Co-combustion kinetics characteristics of semi-coke and bituminous coal under a drop tube furnace

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Abstract. Random pore model was employed to calculate the reaction kinetics parameters of SH bituminous coal and Shenmu semi-coke blended fuels under high-temperature entrained flow combustion, and the reaction kinetics parameters was modified by the intrinsic reaction Arrhenius curve obtained from low temperature thermogravimetric experiments. Then the combustion reaction rate and intrinsic reaction order at different blending ratios of semi-coke were determined. The reaction kinetics parameters and carbon conversion rate of blended fuels were investigated based on the pore structure of fuel, the effects of blending ratio of semi-coke, initial partial pressure of oxygen and reaction temperature. The reaction order decreases with the increasing of semi-coke blended ratio except 100% semi-coke combustion has higher reaction order approximately as char pure burning. The distribution of oxygen along the radius of particle was also calculated in this study. In the low oxygen reduction zone, the blending ratio of semi-coke has little effect on the carbon conversion of blended fuel, and the blending effect is enhanced with the increasing of oxygen content. Carbon conversion rate decreases with the increasing of blending ratio and increases with reaction temperature.

1. Introduction

As a byproduct of coal chemical process, semi-coke has a large reserve in China. It is widely used in metallurgy, chemical industry and adsorbent industry [1-4]. However, with the vigorous development of coal chemical industry in recent years, the annual yields of semi-coke have reached hundreds of millions of tons, far exceeding the consumption of traditional semi-coke utilization market [5]. Due to the high calorific value of semi-coke, the practical method of burning semi-coke in power plant boiler combustion has attracted great attentions in recent years [6]. At present, there are two technical routes for the industrial utilization of semi-coke. The first one is to employ the circulating fluidized bed to pre-heat semi-coke [7, 8], which can improve ignition, inhibit semi-coke quenching and reduce nitrogen oxide emission, however, the auxiliary preheating equipment is needed with its poor economy and high construction cost. Another method is to mix high-volatile raw coal and semi-coke directly in pulverized coal burning system to burnout in furnace [9, 10]. This method usually blends
semi-coke with high volatile coal at certain proportion in the furnace. Rational mixing can improve semi-coke combustion, reduce pollutant emission and save coal resources[11]. As a result, it is important to explore co-combustion behavior characteristics of semi-coke and coal as well as the co-combustion kinetics parameters in order to utilize the semi-coke.

In fact, there are lots of experimental studies have been conducted on the combustion characteristics of coal blending[12-15], however, few studies on the co-combustion characteristics of semi-coke and coal. In addition, most of the semi-coke is derived from the production of oil shale gasification with highly ash content and lower calorific value, which is different from coal pyrolyzed semi-coke[16-18].

The thermogravimetric analysis(TGA) and drop tube furnace(DTF) experiment are the main experimental means[19-20]. There are some combustion parameters in TGA different from those in pulverized-coal boiler, including oxygen concentration, heating rate, which may greatly influence combustion behavior of blended fuel. DTF can simulate the combustion situation of actual boiler furnace, and the burnout and NOx formation characteristics of fuel can be acquired simultaneously [21]. Du et al. [22] observed the burnout ratio of the blends increased with the combustion temperature and blending ratio of high-volatile coal by using DTF experiment. Tianyang Chi [23] used a drop tube furnace with a flame monitoring system to study the ignition characteristics of blended coal.

Most of the researches prefer to focus on the combustion kinetics of coal char in the last decades by using TGA[24-27], in order to discuss the influence of coal type, oxygen concentration, temperature, particle size on the parameters such as activation energy, frequency factor and reaction order. Comparing with the coal, only a few papers focus on the combustion dynamics of semi-coke, and they are all based on the results of TGA experiments at low temperature, which has a certain gap with the actual reaction temperature. Jing et al. [28] use three calculation methods to research the kinetics for different pyrolysis semi-cokes receiving the results that combustion activation energy increases with the increasing of carbon conversion. Zhang et al[29]. investigated the co-combustion characteristics and founded that the higher the mixed bituminous coal is, the apparent activation energy is increases with the increasing proportion of semi-coke, which consists with the study conducted by Chao et al [30].

