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Effects of Superplasticizer on the Hydration, Consistency, and Strength Development of Cemented Paste Backfill

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Abstract: The strength and consistency of cemented paste backfill (CPB) are of key concerns in the stope stability and cost control for underground mines. It is common practice to use additives, such as superplasticizer, to improve the performance of CPB. This study mainly focuses on the effects of superplasticizer on the hydration, consistency, and strength of CPB. In this study, a polynaphtalene sulfonate was used as the superplasticizer. The binder is a mix of 33.3% ordinary Portland cement and 66.7% fly ash. The CPB specimens with a tailings-binder ratio of 3:1 and a solid concentration of 70% were then tested by a low field nuclear magnetic resonance system after different hydration times. Effects of polynaphtalene sulfonate on the hydration, fluidity, and strength were investigated. Results showed that the polynaphtalene sulfonate has a strong influence on short-duration hydration, which may contribute to the strength increase of CPB. It has been demonstrated that the polynaphtalene sulfonate improved the fluidity of the CPB mixture. With the increased dosage of polynaphtalene sulfonate, the slump increased. It was also found that the polynaphtalene sulfonate dosage has a negligible effect on the 1 day (d) strength while it has a strengthening effect on the 7 d, 14 d, and 28 d strength of CPB specimens.

Keywords: cemented paste backfill; superplasticizer; full tailings; hydration; nuclear magnetic resonance; fluidity; strength

1. Introduction

Large amounts of mining tailings are deposited after useful minerals have been extracted by the ore processing plant. In China, 1.2 billion tons of mining tailings are deposited every year. These solid wastes do great harm to the environment, therefore, the mining tailings should be managed properly to protect the environment [1]. Some scholars suggested that one of the most valuable ways to re-use tailings is to generate cemented paste backfill (CPB) materials in underground mines [2]. In practical engineering, CPB is the main method to fill the underground void spaces and support the open stopes. CPB has been widely employed in many mines around the world [3,4].

CPB material is often made by an engineered mixture of binder, tailings, and water. Ordinary Portland Cement (OPC) is often used as the main binder. However, in some cases, fly ash or blast furnace slag substitute part of the OPC to reduce the costs and improve the performance [5–7]. It has been demonstrated that the addition of fly ash increases the amount of calcium silica hydrates (C–H–S), which may contribute to the strength development of the CPB mixture [8].

Cement-mixed materials are subjected to loading conditions [9,10]. The two most important properties of CPB are its strength and consistency. With high strength, the CPB can support the open stopes more effectively and, with good fluidity, the transportation of the CPB mixture will be more cost-effective. Factors that affect the properties of CPB, such as chemical composition and particle size distribution of the tailings, types of binder, water to cement ratio, and temperature, have been presented in many studies [11–17]. The strength and fluidity of CPB materials are also investigated by several scholars [18–23]. In some cases, superplasticizer is added to the CPB mixture to improve its mechanical properties and consistency [24–33]. It was found that the addition of superplasticizers increases the early compressive strength of CPB [34]. In addition, the strength of CPB varies depending on the type and dosage of the superplasticizer [35]. However, few studies focus on the microscopic mechanism of fluidity and strength evolution under the effect of superplasticizers.

On the other hand, the strength and consistency of CPB are strongly affected by the hydration of the binder [36]. The hydration process of cement-water pastes can be divided into four stages, i.e., early reactions (0–30 min), induction or dormant period (1–3 h), acceleration period (3–17 h), and slow reaction period [37]. The hydration of cement in the concrete/mortar mixtures has also been presented in other studies. However, few studies focus on the hydration in CPB mixtures.

This paper presents the effects of superplasticizer on the hydration, consistency, and strength of CPB. To do so, a mix of OPC and fly ash was used as the binder. Polynaphtalene sulfonate was used as the superplasticizer. Other materials include mine tailings and water that is discharged from the ore concentrator. The CPB mixture with a tailings-binder ratio of 3:1, a solid concentration of 70%, and different dosages of polynaphtalene sulfonate was placed into the low field nuclear magnetic resonance (NMR) system to measure the water content evolution and analyse the hydration of CPB. The fluidity and strength evolution of CPB containing the different dosages of polynaphtalene sulfonate was also presented. Finally, the physicochemical mechanism of the polynaphtalene sulfonate on the fluidity and strength evolution of CPB was investigated.

