

Influencing factors and mechanisms involved in the ignition of low-rank coal: Implications for spontaneous combustion

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Abstract. Understanding the ignition mechanism of spontaneous combustion is critical for preventing it. In this work, the effects of different test conditions including oxygen concentration, heating rate, oxidation carrier gas flow rate, and sample amount on the ignition temperature were studied with a thermal gravimetric analyzer. Further, the effects of coal properties on the ignition temperature were also investigated using 15 different low-rank coals. A heterogenous ignition model was proposed that small amount of active species is the key material leading to ignition. The heterogenous ignition mechanism well explained the complex effects of test conditions and coal properties on the ignition temperature of low-rank coal. Finally, an empirical formula for predicting the ignition temperature was derived for the rapid assessment of the spontaneous combustion potential.

1 Introduction

The highly active chemical structure and spontaneous combustion tendency of low-rank coal cause several challenges in its mining, storage, and utilization [1]. several researches have focused on the development of an index for assessing its spontaneous combustion potential, such as absorption of flowing oxygen [2], activation energy of the oxidation reaction [3], adiabatic heating rate [4], characteristic temperatures [5] and indicative gases [6]. Meanwhile, earlier studies [7-9] have indicated that the reaction of the volatiles released from coal with oxygen in the gas-gas phase causes ignition. Some scholars focused on the changes in the active functional groups present in the coal prior to the ignition and suggested that the oxidation of these groups at low temperatures was the main contributor to spontaneous combustion [10]. While the ignition temperature under different test conditions exhibits significant differences, and the known mechanistic knowledge does not adequately illustrate the causes for such variations.

We employed a thermal gravimetric analyzer to simulate the spontaneous combustion conditions. The test conditions including O₂ concentration, oxidation carrier

gas flow rate, heating rate and sample amount were changed to observe the ignition temperature variation. Furthermore, the ignition temperatures of fifteen low-rank coals were also tested to investigate the influence of coal properties. Considering the attribution of the high chemical reactivity from the low-rank coals, a new heterogenous coal particle model was proposed. A heterogeneous mechanism was established in combination of thermodynamics, chemical kinetics, and heat transfer principals to deeply understand the ignition process of low-rank coal. We finally fitted an empirical formula to predict the ignition temperature using ash content, volatile content, and reduction reactivity of coal.

2 Experimental section

2.1 Sample preparation

Fifteen typical low-rank coal samples from the Shendong coalfield in Inner Mongolia of China were used, and their proximate and ultimate analyses are presented in Table 1. The coals were ground to particles of size less than 0.2 mm and dried for 1 h in an anhydrous nitrogen atmosphere at 100 °C before testing.

Table1. Proximate and ultimate analysis of 15 low-rank coal samples.

Sample	Proximate analysis (wt%)				Ultimate analysis (wt%, daf)				
	<i>M</i> (ad)	<i>A</i> (d)	<i>V</i> (daf)	FC (daf)	<i>S</i>	<i>C</i>	<i>H</i>	<i>N</i>	<i>O</i> * _{diff}
BD-8	1.12	44.03	42.35	57.65	0.54	74.22	5.57	1.21	18.46
BET-22	6.60	5.10	31.22	68.78	0.58	82.32	4.53	0.92	11.65
BET-42	5.13	15.57	36.84	63.16	0.20	81.43	4.83	1.03	12.51

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DLT-52	4.89	6.42	32.54	67.46	0.59	82.29	4.59	0.92	11.61
HYC-4	2.01	36.72	41.62	58.38	1.33	74.24	5.37	1.11	17.95
HYC-6	1.98	13.34	39.19	60.81	0.78	80.10	5.01	1.47	12.64
JJ-31	2.80	5.67	39.35	60.65	0.54	80.39	5.18	1.14	12.75
SSLT-3	6.24	11.29	38.43	61.57	2.04	78.22	4.71	0.85	14.18
SSLT-4	6.85	10.21	35.57	64.43	1.87	77.81	4.38	0.78	15.16
SSLT-5	8.68	8.62	34.85	65.15	0.70	78.11	4.28	0.78	16.13
SW-12	6.14	7.39	28.56	71.44	0.25	82.29	3.93	0.76	12.77
WL1-31	4.94	34.00	39.18	60.82	0.70	75.79	4.65	0.74	18.12
WL1-42	4.98	15.85	36.45	63.55	1.47	77.73	4.27	0.77	15.76
YJL-43	4.00	9.48	35.39	64.61	0.36	83.56	4.90	1.19	9.99
YJL-52	2.36	10.09	35.39	64.61	0.31	81.05	4.77	1.08	12.79

