

Controlled-release inhibitor for preventing the spontaneous combustion of coal

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Received: 10 December 2015 / Accepted: 4 February 2016 / Published online: 19 February 2016
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Abstract The existing coal self-heating inhibitors usually have the shortcomings of short effective time and low inhibition effect. This study proposed a kind of controlled-release inhibitor and analyzed their differences in inhibition effects compared with existing inhibitors on coal self-heating. The controlled-release inhibitor is made from the synthesis of halogen inhibitors, catechin, copolymer, solvent and surfactant. The controlled-release inhibitor will not break down quickly until coal temperature reaches a value about 70 °C and can continually inhibit the process of coal self-heating after a longer time. The crossing point temperature, propensity to spontaneous combustion and CO generation of coal samples processed by different inhibition methods were tested separately based on an oxidation dynamic method. The results show that the controlled-release inhibitor can inhibit the coal self-heating more effectively for a longer time than existing halogen inhibitors. This study provides a new method for more efficient prevention of coal spontaneous combustion.

Keywords Coal fire · Propensity of coal to spontaneous combustion · Controlled release · Inhibitor

1 Introduction

Spontaneous combustion of coal is an undesirable phenomenon in coal mining and storage. Almost all the coal mining countries are disturbed by this hazard. It is the most important reason for mine fires that usually result in serious personal injury, huge economic loss and

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environmental pollutions (Deng et al. 2015; Ham 2005; Rosa; Stracher and Taylor 2004; Zeng et al. 2015). At present, many different techniques have been proposed to prevent the spontaneous combustion of coal. Among these techniques, the technique based on inhibitors is one of the prevention techniques used frequently in coal mines. The existing inhibitors can be divided into physical inhibitors and chemical inhibitors. The physical inhibitors are usually made from inorganic salts, which inhibit coal spontaneous combustion by decreasing ambient temperature and forming water film covering coal particles. The chemical inhibitors can react with active groups on coal surface and break the reaction chains existing in the process of coal oxidation, which inhibit the development of coal spontaneous combustion.

The physical inhibitors currently used in coal mines include halogen salts (such as NaCl, MgCl₂ and CaCl₂), ammonium salts, Ca(OH)₂ and Na₂CO₃ (Beamish et al. 2012; Rath 2012; Slovák and Taraba 2012; Smith et al. 1988; Shan et al. 2008; Wang 2008; Yang 1996; Yu et al. 2010a, 2010b; Zheng 2010). Among these physical inhibitors, the halogen salts have been widely used in coal mines because of its moisture effect, harmlessness, economy and environmental protection. However, they are hard to be sprayed evenly on coal surface and are corrosive for mining machines underground. The techniques based on ammonium salts, Ca(OH)₂ or Na₂CO₃ are also have some shortcomings, such as uneven spray on coal surface, harmful gases produced in applications or corrosivity on mining machines. The existing physical inhibitors mainly play inhibition roles through the ways of isolating coal from oxygen and maintaining humid surroundings (Wang 2011; Wang et al. 2012b). Their inhibition effects depend on physical interaction, while few or none chemical change happen during the inhibiting process (Wang et al. 2012b, 2013). As a result, the physical inhibitors cannot eliminate the risk of coal spontaneous combustion. Especially, their inhibition effects cannot maintain for a long time due to the air flow and moisture, which indicates that they cannot continually play efficient prevention roles on coal spontaneous combustion.

Considering the shortcomings of existing physical inhibitors, some investigators tried to improve their inhibition performance through different ways. Yu et al. tried to improve the inhibition efficiency of halogen inhibitor by adding antioxidant (Yu et al. 2010a, b; Yu et al. 2010a, b). Beamish researched the influence of antioxidant on coal self-heating rate and confirmed their inhibition effects (Beamish et al. 2012, 2013). Wang et al. (2013) added corrosion inhibitor and wetting agents to halogen inhibitors in order to improve the inhibition effects and decrease the corrosion influence on machines. Deng et al. (2012) proposed to nebulize and spray the inhibitor solutions for extinguishing mine fire (Chen et al. 2011). Lu (2008) suggested delivering and spraying inhibitors to fire zones by the carrier of foam (Shi et al. 2010). Wang et al. (2012a) investigated the inhibition effects of ionic liquids on coal self-heating and tried to propose a prevention method based on ionic liquids, which is expensive and hard to apply (Zhang et al. 2013a, 2013b). Dou and Wang et al. (2014) analyzed the inhibition effects of some chemical compounds on the reaction of active groups (Dou et al. 2014).

