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Influences of polymers on the properties of cement-sodium silicate grouts with a high water-binder ratio

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This study investigates the influence of redispersible latex powder and hydroxy propyl methyl cellulose on the physical and mechanical properties of cement-sodium silicate grouts with a high W/B ratio. The rheological properties, setting time, compressive strength, autogenous shrinkage sulfate attack resistance and permeability of the grouts were assessed and compared. The microstructures were investigated in terms of crack development. The results show that satisfactory properties and durability, such as an initial setting time extension of 20~55%, an autogenous shrinkage reduction of 0.332~1.21‰, and a permeability addition of 0.1–0.4 MPa, were obtained in cement-sodium silicate grouts with the addition of two polymers. The flexural strength of cement-sodium silicate grouts with redispersible latex powder was improved after curing in water and sulfate attack by 3 wt% sodium sulfate. However, the addition of two polymers reduced the compressive strength of all grouts except for the redispersible latex powder content of 3%. These research results can provide a technical reference for grouting material selection and mixing ratio adjustment.

Key words: Cement sodium silicate grout, Redispersible latex powder, Hydroxy propyl methyl cellulose, Physical and mechanical properties, Microstructure.

Introduction

Cementitious grouts are often used for geological strengthening and water control (GSWC) in grouting reinforcement engineering based on cost, application technology and environmental conservation. For resisting dilution and scour of moving water during grout injection, rapid setting times and accelerators are required [1, 2]. Sodium silicate solution (SSS) is frequently used as an accelerator because of its efficiency and storage and handling benefits to promote agglomeration and solidification of cement grout against gushing water [3-6]. The advantage of cement-sodium silicate (CS) grouts lies in their short gelation time and high compressive strength in the early stage, so they are widely used for strengthening weak strata and plugging leaks. Previous studies on CS grouts have mainly focused on the influence of the change of the double liquid volume ratio or the addition of admixtures and additives on the physical and mechanical properties [7-11]. When CS grouts are in the condition of water enrichment, sodium hydroxide and other substances are easily dissolved, which will easily cause alkali pollution to groundwater and the environment. Moreover, the alkalinity decrease of the concretion can easily lead to the decomposition of hydrated calcium silicate gel and other substances and ultimately lead to structural destruction of the concretion [12, 13]. When the solidified body of CS grouts is in a dry condition, the silica gel will dehydrate, which will result in shrinkage and cracking [14].

Adding a small amount of polymer to the cement mixture can significantly enhance the performance of the final material, which is called polymer-modified cement-based material (PMC) [15]. Polymer can significantly improve the tensile strength, flexural strength, flexibility, compactness and durability of cement-based composite material and has a wider adaptability to engineering compared with ordinary cement-based material slurry [16]. PMC has good flexibility and bonding properties, which can lap the cracks caused by shrinkage between particles and prevent the occurrence of cracks [17].

To date, polymers have become an important component of cement-based materials. However, most studies focus on the effects of polymers on mortar and concrete properties [16-20]; the combined effects of polymer and CS grouts have received less attention, especially concerning high water-to-binder (W/B) ratios. This study experimentally investigated the effects of redispersible latex powder (RLP) and hydroxy propyl methyl cellulose (HPMC) on the physical and mechanical properties of CSG with a high W/B ratio, considering varied mixing amounts of polymers, and performed a microanalysis based on scanning electron microscopy (SEM) to analyze the microstructure. According to the

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Table 1. Chemical compositions of the OPC power

Main oxides and related proportions (%)						
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Loss on Ignition
20.62	6.13	3.45	58.73	6.16	3.8	1.11

Table 2. The mixing ratio design of experiments referenced to OPC mass

Cases	CS-0	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
OPC	1	1	1	1	1	1	1	1	1
water	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
RLP	0	0.03	0.06	0.09	0.12	0.15			
HPMC							0.001	0.002	0.003

main results of this study, some suggestions are also proposed for further studies and engineering applications.

