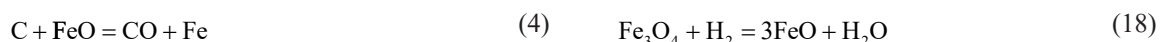
Crystal water desorption:

$$nFe_2O_3 \cdot mH_2O = nFe_2O_3 + mH_2O \qquad (1)$$

Direct reduction：

$$C + 3Fe_2O_3 = CO + 2Fe_3O_4 \qquad (2)$$

$$C + Fe_3O_4 = 3FeO + CO \qquad (3)$$

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

Ash	S	H_2O	Volatile	C
14.29	0.59	0.46	1.64	83.6

Table 1. *Iron ore chemical composition (%)*

w(TFe)	w(FeO)	w(SiO$_2$)	w(S)	w(MnO)	w(TiO$_2$)	w(Pb)	w(Zn)	w(K$_2$O)	w(Na$_2$O)	w(As)	w(Cu)	w(Sn)	w(V$_2$O$_5$)	LoI
54.67	0.29	4.04	0.048	3.47	0.27	0.009	0.018	0.095	0.001	0.007	0.009	0.001	0.04	14.82

Figure 1. *XRD pattern of (a) Guisha limonite and (b) coke*

$$C + FeO = CO + Fe \tag{4}$$

Indirect reduction:

$$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2 \tag{5}$$

$$Fe_3O_4 + CO = 3FeO + CO_2 \tag{6}$$

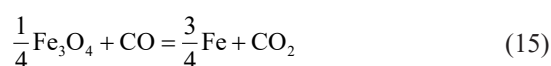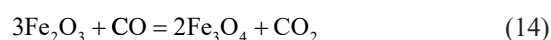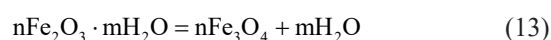$$FeO + CO = Fe + CO_2 \tag{7}$$

Coke gasification:

$$C + CO_2 = 2CO \tag{8}$$

The main reactions occurring under a CO atmosphere were basically the same as under a N_2 atmosphere, but the direct reduction process of CO played a dominant role. The reaction process occurred in a stepwise fashion, and the reaction process is shown in formulas (9) to (15).
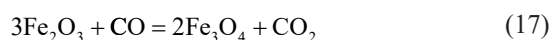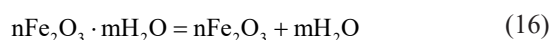
T > 843K

$$nFe_2O_3 \cdot mH_2O = nFe_2O_3 + mH_2O \tag{9}$$

$$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2 \tag{10}$$

$$Fe_3O_4 + CO = 3FeO + CO_2 \tag{11}$$

$$FeO + CO = Fe + CO_2 \tag{12}$$

T < 843K

$$nFe_2O_3 \cdot mH_2O = nFe_3O_4 + mH_2O \tag{13}$$

$$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2 \tag{14}$$

$$\frac{1}{4}Fe_3O_4 + CO = \frac{3}{4}Fe + CO_2 \tag{15}$$
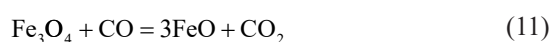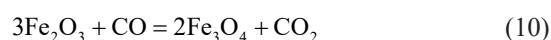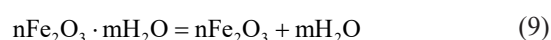
In addition to the reactions that take place in a N_2 atmosphere, the main reactions that took place in a H_2 atmosphere included the direct reduction of H_2, which played a dominant role, as shown in equations (16) to (22).

