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Research Article

Experimental Study on the Influence of PVA Content on the Performance of Grouting Material in Deep Stope

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Based on laboratory experiments, the paper researches the effect of polyvinyl alcohol (PVA) content on the performance of fly ash-based cement grouting materials. The test results show that the addition of PVA has limited effect on the initial and final setting time and brings a certain but minor delay on the appearance of the hydration peak period. It enhances cement hydration during the hydration slow down period. The impermeability of PVA to the grouting material is obviously improved, and it increases with the growing amount of PVA content. PVA improves the flexural strength and compressive strength of the sample block to varying degrees, and the improvement on flexural strength is more obvious than that on the compressive strength. It has a significant effect on the mechanical properties of the grouting material and can effectively improve the rock mass's hydraulic coupling characteristics. It also has great application value and practical significance for the prevention and control of water hazards in deep coal stopes.

1. Introduction

Mine water disasters have always been one of the biggest obstacles restricting the safe coal production in our country. With the continuous extension of mining, the mining depth and intensity continue to increase, resulting in a significant increase of the karst water pressure of the rock formation, and the original microcracks in the rock mass develop into macro cracks and new cracks that cause the rock mass to crack or even break. The pressurized water lift zone will then be forced to expand upwards with the growing water pressure, resulting in serious water inrush disasters. Water inrush often occurs within a period of time after the end of mining, which is often sudden and unpredictable. In view of the seriousness, suddenness, and unpredictability of water inrush disasters in deep stopes affected by the "three highs" of high crustal stress, high water pressure, and high mining disturbances, the most effective way to solve water disasters is to prevent with pregrouting [1–5].

The stress in the deep coal stope is complex. While the vertical stress in the crust increases linearly with depth grows, the horizontal stress and the buried depth are more complicated. According to the statistics of 116 field data worldwide, when the buried depth is within 1000 m, the horizontal stress is 1.5 to 5.0 times the vertical stress. The current state of crustal stress in China varies with depth and is closely related to the mode of fault activity. Therefore, in most areas, the horizontal stress is greater than the vertical one [6–9]. So the horizontal stress in the deep coal stope has a far greater and more complicated influence than the vertical stress. When the deep stope's water barrier is subjected to large horizontal stress, it will inevitably bend and deform. Therefore, the deep stope not only requires good compressive strength for grouting materials but also good flexural strength.

The traditional grouting materials for mining are mainly cement-based ones. But those grouting materials are porous and brittle. Under external forces, especially the high crustal stress, high water pressure, and high mining disturbances, they are prone to crack, affecting the bearing capacity and durability of grouting materials [10–14]. In order to reduce the cost of grouting, improve its performance, and adhering to the concept of green mining to protect the environment, many Chinese experts and scholars have made outstanding achievements by adding fly ash, slag, and industrial waste powder and other methods on the basis of cement-based grouting materials. The research results have been applied in mines. However, the problems of bearing capacity, durability, and impermeability of grouting materials have not been solved, especially in strengthening the flexural strength and water inrush performance of deep stopes [15–27]. Therefore, it is imperative to develop a grouting material with higher compressive and flexural strength, better toughness, and stronger impermeability.

Polyvinyl alcohol (PVA for short) is a low-cost, nontoxic, and safe high molecular polymer. PVA1778, PVA1788, and PVA1799 are the most common specifications. Among them, PVA1799 is hardly soluble in water at room temperature and often used as a fiber material to add to the slurry, therefore unable to give full play to its excellent performance. PVA1799 has good flexibility, durability, and air- and water-resisting properties, which can significantly improve the mechanical properties and impermeability of grouting materials. However, the general theory believes that PVA will form a polymer film in the slurry to wrap the cement particles, thereby inhibiting the hydration process of the cement particles, affecting the performance of the material. Therefore, PVA is rarely used as a grouting material for research. If the shortcomings of PVA could be overcome, we could apply it to the grouting material in the deep coal stope, perfectly solving the problem of lacking impermeability and flexural strength and the weak flexural strength of existing grouting materials [28–37].

The author made PVA solution by heating, mixed with cement and fly ash to make grouting material, and set up different PVA contents to test separately. Based on past experience, a slurry with a mass concentration of 65% was conducted in the experiment to study how different PVA content grouting materials affect cement hydration process, impermeability, flexural strength, compressive strength, and initial and final setting time [38–47].

2. Experimenting Materials, Equipment, and Methods

2.1. Experimenting Materials. Ordinary silicate 325 cement was used for the ratio test, and the chemical composition is shown in Table 1. The fly ash used in the experiment comes from a thermal power plant. The chemical composition of fly ash is shown in Table 2. The quality indicators of the polyvinyl alcohol used in the experiment are shown in Table 3.

