Experimental study on high temperature oxidation spontaneous combustion characteristics of long flame coal in Fushun West Open-pit Mine

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Abstract

In order to study the spontaneous combustion and oxidation characteristics of coal in the Fushun West Open-pit Mine, the spontaneous combustion experiment was carried out on the basis of physical property tests. The macroscopic spontaneous combustion characteristics parameters 30-600℃ were obtained. The characteristic temperature point was determined by analyzing the growth rate of indicator gas. The results show that the water content and sulfur content of coal samples from the West Open-pit Mine are all low, and the volatile matter is as high as 42%. The specific surface area of the coal is larger, and the microstructure shows that macropores and mesopores account are more than 95%, which provides favorable conditions for coal-oxygen synthesis reaction. In the low-temperature oxidation stage, the oxygen concentration and consumption rate changed gently, and changed sharply after 200℃, and the CO and CO_2 gas concentration increased exponentially. The CH_4, C_2H_4, C_2H_6, and C_3H_8 gas concentrations have similar variation laws with temperature. It was relatively small at low temperature and increased slowly with the increase of temperature, but rapidly increased to the peak value after about 350℃. The critical temperature was 75℃, cracking temperature was 120℃, active temperature was 195℃, speedup temperature was 240℃, and ignition temperature was 315℃. The high volatile content in the coal sample lead to an earlier critical temperature, but the ignition temperature was lower than the conventional one. The thermal analysis showed that the DSC and exothermic process can be divided into four stages, and the peak exothermic heat was 4714 J/g. The free radical concentration had a linear increasing relationship with temperature, and the concentration at the active temperature increased by about 50% than which at the critical temperature. The free radical concentration at the ignition temperature was twice the critical temperature moment, and the free radical activation would further promote the coal oxygen synthesis reaction.

Introduction

Coal spontaneous combustion often occurs in the process of mining, and is one of the major disasters causing a series of safety and environmental problems, and waste of resources[1]. Existing studies have shown that coal spontaneous combustion is a complex oxidation process[2,3], affected by various internal and external factors. Scholars have done a lot of research work by theoretical research, experimental analysis, and numerical simulation. Qin et al.[4] summarizes the research results of the mechanism, monitoring and prevention technology on coal spontaneous combustion comprehensively and systematically. Deng et al.[5], Dong et al.[6] have determined the composition and precipitation characteristics of coal spontaneous combustion index gas through experiments respectively. Liang et al.[7] studied the mesoscale of spontaneous combustion of coal by combining macroscopic model with microscopic model, and explained the microscopic internal mechanism of spontaneous combustion of coal. Wang et al.[8] studied the evolution law of free radicals and functional groups with the theoretical method of oxidation kinetics. Chang et al.[9], Wen et al.[10] studied the law of oxygen concentration and its effect on the evolution of spontaneous combustion by the thermogravimetric experimental method. Scholars et al.[11-14] conducted experiments on the characteristics of spontaneous combustion of different types of coal in different regions by using temperature-programmed experiments, field measurements, and thermogravimetric way.

The existing research results are mostly divided into two stages, one is the low-temperature oxidation stage (usually below 200℃), and the other is the high-temperature oxidation stage (until the sample burns out). In terms of low temperature oxidation research, Lu et al. [15] studied the relationship between the law of coal molecular structure and coal spontaneous combustion process through low temperature oxidation experiments. Liang et al. [16] studied the low-temperature oxidation characteristics of metamorphic coal under the condition of igneous rock intrusion, and obtained characteristic parameters. Wang et al. [17] studied the low-temperature oxidation characteristics of mixed minerals of coal and oil shale in Liangjia coal mine by temperature-programmed experiments, and established monitoring and evaluation indexes for spontaneous combustion in the mined-out area of oil shale and coal seam. In terms of high-temperature oxidation research, Chen et a1. [18] developed a high-temperature self-absorption oxygen experimental device for loose coal and carried out experimental application. Deng et al. [19] tested the characteristic parameters of the weathered coal, analyzed the law of the index gas concentration and the influence of the characteristic temperature point and particle size on the oxidation of the weathered coal. Zhao et al. [20] studied the high temperature oxidative spontaneous combustion characteristics of bituminous coal with thermodynamic analysis method, and divided the oxidative spontaneous combustion process into four stages. Li Lin, et al [21], Liu. [22] used experimental methods to study the spatiotemporal evolution law of temperature, oxygen concentration and index gas in the process of coal oxidation, not only considering time and temperature factors. Zhao et al. [23] simulated the spatial migration law of temperature from the surface to the inner area in the process of oxidative spontaneous combustion of loose coal, which is more in line with the actual situation on site.