T > 843K

$$nFe_2O_3 \cdot mH_2O = nFe_2O_3 + mH_2O \tag{16}$$

$$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2 \tag{17}$$

$$Fe_3O_4 + H_2 = 3FeO + H_2O \tag{18}$$

$$FeO + H_2 = Fe + H_2O \tag{19}$$

T > 843K

$$nFe_2O_3 \cdot mH_2O = nFe_2O_3 + mH_2O \tag{20}$$

$$3Fe_2O_3 + H_2 = 2Fe_3O_4 + H_2O \tag{21}$$

$$\frac{1}{4}Fe_3O_4 + H_2 = \frac{3}{4}Fe + H_2O \tag{22}$$

2.4. Kinetics analysis

The reduction of iron oxides is a complex process, and the thermal weight loss method is commonly used to study the reduction reaction kinetics. In order to reasonably study the reduction process of oxidized pellets, previous researchers have established many different methods to investigate the mechanism. Previous studies have shown that under a N_2 atmosphere, coke acts as the only reducing agent, and the reaction involves nucleation and growth. The process can be described by the Avrami-Erofeev model [11]. Under H_2 and CO atmospheres, the reducing gas plays a leading role in the reduction process. At this time, the reduction reaction is controlled by interfacial chemical reactions, which can be modeled using the shrinking core model [7]. Table 4 lists the kinetic models used to describe the above reactions.

2.5. Experimental method

A schematic diagram of the limonite pellet reduction experiment device is shown in Figure 2. The test system mainly included a vertical tube furnace, flowmeter, balance, electric furnace control system, and data processing computer. The computer was connected with the balance to realize real-time data recording.

The tube furnace was heated to the reduction temperature and then purged with N_2 gas with a purity of 99.99%. After the furnace was filled with N_2 gas, the prepared limonite-coke composite pellet particles were suspended in a hanging basket, suspended under the balance by an iron-chromium-aluminum wire, and the reducing gas was simultaneously introduced (gas velocity = 0.382 m/min). The mass of the pellets was

Table 3. *Compound pellet ratio (wt%)*

C/O	0.75	1	1.25	1.5
Limonite wt%	81.4	76.95	72.96	69.37
Coke wt%	17.1	21.55	25.54	29.13
Bentonite wt%	1.5	1.5	1.5	1.5

Table 4. *Reaction kinetics models*

Response model	Equation	Rate control procedure
Avrami-Erofeev Model	$\left[-\ln\left(1-x\right)\right]^{2/3} = kt$	Nucleation and growth control
Shrink Nuclear Model	$1-\left(1-x\right)^{1/3} = kt$	Interface chemical reaction control

automatically recorded by a computer.

The limonite-coke powder composite pellet particles lost oxygen during the reduction process, and the coke content in the particles also decreased as reduction proceeded. The formula for calculating the total weight loss percentage (w) is shown in formula (23) [12-13]:

$$w = \frac{w_0 - w_t}{w_{Fe} + w_C} \times 100\% \qquad (23)$$

where is the initial mass of a pellet, is the mass of a pellet at time t, is the maximum weight loss of limonite, and is the maximum weight loss of coke.

Therefore, the formula for calculating the reaction rate v of limonite-coke powder composite pellets is shown in equation (24):

$$v = \frac{dw}{dt} = \frac{\Delta w}{\Delta t} \qquad (24)$$

where Δw is the increase in the total weight loss percentage at each time interval Δt.

3. Results and discussion
3.1. Reduction kinetics analysis
3.1.1. Percentage analysis of total weight loss

The experiment was carried out at 973–1373 K, with either a N_2, CO, or H_2 atmosphere, and 0.75–1.5 C/O molar ratios. The total weight loss percentage of limonite pellets was calculated using equation (23). Figure 3 shows the relationship between the total weight loss percentage and reaction time. Figure 3 shows that the reduction temperature, reducing atmosphere, and C/O molar ratio significantly affected the total weight loss percentage of limonite pellets. As shown in Figures 3(a), (b), and (c), under a given C/O molar ratio and reducing atmosphere, a higher temperature increased the total weight loss and shortened the reaction time. As shown in Figure 3(b), using a C/O molar ratio of 1 and a CO atmosphere as an

example, the total weight loss of limonite increased from 57.26% to 82.20% as the temperature increased. From 973 K to 1373 K, the reduction time decreased from 62 min to 30 min. This increase occurred because increasing the temperature promoted the endothermic reduction reaction between the iron oxide and reducing agent.