2.2. Experimenting Equipment. The experimenting equipment mainly used in the test includes the following: ULTRATEST ultrasonic tester, SS-1.5 mortar penetration tester, Shanghai Hualong WHY-2000 compression tester, MTS CDT1205-2 bending tester, and Vicat tester. The details are in Figure 1.

2.3. Testing Methods. Firstly, the PVA particles of certain mass are weighed and put into the beaker, and the corresponding quality of pure water is also added into the beaker according to the proportion. The magnetic stirrer is put into the beaker, and the beaker mouth is covered with a board to reduce the water loss. The ika-hs7 heating plate is used to heat and start the stirrer to stir. The heating temperature is set at 200°C, and the stirring speed is set at 220 r/min. With continuous heating and stirring, PVA particles gradually dissolved, forming a completely clear and transparent colloidal solution, and finally, PVA solution with mass concentration of 10% was made. In the experiment, the mass ratio of fixed cement and fly ash is 1 : 2 with mass concentration of slurry being 65%, and 6 sets of tests are designed with different PVA contents. First, weigh the cement and fly ash in the solid phase and mix well, and then, add water and stir for 3 minutes, and in the end, add the corresponding amount of PVA and mix well. Table 4 shows the experimental design plan.

Table 1: Main chemical composition of silicate 325 cement.

Composition	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_2
Percentage	22.63%	5.36%	65.12%	3.52%	1.28%	2.09%

Table 2: Main chemical composition of fly ash.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO
Percentage	56.65%	34.09%	1.42%	6.43%	1.41%

In the ultrasonic test, pour the stirred slurry into the experiment mold. The test will emit an ultrasonic signal every 2 minutes according to the program. We could speculate the hydration process of the grouting material by detecting the propagation speed of the ultrasonic wave. In the impermeability test, pour the slurry into the mold for curing and demolding, apply paraffin wax evenly after heating in the oven and put it back to the mold to seal, and then install the sealed sample on the mortar permeameter, and in the end, follow the set procedure to give a certain water pressure from the bottom and slowly pressurize it to observe the penetration of the sample. In flexural strength and compressive strength test, pour the stirred slurry into the experiment mold and attend to it at room temperature, and then, test the flexural and compressive strengths of the samples for 7 d, 14 d, and 28 d. In the initial setting test, pour the slurry into the mold and place it under the test needle of the Vicat instrument. After zero adjustment, slowly lower the test needle to contact the sample surface, tighten the screw, and suddenly loosen it for 1-2 seconds. The test needle will be inserted vertically and freely. Observe the reading after the test needle stops sinking or release the test needle for 30 seconds. When the test needle reads 4 mm ± 1 mm, the initial setting state is reached. In the final setting test, turn the sample 180° and apply to the same test method. When the test needle trace is ≤0.5 mm, the final setting state is reached. The influence of PVA content on the performance of grouting material is analyzed through the above series of tests; see Figure 2.

3. Results and Analysis

3.1. Ultrasonic Test. Figure 3 shows the trend diagram of the ultrasonic wave propagation velocity with time in the ultrasonic test with different PVA contents.

Table 3: Quality index of polyvinyl alcohol.

Item	Content	Light transmittance	Average degree of polymerization	Nonvolatile matter	Sodium acetate	Acetate
Requirement	≥90%	≥85%	1799 ± 50	≤7%	≤0.5%	≤0.5%

