Oxidation Behavior and Kinetics Parameters of a Lean Coal at Low Temperature Based on Different Oxygen Concentrations

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Abstract: The method of dividing the “three zones” of spontaneous combustion in goaf by oxygen volume fraction is the most widely used and effective method at present. However, the oxygen volume fraction method does not consider the influence of methane concentration in goaf, which is only applicable to low-gas goaf, not high-gassy goaf. In this work, the oxidation behavior and kinetics parameters of a lean coal at low temperatures under five different oxygen concentrations, including methane and its kinetics during low-temperature oxidation, were studied using temperature programming tests and thermogravimetric tests. The results showed that the decrease of oxygen content improves the adsorption capacity of coal to absorb different atmospheres at the initial stage. In the whole reaction process, there is a negative correlation between the strong-to-weak order of exothermicity and adsorption capacity, with a significant increase in apparent activation energy E. A marked hysteresis of the precipitation time of CO and CO\(_2\) and a decrease in their precipitation amount and a rise in the initial temperature for the generation of CO and CO\(_2\) were found.

Keywords: oxygen concentration; temperature programming test; thermogravimetric analysis; index gases; activation energy analysis

1. Introduction

In China, the United States, India, Russia, and other countries and regions, spontaneous combustion of coal seam is common [1,2]. Spontaneous combustion of coal seam seriously threatens the natural environment and human health, causing huge resource loss and environmental pollution [3–5]. High-gas spontaneous combustion mines in China are prone to secondary disasters caused by gas explosion due to coal spontaneous combustion [6–10].

Research on the mechanism of coal spontaneous combustion is mainly based on the theory of coal-oxygen compound action [11]. The process of coal spontaneous combustion is the physical and chemical process between coal and oxygen. Oxygen concentration has a great influence on coal spontaneous combustion [12,13]. The distribution of “three zones” of spontaneous combustion in working face goaf is mainly divided based on oxygen concentration [14]. In the past 20 years, scholars from various countries have studied the mechanism of coal spontaneous combustion using different methods, including experimental simulation and thermal analysis from different perspectives, such as activation energy, characteristic temperature of coal spontaneous combustion, and surface-active groups of coal molecular structure [15–18]. The self-heating temperature method was used to explore the spontaneous combustion tendency of coal. This method reflected the nature of coal spontaneous combustion, but it had many disadvantages—for instance, poor repeatability, complex test process, and long test time. The cross-point temperature method could greatly shorten the test time, but it could not scientifically measure oxidation characteristics of coal at low temperatures. At present, the method of dynamic physical adsorption of oxygen...
used in China has good repeatability, but it only points out the physical adsorption capacity of coal, which is obviously not enough to reflect the essential characteristics of coal spontaneous combustion, especially the oxidation capacity of coal at low temperatures.

In recent years, many studies on the spontaneous combustion characteristics of coal under different oxygen concentrations have been reported. Qi et al. [19] tested the heat generation of coal oxidation under different oxygen concentrations. Wang [20] studied the kinetic parameter of model coal and coal samples under 10% oxygen. Su et al. [21] investigated low-temperature oxidation of coal with different oxygen concentrations ranging from 6% to 21%. Li et al. [22] conducted a TGA-DSC experiment for a comparative study of the kinetic parameter in 5% and 15% oxygen concentrations, respectively. Wang et al. [23] set up a kinetic model that was used for the quantitative description of the oxygen consumption rate and the formation rate of gas oxidation. Onifade [24] tested 28 selected coal and coal-shale samples to obtain the intrinsic properties and spontaneous combustion ability. Moshood et al. [25] tested fourteen coal and four coal-shale samples using the TGA and the Wits–Ehac tests to reveal six different heating rates. The abovementioned investigation used a single method, resulting in a conclusion which was not comprehensive enough [19–25].

