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## Full Length Article

## Investigation of the redox performance of pyrite cinder calcined at different temperature in chemical looping combustion

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## ABSTRACT

As an industrial solid waste, pyrite cinder exhibited excellent reactivity and cycle stability in chemical looping combustion. Prior to the experiment, oxygen carriers often experienced a high temperature calcination process to stabilize the physico-chemical properties, which presented significant influence on the redox performance of oxygen carriers. However, the effect of calcination temperature on the cyclic reaction performance of pyrite cinder has not been studied in detail. In this work, the effect of calcination temperature on the redox activity and attrition characteristic of pyrite cinder were studied in a fluidized-bed reactor using CH<sub>4</sub> as fuel. A series of pyrite cinder samples were prepared by controlling the calcination temperature. The redox activity and attrition rate of the obtained pyrite cinder samples were investigated deeply. The results showed that calcination temperature displayed significant impact on the redox performance of pyrite cinder. Considering CH<sub>4</sub> conversion (80%–85%) and attrition resistance, the pyrite cinder calcined at 1050 °C presented excellent redox properties. In the whole experiment process, the CO<sub>2</sub> selectivity of the pyrite cinder samples were not affected by the calcination temperature and were still close to 100%. The results can provide reference for optimizing the calcination temperature of pyrite cinder during chemical looping process.

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## 1. Introduction

With the rapid development of global society and economy, the demand for energy has increased dramatically. It is facing more and more challenges in the fields of energy utilization, environmental protection and cleaner production [1]. Studies have shown that large amounts of carbon dioxide from human activities are most likely to be the main cause of global warming [2]. Among all sources of carbon dioxide, the extensive use of fossil fuels (coal, oil and natural gas) is the most important source of carbon dioxide emissions [3,4]. From the perspective of long-term sustainable development, alternatives to fossil fuels must be found to solve the problem of carbon emissions. Nowadays, solar energy, water energy, wind energy, tidal energy and other clean and renewable

energy have gradually played a role in human energy utilization [5]. However, limited by the low energy density and utilization technology, the dominant position of fossil energy will not be changed in the next few decades. The development of efficient and clean carbon capture technology in coal-fired power plants is of great significance to the sustainable development of global economy [6].

To date, some technological routes are being developed to reduce CO<sub>2</sub> emissions [7–11], such as: (1) improving energy efficiency, (2) using low-carbon fuels, (3) developing new energy sources such as solar, biomass, wind, etc., and (4) CO<sub>2</sub> capture, utilization and storage (CCUS) technology. CCUS technology is one of the effective ways to solve the excess emissions of CO<sub>2</sub> in the short term. As the basis of CCUS technology, a lot of problems still need to be solved in the CO<sub>2</sub> enrichment process [12]. Among CO<sub>2</sub> capture technologies, three main routes are feasible or expected to be implemented in power plants: pre-combustion, post-combustion and oxy-fuel combustion [13,14]. The above three CO<sub>2</sub> capture technologies are involved in excessive energy

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consumption of gas separation process [15,16], thus limiting the commercialization of these technologies.

Chemical looping combustion (CLC) is a new combustion technology with inherent CO<sub>2</sub> separation [17,18], which uses the lattice oxygen transmitted by solid oxygen carrier (OC) particles to carry out indirect combustion of fuels, thus avoiding direct contact between fuels and air and achieving CO<sub>2</sub> enrichment at near zero energy consumption. Compared with the traditional combustion technology, CLC process decomposes the direct combustion into two relatively independent redox processes, realizing the step utilization of chemical energy and reducing the exergy loss in combustion process [15].

From the above analysis of CLC process, it can be seen that oxygen carrier acts a crucial function in achieving the complete combustion of fuels [19–21]. Iron-based oxides are regarded as the most potential oxygen carriers for commercial applications because of low cost, wide range of sources and environmentally friendly [22–24]. At present, oxygen carriers mainly include synthetic and natural/waste materials [25,26]. In general, synthetic oxygen carriers present the characteristics of porous structure, high reaction activity, poor attrition resistance and long operation life. Considering the problems of crushing, attrition, sintering and poisoning of oxygen carriers in the actual operation process, the cost of using synthetic materials as oxygen carrier is too high [15]. In recent years, more and more researchers began to pay attention to low-cost oxygen carriers, such as iron ore [24,27,28], copper ore [29–31] and manganese ore [32–34]. Ilmenite (FeTiO<sub>3</sub>) [35,36] and hematite [37,38] are two common natural iron-based oxygen carriers.