2. Materials and Methods

In this study, full mining tailings from the Shuiyindong gold mine located in the southeast of Guizhou Province of China were selected as the tailings material. OPC is used as the main binder. The average price of OPC is CNY350/t. Fly ash, which was generated by the power plant of the mine, is also selected to substitute part of the cement to decrease the backfilling costs. Mine water and polynaphtalene sulfonate were also added to the tailings material.

2.1. Tailings

Many researchers observed the important effects of physical and chemical properties of tailings on the strength and fluidity. Particle size and chemical composition were the most influential parameters affecting the properties of CPB. In this study, the particle size is measured by the sieve analysis method (SAM). The results of the particle size distribution are shown in Table 1 and Figure 1. It may be seen that particles with a size less than 0.002 mm comprise 21.4% and particles with a size less than 0.05 mm comprise 84.0%. The parameter d_{50}, which means the median particle size, defined by the particle size when the accumulative percentage reaches 50%, can be calculated from Figure 1. By Figure 1, d_{50} is 0.012.

Table 1. Particle size distribution of selected tailings.

Method	Particle Size (mm)	2–0.5	0.5–0.25	0.25–0.075	0.075–0.05	0.05–0.005	0.005–0.002	<0.002
SAM	Mass Fraction (%)	0.4	0.1	5.5	9.0	50.9	12.7	21.4

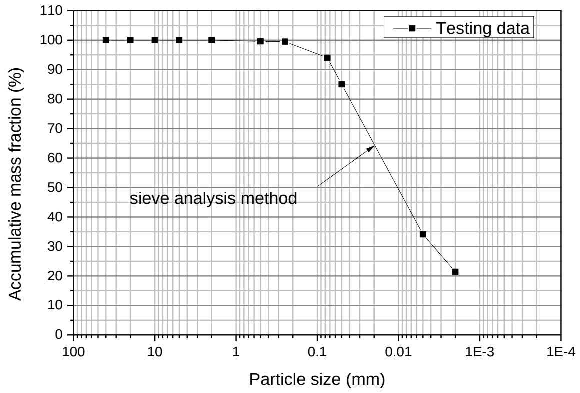


Figure 1. Particle size distribution obtained by the sieve analysis method.

Chemical Composition Analysis Centre at Central South University also tested the chemical composition, and the results are shown in Table 2. Table 2 demonstrates that the content of SiO_2 and Al_2O_3 is 35.42% and 14.65%, respectively. The low content of cementitious oxides is not favourable for CPB strength gain. Therefore, in this study, fly ash with a high content of SiO_2 and Al_2O_3 is added to the CPB mixture.

Table 2. The chemical composition of full tailings.

Chemical Composition	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	Others
Mass percentage (%)	35.42	18.53	6.27	14.65	6.61	18.52

2.2. Binders

The most commonly used binder in CPB is OPC. The cost of the binder is nearly 50–75% of the total costs of a typical CPB plant [37,38]. A substitution binder can reduce the cost of backfilling significantly. As the main solid waste of power plants, fly ash has potential cementitious properties due to the high content of SiO_2, Al_2O_3, and other kinds of oxides. On the other hand, the replacement of part of the cement by fly ash in the CPB mixtures is often seen as an environmental value-added benefit. Moreover, the use of fly ash in CPB can significantly reduce the total costs of a paste backfill plant due to its lower cost [13]. As shown in Table 1, the fine tailings (<20 µm) comprise 21.4%. Fall and Benzaazoua [11] suggested that a high content of fine tailings (<20 µm) is not favourable for CPB strength gain. Therefore, in this paper, a relatively high binder content is adopted. In order to improve the particle size distribution of CPB mixture, as well as to reduce the backfill costs, a mix of 66.7% of fly ash and 33.3% OPC is used as the binder. The chemical compositions of OPC and fly ash are given in Table 3.

Table 3. Chemical compositions of OPC and fly ash.