* Oxygen content is by difference.

2.2 Experimental conditions

A thermogravimetry (TG) instrument (Seiko co., Ltd. 7300, Japan) was employed to measure the ignition temperature. As schematically shown in the Figure 1, a certain amount of the sample was placed in a platinum crucible and heated until a fire was generated. A mixture of O₂ and N₂ was introduced into the thermogravimeter as the carrier-gas at a particular flow rate to maintain the O₂-N₂ atmosphere. Oxygen concentration, carrier-gas flow rate, heating rate, and sample amount were varied to study their impact on the ignition temperature.

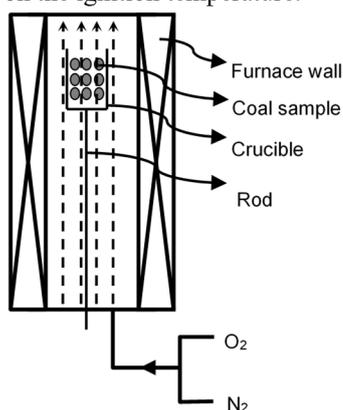


Fig1. Schematic diagram of the experimental apparatus.

2.3 Determination of ignition temperature

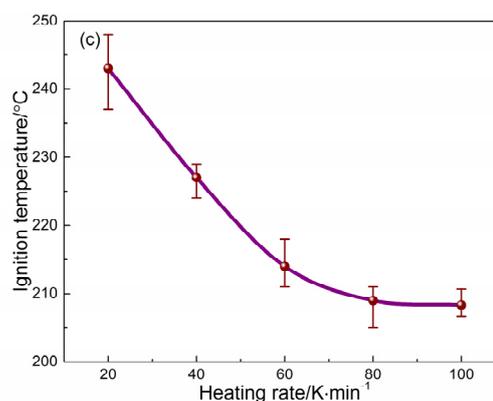
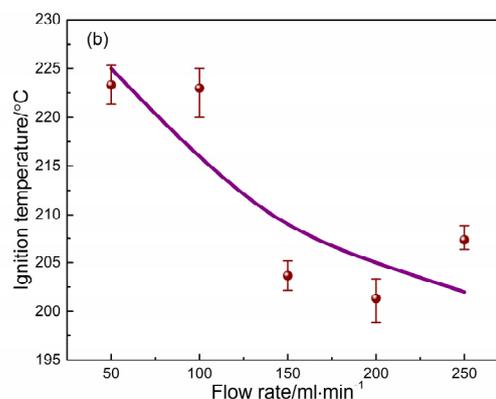
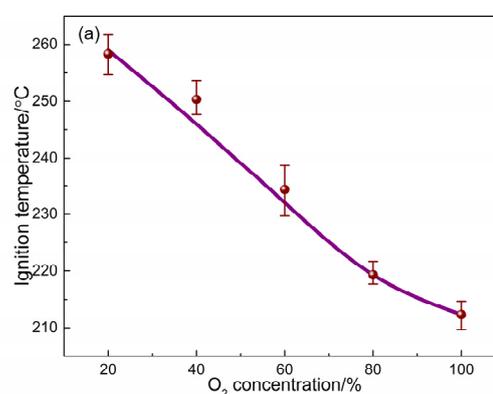
The on-set method [11-12] is not suitable for low-rank coals due to poor reproducibility of results. We defined the temperature corresponding to the point that has maximum slope variation and prior to the combustion peak on the DTG curve as the tested ignition temperature. The method avoided the influence of TG curve's shape and the reproducibility of results was considerably improved (Supporting Information 1).