Calcination is a necessary process for preparing oxygen carriers [34,39]. On the one hand, high calcination temperature is usually beneficial to improve the mechanical strength of oxygen carrier particles. On the other hand, calcination can increase the oxygen carrying capacity of oxygen carriers by oxidizing the active component to high valence state. In order to fully oxidize ilmenite prior to the redox reactions, the calcination process is often needed [40]. The results of Zheng *et al.* [39] showed that low calcination temperature could generate a CeO<sub>2</sub>-ZrO<sub>2</sub> sample with good uniformity and three-dimensional ordered macroporous structure, thus enhancing the redox activity of CeO<sub>2</sub>-ZrO<sub>2</sub> by increasing the diffusion of oxygen species. Appropriate calcination conditions are conducive to the microstructure and cycle stability of oxygen carrier. So far, the studies on the calcination process of oxygen carrier are rare.

In the fluidized-bed CLC system, two main factors affect the stable operation of the whole system [41]: one is the cycling stability of oxygen carrier, the other is the attrition rate of oxygen carrier. The low attrition rate of oxygen carrier material can reduce the supplement of fresh oxygen carrier during operation, thus decreasing the system investment, the cost of operation and waste treatment. Therefore, it is of great significance to study the attrition resistance of OC. At present, the attrition characteristics of Ca-based sorbents in CO<sub>2</sub> capture process are studied extensively [42–44], but the research on the attrition characteristics of OC in CLC process is not enough. If the mechanical strength of oxygen carrier particles can't bear the mechanical stress produced by collision, the OC particles are easy to be broken.

Based on the above analysis, the calcination process has an important influence on the performance of oxygen carrier. Compared with other iron-based oxygen carriers, our previous results showed that pyrite cinder exhibited excellent reactivity and cycle stability in CLC process [45–47]. However, the effect of calcination temperature on the cyclic reaction performance of pyrite cinder has not been studied. In this work, the influence of calcination temperature on the redox activity and attrition characteristics of pyrite cinder were studied in a fluidized-bed reactor. This work provides

an important reference for the selection of proper calcination temperature for pyrite cinder.

## 2. Experimental

### 2.1. Preparation of pyrite cinder samples

Detailed preparation process of pyrite cinder particles (0.180–0.250 mm) was shown in Fig. S1 (see Supplementary Material). In the first step of pyrite cinder particles preparation, the addition of deionized water was to improve the viscosity of pyrite cinder powders. Three calcination temperatures (950 °C, 1050 °C, 1150 °C) were investigated. The obtained pyrite cinder samples named Pc-950, Pc-1050 and Pc-1150, respectively. The calcination time for the three samples was 2 h, the heating rate was 10 °C·min<sup>−1</sup> and the heating atmosphere was air. The chemical composition of pyrite cinder samples were presented in the Supplementary Material (Table S1).

### 2.2. Characterization of oxygen carriers

The microstructure of fresh and spent OCs were characterized by XRF, SEM, XRD and BET. More details of the characterization were described in the Supplementary Material.

### 2.3. Evaluation of oxygen carriers

The cyclic reactivity of OCs were evaluated in a fluidized bed reactor. The schematic diagram of the fluidized bed reactor was shown in Fig. 1. The detailed description of experimental system and procedures were presented in the Supplementary Material. In this study, the mass of OC sample was 50 g. The experimental steps of a reduction–oxidation cycle was shown in Table 1. The flow rate of reaction gases was 2.0 L·min<sup>−1</sup>.

The equations for CH<sub>4</sub> conversion and CO<sub>2</sub> selectivity can be found in the previous study [45]. The attrition rate of different pyrite cinder samples in fluidized bed reactor was studied. The attrition rate can be obtained through the following equation:

$$\text{Attrition rate} = \frac{m_f}{\Delta t \times m} \quad (1)$$

In the above equation,  $m_f$  is the mass of pyrite cinder fines collected after the experiment.  $m$  is the mass of pyrite cinder.  $\Delta t$  is the time interval.

## 3. Results and Discussion

### 3.1. Microscopic physico-chemical properties of fresh pyrite cinder samples

The physico-chemical properties of fresh pyrite cinder samples were characterized by XRF, BET and SEM, as shown in Fig. 2 and Fig. 3.

Pyrite cinder mainly contains iron oxide, calcium sulfate and some inert components. Calcium sulfate (CaSO<sub>4</sub>) can decompose into CaO and SO<sub>2</sub> at high temperature, as shown in Eq. (2). Fig. 2 (a) showed the content of CaSO<sub>4</sub> in different pyrite cinder samples from XRF analysis.



