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THERMAL BEHAVIOR AND KINETICS OF SULFIDE CONCENTRATES

by

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Thermal behavior of sulfide concentrates can give rise to many serious problems in its storage and transportation. In order to uncover the thermal behavior of sulfide concentrates, as well as obtain the kinetic parameters, four representative samples were tested using TG-DTG-DSC techniques in the presence of oxygen from 20 °C to 1000 °C and with three different heating rates of 5, 10, and 15 °C per minute. Meanwhile, corresponding activation energies of sulfide concentrates were also determined by the Coats-Redfern method (199.4-234.9 kJmol⁻¹ for Sample 1, 203.6-235.9 kJmol⁻¹ for Sample 2, 234.7-255.6 kJmol⁻¹ for Sample 3, and 199.7-254.9 kJmol⁻¹ for Sample 4). Results indicate that the heating rate is an important factor affecting the thermal behavior of sulfide concentrates. The peak temperature corresponding to the maximum mass loss rate of the ore sample at different heating rates is different and the sulfide concentrates at heating rate of 5 °C per minute is more prone to spontaneous combustion. Furthermore, this thermodynamic method was demonstrated to be effective for investigating and predicting the thermal behavior of sulfide concentrate and the activation energy index was reasonable for determining its spontaneous combustion tendency.

Key words*: sulfide concentrates, coats-Redfern integration method,
         thermal behavior, kinetics, activation energy*

Introduction

When the sulfide ores are in contact with air, oxidation will occur and release lots of heat. The continuous accumulation of heat can accelerate the oxidizing reaction and cause the ore temperature reaching the auto-ignition point [1-3]. Sulfide concentrate is the term of selected metal sulfide ore which is a higher-grade powdery substance obtained by separating undesired components from sulfide metal ore, such as iron sulfide, copper sulfide, lead sulfide and zinc sulfide [4]. Due to the increase of specific surface area, the chemical activity of sulfide concentrates is largely increased, which make them easily to cause spontaneous combustion after a period of time of storage. Once a fire broke out in stored sulfide concentrates, in the spontaneous combustion process will release a large amount of SO_2 and other toxic gases (H_2S, SO), which will result in anoxia and asphyxiation in the ore bin as well as seriously threaten the workers' lives and health. In the humid environment, a large amount of acidic substances

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will be generated to corrode many metal facilities [4]. During the stacking period, the knotting phenomenon often occurs so that the sulfide concentrate loses its commercial value and its oxidation product will be a new pollution source of acidic wastewater. The occurrence of fire not only worsens the working environment, pollutes the surface air, causes huge waste of mineral resources, but also destroys production equipment, affects the normal operation and even causes major casualties. The thermal behavior of sulfide concentrates is one of the important indicator reflecting the propensity of spontaneous combustion. Therefore, revealing the thermal behavior during the oxidative decomposition of sulfide ore, and further determining its reaction kinetic parameters will provide an important basis for the fire prevention classification of sulfide concentrate in storage and transportation, and has important guiding significance for the prevention of spontaneous combustion disaster of sulfide concentrates.

Thermal analysis is a technique for measuring the physical properties of a substance in relation temperature at a programmed temperature. Thermal analysis kinetics is an approach of studying the physical properties of a substance and the rate and mechanism of a chemical reaction using thermal analysis techniques to obtain the kinetic parameters and the corresponding reaction mechanism functions. Because of its convenient operation and low sample consumption, the method is widely used in the field of kinetic parameter testing such as material polymerization, petroleum pyrolysis, and coal pyrolysis [5-9]. With the advancement of thermal analysis technology, many scholars have used thermal analysis methods to determine the kinetic parameters of oxidative decomposition of sulfide ore, of which, the differential method and integral method are most commonly adopted. Chen *et al.* [10] solved the reaction kinetic parameters of pyrite thermal decomposition process before and after mechanical activation by Friedman method. Yang *et al.* [11] calculated the apparent activation energy of sulfide ore before and after pre-oxidation based on Ozawa method. Some references have been carried out with TG to analyze the thermal behavior of coal and coal-biomass blending [12-14]. Li *et al.* [15] solved the oxidation kinetic parameters of mechanically activated pyrite based on the Bagchi method for apparent activation energy and reaction order.