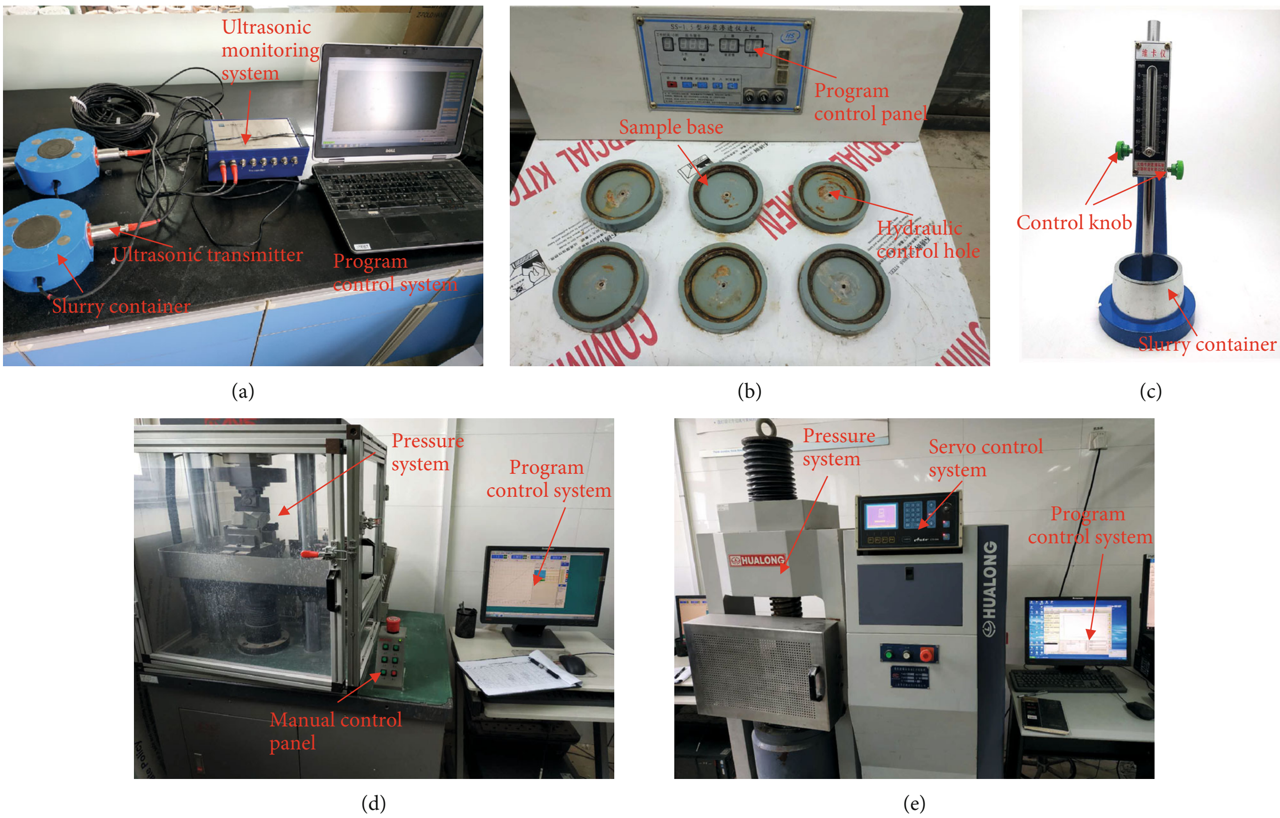

Figure 1: Diagram of test equipment: (a) ULTRATEST ultrasonic tester; (b) SS-1.5 mortar penetration tester; (c) Vicat tester; (d) Meters CDT1205-2 flexural testing machine; (e) Shanghai Hualong WHY-2000 compression testing machine.

Table 4: Experimental design scheme.

Group	PVA added amount	Solid phase mass ratio (cement : fly ash)	Concentration	Antifoaming agent (silicone oil) dosage
1#	0%	1 : 2	65%	0.1%
2#	0.1%	1 : 2	65%	0.1%
3#	0.3%	1 : 2	65%	0.1%
4#	0.5%	1 : 2	65%	0.1%
5#	0.8%	1 : 2	65%	0.1%
6#	1%	1 : 2	65%	0.1%

(1) According to the figure, the author divides the preliminary hydration process into 5 stages, the stationary period (0–600 min), the peak period (600–1000 min), the slowing period (1000–1500 min), the prestable period (1500–2500 min), and the stable period (after 2500 min)

(2) The inflection point appears at about 600 min and then the hydration peak comes. With the increase of PVA content, the inflection point is slightly delayed but not much. Generally speaking, it has no effect on the delay of the inflection point during the peak of hydration. It can be seen that theoretically PVA film enveloping the cement particles has a minor effect in inhibiting the hydration process

(3) From 1000 min to 1500 min, it is the hydration slowdown period. It can be seen from the figure that as the content of PVA increases, the slope of the ultrasonic propagation velocity curve gradually increases. The steeper the slope, the faster the hydration speed is. It can be seen that PVA promotes hydration in the slowdown period. As more PVA content comes in, the long carbon chain macromolecular structure of PVA plays an important role, making the distribution of cement particles more even, opening up the agglomeration structure of more cement particles, and enabling a better hydration reaction of cement particles so that the hydration speed in the slowdown period could be faster

(4) From 1500 min to 2500 min, it tends to enter the stable stage. The test curve without PVA is smooth with