In general, though the previous investigations have improved the inhibition effects in different degrees, the existing inhibition techniques still cannot meet the requirements of efficient prevention of coal spontaneous combustion. The applications of existing inhibitors are usually influenced by humid underground environment. The inhibitors sprayed on coal surface will dissolve in water or volatilize into air. As a result, the inhibition effects will decrease or even disappear gradually, which results in the low inhibition efficiency and short inhibition lifetime. Therefore, people have no ways but to spray inhibitors repeatedly, especially in coal mines facing serious coal spontaneous combustion. This approach costs

much labor and expense, which is unrealistic in the prevention of coal spontaneous combustion. Consequently, this study proposed a kind of controlled-release inhibitor and analyzed the differences in inhibition effects compared with other inhibitors on coal self-heating. The controlled-release inhibitor can continually inhibit the process of coal self-heating after a longer time and will be easy to use through the grouting pipeline systems. This study provides a new method for more efficient prevention of coal spontaneous combustion.

2 Preparation of the controlled-release inhibitor

The controlled-release inhibitor was proposed based on the available halogen inhibitors currently used in coal mines, such as NaCl, CaCl₂ and MgCl₂. For improving the inhibition performance, we also added catechin in the production of controlled-release inhibitor. During the production process, the key process is the surface treatment, which makes the traditional halogen inhibitors to have a controlled-release property. The production procedures of controlled-release inhibitor are listed as follows.

1. Mix poly (ethylene glycol) (PEG) 200, acrylonitrile–butadiene–styrene copolymer (ABS), alkyl polyglucoside (APG). The mass ratio for PEG 200, ABS and APG was 84:15:1.
2. Put the mixed solution into a three-necked flask. Then stir the mixed solution with a blender at room temperature for 30 min.
3. Add halogen inhibitors and (+)-catechin into the mixed homogeneous emulsion. The mass ratio for halogen inhibitors, (+)-catechin and emulsion was 1:1:2. Moreover, stir the mixture at 40 °C by a blender for 3 h.
4. Put the mixture containing halogen inhibitor and (+)-catechin into a programmed temperature controlling vessel, and run it in a constant 70 °C mode. Then the controlled-release inhibitors will be obtained after drying the mixture for 8 h at this temperature.

3 Experimental

3.1 Coal sample

A gas coal sample collected from the Pansan Coal Mine in China was processed to remove the surface layers, and the core material was then crushed in an oxygen-free glove box. The coal particles ranging in 0.18–0.38 mm in size were sieved and used as the experimental coal samples. The coal particles were kept under an inert atmosphere. Its proximate and ultimate analysis is listed in Table 1.

Table 1 Proximate and ultimate analyses for the coal sample studied

Coal sample	Proximate analysis, wt% (air-dry basis)				$Q_{\text{net, ad}}$, MJ/kg	Ultimate analysis, wt% (air-dry basis)				
	Moisture	Ash	VM	FC		C	H	N	S _t	O
Pansan gas coal	1.62	24.83	27.42	46.13	29.06	70.33	3.26	1.51	0.25	11.36

The coal particles were mixed with the controlled-release inhibitor powder and 20 % MgCl_2 solution separately. The controlled-release inhibitor powder was added into coal particles at a ratio of 5 wt%. Then the coal samples mixed with different types of inhibitors were stored for 1 or 7 days, which were used in later testing. Then the initial and processed coal samples were kept under a vacuum condition. Considering the effects of storage on coal self-heating (Beamish et al. 2000), the coal samples should be tested no longer than 1 week.

3.2 Testing of crossing point temperature (CPT) and the propensity of coal to spontaneous combustion

Crossing point temperature (CPT) and propensity of coal to spontaneous combustion are two important parameters for evaluating the risk of coal self-heating. As known, spontaneous combustion of coal is resulted from the accumulation of heat released from coal oxidation. If the surrounding is appropriate for coal oxidation and heat accumulation, the rising rate of coal temperature will become greater step by step. This means that coal temperature will exceed surrounding temperature at last if maintaining coal samples in an appropriate surrounding. During coal self-heating process, the temperature at the point when coal temperature begins to exceed surrounding temperature is the so-called CPT, as shown in Fig. 1.

