Kinetic Study on Coal Char Combustion in a Microfluidized Bed

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ABSTRACT: The intrinsic kinetics of char combustion were commonly investigated using thermogravimetric analysis (TGA) in previous works at low temperatures to prevent oxygen limitations and temperature deviation. However, the low temperatures caused excess test time because the reaction rate was too slow. In this study, the microfluidized bed (MFB), which has effective heat and mass transfer, was used to investigate the intrinsic kinetics of char combustion at a higher temperature within less time. TGA was used to check the reliability of the MFB. The results suggested that, for fine particles (74–100 μm), the particle temperature deviation and gas distortion could be disregarded in the MFB. For four coal samples, the activation energy measured by the MFB was similar to the result measured by TGA, but the test time using the MFB was greatly shortened as a result of the higher temperatures. The kinetic parameters measured by the MFB were used to predict the char oxidation rate. The predicted rate fit the TGA experiment well at lower temperatures. These results demonstrated the reliability of the MFB and TGA measurements. However, at higher temperatures, the combustion rate in TGA was limited by oxygen diffusion, suggesting that the TGA measurement should be carried out at relatively low temperatures to prevent oxygen diffusion limitation. Instead, the MFB measurement was still valid at higher temperatures as a result of the effective mass and heat transfer.

1. INTRODUCTION

Char combustion plays an important role in the overall coal combustion process. It is the rate-determining step, taking up 70–90% of the whole combustion time.1–3 A comprehensive understanding of char combustion is helpful for the design and operation of the actual combustion systems.1–3 Smith5 proposed an intrinsic reaction model, in which the combustion rate of pulverized coal in actual combustion systems was estimated on the basis of the intrinsic reaction rate. However, different types of coal have a wide range of intrinsic reaction rates. Hargrave et al.6 took the petrographic structure into account and correlated the intrinsic reactivity with coal properties. Hurt et al.7 pointed out that the char intrinsic reactivity decreased by a factor of 100–1000 after thermal annealing in pulverized-coal-fired boilers (1400–1800 K). The intrinsic reaction rate plays an important role in predicting char burnout; therefore, it is essential to investigate intrinsic char reactivity in the absence of the heat and mass transfer limitations.2,3,6

Thermogravimetric analysis (TGA), which is a common instrument in thermal analysis, has been widely used to estimate char oxidation kinetics. There are two main methods adopted by TGA to measure char intrinsic reactivity: (i) non-isothermal method, where char is heated at a constant rate,10–12 and (ii) isothermal method, where char reacts with oxygen at a constant temperature.13–15 Char combustion is a rapid exothermic reaction, and the released heat during the test would cause the particle temperature to differ from the preset temperature.8,16 Char samples are placed in a crucible during the TGA test. The pile-up effect inhibits the transport of oxygen to the char surface.17 To minimize oxygen diffusion limitations and temperature deviation, char must be tested at relatively low temperatures.18 At such low temperatures (<773 K), it may take several hours for char to reach 50% conversion. In summary, the disadvantages mentioned above have restricted the further application of TGA to measure intrinsic kinetics of char combustion at higher temperatures.

On the basis of the Arrhenius equation, if the reaction temperature increased from 693–773 to 833–923 K in the chemically controlled regime, the test time would decrease by a factor of 100. The activation energy of char combustion was evaluated as 160 kJ/mol, as reported in previous works.6,19 Then, the problem is how to prevent temperature deviation, oxygen diffusion limitations, and unstable atmosphere at the initial combustion stage after increasing the reaction temperature.

The microfluidized bed (MFB), which has the advantages of mass and heat transfer and instant online feeding samples, has been applied to the investigation on the kinetics of gas–solid reactions for various solid fuels.20–29 During lignite char gasification at a constant temperature, the reaction rate in the MFB was higher than that in TGA, indicating the less diffusion limitation in the MFB.21–23 Yu et al.25 investigated combustion kinetics of graphite powder at a wide range of temperatures from 973 to 1273 K. Mueller et al.23 found that the measured kinetic parameters of lignite char gasification between the MFB and TGA were different. This was because the heating rate of the MFB was much higher. The produced char showed high reactivity and experienced less thermal deactivation. As shown in previous works, it was possible for the MFB to measure the intrinsic reactivity of char combustion at higher temperatures within less time. However, the char reaction with O2 was faster than the char reaction with CO2 by a factor of 100 000,30 suggesting that char combustion was more easily limited by...
oxygen diffusion and heat transfer than char gasification or graphite combustion. Fennell et al.\textsuperscript{31} and Ramos et al.\textsuperscript{32} investigated char combustion below 1173 K in a fluidized bed, and the combustion rate was found to be limited by oxygen diffusion, owing to the high reactivity of char. Char reacted with oxygen around the char surface so quickly that the concentration of gas products varied rapidly. It was then difficult to measure the real-time carbon consumption rate by analyzing the outlet gas product concentration, which might have become distorted after going through the bed. In addition, the rapid exothermicity of char combustion led to uncertainty in the particle surface temperature and active sites.\textsuperscript{31–33} Ramos et al.\textsuperscript{32} reported that the particle surface temperature was 41–82 K higher than the bed temperature when different types of char were combusted at different atmospheres in the fluidized bed.