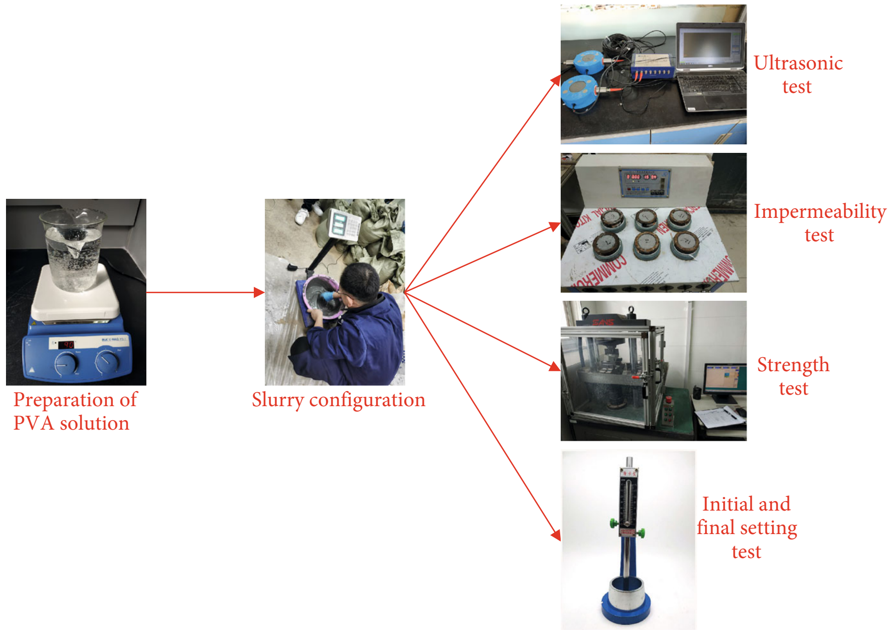

Figure 2: Record diagram of the experimental process.

no fluctuations; the test group with PVA content has fluctuations more or less, which shows PVA in that process has a certain inhibitory and fluctuating effect

(5) After 2500 min, the stable period comes. PVA content has no obvious influence on the ultrasonic propagation speed after that period. After 2500 min, the sample block is completely set and the hydration reaction is basically completed. The subsequent hydration reaction of cement particles will be very slow so that no fluctuation can be seen in the ultrasonic curve

3.2. Impermeability Test. Set up the test program. First, seal the sample with melted paraffin to an impermeable mold and fix to the impermeable equipment. Then, give a certain water pressure from the bottom of the sample, which gradually goes up from 0.1 MPa, increasing by 0.1 MPa every 30 min, and then observe the penetration of the sample. Record the pressure and time when water droplets ooze out, and record the time from when the water droplets appear to the time when they converge to cover the entire sample. The test results are shown in Table 5.

(1) It can be seen from the results of impermeability test that PVA added grouting material significantly improves the karst water pressure impermeability of the original material, and the performance gets better as PVA content increases. This is because PVA is evenly distributed in the grouting material sample in the form of a polymer film, filled in the gaps of cement hydration products, blocking pore channels and improving the compactness of the cement gel. Moreover, the physical properties of PVA are stable and with great waterproof properties that is why grouting material added with PVA can significantly improve the impermeability

(2) When the PVA content is less than or equal to 0.6%, the impermeability performance increases rapidly as the PVA content goes up. When the PVA content is higher than 0.6%, the improvement of the impermeability performance with the increase of the PVA content significantly slows down. The test results show that impermeability will be in the best state when PVA content is from 0.6% to 1%. This shows that there is a certain upper limit for the improvement of the impermeability of PVA to the grouting material, which will gradually become stable as the PVA content increases. This is because a small amount of addition of PVA has a certain promotion effect on the hydration and coagulation of the slurry and improves sample's compactness and water resistance. If PVA content is more than needed, it will focus more on wrapping the cement particles, thereby inhibiting the hydration process and affecting the compactness and impermeability of the sample

(3) From the experiment, we could know that when the high-pressure water at the bottom penetrates through the sample block, it first appears in the form of dense small water droplets on its surface, and then, the water droplets gradually converge into clusters. Under the same osmotic pressure, the more PVA content there is, the less time water droplets need to converge into clusters, that is to say, the penetration rate slows down. It is fair to say that the increase of PVA content helps to slow down the penetration rate of high karst water pressure to the sample block. This is because PVA is a high-molecular water-soluble polymer that is evenly

