



Full Length Article

A superabsorbent hydrogel–ascorbic acid composite inhibitor for the suppression of coal oxidation



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HIGHLIGHTS

- Pioneering study on prevention of coal oxidation by composite inhibitors.
- Composite inhibitor mechanism was proposed.
- The surface structure of composite inhibitor was analyzed.

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ABSTRACT

Composites of physical (superabsorbent hydrogel) and chemical (ascorbic acid) inhibitors were investigated to ascertain their ability to prevent the spontaneous combustion of coal. CO emissions from the oxidation of the composite inhibitor-treated coal were markedly lower than those for tests with raw coal. Less oxygen was also consumed during low-temperature oxidation, while the crossing point temperature of the inhibitor-treated coals was higher than that of raw coal. Together, these results demonstrate that the composite inhibitors can effectively mitigate coal oxidation. However, variation in the effectiveness was observed depending on the inhibitor-loading and the blend of the individual inhibitors in the composite. The composite inhibitor was shown to more effectively prevent oxidation than use of an individual inhibitor. The network structure of the composite inhibitor, revealed by SEM analysis, was suggested to explain the suppression of the coal oxidation.

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1. Introduction

The oxidation of coal has become an increasingly serious worry during the coal mining and storage [1–3]. Low-temperature oxidation of coal is the primary heat source that leads to uncontrolled spontaneous combustion, which can result in safety concerns and financial losses [4–8]. Adding inhibitors is one way of preventing spontaneous combustion of coal, especially so called ‘green inhibitors’ [9,10]. Physical inhibitors including suction salts (such as NaCl, MgCl₂ and CaCl₂), three-phase foam, and water glass, which can all absorb water and prevent oxygen from reaching active centers on the coal surfaces, are widely applied in coal mines for the prevention and extinguishing of fires [11–15]. However, these inhibitors exhibit low efficiencies and short active lifetimes. A number of chemical inhibitors also exist—including oxidizing agents, ionic liquids and several inorganic salts, such as Na₃PO₄ and Mg(Ac)₂—

which can react with active functional groups on the coal surface so as to decrease the formation of active groups or decrease their activity during reactions with oxygen [13,16,17]. These chemical inhibitors are highly efficient, although some are expensive or not applicable to all types of coal. Polymer-based inhibitors like polyethylene work by forming a thin membrane on the coal's surface, thereby preventing the reaction between coal and oxygen [18]. However, polymer inhibitors tend to be expensive and display low efficiencies. Therefore, the development of new inhibitors that are economic and highly efficient is desirable [19–33]. Moreover, it is useful to investigate the potential of composite inhibitors that may overcome the disadvantages of individual inhibitors discussed above.

This work investigates the combination of a physical and a chemical inhibitor. A superabsorbent polymer (SP), hydrogel, was chosen as the physical inhibitor on account of its ability to absorb and robustly retain large amounts of water (up to 10–1000 times its own mass) and its propensity to form an oxygen less permeable film on the coal's surface. Ascorbic acid (A) was chosen to

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chemically inhibit coal oxidation because it is a known major antioxidant additive used in the food industry and has been shown to decrease the oxidative stress in animals [34,35].

2. Material and methods

2.1. Experimental reagents and coal samples

The majority of reagents used (including, ascorbic acid at a purity of 99%, acrylic acid (AA), *N,N'*-methylenebisacrylamide (MBA), ammonium persulfate (APS) and acrylamide (AM)) were purchased from a local medical station. Polyacrylic acid resin, which was prepared in our laboratory, was used as the physical inhibitor for the experimental trials. Coal samples were collected from the Beizao (BZ) Colliery in Shandong Province, the Renlou (RL) Colliery in Anhui Province and the Tunbao (TB) Colliery in Xinjiang Province. During sample preparation, lumps of these coal samples were milled and sieved with fragments in the 0.25–0.80 mm range used for the experimental investigations. Table 1 summarizes the basic characteristics of these coals.

