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ghg GREENHOUSE GASES SCIENCE AND TECHNOLOGY

A novel double-doped polymer-modified cement-based sealant to enhance borehole seals and reduce methane emissions

Aitao Zhou, China University of Mining and Technology, Beijing, China, Henan Polytechnic University, Henan, China, and Pennsylvania State University, University Park, PA, USA
Jiawen Wang and Kai Wang, China University of Mining and Technology, Beijing, China
Derek Elsworth, Pennsylvania State University, University Park, PA, USA
Meng Zhang, China University of Mining and Technology, Beijing, China

Abstract: This study was conducted to develop a new type of sealing material: double-doped polymer-modified cement-based sealant (DPCS) and to verify its sealing performance. The aim is to improve the sealing effect of borehole, improve the gas extraction efficiency, and reduce methane emissions. Two kinds of polymers are doped in DPCS, namely the water-soluble polymer emulsion (A1) and the cationic polymer (A2). The fluidity, expansion, compactness, and mechanical properties of DPCS and the single-doped polymer-modified sealant were tested and compared. Additionally, the effects of the polymer–cement ratio and the dispersant dosage on the sealing performance of DPCS were analyzed. To evaluate these competing effects of multiple factors on sealing performance of DPCS comprehensively, an orthogonal test was designed with range and variance analyses. The results show that with a polymer–cement ratio of 6%, 0.10% expansion agent dosage, and 1.2% dispersant dosage, the DPCS performs best on mechanical and expansion properties. A field site test at the working face 15201 of the Baiyangling coal mine was conducted to verify DPCS's sealing performance. The test results indicate that the novel DPCS can improve the concentration of extracted gas, and is also a better choice than the common sealing materials used in the mine economically. © 2020 Society of Chemical Industry and John Wiley & Sons, Ltd.

Keywords: methane emissions; double-doped polymer-modified; sealing material; gas drainage

Introduction

Methane is a high-quality clean energy source associated with coal, and is 21 times more harmful than CO_2 as a greenhouse gas.[1] The majority of Chinese high-methane coal seams have poor gas permeability, poor drainage effect, low gas utilization rate, and serious gas leakage.[2] Coalbed methane (CBM) emissions have become one of the main contributors of China's greenhouse gas emissions, therefore methane emissions reduction will

Correspondence to: Kai Wang, College of Emergency Management and Safety Engineering, China University of Mining & Technology, Beijing 100083, China.
E-mail: kaiwang@cumtb.edu.cn

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be the most critical step for China to cope with global climate change and solve environmental problems for a long period of time. For the purpose of reducing methane emissions and ensuring the safe and efficient production in mines, gas drainage should be carried out when the methane content of coal bodies reaches a certain value. Methane drainage involves drilling into coal seams and gas accumulation areas, connecting the boreholes to special pipelines, and using gas extraction equipment to pump CBM from coal seams and goafs to the ground for utilization or for discharging CBM to the total return air flow. It is an effective way of reducing gas content in coal seams.[3] To improve gas drainage efficiency, people use sealing materials to seal boreholes and cracks around them. Thus, borehole sealing plays a key role in the effect of gas drainage, enabling effective segregation of combustible gas from an oxidant.[4,5] Poor borehole sealing will cause low gas drainage efficiency and inaccurate gas pressure measurement, which will affect the entire gas drainage process.[6–8]

To improve the sealing effect, sealing technology and materials have been developed rapidly. Common sealing materials used in coal mines are clays, cement slurry, three-phase foams and polyurethane.

As a sealing material, clay has advantages like availability, convenient transportation, low cost, simple operation,[9] but clay slurry has poor fluidity and high shrinkage after dehydration, which leads to dry cracking that causes easy air leakage. Aiming at these shortcomings of the clay sealing method, many researchers have improved this method. In flexible nylon tube grouting method, clay slurry is used to seal pores and to solve problem of water seepage in the mining area.[10] Hydration processes using clay–cement mixtures introduced with coal fly ash and slag has been shown to produce a low hydration-heat cement slurry system.[11] Mud–polyurethane mixtures have been shown to be a fast and effective sealing method.[12] It makes good use of the advantages such as good expansion, filling property, and fast solidification of polyurethane, and integrates the characteristics of compact texture and simple operation of yellow mud to quickly seal boreholes, effectively limiting the expansion and overflow of polyurethane. It also gives full play to the advantages of rapid filling and sealing properties of polyurethane to improve the sealing efficiency.

Cement slurry sealing material is cheaper and easy to operate. It has long sealing depth but poor expansion properties and poor mechanical properties such as compression resistance and crack resistance. The cement slurry injected into the borehole has difficulties in sealing the cracks around the borehole effectively. The sealing effect on rock fractures is limited. In view of the shortcomings of ordinary cement-based materials in sealing boreholes, scholars add additives such as polymers to the cement slurry form polymer mortar that can modify the ordinary cement to optimize its sealing performance. Alkali-treated jute fiber has been used as a reinforcing agent for polymer-modified cement slurries. Experiments have shown that the physical and mechanical properties of the modified cement slurry are significantly improved by using alkali-treated jute fiber; the water absorption rate and apparent porosity are significantly reduced, and the compressive and flexural strength are also strengthened.[13] However, the modified cement slurry exhibits poor fluidity due to the viscosity-enhancing effect of the additive polyacrylamide, which causes difficulty in grouting.[14] The presence of a polymer membrane in a polyvinyl alcohol or methyl cellulose modified cement mortar has also been verified.[15] These experiments proved the feasibility of improving the sealing effect by modifying cement-based materials, but the fluidity of cement-based modified materials still needs to be improved.

In order to seal air leakage efficiently, researchers developed solidified foam based on the foaming characteristics of the physical and mechanical system, the foam sealing material has better flow properties than the polymer-modified cement slurry. Using the cement slurry with a polymer–cement ratio of 0.75:1 and water–cement ratio of 2:1, a solidified foam with high properties and density of only 516 kg m^{-3} and compressive strength of up to 12.68 MPa was prepared.[16] After the determination of the material ratio and the approximate range of the solidified foam, the experimental data of a simulation test was fitted by a stepwise regression analysis to obtain the optimal mixture ratio of the foaming agent FP, foam stabilizer WP, solid filler RX to solid filler GX and defined as 1.5:2:3:3.[17] However, such three-phase foams have limitations relegating them as the only most suitable for sealing boreholes in coal seams and soft rock.

Polyurethane is a new type of sealing material with good sealing effect. Compared with the conventional sealing process, it increases the gas drainage concentration by about 20%.[18] Various exploratory studies include adding allyl double glycol carbonate

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esters to the polyurethane system to examine improvements in mechanical properties and in mixing polyblends with siloxane and some fiber reinforcing agents or with plasticizers.[19,20] However, as an organic material, polyurethane is unstable and toxic. Thus, it is not ideal to use only polyurethane for borehole sealing.

In general, the main shortcomings in the current state of the art/practice in borehole sealing methods are as follows:

(1) sealing materials such as clay and cement have poor cracking resistance and correspondingly reduced effectiveness as a sealing agent, while organic sealing materials such as polyurethane have poor stability, high cost, and high toxicity, posing a threat to the health of underground workers.
(2) Polymer-modified cement materials are rarely used as sealing materials during boreholes sealing. In addition, there are few data on the improvement of double-doped polymer cement slurries as most studies involve the addition of only a single polymer.
(3) The effect of single-doped polymer cement sealing materials in modification is limited. They cannot meet the grouting requirements during sealing process while ensuring its mechanical properties, and it is impossible to obtain sealing materials with good fluidity.[10–13,15–20]

The direct use of pure polymers as sealing materials does have characteristics such as poor stability, high cost, and high toxicity. To improve borehole-sealing effect, many researchers choose to use a small amount of polymer as an additive to modify cement-based materials, which is confirmed to have an excellent improvement in sealing performance. Therefore, the polymers, a water-soluble polymer emulsion A1 (polyacrylamide) and a cationic polymer A2 (epoxy resin), were also selected for our modification test of the cement. They have the characteristics of low toxicity, low cost, and a wide range of sources than other polyurethane additives. The cost and toxicity are within acceptable ranges, which meet the requirements of mine sealing materials.