Fushun West Open-pit Mine has a mining history of more than 100 years. Countless spontaneous combustion accidents have occurred in the mine, and until now spontaneous combustion still occurs from time to time with serious impact on the environment, as shown in Figure 1. The mining environment of open-pit mines is very different from that of underground mining, and the spontaneous combustion evolution process is closely related to the environment. Therefore, it is necessary to conduct in-depth research on the laws of coal spontaneous combustion and oxidation under specific environmental conditions in open-pit mines [24,25].

Coal physical properties such as specific surface area and pore size were obtained by physical adsorption experiment. Spontaneous combustion characteristic parameters including oxygen consumption, concentration of index gas, characteristic temperature and heat release parameters are obtained by high temperature programmed experiment. It is very significant for the prevention and control of coal spontaneous combustion in the process of open-pit mining.

Experiment Plan And Method

Fresh coal samples (as large as possible) collected from the Fushun West Open Pit Mine were sealed on site and transported to the laboratory. When preparing the experimental samples, the oxide layer on the surface was stripped first, and the samples were crushed to 80-120 mesh (particle size is 0.124-0.178 mm) for experiment followed.

(1) Coal quality analysis. Moisture and volatile content were measured by industrial analyzer (5E-MAG6700, China). The element components of samples were determined by test using element analyzer (Vario EL □, Germany).

(2) Analysis of specific surface area and pore size. The automatic physical and chemical adsorption analyzer (Autosorb-iq-c, American) was used to test the specific surface area and pore size distribution characteristics.

(3) Temperature-programmed experiment at high temperature. The coal samples were crushed and sieved into 5 different particle sizes including 0~0.9mm, 0.9~3mm, 3~5mm, 5~7mm and 7~10mm, and 1 kg each was taken to form a mixed particle size sample. The high-temperature-programmed experimental test was carried out in the spontaneous combustion experimental system of (XKGW-1, Xi'an University of Science and Technology, China). The heating rate was 1℃/min, the air flow was set at 120 mL/min, and the temperature rising range was 30-600℃. The gas was pumped every 15℃ increasing, and sent to gas chromatograph (SP-3430, American) for analyzing gas component.

(4) Thermal analysis experiment. Differential scanning calorimetry (DSC, TA-SDTQ600, American) experiment was done by using thermal analyzer to study heat flow curve and the exothermic characteristics of the sample.

(5) Free radical concentration test. The electron spin resonance spectrometer (MS5000, Germany) was used to test the free radical concentration at different characteristic temperature points.

Test results and discussion

Coal quality analysis. The coal seam in the West Open-pit Mine was formed at Jurassic Cretaceous period, and is a kind of long-flame coal. The coal quality analysis results is shown in Table 1, where M_{ad} is moisture content on an air-dry basis (ad), A_{ad} is ash content, V_{ad} is volatile matter content, FC_{ad} is fixed carbon content, and C, H, O, N, S are carbon, hydrogen, oxygen and nitrogen, sulfur element respectively.