3. Results and discussion

3.1 Effect of experimental conditions on ignition temperature

DLT-52 was used as a typical sample. The ignition

temperature was tested under different conditions: O₂ concentration ranged from 20 to 100%, carrier-gas flow rate was varied from 50 to 250 mL/min, the heating rate was varied from 20 to 60 °C/min, and sample amount was varied from 5 to 16.5 mg.



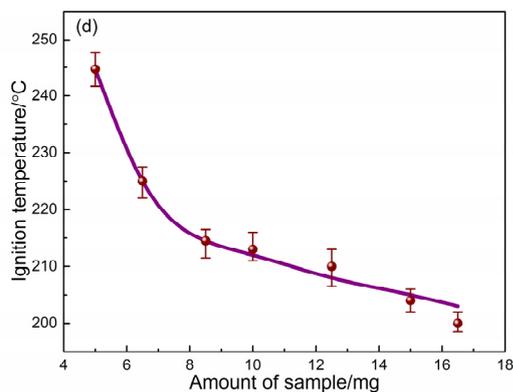


Fig2. Influence of various conditions on ignition temperatures. (a) O₂ concentration, (b) Carrier-gas flow rate, (c) Heating rate, (d) Amount of sample

3.1.1 Effect of O₂ concentration

The other conditions in Figure 2(a) are 10 mg samples, heating rate of 20 °C/min and flow rate of 100 mL/min. When oxygen concentration is 20%, the ignition temperature reaches to 260 °C, when pure oxygen is used, the ignition temperature drops to 212 °C. The difference between the maximum and the minimum temperature is 50 °C. With the increase in oxygen concentration, it can be inferred that the reaction rate of coal and oxygen will accelerate causing an increase in the heat release rate, that will lead to the ignition point of coal reaching earlier resulting in a decrease in the tested ignition temperature.

3.1.2 Effect of oxygen flow rate

The other conditions in Figure 2(b) are a heating rate of 20 °C/min for a sample amount of 10 mg. As the flow rate increased, the ignition temperature decreased. The oxygen flow rate impact the diffusion of the external oxygen to the coal surface. With an increase in the flow rate, the external diffusion is reduced, and the reaction rate is increased, lowering the tested ignition temperature [13].

The range of the tested ignition temperature was 20 °C as the flow rate varied from 50 to 250 mL/min. When the flow rate was greater than 150 mL/min, the range was less than 2 °C, indicating that the influence of the external diffusion is minimized. Extremely high flow rates may disturb the thermos-balance or take away more heat from the system. This causes new challenges, such as an increase in the ignition temperature upon the increase of the flow rate to 250 mL/min.

3.1.3 Effect of heating rate

The other conditions in Figure 2(c) are an O₂ flow rate of 200 mL/min for a sample amount of 10 mg. The tested ignition temperature decreases with an increase in the heating rate. When the heating rate is 20 °C/min, the ignition temperature is 243 °C, which then reduces to 208 °C upon an increase of the heating rate to 100 °C/min, over the 35 °C range. The change in the ignition temperature is much larger when the heating rate varying

from 20 to 60 °C /min than it varying from 60 to 100 °C /min, indicating that the sensitivity is minimized when the heating rate is greater than 60 °C/min.