As shown in Figure 3 (d), (e), and (f), at 1375 K, when the C/O molar ratio increased from 0.75 to 1.5, under an N_2 atmosphere, the total weight loss of limonite pellets increased from 53.28% to 66.67%, which was proportional to the C/O molar ratio. Under CO and H_2 atmospheres, the total weight loss percentage of limonite pellets was the same. It can be seen that under the N_2 atmosphere, increasing the C/O molar ratio increased the reduction rate of limonite pellets. The C/O molar ratio in CO and H_2 atmospheres did not affect the total weight loss percentage of the pellets.

3.1.2. Response rate analysis

By substituting the total weight loss percentage and reaction time into equation (24), the reaction rate of the limonite pellets was calculated. Figure 4 shows the relationship between reaction rate and time under different reducing temperatures, reducing atmospheres, and C/O molar ratios.

As shown in Figure 4, the reduction temperature, reducing atmosphere, and C/O molar ratio had a significant effect on the reaction rate. From Figure 4 (a), (b), and (c), when the atmosphere and the C/O molar ratio were the same, the maximum reaction rate increased with the temperature. For example, as shown in Figure 4(b), in a CO atmosphere with a C/O molar ratio of 1, the temperature increased from 973 K to 1373 K, and the maximum limonite reaction rate increased from 6.510521 s^{-1} to 14.47173 s^{-1}. The results showed that a higher temperature promoted the reduction of limonite. Figure 4(d), (e), and (f) show that in N_2 and H_2 atmospheres, upon increasing the C/O molar ratio, the maximum reaction rate first
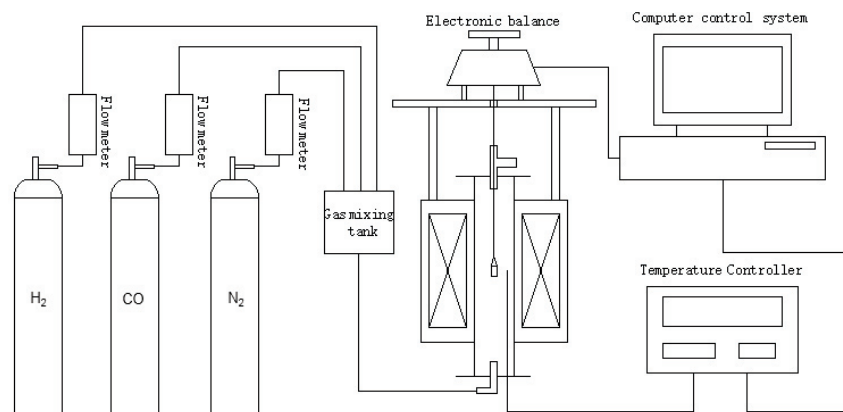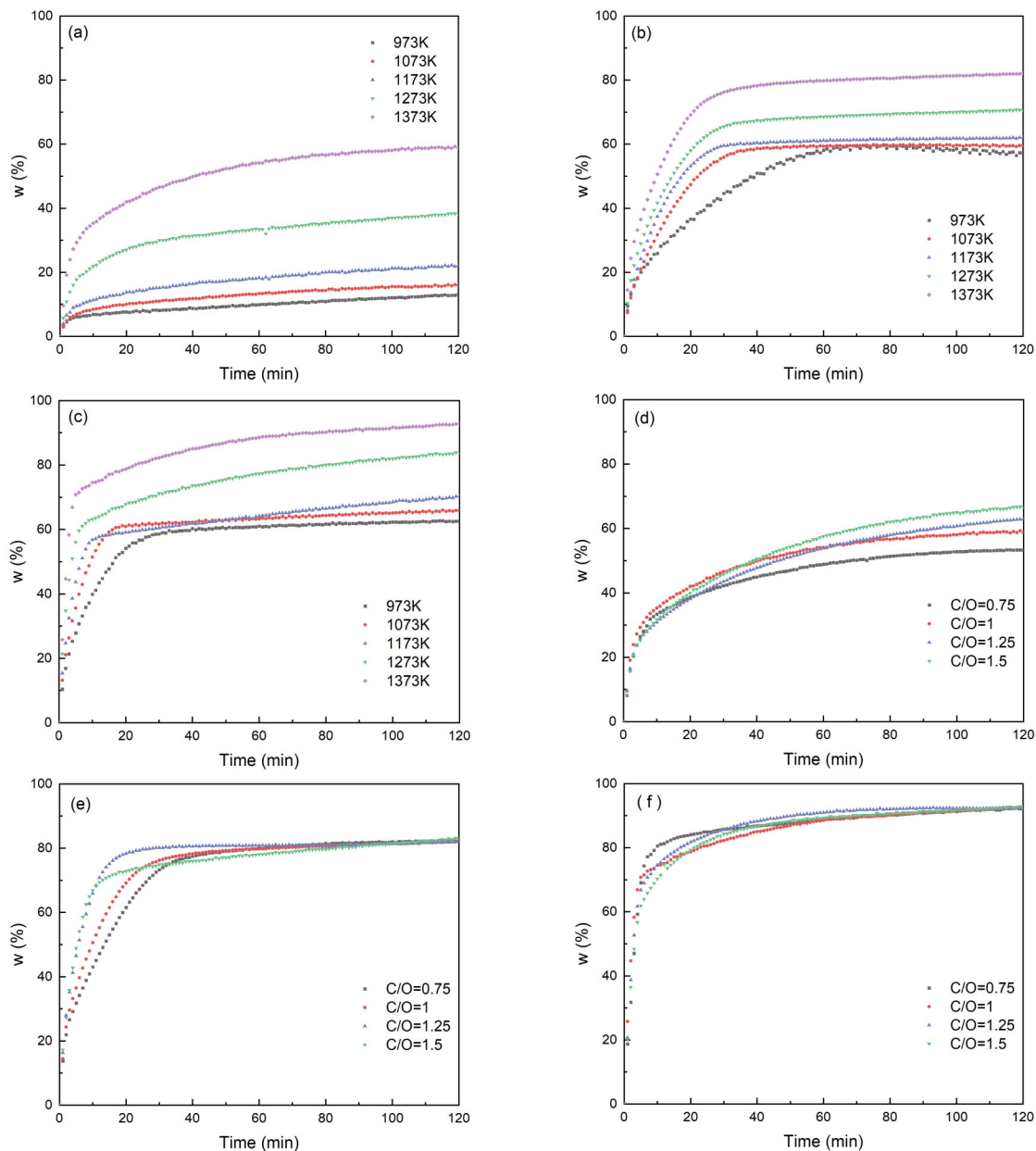