In this study, researchers developed a novel double-doped polymer-modified cement-based sealant (DPCS) by adding A1 and A2, and improved the sealing process. In view of the inconsistency and the poor fluidity of single-doped polymer-modified cement slurry,[14,15] it is required to choose the right polymers' ratio to improve the sealing properties.[16] In this study, researchers designed an experiment to determine the polymers' ratio of the DPCS. Besides, considering the poor expansion and mechanical properties of clay and cement sealing materials, the properties of fluidity, expansion, mechanical behavior, and compactness, as well as the effect of the polymer–cement ratio on the properties of mechanical behavior and compactness of these composites, were tested. Then, the orthogonal test was designed to test the comprehensive influence of multiple levels and multiple factors (the water–cement ratio, the dispersant dosage, and the polymer–cement ratio) on the expansion and mechanical performance of DPCS, the optimal ratio combination was selected by the range and variance analysis. The sealing process was optimized by comparing the advantages and disadvantages of cement mortar sealing and polyurethane sealing. Finally, the sealing effect of DPCS, the sealing process, and the gas drainage effect were verified by the field sealing application experiments.

Materials and methods

The sealant is used to seal the gap between the borehole wall and the interior drainage pipe and fill the fractures generated in the coal body around boreholes. Therefore, any suitable sealant should not only ensure its normal solidification, but also exhibit high fluidity, expansion, strength, and durability. Experiments are conducted to define optimal ratio of mixture components in comparison to a single-doped mixture.

Prototype development

Raw materials and process workflow

According to the requirements of the borehole sealing principle and the material in the gas drainage process,[21] the polymer-modified cement-based sealing material should meet the following points:

(1) A good sealing material should have suitable bonding strength and fluidity to reduce the leakage of slurry and meet the grouting requirements.[22,23]
(2) The sealing material should have a certain expansion property to offset the shrinkage deformation of the cement material during the hardening process to facilitate better sealing of microcracks around the borehole[24].

(3) The sealing material should have good compactness[25].
(4) The raw materials for the development of sealing material should be widely available and at cheap price, which could meet the practicality of borehole sealing in coal mines.

According to the problems and the design principles of the sealing material, the raw materials of the sealant include polymers, water, dispersant, cement, expansion agent, and microcapsule wall material.

The polymers are A1 and A2. They have low toxicity, low cost, and a wide range of sources than other polyurethane additives. The cost and toxicity are within acceptable ranges.

At a suitable low concentration, the polyacrylamide emulsion can be regarded as a network structure with good flocculation properties, which can reduce the frictional resistance between liquids. When the concentration is high, it is easily gelatinous. Epoxy resin cures easily, having strong adhesion and low shrinkage. The cured epoxy resin system has excellent mechanical properties.

The water used is the Beijing tap water. The cement is number 425 ordinary Portland cement from Jingluo cement factory in Yutian county, Tangshan city. The number 425 ordinary Portland cement has the characteristics of high strength, large hydration heat, good frost resistance, small shrinkage, good abrasion resistance, good carbonization resistance, poor corrosion resistance, and high temperature resistance. Its compressive strength at 28 days age ≥ 42.5 MPa, flexural strength ≥ 6.5MPa. The dispersant B(NNO) is a common cement slurry dispersant making the polymer cement system more stable. There is no chloride salt in the test, thus the expansion agent C (aluminum powder) is selected to make the cement expand and to compensate for volume shrinkage. The methyl cellulose solution, a conventional mucus (adjustable concentration), is used as the microcapsule wall material. Liquid microcapsules can be obtained by uniformly stirring the expansion agent in the mucus.

These raw materials come from a wide range of sources and are cheap, it is very consistent with the selection principles of the development of polymer-modified cement-based sealing material in this research.

The process workflow of optimization test is as follows; first, the dispersant B is added to the cement slurry to increase the dispersion, next, methyl cellulose is added to the water at 50–60°C to prepare the microcapsule wall material, and then the expansion agent C is added for microencapsulation treatment. Finally, the liquid microcapsules, the high-dispersion slurry, and the polymers are mixed to produce the required cement sealing slurry.

Polymers' ratio

In view of the inconsistency and poor fluidity of single-doped polymer-modified cement slurry, the water-soluble polymer emulsion A1 and the cationic polymer A2 were selected to modify the cement-based sealing material, wherein the polymer emulsion A1 (according to its own water content) can improve the fluidity of the slurry and further improve the mechanical properties of the composite material on the basis of the polymer A2. However, as the amount of A1 increases, the properties of the material do not increase blindly, so it is also crucial to choose the right polymer ratio.

In order to determine the optimal ratio of the two kinds of polymers, the cement slurry with a polymer–cement ratio of 6% and water–cement ratio of 1:1 was first prepared (the mass ratio of A1 to A2 was 1:2, 1:1, 2:1 respectively, and they were marked as three groups), these different cement slurry mixtures were then poured into three separate containers (plastic cups) for maintenance under identical environmental conditions as shown in Fig. 2.

Figure 1 shows that both cup 2 and cup 3 exhibited a "water-bleeding" phenomenon, which will affect hardening and durability of cement. However, this phenomenon did not appear in cup 1. The reason is that polymer emulsion A1 itself contains water that can improve the fluidity while the polymer A2 will increase the viscosity. Since polymer emulsion A1 is dispersed uniformly in the cement slurry it forms tiny "liquid beads" filling the space of the cement hydrated product and the unhydrated cement particles, friction between the cement particles and the hydration product is reduced. Macroscopically, the flow properties of the slurry are improved. If the polymer ratio exceeds 1:2, as the addition dosage of A1 increases, the free water content in the entire cement slurry system increases gradually and leads to excessive water–cement ratio, which results in the "water-bleeding" phenomenon ultimately. If the polymer ratio is less than 1:2, the A2 will cause the cement slurry to be too viscous, so in a limited number of tests, we conclude that the mass