In China, most mines are gassy mines, and the spontaneous combustion of coal in mine goaf involves methane-containing mixed gases. In previous studies, most TG-DSC experiments and programmed temperature experiments were carried out in a mixed atmosphere of oxygen and nitrogen, and few studies were carried out under the condition of methane participation. Nevertheless, the adsorption capacity of coal for methane, nitrogen, oxygen, carbon dioxide, and other different gases was different. Under the condition of the same oxygen concentration, different gas components would generate different effects on the spontaneous combustion of coal, including macroscopic thermal effects, oxidation products, and dynamic characteristics. Thus, research on the mechanism of competitive adsorption–spontaneous combustion oxidation of gas containing different methane concentrations is of great importance to the prevention and treatment of coal spontaneous combustion and gas control.

Therefore, this work focuses on the oxidation product (index gas), TG-DSC, and activation analysis of coal under different gas conditions with a thermal gravimetric analyzer and programmed temperature controller. The aim of this work is to forecast the spontaneous combustion of coal in highly gassy goaf, so as to ensure safe production of the working face of the coal seam with high gas and easy spontaneous combustion.

2. Materials and Methods

2.1. Sample Collection and Preparation

Coal samples were collected from the large chunk of coal stripped of surface oxidation layer of K₃ coal seam in Donglin Coal Mine. The coal type was lean coal. Before the experiment, a large piece of fresh coal was selected to strip the surface oxide layer, and the coal sample was crushed and screened. After screening, particle samples of a size of 0.1–0.2 mm were obtained as experimental coal samples, which were placed in coal sample bottles that were then sealed and reserved to prevent oxidation. According to the proximate analysis method of coal, moisture, ash, and volatiles were measured. The content of sulfur in coal was determined in accordance with the method of total sulfur in coal. The grade of coal spontaneous combustion ability was determined via fluidized chromatography using the oxygen absorption method. Coal sample test results showed a moisture content of 0.63%, ash content of 11.40%, volatiles of 19.15%, true density of 1.40 g·cm⁻³, sulfur content of 1.95% and 0.66 cm³·g⁻¹ of physical oxygen intake of dry coal. The K₃ coal seam was a Class-II spontaneous combustion coal seam.

2.2. Composition of Oxygen Concentration

The experimental gas was purchased from Chongqing Lituo Gas Co., Ltd. and configured in accordance with the different proportions of gas. The specific ratio of oxygen concentration volume and different oxygen concentration ratio is presented in Table 1.
2.3. Temperature Programming Test

The prediction methods of coal spontaneous combustion mainly include oxidation product (index gases), temperature measurement, tracer gas, odor detection, and so on. The oxidation product yield will change regularly with the rise of coal temperature. The oxidation product (index gases) analysis method is widely used in the early prediction of coal seam fire in China and other countries. Compared with the adiabatic oxidation experiment, programmed temperature has obvious advantages, such as low time–cost and low energy consumption. Most importantly, the influence of different environmental factors on the spontaneous combustion characteristics of coal can be investigated [26].

This experiment consists of preheating the gas circuit, heating the transfer coal sample tank, and programming the temperature control box, temperature control system, and data collection system. Experimental process: A 20 g coal sample with the granularity of 0.12–0.15 mm was placed in the coal sample tank at a flow rate of 100 mL·min\(^{-1}\), with the temperature programmed at a heating rate of 1 °C·min\(^{-1}\) in the temperature control box. Gas analysis was conducted at each additional increase of 10 °C, and composition and concentration of coal oxidation products were determined using a gas chromatograph (Figure 1, MK-6000, Shandong rainbow Chemical Co., Tengzhou, China).

| Gas Composition | 1#   | 2#   | 3#   | 4#   | 5#   |
|-----------------|------|------|------|------|------|
| CH\(_4\) (%)    | 100.0| 50.0 | 30.0 | 0    | 0    |
| O\(_2\) (%)     | 0    | 10.5 | 14.7 | 21.0 | 100.0|
| N\(_2\) (%)     | 0    | 39.5 | 55.3 | 79.0 | 0    |

2.4. Thermogravimetric Analysis

Thermal analysis technology is divided into isothermal analysis technology and non-isothermal analysis technology depending on the different temperature control methods. Isothermal analysis technology has become the most widely used thermal analysis technology in the study of coal spontaneous combustion due to its simple application method, as well as the fact that it takes little time and has strong practicability. In the research field of coal spontaneous combustion, TG and DSC can be used to obtain the enthalpy change law and oxidation kinetic parameters at different stages of coal spontaneous combustion oxidation.