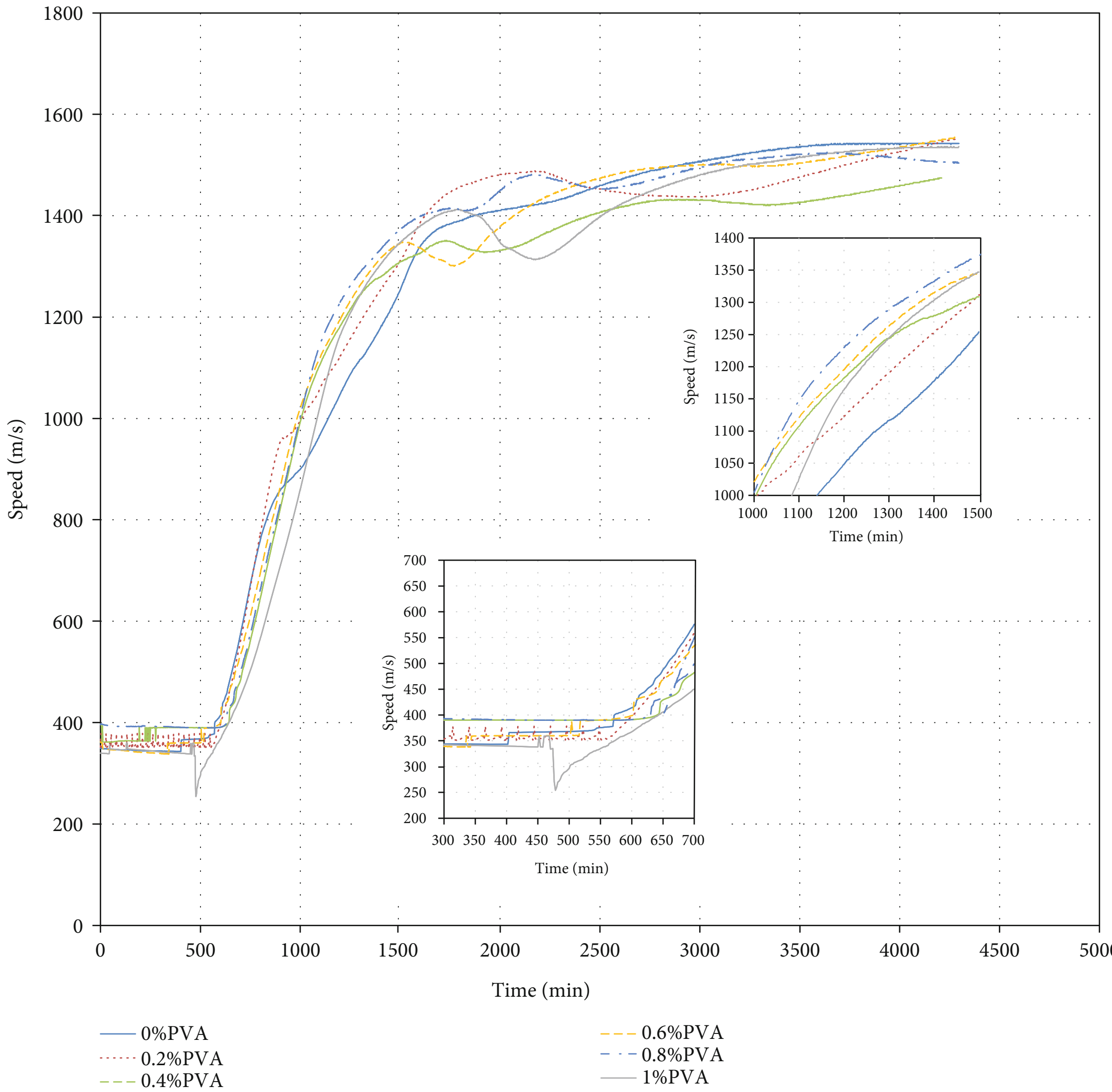

Figure 3: Trend chart of ultrasonic propagation velocity over time.

Table 5: Record table of impermeability test results.

Group	PVA content	Penetrating water pressure	Impermeability time	Time for water droplets to converge
1#	0%	0.3 MPa	5 min	27 s
2#	0.2%	0.3 MPa	28 min	49 s
3#	0.4%	0.4 MPa	7 min	25 s
4#	0.6%	0.4 MPa	21 min	33 s
5#	0.8%	0.4 MPa	20 min	32 s
6#	1%	0.4 MPa	23 min	40 s

distributed in the sample block, which is well connected to the cement particles, reducing the volume shrinkage during the hydration and coagulation process of the cement, thereby reducing the number of microcracks and size, improving the structure of the sample block matrix

3.3. Flexural and Compressive Strength Test. Figure 4 is a graph showing the changing trend of flexural strength of different contents of PVA. Figure 5 is a histogram of the improvement on flexural strength of different contents of PVA relative to no PVA, and Figure 6 is the changing trend of compressive strength of different contents of PVA. Figure 7 is a histogram of the improvement of the compressive strength of different contents of PVA relative to no PVA.

(1) The 7 d flexural strength increases first and then weakens with the growing of the PVA content. The peak value is 1.48 MPa when the PVA content is 0.4%, which is a 42.3% increase compared to the sample without PVA; 14 d flexural strength increases as PVA content goes up, but the increasing speed slows down later. When the PVA content reaches 0.6%, the strength is basically stable at about 2.6 MPa, with a peak value of 2.62 MPa, a 37.2% increase compared to the sample without PVA; the 28 d flexural strength first increases