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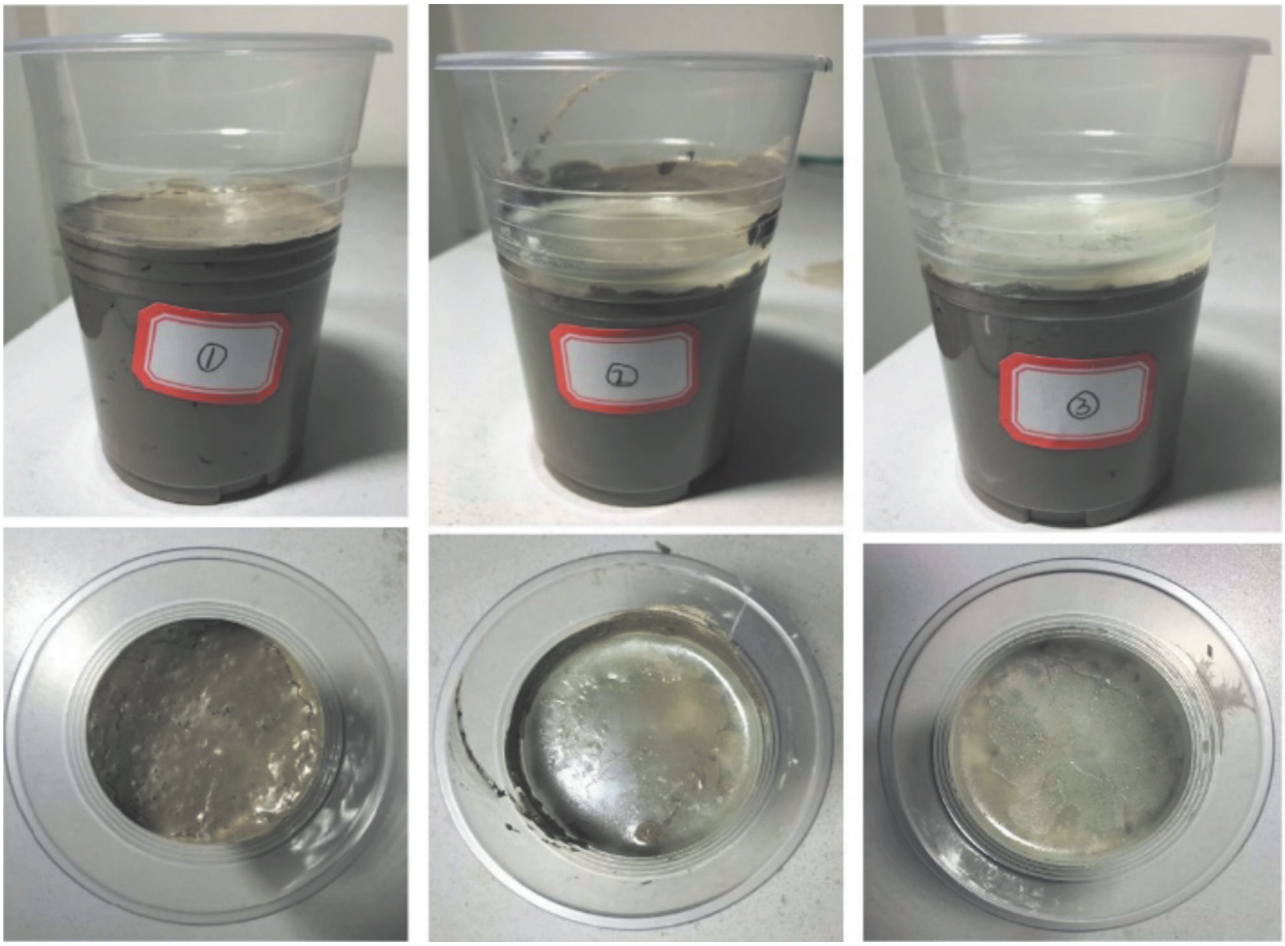

Figure 1. Hydration of the three mixes after one day.

ratio of water-soluble polymer emulsion A1 to the cationic polymer A2 should be 1:2.

Performance characteristics

Fluidity

According to the boreholes' sealing mechanism, the sealing performance is mainly measured by the amount of cement slurry leakage, and this leakage amount is determined by the fluid flow state in the cracks around the borehole. The calculation formula for the leakage amount Q of sealing cement slurry is deduced as follows[26,27]:

$$Q = \frac{\pi D (P_1 - P_2)}{12\mu L} h^3 \left(1 + 1.5\delta^2\right) \quad (1)$$

where Q is the amount of cement slurry leakage, $m^3\ s^{-1}$; D is the borehole diameter, m; P_1, P_2 are the pressures of sealing medium and the external, Pa; μ is the viscosity of sealing medium, $Pa \cdot s$; L is the length of the seal body; h is the average width of the cracks; δ is the eccentricity.

From the formula (1), it can be identified that the leakage amount Q of cement slurry decreases as the viscosity μ increases. However, in the actual sealing, ensuring that the slurry has an appropriate viscosity helps reduce the leakage amount of slurry and reduce the loss of the sealing material. Nevertheless, the increased viscosity of the slurry will affect the fluidity and make it difficult to meet the sealing requirements of cracks around the borehole.[27] Therefore, it is necessary to test the fluidity of the developed cement slurry. According to the traditional cement fluidity test method,[21] a simple device was designed to test the fluidity of cement slurry under the existing equipment conditions in the laboratory (Fig. 2).

The test method is to first block the lower end of the funnel, pour the developed cement mortar into the

Figure 2. Testing device to define slurry flowability.

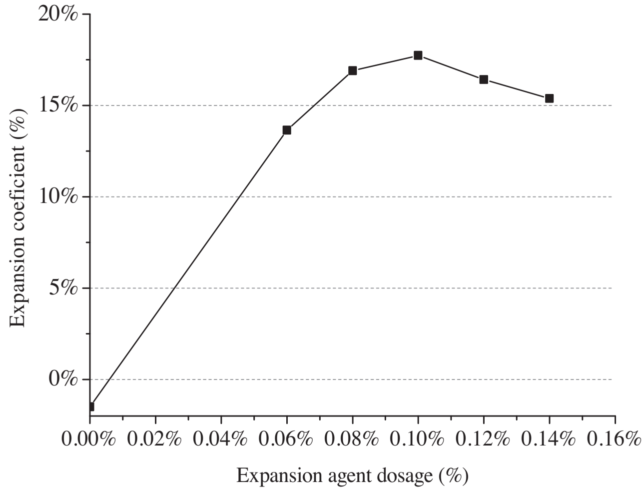

Figure 3. Expansion coefficient variation curve of the modified cement mortar with different dosages of expansion agent.

Table 1. Time required for modified cement slurry to flow out of the funnel.

Type	Number	Time(s)	Average time(s)
Single-doped	1	47.67	52.41
	2	51.08	
	3	58.47	
Double-doped	1	32.44	33.01
	2	28.57	
	3	38.02	

funnel, and ensure that the funnel is vertically down without position migration, and then use a glass rod to remove the plug at the lower end of the funnel and start timing at the same time, finally, stop timing until all the mortar in the funnel flows into the plastic bottle. The liquidity experiment of the cement slurry of the double-doped polymers A1 and A2 (A1:A2–1:2) and the single-doped polymer A2 was compared, each group was done three times, and the time taken by slurry to flow into the breaker completely was recorded in every test (Table 1).

From Table 1, we can see that the time taken to flow out from the funnel by the double-doped polymer-modified cement slurry is shorter by about 20 s that of the single-doped sample, indicating that the double-doped modified cement slurry flows faster, and the polymer emulsion A1 can improve the fluidity. Since polymer emulsion A1 is dispersed uniformly in the cement slurry and forms tiny "liquid beads" filling the space of the cement hydrated product and the unhydrated cement particles,[25] friction between the cement particles and the hydration product is reduced.[28] Macroscopically, the flow properties of the slurry are improved.

Expansion property

The hydration of the cement with water causes the hardened paste to shrink in volume for a long time, thereby forming a void space around the cement body. These developed pore spaces and mechanical shocks can lead to poor bonding between the cement and the casing, which in turn causes the leakage into the well.[29] The expandable cement can minimize these negative effects and improves the quality of the bond between the cement and the casing.[30]

The expansion property testing process is as follows; first, the developed modified cement slurry was poured into several 100 mL cylinders. Then, both the initial volume and the volume change of the slurry in the cylinders were recorded every 24 hours until the cement slurry solidified and there was no more volume expansion. The volume expansion coefficient of the cement slurry can be obtained by the ratio of the final volume to the initial volume, as shown in Fig. 3.