![Figure 1](image-url)

Figure 1. Temperature programming oxidation system. (a) Test system; (b) heat transfer coal sample tank.
A thermogravimetric analyzer (Figure 2, STA449F3, NETZSCH-Gerätebau GmbH, Selb, Germany) was used for thermogravimetric analysis and differential scanning calorimetry of coal sample. A 6 mg coal sample was placed in the aluminum crucible in the thermal analyzer. Nitrogen was introduced for a 1-hour purge of the coal sample. The software test system was turned on with a test temperature of 35–200 °C, heating rate of 1 °C·min⁻¹, reaction atmosphere flow of 60 mL·min⁻¹, and flow rate of shielding gas of 20 mL·min⁻¹. This experiment was carried out under the inlet of mixed gas with different methane content. Each atmosphere experiment was conducted three times to obtain the mean value, ensuring the authenticity of the experimental results.

![Figure 2. STA449F3 synchronous thermal analyzer.](image)

The low-temperature oxidizability of coal (less than 70 °C) can reflect the intensity of the spontaneous combustion characteristics of coal [27,28]. Poland and other countries use activation energy as the main index for the classification of spontaneous combustion tendency [29–31]. Therefore, the spontaneous combustion characteristics of coal under different reaction atmospheres were evaluated applying the apparent activation energy at 40–60 °C. The kinetic data of the coal oxidation reaction were obtained through the thermogravimetric test curve and fitted using the mechanism model function of the kinetic reaction. The Coats–Redfern integral method [32,33] adopting the single scanning rate method was used to acquire the activation energy and pre-exponential factor.

3. Results and Discussions

3.1. Oxidation Product Generation Characteristics

The low-temperature oxidation characteristics of coal could be characterized and evaluated through the low-temperature oxidation products (significant gas) [34]. The results indicated that the oxidizing reaction of the active structure on the coal surface was directly influenced by the oxygen concentration of the environment. Different oxygen concentrations gave rise to different oxygen uptake rates of coal and different heat release intensities of coal. It was found through experimental research that oxidation products such as CO and CO₂ serve as major low-temperature oxidation products that help to analyze the influence of different gas atmospheres on their generation rules.
3.1.1. Initial Temperature for the Generation of CO and CO$_2$

The initial temperature of CO and CO$_2$ generation under different oxygen concentrations is shown in Figure 3. The results revealed that along with the reduction of oxygen concentration, the initial temperature of CO and CO$_2$ increased significantly. When oxygen concentration exceeded 14.7%, the initial temperature of CO$_2$ generation was lower than that of CO generation. However, when oxygen concentration was lower than 14.7%, the low-temperature oxidative activity of coal reduced significantly because the active functional groups in coal were related to the oxygen content of reaction. With the constant increased in methane concentration, there was much less oxygen attached to the coal surface through competitive adsorption in the mixed gas. However, when methane occupied more active sites on the coal surface, thus causing less oxygen to participate in the oxidation reaction and further significantly weakening the low-temperature oxidation reaction, a higher temperature was required to activate the participation of more active groups in the oxidation reaction as a result.

![Figure 3. Initial temperature of CO and CO$_2$ generation at different oxygen concentrations.](image)

3.1.2. Quantity of CO and CO$_2$ Generation

Due to the large differences in the amount of CO and CO$_2$ evolution under different oxygen concentrations, logarithmic coordinates were used to analyze the data. The relationship between the yields of CO and CO$_2$ under different oxygen concentrations and temperatures is presented in Figures 4 and 5. They show that as temperature rises, the yields of CO and CO$_2$ also increase under different oxygen concentrations; likewise, as the oxygen concentration increases, the quantity of CO and CO$_2$ also increases under different temperatures. Because different active structures in coal are activated at different temperatures, an oxidation reaction occurs, which brings about the formation of new free radicals. These free radicals combine with other free radicals already existing in coal to form an index gas. As temperature increases, the concentration of free radicals increases, too. Therefore, the concentrations of CO and CO$_2$ at the low-temperature stage generally increases with temperature rising.
Therefore, the concentrations of CO and CO\textsubscript{2} at the low-temperature stage generally increases with temperature rising. When the oxygen concentration reaches 21% and 100% in the mixed atmosphere, the concentrations of CO and CO\textsubscript{2} gradually increase at the initial stage of coal oxidation. However, when the temperature exceeds 100 °C, the yields of CO and CO\textsubscript{2} under two atmospheres quickly increase.