The absorption and exothermic properties of the sulfide concentrate during oxidative decomposition should be the combined thermal properties of all minerals in the ore, not a single mineral thermal behavior. Current thermal analysis of sulfide ores has been focused on a single way. For example, thermogravimetric (TG) analysis was used to study the thermal decomposition kinetics of unactivated pyrite under high purity argon gas protection and pyrite with different mechanical activation time at different heating rates [4]. The oxidative behavior of sphalerite in oxygen atmosphere was studied by TG means [10]. The thermogravimetric-differential thermal analysis (TG-DTA) was used to deal with roasting process of pyrite in [11]. The thermal analysis specifically for sulfide concentrates in air atmospheres is rare. Researchers [16] used DTA to determine the thermal spectrum of sulfide ore. In view of the fact that different minerals in the ore exhibit large differences in temperature and heat release of oxidative exothermic reaction, there may be overlaps in each reaction, which may set-off each other, so a single thermal analysis method for the thermal properties of sulfide ore has certain limitations and it is hard to have an essential presentation for the oxidation reaction. To our knowledge, there has been little research to combine TG with differential scanning calorimetry (DSC) analysis to study the oxidative combustion characteristics of sulfide concentrates. Therefore, an advanced integrated thermal analyzer to perform TG-DTG-DSC thermal analysis on four sulfide concentrate samples were used in our work for revealing the thermal behavior and its corresponding kinetic parameters of spontaneous combustion.

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Materials and methods

Sample Preparation

Four representative sulfide concentrate samples (named as: Sample 1, 2, 3, and 4, respectively) were taken from a typical sulfide mine in Guangdong Province, China. When the samples were transported to the lab, they were first dried at 60 °C for 10 hours in a constant temperature oven. Then, the ore samples are crushed in an air environment and sieved to 300 mesh (50 μm). The particles size distributions of the sieved samples for tests are shown in fig. 1, from which it can be found that the particles size distributions for the four ore samples are basically uniform. The chemical compositions of ore samples are peovided in tab. 1, from the table it is noticed that iron and sulfur contents are high in four samples (Sample 1 contains the most sulfur content, followed by Sample 2, 4, and 3, respectively), which also indicates that each sample contains a variety of impurities.
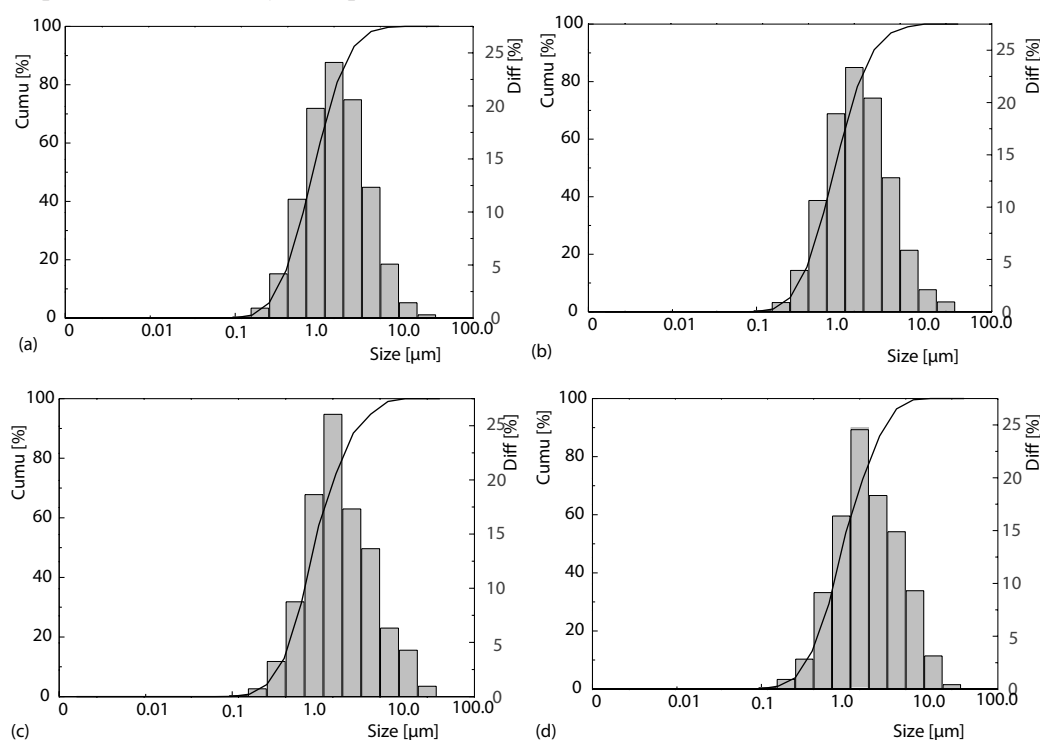

Figure 1. Particle size distribution of samples; (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

Table 1. Main chemical compositions of the samples, mass fraction [%]

Sample	Fe	O	S	Si	Ca	Ag	Se	Ga	Cu
1	40.12	23.62	21.37	–	–	–	5.01	5.39	4.44
2	39.36	24.58	18.66	3.38	8.37	0.65	–	–	–
3	34.55	23.21	15.02	1.51	6.77	–	4.15	4.48	6.57
4	33.69	20.80	17.35	1.27	10.31	–	6.34	4.62	5.11

Test equipment

In our work, the XRD were operated to test microtextures of sample and its mineralogy on semiquantitative scale, resepectively. the TG-DSC experiments were carried out using a synchronous thermal analyzer of American TA company (SDT Q600 V20.9 Build 20) which is equipped with a power compensation system, a digital acquisition system, a control system, and a cooling system. The heating rate and the test termination temperature can be controlled by the test system internal program.