	Chemical Composition	SiO_2	CaO	Fe_2O_3	Al_2O_3	K_2O	Others
OPC	Mass percentage (%)	50.61	5.35	7.22	31.33	0.34	5.15
Fly ash	Mass percentage (%)	23.20	57.59	3.87	7.25	0.68	7.41

2.3. Superplasticizer and Water

In the cement and concrete industry, the mechanical strength of cemented pastes can be improved by reducing the moisture content of the paste, while maintaining the fluidity of the paste by adding a superplasticizer [39]. In this study, a polynaphtalene sulfonate is used as a superplasticizer. The dosages of additive superplasticizer are 0.2%, 0.4%, and 0.8% of the binder by weight based on the backfill practice of this mine. The water discharged from the ore concentrator is used to make the CPB mixtures.

2.4. Specimen Preparation

In this study, the tailing-binder ratio is 3:1, and the solid concentration of CPB is 70%. All the specimens were prepared by a standard cubical mould (i.e., 7.07 × 7.07 × 7.07 cm^3) suggested by the Ministry of Construction of the PRC [40]. The specimens were then cured in the curing box, being wrapped after a 6 h knockout time. The curing temperature was kept at 20 °C, and the humidity at 95%. Curing times for strength test specimens were 1 day (d), 7 d, 14 d, and 28 d, respectively. For each curing time, three specimens were prepared.

2.5. Nuclear Magnetic Resonance Tests

Previous studies demonstrated that NMR technology is suitable for evaluating the hydration of cement [41–43]. In this study, a low-field NMR system (see Figure 2a) was used to evaluate the hydration process of the CPB specimens.

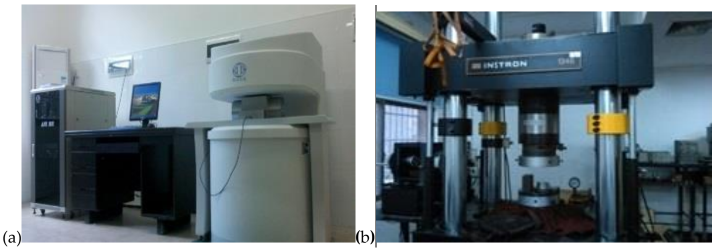


Figure 2. Test apparatus (**a**) low-field NMR system and (**b**) a uniaxial compressive strength testing machine.

For the short-duration tests, the CPB mixture was placed into a plastic cylinder tube and put into the NMR system to measure the T_2 relaxation time at 10 min, 1 h, 2 h, and 8 h after mixing. For the long-duration tests, after 1 d, 7 d, 14 d, and 28 d of curing the CPB specimens were tested by the NMR system.

Before the test, the T_2 relaxation time of pure water was measured and presented in Figure 3. The figure shows that the T_2 relaxation time of pure water is about 800–1600 ms.

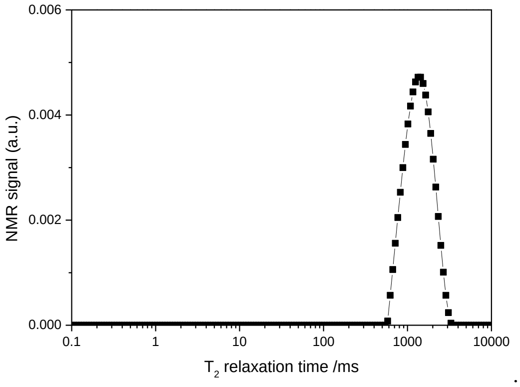


Figure 3. T_2 relaxation time of pure water.

2.6. Unconfined Compressive Tests

After the NMR tests, all the specimens cured for the strength test were placed in a servohydraulic material test system (see Figure 2b) after 1 d, 7 d, 14 d, and 28 d. The force-displacement curve during loading was recorded automatically by the computer. The specimens were loaded by the displacement control method with a loading rate of 0.01 mm/s. Then the uniaxial compressive strength can be calculated. The capacity of the testing machine is 200 KN and the accuracy is 0.5%.