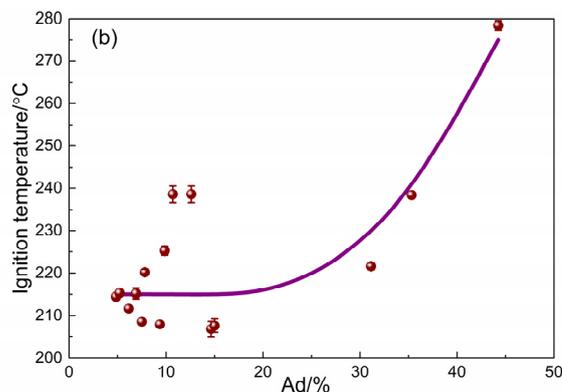
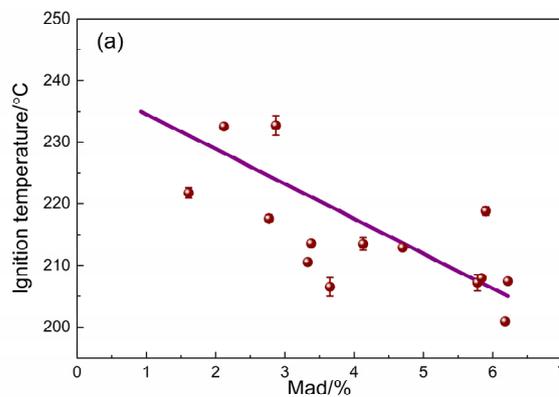
3.1.4 Effect of sample amount

The other conditions in Figure 2(d) are a flow rate of 200 mL/min and a heating rate of 60 °C/min. The effect of the coal sample amount and the tested ignition temperature decreased as the sample amount increased. Variations in the sample amounts cause variations of the sample cylinder height in a special crucible. With an increase in the sample height, the heat transfer from the surface to the interior of the sample stack becomes more difficult, leading to a decrease in the tested ignition temperature.

Apparently, when the amount of sample increased from 5 to 10 mg, the ignition temperature reduced by 35 °C, and when the sample amount increased from 10 to 16.6 mg, the ignition temperature reduced by only 15 °C.

10 mg of the sample with pure oxygen at a flow rate of 200 mL/min and a heating rate of 60 °C/min were chose as the optimal conditions. The ignition temperatures of 15 low-rank coals were measured by these conditions, and the average error was reduced within ±0.5 °C in most cases (Supporting Information 2) .

3.2 Effect of coal chemical properties on ignition temperature



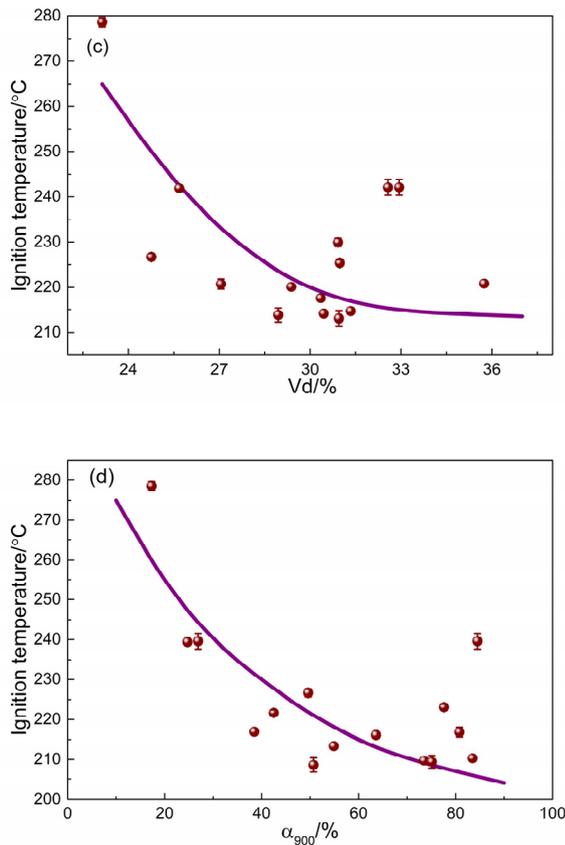


Fig3. Ignition temperature corresponding to the properties of the coal sample.
 (a) Moisture content, (b) Ash content, (c) Volatile content, (d) Reduction rate of CO₂ by coal at 900 °C (α_{900})

3.2.1 Effect of moisture

To minimize the influence of the moisture content, the samples were dried before testing and dried gas was used in this work. In Figure 3(a), a relationship between the moisture content (air-dried basis) and ignition temperature was found that the ignition temperature decreased as the air-dried moisture increased. The residual water in the coal under dried air is related to the number of pores, and higher moisture indicates a more developed pore system. The characteristics of pore in coal are indicative of its age. Therefore, the presence of higher moisture indicates that the coal is younger, which have more active groups and may have a significant influence on its ignition temperature.