Figure 2. Test device schematic diagram

Figure 3. *The effect of reaction conditions on the total weight loss percentage of limonite pellets:*
(a) C/O molar ratio is 1, N₂ atmosphere; (b) C/O molar ratio is 1, CO atmosphere;
(c) C/O molar ratio is 1, H₂ atmosphere; (d) reaction temperature is 1375 K, N₂ atmosphere;
(e) reaction temperature is 1373 K, CO atmosphere; (f) reaction temperature is 1373 K, H₂ atmosphere

increased and then decreased because coke was added with N_2 as a reducing agent. When the amount of coke increased, its reducibility rapidly increased. When the coke reached a certain amount, unreacted coke prevented the generated CO from contacting the limonite, which decreased the reaction rate. Under the H_2 and H_2 atmospheres, increasing the amount of coke provided more reducing agent to generate more CO gas, which accelerated the reaction. When the amount of coke exceeded a certain ratio, it affected

the inward diffusion rate of H_2, and unreacted carbon also reduced the specific surface area of the ore. As a result, the reaction rate was reduced. In a CO atmosphere, the reaction rate gradually increased upon increasing the C/O molar ratio, which showed that adding the proper amount of coke increased the maximum reaction rate. In N_2 and H_2 atmospheres, the optimal C/O molar ratio was between 0.72 and 1.5. In a CO atmosphere, the optimal C/O molar ratio was greater than or equal to 1.5.
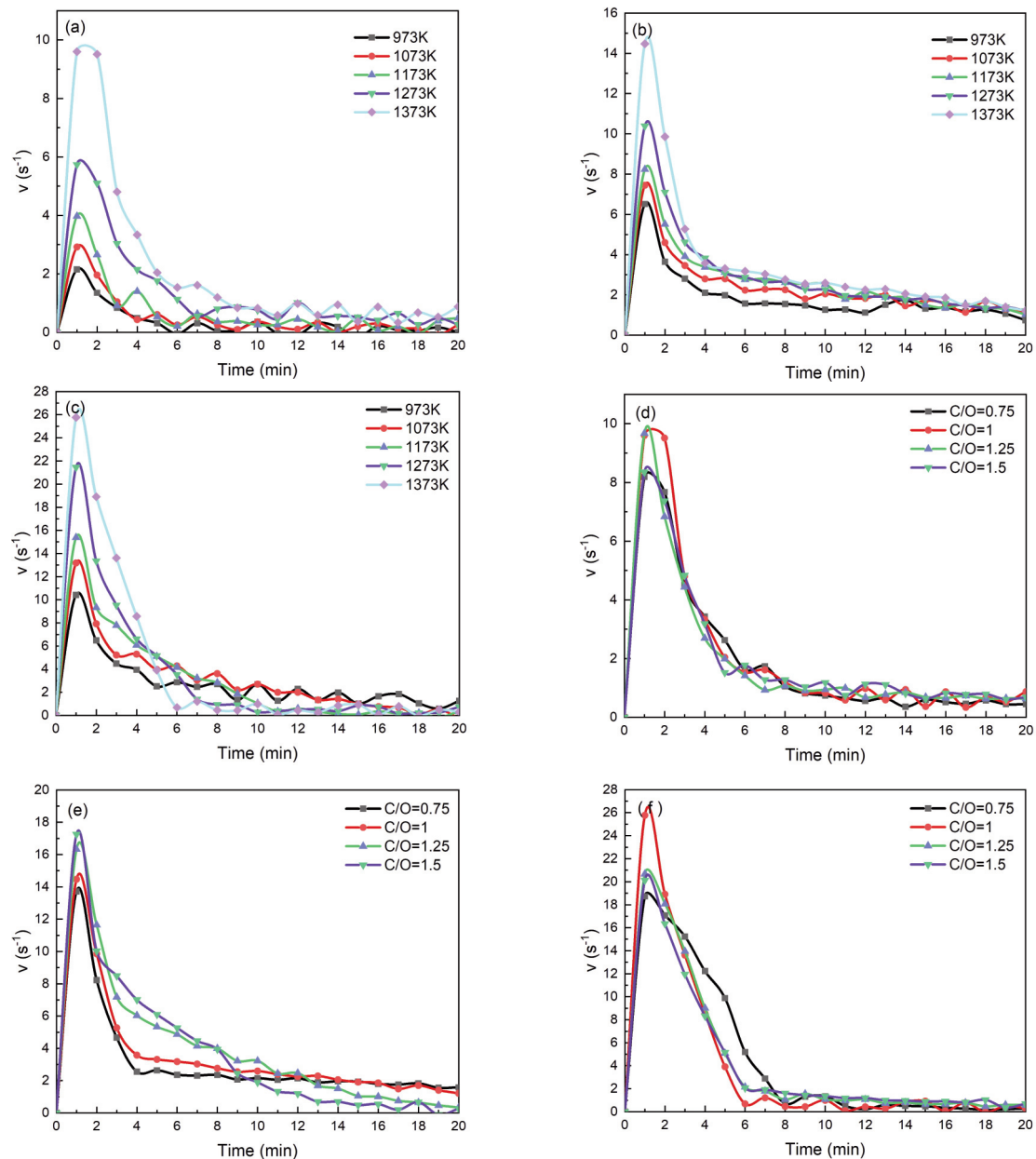
Figure 4. *The influence of experimental conditions on the reaction rate of limonite pellets:*
(a) C/O molar ratio is 1, N_2 atmosphere; (b) C/O molar ratio is 1, CO atmosphere;
(c) C/O The molar ratio is 1, H_2 atmosphere; (d) reaction temperature is 1375 K, N_2 atmosphere;
(e) reaction temperature is 1373 K, CO atmosphere; (f) reaction temperature is 1373 K, H_2 atmosphere