Although MFB has been successfully used to measure intrinsic parameters of char gasification, the high reaction rate during char combustion renders it difficult to prevent oxygen diffusion limitations. The gas concentration distortion makes it hard to determine the real-time oxidation rate at the outlet. The rapid exothermicity creates difficulty in ensuring a constant temperature of the particle surface. In this way, the intrinsic kinetics of char combustion using the MFB require further investigation.

This paper focused on studying intrinsic kinetics of char combustion in the absence of mass and heat transfer limitations using the MFB at higher temperatures, at which the test time was greatly shortened. The particle surface temperature and gas concentration distortion in the MFB were also studied to obtain the reliable kinetic data. TGA at lower temperatures was used to check the reliability and practicability of the MFB. Four Chinese coals were selected for this study. The kinetic parameters and behavior of coal char combustion obtained using two instruments were compared.

2. EXPERIMENTAL SECTION

2.1. Char Samples. Four Chinese coals, Shenhua (SH), Huolinhe (HLH), Pingdingshang (PDGS), and Heshan (HS), were selected as samples for this study. Their proximate analyses are given in Table 1. The volatile content of four coal samples varied from 13.85 to 37.19 wt %, and the ash content varied from 7.59 to 40.20 wt %. Before the test, the raw coal was ground to a desired size. Then, the fine sample was pyrolyzed in a tube furnace at 1273 K for 2 h to completely remove the volatile. Because the temperature and time of pyrolysis were markedly higher than those of later char combustion, char deactivation owing to thermal annealing during combustion was prevented.\textsuperscript{13,5}

2.2. Kinetic Test by the Isothermal Method. 2.2.1. Test in the MFB. The schematic diagram of the MFB is shown in Figure 1. This system consists of three key parts: (i) a quartz reactor with a diameter of 20 mm, a height of 45 mm, and two gas distributors, (ii) mass spectrometry (Ametek Dycor LC-D 100) analyzing gas products CO and CO\textsubscript{2}, with the capillary with an inner diameter of 50 \( \mu \)m used to collect gas products, in which this sampling method was found to prevent gas distortion during gas analysis, and (iii) the online feeding device, including the solenoid valve and programmable logic controller (PLC). A small amount of pulse gas blew the char sample placed in the injector into the reaction zone instantly. A total of 3 g of neutral alumina particles with diameters of 74–150 \( \mu \)m was used as the bed material, and a thermocouple was inserted into the dense zone to measure the bed temperature in real time. At the time of testing, the measured bed temperature was constant.

Before the test, 10 mg of char particles was loaded in the injector. The reactor was then heated to the prescribed temperature ranging from 833 to 1003 K. The mixture gas of 80 vol % Ar and 20 vol % O\textsubscript{2} was introduced into the reactor at the rate of 800 standard cubic centimeters per minute (SCCM). Such high-velocity gas flow ensures a high excess air coefficient and minimizes gas diffusion.\textsuperscript{5} When the temperature and gas flow rate were stable, char particles were blown into the dense zone, where char reacted with oxygen, by a small amount of pulse gas. The variation of the gas product concentration of CO and CO\textsubscript{2} at the outlet was measured by online mass spectrometry. When char burned out, a new sample was loaded in the injector and the next set of experiments was started straightforwardly. There was no need to cool the hot furnace and reactor. In this study, the test temperatures of different coals were not the same. For example, char combustion experiments of SH coal were conducted between 833 and 923 K, but those of HS coal were carried out between 913 and 1003 K. This was related to the various reactivities of different coals. The reactivity of HS coal was much worse than that of SH coal. The HS char combustion rate was still chemically controlled with raising the test temperature to 1003 K, while the combustion of high-reactive SH char was easily affected by oxygen diffusion limitation at 1003 K.