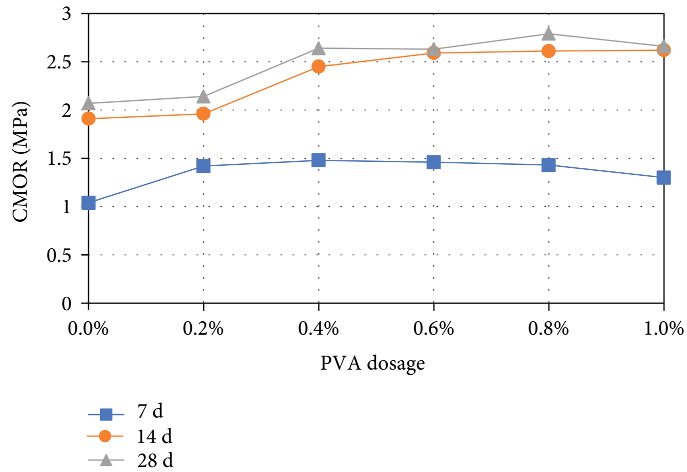

Figure 4: Trend chart of flexural strength with PVA content.

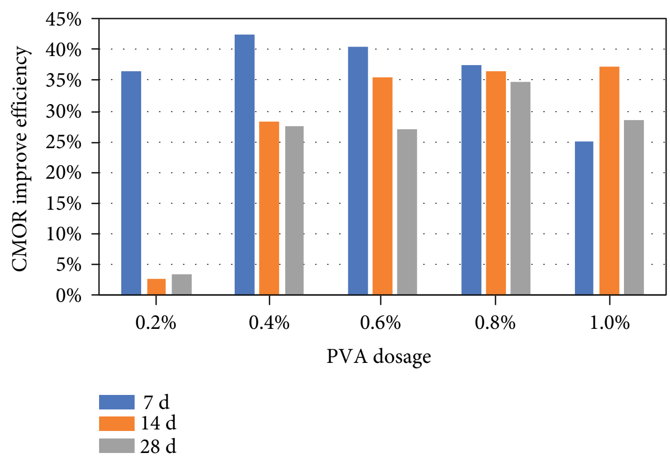

Figure 5: Histogram of the improvement effect of different PVA contents on flexural strength.

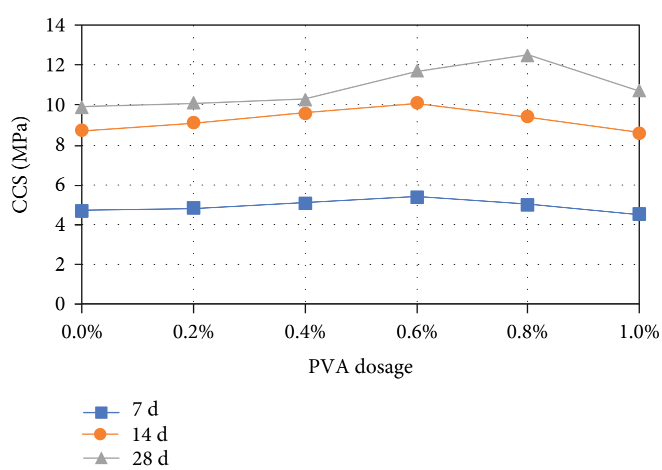

Figure 6: Trend chart of compressive strength with PVA content.

and then decreases as PVA content goes up. The peak value of 2.79 MPa appears when the PVA content is 0.8%, a 34.7% increase on the flexural strength compared with the sample without PVA. We could see that PVA has a significant effect on improving the flexural strength, and it can effectively prevent the bending deformation and damage of the deep stope's waterproof layer under relatively large horizontal stress

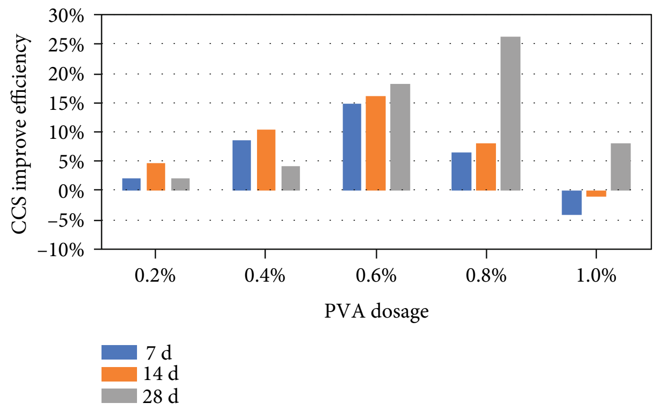

Figure 7: Histogram of the improvement effect of different PVA contents on compressive strength.