Table 1. Coal analysis result (%)

Index	M_{ad}	A_{ad}	V_{ad}	FC_{ad}	N	C	H	O	S
Quantity	2.94	8.12	42.01	46.93	2.29	74.27	5.88	10.91	0.33

It can be seen that the M_{ad} content is only 2.94%, less than 5%. The V_{ad} content is much higher than some conventional data, which is very helpful for spontaneous combustion. The sulfur content is only

0.33%, which is good for use.

Analysis of specific surface area and pore size. As shown in Table 2, the specific surface area of coal sample is up to 39.3788 m2/g, increasing the chance of a reaction in contact with oxygen. It can be seen that the macropores (pore size >50 nm) and mesopores (2-50nm) of the coal sample is more than 95%, and it is very helpful for oxygen adsorption beneficial for low temperature oxidation. The apparent characteristics is very helpful to explain spontaneous combustion is easy happening.

Table 2. Specific surface area and pore size distribution

Index	Specific surface area（m^2/g）	Micropore (%)	Mesopore (%)	Macropores (%)
Value	39.3788	0.62	56.15	43.23

Variation of oxygen concentration. The law of oxygen consumption is one of the most important macro parameters to reflect the severity of spontaneous combustion. The variation curves of oxygen concentration and consumption rate with temperature are shown in Figure 2. In the low-temperature stage, the oxidation reaction of coal samples was relatively gentle, and there is only a little active functional groups reacting with oxygen. A large number of active functional groups had not been activated, and the consumption of oxygen was low.

The oxygen concentration decreased sharply and oxygen consumption rate increased rapidly after 150 °C. At 270 °C, the oxygen concentration reached the point of 6.986 %. At this time, the active functional groups in coal molecules were gradually activated, resulting in chemical reactions of coal samples, and the reaction rate was accelerating. A large number of active functional groups participated in the reaction and consumed a lot of oxygen. When reaching 330 °C, the oxygen concentration began to rise significantly, and rose to 16.04 % at 390 °C. At this temperature, a large number of functional groups inside the mineral molecules began to decompose. The sample has just reached the ignition point, and the heat generated by combustion is low. However, the decomposition of functional groups requires a lot of energy, resulting in a slight decrease in the combustion effect of the sample and the oxygen concentration rose slightly. After 390 °C, the oxygen concentration began to decline sharply, until the end of the experiment, it fell to 6-7 %. The oxygen consumption rate is in the opposite trend to the oxygen concentration.

The law of indicator gas. Experiments show that the coal will release carbon oxide and hydrocarbons (olefins, alkanes) in the process of the oxidation and spontaneous combustion. The composition and concentration of gas have a corresponding relationship with temperature. In this experiment, the concentrations of CO, CO_2, CH_4, C_2H_4, C_2H_6 and C_3H_8 generated and their variation laws were measured.

(1) Carbon oxide. The gas of CO and CO_2 were detected from the beginning in the low temperature oxidation stage (before 200 °C), the gas precipitation changed slowly, as shown in Figure 3. When the coal temperature was lower than 180 °C, the CO concentration increased slowly, and a large number of

water oxygen complexes were generated by the reaction of coal with water and oxygen. With the increase of temperature, some water oxygen complexes were converted into CO and CO_2. After 200 °C, the gas concentration increased rapidly. The CO concentration reached the first max point at 350 °C. At this stage, the coal sample reacted strongly with oxygen, resulting in an increase in oxygen consumption rate, a rapid decline in oxygen concentration, and a large amount of CO was generated. The gas concentration dropped a little because of decreasing of the content of functional groups such as carboxyl and aliphatic hydrocarbons. As the oxidation reaction of the sample gradually intensified, the C=C double bonds and some oxygen-containing heterocycles in the coal molecule were cracked, the production of CO increased again reaching a peak of 60760 ppm at 555 °C, then began to drop until the end of the experiment.

