



Effects of CO₂ and heating rate on the characteristics of chars prepared in CO₂ and N₂ atmospheres



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HIGHLIGHTS

- CO₂ influences the char structures in the initial stage of oxy-fuel combustion.
- The char becomes more condensed and oxygenated with increasing CO₂ concentration.
- Fast-heating char is less condensed but more oxygenated than slow-heating char.
- O-holding capacity of fast-heating char in CO₂ decreases with char condensation.

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ABSTRACT

This paper reports an experimental study to assess the effects of CO₂ and heating rate on the characteristics of the chars formed in CO₂ and N₂ atmospheres in the initial stage of oxy-fuel combustion. The chars of a Chinese bituminous coal were prepared in a fluidised-bed/fixed-bed reactor at 900 °C and their chemical structural features were further characterised with FT-Raman spectroscopy. Our results indicate that changes in CO₂ concentration influence not only the char yield but also the char chemical structural features, which become more condensed and increasingly oxygenated with increasing CO₂ concentration. The chars formed in CO₂ atmosphere at fast heating rates are less condensed but more oxygenated than those formed at a slow heating rate. The O-holding capacity of the fast heating char decreases with the aromatic condensation as the char is held in CO₂ atmosphere at 900 °C.

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1. Introduction

Among the most recent developments for reducing CO₂ emissions, oxy-fuel combustion is considered to be one of the most promising and cost-effective CO₂ capture strategies for coal combustion systems. It is suitable for both retrofitted and new coal-fired power plants [1–3]. Research, development and demonstration of oxy-fuel combustion technologies have been advancing in recent years [1–5]. However, there are still fundamental issues and technological challenges that must be addressed before this technology can reach its full potential [1,6–12].

Combustion can be conceptually viewed as the pyrolysis of coal followed by the combustion of the residual char and volatiles. Although pyrolysis is a quick process and virtually completes below 900 °C, the pyrolysis process will greatly influence the whole life of the coal particles from their injection to burnout in

the furnace. In view of the differences in thermal and chemical properties between N₂ and CO₂, the replacement of N₂ by CO₂, which is typical of oxy-fuel combustion, could make the behaviour of coal in the initial stage of oxy-fuel combustion quite different from that in conventional air combustion and thus may affect nearly all subsequent processes of coal particle combustion [13–18]. The key is to understand the roles of CO₂, especially in the initial stage of oxy-fuel combustion. The char structure is particularly important for the fluidised-bed (oxy-fuel) combustion that takes place at much lower temperatures than the pf combustion.

Unfortunately, the reports in this area do not always agree with each other. Some observed that the volatile yields in CO₂ were higher than those in N₂ and attributed the difference to the char-CO₂ gasification [19,20]. However, others observed a lower mass release [21] and a lower pyrolysis rate [22,23] in CO₂ and suggested that CO₂ could be involved in the cross-linking reactions, which reduced the swelling of the particles and inhibited the volatile release [21]. Furthermore, it has also been reported [24] that no remarkable differences existed in char

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morphology and volatile yields in N₂ and CO₂, concluding that a shift from air to oxy-fuel combustion would not alter the pyrolysis process significantly.

There are clearly needs for further study on this aspect, especially in the initial stage of oxy-fuel combustion. Although some efforts have been made to compare the physical structure of the chars between oxy-fuel and conventional conditions [10,12,19–21], the effects of CO₂ on the chemical structural features of the chars formed in the initial stage of oxy-fuel combustion remain unclear. Quantitative information about the evolution of char chemical structure in the initial stage of oxy-fuel combustion is vital for the fundamental understanding of oxy-fuel combustion process but very little information is available in literature. In addition, the heating rate of coal particle is a key parameter influencing the pyrolysis products and the char characteristics. Despite the large number of publications that have been devoted to oxy-fuel combustion in recent years [1–5], there are few works [7,8] studying the effects of heating rates on oxy-fuel combustion. Neither is there any reference focusing on the potential effects of heating rate on the chemical structure of chars generated in the initial stage of oxy-fuel combustion.

This study focuses on the initial stage of oxy-fuel combustion process of a bituminous coal. The experiments have been carried out in a fluidised-bed/fixed-bed reactor at 900 °C and the chars prepared in the mixtures of CO₂ and N₂ have been collected and further characterised with a FT-Raman spectrometer in order to obtain the quantitative information about the chemical structural features of the chars. The potential effects of CO₂ and heating rate on the characteristics of chars formed in the initial stage of oxy-fuel combustion have been assessed through the char yields and the char structure.