3.1.3. Kinetics analysis

The apparent reaction rate and correlation coefficient under different conditions were calculated by the kinetics models in Table 4 and the results are shown in Table 5. The correlation coefficients in Table 5 were all above 0.95, indicating that the response model had a good linear relationship. The results further confirmed the correctness of the response model. Table 5 also shows that the apparent reaction rate of limonite

pellets increased as the reduction temperature increased. At the same reduction temperature, the apparent reaction rate constant in the H_2 atmosphere was the largest, and the apparent reaction rate constant in the N_2 atmosphere was the smallest.

According to the Arrhenius equation, the relationship between reaction (T) and reaction rate constant (k) can be obtained, as shown in equation (3) [15]. In equation (3), E is the activation energy (kJ/mol), and k_0 is the frequency factor.

Table 5. *Apparent reaction rate and correlation coefficient of limonite pellets*

Atmosphere	T (K)	Response model	K (min⁻¹)	R²
N₂	973	$\left[-\ln\left(1-x\right)\right]^{2/3} = kt$	0.00427	0.96973
	1073		0.00513	0.99039
	1173		0.00711	0.97605
	1273		0.01665	0.98125
	1373		0.02308	0.97722
CO	973	$1-\left(1-x\right)^{1/3} = kt$	0.01326	0.95125
	1073		0.01801	0.96215
	1173		0.02079	0.96816
	1273		0.02345	0.95772
	1373		0.03692	0.95468
H₂	973	$1-\left(1-x\right)^{1/3} = kt$	0.02265	0.95704
	1073		0.02888	0.95596
	1173		0.03684	0.9753
	1273		0.05226	0.96688
	1373		0.07745	0.9872

$$k = k_0 \exp\left(-\frac{E}{RT}\right) \tag{25}$$

Combined with equation (3), and the logarithms of both sides of the equation, as shown in equation (4):

$$\ln k = -\frac{E}{RT} + \ln k_0 \tag{26}$$

The linear fit between ln*k* and 103/*RT* is shown in Figure 4, and the fitted kinetic parameters are listed in Table 6.

Table 6 shows that the reaction was the most difficult in the N₂ atmosphere, and the reaction was most likely to occur in the CO atmosphere.

3.2. Microstructure changes of reduced particles

Using EPMA and XRD, taking C/O = 1.5 as an example (reduction time = 120 min), the microstructures of the particles reduced in N₂, CO, and H₂ atmospheres were studied and analyzed. The results are shown in Figure 5.

First, the EPMA image in Figure 6 shows that the porosity of the pellets reduced in the N₂ atmosphere was the lowest, while the porosity of the pellets reduced in the H₂ atmosphere was the highest. The porosity of the pellets reduced in the CO atmosphere was between the two. This indicated that during the reduction of carbon-containing pellets, the introduction of a reducing gas increased the porosity of the pellets, which improved the diffusion of coke after gasification. The reducing gas would also directly reduce the pellets, which promoted the reaction progress [15]. The XRD pattern in Fig. 5 shows that the pellets had the same composition after the reaction,
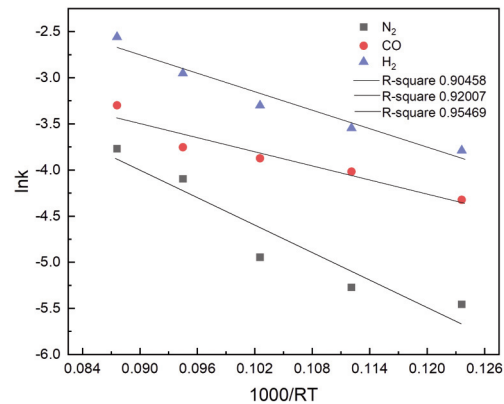
Figure 5. *The fitting curve of lnk and 1000/RT under different conditions*

Table 6. *Kinetics parameters of the reduction of limonite particles*

Atmosphere	N₂	CO	H₂
E (kJ/mol)	49.42836	25.31582	33.31605
ln*k* (min⁻¹)	0.43608	-1.21843	0.23853

and all were composed of Fe, SiO₂, and Fe₃O₄.

