1. INTRODUCTION

Mine fires are one of the main natural disasters in coal mining.\(^1\) Their occurrence will not only damage coal resources and equipment but also produce large volumes of smoke and toxic gases. Numerous mine fire accidents occur in China every year, resulting in significant economic losses and human casualties. The spontaneous combustion of coal is the main cause of mine fires. When coal comes into contact with sufficient oxygen, oxidation will be initiated and continuous oxidation leads to head accumulation.\(^2\) In order to prevent spontaneous combustion of coal, inhibitors such as inorganic salts (CaCl\(_2\), MgCl\(_2\)),\(^3-5\) free radical scavengers (2,2,6,6-tetramethyl-1-piperidinyloxy, procyanidine),\(^6\) and ionic liquids\(^7-9\) are used. These inhibitors played a positive role in preventing coal oxidation by altering the physical properties of the coal or eliminating free radicals existing in the coal oxidation process. However, physical inhibitors are easily run off with temperature increase, and free radical scavengers are mostly expensive; moreover, it is also hard to ensure that they react completely with the active free radical in the coal piles. Furthermore, some inorganic gels including water glass\(^10\) and composite slurry\(^11\) have been used in coal mines. These techniques are relatively simple and low-cost, but there are also some problems with these materials. The inorganic gel is easy to crack in the water loss phase and has a fast water loss rate. Particularly for the ammonium salt gel, it can release toxic gas when used. The strength of composite slurry is not high.\(^12\) Some chemical slurry expands rapidly, which leads to blockages in pipelines.\(^13\)

In order to resolve these problems, it is necessary to find new materials to replace the existing mine fire prevention and extinguishing materials. Hydrogels, as high-molecular-weight polymers, have characteristics capable of absorbing a large amount of water and retaining moisture. It is already widely applied to aspects of sanitary products, agriculture, medicine, and other applications.\(^14-16\)

Moreover, due to their characteristics, hydrogels are also proposed for use in fire prevention and extinguishing.\(^17,18\) For example, Tsai et al.\(^19\) prepared a thermosensitive hydrogel (P(NIPA-co-SA)). This gel is liquid at low temperatures and converts to solid at high temperatures. The experimental results show that the gel has a good inhibitory effect on anthracite. Hu et al.\(^20\) synthesized authigenic gas-foaming hydrogels by using chitosan (CS), acrylic acid (AA), and attapulgite (APT) and studied the effect of different synthetic conditions on the properties of the hydrogel. Huang et
with special properties. Chitosan is a natural polymer and can be grafted together to form grafting materials which will branch or functional side group via a chemical bond. By reaction of a macromolecular chain with an appropriate monomer, the hydrogel in this work. Graft refers to the coupling of two monomers that are degradable and thus do not pollute underground environments. Therefore, these two monomers were selected due to their excellent properties such as low cost, microbial degradability, compatibility, and nontoxicity. Some studies have shown that methacrylamide can enhance the water absorption capacity of hydrogels. The polymers synthesized by these two monomers are hydrophilic and thus do not pollute underground environments. Acrylic acid (AA) is a common monomer used to synthesize hydrogels. The hydroxyl groups in acrylic acid can form hydrogen bonds when exposed to water, causing them to have strong hydrophilicity. Methylacrylamide (MAA) is known as a biocompatible compound. Some studies have shown that methacrylamide can enhance the water absorption capacity of hydrogels. The polymers synthesized by these two monomers are degradable and thus do not pollute underground environments. Therefore, these two monomers were selected to prepare the hydrogel in this work. Graft refers to the reaction of a macromolecular chain with an appropriate branched or functional side group via a chemical bond. By means of polymerization, two kinds of polymers with different properties can be grafted together to form grafting materials with special properties. Chitosan is a natural polymer product obtained by deacetylation of chitin and widely exists in nature. It has been commonly used in medicine, food, and chemical industries due to its excellent properties such as low cost, microbial degradability, compatibility, and nontoxicity. Thus, in order to improve the adaptability of hydrogel in a solution with different pH values and make the hydrogel degradable, chitosan grafted onto P(AA-co-MAA) was synthesized and characterized in this paper. The thermal stability and the inhibitory performance of chitosan-grafted P(AA-co-MAA) were investigated by simultaneous thermogravimetry and differential scanning calorimetry (TG–DSC) measurement.