We used the method based on oxidation kinetics, which is a Chinese industry standard for production safety (AQ/T 1068-2008), to test the CPT and the propensity of coal to spontaneous combustion. The method suggests that there are two different stages in the process of spontaneous combustion of coal, i.e., the slow oxidation stage at low temperatures and the accelerated oxidation stage at high temperatures (Wang et al. 2009; Zhong et al. 2009). Two parameters were selected as the fundamental parameters for the two stages separately. The oxygen concentration (C_{O_2}) at the outlet of coal oxidation vessel when the coal temperature is 70 °C was selected as the fundamental parameter of slow oxidation stage. And the CPT was selected as the fundamental parameter of accelerated

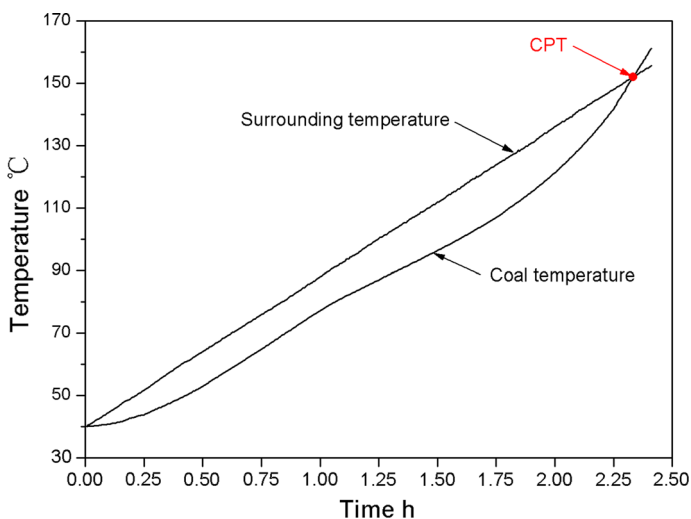


Fig. 1 Crossing point temperature (CPT) during coal spontaneous combustion

oxidation stage. The testing facility and testing procedures of C_{O_2} and CPT were described clearly in the previous papers (Qi et al. 2011; Wang et al. 2009; Zhong et al. 2009). Based on the two fundamental parameters selected, a comprehensive evaluating index was determined using the following formulas.

$$I_{C_{O_2}} = \frac{C_{O_2} - C_{O_2,\min}}{C_{O_2,\min}} \times 100 \tag{1}$$

$$I_{T_{\text{cpt}}} = \frac{T_{\text{cpt}} - T_{\text{cpt},\min}}{T_{\text{cpt},\min}} \times 100 \tag{2}$$

$$I = \phi \left(\varphi_{C_{O_2}} I_{C_{O_2}} + \varphi_{T_{\text{cpt}}} I_{T_{\text{cpt}}} \right) - I_0 \tag{3}$$

where $I_{C_{O_2}}$ is the evaluating index for the slow oxidation stage at low temperatures, dimensionless; $I_{T_{\text{cpt}}}$ is the evaluating index for the accelerated oxidation stage, dimensionless; I is the comprehensive evaluating index, dimensionless; I_0 is the amendment index which is valued at 300, dimensionless; C_{O_2} is the oxygen concentration in the outlet of coal reaction vessel when the coal temperature is 70 °C, v/v %; $C_{O_2,\min}$ is the calculation parameter of oxygen concentration whose value is 15.5 %; T_{cpt} is the CPT, °C; $T_{\text{cpt},\min}$ is the calculation parameter of CPT whose value is 140 °C; $\varphi_{C_{O_2}}$ is the percentage of slow oxidation stage in the whole process of coal spontaneous combustion at low temperatures whose value is 0.6; $\varphi_{T_{\text{cpt}}}$ is the percentage of accelerated oxidation stage whose value is 0.4; ϕ is the amplifying parameter which is valued at 40, dimensionless.

Then the I obtained from calculation is used to evaluate the propensity of coal to spontaneous combustion. The propensity of coal is classified into three kinds, as given in Table 2.

3.3 Testing of CO generation during coal self-heating

The testing facility is made up of gas preheating tube, coal reaction vessel, programmed temperature enclosure, gas chromatography and temperature logger. It is shown in Fig. 2. The gas preheating tube is a 50-m-long copper tube, which is used to preheat the gas flowing into the coal reaction vessel. The programmed temperature enclosure can provide a reaction surrounding with an accurate and uniform temperature. The gas chromatography is used to analyze the gas sample collected from the outlet of coal reaction vessel. The temperature logger can test the temperatures of coal sample and surrounding online.