Test condition

Because the sample mass will lead to a slow heat transfer and a high temperature gradient in its interior, which will affect the accuracy of DSC test. Therefore, the sample mass was controlled to about 10 mg. The heating rates of the test conditions were set to 5, 10, and 15 °C per minute, respectively, and the test temperature was from ambient temperature (20 °C) to 1000 °C. During the test, synthetic air (20.5% O_2 in N_2) was used as the purge gas with a flow rate of 40 mL per minute.

Results and discussion

Micromorphology and microcrystalline structure

The microcrystalline structure of the crushed samples were analyzed by the XRD. Figure 2 shows the XRD pattern of the four ore samples and the XRD analysis results of the samples have shown that the main composition in the ore sample was pyrite (FeS_2) while the impurities contained in the sample were silica, calcium carbonate, *etc*.
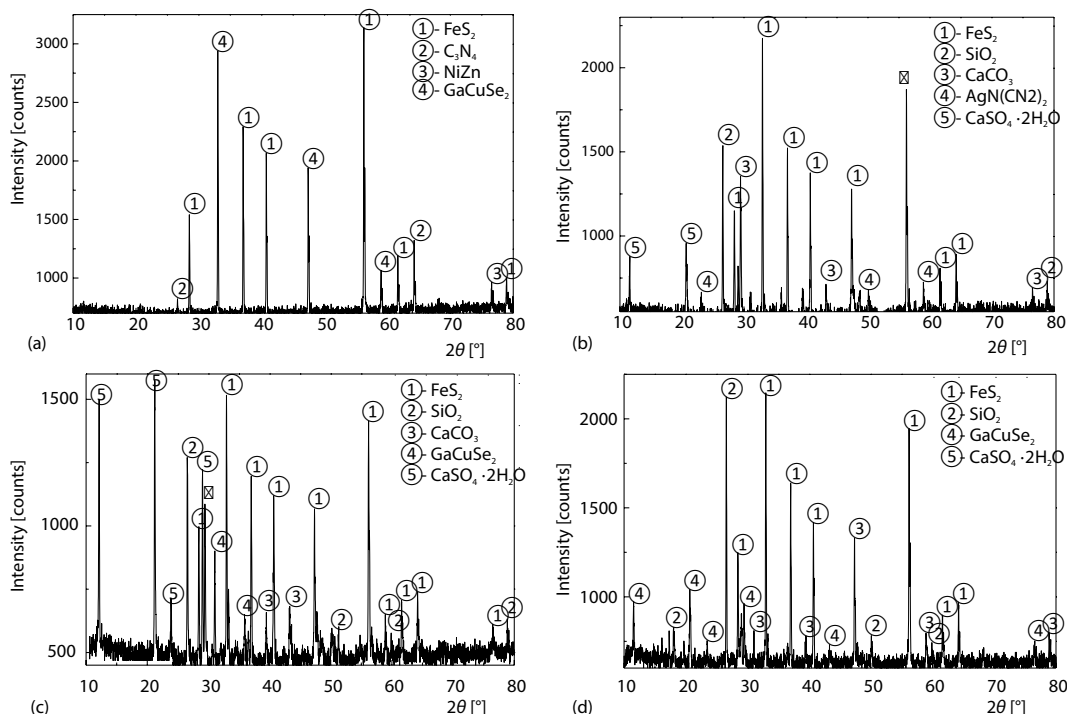

Figure 2. The XRD analysis of the ore samples; (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