Experimental Materials and Methods

Experimental materials

In this study, ordinary Portland cement (OPC) with the strength grade of P. O 42.5 according to the China National Standards GB175-2007 was used, and its chemical composition, which was provided by the manufacturer, is listed in Table 1. A commercial SSS was adopted as the accelerator for the grout. The density was 1.26 g/cm^3, and the concentration and modulus values (Ms, SiO_2 to Na_2O ratio) of SSS used were 25 wt% and 2.6, respectively. In addition, two main types of polymers with a solid content of 99%, including RLP, which is based on the copolymer of vinyl acetate and ethylene, and HPMC, which has a viscosity of 100,000, were used.

Based on practical engineering and commonly used mixtures of CS grouts, the W/B ratio of the cement slurry was 0.8, and the designed volume ratio (VR, OPC paste to sodium silicate) was 3. During the experiment, the mixing amount of RLP and HPMC was changed to investigate their effects on the physical and mechanical properties of CS grouts, while the amounts of other components remained constant. To conveniently assess and compare the results, the mix proportions were converted and expressed in Table 2, in which the data denote the proportions relative to the mass of OPC.

Mix design and specimen preparation

CS grouts adopt a dual-liquid grouting process. OPC slurry and SSS are used as two liquids, which are then mixed and injected into the formation. OPC slurry with or without polymer is used as the A liquid, and alkaline SSS is used as the B liquid in this paper. The OPC powder, polymer powder and water were fully blended for 240 s in a cement slurry mixer in accordance with the predetermined W/B ratio. Then, a certain volume of SSS was poured into the mixer and blended with the prepared OPC slurry for 5 s to obtain fresh CS grouts. The stirring rate was approximately 285 r/min to ensure thorough mixing of the components. After stirring, the fresh slurry was cast into molds and kept in the molds for 2 h to avoid breaking when the specimen was exposed to air for a long time. The molds used to create the following specimens: a height of 40 mm, an inside diameter at the bottom of 70 mm, and an inside diameter at the top of 60 mm conical ring for final setting time; a 40×40×40 mm cube for the compressive strength test; a 25×25×280 mm prism (with two copper measuring heads embedded 15 mm at both ends) for the shrinkage test; a truncated cone shape with a 70 mm top diameter, an 80 mm bottom diameter and a 30 mm height for the penetration test; and a 40×40×160 mm prism for the rapid sulphate resistance test.

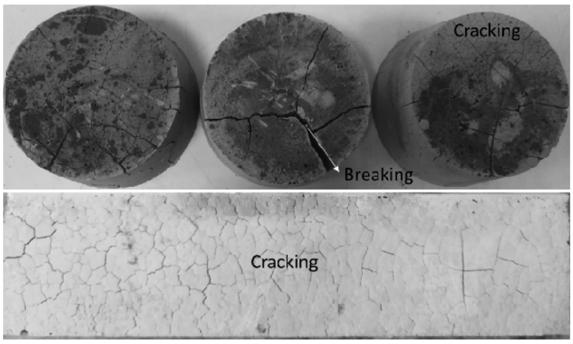


Fig. 1. Cracking and breaking of specimens in the air during the curing process.

Then, the specimens were demolded and cured in water at a temperature of 20±2 °C for a certain period because the specimens that were exposed to air suffered cracking and breaking (Fig. 1). Three specimens were made for each mixture to reduce deviations. The rapid stirring time used for the CSG was mainly because of their rapid setting time, which might lead to layered gelation of the specimens without adequate time for casting.

Methods

The initial setting time cannot be obtained by using the method of the ASTM C191-13 standard because the real W/B ratio of the proposed CS grouts was 1.15 and was rapid-setting. However, the final setting of the CS grouts adopts the method of this standard for testing. The initial setting time is usually determined by the "pour cup method". A and B liquid that had been weighed were placed in two plastic cups. Liquid A was added to liquid B, the timer was started, and then the liquid was poured back into the A cup and repeated at the specified frequency. The initial setting time was the time at which the surface of the CS grouts remained at 45° once the plastic cup was straightened [9]. The gelatinized CS grouts with inclined surfaces in plastic cups are shown in Fig. 2a.