It indicated that the content of CaSO<sub>4</sub> in pyrite cinder decreased gradually with the increase of calcination temperature. Pc-950 sample presented the highest content of CaSO<sub>4</sub> (14.28%). When the calcination temperature increased to 1150 °C, the content of CaSO<sub>4</sub> in Pc-1150 was 5.69%. Some studies have shown that a syn-

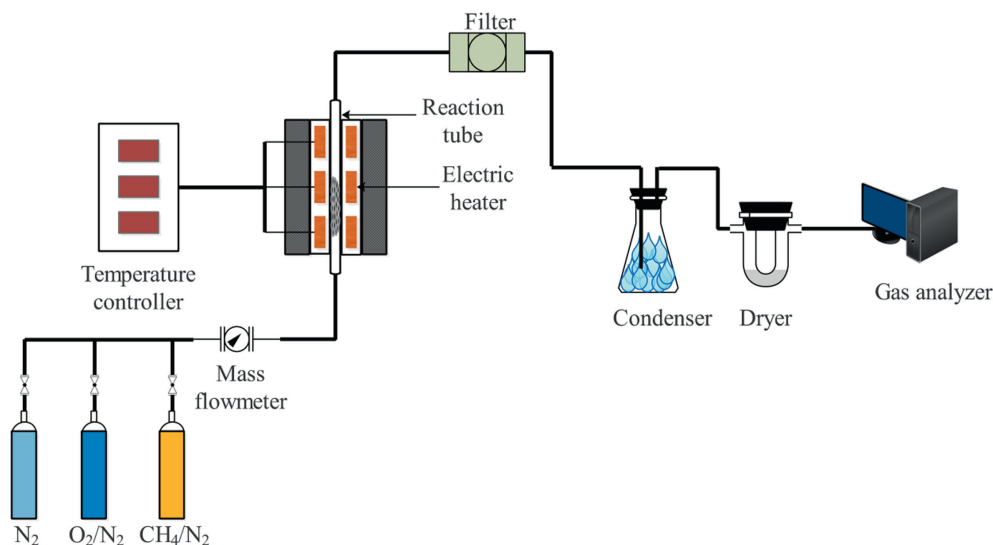


Fig. 1. Simplified schematic of fluidized-bed system.

**Table 1**  
Experimental steps of a reduction-oxidation cycle

| No. | Stage       | Temperature/°C | Duration/min | Reaction gas                      |
|-----|-------------|----------------|--------------|-----------------------------------|
| 1   | Reduction   | 900            | 10           | 5%CH <sub>4</sub> /N <sub>2</sub> |
| 2   | Inert purge |                | 5            | N <sub>2</sub>                    |
| 3   | Oxidization |                | 10           | 5%O <sub>2</sub> /N <sub>2</sub>  |

ergistic effect is existed between Fe<sub>2</sub>O<sub>3</sub> and CaSO<sub>4</sub>, which can improve the cyclic reaction performance of composite Fe<sub>2</sub>O<sub>3</sub>/CaSO<sub>4</sub> oxygen carrier [48,49].

The BET surface area of different pyrite cinder samples were presented in Fig. 2(b). It can be seen that the surface area of the three samples follow the order: Pc-950 > Pc-1050 > Pc-1150. It showed that the surface area decreased with the increase of calcination temperature, which was attributed to the agglomeration of pyrite cinder particles at high calcination temperature. Many studies have shown that high surface area and rich pore structure are beneficial to improve the cyclic activity of OC [50,51]. The porous microstructure can promote the diffusion of reaction gas into OC. High surface area is also conducive to the adsorption of reactants on the surface of OC.

Fig. 3 showed the characterization results of surface microstructure of the three pyrite cinder samples. For Pc-950 sample, obvious pores could be observed on the surface, indicating that the sample

presented rich pore structure. Moreover, agglomeration for the surface grains of Pc-950 occurred, which was because the content of iron oxide in pyrite cinder was as high as 65.5% [45], thus making pyrite cinder easy to sintering at high calcination temperature and resulting in agglomeration of surface grains. With the increase of calcination temperature to 1050 °C, the agglomeration of grains on the surface of pyrite cinder was more obvious, leading to forming many large grains. When the calcination temperature continued to rise to 1150 °C, the surface pores of Pc-1150 were disappeared. A dense surface structure was formed for this sample. This microstructure was disadvantageous to the gas-solid reaction in cyclic reactions. When the temperature exceeds the Tamman temperature (half of the temperature of melting point), the surface grains of material are much easier to merge and grow [52]. The melting point of Fe<sub>2</sub>O<sub>3</sub> is 1565 °C. Therefore, the high content of active components in pyrite cinder resulted in a serious agglomeration under high calcination temperature. The characterization results in Fig. 3 were completely consistent with that in Fig. 2(b). It indicated that the pyrite cinder presented relatively dense structure after high temperature calcination.