According to Fig. 6, consolidated products precipitated in the pellets reduced in the N₂ atmosphere, but no crystalline products precipitated when reduced in CO and H₂ atmospheres. The spectrum of the consolidated products was detected, and the results are shown in Figure 7 and Table 7.

As shown in Figure 7, the analysis of the results of the test shows that when reduced in an N₂ atmosphere, some unreduced Si, Al, Mn, Ca, and Fe precipitated in the form of oxides. When a reducing gas (CO, H₂) was used, no precipitation occurred.
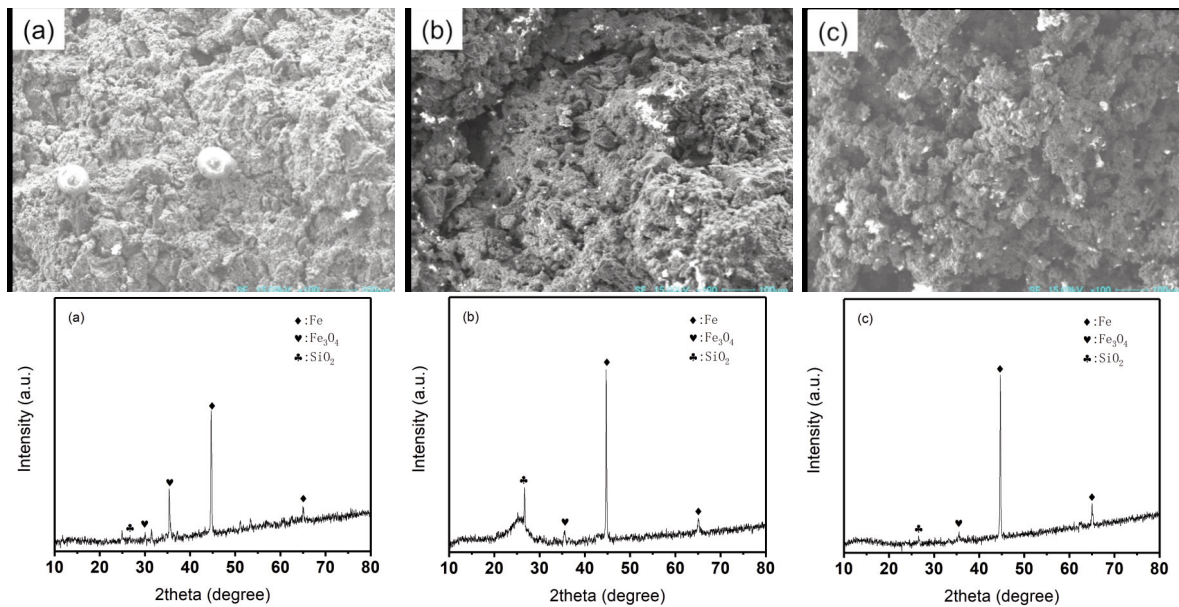
Figure 6. *EPMA Images and XRD patterns of limonite pellets reduced at 1373 K under different conditions: (a) C/O = 1.5, N_2 atmosphere, (b) C/O = 1.5, CO atmosphere, (c) C/O = 1.5, H_2 atmosphere*
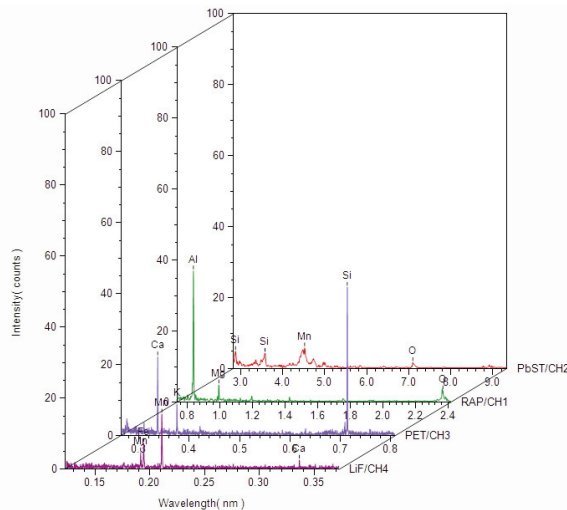