2. RESULTS AND DISCUSSION

2.1. Fourier Transform Infrared Spectroscopy of Samples. The FTIR spectra of P(AA-co-MAA) and CTS-g-P(AA-co-MAA) hydrogels are shown in Figure 1. In Figure 1b, a wide absorption band can be observed from 3500 to 3000 cm\(^{-1}\), which represents hydroxyl groups of acrylic acid and N—H groups of methacrylamide. The peak at 2946 cm\(^{-1}\) represents methylene groups, and the peak at 1715 cm\(^{-1}\) is due to C=O groups of acrylic acid. The peak at 1563 cm\(^{-1}\) represents —COOH groups. There are two obvious absorption peaks at 1450 and 1408 cm\(^{-1}\), which represent —COO\(^{−}\) groups. In Figure 1a, the position of each absorption peak is similar to that in Figure 1b, which represents a similar molecular structure for both gels. The peak at 1169 cm\(^{-1}\) represents the ether bond in chitosan, suggesting that the CTS-g-P(AA-co-MAA) hydrogel had been prepared.

2.2. Effect of pH Value on Equilibrium Water Absorbency. Equilibrium water absorbency represents the water absorption of hydrogels when the hydrogel is saturated. Due to the complexity of underground environments, the prepared hydrogel needs to work under both acidic and alkaline conditions. The different pH values of the solution will lead to the expansion ratio of the hydrogel being different, which will affect the leakage blocking and cooling properties of the hydrogel. Therefore, it is necessary to test the water absorption and swelling properties of the hydrogel in different pH solutions.

The equilibrium water absorbency of P(AA-co-MAA) and CTS-g-P(AA-co-MAA) hydrogels in different buffer solutions with different pH values is shown in Figure 2. The water absorbency of both hydrogels clearly decreased when the solution was acidic or alkaline. This was because when the pH value of the solution was low, most of the ionizable groups were not dissociated and there was no electrostatic repulsive force in the system. The hydrogel shrinks, and the moisture cannot enter the interior by osmotic pressure. With the pH value increasing to neutral, the dissociable groups are rapidly dissociated, the electrostatic repulsive force between ions makes the molecular chain expansion gel network larger, and the degree of swelling begins to increase. When the pH value continues to rise to alkaline conditions, the ionizable groups have been completely dissociated and the concentration of external ions is basically the same as internal ions. The
decrease in osmotic pressure leads to the decreased swelling properties of the hydrogel. In Figure 2, it is evident that the CTS-g-P(AA-co-MAA) hydrogel has improved swelling properties in acidic and neutral solutions as compared with the P(AA-co-MAA) hydrogel. This may be caused by the fact that the chitosan contains a large number of hydrophilic groups such as amino (−NH₂) and hydroxyl (−OH). Amino groups produce electronic repulsive forces on the hydrogel network, which enhance the swelling performance of the hydrogel. It is known that stronger water absorption can make the hydrogel adapt to more complex environments and improve the endothermic ability; thus, the CTS-g-P(AA-co-MAA) hydrogel has stronger adaptability to its surroundings than the P(AA-co-MAA) hydrogel.

2.3. Water Retention of P(AA-co-MAA) and CTS-g-P(AA-co-MAA) Hydrogels. Water retention determines whether the hydrogels can absorb heat for a long time through the evaporation of water. Figure 3 shows the water retention properties of P(AA-co-MAA) and CTS-g-P(AA-co-MAA) at high temperatures. The water loss rate for P(AA-co-MAA) is relatively fast. A 4 h period was required for the weight of the hydrogel to no longer change. It is clear that the grafting of chitosan prolonged this process. The water in CTS-g-P(AA-co-MAA) was completely lost after a 5 h period. It can be concluded from this study that the CTS-g-P(AA-co-MAA) had a good water retention capacity even at a high temperature. This ability will help the hydrogel reduce water loss and prolong the endothermic time in practical applications.

2.4. Surface Structure of Samples. The scanning electron microscopy diagrams of the two hydrogels are shown in Figure 4. The surface structures of the two gels are very different. Compared with P(AA-co-MAA), the surface of the CTS-g-P(AA-co-MAA) hydrogel in Figure 4b has more complex grains and folds. It can be inferred from the results of the water absorption test that this structure expands the surface area of the hydrogel and makes it easier for moisture to spread into the hydrogel, which strengthens the swelling properties of the hydrogel. The reason for this phenomenon may be that acrylic acid is an anionic monomer and chitosan is a cationic polymer. The molecular repulsive force in the graft product is small, which leads to the curling of the molecular chain. Better water absorption enables the hydrogel to absorb more heat by the evaporation of water.