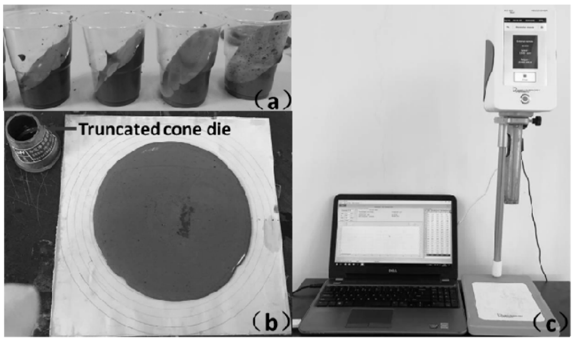
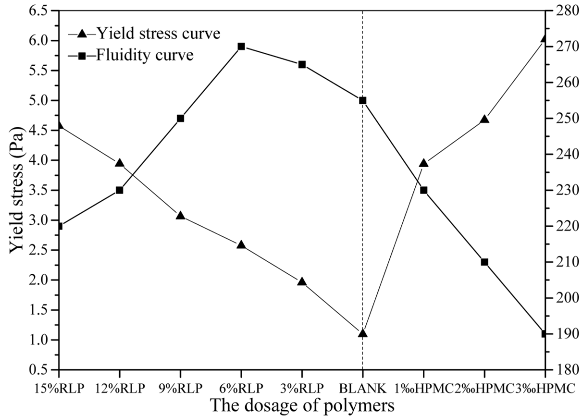
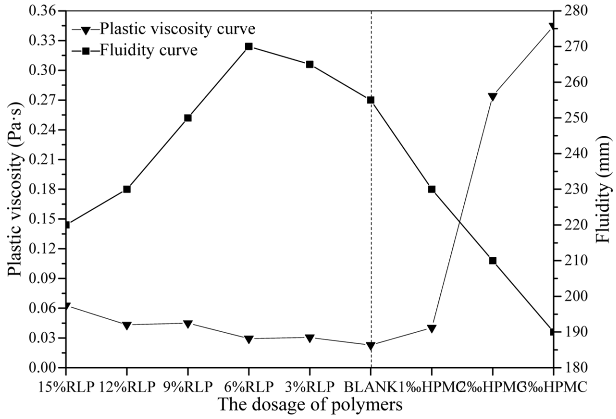


Fig. 2. The experimental instruments for the (a) setting time test, (b) fluidity test and (c) viscosity test.



(a) The yield stress and fluidity



(b) The plastic viscosity and fluidity

Fig. 3. The rheological properties of fresh slurry.

Although the incorporation of the polymer might prolong the setting time of the CS grouts, the slurry loses fluidity in only a few tens of seconds. The time and accuracy requirements required for the viscometer to test the rheological properties of the slurry cannot be met. Therefore, the yield stress and plastic viscosity of OPC slurry incorporating RLP and HPMC were tested to illustrate the effect of the polymers on the rheological properties. The fluidity was determined by a truncated cone die according to the national standard (GB/T 8077-2012) of China, and it was conducted using smooth copper cones with dimensions of 60 mm in height, 36 mm in bottom diameters and 60 mm in top diameters. The mixed A liquid was injected into the truncated cone die, and the truncated cone die was lifted to determine the maximum diameter of the slurry flowing freely on the glass plane (Fig. 2b). The rheological characteristics were tested with a VISCO-RM100 from LAMY RHEOLOGY (Fig. 2c). The slurry was mixed evenly and then moved to the test container. The shear stresses at speeds of 5, 20, 30 and 50 rpm were tested in 30 seconds, and then plastic viscosity and yield stress were obtained through data analysis.