Fig. 4 displayed the XRD characteristics and particle crushing strength of fresh pyrite cinder samples. In Fig. 4(a), all the three samples were composed of CaSO<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>. With the increase of calcination temperature, the intensity of CaSO<sub>4</sub> diffraction peaks decreased gradually, which was attributed to the

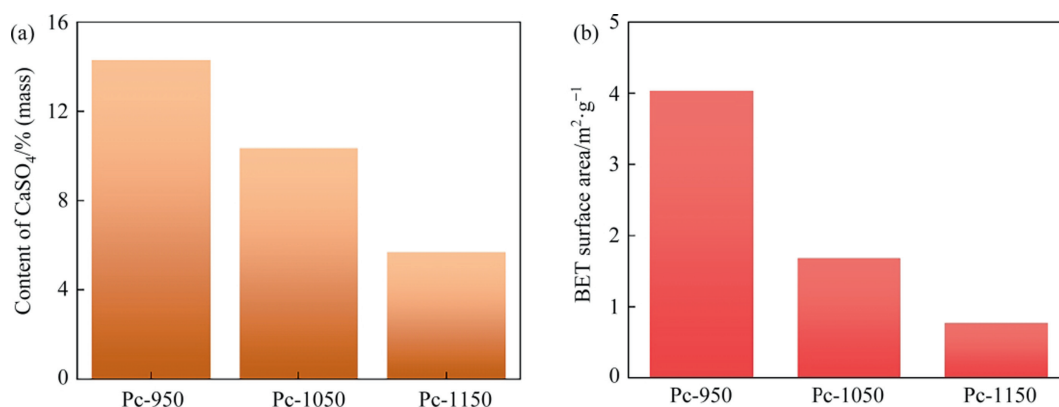


Fig. 2. (a) CaSO<sub>4</sub> content from XRF analysis and (b) BET surface area of fresh pyrite cinder samples.

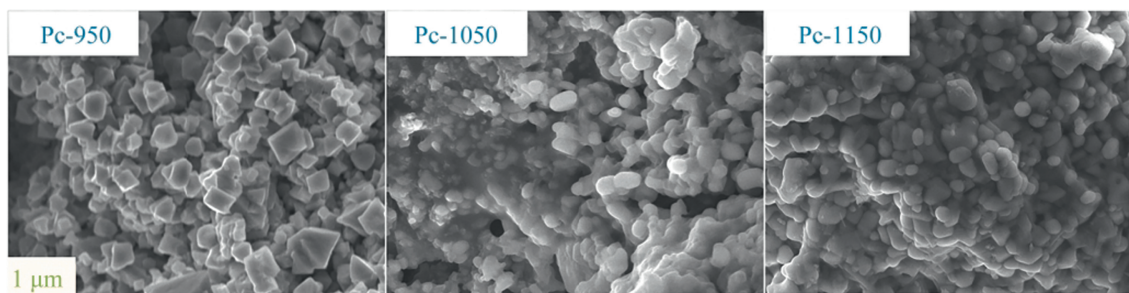


Fig. 3. Surface microstructure of fresh pyrite cinder samples.

decomposition of  $\text{CaSO}_4$  at high temperature. In addition, the intensity of diffraction peaks of  $\text{Fe}_2\text{O}_3$  in XRD patterns increased with the increase of calcination temperature. It is because high calcination temperature can improve the crystallinity of crystal phase. Due to the low content of  $\text{CaSO}_4$  in Pc-1150 sample, the diffraction peak intensity of  $\text{CaSO}_4$  in the XRD pattern of this sample significantly decreased. Fig. 4(b) provided the crushing strength of pyrite cinder particles. In the CLC reaction system, the oxygen carrier particles need to be circulated between the fuel reactor and air reactor. In order to avoid the breakage of oxygen carrier particles by the collision, the oxygen carrier particles should possess sufficient mechanical strength to maintain a complete physical structure in reaction system. The crushing strength of the three samples are 1.10 N (Pc-950), 2.11 N (Pc-1050) and 3.24 N (Pc-1150), respectively. It suggested that increasing the calcination temperature could effectively improve the crushing strength of pyrite cinder particles. It was attributed to that the high temperature calcination decreased the porosity and formed a compact structure for the pyrite cinder. The increase of crushing strength could enhance the attrition resistance of pyrite cinder particles in fluidized-bed system.

### 3.2. Reactivity of pyrite cinder samples

For studying the effect of calcination temperature on the reactivity of pyrite cinder, the reactivity of pyrite cinder samples were investigated in the 1st reduction process of  $\text{CH}_4$  CLC.