3. Results and Discussion

3.1. Effects of Polynaphtalene Sulfonate on the Fluidity

Slump tests were undertaken on CPB mixtures to investigate the effects of polynaphtalene sulfonate on the consistency. Figure 4 shows the sample with different dosage of polynaphtalene sulfonate in a slump test. The average slump of CPB mixtures with different polynaphtalene sulfonate dosages is given in Table 4. As shown in Figure 4 and Table 4, the addition of polynaphtalene sulfonate improves the fluidity of CPB significantly. Similar to previous studies, it can be concluded that the superplasticizer has a positive effect on the fluidity of CPB [27,29]. The fluidity increased with an increase in the polynaphtalene sulfonate dosage.

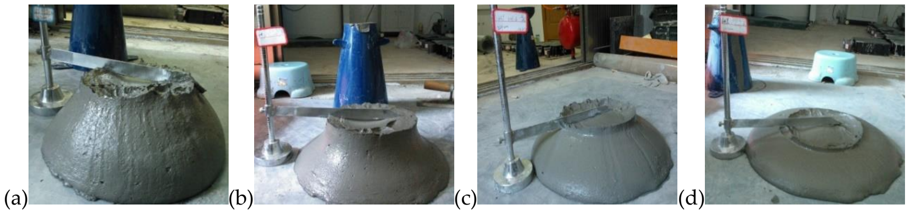


Figure 4. Slump tests of CPB mixture with different polynaphtalene sulfonate dosage: (**a**) 0, (**b**) 0.2%, (**c**) 0.4%, and (**d**) 0.8%.

Table 4. The average slump of CPB with different dosages of polynaphtalene sulfonate.

Polynaphtalene Sulfonate (%)	**0**	**0.2**	**0.4**	**0.8**
Slump (cm)	16.5	20.5	22	24.5

3.2. Effects of Polynaphtalene Sulfonate on the Strength

The correlation between the strength of CPB and dosage of polynaphtalene sulfonate is shown in Figure 5. It shows that the strength of CPB increases with the increase of curing time. Additionally, with the increased dosage of polynaphtalene sulfonate, the 1 d strength remained nearly constant. However, the 7 d, 14 d, and 28 d strengths increase rapidly with an increase in polynaphtalene sulfonate dosage. This means that the polynaphtalene sulfonate has a negligible effect on the 1 d strength of CPB, while it has a positive effect on the 7 d, 14 d, and 28 d strength gain.

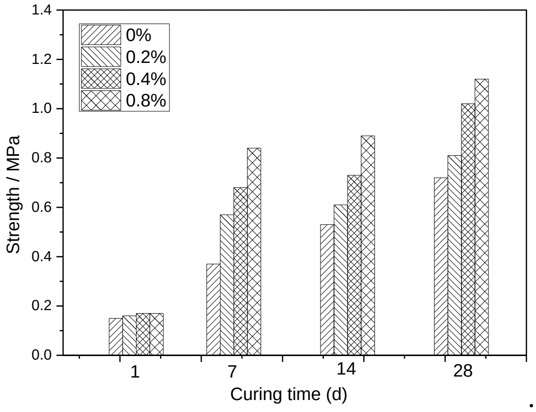


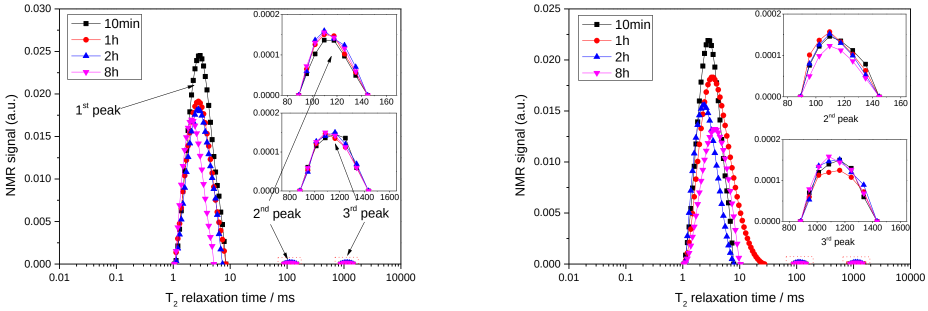
Figure 5. Strength evolution of CPB specimens with different polynaphtalene sulfonate dosages.