Figure 7. *Spectrum detection results*

Table 7. *Element ratio*

O	Al	Si	Ca	Mn	Fe
41.77	6.19	26.97	3.42	14.84	6.81

4. Conclusion

(1) In pellets reduced with a C/O molar ratio of 1 under the same reducing atmosphere, increasing the temperature increased the total weight loss percentage and shortened the reduction time.

(2) In pellets reduced at 1373 K under the N_2 atmosphere, increasing the C/O molar ratio promoted the reduction rate of limonite pellets. In CO and H_2 atmospheres the C/O ratio did not affect the total weight loss percentage of the pellets.

(3) In N_2 and H_2 atmospheres, the optimal C/O molar ratio was between 0.72 and 1.5. In a CO atmosphere, the optimal C/O molar ratio was $\geq$ 1.5.

(4) In pellets reduced with a C/O molar ratio of 1 at 1373 K, the E values in N_2, CO, and H_2 atmosphere were 49.42836 kJ/mol, 25.31582 kJ/mol, and 33.31605 kJ/mol, respectively, and the ln$k$ values were 0.43608 min^{-1}, -1.21843 min^{-1}, and 0.23853 min^{-1}, respectively.

(5) In pellets reduced with a C/O molar ratio of 1 at 1373 K, the porosity of pellets reduced in the N_2 atmosphere was the smallest, the porosity of pellets reduced in a H_2 atmosphere was the largest, and the porosity of pellets reduced in CO atmosphere was somewhere in between. The composition of the pellets after reduction was the same.

(6) In pellets reduced with a C/O molar ratio of 1 at 1373 K under the N_2 atmosphere, some unreduced Si, Al, Mn, Ca, and Fe precipitated in the form of oxides. When a reducing gas (CO, H_2) was introduced, no precipitation occurred.

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ISPITIVANJE KINETIKE SINERGISTIČKE REDUKCIJE KOD PELETA LIMONITA NA BAZI GAS-ČVRSTO KOJI SADRŽI UGLJENIK

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Apstrakt

Sinergistička redukcija peleta na bazi gas-čvrsto predstavlja inovativnu i efikasnu metodu za topljenje rude železa. Sa razvojem i iskorišćenjem resursa železa, rezerve rude su znatno iscrpljene, stoga, široka upotreba limonita postaje sve važnija. Da bi se sveobuhvatno proučila redukciona kinetika peleta, za potrebe ovog ispitivanja su korišćeni koks i redukcioni gasovi (CO i H_2) kako bi se ispitala svojstva redukcije i promene koje se javljaju u peletu limonita koji sadrži ugljenik. Rezultati su pokazali da se ukupan procenat gubitka težine kod peleta postepeno povećavao kako se povećavala temperatura. Molarni odnos C/O je imao veći uticaj na ukupan procenat gubitka težine kod peleta u atmosferi sa N_2, ali nije imao značajniji efekat u atmosferi sa CO i H_2. Maksimalna brzina reakcije se povećavala sa porastom temperature. Reakcija redukcije se najteže odvijala u atmosferi sa N_2, a najlakše se odvijala u atmosferi sa CO. Elektronska mikro analiza – EPMA, kao i XRD analiza su pokazale da su peleti redukovani u atmosferi sa N_2 imali najmanju poroznost, a da su peleti redukovani u atmosferi sa H_2 imali najveću poroznost, što je pogodnije za difuziju gasova. Jedan deo neredukovanog Si, Al, Mn, Ca i Fe u peletu koji je redukovan u atmosferi sa N_2 se nataložio u obliku oksida, ali kada je uveden redukcioni gas (CO, H_2), taloženja nije bilo.

Ključne reči: Limonit; Pelet; Redukcija