The compressive strength test was conducted according to ASTM Standard C942-15. Strength at 3, 7, 14, 28 and 56 d was tested to determine the development of compressive strength.

The period was 28 d for the penetration test according to the industrial standard (JGJ/T 70-2009) of China.

The shrinkage test was performed after demolding according to ASTM C157. The lengths (*Li*) at 1, 2, 3, 7, 14, 28, 35, 42, 49, 56 and 63 d were measured to determine the development of shrinkage with the length change rate (LCR) using the standard prescribed formula: LCR = (the initial lengths- *Li*)/250. However, the curing method of the specimen was changed to water curing since the CS grouts for GSWC in projects were always immersed in water and seldom suffered drying-wetting cycles. Additionally, specimens with a high W/B ratio, as shown in Fig. 1, were easily cracked and unable to mold in the air. Hence, the volume stability of the CS grouts was performed through autogenous shrinkage instead of natural drying shrinkage in this paper, which had been previously used in the literature [6]. Finally, the microstructures of the 28 d hardened CS grouts were investigated using SEM (Thermo Fisher 250 from FEI). The appearance of crystals was examined under a magnification of 2000 times.

Results and discussion

Rheological properties

The rheological properties of cement slurry with and without polymers with a W/B ratio of 0.8 can be approximated by the Bingham model [21], which can be characterized by yield stress and plastic viscosity. It can be seen from Fig. 3 that when the content of RLP is increased from 3% to 15%, the yield stress and plasticity viscosity of the slurry show an increasing trend, the yield stress increases by 79~319%, and the growth amplitude of the plastic viscosity reaches 29~175%. RLP has certain water-reducing and air-entraining effects [16, 18]. When the mixing amount of RLP is less, the introduction of a proper amount of bubbles and the formation of the emulsion by dissolving RLP during the mixing of slurry will result in the "rolling ball" and lubricating effect, which will increase the fluidity of the slurry. However, the introduction of more than a certain amount of bubbles will reduce the fluidity of the slurry. RLP will absorb more free water with the increase of the mixing amount, which will lead to the reduction of the fluidity and the increase of the cohesion of the slurry. Therefore, the yield stress and plastic viscosity tend to increase with increasing dosage of RLP. This also explains why the grout fluidity first increases and then decreases.

When the amount of HPMC increases from 1‰ to 3‰, the slope of the yield stress and plastic viscosity curves increases more obviously compared with the effect of RLP. The increase amplitude of yield stress reaches 259~449%, and the increase amplitude of plasticity viscosity reaches 77~141%. This is mainly because the hydroxyl group and the oxygen atom on the ether bond of the HPMC molecule form the hydrogen bond with the water molecule, which will lead to the reduction of the free water in the slurry. The polymer's long chain will adsorb and fix more water with the increase of the mixing amount, which leads to the weakening of the lubrication effect of the cement particles and the increase of the frictional force. Therefore, the yield stress and plastic viscosity of the slurry increase, and the fluidity of the slurry decreases.

Setting time

As shown in Fig. 4, the setting time of CS grouts greatly affects their diffusion distance in strata and the anti-dilution performance in moving water. When the amount of RLP increases from 3% to 15%, the initial and final setting time of the slurry show an increasing trend, but the change of initial setting time is smaller, and the final setting time is up to 44% longer than that of the blank group (CS-0). When the dosage of HPMC increases from 1‰ to 3‰, the initial setting time is 20~55% longer than that of the blank group, and the final setting time is prolonged 67~78%. Polymer particles form emulsions in water that increase the viscosity of the slurry, but this has a very weak effect on the reaction of SSS and calcium hydroxide (CH) formed by the hydration of cement. The polymer particles in the emulsion are adsorbed on the surface of hydration products and nonhydrated cement particles that hinder the contact between the SSS and CH produced by the hydration of cement, thus delaying the hydration of CS grouts. In addition, the high amount of polymer has a more serious effect on the setting time of the slurry.