(2) 7 d compressive strength increases in the first place and then weakens as the PVA content rises. The peak value 5.4 MPa occurs when the PVA content is 0.6%. Compared with the sample without PVA, the compressive strength improved by 14.9%; 14 d compressive strength increases in the first place and then weakens as the PVA content goes up. The peak value of 10.1 MPa occurs when the content of PVA is 0.6%. Compared with the sample without PVA, the compressive strength improved by 16.1%; 28 d compressive strength increases in the first place and then weakens as the PVA content goes up. The peak value 12.5 MPa occurs when the PVA content is 0.8%. Compared with the sample without PVA, the compressive strength improved by 25.7%. It should be noted that when the content of PVA is 1%, the compressive strength of 7 d and 14 d does not improve but reduced by 4.3% and 1.1%, respectively, instead. It shows that when the content of PVA is ≥1%, it will bring negative effects to the compressive strength. When the PVA content is too high, the polymer film has an obvious wrapping effect on the cement particles, which inhibits the hydration and coagulation process of the cement particles, thereby reducing the compressive strength of the sample

(3) The best improvement on flexural strength of 7 d, 14 d, and 28 d is 42.3%, 37.2%, and 34.7%, respectively, whereas the best improvement on compressive strength of 7 d, 14 d, and 28 d is 14.9%, 16.1%, and 25.7%, respectively. PVA content's enhancing effect on the flexural strength decreases as time goes by, and its enhancing effect on the compressive strength increases with the passage of time. The reason for the decrease of flexural strength is that the hydration rate of cement is slow, the early cement has little effect on the strength improvement of sample block, and PVA has good toughness and high tensile strength, which reflects that PVA has a greater promotion effect. With the passage of time, the hydration process of cement is gradually completed, and the strength of the sample block is greatly improved. In this process, PVA does not further strengthen the sample block, so the effect of contrast is weakened. The overall improvement brought by PVA content to the sample block on flexural strength is larger than that on the compressive strength. This is because the PVA long carbon chain macromolecular structure is evenly distributed in the grouting material to form a polymer film. This film has good toughness and a certain tensile strength, which is a good complement to rock's nonflexural but compressive characteristics. It is also in the position to better cope with the situation that bending deformation is bound to occur when the water barrier of the deep stope is subjected to a large horizontal stress. It has a very significant effect on the improvement of the mechanical properties of the grouting material

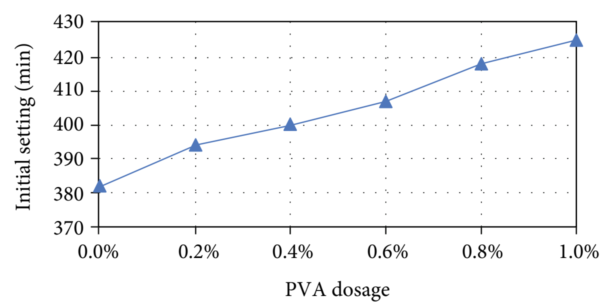

Figure 8: The change trend graph of the initial setting time of different contents of PVA.

(4) 7 d, 14 d, and 28 d flexural strength peak at 0.4%, 0.6%, 0.8%, respectively; 7 d and 14 d compressive strength peak at 0.6% whereas 28 d compressive strength peaks at 0.8%. Taking a comprehensive consideration for flexural and compressive strength, 0.6% PVA content should be the best choice

3.4. Initial and Final Setting Test. When conducting the initial and final setting time, one should be careful that the test needle should be lowered slowly during operation to prevent from being bent. For more accurate results and a shortened test time interval, it is measured every minute when

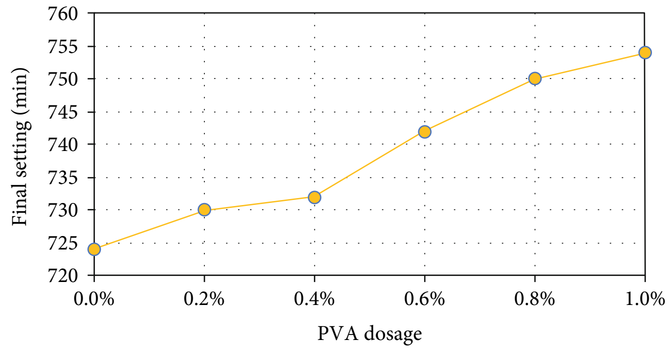

Figure 9: The change trend graph of the final setting time of different contents of PVA.

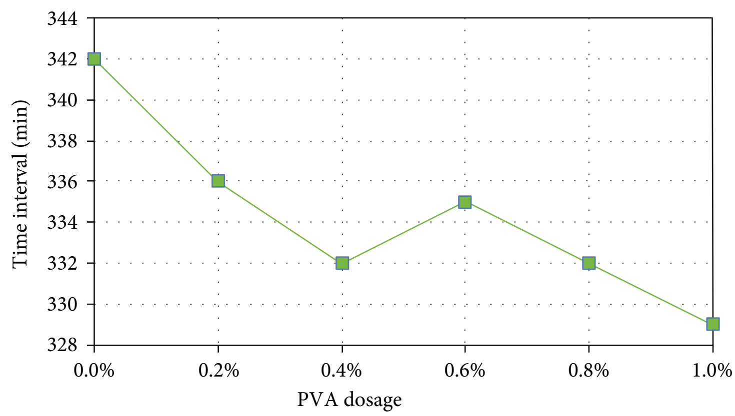

Figure 10: The change trend graph of the initial and final setting time intervals.