Mechanical behavior

In the actual gas drainage process, the sealing material in the borehole will be affected by the gas pressure and the wall pressure of the borehole. If the strength of the sealing material is too low, the borehole wall will collapse. Therefore, good compressive strength and

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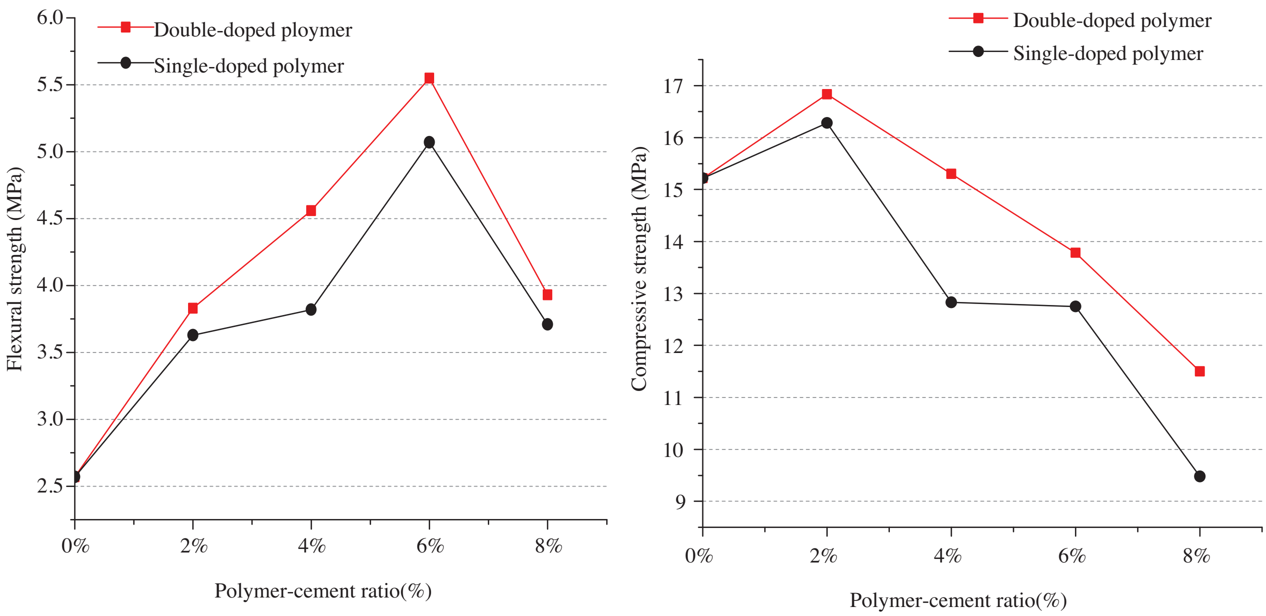

Figure 4. Flexural and compressive strength of cement specimens of different polymer–cement ratios.

flexural strength are the properties that must be present in the sealing material.[31] The polymer–cement ratio has a significant effect on the mechanical properties of the modified sealing material. Therefore, in this experiment, the mechanical properties such as the compressive strength and the flexural strength of the sealing materials were measured under different polymer–cement ratios, and the optimum polymer–cement ratio was obtained by analyzing the bend-press ratio of the test block.

In this experiment, the cement slurry was modified by double-doped water-soluble polymer emulsion A1 and cationic polymer A2 (A1:A2 = 1:2) with a water–cement ratio of 1.5:1 and a polymer–cement ratio of 0, 2, 4, 6 and 8%, respectively. The modified cement slurry was placed in triple molds of 40 × 40 × 160 mm. The flexural strength and the compressive strength of the cement specimens were tested after 7 days of curing under natural conditions. The results were compared with the cement slurry modified by single-doped cationic polymer A2 (Figs. 4 and 5).

Compactness

According to the development goal of the sealing material mentioned above, it can be found that the compactness of the material is crucial for the sealing of borehole, and a poor compactness could result in a bad sealing effect and gas leakage in the gas drainage

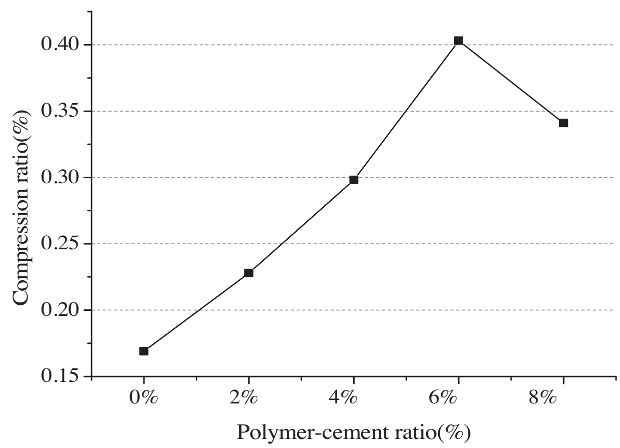

Figure 5. Bend–press ratio of cement specimens of different polymer–cement ratios.

process.[32] The material compactness is reflected by the porosity calculation formula as follows[33]:

$$n = \frac{\rho_t - \rho_v}{\rho_t} \tag{2}$$

where n is porosity; ρ_t is true density; and ρ_v is visual density.

The main test equipment includes a MDMDY-350 automatic density meter, a drying oven, liquid paraffin, and an electronic balance. The test method is as follows:

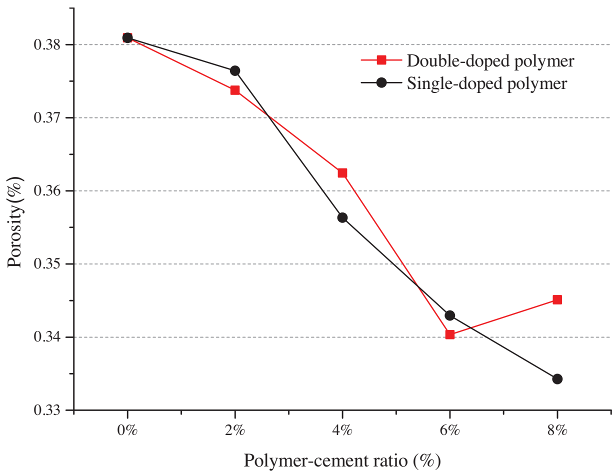

Figure 6. Porosity variation in cement specimens for different polymer–cement ratios.

(1) According to the ratio of the experiment, the cement mortar was disposed and cured at room temperature for 7 days.
(2) The formed cement test blocks were crushed into pieces. Then the samples with a particle size that meet the requirements of the density meter were selected by using a sieve.
(3) The selected samples were dried in a dry box, and weighed with an electronic balance.
(4) The true density and visual density of the weighed samples were measured by a densitometer.
(5) Calculating the porosity according to the formula (2).

The polymer will form a three-dimensional network structure in the hydration process of the cement and fill the pores between the hydration products, thus the polymer dosage can affect the quantity of pores inside the material, thereby affecting its compactness.[34] In this research, the experiments were carried out with a water–cement ratio of 1:1, and both the double-doped polymer cement and the single-doped polymer cement were prepared with polymer–cement ratios of 0, 2, 4, 6 and 8%, respectively. After 7 days of curing, the true density and the apparent density of the specimens were measured to calculate the porosity (Fig. 6).

Orthogonal tests to optimize mix sealing material

Orthogonal testing is a common test method in scientific research. It selects some uniform dispersions and comparable fluffy points from a comprehensive test, and conducts experimental research on them. These points are used to study the combined effects of different factors in different levels on the test results. The orthogonal test can find the best experimental ratio efficiently and conveniently without extensive and comprehensive experiments.[35,36] Therefore, by designing the orthogonal test, the influence of various factors on DPCS can be analyzed effectively and accurately, so as to obtain the best ratio of materials.

Orthogonal test design

To evaluate the sealing effect of the DPCS with multiple factors, the polymer–cement ratio, the expansion agent dosage, and the dispersant dosage, are selected to analyze the relations affecting the sealing performance at three levels. The water–cement ratio is retained at 1.0. Since there are three levels for each factor in this test, representative testing groups are selected by an L9 (3^4) orthogonal design table (Table 2). The expansion and mechanical properties in each group are tested to explore the comprehensive impact of the three factors on cement slurry.