Compressive strength

As shown in Fig. 5, the compressive strengths of CS grouts show a trend of increasing first and then decreasing with the increase of the mixing amount, which is similar to other results noted in the literature [19]. The peak value appears at the dosage of 3%. The compressive strengths increase by 12~18% from 7 d to 56 d before the peak compared with the blank group. Then, the compressive strengths show a decreasing trend. With the amount of RLP ranging from 6% to 15%, compressive strengths at 3 d reduce the range from 10% to 25%, compressive strengths at 7 d reduce the range from 6% to 28%, compressive strengths at 14 d reduce the range from 2% to 25%, compressive strengths at 28 d reduce the range from 6% to 29%, and compressive strengths at 56 d reduce the range from 8% to 26% compared with the blank group.

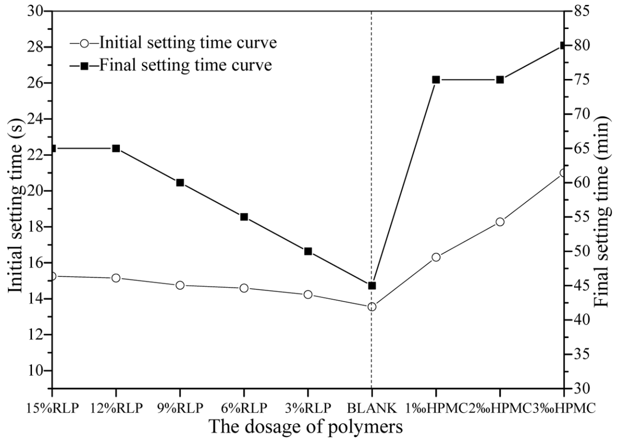


Fig. 4. The setting times of grouts.

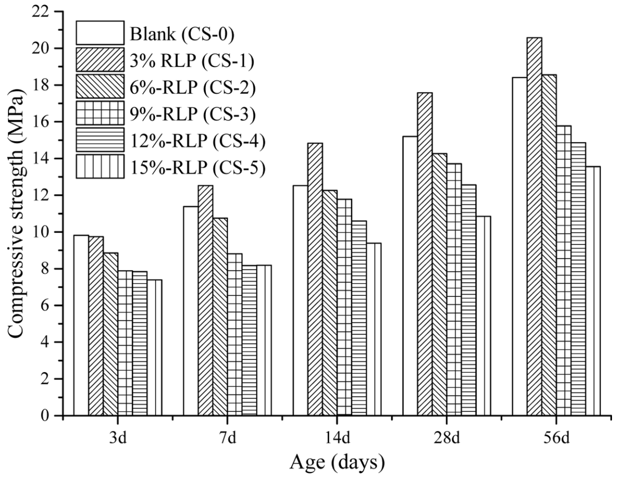


Fig. 5. Compressive strengths of specimens with different amounts of RLP.

The compressive strengths of CS grouts can be affected by the following factors. First, some RLP particles fill smaller pores, which will make the material more compact and improve the compressive strengths. Second, the polymer forms thin films between cement particles to increase the flexibility, which has a negative impact on the compressive strength. Finally, RLP introduces a large number of bubbles, which will reduce the strength. When RLP is mixed with a low amount (3%), the compressive strength is improved

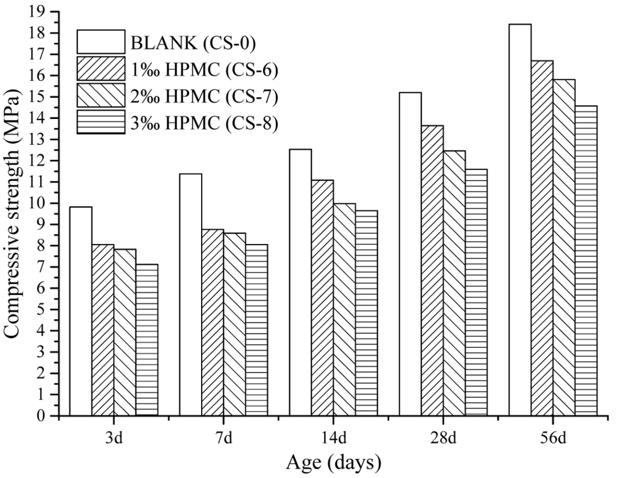


Fig. 6. Compressive strengths of specimens with different amounts of HPMC.