3.2.2 Effect of ash content

The results presented in Figure 3(b) show that the higher the ash content, the higher the ignition temperature. The higher ash in coal, the combustible components to be more likely wrapped in ash that acts as flame retardants [14]. In addition, higher ash content reduces the concentrations of the flammable components that further reduces the rate of the oxidation reaction. BD-8 has a high ash of 45%, and its ignition temperature reaches to 280 °C, which is clearly higher than that of the other coals. However, when the ash

content was less than 20%, the ignition temperatures presented no regular change, indicating the involvement of more complex factors impact the ignition temperatures of the low-rank coal.

3.2.3 Effect of volatile content

Figure 3(c) shows that the higher the volatile content, the lower the ignition temperature. To a certain extent, the content of the volatile reflects the content of the active components, which oppositely affects the ignition temperature compare with the flame-retardant effect of ash. Higher volatility leads to a higher coal-oxygen reaction rate and easy ignition.

When the volatile content is higher than 30%, the influence on the ignition temperatures becomes insignificant. It indicates that more complex factors that exert influence on the ignition temperatures need be considered.

3.2.4 Effect of chemical reactivity

The reduction degree of CO₂ by coal at 900 °C (α_{900}) was used as the value that presents the chemical reactivity of the low-rank coal. Figure 3(d) exhibits that the ignition temperature decreases with the increase in α_{900} . The strong correlation between chemical reactivity and the ignition temperature indicates the importance of this parameter in evaluating ignition temperature tendency. Reduction degree of CO₂ by coal is a parameter commonly used for coal gasification and combustion in industry. Higher chemical reactivity is indicative of lower activation energy of ignition, therefore, the ignition could occur at a lower temperature.

In summary, O₂ concentration and flow rate were related to chemical kinetics, heating rate and sample amount were related to heat conduction, and coal properties were related to the thermodynamics of the process. The ignition process is complexly affected by these factors.

3.3 Ignition mechanism and prediction of spontaneous combustion potential

3.3.1 Thermogravimetric behavior around ignition moment

To probe the phenomena that prevail prior to ignition, we focused on the changes on the weight and heat flow online by thermogravimetric analysis. In Figure 4, curves (a), (b) and (c) indicate the typical weight changes of DLT-52 under optimized conditions by TG, DTG, and differential thermal analysis (DTA).

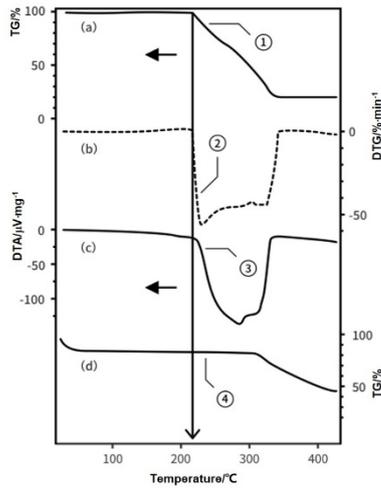


Fig4. Behavior of low-rank coal as per thermogravimetric analyses. (a) TG curve, (b) DTG curve, (c) DTA curve, (d) TG curve in N₂ atmosphere.

The TG plot shows a typical weight loss trend, which indicated the initiation of the weight loss at the location marked ① at around 230 °C; the loss ended at approximately 350 °C where the plot became flat again. The weight loss can be described further by the DTG plot. At the location marked ②, the loss rate became higher within a very short time at around 230 °C, indicating that the weight loss occurred suddenly. The change in the DAT plot corresponding to the weight loss temperature (Location ③) testified that the weight loss is an exothermic process. They are typical ignition process that ignition happens at an ignition temperature recorded by thermogravimetric.