The TG-DTG curve analysis

The TG curve represents the relationship between sample's weight and experimental temperature, and the DTG curve is obtained by taking derivative of the TG curve with respect to time, indicating the change rate of weight with time. It can be seen from fig. 3 that the combustion behavior of the ore sample can be generally divided into three-stages. Stage I (<400 °C) is the water evaporation stage. The mass of sample is slightly reduced after the start of temperature rise. At this stage, the water evaporation process of the physical adsorption of the sulfide concentrate is basically no chemical reaction, and the mass loss rate is about 5% (for Sample 4 is nearly 10%). Among them, Samples 2-4 taken on the pattern of step weightlessness which was related to the crystallization water precipitation in the ore sample and the $CaSO_4 \cdot 2H_2O$ losing crystal water step by step with gradually increasing temperature. Stage II (400-600 °C) is the ore sample combustion stage. When the temperature is continuously raised to about 400 °C, the ore sample begins to enter a very fast mass loss stage, and a maximum mass loss peak occurred between 400 °C and 600 °C. At this time it should be the precipitation and combustion process for sulfide concentrate, and the mass loss rate for ore sample also reach its maximum value. Stage III (>600 °C) is the gradual stabilization phase of the burning of the ore sample. And the combustion content mass of the four kinds ore samples is maintained between 65% and 75%.
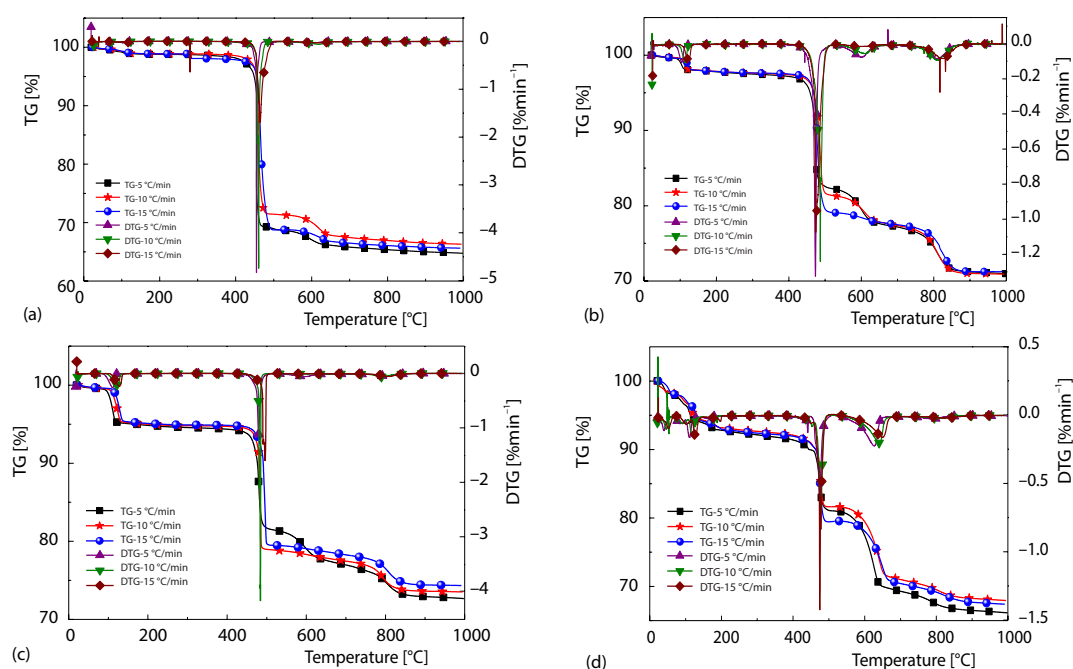

Figure 3. The TG-DTG curves of four ore samples at heating rates of 5, 10, and 15 °C per minute; (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

Form the DTG curves, the mass loss peak due to crystal dehydration for ore Samples 2-4 is noticed in Stage I. The maximum mass loss peak for all of ore samples appeared in the Stage II at which SO_2 were released because of the rapid combustion reaction of the sulfide concentrates under the air environment. In the Stage III, the reaction process had been basically stable. Interestingly, it was found that both of the Samples 2 and 4 had mass loss peaks here,

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which may be due to the fact that the product of the previous oxidation, Fe_nO_m, reacted further with SO_2 or O_2, and then there was no mass loss peak. In addition, fig. 3 shows that the maximum mass change rate of the four samples is not directly related to the heating rate. As matter of fact, the maximum mass change rate of the ore Samples 1 and 2 decreases with the increase of heating rate. However, which for Sample 3 occurs at a heating rate of 10 °C per minute, and the maximum mass change rate of the ore Sample 4 increases with the increase of the heating rate.