Fig. 5 displayed the redox activity of different pyrite cinder samples. It can be found that the  $\text{CO}_2$  concentration increase rapidly for both Pc-950 and Pc-1050, which indicate that the two samples present high reactivity with  $\text{CH}_4$ . The peak values of  $\text{CO}_2$  concentration for Pc-950 and Pc-1050 are 4.6% (vol) and 4.5%

(vol), respectively. For Pc-950 sample, the concentration of  $\text{CH}_4$  increased rapidly after 200 s. Pc-1150 sample presented relative low  $\text{CO}_2$  concentration with a peak value of 2.4% (vol). For the Pc-1150, the concentration of  $\text{CH}_4$  increased rapidly after the beginning of the reaction, indicating that the reaction activity of the sample with  $\text{CH}_4$  was low, resulting in a large amount of  $\text{CH}_4$  not participating in the reaction. It suggested that the lattice oxygen in Pc-1150 was inactivity and presented low reactivity with  $\text{CH}_4$ . In Fig. 5(a), (b) and (c), the content of by-products ( $\text{CO}$ ,  $\text{H}_2$ ) in the product gas for the three samples were very low, which meant that the reacted  $\text{CH}_4$  was completely converted into  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . In Fig. 5(d), the  $\text{CH}_4$  conversion of Pc-950 and Pc-1050 are 88.13% and 85.27%, respectively. It indicated that Pc-950 sample possessed the highest  $\text{CH}_4$  conversion, which was attributed to this sample exhibited the highest surface area. However, Pc-1150 sample presented the lowest  $\text{CH}_4$  conversion (58.57%), which was mainly ascribed to the severe grains agglomeration after high temperature calcination. In addition,  $\text{CaSO}_4$  also has the ability to provide lattice oxygen in the cyclic reactions. The low content of  $\text{CaSO}_4$  in Pc-1150 sample could reduce the  $\text{CH}_4$  conversion. The  $\text{CO}_2$  selectivity of the three samples were close to 100%. This showed that the lattice oxygen in pyrite cinder possessed excellent  $\text{CO}_2$  selectivity in  $\text{CH}_4$  CLC process. The similar results have been found in our previous study [45].

### 3.3. Redox stability test of fresh pyrite cinder samples

Fig. 6 showed the effect of calcination temperature on the cyclic stability and attrition rate of pyrite cinder samples. By comparison, the  $\text{CH}_4$  conversion and  $\text{CO}_2$  selectivity of the three samples can remain stable in 20 consecutive cycles. Among these samples, both Pc-950 and Pc-1050 maintained high  $\text{CH}_4$  conversion (around 85%)

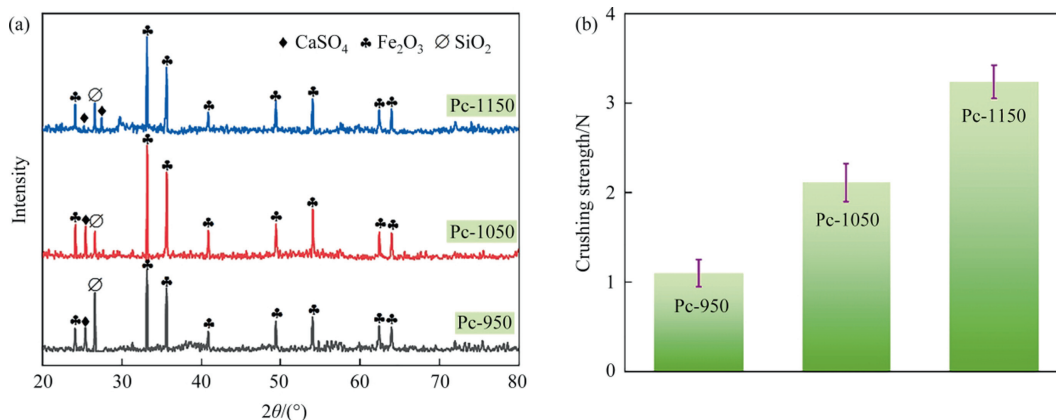
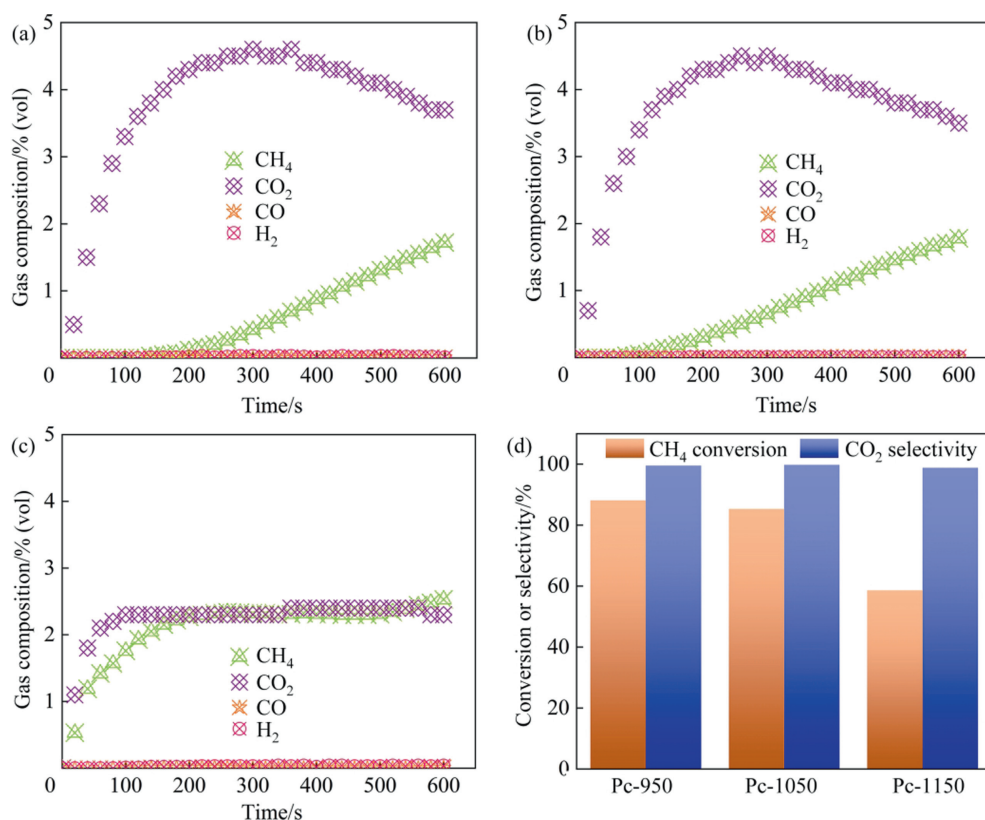