Totally, semi-coke is a special kind of carbon-based fuel and the physical and chemical structure are quite different from coal, however, the current research has rarely pointed out the influence of pore structure on blended combustion characteristics. In addition, blended combustion is a very complex chemical reaction and two kinds of blended materials interact. The relationship between combustion reaction rate and semi-coke and raw coal is not clear yet and it is necessary to study the combustion kinetics of blended fuel systematically.

In this paper, the combustion characteristics of blended fuel are investigated in low temperature reaction region (kinetics control zone) by TGA and high temperature reaction region (diffusion control zone) by DTF. Combining with the pore structure analysis and combustion reaction kinetics calculation, the effect of blending ratio of semi-coke, oxygen concentration and combustion temperature on carbon conversion rate are studied. More importantly, the reaction order, frequency factor and activation energy at different blending ratios of semi-coke are obtained, which has important reference value for the blending combustion of semi-coke.

2. Sample preparation and experiment methods

2.1. Sample preparation

In this paper, The Shenhua(SH) raw coal was crushed and later sieved to obtain 60-75μm coal particles. To remove the volatile components thoroughly, the coal particles were pyrolyzed in an Ar atmosphere at 1273K for 30min using a horizontal tube reactor to obtain the SH char samples. After pyrolysis process in the reactor, the coal char particle size is controlled in 60-75μm. The semi-coke (SC) was obtained from Shanxi Coal and Chemical Industry Group CO. Ltd, as a by-product of the coal carbonization process heated at 700 °C, which was grinded to the same size as coal char.
The ultimate analysis and proximate analysis of SH raw coal, coke and SM semi-coke are shown in Table 1.

| Sample       | Elemental analysis (%) | Industrial analysis (%) | Qnet,v,ar (KJ/Kg) |
|--------------|------------------------|-------------------------|-------------------|
|              | Cdaf | Hdaf | Ndaf | Sdaf | Odaf | Mar | Var | Fcar | Aar |          |
| SH coal      | 82.97 | 4.68 | 1.08 | 0.49 | 10.78 | 9.36 | 28.11 | 56.05 | 6.48 | 26422    |
| SH char      | 97.35 | 0.41 | 1.32 | 0.24 | 0.68  | 3.56 | 2.54  | 80.73 | 13.17 | 27386    |
| Semi-coke    | 93.32 | 1.20 | 0.98 | 0.37 | 4.13  | 6.48 | 8.75  | 73.12 | 11.65 | 26535    |

2.2. Pore structure measurements

The pore size structure of SH char and SM semi-coke was analyzed by ASAP 2020 (Micromeritics, USA) physical adsorbent, and the specific surface area of N₂ and CO₂ adsorption was determined respectively. N₂ adsorption and CO₂ adsorption were used to determine the specific surface area of micropore and micropore in the sample respectively. The adsorption of N₂ and CO₂ was studied at 77K and 273K, respectively. All samples were dried for 12 hours in a vacuum drying chamber at 80℃ before determination. The pressure of the adsorption system is less than 1.33×10⁻⁴ Pa after vacuum treatment at 150℃. The specific surface area of the sample was calculated by Brunauer-Emmett-Teller (BET) method. Pore volume and pore size distribution were obtained by adsorption-desorption curve (Barrett-Joyner-Halenda (BJH) model). The total pore volume was estimated at the relative adsorption pressure P/Pₒ of 0.99.

2.3. Isothermal TG co-combustion tests

In order to obtain the intrinsic reaction kinetics parameters of SH char and SM semi-coke co-combustion reaction, the TGA/DSC3+ thermogravimetric analyzer produced by METTLER-TOLEDO Company in Switzerland was used to carry out co-combustion experiments of semi-coke and char in different proportions. The co-combustion proportions of semi-coke were 0%, 20%, 30%, 40%, 50% and 100%. Pure N₂ was used as protective gas in the experiments. Approximate 5mg blending samples in alumina crucible was dried at 105℃ in N₂ atmosphere for 20 minutes and then maintained at the reaction temperature (698K, 748K, 798K, 848K) with heating rate of 20K/min. Next, the protective gas was switched to high purity oxygen reaction atmosphere until the reaction ended. The gas flow rate was set at 60mL/min and the reaction temperature was maintained for 2 hours.