2.2.2. Test in TGA. Kinetics of char combustion were also conducted in TGA (PerkinElmer) using the isothermal method. To prevent limitations of oxygen diffusion, char combustion using TGA was conducted at lower temperatures. For each test, 10 mg of the char sample was placed on the platinum crucible and heated to the reaction temperature at 20 K/min at Ar flow. When the temperature was stable, the Ar/O\textsubscript{2} mixture at the rate of 200 SCCM was introduced. Char began to react with O\textsubscript{2}. The weight loss data of the sample as the reaction proceeded were recorded. When char burned out, the furnace should be cooled to change the sample.

2.3. Kinetic Approach. The reaction rate was given

\[
\frac{dX}{dt} = kf(X) = A \exp\left(-\frac{E}{RT}\right)f(X)
\]  

(1)

where \( X \) is the carbon conversion degree, \( t \) is the reaction time, \( k \) is the reaction rate constant, \( f(X) \) is the reaction model function, \( E \) is the activation energy, \( A \) is the pre-exponential factor, and \( T \) is the reaction temperature. For TGA, the carbon conversion degree \( X \) was calculated using the weight loss data as shown below

![Figure 1. Schematic diagram of the microfluidized bed.](image-url)
The carbon conversion degree versus reaction time for different sizes of char.

![Figure 2](image)

3. RESULTS AND DISCUSSION

3.1. Measurement Condition in the MFB. 3.1.1. Particle Size of Coal. To measure the intrinsic kinetics of char combustion, coal samples were grounded to 74–100 μm in this study. The internal diffusion limitation could be disregarded for fine particles (<100 μm) as a result of the short intraparticle diffusion trajectories. Figure 2 shows the carbon conversion degree versus reaction time for different sizes of SH char in the MFB. The combustion temperature was set as 893 K. Three different particle sizes (22–45, 45–74, and 74–100 μm) were compared. The results show that the carbon consumption rate did not increase with decreasing the particle size, indicating that the internal diffusion limitations could be disregarded when the particle size was less than 100 μm.

![Figure 3](image)

The terminal velocity $u_t = 15.83$ cm/s, and the relevant flow rate was about 1120 SCCM. Figure 3 shows the carbon burnout curve at different gas flow rates.
heat, and the heat exchange with bed material. Thus, the particle temperature was calculated using the following heat balance for a single particle:

\[
\frac{dT_p}{dt} = \frac{m}{C_p} \left( Q_{\text{in}} - hS(T_p - T_b) - \varepsilon \sigma (T_p^4 - T_b^4) - Q_{\text{coll}} \right)
\]

where \(T_p\) and \(T_b\) are the particle temperature and bed temperature, respectively, \(m\), \(C_p\), and \(A\) are the mass, specific heat capacity, and external surface area of the char particle, respectively, \(h\) and \(\varepsilon\) are the emissivity and Stefan–Boltzmann constant, respectively, \(T_b\) is the convective heat transfer coefficient and is given in a previous work, \(Q_{\text{in}}\) is the heat release during particle combustion and reported in many studies, and \(Q_{\text{coll}}\) is the heat exchange with bed material and calculated on the basis of the research of Sun and Chen.\(^{43}\)

Figure 4 shows the burning char temperature versus the reaction time when the bed temperature is set at 1000 K. The bed temperature was a constant with the reaction proceeding as the thermocouple was measured. These results show that the particle (100 \(\mu\)m) was rapidly heated within 0.1 s.\(^{25}\) As the char burnout proceeded, the calculated particle temperature was almost the same as the bed temperature and varied only slightly. This was related to the rapid heat transfer for the fine particle (<100 \(\mu\)m). Thus, the particle temperature in the MFB was considered as the bed temperature measured using the thermocouple, which was inserted into the dense zone.