Fig. 4. (a) XRD patterns and (b) Particle crushing strength of fresh pyrite cinder samples.



**Fig. 5.** Evolution of gas composition for pyrite cinder samples in the 1st reduction process: (a) Pc-950, (b) Pc-1050, (c) Pc-1150. (d) CH<sub>4</sub> conversion and CO<sub>2</sub> selectivity of different pyrite cinder samples.

during the whole experiment process. The CH<sub>4</sub> conversion of Pc-950 sample was slightly higher. The Pc-1150 sample possessed the lowest CH<sub>4</sub> conversion in the successive reactions. The CO<sub>2</sub> selectivity of the three samples were maintained around 100%, indicating that pyrite cinder could keep stable reactivity and CO<sub>2</sub> selectivity in the continuous cycle reactions. It showed that the calcination temperature only affected the CH<sub>4</sub> conversion of pyrite cinder and had little effect on the cycling stability and CO<sub>2</sub> selectivity of pyrite cinder in CH<sub>4</sub> CLC.

Fig. 6(d) presented the attrition rate of pyrite cinder samples in continuous cycle reactions. It indicated that the attrition behavior of the three samples were similar in the whole reactions. The samples presented the largest attrition rate in the initial five cycles. The attrition rate of the three samples decreased with the number of cycles. After 20 cycles, the attrition rate of Pc-950, Pc-1050 and Pc-1150 are 0.19%, 0.10% and 0.05%, respectively. It indicated that the attrition resistance of pyrite cinder was improved by increasing the calcination temperature. The high irregularity and roughness of the surface for fresh samples often result in the collision of sample particles in the fluidized-bed reactor, which lead to high attrition rate for fresh samples. As the number of redox cycle increased, the surface of pyrite cinder particles became smooth and the attrition rate decreased accordingly. The similar phenomenon has also been reported in other studies [46,53]. According to the results of Fig. 4(b), high calcination temperature increased the crushing strength of pyrite cinder. Therefore, the Pc-1150 sample showed the lowest attrition rate. The order of the attrition resistance for the three samples is: Pc-1150 > Pc-1050 > Pc-950.