2.4. Drop tube furnace co-combustion tests

The high temperature co-combustion experiments were carried out in a DTF system. As shown in Figure 1, the experimental system consists of gas distribution system, micro-powder feeder, preheating furnace, reaction furnace and sampling system. Before the experiments, furnace was electrically heated to the experimental temperature (1473K, 1673K, 1873K), sample particles of different semi-char blending ratio were fed by a vibratory powder feeder at a feeding rate of 0.1mg/min.

In this paper, the initial partial pressure of oxygen Pᵢₐ,O₂ refers to the mixture of primary and secondary reaction air. Pᵢₐ,O₂ is realized by changing the mixing ratio of O₂ and Ar in the reaction air, including four oxygen concentrations: 0.5%, 1.0%, 5% and 10% atm. The blended powder of char and semi-coke(semi-coke proportion is 0%, 20%, 30%, 40%, 50% and 100%) are brought into the reactor by primary air and mixed with the secondary air straightened by the rectifier at the reactor inlet and then burned in the reactor. The constant temperature distance of the reaction is about 700mm. It is assumed that the settling velocity of the reactant particles is the same as that of the reaction gas flow, and the designed residence time is 2s. Finally, the gas-solid mixture was cooled to 573K by a water-cooled probe gun at the tail of the reactor, and the solid particles were filtered and collected by stainless steel metal mesh. The composition of the reaction tail gas was measured by MG5+ gas analyzer.
3. Reaction kinetics calculation

3.1. Thermogravimetry analysis model

This paper combines the random pore model[17] and combines half-life reaction time method to calculate the intrinsic reaction kinetics of the coke constant temperature thermogravimetric experiment [24]. The expression of the random pore model is as follows:

\[
\frac{dx}{dt} = (1 - X) \sqrt{1 - \psi \ln(1 - X)}
\]

\[
X = 1 - \exp\left(-\tau \left(1 + \frac{\psi L_0}{4}\right)\right)
\]

(1)

(2)

Where \(X\) is the carbon conversion ratio, \(\tau\) is the dimensionless time:

\[\tau = A_0 t\]

(3)

Where \(A_0\) is the initial reaction rate and the expression is

\[A_0 = R_i A_g (P_{O_2})^m\]

(4)

Where \(R_i\) is the intrinsic reaction rate constant, \(A_g\) is the initial specific surface area of coke, \(P_{O_2}\) is the partial pressure of oxygen, \(m\) is the intrinsic reaction order, \(t\) is the reaction time, \(\psi\) is the structure parameters of coke particles, and \(\psi = \frac{4\pi L_0 (1 - \epsilon_0)}{8S_0}\), where \(L_0\) is the total length of the initial pores per unit volume overlap, \(S_0\) is the surface area of the initial pores per unit volume overlap, and \(\epsilon_0\) is the initial porosity of coke.

Integrate equation (1) and combine equations (3) and (4) to get

\[R_i = \frac{2[(1 + \psi \ln 2)(0.5 - 1)]}{A_g (P_{O_2})^m \psi_{t_{0.5}}}\]

(5)

Where \(t_{0.5}\) is the reaction time at \(X = 0.5\),

According to the Arrhenius empirical equation

\[R_i = A \exp\left(-\frac{E}{R_g T}\right)\]

(6)

Where \(A\) is frequency factor, \(E\) is the intrinsic activation energy, \(R_g\) is the gas constant and \(T\) is the reaction temperature.

A linear fitting of the \(\ln R_i - \frac{1}{T}\) relation diagram can be used to obtain the intrinsic reaction kinetics Arrhenius curve of sample combustion under different semi-coke blending ratios under low and constant temperature thermogravimetric combustion conditions. It should be pointed out here that the reaction atmosphere of the thermostatic thermogravimetric experiment is oxygen at 1atm, so it is unnecessary to assume the intrinsic reaction order \(m\) for calculation.
3.2. Drop tube furnace model

Thermogravimetric analyzers are suitable for studying the intrinsic reaction kinetics characteristics of low-temperature combustion condition, and a drop tube furnace (DTF) experiment is required for high-temperature test. Assuming that the primary product of the combustion is CO, from mass balance consideration the burning rate $\rho$ can be expressed as [19].

$$\rho = h_m (C_g - C_0)$$  \hspace{1cm} (7)

Where $\rho$ is the reaction rate per unit surface area of char, $h_m$ is the CO mass transfer coefficient, $C_g$ is the atmosphere oxygen concentration.