In the low temperature oxidation stage, a large amount of CO_2 gas was adsorbed between the sample macromolecules due to van der Waals force. In the process of heating, CO_2 gas would gradually desorb, and the gas concentration increased rapidly in the rapid heating stage. In the high temperature stage, the growth rate slowed down slightly, and then increased rapidly. The CO_2 concentration was up to 168704 ppm at 465 °C, and then fluctuated slightly because of the oxidation of aromatic hydrocarbons and aliphatic hydrocarbons, and some CO_2 gas was produced again. Then all kinds of functional groups were exhausted, and the gas concentration decreased rapidly.

(2) Hydrocarbon. The variations of CH_4, C_2H_6, C_3H_8 and C_2H_4 concentrations with coal temperature were generally similar, as shown in Figure 4. The concentration of these four gases were lower before 300 °C, but gradually increased with temperature. Since CH_4 was easy to desorb from coal, it was detected at lower temperature. The gas of C_2H_6, C_3H_8 and C_2H_4 appeared at 120 °C, 195 °C and 135 °C, respectively, and their concentration were relatively low, and increased slowly with the increase of coal temperature.

The concentrations of CH_4, C_2H_6, C_3H_8 and C_2H_4 increased sharply from 340 °C, 330 °C, 350 °C and 345 °C, and reached the peak value at 480 °C, 430 °C, 435 °C and 435 °C respectively. In addition, a certain amount of humic acid contained in coal also produced hydrocarbon gases by thermal decomposition reaction. After that, the decomposition of active functional groups ended, and the concentration of CO was in a high rising state, which inhibited the precipitation of hydrocarbon gases. The concentration of CH_4 dropped after 480 °C, but it was still at a high level until the end of combustion. The concentration of C_2H_6, C_3H_8, and C_2H_4 gas decreased sharply after 435 °C, 480 °C, and 480 °C, respectively, and remained at a low level after 530 °C as it was in the low temperature oxidation stage.

Characteristic temperature. Characteristic temperature is a macroscopic representation of the influence of external environment on the reaction process in the micro-process of coal sample oxidation. Using the growth rate analysis method of index gas CO, as shown in Figure 3. The characteristic temperature points of high temperature oxidation of coal samples were obtained, such as critical temperature (T_1), crack temperature (T_2), active temperature (T_3), speed-up temperature (T_4) and ignition temperature (T_5), as shown in Table 3.

Table 3. Characteristic temperatures (℃)

Index	Critical temperature(T_1)	Crack temperature(T_2)	Active temperature (T_3)	Speedup temperature(T4)	Ignition temperature (T_5)
Value	75	120	195	240	315

The critical temperature stage is the stage of water evaporation and desorption, corresponding to the stage before the critical temperature point. Crack – active – speedup temperature stage is the stage of oxygen inhalation and weight gain, which corresponds to process between the critical temperature and the speedup temperature. The speedup – ignition temperature stage is the stage from the speed-up temperature to the ignition temperature, which indicates that the coal samples undergo thermal decomposition and start violent oxidative cracking. The combustion stage is the stage from ignition temperature to burnout. The critical temperature, crack temperature and active temperature occurred in the low temperature oxidation stage. The CO concentration of the sample increased slowly before the active temperature T_3, and a large number of CO and CO_2 precipitated after the active temperature, and the corresponding gas concentration increased sharply.

Exothermic properties. The oxidation spontaneous combustion is a continuous exothermic process, and the exothermic characteristics determine the spontaneous combustion properties. The DSC curve of the sample is shown in Figure 5, and the change of heat release with temperature is shown in Figure 6. According to the DSC results of coal samples, it can be seen that the heat flow values decreases at the initial stage (the first stage), this is due to the evaporation of water absorbing heat, while the released heat is generated by the physical adsorption of coal. The heat release is relatively small, so this stage is in the endothermic process. In the rapid heating stage (the second stage), the heat flow value increased with temperature, reaching the first peak of heat flow at about 320 ℃. At this time, the active functional groups, such as aliphatic hydrocarbons and oxygen-containing functional groups, reacted with oxygen first, producing a large amount of gas and releasing a large amount of heat. In the high temperature combustion stage (the third stage), the temperature was at about 490 ℃, and the heat flow value began to decrease after reaching the second peak. A large number of small molecule active agent structures and combustible materials reacted with oxygen, and the concentration of carbon oxides increased continuously. Finally, the exothermic peak and the peak of CO and CO_2 gas concentrations of DSC curve appeared. The heat release intensity of the sample was very weak, and the heat release increased sharply. After the burnout temperature (600 ℃) (the fourth stage), the heat flow fall rapidly. At this time, the heat release intensity became weak, but the heat release still increased slowly, and finally reached the peak value of 4714 J/g.