Orthogonal test on the expansion property

The number 1–9 specimens of polymer cement mortar were prepared according to the designed orthogonal test, and the prepared specimens were placed in nine 100 mL measuring cylinders, the initial volume was recorded, as well as the final volume after standing for 48 hours, so the volume expansion coefficient of the material could be obtained by the ratio of the initial volume and the final one. The data were recorded and a range and variance analysis was performed on the expansion property (Tables 3 and 4). K_i is the sum of three tests of the level i in every column; k_i is the average value of K_i; and R is the range difference. An interrelationship is developed by using the corresponding k_i of every factor in Table 3, as shown in Fig. 7.

Orthogonal test on the mechanical properties

According to the ratio of the designed orthogonal test above, the number 1–9 specimens of polymer cement mortar were prepared, and the obtained polymer cement mortar was placed in the 40 × 40 × 160 mm

Table 2. Orthogonal design table.

Number	A Polymer-cement ratio	B Expansion agent dosage	C Dispersant dosage	Water-cement ratio
1	1(4%)	1(0.08%)	1(0.8%)	1.0
2	1(4%)	2(0.10%)	2(1.0%)	1.0
3	1(4%)	3(0.12%)	3(1.2%)	1.0
4	2(6%)	1(0.08%)	2(1.0%)	1.0
5	2(6%)	2(0.10%)	3(1.2%)	1.0
6	2(6%)	3(0.12%)	1(0.8%)	1.0
7	3(8%)	1(0.08%)	3(1.2%)	1.0
8	3(8%)	2(0.10%)	1(0.8%)	1.0
9	3(8%)	3(0.12%)	2(1.0%)	1.0

Table 3. Range analysis on the expansion coefficients and the bend-press ratios.

Test group	A	B	C	Initial volume (mL)	Final volume (mL)	Expansion coefficient	Bend–press ratio
1	1 (4%)	1 (0.08%)	1 (0.8%)	60	68	1.14	0.304
2	1 (4%)	2 (0.10%)	2 (1.0%)	66	77	1.17	0.312
3	1 (4%)	3 (0.12%)	3 (1.2%)	74	85	1.15	0.299
4	2 (6%)	1 (0.08%)	2 (1.0%)	70	87	1.24	0.401
5	2 (6%)	2 (0.10%)	3 (1.2%)	66	83	1.26	0.398
6	2 (6%)	3 (0.12%)	1 (0.8%)	73	89	1.22	0.433
7	3 (8%)	1 (0.08%)	3 (1.2%)	61	72	1.18	0.352
8	3 (8%)	2 (0.10%)	1 (0.8%)	68	82	1.21	0.367
9	3 (8%)	3 (0.12%)	2 (1.0%)	73	85	1.16	0.341
K_1	3.46/0.915	3.56/1.072	3.57/1.054				
K_2	3.72/1.232	3.64/1.097	3.57/1.059				
K_3	3.55/1.065	3.53/1.043	3.59/1.099				
k_1	1.153/0.305	1.187/0.3573	1.190/0.351				
k_2	1.240/0.4107	1.213/0.3657	1.190/0.353				
k_3	1.183/0.355	1.177/0.3477	1.197/0.366				
R	0.087/0.1057	0.036/0.018	0.007/0.015				

Note: The value before “/” is the expansion coefficient, and the value after is the bend–press ratio

Table 4. Variance analysis on the expansion coefficients and the bend–press ratios.

Variance source	Sum of squares (S_ε)	Degree of freedom (f)	Variance value	F value	Threshold value
A	0.0116/0.01678	2	0.0058/0.008388	116/106.88	$F_{0.01}(2,2) = 99$
B	0.0022/0.0005	2	0.0011/0.000025	22/3.18	$F_{0.05}(2,2) = 19$
C	0.0001/0.0004	2	0.00005/0.0002	1/2.55	$F_{0.1}(2,2) = 9$
Error	0.0001/0.000157	2	0.00005/0.0000785		
Sum	0.014/0.017837	8			

Note: The value before “/” is the expansion coefficient, and the value after is the bend–press ratio

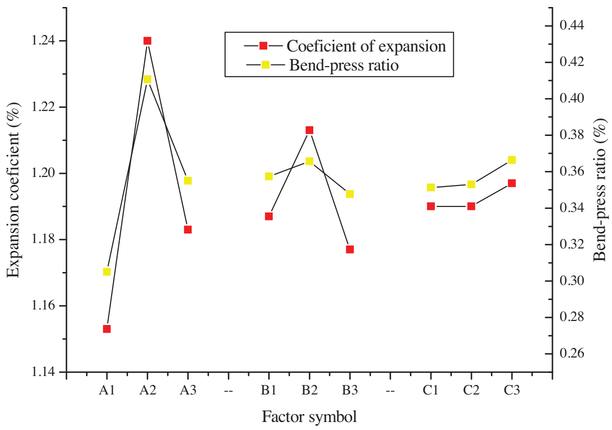

Figure 7. Influence curves of three factors on the expansion coefficient and bend–press ratio.

triple molds, and those molds were removed after seven days of natural curing. In accordance with the compression and bending test methods mentioned above, the mechanical properties tests were carried out with the range and variance analysis. See the results in Tables 3 and 4, and Fig. 7.

Field site sealing application

The boreholes' sealing performance of DPCS can be examined by a field site test in a coal seam. For comparison, the common sealant used in coal mines is used as the comparative material to seal the boreholes under identical application conditions. Comparative indices, including the methane concentration, the pure methane volume, and the negative pressure in the drainage pipe are used as metrics to evaluate the relative sealing performance among these systems.

Filed site situation

The coal seam bedding predrilling sealing test was carried out in the transport entry of the working face 15201 of the Baiyangling mine in Jinzhong, Shanxi province. The 15201working face has a strike length of 1640 m, an inclination length of 200 m, and a coal layer inclination angle of 3°–9°. The geological conditions of number 15 coal seam are simple with a coal seam thickness of 4.4–4.9 m and an average thickness of 4.6 m. The original gas content of the coal seam is 7.86 m^3 t^{-1}, and the permeability coefficient is 0.0721 m^2 $(MPa^2 \times d)^{-1}$ showing that the coal seam is difficult to drain.

Drill cutting method for measuring the desired depth of sealing in the borehole

The sealing behavior directly affects the compactness of the broken coal cemented with DPCS around the drainage pipe. The sealing is to both prevent outside air from entering the pipe and thereby improving the concentration of extracted gas as well as for maintaining enough negative pressure in the drainage pipe to accelerate drainage efficiency.[37] Before embarking on a sealing process design, a reasonable sealing depth must be defined.

The borehole depth is generally determined by the drill cuttings method. The drill cutting method is used to drill a borehole of a 42 mm diameter on the coal wall vertically with a coal electric drill, and to analyze the stress distribution state in the coal body according to the variation law of the amount of drill cuttings and its accompanying dynamic phenomenon. The theoretical basis for the drill cutting method is as follows:

(1) There is a functional relationship between the volume of drill cuttings and the stress distribution in the coal.
(2) The dynamic phenomenon during the drilling process reflects the degree of stress concentration in the coal.

The relationship between the volume of drill cuttings and the stress in the coal is[38,39]

$$G = \gamma(\pi\alpha^2 + 2\pi RU_R) + \frac{1}{2}(R^2 - \alpha^2) \quad (3)$$

$$U_R = \frac{1+\mu}{2E}R\left(\sigma_c + \frac{q-1}{q+1}(2p - \sigma_c)\right) \quad (4)$$

where G is the volume of drill cuttings; γ is the coal bulk density; α is the hole radius; R is the inelastic zone radius; U_R is the radial displacement of the inner wall of the borehole considering expansion; μ is the Poisson's ratio; E is the elastic modulus of coal; σ_c is the uniaxial compressive strength of coal; q is a coefficient; φ is the internal friction angle; and p is the stress in the coal. Combining Eqns (3) and (4) defines the stress in the coal as proportional to drill cutting volume.

A total of eight boreholes were drilled in the haulage entry of the working face 15201 for the test, defined as number 1–8, the results are drawn in Fig. 8 with the test data.