2.5. Results from the Thermal Stability Analysis of Samples. The thermal stability of chitosan is poor, so it is necessary to study the effect of grafting on the thermal stability of the copolymer. Considering the practical application in mines, TG and differential thermogravimetry (DTG) were used to determine the thermal stability of the hydrogels. The TGA data obtained from the measurements for CJS coal samples containing different weight percentages of P(AA-co-MAA) and CTS-g-P(AA-co-MAA) under nitrogen are shown in Figures 5 and 6, respectively. As observed from the figures, the TG curves experience a slight decline at the initial stage from room temperature to approximately 100 °C, which is attributed to the loss of bound moisture at this stage. As the temperature is increased further, the mass of samples remaining decreases slowly until approximately 350 °C, which is then followed by a rapid decrease. For raw CJS coal, it is evident that there is a mass loss peak at ~450 °C, implying the thermal decomposition of coal. While for CJS coal samples containing hydrogels, apart from the mass loss peak at 450 °C, another mass loss peak appeared at approximately 350 °C, which can be ascribed to the destruction of the cross-linked structure and the loss of hydroxide and amine groups. As the hydrogel used increased, the mass loss rate increased; and when 10 wt % CTS-grafted hydrogel was mixed with the coal, the DTG curve showed a lower mass loss peak at 350 °C than P(AA-co-MAA) hydrogel-mixed coal, and heat stability of the sample was close to that of the original coal sample.
This indicates that the thermal stability of the sample containing 10% CTS-grafted hydrogel is better than that of the sample with more hydrogel content. Thus, 10 wt % CTS-grafted hydrogel could be chosen as optimal.

2.6. Inhibition of Coal Oxidation at an Early Stage by CTS-g-P(AA-co-MAA). In order to test the effect of the CTS-g-P(AA-co-MAA) hydrogel on coal–oxygen complex action, the thermal analysis experiment was carried out in air. Considering the thermal stability of the mixture, the ratio of coal to gel was chosen according to the results of the previous thermal analysis experiments. The CTS-g-P(AA-co-MAA) hydrogel was mixed with CJS coal at a weight ratio of 1:10, and the mixture was analyzed by thermal analysis in air. The results of the thermal analysis are shown in Figure 7. For raw CJS coal, a slight decline appeared at the initial stage due to dehydration. As the temperature increased further, the mass of raw CJS coal started to increase and reached a maximum at 315 °C. The reason for this phenomenon is that there was chemical adsorption between coal and oxygen in air. Followed by the increase, the coal mass started to drop rapidly. During this stage, the oxidation speed of the coal was accelerated, and as the temperature continued to rise, the coal reached the burning point. Different to raw coal samples, there was no obvious mass increase followed by dehydration for the CJS coal containing 10 wt % CTS-g-P(AA-co-MAA), and a plateau was found at temperatures between 130 and 340 °C. That indicated that the coal oxidation was inhibited by the 10 wt % CTS-g-P(AA-co-MAA) hydrogel. Figure 8 displays the DSC curves for raw CJS and 10 wt % CTS-g-P(AA-co-MAA) hydrogel-mixed CJS coal samples in air. For raw CJS coal, a small exothermic peak appeared at
temperatures between 283 and 409 °C followed by a significant exothermic peak. However, the DSC curve for the 10 wt % CTS-g-P(AA-co-MAA) hydrogel mixed with the CJS coal sample showed an apparent difference compared to raw CJS. There was only a significant exothermic peak observed at temperatures from 366 to 635 °C, indicating that the oxidation of CTS-g-P(AA-co-MAA) hydrogel-treated CJS coal was slower than that of raw coal. This was because the oxygen was isolated from the surface of the coal sample by hydrogel layers, thus reducing diffusion rates of oxygen and inhibiting the combustion. The above phenomena indicated that coal oxidation could be potentially prevented by the CTS-g-P(AA-co-MAA) hydrogel in low-temperature environments.

2.7. In Situ FTIR Analysis of Coal Oxidation. The infrared spectra of raw CJS and 10 wt % CTS-g-P(AA-co-MAA) hydrogel-mixed CJS coal samples in air at different temperatures are shown in Figure 9.