2.2. Preparation of the superabsorbent polymer

A kaolinite micropowder-based superabsorbent polymer was synthesized according to the following procedure. Kaolinite micropowder (2.13 g) was mixed with distilled water (7.10 g) in a 250 mL, three-neck flask, which was equipped with a stirrer, a reflux condenser and a nitrogen line. AA (7.21 g) was neutralized at 5 °C with a sodium hydroxide solution (2 M, 16 g) and was then added to the above solution. MBA (4 mg) and AM (3.55 g) were dissolved in distilled water (20 mL) and then added to the above mixed solution. After being purged with nitrogen for 30 min to remove dissolved oxygen, the mixed solution was gradually heated to 70 °C with APS (10 mg) then introduced into the flask to initiate the reaction. The solution was stirred vigorously and a nitrogen atmosphere was maintained throughout the 2 h polymerization. The product was then washed with ethanol and water and dried in an oven at 80 °C until no further change in mass was observed. Finally, the dried products were milled to pass through a 40–80 mesh.

2.3. Preparation of the composite inhibitor

The synthesized superabsorbent polymer was mixed with ascorbic acid in certain proportions depending on the experiment (SP:A = 1:3, 1:5, 1:7, 1:9). The mixture was then immersed in sufficient water and allowed to soak for 10 h. The swollen mixture was dried in an oven at 80 °C until no further change in mass was observed. The dried products were then milled to create a powder.

2.4. Experimental

Coal specimens were dried overnight in a vacuum oven at 40 °C before the moisture contents shown in Table 2 were measured by a coal proximate analyzer. The composite inhibitors were then blended with the raw coal particles in a beaker using mechanical stirring. Various inhibitor-loadings were investigated. As we all

Table 2

Moisture content of coal samples after being dried at 40 °C for 4 h in a vacuum oven.

Sample	Moisture content (%)
BZ coal	19.85
RL coal	1.05
TB coal	5.51

know, the moisture in the coal can affect coal oxidation, so in order to compare and study the effectiveness of different loads of composite inhibitors, the moisture content of the raw coal sample and the inhibitor treated coal sample has to be the same, therefore, the raw coal and composite inhibitor treated coal samples were then dried in a vacuum oven until their moisture content was similar to that of dried raw coal samples and stored in desiccators until measurements were taken. The cross point temperature (CPT), low temperature oxygen consumption and emissions of CO from raw and inhibitor-treated coal samples were then measured (see Sections 2.5 and 2.6). Meanwhile, changes in a sample's functional groups were determined by in situ analysis using a Nicolet 6700 Fourier transform infrared (FTIR) spectrometer (see Section 2.7). Finally, the micro-topography of the composite inhibitor's surface was measured by a scanning electron microscope (SEM) (Quanta 250, FEI, USA). Similar analytical procedures were previously reported [36–38,25,39–45,24,46–56].

2.5. Cross point temperature (CPT) and evolution of CO with increasing temperature

Widely used as a means of categorizing the propensity of coals for self-ignition, the point at which the temperature of a bed of coal exceeds the temperature of the heating bath surrounding it is referred to as the CPT and marks the onset of self-heating. CPT values were determined using an experimental apparatus of our own design consisting of a spontaneous combustion simulation system and a temperature data acquisition system. For each test, 50 g of sample was positioned in the apparatus reactor in such a way as to ensure that there was sufficient airflow within the sample. Dry air with an oxygen concentration of 20.96% was then passed through the sample for half an hour to stabilize the test specimen while the reactor was heated to a predetermined initial temperature. The temperature of the reactor was then set to increase by 0.8 °C/min while dry air was entered the reactor at 8 mL/min for 160 min. During this process, the temperatures of the reactor and the sample were recorded. Because the emission of CO and CO₂ from coal is temperature-dependent, monitoring the evolution of these gases is recognized as an effective means of tracking or predicting the onset of self-heating. Meanwhile therefore, exhaust gases from the reactor outlet were analyzed for CO content by gas chromatography at 10 °C intervals, beginning at 40 °C.

2.6. Oxygen consumption during low temperature coal oxidation

The same apparatus used to measure CO content was also employed to measure the consumption of oxygen by the samples between 40 and 70 °C. Here, the enclosure was set to a constant temperature of 40 °C while dry air entered the reactor at 50 mL/

Table 1
Properties of the coal samples.

Sample	Proximate analysis, %				Elemental analysis, %		Real density, g/cm ³
	M _{ad}	A _{ad}	V _{ad}	F _{cad}	H _{ad}	S _{td}	
BZ coal	22.98	9.77	29.93	37.32	4.25	0.61	1.47
RL coal	1.44	14.73	32.53	51.30	4.89	0.60	1.47
TB coal	8.14	13.48	31.49	46.90	3.06	0.70	1.44

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