Variation of free radicals. There were a certain amount of free radicals in coal formation, and a large number of free radicals will be generated in the process of spontaneous combustion. The concentration of free radicals has a great effect on spontaneous combustion. As shown in Figure 7. the concentration of free radicals at different characteristic temperature are increasing linearly with temperature.

The temperature increasing will promote production of the free radicals. The free radicals concentration at the active temperature is about 50% higher than which at the critical temperature point. At this time, some structural basic units, bridge bonds, and chemical bonds began to decompose and break, resulting in a large number of free radicals, and whose concentration at the ignition temperature is about twice as high as which at the critical temperature. The activity of free radicals will promote the coal-oxidation reaction and heating until combustion, and meanwhile it will also promote the precipitation of the indicator gas.

Conclusions

1. The physical property test of sample in the West open pit shows that the sulfur content of the coal is only 0.33%, the volatile matter is as high as up to 42%. The specific surface area is much larger, and the macropores and mesopores is more than 95%, which provides favorable conditions for coal-oxygen contacting oxidation and heating.
2. In the low-temperature oxidation stage, the oxygen concentration and oxygen consumption rate changed gently, and changed sharply after 200 ℃, meanwhile the precipitated CO and CO_2 gas concentration increased exponentially. The CH_4, C_2H_4, C_2H_6, and C_3H_8 gas concentrations have similar variation with temperature, that is relatively small at low temperature, and increasing slowly. After about 350 °C, the gas concentrations increase to the peak value. In the combustion stage, with the consumption and cracking of the active functional groups inside the coal molecules, the concentration of the gases decreased.
3. The characteristic temperature were obtained respectively by experiment, that is critical temperature (75℃), crack temperature(120℃), active temperature(195℃), speedup temperature(240℃) and ignition temperature is (315℃). The high volatile content leads to an earlier critical temperature, but the ignition temperature is lower than convention.
4. The thermal analysis results shows that the DSC curve and exothermic processes can be divided into four stages, and the exothermic heat of the coal sample reaches 4714 J/g. The free radical concentration has a linear increasing relationship with temperature. The concentration at the active temperature is about 50% higher than which at the critical temperature stage, and the free radical concentration at the ignition temperature is about twice as high as which at the critical temperature. The oxidative heat release and free radical activity will positively promote the coal oxygen synthesis reaction, and then achieve the state of spontaneous combustion.

Declarations

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Author contributions

Z.Z. and K.L. Mainly responsible for providing the materials for the research, X.L. Mainly responsible for designing experimental schemes; Y.L. and K.G. Mainly responsible for doing experiments and analyzing experimental data; W.Z. Mainly responsible for writing thesis; X.C. and M.W. Mainly responsible for revising thesis.

Competing interests

The authors declare no competing interests.