Apparent from Fig. 8 is that the volume of cuttings peaks at a depth of 8–l0 m. According to the variation of the cuttings along the drilling depth with the stress

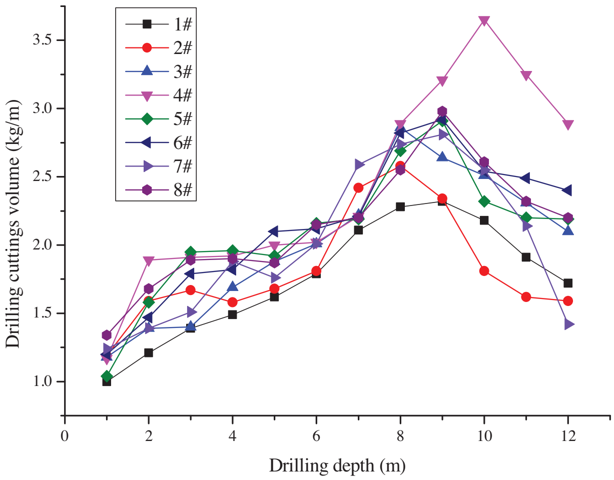

Figure 8. Relationship between drilling depth and drilling cutting volume.

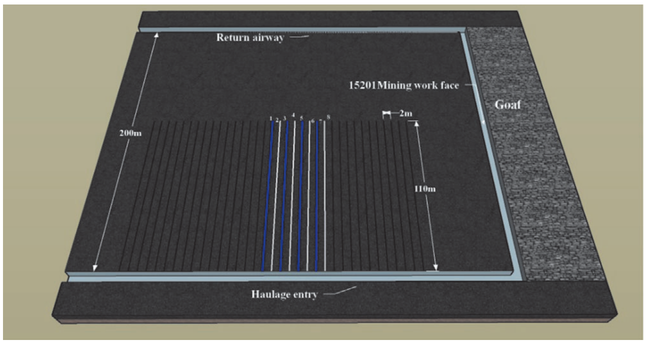

Figure 9. Layout of gas drainage boreholes in the transport entry.

distribution in the coal, it can be deduced that the range of l–8 m represents the pressure relief area of the roadway. In this range, the stress of the coal is completely relieved, and the volume of cuttings remains stable. The range of 8–10 m represents the stress increase zone with the average stress peak at a depth of 9 m, indicating that the average width of the damaged zone is 9 m. It is known from the gas flow characteristics of borehole sealing that the sealing depth should exceed the extent of the pressure relief zone, so the sealing depth is taken as 9 m here. In the actual sealing process, the sealing depth can be extended to 10 m considering different geological conditions and a safety factor.[22,39,40]

Borehole arrangement

Boreholes are drilled in parallel as shown in Fig. 9. The methane extraction boreholes are perpendicular to the center line of the transport entry with the drilling angle

Table 5. Borehole parameters for gas drainage in the transport entry.

Borehole	Vertical angle (°)	Borehole diameter (mm)	Depth (m)
1	−4	113	110
2	−4	113	110
3	−4	113	110
4	−2	113	110
5	−2	113	110
6	−2	113	110
7	−2	113	110
8	−2	113	110

determined according to the inclination angle of the coal seam. The height of the borehole is not less than 1.6 m from the bottom plate. The number1, 3, 5, and 7 boreholes are sealed with DPCS, and the number 2, 4, 6, 8 boreholes are used as the comparative groups and sealed with a common sealing material. The drilling parameters are shown in Table 5.

Sealing process

Considering the advantages and disadvantages of sealing with cement slurry or polyurethane, the two methods are combined to design the sealing process for DPCS. The specific sealing process is as follows. First, staggered holes are arranged evenly at the front end of the pumping pipe to prevent coal debris from being entrained into the pumping pipe and blocking the pipe during gas extraction. The gas can then enter the pipeline through these staggered holes. Second, burlap is wrapped about 1 m from the staggered holes at the front end of the pipe, and the newly mixed polyurethane liquid is poured onto the burlap, fixed with a wire. Then the pipe is immediately set into the borehole. After the grouting pipe and the slurry return pipe are inserted into the borehole, the borehole is inserted with the burlap to prevent the slurry from flowing out. When the polyurethane liquid seal solidifies, the grouting pipe and the grouting pump are connected and the prepared double-doped polymer-modified cement-based sealing material is injected into the borehole through the grouting pump at a prescribed ratio. Once slurry flows from the return pipe, the return pipe is clamped and the slurry is injected continuously for a period so that the slurry can enter the fractures around the borehole. The draining

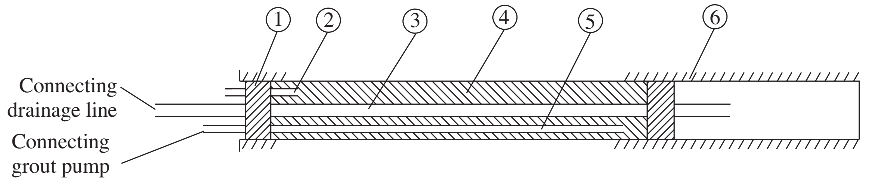

Figure 10. Schematic diagram of improved sealing process.

pipe is connected to the gas drainage branch after grouting. The gas drainage begins after completely curing for 24 hours. The sealing process is shown in Fig. 10.

The polyurethane bonds securely to the borehole wall, thus the two ends of the borehole are sealed with polyurethane, and the long-distance closed space formed in the center is filled with DPCS. The positive expansion property of DPCS aids the slurry in filling the fractures around the borehole. The polyurethane acts as a rubber stopper to prevent gas outflow and axial expansion.

According to the optimized sealing process described above, the gas parameters of the test boreholes were measured by using the CJZ7 gas drainage comprehensive parameter measuring instrument. To prevent the spontaneous combustion of the coal seam, gas drainage in the other boreholes in the transport entry was stopped due to the low methane concentration and the high carbon monoxide concentration.[41] The methane concentration, the pure methane flow volume, and the recorded negative pressure are shown in Fig. 11.

Results and discussion

Performance characteristics of DPCS

Expansion agent effect on the volume expansion

From Fig. 3, it is clear that the cement slurry will undergo volume shrinkage for the group of nonadditive mixes. As the expansion agent dosage increases, the expansion coefficient of the material will first increase and then decrease, rather than increasing monotonically. When the expansion agent dosage was 0.10%, the expansion coefficient reached the maximum at 17.74%. To analyze the causes of this phenomenon, we believe that there are two factors affecting the volume change of the modified cement mortar. One is the volume shrinkage caused by the evaporation and the absorption of water during the solidification process of cement mortar itself. The other is the expansion deformation of the cement mortar caused by the expansion agent.[30] The reason is that when the expansion agent dosage is relatively small, the volume shrinkage produced by the cement slurry itself is much smaller than the expansion deformation generated by the expansion agent. Thus, the slurry volume increases as the expansion agent dosage increases and the volume expansion coefficient progressively increases until it reaches a maximum.[42] Subsequently, the expansion rate further increases by so much that the expansion reaction cannot be completed. At the same time, the excess expansion agent will cause the evaporation and consumption of water, and a further shrinkage of slurry, resulting in a decrease in the expansion coefficient. An expansion agent dosage of 0.1% results in the maximum expansion coefficient at 17.74%. Therefore, the expansion agent dosage selected in this experiment is 0.1%.