Before the testing, we checked and debugged gas tightness of the testing facility and adjusted the gas chromatography. Then we put 50 g (± 0.01 g) of coal sample in the coal reaction vessel and laid a thin bed of asbestos on top of the coal sample for preventing the gas tube from becoming jammed. Firstly, the programmed temperature enclosure was set to run at a constant temperature of 25 °C. We let pure nitrogen, with a nitrogen concentration of 99.999 %, flow into the coal reaction vessel at a flow rate of 100 mL/min for

Table 2 Classification of the propensity of coal to spontaneous combustion

Classification	Propensity	Index I
I	Easy to spontaneous combustion	$I < 600$
II	Spontaneous combustion	$600 \leq I \leq 1200$
III	Difficult to spontaneous combustion	$I > 1200$

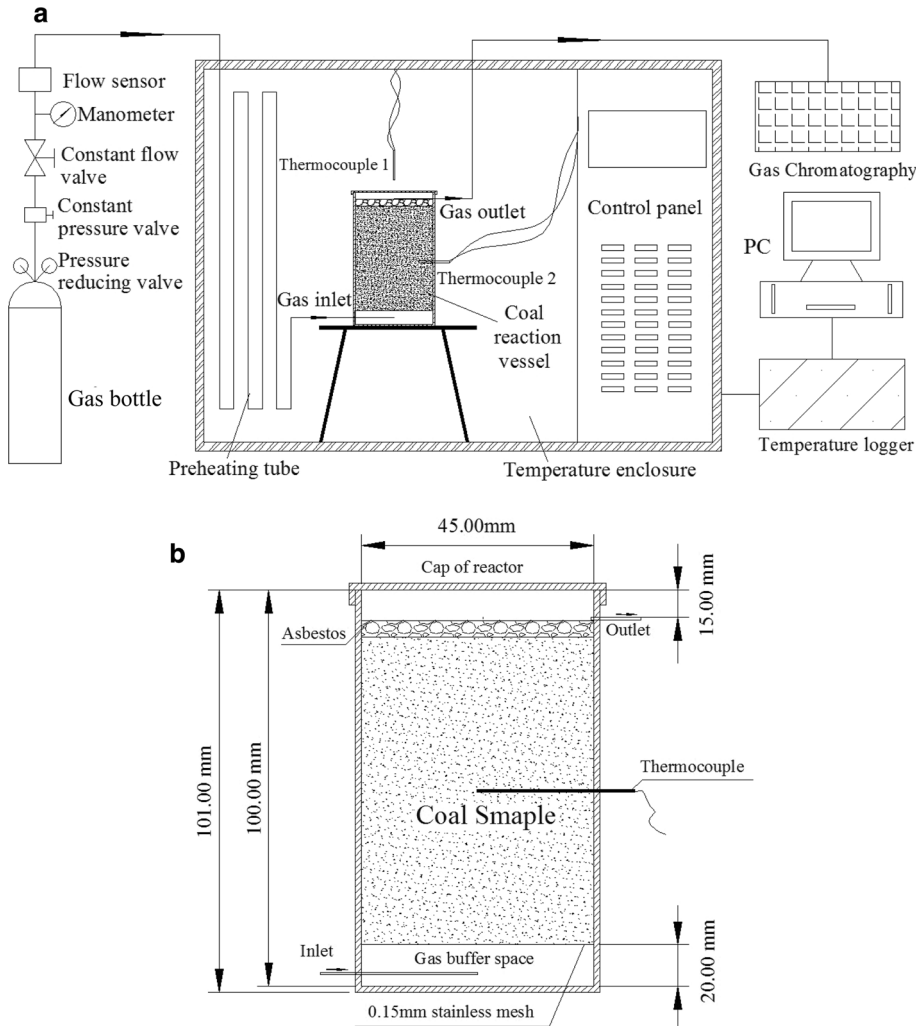


Fig. 2 Schematic diagram of the testing facility (**a** testing system; **b** coal reaction vessel)