From the data collected from in situ FTIR, we found that the peaks ranging from 3200 to 3600 cm⁻¹ (specific to hydroxyl group) and 2800 to 3000 cm⁻¹ (specific to CH₂ and CH₃) of raw and 10 wt % CTS-g-P(AA-co-MAA)-treated CJS decreased with the increased temperature, and those for raw CJS decreased faster than CTS-g-P(AA-co-MAA)-treated coal, indicating that although hydroxyl and fatty acid groups participated in oxidation, CTS-g-P(AA-co-MAA) could slow the oxidation.

3. CONCLUSIONS
This study investigated P(AA-co-MAA) and CTS-g-P(AA-co-MAA) hydrogels, prepared by solution polymerization, and found a contrast between them. The results of swelling experiments and water retention tests indicated that the grafting of chitosan enhanced the absorbing ability of the hydrogels in buffer solutions with different pH values (5.8, 7.0, and 8.1) and prolonged the water retention time of the hydrogels. The thermal decomposition temperature of the two hydrogels was about 350 °C, much higher than the environmental temperature used to prevent and control spontaneous combustion of coal. The thermogravimetric experiment in nitrogen showed that the CJS coal containing 10 wt % CTS-g-P(AA-co-MAA) has the best thermal stability. The thermogravimetric and DSC curves revealed that the CTS-g-P(AA-co-MAA) hydrogel can prevent the initial oxidation of coal. The in situ FTIR spectra of coal oxidation indicated that CTS-g-P(AA-co-MAA) could slow down the oxidation of hydroxyl and fatty acid groups. Thus, the results of this work show that CTS-g-P(AA-co-MAA) is a good mine fire-resistant material and can be of useful application in the inhibition of coal oxidation.

4. MATERIALS AND METHODS
4.1. Materials. The majority of reagents used (including acrylic acid (AA, A.R. grade), methacrylamide (MAA, A.R. grade), chitosan (CTS), N,N,N′,N′-tetramethylethylenediamine (TEMED, A.R. grade), ammonium persulfate (APS, A.R. grade), N,N-methylene diacrylamide (MBA, A.R. grade), and sodium hydroxide (NaOH, A.R. grade)) were purchased from a local medical station. The coal samples were collected from the Chenjiashan (CJS) Colliery in Shaanxi province. In sample preparation, lumps of coal were milled and sieved with fragments in the 0.25–0.80 mm range then dried at 110 °C under nitrogen to a
constant mass and placed in sealed containers in preparation for the experimental investigations. Table 1 summarizes the basic characteristics of the coal.

| Table 1. Properties of the Coal Samples |
|----------------------------------------|
| sample                 | proximate analysis (air-dried basis) (%) |
|                        | M_d | A_d | V_d | FC_{ad} |
| CJS coal               | 5.82 | 8.58 | 28.74 | 56.86 |

4.2. Preparation of P(AA-co-MAA) and CTS-g-P(AA-co-MAA). Initially, 1.28 g of sodium hydroxide was dissolved in deionized (DI) water to prepare the sodium hydroxide solution, and 5.76 g of acrylic acid (AA) was slowly added to the solution. In order to prevent the self-polymerization of acrylic acid, the process was carried out in an ice water bath. Subsequently, the partially neutralized acrylic acid solution was transferred to a three-neck flask, oxygen was removed, and 0.228 g of APS was added to initiate the free radical. After 10 min, 1.70 g of MAA, 0.154 g of MBA, and 0.228 g of TEMED were added to the three-neck flask. The temperature was kept at 70 °C for 1 h to complete the polymerization reaction. The product was then washed with water to remove monomers that were not involved in the reaction. Finally, the product was dried at 90 °C for 12 h, crushed, and sieved through a 40-mesh screen.

The preparation method of CTS-g-P(AA-co-MAA) was similar to that of P(AA-co-MAA). CTS (0.1 g) was dissolved in 60 mL of 1 wt% glacial acetic acid. At the same time, 1.28 g of sodium hydroxide was dissolved in water to prepare the sodium hydroxide solution, and 5.76 g of acrylic acid was slowly added to the solution. In order to prevent the self-polymerization of acrylic acid, the process was carried out in an ice water bath. The CTS solution and the partially neutralized acrylic acid solution were then transferred to a three-neck flask with oxygen removed, and 0.228 g of APS was added to produce the free radical. The following steps were used as per the preparation of P(AA-co-MAA). Again, the product was dried at 90 °C for 12 h, crushed, and passed through the 40-mesh screen.