However, a careful comparison of the curves revealed that the evident change in DTG is prior to the evident change in TG, which is a key evidence that at the point of ignition, the weight loss is not evident, implying that only a small amount of substance initially undergoes ignition. Therefore, this result clearly indicates that a small amount of substance exists in the coal that is more active than the rest of the sample and is the origin of the ignition. It is considerably different from the theory of homogeneous ignition, wherein the volatiles in the coal firstly react with oxygen in the gas phase [15-18]. The TG plot of the same sample in N₂ under the same heating conditions was compared with the TG plot; no weight loss was detected in coal around the ignition temperature (Location ④ in Curve (d)).

3.3.2 Heterogeneous ignition model of the coal surface

A low-rank coal particle model was illustrated in Figure 5(a). A small amount of active species heterogeneously covers the surface of the coal particles (assumed as spherical particles). The species could be the side chains with active chemical structures, which have different physical and chemical properties from the coal particles, and can be considered as another phase that adheres to the

coal particle surface.

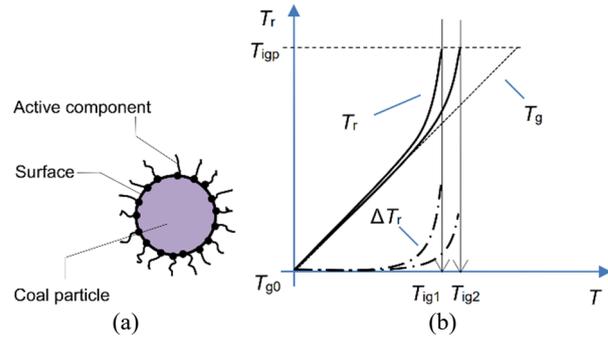


Fig5. Model for the heterogeneous ignition mechanism of low-rank coal. (a) Particle model demonstrating the heterogeneous distribution of active ignition matter over the coal surface, (b) Temperature profiles of the ignition matter before ignition.

In a thermogravimetric test, the tested temperature (T) is controlled by employing a heating rate that equals the temperature of the carrier gas (T_g) and is expressed by Eq. (1).

$$T = T_g \quad (1)$$

In the heating process, the heat was transferred to the coal by the carrier gas. T_g is the temperature of the active species (T_r) on the surface. The case of no activity is depicted by a straight dotted line shown in Figure 5(b). After the active species react with oxygen, reaction heat is generated and transferred to coal particles. In the non-equilibrium state discussed above, the time for the transfer of the reaction heat to the coal particles is limited, then the accumulated heat increases the temperature of the active species. This increment in T_r (ΔT_r) is described by the dot-dash line in Figure 5(b). Therefore, T_r is the sum of T_g and ΔT_r , depicted by the solid line shown in Figure 5(b), and is expressed by Eq. (2).

$$T_r = T_g + \Delta T_r = T + \Delta T_r \quad (2)$$

Once T_r reaches the temperature of the ignition point (T_{igp} , a constant), the active species ignite and then induce the subsequent ignition of coal particles. T at this moment is the tested ignition temperature T_{ig} (expressed by Eq. (3)). Different conditions have impact on the heat generation and heat loss, resulting in different ΔT_r profiles before ignition, and then the value of T_{ig} also differ.

$$T_{igp} = T_{ig} + \Delta T_r \quad (3)$$

The concentration of the active species is C_r , concentration of oxygen is C_{O_2} , coal-oxygen reaction is a first-order reaction with k as the apparent rate constant, and reaction enthalpy is ΔH . The heat generation rate is represented by $kC_rC_{O_2}\Delta H$. When oxygen concentration is increased, C_{O_2} increases. Further, with an increase in flow rate, the collision probability between O_2 and coal increases. These lead to an increase in k , that enhances the heat generation rate. Thus, ΔT_r becomes larger at the same heating rate, and T_{ig} decreases as observed in Figure 2(a) and Figure 2(b). When the heating rate increases, the heat generation rate also increases and the heat transfer time reduces, which cause increase of ΔT_r , and then T_{ig} decreases as observed in Figure 2(c). When the heating

rate, gas flow rate, and oxygen concentration are fixed, thermal conductivity of coal becomes the factor influencing ΔT_r . Higher volumes of the sample cause greater heat accumulation, leading to lower ignition temperatures as shown in Figure 2(d). In summary, the effects of oxygen concentration and carrier-gas flow rate ascribe to the oxygen-coal reaction kinetics, and the effects of heating rate and amount of sample ascribe to the heat conduction process.