Polymer–cement ratio effect on the bend–press ratio

From Fig. 4, we find that the compressive strength changes consistently for the double-doped and single-doped cements. The compressive strength increases first and then decreases with an increase of the polymer-cement ratio. When the polymer–cement ratio is 2%, the compressive strength of the cement specimen reaches a peak, and the flexural strength increases as the polymer mass increases. When the polymer–cement ratio is 6%, the flexural strength of the cement specimens reaches its maximum and then the flexural strength decreases slightly. The compressive strength of the modified cement material is superior to the ordinary cement. At the same polymer–cement ratio, the compressive strength of DPCS is larger than that for the single-doped polymer-modified cement, indicating that the modification effect of the single-doped polymer is

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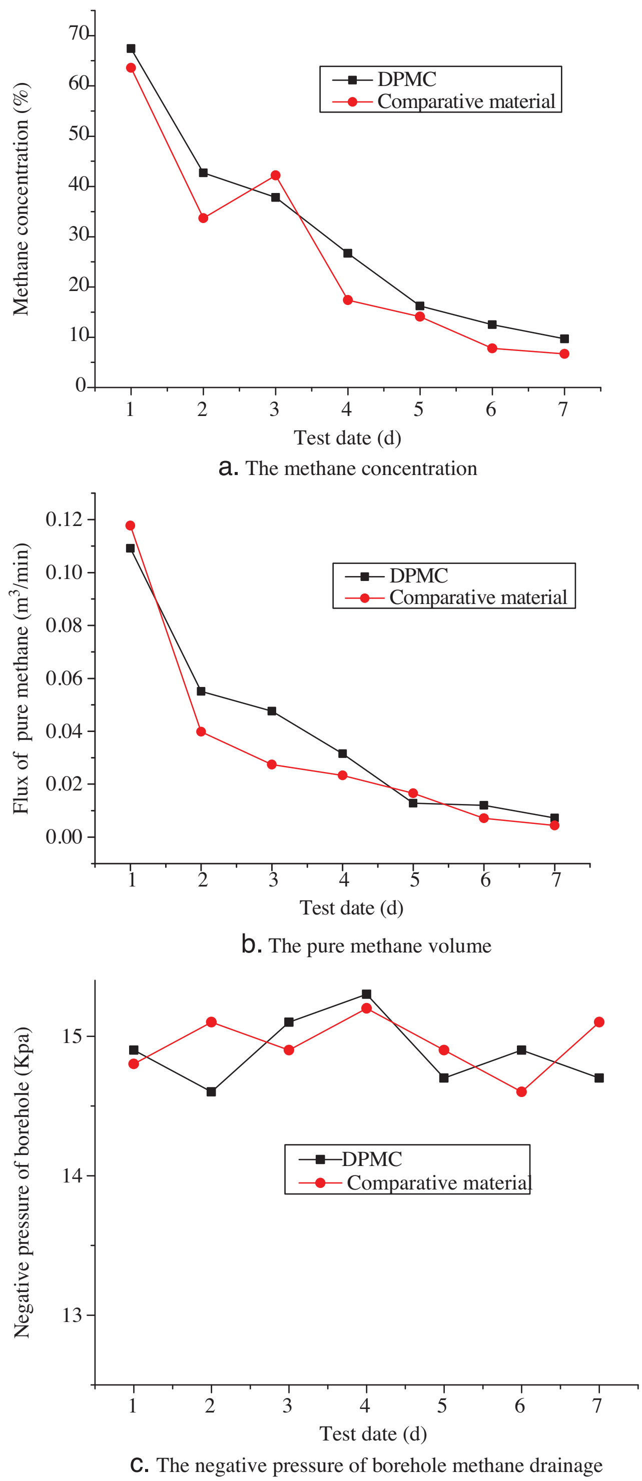

Figure 11. Gas drainage parameters in the test boreholes.

limited, and that the double-doped polymer has the superior strength of the cement specimens.

From Fig. 5, we find that as the polymer dosage increases, the bend–press ratio of the specimens increases remarkably. When the polymer–cement ratio is 6%, the bend–press ratio reaches its peak of 0.403 with reduced brittleness. This suggests that the double-doped polymer-modified cement material can better meet the desired sealing needs.

The reasons for the same are as follows: the polymer A2 is uniformly dispersed in the cement slurry, and as the cement hydration reaction proceeds, the polymer membrane is formed and filled in the micropores of the cement hydration product. The hydration reaction consumes a large amount of free water, in which case the polymer and the hydration product interweave to form a network structure.[11,15] Due to the addition of the emulsion polymer A1, the emulsion particles are uniformly dispersed and deposited on the surface of the hydrogel and ettringite. As the hydration reaction proceeds, the resin emulsion attached to the surface of the hydrogel and the ettringite crystal forms a layered or agglomerated material that further fills the micropores in the cement hydration product,[28,30] and then, together with the polymer membrane of A1, closely bonds the cement hydration product and the unhydrated cement particles to a certain extent. The cracking of the cement is hindered to a certain degree, and the improvement of the mechanical properties of the cement material is relatively more significant.

Compactness

From Fig. 6 it is apparent that the porosity of the single-doped polymer cement sample decreases with an increase in the content of polymer A2, while the porosity of the double-doped polymer sample decreases first and then increases with an increase in polymers dosage. When the polymer–cement ratio is 6%, the porosity of the specimens is the smallest at 0.3403, then, as the polymer–cement ratio increases, the porosity of the material increases slightly. The results show that polymer A2 can significantly improve cement densification, and its combination with polymer emulsion A1 can bring better densification effect when the polymer–cement ratio is 6%.

Further analysis of the experimental phenomena reveals that when polymer A2 is added the cement particles undergo a hydration reaction, and a continuous three-dimensional network structure film is formed in the polymer slurry system intertwined with the cement material to form a dense structure.[28] The porosity is reduced and the compactness is improved.[43] On the basis of this, when the polymers are added, the emulsion particles continue to crosslink and solidify into a membrane, further filling the pores in the cement, and adhering the unhydrated cement particles. The hydration products and the polymer particles combine together, increasing the density. However, as the polymer dosage increases, the cement hydration remains incomplete and the continuity of the internal phase is interrupted. The polymer then cracks due to excessive shrinkage, forming a weaker structure and resulting in an increased porosity.

Optimized results of orthogonal test

Range and variance analysis on expansion coefficient

Since the k_i value in Table 3 is the mean value of the expansion coefficient of the corresponding factor at the respective level, the curves of expansion coefficient in Fig. 7 can reflect the influence of the three factors on the expansion coefficient of the prepared cement sealing materials.

(1) Range analysis

The range value R can be used to measure the effect degree of corresponding factors on the test results. If the R value of a certain column is larger, the influence of the factor corresponding to that column on the test results is greater. Under normal circumstances, the factor with large range is the main factor.

From Table 3, it is apparent that the test group number 5 has the largest expansion coefficient of 1.26 among the nine orthogonal test groups.

As shown in Fig. 7, when the polymer–cement ratio, the expansion agent dosage, and the dispersant dosage are 6, 0.10, and 1.2% respectively, the sealing material has the largest expansion coefficient and the best expansion performance. According to the range result: 0.087 > 0.036 > 0.007, factor A has the largest range, factor B the second, and factor C the smallest. Thus, the polymer–cement ratio is the main factor affecting the expansion performance. The expansion agent dosage is the secondary factor, and the dispersant dosage has the least influence on the expansion coefficient. In the test group number 5, when the polymer–cement ratio is 6% and the expansion agent dosage is 0.10%, the developed sealing material has the largest expansion coefficient and the best expansion performance which is consistent with the conclusions of the previous single-factor test.

(2) Variance analysis

The variance analysis is used to study which variables have significant effects on the observed variables according to the variance of the observed variables. From Table 4, $F_A = 116 > F_{0.01}(2,2)$, indicates that the effect of A (polymer-cement ratio) on expansion performance is particularly significant. $F_{0.01}(2,2) < F_B < F_{0.05}(2,2)$, shows that B (expansion agent dosage) has a significant effect on expansion performance. $F_C < F_{0.1}(2,2)$, shows that C (dispersant dosage) has the least impact on expansion performance and can be ignored. This analysis of variance is consistent with the range analysis. In summary, the ratio type of A2B2C3 (the test ratio of number 5) is the best choice.