approaching the initial setting state and every 2 minutes when approaching the final setting state. An immediate test should be done when it reaches the initial and final setting state. It can be decided as the final initial or final setting state when two conclusions become identical. The following figures are based on test results. Figure 8 is the change trend graph of the initial setting time of different contents of PVA. Figure 9 is the change trend graph of the final setting time of different contents of PVA, and Figure 10 is the change trend graph of the initial and final setting time intervals.

(1) It can be seen from the figure that both the initial setting time and the final setting time rise with the increase of PVA content. It shows that the polymer film formed by PVA in the slurry envelops the cement particles and prevents the cement particles from contacting with water, thereby inhibiting cement hydration to a certain extent

(2) The time interval between initial and final setting has a tendency to decrease with the increase of PVA content, indicating that during the period from initial setting to final setting, the presence of PVA has a certain promotion effect on cement hydration reaction and the speeding up of the hydration process shortens the time interval between initial and final setting time. It shows that the long carbon chain macromolecular structure of PVA fully opens the agglomeration of cement particles and makes them evenly distributed, making the cement particles fully contact with water and promoting the hydration process of cement particles. That is how PVA promotes the hydration process from the initial setting to the final setting time

(3) The addition of PVA did not have a major impact on the initial and final setting time of the grouting material, especially that the gap in the final setting time is quite narrow. Traditional ideas believe that the polymer film prevents cement particles from contacting water, thereby preventing their hydration. That is not entirely correct. PVA has helped to delay the hydration but also accelerate it in midterm process

4. Conclusions

In this study, indoor tests were conducted on the hydration process, impermeability, flexural strength, compressive strength, and initial and final setting time of the grouting material added with PVA. The mixing ratio of PVA content was also been optimized for a better engineering application in the prevention of water disaster in the deep stope. The research indicates the following:

(1) PVA content has a certain but limited delaying effect on the appearance of the inflection point during the

peak period of hydration. Therefore, the PVA film enveloping the cement particles to inhibit the cement hydration process has quite limited effect. With the increase of PVA content during hydration's slowing down period, the slope of ultrasonic propagation velocity curve gradually increases with faster hydration. It can be conducted that PVA can promote cement hydration in the slowdown period because the long carbon chain macromolecular structure of PVA opens the agglomeration of more cement particles, making the distribution of cement particles more even and the hydration rate during the slowdown period faster

(2) PVA can significantly improve the karst water pressure impermeability of the grouting material, and it will be enhanced with the increase of PVA content. The impermeability effect is better when the PVA content being 0.6% to 1%. This is because PVA is evenly distributed in the grouting material sample in the form of a polymer film, filled in the gaps of cement hydration products, blocking the pore channels and connecting cement particles perfectly. Therefore, the cement hydration will coagulate in smaller volume thereby reducing the number and size of microcracks, improving the structure of the sample block matrix and the compactness of the cement gel. What is more, the physical properties of PVA are stable with great water-resisting property. Therefore, the grouting material with PVA can significantly improve its impermeability. PVA could be used in deep stope grouting material for better dealing with the penetration damage of high karst water pressure to the water-resisting layer

(3) PVA could improve the flexural strength by 42.3%, 37.2%, and 34.7%, respectively, and enhance the compressive strength by 14.9%, 16.1%, and 25.7%, respectively. The flexural and compressive strengths of the sample block are all improved with PVA but with more obvious improvement in flexural strength than the compressive strength. The polymer film formed by PVA has good toughness and certain tensile strength, which is a good complement to rock's nonflexural but compressive characteristics, bringing an obvious improvement to grouting material's mechanical property, especially in flexural strength. We could know that PVA content could cope with the complex stress environment of the deep stope and effectively prevent the bending deformation and damage of the deep stope's waterproof layer under relatively large horizontal stress

(4) From initial setting to final setting, the presence of PVA has a certain promotion effect on the cement hydration reaction, and when the hydration process is accelerated, it will shorten the time interval between initial and final setting. The long-chain macromolecular structure of PVA fully opens the agglomeration of cement particles and makes them evenly distributed so that the cement particles are in full contact with water and the hydration process is promoted. PVA has both the effect of delaying hydration in the initial stage and promoting hydration in the medium term

(5) Considering the complex stress field and high karst water pressure in the deep stope of the coal mine and the grouting material's hydration process, impermeability, flexural and compressive strength, and the initial and final setting time, the final optimized ratio for PVA content is 0.4% to 0.8%, and the best ratio is 0.6%

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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