more than 12 h. This step is necessary for removing some of the absorbed gases that can desorb easily at a low temperature and maintaining a uniform coal temperature. After this step, the gas concentration at the outlet of coal reaction vessel would be zero or constant at a very low level. Then the programmed temperature enclosure was set to run at a programmed rate of $0.8\text{ }^{\circ}\text{C}/\text{min}$ and the pure nitrogen flow at a flow rate of $100\text{ mL}/\text{min}$ was changed to dry air flow at a flow rate of $8\text{ mL}/\text{min}$. When the coal temperature reached $70\text{ }^{\circ}\text{C}$, the programmed temperature enclosure continually ran at the heating rate of $0.8\text{ }^{\circ}\text{C}/\text{min}$, but the flow rate of dry air was changed to $96\text{ mL}/\text{min}$. During the whole oxidation process, the gas samples at the outlet of coal reaction vessel were analyzed at every $10\text{ }^{\circ}\text{C}$ rise of coal temperature from 30 to $180\text{ }^{\circ}\text{C}$. The testing was ended when the coal temperature reached $180\text{ }^{\circ}\text{C}$. When the temperature was cooled down, the coal sample was removed and the coal reaction vessel was cleaned for the preparation of next testing.

4 Results and discussion

4.1 CPT Changes in coal samples processed by different inhibition methods

As known, CPT is a very important parameter during the developing process of coal spontaneous combustion. Based on the testing results, the CPTs of initial coal sample and coal samples processed by different types of inhibitors are given in Table 3.

The results indicate that the traditional halogen inhibitors and controlled-release inhibitors play different inhibition effects on the dynamic process of spontaneous combustion of coal. The CPT of the initial Pansan coal sample is 158 °C, which means it is a coal sample easy to spontaneous combustion. For the coal samples processed by 20 % MgCl₂ solution, if they were tested 1 day after blending with 20 % MgCl₂ solution, the CPT is 180 °C, but this value will be only 163.5 °C if tested 1 week later. The reason is that the inhibition effect of MgCl₂ solution decreases day by day and is not very strong after 1 week. It indicates that the inhibition effects of existing halogen inhibitors decrease with the time and will almost disappear if exposed for an enough time. In contrast, for the controlled-release inhibitor, their inhibition effect can maintain for a longer time and efficiently preventing the process of coal self-heating. For coal samples processed by controlled-release inhibitor, if they were tested 1 day after blending with controlled-release inhibitor, the CPT is 181 °C, and this value is still 179 °C if testing 1 week later.

4.2 Effects on the propensity of coal to spontaneous combustion

Based on the results, the comprehensive evaluating index (I) of the propensity to spontaneous combustion of initial coal sample and blended coal samples is given in Table 4. The results indicate that the inhibition effects of traditional halogen inhibitors and controlled-release inhibitors differ from each other very much after the same time.

The results indicate that the decreases in inhibition effects after the same time are different for MgCl₂ solution and controlled-release inhibitor. The comprehensive

Table 3 CPTs of initial and processed coal samples

Coal samples	CPT/°C
Initial coal sample	158
One day after mixing with 20 % MgCl ₂ solution	180
Seven days after mixing with 20 % MgCl ₂ solution	163.5
One day after mixing with controlled-release inhibitor	181
Seven days after mixing with controlled-release inhibitor	179

Table 4 Propensity of initial and processed coal samples

Coal samples	C _{O₂} /%	CPT/°C	I
Initial coal sample	19.8	158	571.5
One day after mixing with 20 % MgCl ₂ solution	20.7	180	962.3
Seven days after mixing with 20 % MgCl ₂ solution	19.85	163.5	642.1
One day after mixing with controlled-release inhibitor	20.76	181	983
Seven days after mixing with controlled-release inhibitor	20.68	179	947.8

evaluating index of the initial Pansan coal sample is 571.5. For coal samples processed by 20 % MgCl_2 solution, if they were tested 1 day after processed by 20 % MgCl_2 solution, the comprehensive evaluating index is 962.3, but it will be only 642.1 if tested 1 week later. The inhibition effects of MgCl_2 solution become weak after 1 week. This means the inhibition effect of MgCl_2 solution quickly decreases with the time and nearly disappears after an enough time. In contrast, for controlled-release inhibitor, the inhibition effect can maintain for a longer time and prevent the process of coal self-heating more efficiently. For the coal samples processed by controlled-release inhibitor, if they were tested 1 day after mixed with controlled-release inhibitor, the comprehensive evaluating index of is 983, and it is 947.8 if tested 1 week later. It indicates that the inhibition effect of controlled-release inhibitor can maintain for a longer time than the existing halogen inhibitors.