Additional information

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Figures

Figure 1

Spontaneous combustion in West open pit

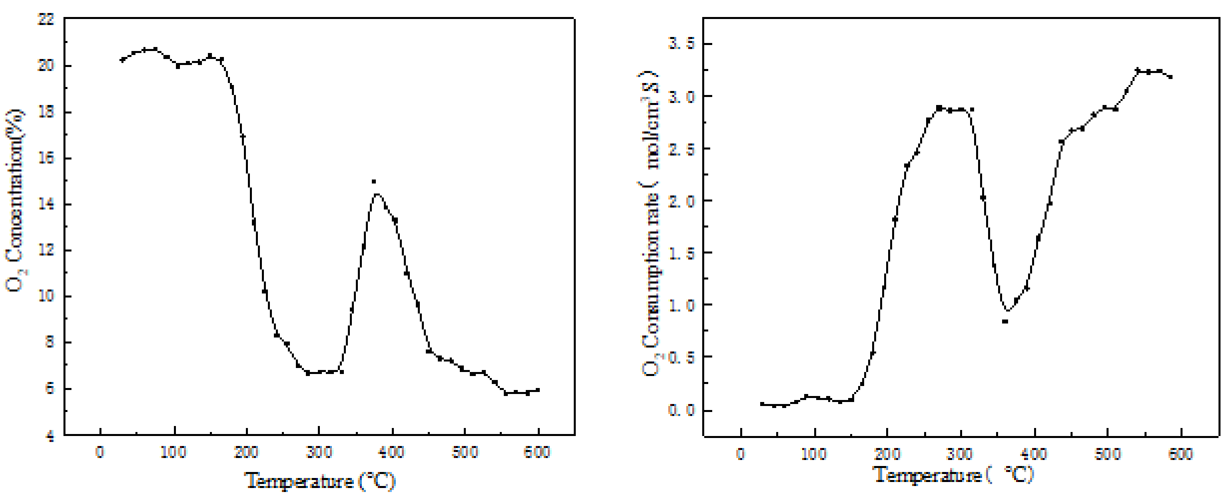

Figure 2

Chang of oxygen concentration and oxygen consumption rate with temperature

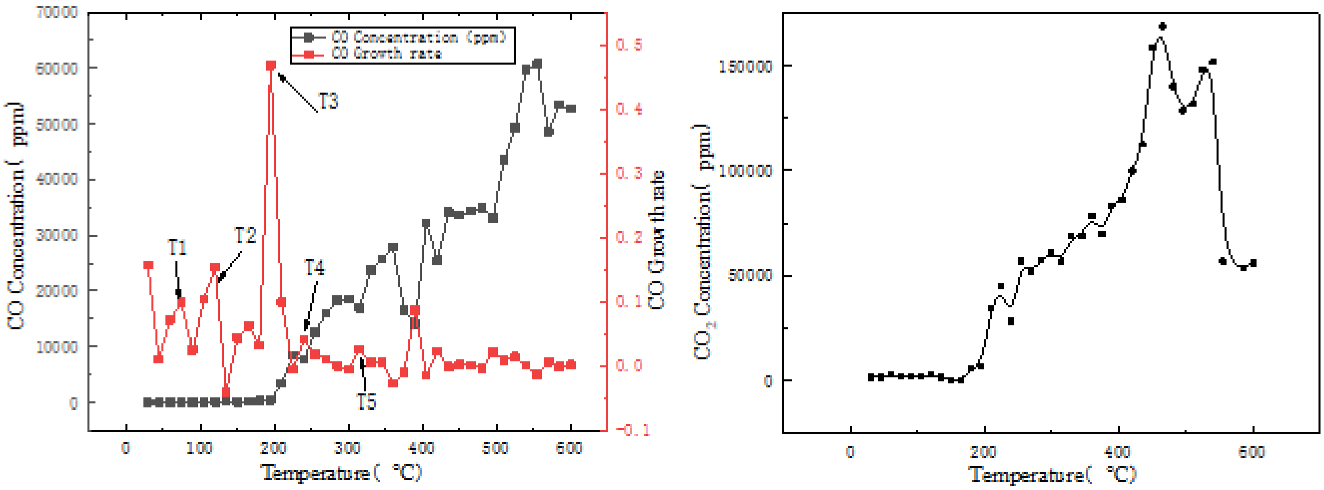

Figure 3

Chang of CO concentration and growth rate and CO_2 concentration with temperature