According to Smith's research [27], the following relationship exists between the external surface reaction rate $\rho$ and the intrinsic reaction rate $R_i$:

$$\rho = \eta A_e R_i \left[ \frac{C_g}{1 - \chi} \right]$$  \hspace{1cm} (8)

Where $\eta$ is the effective factor, $\gamma$ is the characteristic size of particle, $\sigma$ is the particle density, $A_e$ is the specific pore-surface area, $C_g$ is the oxygen concentration, $\chi$ is the proportional coefficient, $m$ is the reaction order.

The following relationship exists between the effective factor $\eta$ and the Thiele modulus $\Phi$:

$$[\eta\Phi^m (m+1)/2] = e^{-\gamma (m+1)/[8D_e C_g (1-\chi)]}$$  \hspace{1cm} (9)

Then $\eta$ can be determined using the relations between $\eta$ and $\Phi$ given by Mehta and Aris [30].

3.3. Oxygen diffusion in the particle with the size

Furthermore, the distribution of oxygen in particle can be obtained by using Gas transport equation [31] as follows:

$$\frac{1}{r_p^2} \frac{d}{dr_p} \left( r_p^2 \frac{dC_g^*}{dr_p} \right) = \frac{\beta \rho d T C_g^*}{D_{eff} M_c}$$  \hspace{1cm} (10)

Where $r_p$ is the radius of a particle (m); $\rho$ is the bulk density of a particle (kg/m³); $\beta$ is the molar stoichiometric coefficient of the reaction; $R_u$ is the universal gas constant (J/Kꞏmol); $n$ is the reaction order; $D_{eff}$ is the effective diffusion coefficient inside a particle (m²/s); and $M_c$ is the molar mass of carbon (kg/mol).

4. Results and discussion

4.1. Pore structures of char and semi-coke

Figure 2 shows the calculated specific surface area and pore volume distribution for the samples measured by the nitrogen adsorption method. From Figure 2a it can be found that the pore volumes for both semi-coke and char first increase and then decrease with the increasing of the pore size. In addition, the differential pore volume of semi-coke is larger than that of char. This is because the
semi-coke is a low temperature pyrolysis product at 700℃ and the pore structure is better preserved, so the pore volume of semi-coke is larger than that of char.

Figure 2b shows the distribution of specific surface area with pore size. It is found that the specific surface area of semi-coke and char increases first and then decreases with increasing particle size. In addition, the specific surface area of semi-coke is larger than that of char significantly, which is consistent with the analysis results of pore volume. The distribution of pore volume and specific surface area shows a positive correlation.

4.2.  Combustion reaction kinetics
A linear fit was performed on the experimental values of temperature 698~848K to obtain the Arrhenius curve of the intrinsic reaction rate Ri, the expression and kinetic parameters are shown in Figure 3.

It can be seen that the activation energy E gradually decreases with the increase of blending ratio which illustrates the semi-coke has better reactivity. It is because the volatile content of the blended fuel increases with the increasing proportion of the semi-coke. Moreover, the specific surface area of the mixed fuel increases significantly with the increase of blending, and the contact between the fuel and the reaction gas becomes more sufficient, and the reaction activity increases gradually.

4.3. Diffusion characteristics of oxygen in particles
In Figure 4, such conclusions can be obtained as follows. Considering the effect of oxygen concentration on the effective factors, it is found that the effective factor η of different oxygen concentration is 10% > 5% > 1% > 0.5% in turn. The effective factors increase with the increase of oxygen concentration, which indicates that oxygen more easily to diffuse in the particles with the increasing of oxygen partial pressure.
With the increase of the proportion of semi-coke, the average effective factor increases. Especially when the blending ratio is between 0-40%, the increase is significant. The diffusion characteristic of char dominates the diffusion characteristic of mixed fuel. As mentioned above, the pore diffusion characteristic of semi coke is better than that of char, and the blending of semi coke will significantly improve the average diffusion characteristic. While the blending ratio is between 40-100%, the diffusion characteristic of semi-coke dominates the diffusion characteristic of mixed fuel and the increasing of the semi coke has little effect on the effective factors. Comparing the effective factor of combustion of pure semi-coke and pure char, it can be seen that the effective factor of pure semi-coke with larger specific surface area are significantly greater than that of char, indicating that the pore structure of particles plays an important role in oxygen diffusion, and the diffusion of oxygen in the pores of semi coke is significantly better than that of coke under the same conditions.