In the MFB, the reaction occurred on the char surface but the gas products were analyzed at the outlet using mass spectrometry. The distortion of the gas concentration signal after gas mixing in the bed reactor might affect the measurement of the high combustion rate. The gas distortion in the MFB was simulated using computational fluid dynamics (CFD). A gas concentration wave at the rate of 8 cm/s was input at the air distributor to simulate reaction on the char surface. The gas velocity was determined using the gas flow rate and diameter of the bed reactor. The gas concentration wave went through the MFB reactor and then was output at the outlet to simulate the capillary sampling of mass spectrometry. The simulation results are displayed in Figure 5. Figure 5a suggests that the gas concentration wave of the output was different from that of the input when the wave period was 1 s. When the wave period was raised to 10 s, it was found that the distortion of the gas concentration was negligible, as shown in Figure 5b. The characteristic combustion time exceeded 10 s at temperatures below 923 K; therefore, the gas distortion in the MFB could be disregarded and the measured gas concentration at the outlet could reflect the real-time combustion rate.

To further verify the reliability of the measured gas concentration at the outlet, experiments were conducted. Before the experiment, the reactor was heated to 773 K and 800 SCCM fluidizing gas was introduced. When the temperature and gas flow were stable, a stream of tracer gas was introduced. The gas concentration of the tracer gas at the inlet and outlet, experiments were conducted. The carbon conversion degree of four coals versus reaction time at different prescribed temperatures is shown in Figure 7. The char reaction time decreased rapidly as the reaction temperature increased because the char oxidation was dependent upon the reaction temperature in the chemical-controlled regime. When the SH coal in panels a and e of Figure 7 is taken as an example, the char reaction time using the MFB at 833–923 K was about 60–500 s. However, the reaction time of SH coal using TGA at 693–783 K increased to 4000–60 000 s. The test time using TGA was about 100 times that using the MFB, and the other three coals showed a similar result, which meant that the test time required in the MFB was far shorter. This was because the combustion temperature in the MFB was higher than that in TGA. For all coals, the test could be completed within less time.

Figure 4. Particle surface temperature in the MFB.

Figure 5. Gas distortion in the MFB by simulation.
Figure 8 displays the char combustion rate versus carbon conversion degree. In both instruments, the char reaction rate first increased and then decreased, owing to the pore enlargement and pore coalescence. During the initial combustion stage, pore enlargement was dominant, leading to an increase in the internal surface. As burnout proceeded, the pore coalescence became dominant and the char combustion rate decreased. For SH coal in panels a and e of Figure 8, the...
maximum reaction rate in both instruments appeared around the conversion degree of 0.33 at different temperatures. For HLH coal in panels b and f of Figure 8, it appeared around the conversion degree of 0.32. For PDSG and HS coals, it appeared around the conversion degree of 0.19 and 0.31, respectively. The char combustion behavior in two instruments was similar. However, the combustion rate using the MFB was 2 orders of magnitude higher than that using TGA because the test temperature in the MFB was higher.

3.3. Char Combustion Kinetics. The char combustion rate first increased and then decreased in the MFB and TGA experiments as a result of pore enlargement and pore coalescence. The random pore model developed by Bhatia and Perlmutter\textsuperscript{36} has been widely used to describe the relationship between the reaction rate and reactive surface area.\textsuperscript{37–39,44} The structure parameter $\Psi$ of each coal was determined according to eq 7. The four coals have different values of $\Psi$. Then, the fitting results of $G(X)$ versus reaction time based on eq 8 are shown in Figure 9. The reaction rate constant $k$ was the slope of the fitting straight line. Table 2 shows the values of the reaction rate constant and structure parameter for four coals. The results suggested that the random pore model fitted the experiment well at all temperatures in the MFB and TGA at the conversion from 0 to 0.95.

When the values of $k$ at different temperatures were determined, the kinetic parameters could be obtained according to eq 9. Figure 10 shows the linear fitting between $\ln k$ and $1000/T$. The calculated values of activation energy $E$ and pre-exponential factor $A$ are summarized in Table 3. For the four coals, the intrinsic activation energy $E$ measured by the MFB...
was similar to that measured by TGA. It could be concluded that the sensitivity of char combustion in the MFB and TGA to temperature was similar. Besides, measured $E$ was within the range of previous studies.\textsuperscript{6,19,42} This result indicated the reliability of the MFB and TGA on measuring intrinsic kinetics of char combustion, but the test time using the MFB was shortened by a factor of 100. The HS coal using TGA was analyzed at 782–863 K, which was a little higher, and the combustion rate was limited by oxygen diffusion. Therefore, $E$ of HS coal measured by TGA was 129.35 kJ/mol, lower than 137.73 kJ/mol measured by the MFB. For the other three coals, $E$ measured by the MFB and TGA was closer or the same. The value of $A$ measured by the MFB was much higher than that by TGA for four coals. This was because the accessible surface with oxygen was different in two instruments. In TGA, char particles were piled up together, while in the MFB, char particles were highly dispersed. Then, more oxygen was transported to the char surface in the MFB. Thus, the accessible area was larger, and $A$ measured by the MFB was higher.