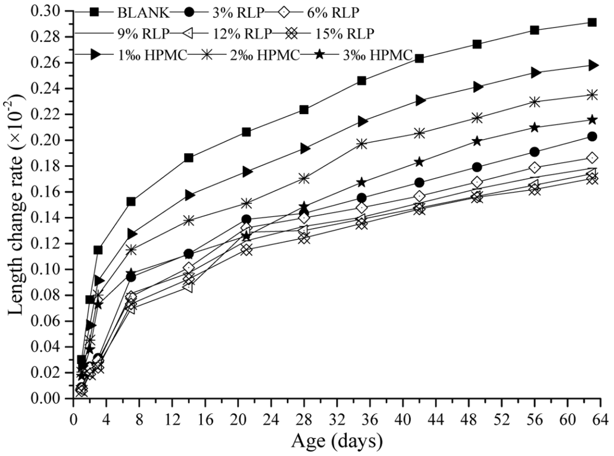


Fig. 7. The autogenous shrinkage characteristics of grouts.

because of the filling effect of RLP particles on smaller pores. With the increase of the mixing amount, the compressive strength decreases gradually due to the increasing air-entraining content and monolithic flexibility.

Fig. 6 shows the compressive strengths of specimens with different amounts of HPMC. The compressive strengths of the CS grouts decrease gradually with increasing dosage of HPMC. When the dosage of HPMC increased from 1‰ to 3‰, compressive strengths at 3 d reduced the range from 18% to 27%, compressive strengths at 7 d reduced the range from 23% to 29%, compressive strengths at 14 d reduced the range from 12% to 23%, compressive strengths at 28 d reduced the range from 10% to 24%, and compressive strengths at 56 d reduced the range from 9% to 21% compared with the blank group. The influencing factors of HPMC are similar to those of RLP on the compressive strength. However, the molecular structure of HPMC has a large number of hydrophilic groups (hydroxyl ether group) and hydrophobic groups (methyl glucose ring) [22]. The effect of air entrainment is stronger than that of RLP, which leads to an increase in air content in the concretion body and a decrease in mechanical properties.

Autogenous shrinkage

Shrinkage is an important parameter affecting the long-term durability of grouting. The shrinkage causes cracking of the concretion body, and the connecting cracks will produce seepage channels. Therefore, the development of autogenous shrinkage at 63 d was studied (Fig. 7). Approximately half of the shrinkage in the blank group occurred in the first week, which was similar to that observed for CS grouts with W/B ratios in the range of 1.1–2.5 and the dosage of SSS in the range of 4.4–7.5 wt% [14]. The addition of RLP significantly reduces the shrinkage of CS grouts, but the increase in amplitude of the shrinkage becomes small, which can be seen from the fact that the slope of the curve becomes flatter. With the increase of RLP, LCR at 63 d decreases by 0.883–1.21% compared with the blank group. Although the mixing of HPMC also reduces the shrinkage of CS grouts, the magnitude of the reduction is smaller than that of RLP. When the dosage of HPMC increases from 1‰ to 3‰, the LCR of CS grouts shows a decreasing trend, and the LCR at 63 d decreases by 0.332–0.754% compared with CS-0.

The addition of the two polymers reduces the number of large cracks, which was verified in microstructure analysis. This is mainly because the tiny pores introduced by the polymer are uniformly distributed in the slurry, which can reduce the stress concentration inside the concretion body. The polymer film formed on the surface of cement particles and hydration products improves the bonding strength between particles, which can also effectively absorb the energy required by microcrack growth because of its relatively low elastic modulus and high deformation capacity [23-25], thus significantly improving the crack resistance and shrinkage performance of CS grouts.