Range and variance analysis on the mechanical properties

(1) Range analysis

As is shown in Fig. 7, the modified sealing material of test group number 6 (A2B3C1) has the largest bend–press ratio and the best mechanical properties. In addition, the range difference of factor A > factor B > factor C, identifying that the polymer–cement ratio is the main factor affecting the bend–press ratio, that the dispersant dosage is the secondary factor, and that the expansion agent dosage has the least influence on the bend–press ratio. It can be seen that the bend–press ratio first increases and then decreases with an increase in the poly–ash ratio. This reaches a peak when the polymer–cement ratio is 6% consistent with the conclusions in the previous single-factor test.

(2) Variance analysis

From Table 4, $F_A = 106.88 > F_{0.01}(2,2)$, indicates that factor A (polymer–cement ratio) has a significant effect on the mechanical properties, while F_B and F_C $< F_{0.1}(2,2) = 9$, indicate that factor B (expansion agent dosage), and factor C (dispersant dosage) have little effect on the mechanical properties and the effect of them can be ignored. The result of the variance analysis is consistent with that of the range analysis.

In summary, from the expansion property analysis, the best ratio is determined as 6% polymer–cement, 0.10% expansion agent, and 1.2% dispersant. From the mechanical property analysis, the best ratio is determined as 6% polymer–cement, 0.12% expansion agent, and 0.8% dispersant. According to the results of range and variance analyses, it is found that the polymer–cement ratio is the most important factor affecting the expansion and mechanical properties of

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the developed material. The expansion agent has a significant effect on the expansion performance, while the effect of the dispersant on the expansion property and mechanical property of the sealing material is the smallest. The expansion with a 0.10% dosage of the expansion agent is larger than that at a dosage of 0.12%, thus the optimal ratio of the sealing material is decided as 6% polymer-cement ratio, 0.10% expansion agent dosage, and 1.2% dispersant dosage.

Field test results analysis

Apparent from Fig. 11(a) is that the gas concentration of the two alternate sealing materials decreases throughout the duration of drainage. The initial gas concentration reaches a maximum of 70% and then continuously decreases to 10% when drainage is stopped. The gas concentration of the boreholes sealed with DPCS is higher than that of the comparative material and is more stable. From the gas flow curves in Fig. 11(b), it can be seen that with the progress of gas drainage, the pure gas flow continues to decline. At the beginning, the pure gas flow is about 0.12 m^3/ min, then the curves decrease rapidly, indicating that the two sealing materials can both play a good sealing effect. On the second day, the decrease rate of the pure gas flow becomes smaller, and a gentle decline is maintained. The pure gas flow in the boreholes using DPCS sealing material is significantly higher than that of the comparative material up to about 60%, showing that DPCS has better sealing performance in the middle of gas extraction. By the 5th day, as the gas content in the coal seam decreases, the pure gas flow also drops to 0.01 m^3 min^{-1} and tends to be stable. As is shown in Fig. 11 (c), the negative pressures in the two borehole groups change little over time and remain a high level of negative pressure with only a small difference between them. The reason may be that the test boreholes are not enough, the sealing conditions of the boreholes are quite good, and no serious air leakage has occurred.

Due to the low permeability of the coal seam at the 15201working face, it was difficult to drain gas in the past. The gas drainage in mine needs to consider the time cost, that is, completing the gas drainage in a shorter time to ensure efficient and safe mine production. In the DPCS field site sealing experiment, it can be concluded that the performance of DPCS is better than the comparative sealing materials conventionally used in this mine, especially in the medium-term of gas extraction process. In addition, from the auxiliary analysis of economic factor, the comparative sealing material conventionally used in this mine sells for more than 600 Yuan per bag while the raw materials of DPCS costs about 150 Yuan per bag, the cost of the original sealing material is much higher than the novel sealing material developed in this study even considering the material processing costs. And under normal circumstances, one bag of this comparative sealing material can only seal one borehole while using DPCS to seal boreholes saves more material. In summary, DPCS is a better choice than the comparative sealing material commonly used in this mine.

Conclusions

A mixture optimization study is completed for a novel DPCS to enhance borehole seals and gas drainage. The following conclusions may be drawn:

(1) The ratio of the two polymers (A1 and A2) in the cement slurry is optimally determined as 1:2. The expansion, the compactness, and the mechanical properties are selected as the key performance evaluation indices. The experimental program establishes that the expansion coefficient is a maximum of 17.74% when the expansion agent dosage is 0.10%. The maximum bend–press ratio is 0.403 and the optimal porosity is 0.34034 when the polymer–cement ratio is 6%.
(2) Analysis of the expansive properties in an orthogonal experiment identifies that the optimal group number 5 (A2B2C3) has the largest expansion coefficient and the best expansion performance. In the analysis of the mechanical property test, it is found that sample group number 6(A2B3C1) has the largest bend–press ratio and the best mechanical performance. In addition, the range and variance analyses show that the polymer–cement ratio is the main factor influencing the expansion and mechanical properties. The expansion agent dosage remains a secondary factor while the dispersant dosage has the least impact. Considering these comprehensive effects, the optimal ratio of the sealing material is determined as 6% for the polymer–cement ratio, 0.10% for the expansion agent dosage, and 1.2% for the dispersant dosage.

(3) In an experimental evaluation of sealing, *in situ*, the length of the sealing hole is determined to be 10 m by the drill cutting method. The result of field test shows that the comprehensive performance of DPCS on the gas drainage concentration, the pure gas flow, and the negative pressure is better than that of the comparative sealing material that is commonly applied in the mine though the cost of using DPCS is substantially less expensive.

Acknowledgments

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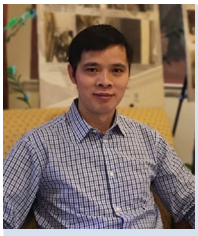


Aitao Zhou

Aitao Zhou is an Associate Professor in the College of Emergency Management and Safety Engineering, China University of Mining and Technology, Beijing, China. His research interest includes mining safety, especially in the area of mining ventilation induced by coal and gas outbursts. His current research projects include multiscale model of outburst shock waves and induced disorder law of mine airflow and methane drainage and utilization in coal mines.

Jiawen Wang

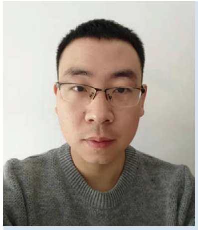
Jiawen Wang is pursuing his Master's in the College of Emergency Management and Safety Engineering, China University of Mining and Technology, Beijing, China. His research interests include the areas of computational numerical simulation, rock mechanics, and in the mechanical and transport properties of fractured coal rocks, with application to mine gas prevention and mine ventilation.

Kai Wang

Kai Wang is a Professor in the College of Emergency Management and Safety Engineering, China University of Mining and Technology, Beijing. His research interest includes safety engineering, safety and emergency management, mine safety engineering, coal and gas outburst disaster prevention, mine ventilation, etc.

Derek Elsworth

Derek Elsworth is a Professor in the Departments of Energy and Mineral Engineering and of Geosciences and the Center for Geomechanics, Geofluids, and Geohazards at Pennsylvania State University, University Park, USA. His research interests include the areas of computational mechanics, rock mechanics, and in the mechanical and transport characteristics of fractured rocks, with application to geothermal energy, the deep geological sequestration of radioactive wastes and of CO_2, and recovery of unconventional hydrocarbons including coal-gas, tight-gas-shales, and hydrates.



Meng Zhang

Meng Zhang is a PhD candidate in the School of Emergency Management and Safety Engineering, China University of Mining and Technology, Beijing. His research interests include the areas of computational fluid dynamics, two-phase flow propagation characteristics of coal and gas outburst, and emergency rescue of coal and gas outburst.

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