The XRD and SEM results of the samples after 20 cycles were given in Fig. 7. In Fig. 7(a), the XRD patterns of the three samples were basically the same, which only contained the diffraction peaks of Fe<sub>2</sub>O<sub>3</sub> and CaSO<sub>4</sub>. The diffraction peak of CaSO<sub>4</sub> was not

observed, which was attributed to the decomposition of CaSO<sub>4</sub> in the redox cycles. According to the actual composition of pyrite cinder, the content of Fe<sub>2</sub>O<sub>3</sub> is much higher than that of CaSO<sub>4</sub>. Although CaSO<sub>4</sub> can provide lattice oxygen at high temperatures, the content of CaSO<sub>4</sub> in pyrite cinder is relatively less. In addition, CaSO<sub>4</sub> was unstable and decomposed after multiple cycles. So, CaSO<sub>4</sub> was not the main active component in pyrite cinder. Compared with the results in Fig. 3, the surface microstructure of Pc-950 has changed after 20 cycles. However, the 20th cycled Pc-950 still maintained small grain size and porous structure. For Pc-1050, the surface of the 20th cycled sample showed compact structure without pores. After 20 cycles, the Pc-1150 presented a very dense surface microstructure. Although the surface microstructure of both Pc-1050 and Pc-1150 changed significantly during the cyclic reactions. The two samples could maintain stable activity and cycle stability in continuous cycle reactions (Fig. 6). The lattice oxygen in pyrite cinder revealed high redox activity, and the thermal sintering in redox cycles had no obvious effect on the cyclic reactivity of pyrite cinder. When pyrite cinder was used as OC in CH<sub>4</sub> CLC process, the CO<sub>2</sub> selectivity was not affected by the calcination temperature of pyrite cinder and was still close to 100%. When considering the reactivity and attrition resistance of pyrite cinder, 1050 °C is the suitable calcination temperature.

Based on the above results, increasing the calcination temperature was beneficial to the attrition resistance of pyrite cinder. However, a contradiction between improving the attrition resistance and maintaining high cyclic reactivity of pyrite cinder is existed. When the calcination temperature is raised to 1150 °C, serious grain aggregation was appeared for pyrite cinder, which reduced the surface area and pore volume of the material, thus decreasing the cyclic reactivity of pyrite cinder. In industrial applications, oxygen carrier particles need to be circulated in circulating

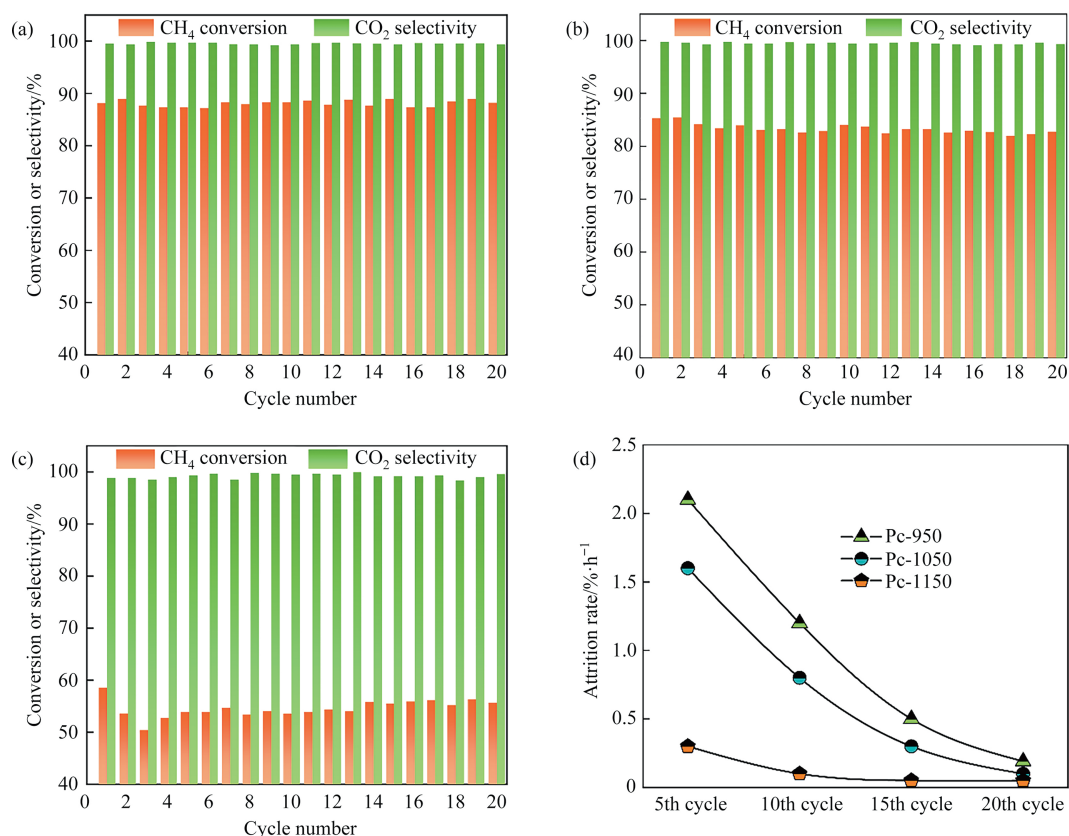


Fig. 6. Redox stability of pyrite cinder samples in continuous 20 cycles: (a) Pc-950, (b) Pc-1050, (c) Pc-1150. (d) Attrition rate of different pyrite cinder samples.