4.3. Characterization. The P(AA-co-MAA) and CTS-g-P(AA-co-MAA) hydrogels prepared were characterized by recording FTIR spectra on a Nicolet 6700 Fourier transform infrared spectrometer (Thermo Fisher Scientific Company, USA). The samples were dried prior to measurement. The surfaces of the samples were sprayed with gold, and the morphology of the samples were observed under a Quanta 250 scanning electron microscope (FEI Company, USA).

4.4. Swelling Experiment. Samples (0.5 g) were immersed in 100 mL of DI water for 12 h to ensure that the hydrogel fully absorbs water. The excess moisture was then filtered using an 80-mesh screen, and the hydrogel was set aside for 15 min. Subsequent to that, the swollen sample was weighed. The equilibrium water absorbency of the swollen sample was calculated using the following equation (eq 1)

\[ Q_{eq} = (w_e - w_d)/w_d \]

where \( w_e \) and \( w_d \) are the weights of the swollen sample and dry sample, respectively. \( Q_{eq} \) was calculated on the grams of water per gram of sample basis.

The effect of pH value on adsorption was also measured at the same time. Two 100 mL buffer solutions with pH values of 5.3 and 8.1 were prepared by the following steps: Solution 1 was prepared through weighing 7.099 g of NaH_{2}PO_{4} and adding distilled water to 1000 mL. Solution 2 was prepared by weighing 6.803 g of KH_{2}PO_{4} and adding distilled water to 1000 mL. Five milliliters of solution 1 and 95 mL of solution 2 were used to configure the acid buffer solution and the test results for a pH value of 5.3. Ninety-five milliliters of solution 1 and 5 mL of solution 2 were used to configure the alkaline buffer solution and the test results for a pH value of 8.1. Deionized water was selected as the neutral solution. The 0.5 g hydrogel samples were immersed in the two buffer solutions and set aside for 12 h. The excess moisture was then filtered using an 80-mesh screen, and the samples were set aside for 15 min. Subsequently, the samples were weighed, and the equilibrium water absorbency was calculated.

4.5. Water Retention of the Hydrogel. The hydrogel samples were immersed in ample DI water for 12 h to ensure that the hydrogel fully absorbs water. The saturated water-absorbing hydrogel samples were weighed and then dried at 90 °C until the mass no longer changed. The weight of the sample was recorded every hour. Water retention of the hydrogel was indicated using the following equation (eq 2)

\[ R = w_t/w_i \]

where \( w_t \) and \( w_i \) represent the weight of the hydrogel subjected to the drying process and the weight of the initial hydrogel, respectively. \( R \) represents the water retention rate.

4.6. Thermogravimetric Analysis. Two hydrogel samples (P(AA-co-MAA) and CTS-g-P(AA-co-MAA)) were mixed separately with CJS coal in different mass ratios (1:10, 1.5:10, and 2:10). Deionized water was added to the mixture, and the mixture was stirred to make the sample homogeneous. The mixture was then dried at 90 °C for 12 h. Ten milligrams of the mixture was weighed as a sample for thermal analysis. Thermogravimetric analysis was performed using a Q-600 synchronous thermal analyzer (TA Company, USA) at a heating rate of 15 °C/min from 20 to 800 °C. Two groups of experiments were carried out, one with nitrogen and the other in dried air. The gas flow in the experiments was 100 mL/min.

4.7. In Situ FTIR. In situ FTIR was employed to determine changes in functional groups in the coal samples with a KBr powder background used as a reference. CTS-g-P(AA-co-MAA) was mixed with CJS coal in the mass ratio of 1:10, and the raw and mixed samples were dried at 40 °C in a vacuum oven overnight under N_{2}. In situ infrared spectrometer was used to perform the test. Dry air flowed into the reaction chamber from its base and exited out to the top. A temperature controller was connected to the reaction chamber, and the chamber was heated to 140 °C. The scanning range was 4000–400 cm\(^{-1}\) while the resolution was 4 cm\(^{-1}\), and 64 scans were summed to produce each spectrum.

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Notes
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