The DSC curve analysis

The DSC curves of ore samples in air at the heating rates of 5, 10, and 15 °C per minute are shown in fig. 4. The ordinate of the DSC curve indicates that the heat supply required for zero temperature difference between the combustion sample and the reference substance (air heated without samples). The part of above zero scale means exothermic process, and that of below zero scale means endothermic process. In theory, the two curves of DTG and DSC are related to each other, that is, the mass loss peak has a one-to-one correspondence with the exothermic peak (or endothermic peak), but since the ore sample is precipitated firstly and then burned. Therefore, there may have a slightly delayed phenomenon among sample exothermic peak and its mass loss peak.
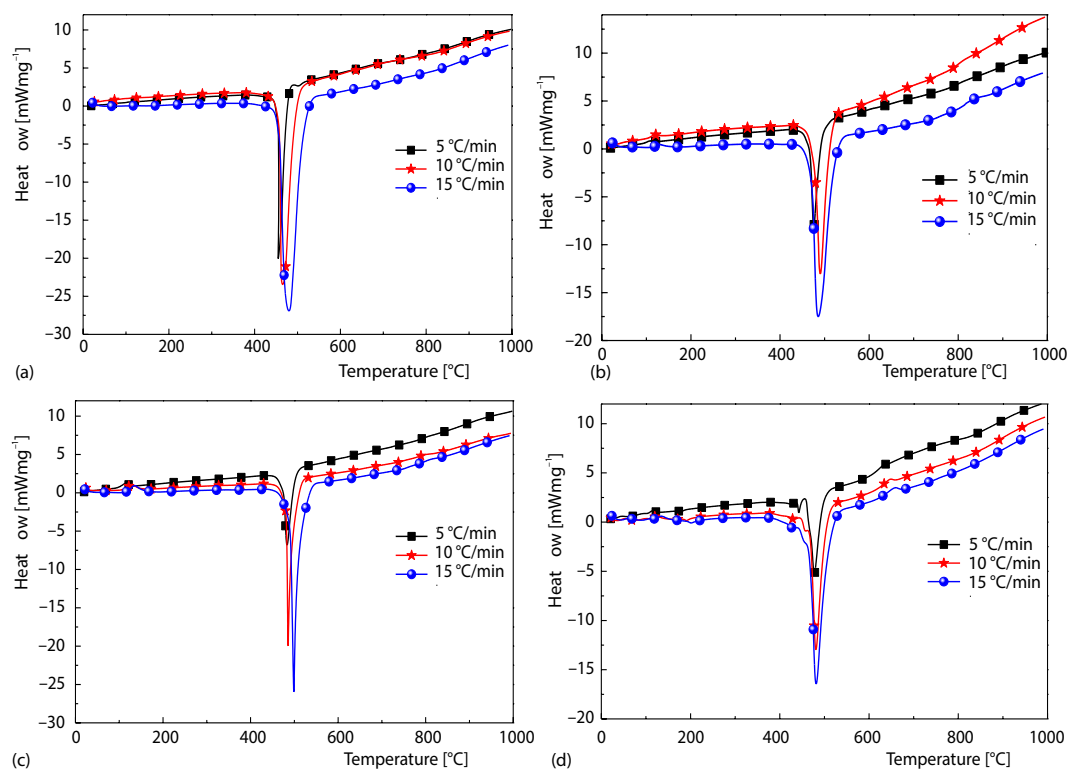

Figure 4. The DSC curves of four samples at heating rates of 5, 10, and 15 °C per minute; (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

As can be seen from fig. 4, the combustion of the ore sample mainly presents an obvious endothermic process and a slow exothermic process. The endothermic process (400-600 °C) has an obvious endothermic peak corresponding to the mass loss peak of the DTG curve in combustion reaction of the stage II. The exothermic process (>600 °C) is mainly a steady upward trend,

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and the ore sample continues to occur an oxidative exothermic reaction at high temperatures. It can be found that the smaller the heating rate, the earlier the peak of the endothermic heat appears. As the heating rate increases, the heat absorption of the ore sample also increases.