The relationship between the quantities of CO and CO\textsubscript{2}, two low-temperature oxidation products of coal, and temperature shows that higher oxygen content and furnace temperature give rise to a more volatile coal reaction.

Figure 4. CO generation with temperature under different oxygen concentrations.

Figure 5. CO\textsubscript{2} generation curves with different oxygen concentrations.

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The adsorption capacity of coal to different gases is different. The order of coal adsorption capacity is \( \text{CH}_4 \succ \text{O}_2 \succ \text{N}_2 \), so the probability of coal–oxygen compound action strengthens with the increase in oxygen concentration. When the temperature and concentration of oxygen rise, there are more active groups participating in the coal-oxygen compound action. Coals are prone to spontaneous combustion in an atmosphere due to high heat release and volatile coal reaction.

3.2. TG-DSC and Activation Analysis

3.2.1. TG Analysis

Figure 6 presents the TG difference spectrum of different atmospheres compared with the atmosphere under an oxygen concentration of 21%. It is shown that the sample mass increases under oxygen concentrations of 10.5% and 14.7%, respectively, while the mass decreases under an oxygen concentration of 100%. On the one hand, the heat production of coal–oxygen compound action directly causes coal spontaneous combustion. The reaction heat of coal–oxygen compound action is divided into physical adsorption heat, chemical adsorption heat, and chemical reaction heat. Physical adsorption is the first step of the coal-oxygen compound action, which is the basis of chemical adsorption and chemical reaction [34,35]. The physical adsorption of gas decreases along with the rise in temperature. A stronger adsorption at the initial stage usually results in more reduction in mass. On the other hand, as temperature rises, chemical adsorption gradually increases. This means that with the increase in oxygen concentration, there are more radical groups participating in the active reaction. Fuller chemical adsorption and chemical reaction are accompanied by the generation of more oxidation products from the oxidation reaction and the decrease in mass, which is consistent with the trend of indicator gases produced via coal oxidation under different oxygen concentrations.

![Figure 6. TG difference spectrum curve of coal sample.](image)

3.2.2. DSC Analysis

Figure 7 shows the DSC differential spectrum under different atmospheres compared with an oxygen concentration of 21%. It can be seen that along with the increase in temperature, the differential spectra of three samples show a heat adsorption state under oxygen concentrations of 0%, 10.5%, and 14.7%, but a heat release state under an oxygen concentration of 100%.

At an oxygen concentration of 0%, there is no oxygen participating in the coal oxidation reaction but simply the gas desorption and autooxidation process. In the process of coal oxidation with different methane atmospheres, there is a different degree of participation in chemical adsorption and oxidation reaction. The heat release reaches its highest level at an oxygen concentration of 100%. The compound reaction of coal and oxygen is boosted along with the increase in oxygen concentration in the process of coal–oxygen reaction. The heat release curve in Figure 7 shows that the ranking of the heat release intensity in descending order is 100% > 14.7% > 10.5% > 0 based on oxygen concentration. The reaction intensity between the active group and oxygen in coal is significantly affected by the oxygen concentration, as heat release increases with the increase in oxygen concentration. This also shows that a reduction in oxygen concentration can significantly inhibit the heat release of coal in the process of low-temperature oxidation.
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![Figure 7. DSC differential spectrum curve.](image)

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### 3.2.3. Activation Energy Analysis