The kinetic parameters $E$ and $A$ of SH coal measured by the MFB were used to predict the char combustion rate in TGA, ignoring the oxygen diffusion limitation. The prediction rate was calculated based on eq 1. The comparison between the experimental data in TGA and model prediction of SH coal at different temperatures is shown in Figure 11. The curve of the prediction rate fits the experimental rate well at 693 and 755 K. The measured values of $E$ and $A$ by the MFB could also be

Figure 9. Linear fitting of $G(X)$ versus reaction time using the random pore model.
applied to the TGA experiment. This result indicated the reliability and practicality of the MFB measurement on combustion kinetics. This also suggested that the oxygen diffusion in TGA was negligible at low temperatures. Therefore, the measurement of TGA at low temperatures was reliable.

Figure 12 shows a comparison of the combustion rate of prediction and experiment at 833 and 863 K. Figure 12a shows that the prediction rate fits the MFB experiment well. However, it was about 2–3 times that of the TGA experiment in Figure 12b. It could be seen that the deviation between prediction and experimental values increased as the reaction temperature in TGA increased. This was because the char combustion rate was limited by oxygen diffusion for the pile-up effect in TGA at higher temperatures. In all, the kinetic parameters of char combustion measured by the MFB were more similar to intrinsic parameters because char combustion in the MFB was chemically controlled at higher temperatures. The char

Table 2. Reaction Rate Constants at Different Temperatures in the MFB and TGA

<table>
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<th>Ψ</th>
<th>instrument</th>
<th>T (K)</th>
<th>k (s⁻¹)</th>
<th>instrument</th>
<th>T (K)</th>
<th>k (s⁻¹)</th>
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Figure 10. Linear fitting between ln k and 1000/T for four coals.

Table 3. Kinetic Parameters Measured by the MFB and TGA

<table>
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<tr>
<th>coal</th>
<th>instrument</th>
<th>E (kJ/mol)</th>
<th>A (s⁻¹)</th>
<th>fitting coefficient</th>
</tr>
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<td>TGA</td>
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<tr>
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<td>2.76 × 10⁴</td>
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</tr>
<tr>
<td></td>
<td>TGA</td>
<td>129.35</td>
<td>5.8 × 10⁴</td>
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</table>

Figure 11. Comparison of the experimental data in TGA and model prediction at different temperatures for SH coal.
combustion rate in TGA was visibly limited by oxygen diffusion as the reaction temperature increased. The poor mass transfer in TGA limited its application in kinetic studies at higher temperatures; thus, the TGA test should be carried out at lower temperatures to avoid the limitation of oxygen diffusion. The MFB provides the homogeneous atmosphere, and char particles were highly dispersed, which strengthened the mass transfer around the particles. Therefore, the MFB could cover the limitations of TGA on measuring char combustion kinetics at higher temperatures as a result of the effective mass transfer.

4. CONCLUSION
The intrinsic kinetics of coal char combustion were studied in the MFB free from mass and heat transfer limitations. The particle temperature for pulverized coal (<100 μm) was equal to the bed temperature and varied little as the reaction proceeded. The gas distortion and secondary reaction also did not affect measuring the carbon consumption rate in real time. A similar test was conducted in TGA at lower temperatures to check the reliability. For the four coal samples, E measured by the MFB was similar to that measured by TGA. However, A measured by the MFB was much higher. This was because accessible surface with oxygen was larger in the MFB as a result of the strengthened mass and heat transfer. The random pore model was used to predict the char reaction rate using the kinetic parameters measured by the MFB. The prediction fits the MFB and TGA experimental data well, indicating the reliability of the MFB and TGA measurements. However, when the TGA test was carried out at higher temperatures, the prediction was about 2 times higher than the TGA result, suggesting that the char reaction rate in TGA was limited by oxygen diffusion as a result of the pile-up effects. For the MFB, the effective mass and heat transfer ensured that char combustion still occurred in the kinetic regime at higher temperatures. The test time was shortened by a factor of 100 compared to TGA.

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**Notes**
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