Ignition characteristic analysis

The ignition point temperature is the main indicator of the thermal behavior of materials. The lower the ignition point temperature, the better the ignition performance of the material, and the easier it is to catch fire. Thus finding the ignition point of the ore sample is of great significance for the prevention and control of spontaneous combustion of sulfide concentrate heap in storage and transportation. To determine the ignition point, the TG-DTG method was used (the ignition point data T_i is shown in tab. 2). The ignition point temperature is obtained by the mapping method. Taking the ore samples at heating rate of 5 °C per minute as an example. As shown in fig. 5, DTG_m (the maximum burning speed) and its corresponding temperature T_m at the crossing-point are obtained by the TG-DTG curves. Then the ignition point temperature of the ore sample can be calculated. The main works are firstly, finding the peak-point A on the DTG curve where the mass loss rate is the largest, then passing the point A to make the vertical line TG curve at point B, and passing the point B to make the tangent line of the TG curve. Obviously, the DTG of point C is 0, and passing point C to make a vertical line TG curve at point D where indicates that the ore sample gradually loses weight from point D, and the temperature corresponding to points C and D is the mass loss starting temperature T_0. Finally, passing point D make the horizontal line through the tangent of point B and the cross-point is at point E, where the temperature corresponding to point E is the ignition temperature T_i.
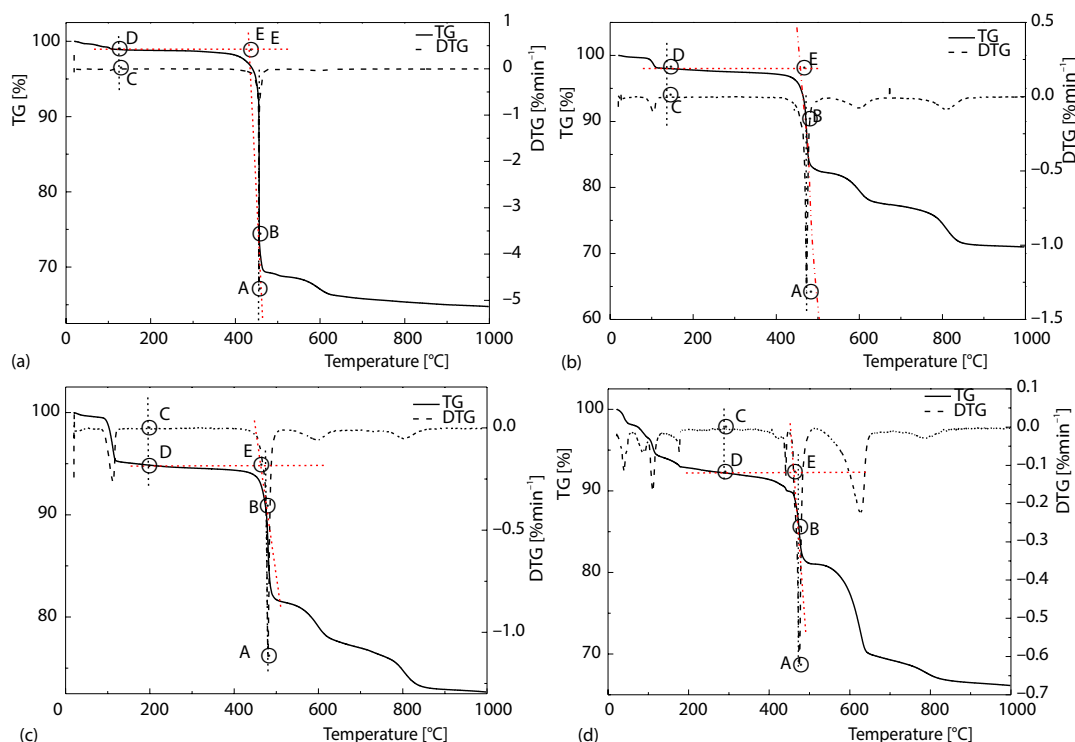

Figure 5. Determination of ignition temperature of four samples at a heating rate of 5 °C per minute: (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

Table 2. Combustion characteristic parameters of samples at different heating rates, [°C]

Samples	T_m			T_0			T_i			
	5	10	15	5	10	15	5	10	15	Average
1	455.3	469.3	465.2	124.0	133.0	157.2	452.6	468.4	464.2	461.8
2	472.8	486.4	474.8	136.7	241.2	153.0	472.8	486.4	473.7	477.6
3	480.4	484.8	496.7	194.1	159.8	211.1	480.3	484.8	496.7	487.2
4	474.8	477.0	474.6	287.9	291.8	295.9	478.4	481.6	476.3	478.8

Table 2 shows the ignition characteristic parameters of the ore sample at the heating rates of 5, 10, and 15 °C per minute obtained according to the TG-DTG analysis result, including the maximum burning temperature, T_m, the mass loss starting temperature, T_0, and the ignition temperature, T_i. The lower the ignition temperature of sulfide concentrate, the lower the maximum ignition temperature T_m is, as well as the lower weight loss starting temperature T_0, which indicates that these ignition characteristic parameters are related to the spontaneous combustion tendency of the sulfide concentrate. Furthermore, the ignition temperature is approximately equal to the temperature of sample's maximum mass change rate, so that the temperature at which the maximum mass change rate was found can be approximated as the ignition temperature under the corresponding conditions. It can be seen from tab. 2 that, as a whole, T_i increases with the increasing heating rate, which may be due to the thermal hysteresis effect caused by an increase in heating rate. As the rate of temperature continues to increase, T_i begins to decrease. All of samples showed the phenomenon where the ignition temperature at 10 °C per minute was greater than 5 °C per minute and 15 °C per minute, and in the case of the same heating rate, all of samples show the ignition temperature from high to low as: $3 \rightarrow 4 \rightarrow 2 \rightarrow 1$. For the reason that the order of reaching the ignition point from difficulty to ease can be described: $3 \rightarrow 4 \rightarrow 2 \rightarrow 1$.