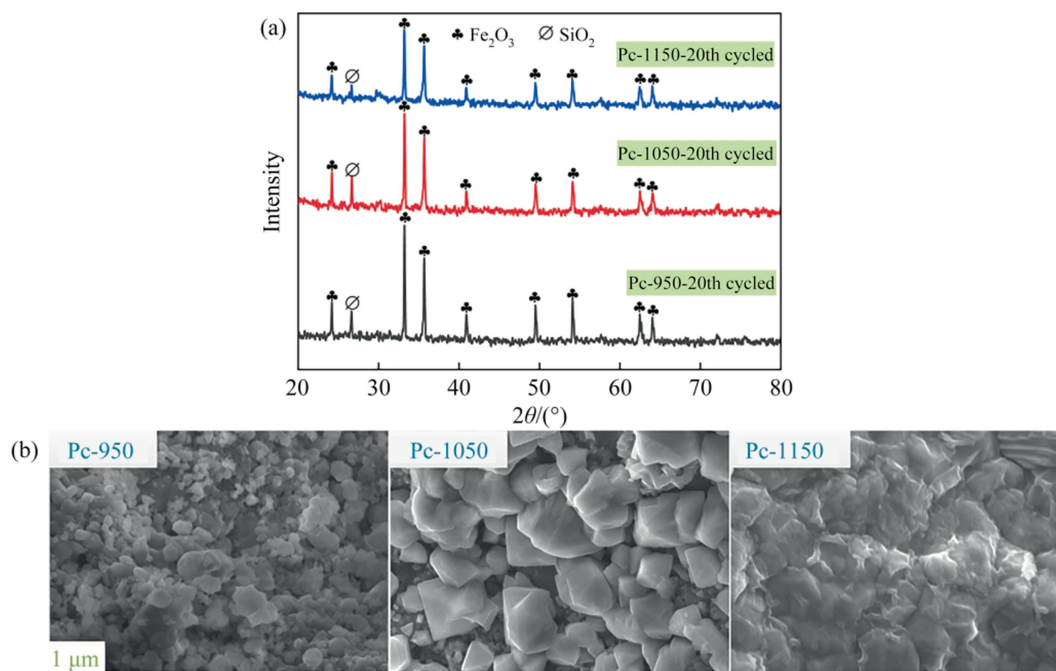


Fig. 7. (a) XRD and (b) SEM results of pyrite cinder samples after 20 cycles.

fluidized-bed reactors for a long time, which require oxygen carrier particles must have sufficient mechanical strength to deal with friction, thermal stress and volume stress [54]. Otherwise, the oxygen carrier particles experience serious attrition, which decrease

the efficiency of the whole reaction system. If the mechanical strength of particles is improved by directly increasing calcination temperature, the cyclic reactivity of pyrite cinder is decreased. Then large holding capacity of pyrite cinder is needed in reactor,

thus increasing the reactor size and the oxygen carrier cost. Therefore, it is necessary to take effective measures to obtain pyrite cinder particles with enough mechanical strength and high redox activity in the near future study.

#### 4. Conclusions

The effect of calcination temperature on the cyclic reactivity of pyrite cinder were studied deeply. The results indicated that the calcination temperature presented an important influence on the physico-chemical properties and reactivity of pyrite cinder. With the increase of calcination temperature, the surface structure of pyrite cinder became dense and the pores were gradually disappeared, thus leading to high crushing strength of pyrite cinder particles. Among the studied samples, the pyrite cinder sample after high temperature calcination (1150 °C) exhibited the lowest CH<sub>4</sub> conversion (58.57%) and highest crushing strength (3.24 N). Both Pc-950 and Pc-1050 presented higher CH<sub>4</sub> conversion of around 85%. It indicated that high calcination temperature could decrease the redox activity of pyrite cinder. However, calcination temperature had little effect on the CO<sub>2</sub> selectivity of pyrite cinder in CH<sub>4</sub> CLC. All the samples displayed a CO<sub>2</sub> selectivity of approximate 100%. In this study, 1050 °C was the best calcination temperature for pyrite cinder. In redox cycles, surface sintering occurred for the most studied samples. Nonetheless, the cyclic reactivity of pyrite cinder was not affected by sintering, which indicated that the lattice oxygen in pyrite cinder possessed high reactivity.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Supplementary Material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cjche.2021.11.015>.

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