Sulfate attack resistance

Sulfate erosion has become an important factor affecting the durability of cement-based materials. Erosion of cement-based materials by external sulfates may lead to cracking, breaking and spalling because sodium sulfate reacts with calcium hydroxide to generate newly expanded ettringite and gypsum [26]. The concretion body of CS grouts is easily eroded by sulfate ions in groundwater. The resistance to sulfate, which relates to the durability of CS grouts, is evaluated by measuring the flexural strength after sulfate attack. Two groups of samples were cured in water and 3 wt% sodium sulfate solution. The influences of polymer and sodium sulfate on the flexural strength of CS grouts at 28 d were tested and analyzed. During the test, no cracks were found on the surfaces of all samples.

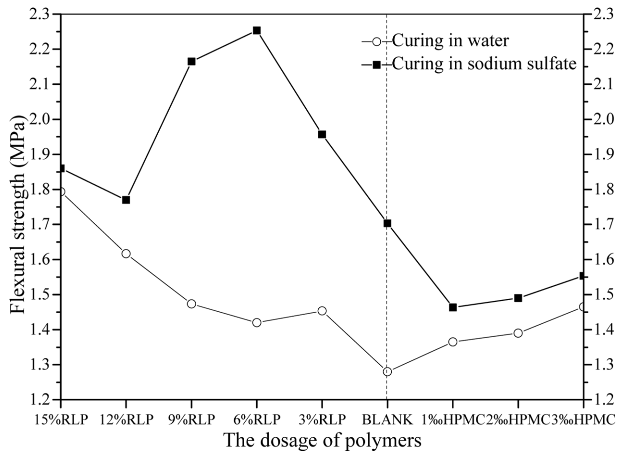


Fig. 8. The 28 d flexural strength after curing in water and sodium sulfate with 3 wt%.

It can be seen from Fig. 8 that when the sample is cured in water, the flexural strength of samples mixed with RLP and HPMC is higher than that of the blank group. In addition, the flexural strength shows an increasing trend with increasing RLP, and the increase range is from 11% to 40%. The effect of HPMC on the flexural strength is significantly weaker than that of RLP, which only increases by 7~14%. In sodium sulfate solution, RLP also increases the flexural strength, but the flexural strength first increases and then decreases with increasing RLP. Moreover, the incorporation of HPMC reduces the flexural strength compared with the blank group. It should be noted that sodium sulfate does not have a negative effect on the flexural strength through the comparison of the test results of the two curing methods. The main reason is that there are a large number of pores in the concretion body because of the high W/B ratio of CS grouts. Although sodium sulfate reacts with calcium hydroxide and calcium aluminate to generate newly expanded ettringite, its volume expansion does not cause the volumetric instability, but fills and compacts the pores which will make the concretion body denser and improve the flexural strength.

In brief, adding a proper amount of RLP can significantly improve the flexural strength and sulfate resistance of CS grouts. However, CS grouts mixed with HPMC are not suitable for the formation with high sulfate content.

Permeability

The permeability of CS grouts is the most basic factor that determines their durability under a certain underground water pressure. The leakage of underground water through grouts will seriously affect the quality of the grouting reinforcement area. Therefore, the permeability of CS grouts at 28 d was evaluated by testing the ultimate hydraulic pressure before leakage or failure.