When the ash content in the coal increases, the concentration of the active species decreases and the heat generation rate is reduced. The ignition temperature is then elevated as described in Figures 3(b). The effect of volatiles on the ignition temperature shown in Figure 3(c) similarly can be explained.

To sum up, the ignition mechanism is very useful for understanding the special ignition behavior of low-rank coal and is valuable for managing the safety aspects of coal stacks to preventing their spontaneous combustion.

3.3.3 Prediction of spontaneous combustion potential

The inherent properties including A_d , V_d , and α_{900} are associated with the complexity of the analyzed results. Using a statistical software (JMP), we fitted the data to a formula which is expressed by the following equation and is based on the ignition temperatures of the 15 coals.

$$T_{ig} = 290 + 0.54e^{2.27\frac{A_d}{V_d}} - 19\ln(\alpha_{900}) \quad (4)$$

where T_{ig} is the ignition temperature (°C), A_d is the dry basis ash content (wt%), V_d is the dry volatile content (wt%), and α_{900} is the reduction degree of CO₂ by coal at 900 °C.

The comparison of residuals between the predicted and experimental values appears to present a good correlation ($R^2=0.91$); the probability of correct prediction is 0.95 when the confidence interval of error is within ± 11.0 °C, and the probability could be 0.997 if the confidence interval allowed is within ± 14.4 °C (Supporting Information 4).

4 Conclusion

Low-rank coal is likely to become the main energy resource in the future, especially for China. The use of low-rank coal, however, causes challenges in terms of its spontaneous combustion. In this work, the influence factors including the test conditions in thermal gravimetric analyzer and coal properties were studied. The results demonstrated that the spontaneous combustion of low-rank coal was complexly affected by those factors. Combined with the typical behavior in thermal gravimetric analyzer, a heterogeneous ignition model was proposed for low-rank coal. Based on the presence of a small amount of active species that heterogeneously adhere to the surface of the coal particles, it revealed that the active matter, rather than coal particles, firstly ignites. Further, the change tendency of ignition temperature was well explained by three Equations deduced from the

heterogeneous mechanism. In addition, an empirical formula to predict the ignition temperature using ash content, volatile content, and reduction reactivity of coal was established.

In a word, this paper provided a new understanding of the special ignition performance of low-rank coal, which will aid in safe and effective management of coal stacks in terms of preventing their spontaneous combustion.

Supplementary Material

S11: Method for determining the measured ignition temperature

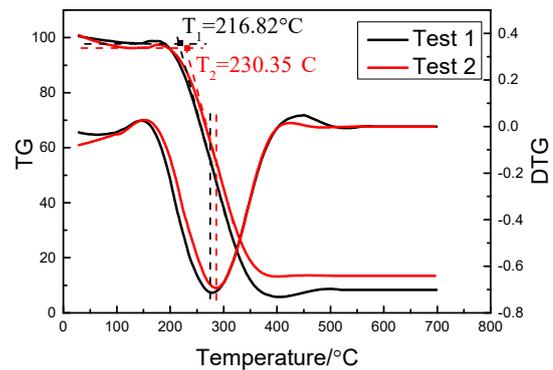


Figure S1. TG-DTG tangent method for determination of ignition temperature.