4.3 Effects on the generation of CO

During the process of coal self-heating, the reaction between coal and oxygen can generate CO, CO_2 , H_2O , CH_4 , C_2H_4 , C_2H_6 and C_3H_8 (Wang et al. 2003; Wang 2008). The CO_2 , H_2O and alkane gases obtained in the testing are usually influenced by gas desorption or water evaporation (Buzek and Lnenickova 2010; Crosdale et al. 2008; Küçük et al. 2003; McCutcheon and Wilson 2003; Qi et al. 2010; Wang et al. 2003; Yuan and Smith 2011). The C_2H_4 is usually generated at a temperature higher than 100 °C, which is not fit for determining the risk at low temperatures, too (Carras et al. 2009; Wang et al. 2003; Wang 2008). In contrast, the CO generation during coal self-heating will not be influenced by these factors (Carras et al. 2009; Qi et al. 2010; Wang et al. 2003; Wang 2008; Yuan and Smith 2011). Therefore, CO is usually used as a good gas index for early prediction of coal spontaneous combustion.

The experimental results show that the CO generation differs from each other for coal samples processed by different inhibition methods, as shown in Fig. 3. The initial Pansan coal sample begins to generate CO at 40 °C. The coal sample processed by 20 % MgCl_2 solution begins to generate CO at 80 °C and the quantity of generated gas decreases

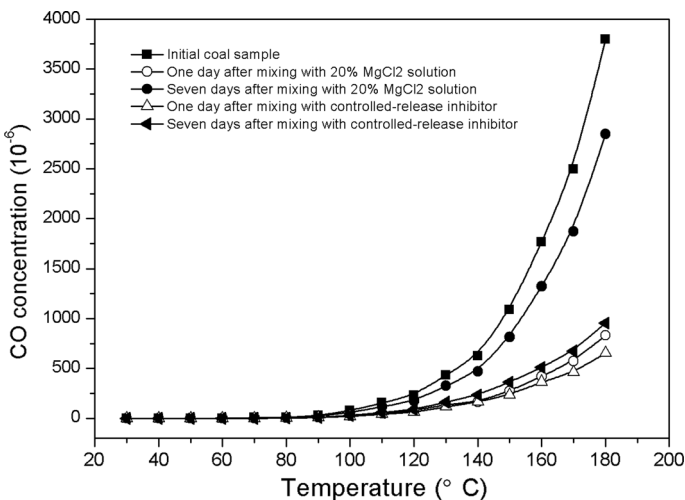


Fig. 3 CO generated during the oxidation process of different coal samples

obviously if tested 1 day after mixed with $MgCl_2$ solution. Comparatively, if tested 7 days after mixed with $MgCl_2$, it begins to generate CO at 50 °C and the quantity of generated gas is close to the initial coal sample. It means that the inhibition effects of $MgCl_2$ solution obviously decrease with the time and almost disappear after an enough time. In contrast, for the coal samples processed by controlled-release inhibitor, if tested 7 days later, the beginning temperature of CO generation is still the same as that tested after 1 day. And the generated quantity is very close for 1 or 7 days later. It indicates that the inhibition effect of controlled-release inhibitor can kept for a longer time than existing halogen inhibitors. This will be helpful for increasing the fire prevention efficiency.

5 Conclusions

The inhibition effects of existing halogen inhibitors decrease with the time and will almost disappear after some days. Considering the shortcomings of existing coal self-heating inhibitors, a kind of controlled-release inhibitor is proposed in this study. The controlled-release inhibitor is made from the synthesis of halogen inhibitors, catechin, copolymer, solvent and surfactant. During the production of controlled-release inhibitor, the key process is surface treatment, which will make the traditional halogen inhibitor have a controlled-release property. The controlled-release inhibitor will not break down quickly until coal temperature reaches a value about 70 °C and can continually inhibit the process of coal self-heating after a longer time. The crossing point temperature, propensity to spontaneous combustion and CO generation of initial and processed coal samples were tested separately based on an oxidation dynamic method. The changes in these parameters are small if we tested the coal samples processed by controlled-release inhibitor after 1 or 7 days, while the corresponding changes are large for coal samples processed by $MgCl_2$ solution. The results show that the inhibition effect of the controlled-release inhibitor can maintain for a longer time and inhibit the process of coal self-heating more effectively than existing halogen inhibitors.

Acknowledgments The authors deeply appreciate the financial support provided by “the Fundamental Research Funds for the Central Universities (No. 2014QNA06).”

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