Reaction kinetics analysis

Thermal analysis kinetics is an approach to study the physical properties of substances, the reaction rate and mechanism of chemical reactions using thermal analysis techniques to obtain reaction kinetic parameters and mechanism functions [14]. In the air atmosphere, the main mineral(pyrite) in the sulfide concentrate will undergo a typical gas-solid reaction, as shown in eq. (1):

$$4FeS_2 + 11O_2 = 2Fe_2O_3 + 8SO_2 \tag{1}$$

According to the theory of thermal analysis kinetics, the expression of reaction kinetic rate for sulfide concentrate can be expressed:

$$\frac{d\alpha}{dt} = Ae^{-E/(RT)}f(\alpha) \tag{2}$$

where α is the conversion rate of oxidative decomposition reaction of sulfide concentrate, $\alpha = (m_0 - m_t)/(m_0 - m_\infty)$, m_0, m_∞, and m_t [g] represent initial mass, final mass, and current mass of the system, respectively, t [s] – the reaction time, T [K] – the reaction thermodynamic temperature, A [s^{-1}] – the pre-exponential factor of the reaction, E [kJmol^{-1}] – the activation energy of the reaction, R [8.314 Jmol^{-1}K^{-1}] – the molar gas constant the universal gas constant, and $f(\alpha)$ is the conversion function reflecting the mechanism of decomposition reaction of sulfide concentrate, a simplified form of $f(\alpha) = (1 - \alpha)^n$ is often used, where n – the reaction order, usually takes values between 0 and 2.

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The eq.(2) is processed by the Coats-Redfern method [16], and the heating rate $\beta = dT/dt$ is substituted into the eq. (2), thus the following eq. (3) is obtained:

$$\frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} e^{-E/(RT)} dT \tag{3}$$

Equation (3) is expressed as an nth order exponential mechanism function expression:

$$\frac{d\alpha}{(1-\alpha)^n} = \frac{A}{\beta} e^{-E/RT} dT \tag{4}$$

For both sides of eq. (4), move the item, integrate it and take the logarithm:

$$\ln\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \ln\left[\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT}, \quad \text{when} \quad n \neq 1 \tag{5}$$

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln\left[\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT}, \quad \text{when} \quad n = 1 \tag{6}$$

The equations set of eqs. (5) and (6) are known as the Coats-Redfern equations. It is necessary to calculate the value of n since the number of reaction stages n for different reactions is different. When $n \neq 1$, $\ln[1 - (1 - \alpha)^{1-n}/T^2(1 - n)]$ is plotted against $1/T$, when $n = 1$, $\ln[-\ln(1 - \alpha)/T^2]$ is plotted against $1/T$, and the experimental data is fitted by least squares method, then a straight line can be obtained with a slope of $-E/R$ and the best value for the linear correlation is chosen as the final reaction order. The experimental results show that the temperature range calculated by high temperature oxidation kinetic parameters is 670~770 K, 720~820 K, 670~770 K, 700~800 K, respectively. Coats-Redfern integration method was used to acquire the best correlation coefficient and corresponding reaction order under different heating rates. The results are shown in tab. 3.

Table 3. The best correlation coefficient and corresponding reaction order of ore samples at different heating rates

Heating rate [°Cmin⁻¹]	1		2		3		4	
	R^2	n	R^2	n	R^2	n	R^2	n
5	0.9554	0	0.9468	2	0.9309	0	0.9217	2
10	0.9417	0	0.9725	0	0.8892	0	0.9410	2
15	0.8926	2	0.9695	1.5	0.8716	0	0.9291	2

By plotting $\ln[1 - (1 - \alpha)^{1-n}/T^2(1 - n)]$ to $1/T$, the fitting straight lines of the corresponding temperature intervals at three heating rates are obtained, as shown in fig. 6. The pre-exponential factor A and the activation energy E of the reaction were calculated from the corresponding intercept and slope, respectively.

The specific results are shown in tab. 4. Since the ore samples were taken from four different positions of the same mining area, the thermal analysis kinetic parameters showed similarity in general. Due to the fact that Samples 2 and 3 were collected from more adjacent positions, their thermal analysis kinetic parameters were more similar. It can also be seen from tab. 4 that the thermal analysis kinetic parameters of same sulfide concentrates are different under three kinds of temperature rising rates (5, 10, and 15 °C per minute), and the thermal analysis kinetic parameters of four ore samples at the same heating rate are also different. Furthermore, from the table it is found that the different heating rates will affect the activation energy of the sulfide concentrate sample. The activation energy at a heating rate of