2. Experimental

2.1. Coal sample preparation

A Chinese bituminous coal was used in this study, which was one of the candidate coals for an oxy-fuel combustion full-scale 35 MW demonstration boiler. The coal was partially dried at <35 °C and then ground and sieved to obtain a sample of particle sizes between 106 and 150 μm. The properties of the coal sample are shown in Table 1. The ash yield of the coal sample on an air dried basis is about 31.4 wt%.

2.2. Char preparation

2.2.1. Char prepared at fast heating rates

The preparation of char was carried out in a quartz fluidised-bed/fixed-bed reactor, which has been described in detail elsewhere [25,26]. Briefly, two frits were installed in the reactor body. The lower frit served as a support for the bed material (silica sand) and as a distributor for the fluidising gas. The “fluidised-bed” feature refers to the silica sand bed, which was fluidised by the incoming gas. During an experiment, some char particles would be elutriated out of the silica sand bed at high temperatures due to the upwards fluidising gas and feeding gas fed into the reactor. Therefore, a top frit was installed at the upper part of the reactor body to prevent char from being elutriated out of the reactor. Any char particles which were elutriated out of the silica sand bed due to the lighter density of chars than the sand would be kept

underneath the top frit in the freeboard. Therefore, a “fixed-bed” was formed underneath the top frit.

The reactor was heated up to the required reaction temperature using an external electrical furnace. About 60 g of silica sand (300–355 μm) was placed on the lower frit. After the temperature inside the reactor was stabilised at 900 °C for at least 15 min, about 1.5 g of coal was continuously entrained with the feeding gas and fed into the hot silica sand bed (at a rate of around 150 mg min⁻¹) through a water-cooled probe. The coal particles were believed to be heated up at a rate in excess of 10³ ~ 10⁴ K s⁻¹ [27,28]. These reaction conditions have been chosen to achieve a sufficiently low temperature at which combustion is slow enough for significant amounts of char to be collected but high enough to allow the reactions involving CO₂ and char to progress significantly [13].

After the pre-set amount of coal had been fed into the reactor, the whole reactor was lifted out of the furnace immediately for the reactor to cool down naturally, which is termed as a “zero holding time” experiment. For an experiment with “holding time”, the reactor was further left inside the furnace for a pre-set amount of “holding time” after the coal feeding was completed and before it was lifted out of the furnace. Char samples were prepared in CO₂ and N₂ mixtures, including 100% CO₂, 90% CO₂ plus 10% N₂, 80% CO₂ plus 20% N₂, 70% CO₂ plus 30% N₂, 50% CO₂ plus 50% N₂ and 100% N₂. The total flow rate of fluidising gas and feeding gas was 2 L min⁻¹ (measured under ambient conditions). In all cases, CO₂ and N₂ were completely pre-mixed. The mass flow controllers (MFC) for the fluidising and feeding gas were calibrated for each experiment using a digital bubble flow meter.

2.2.2. Char prepared at slow heating rate

For comparison purposes, chars were also prepared in the fluidised-bed/fixed-bed reactor at a slow heating rate. About 60 g of silica sand and 1.5 g of coal, which were the same respectively as those in the fast heating experiments, were firstly fed into the reactor at room temperature before the reactor was heated up to 900 °C at a heating rate around 30 °C min⁻¹. When the temperature inside the reactor reached 900 °C, the whole reactor was lifted out of the furnace immediately and the experiment was termed as a “zero holding time” experiment. A “holding time” experiment refers to that where the reactor was further left inside the furnace for a pre-set amount of time after the temperature reached 900 °C.

The atmospheres for preparing chars with a slow heating rate included 100% CO₂, 70% CO₂ plus 30% N₂ and 100% N₂ at the same flow rates as those in the fast heating experiments.

The char yields were determined by the differences in the mass of the reactor and coal/char before and after each experiment. In order to collect char for further analyses, all the outlets of the reactor were blocked except the fluidising gas inlet and a quartz probe underneath the top frit. A cellulose thimble was connected to the quartz probe underneath the top frit. The fluidising gas inlet was connected to compressed air and the flow rate was adjusted until the char particles could be elutriated out of the sand bed due to the lighter density of char and collected in the thimble. The collected char was placed into a plastic vial and stored in a freezer until the char was required for analyses.

2.3. Char characterisation

The quantification of the char characteristics, especially the chemical structural features of char, from low-rank coal has been a major challenge [29]. Many commonly used analytical techniques, such as Fourier transform infrared (FT-IR) and X-ray diffraction (XRD), could only provide very limited information about char structure [27]. Raman spectroscopy has been widely used to characterise the structural features of carbonaceous matters and demonstrated as a very powerful tool to quantitatively

Table 1
Properties of bituminous coal sample in weight percentage (dry and ash free basis).

Volatile matter	C	H	N	S	O (by diff.)
30.9	84.8	3.6	1.2	0.9	9.5

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