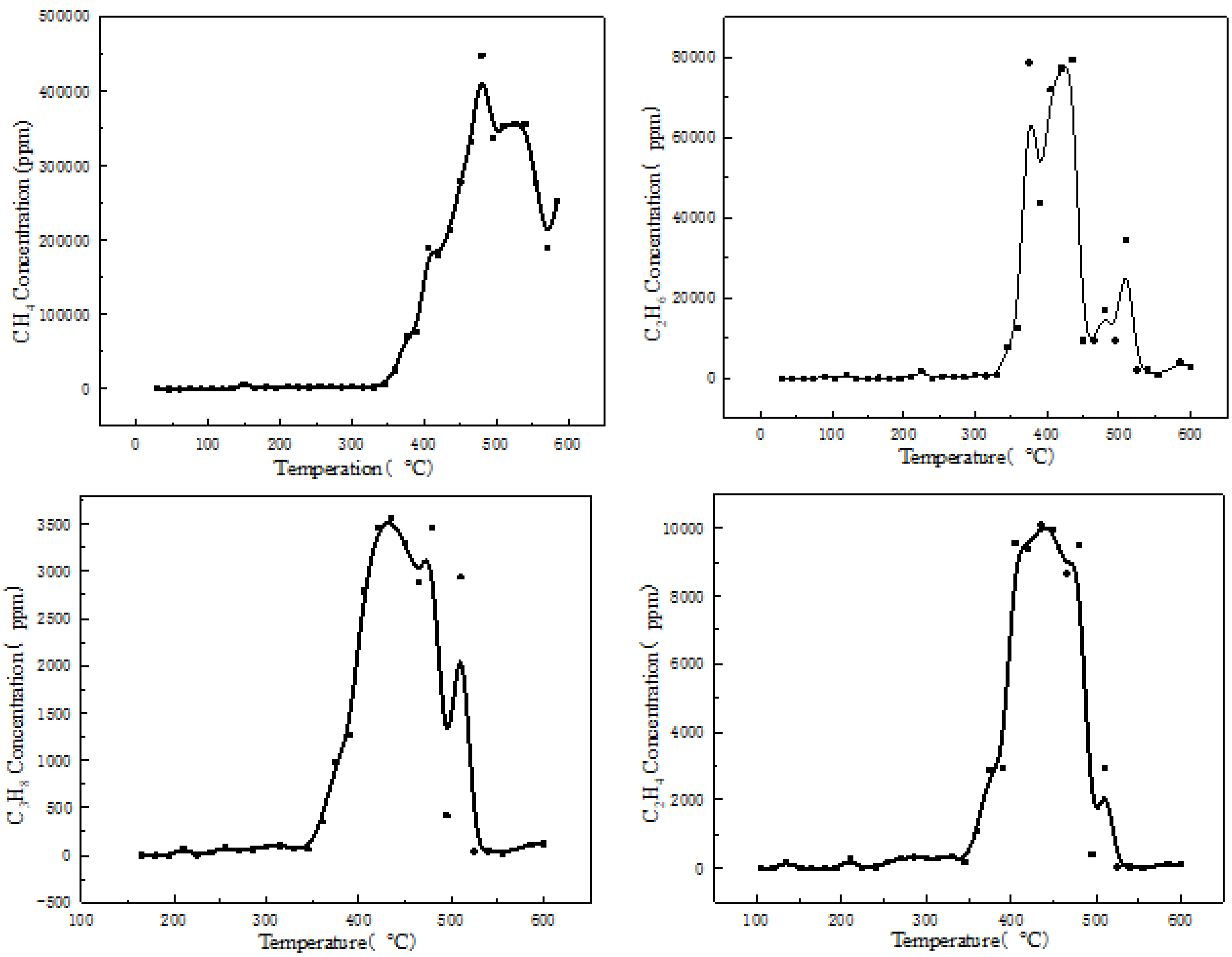

Figure 4

Change of CH_4, C_2H_6, C_3H_8 and C_2H_4 concentration with temperature

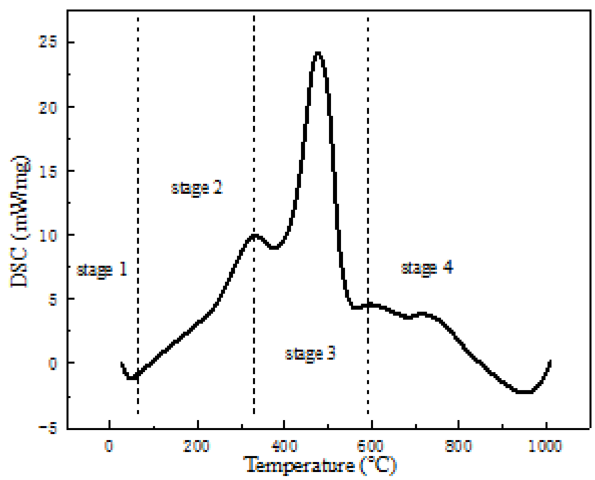

Figure 5

Coal sample DSC curve