The calculated kinetics parameters of coal oxidation reaction under different reaction atmospheres are shown in Table 2. The fitting correlation coefficient of first-order reaction in a low-temperature oxidation reaction under five atmospheres all exceed 0.99, which shows that its low-temperature oxidation reactions meet first-order chemical reaction. At oxygen concentrations of 100%, 21%, 14.7%, 10.5%, and 0%, the corresponding apparent activation energy of coal is 52.68 kJ·mol⁻¹, 34.06 kJ·mol⁻¹, 26.81 kJ·mol⁻¹, 11.37 kJ·mol⁻¹, and 10.35 kJ·mol⁻¹, respectively. With the oxygen concentration increasing from 0% to 14.7%, the active energy of coal reduces significantly. However, with the concentration of oxygen increasing from 21% to 100%, the active energy of coal reduces from 11.37 kJ·mol⁻¹ to 10.35 kJ·mol⁻¹, signifying that the low-temperature oxidative activity under the two conditions is similar. When the oxygen concentration is more than 21%, the surface-active group of coal that participates in the reaction and the recombination action of oxygen almost reaches saturation, which is consistent with the aforesaid TG curve change trend and precipitation characteristics of significant gas. Therefore, the ranking of apparent activation...
energy of oxygen concentrations in descending order is 0% > 14.7% > 10.5 > 21% > 100%. As the O\textsubscript{2} content increases, the activation energy of coal in low-temperature oxidation reduces significantly.

| Experimental Group | Fitting a Straight Line | Activation Energy /kJ·mol\(^{-1}\) | Correlation Coefficient |
|--------------------|-------------------------|-----------------------------------|------------------------|
| 1#                 | Y = -4766.70X - 1.6751  | 52.68                             | 0.9981                 |
| 2#                 | Y = -4097.64X - 4.004   | 34.06                             | 0.9918                 |
| 3#                 | Y = -3225.82X - 5.594   | 26.81                             | 0.9967                 |
| 4#                 | Y = -1388.848X - 10.919 | 11.37                             | 0.9958                 |
| 5#                 | Y = -1245.40X - 11.171  | 10.35                             | 0.9931                 |

4. Conclusions and Outlooks

The adsorption capacity of coal at the initial stage reduces as the oxygen concentration increases. The mass increases by 6.52%, 2.89%, and 1.15% at oxygen concentrations of 0%, 10.5%, and 14.7%. However, coal mass decreases by 1% and 2% at oxygen concentrations of 21% and 100%, respectively. Exothermicity strengthens as oxygen concentration increases in the whole process. With the increase in oxygen concentration, the initial temperatures of CO and CO\textsubscript{2} generation decrease while the yields increase, demonstrating that a decrease in oxygen concentration can significantly inhibit the low-temperature oxidation of coal. Along with the increase in oxygen concentration, the low-temperature apparent active energy of coal reduces significantly and enhances the tendency of spontaneous combustion of coal. With an oxygen concentration of more than 21%, the surface-active groups that participate in the reaction and the compound oxidation of oxygen tend toward a saturation state. A reduction in oxygen concentration increases the difficulty of coal–oxygen compound action and inhibits coal oxidation, the production of oxidation products and heat is reduced, and the activation energy of coal in low-temperature oxidation increases significantly. There are more active groups participating in coal–oxygen compound action with temperature rising, so more oxidation products and more heat are generated.

Combined with low-temperature oxidation under the condition of gas flow, the CO volume fraction corresponding to different combustion stages of the goaf left coal is determined. The goaf left coal has entered the dangerous stage of spontaneous combustion, and measures must be implemented to control the further development of spontaneous combustion and oxidation of the goaf. This is helpful for the early prediction of goaf safety and comprehensive treatment of high-gassy and spontaneously combustible mines. Combined with low-temperature oxidation under the condition of different gas flow, the CO volume fraction corresponding to different combustion stages of the mined-out coal in goaf is determined. When the mined coal in goaf is in the dangerous stage of spontaneous combustion, measures must be taken to control the further development of spontaneous combustion and oxidation in goaf.

The next step is to determine the distribution range of the three zones in the high gas mine, integrating experimental results with field monitoring. Further, targeted technical measures should be put into effect to prevent and extinguish fires in different stopping periods, which is of great significance for industrial production.

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