In addition, comparing the effective factors under the same oxygen condition at three temperatures, it is seen that the effective factors gradually decrease with the increase of temperature. This is because particle combustion is mainly controlled by diffusion reaction under this condition. Although the effective diffusion coefficient increases with the increase of temperature, the chemical reaction rate of particle combustion will also be accelerated with the increase of temperature. Moreover, the effect of temperature on the chemical reaction rate is greater than that on the effective diffusion coefficient. Therefore, the combustion reaction of pores is controlled by the diffusion more obviously and the oxygen diffusion depth becomes shallow with the increasing of temperature.

Both the pore structure characteristics and oxygen concentration of coal char will affect the diffusion of O$_2$ in the particle pores[24]. For a clearer comparison of this effect, the concentration gradient of O$_2$ along the particle radius direction can be calculated directly and presented in Figure 5.

![Figure 5. Distribution of oxygen concentration along the radius of char particles.](image)

For any proportion of semi coke, the oxygen concentration at the same radius is 10% > 5% > 1% > 0.5%, which indicates that increasing reaction oxygen concentration can promote the diffusion of oxygen. In addition, with the increase of the blending proportion of semi coke, the oxygen concentration in the same radius is in the order of 100% > 50% > 0%, which indicates that with the increase of the blending proportion of semi coke, oxygen is more easily diffused into the pores of particles. It means that both the pore structure characteristics and oxygen concentration of coal char will affect the diffusion of O$_2$ in the particle pores.

4.4. Carbon conversion of blended fuel in DTF

It is seen in Figure 6 that carbon conversion rate increases significantly with the increase of oxygen concentration. This is because with the increase of oxygen concentration, it is easier to promote the diffusion of oxygen inside the particles. It can be found that under the same oxygen concentration, the carbon conversion rate gradually increases with the increase of temperature. It is reasonable because the particle combustion temperature is increases with the reaction temperature, so the C-O$_2$ combustion reaction rate is faster and the carbon conversion rate is higher under the same residence time.
In addition, the conversion of carbon first increases and then decreases with the increase of the proportion of semi-coke. The maximum and minimum conversion rate occurs at 30% and 100% semi-coke blending respectively. This is because when the proportion of semi-coke changes from 0% to 30%, the reaction of char is dominant, but the reaction is very sensitive to the change of the proportion of semi coke. When the proportion of semi coke continues to increase, the promotion of semi coke weakens, and even inhibits the combustion of char. When the proportion of semi coke reaches 100% the conversion rate reaches the minimum value, which fully shows the competitive relationship between the char and semi-coke.

5. Conclusions

1) Both the semi-coke and char first increase and then decrease with the increasing of diameter of particles, furthermore, the volume and specific surface area of semi-coke are larger than that of char particles.

2) Under low temperature thermogravimetric reaction conditions, as the blending ratio of the semi-coke increases, both the activation energy and frequency factor of the mixed fuel decrease gradually.

3) The kinetic parameters of sample combustion in the high temperature region was stability and has good agreement with the Arrhenius curve of the intrinsic reaction rate constant linearly fitted in the low temperature region. After correcting the intrinsic reaction kinetic parameters in the low temperature region, it can be determined that reaction order decreases with the blended ratio while pure semi-coke combustion has the same reaction order with pure char combustion condition.

4) At the same radius, the oxygen concentration of particle increases with the reaction oxygen concentration for any semi-coke blending ratio, furthermore, the oxygen concentration of particle increases with the blending ratio for the same reaction oxygen concentration.

5) At any temperature, the burn out rate increases with the increase of oxygen content, and the carbon conversion rate increases first and then decreases with the increase of semi coke blending ratio. When the semi coke blending ratio is 30%, the burn out rate is the highest.

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