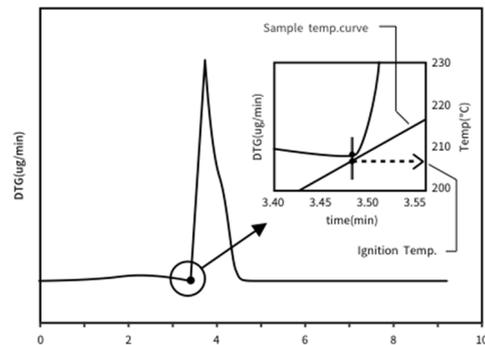


Figure S2. Schematic diagram of determination method of ignition temperature.

Table S1. Ignition temperature of YJL-52 measured with optimized conditions.

Method	Test1	Test2	Range
TG-DTG tangent method	216.82	230.35	13.53
DTG inflection point method	227.6	225.5	2.1

The details of TG-DTG tangent method is showed in Figure S1. The TG curve and DTG curve of YJL-52 under the same experiment conditions in test 1 and test 2 are showed as red line and black line. Apparently, as shown in Table S1, two groups curves are slightly different in shape, resulting in a difference of 13.53 °C in the ignition temperature.

As shown in Figure S2, the temperature corresponding to the maximum slope point on the DTG curve is defined

as the ignition temperature. The definition avoids the effect of curve shape, and the measuring range is reduced to 2.1 °C, which exhibits good repeatability.

S12: Validation of optimized operation conditions

Table S2. Ignition temperature of 15 low rank coals measured with optimized conditions.

Sample	Average	Range	Sample	Average	Range
BD-8	278.5	±2.2	SSLT-4	209.65	±0.5
BET-22	216.1	±0.2	SSLT-5	210.35	±0.1
BET-42	208.6	±3.6	SW-12	216.8	±2.4
DLT-52	213.4	±0.4	WL1-31	223.05	±1.7
HYC-4	239.4	±0.4	WL1-42	209.35	±3.1
HYC-6	239.55	±3.9	YJL-43	221.65	±1.3
JJ-31	216.9	±0.2	YJL-52	226.55	±2.1
SSLT-3	201.95	±0.1			

S13: Reduction degree of CO₂ by coal at 900 °C

Table S3. Reduction rate of CO₂ by coals at 900 °C.

Sample	α_{900}	Sample	α_{900}	Sample	α_{900}
BD-8	17.3	HYC-6	26.9	SW-12	80.8
BET-22	63.6	JJ-31	38.5	WL1-31	77.6
BET-42	50.7	SSLT-3	84.5	WL1-42	75.1
DLT-52	54.9	SSLT-4	73.5	YJL-43	42.5
HYC-4	24.7	SSLT-5	83.5	YJL-52	49.6

S14: The reliability of ignition temperature prediction formula

As shown in Figure S3, it appears a good correlation ($R^2=0.91$) between the predicted value and the measured value, and the residual is a normal distribution, which indicate the reliability of the prediction. The eigenvalues of the normal distribution curve are $\mu=0$ and $\sigma=5.6$, which means that the confidence interval of the prediction error is ± 11.0 °C when the probability of correct prediction is 0.95, and the confidence interval of the prediction error is ± 14.4 °C when the probability of correct prediction is 0.997. This prediction formula can be used to rapidly estimate the ignition temperature of low rank coal, thus helping to evaluate the tendency of spontaneous combustion.

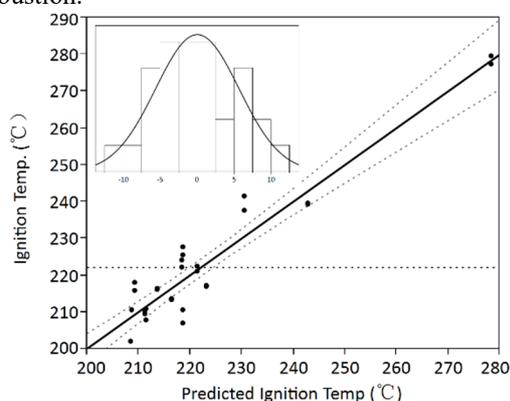


Figure S3. Residual analysis of predicted and experimental values.

Acknowledgments

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