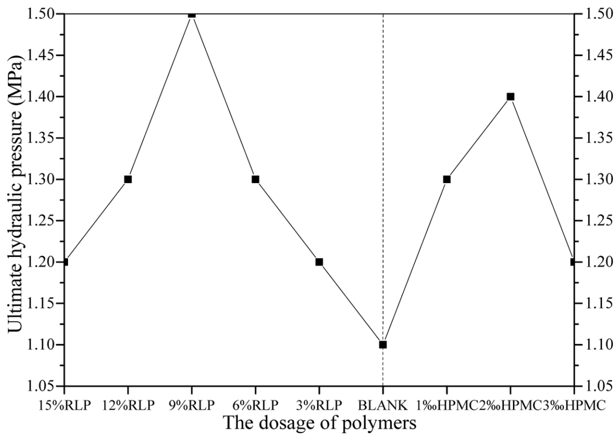


Fig. 9. The ultimate hydraulic pressure of the grouts at 28 d.

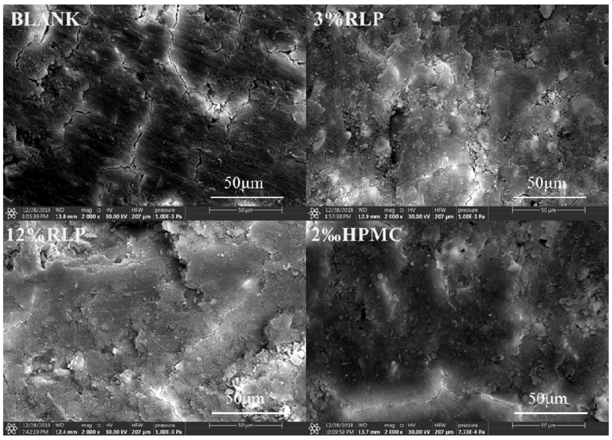


Fig. 10. Micrographs of specimens under a magnification of 2000 times.

As shown in Fig. 9, the ultimate hydraulic pressure of CS grouts doped with polymer increases by 0.1–0.4 MPa, significantly improving the permeability. The optimal dosages of RLP and HPMC are 9% and 2‰, respectively. In the concretion body, the film formed by the polymer is distributed among the cement hydrates to form a network and effectively fill the pores. These two actions prevent the connection of pore channels and thus improve the compactness of CS grouts.

Microstructure analysis

The fresh uncarbonized concretion body was observed to obtain the microstructure of the sample section. The microstructure of CS-0 (BLANK), CS-1 (3% RLP), CS-4 (12% RLP) and CS-7 (2‰ HPMC) at 28 d, which was imaged at 2000-fold magnification by SEM, is shown in Fig. 10.

The adhesion between particles is improved because the addition of polymer significantly improves the roughness of the fracture surface compared with the blank group, which can be used to explain why the flexural strength is increased to some extent. The addition of RLP and HPMC significantly reduces the number and width of cracks compared with the blank group in Fig. 11, which also explains why the autogenous shrinkage is improved.

Conclusion

This paper mainly studies the influence of RLP and HPMC on the physical and mechanical properties of CS grouts with a high W/B ratio. With the variation of mixing amounts of polymers, the rheological properties, setting time, compressive strength, autogenous shrinkage, sulfate attack resistance and permeability are tested and assessed. The main conclusions are summarized as follows:

(1) The yield stress and plastic viscosity of the cement slurry incorporating RLP and HPMC increased significantly, which would be beneficial for improving the resistance to dispersion in water.

(2) The effect of HPMC on CS grout setting time was better than that of RLP. The initial setting time was prolonged by 20~55%, and the final setting time was prolonged by 67~78%. These time could provide references for grout material selection in the grouting project.

(3) The reduction of autogenous shrinkage was more significant when RLP was incorporated into CS grouts. The addition of the two polymers significantly inhibited the formation of new cracks.

(4) Although two polymers had a negative impact on the compressive strength of CS grouts except for the RLP content of 3%, they increased the flexural strength. CS grouts with the proper amount of RLP had a superior sulfate attack resistance when exposed to sulfate-rich environments.

(5) The ultimate hydraulic pressure increased 0.1–0.4 MPa when RLP and HPMC were incorporated into CS grouts. The durability under a certain underground water pressure was improved.

Acknowledgments

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