5 °C per minute is less than that at a heating rate of 10 and 15 °C per minute, indicating the sample at heating rate of 5 °C per minute is more prone to spontaneous combustion. Furthermore, the values of the activation energy and ignition temperature for each ore sample have been consistent with its sulfur contents. The more sulfur contents of the ore sample, the lower the activation energy and the ignition point temperature. Thus, the thermal behavior of sulfide concentrate and the activation energy index was reasonable for determining its spontaneous combustion tendency.
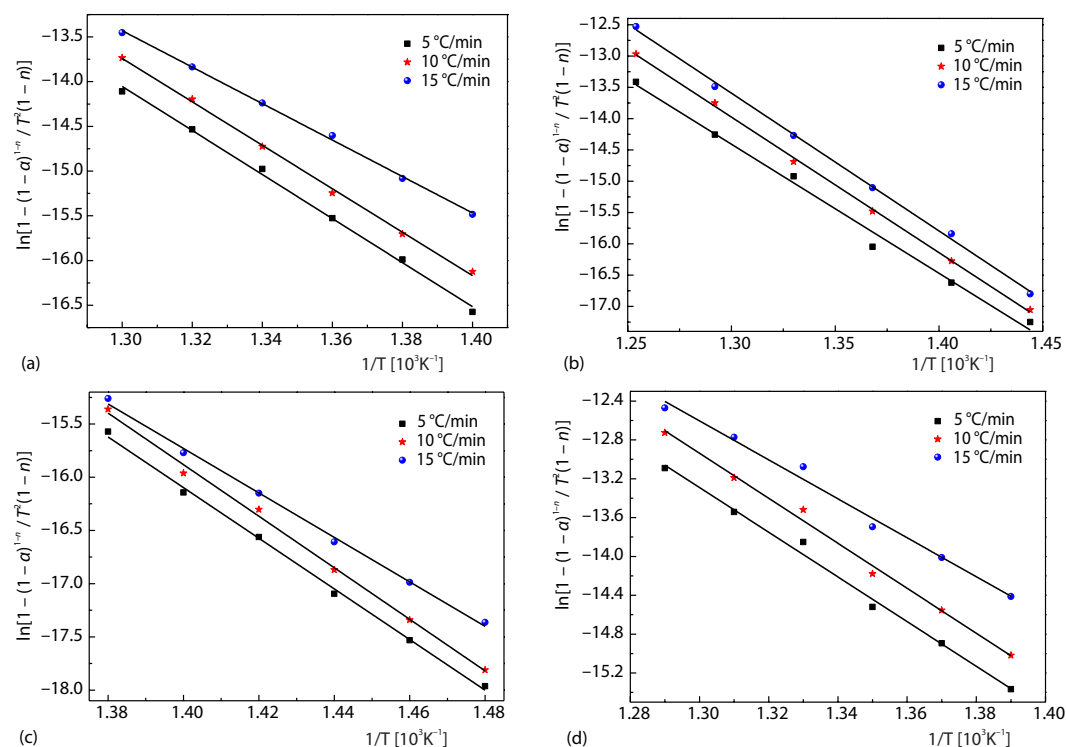

Figure 6. Fitting curves of 1/T of ore samples ln[1 − (1 − α)$^{1−n}$/T^2(1 − n)] at three heating rates; (a) Sample 1, (b) Sample 2, (c) Sample 3, and (d) Sample 4

Table 4. Thermal analysis kinetic parameters of sulfide concentrate

Sample	The pre-exponential factor [s^{-1}]			The activation energy [kJmol^{-1}]			
	5	10	15	5	10	15	Average
1	$1.869 \cdot 10^{13}$	$3.713 \cdot 10^{13}$	$4.336 \cdot 10^{13}$	217.8	234.9	199.4	217.4
2	$1.738 \cdot 10^{13}$	$3.816 \cdot 10^{13}$	$4.933 \cdot 10^{13}$	221.3	235.9	203.6	220.3
3	$1.721 \cdot 10^{13}$	$3.802 \cdot 10^{13}$	$4.352 \cdot 10^{13}$	234.7	255.6	252.9	247.7
4	$1.862 \cdot 10^{13}$	$3.718 \cdot 10^{13}$	$4.347 \cdot 10^{13}$	234.2	254.9	199.7	229.6

Conclusions

Four kinds of sulfide concentrates samples from different positions in the same mining area were analyzed by simultaneous thermal analysis from 20-1000 °C, and TG-DTG-DSC curves for each ore sample were obtained at three heating rates of 5, 10, and 15 °C per minute.

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The heating rate is an important factor affecting the thermal behavior of sulfide concentrates. The peak temperature corresponding to the maximum mass loss rate of the ore sample at different heating rates is different.

The activation energies for four samples are calculated, from 199.4-217.8 kJmol^{-1} for sample 1 to 199.7-254.9 kJmol^{-1} for Sample 4 by the Coats-Redfern method. These results show sulfide concentrates at heating rate of 5 °C per minute is more prone to spontaneous combustion, and their thermal analysis kinetic parameters take on similarity in general which are consistent with the factual situations. So it is reasonable to use the activation energy index to determine the spontaneous combustion tendency of sulfide concentrates.

Acknowledgment

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