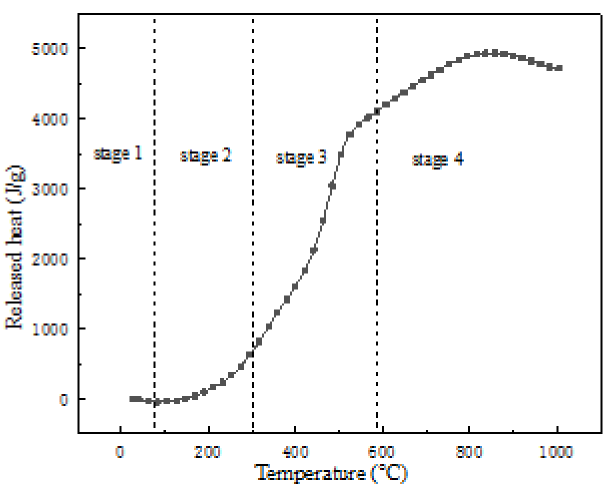

Figure 6

Change of heat release with temperature

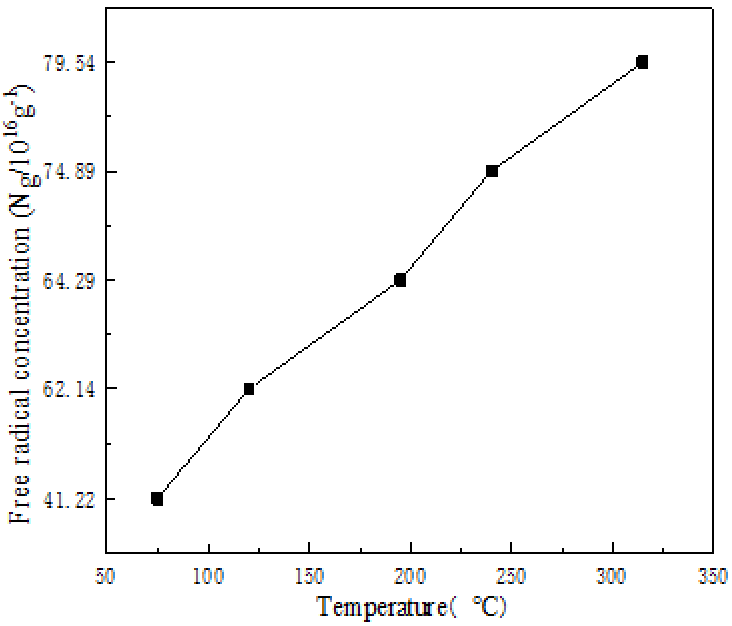

Figure 7

Chang of free radical concentration and characteristic temperature