3.3. Effects of Polynaphtalene Sulfonate on the Hydration

In CPB mixtures there are four different water phase states, i.e., chemically-bound water, which corresponds to the water that is combined with chemical compounds with hydrogen bonds, bonded water in the gel phase, which corresponds to the water that is enveloped by the C–H–S structure, pore water, which corresponds to the water physically bound between particles, and free water. According to the theory of NMR, different water phase states have different T_2 relaxation times. For example, the T_2 relaxation time of chemically-bound water in the cement mixture is about 12 μs [43] which is difficult to obtain by NMR [44,45]. Therefore, in this study, only three water phase states (i.e., bonded water in the gel phase, pore water, and free water) were detected by the NMR system.

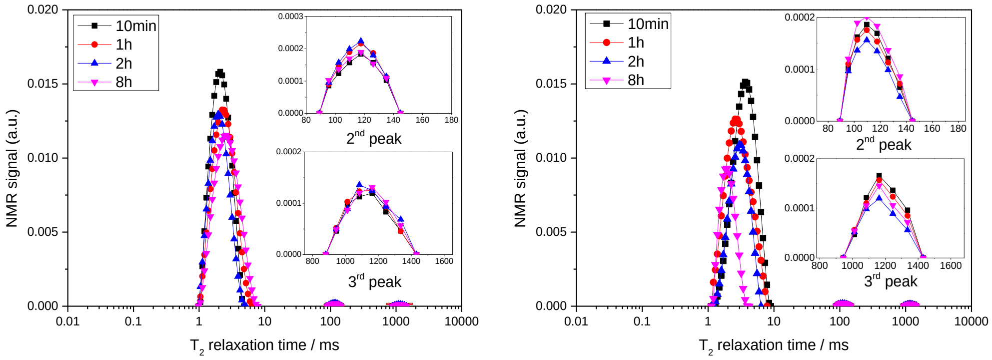
The T_2 relaxation time of short-duration (10 min–8 h) hydration of CPB mixture is shown in Figures 6 and 7. It can be seen that the T_2 relaxation time curve has three peaks, indicating three main water phase states. The three peaks, which are shown in Figure 6a, are named here from left to right as the 1st peak, the 2nd peak, and the 3rd peak. It demonstrated that the centre of the 1st peak is 1 ms–10 ms, while the centre of the 2nd and the 3rd peaks are about 100 ms and 1000 ms, respectively. Thus, it can be judged that the 1st peak is the signal of bonded water in the gel phase, while the 2nd and the 3rd peaks are the signals of pore water and free water, respectively. Figure 6 demonstrated that the peak value of the 1st peak decreased with an increase in hydration time, which means that the amount of the bonded water in the gel phase decreased. The decrease in bonded water in the gel phase is mainly due to the reaction with the binder, leading to the decreasing of a detectable water molecule. Additionally, it can be seen that the 2nd and the 3rd peaks do not change much, identifying that the amount of pore water and free water changes little during the short-duration hydration. In addition, it is implied that during the hydration time of 10 min to 1 h, the peak value of the 1st peak decreases more than during 1 h–8 h. This is mainly because during the hydration process of CPB mixture the reaction between water and binder is stronger in the first 30 min [22].

As shown in Figure 7, an increase in the dosage of polynaphtalene sulfonate leads to the decrease in the peak value of the 1st peak, indicating that the bonded water in the gel phase decreased. However, the 2nd and the 3rd peaks showed almost no changes, which means that the amount of pore water and free water has little change with an increased dosage of polynaphtalene sulfonate. It was also observed that, during hydration times of 10 min to 1 h, when the dosage of polynaphtalene sulfonate increased from 0% to 0.2%, the peak value of the 1st peak slightly decreased. However, when the dosage of polynaphtalene sulfonate increased from 0.2% to 0.4%, the peak value of the 1st peak decreased significantly. On the other hand, during the hydration time of 2 h to 8 h, the peak value of the 1st peak decreased in a nearly uniform manner. This result demonstrated that the addition of 0.4% polynaphtalene sulfonate promoted the hydration of this CPB material during the 10 min to 1 h hydration time, which may contribute to the strength increase. Furthermore, the addition of polynaphtalene sulfonate has a stronger influence on the hydration of this CPB material during the hydration time of 10 min to 1 h than from 2 h to 8 h.



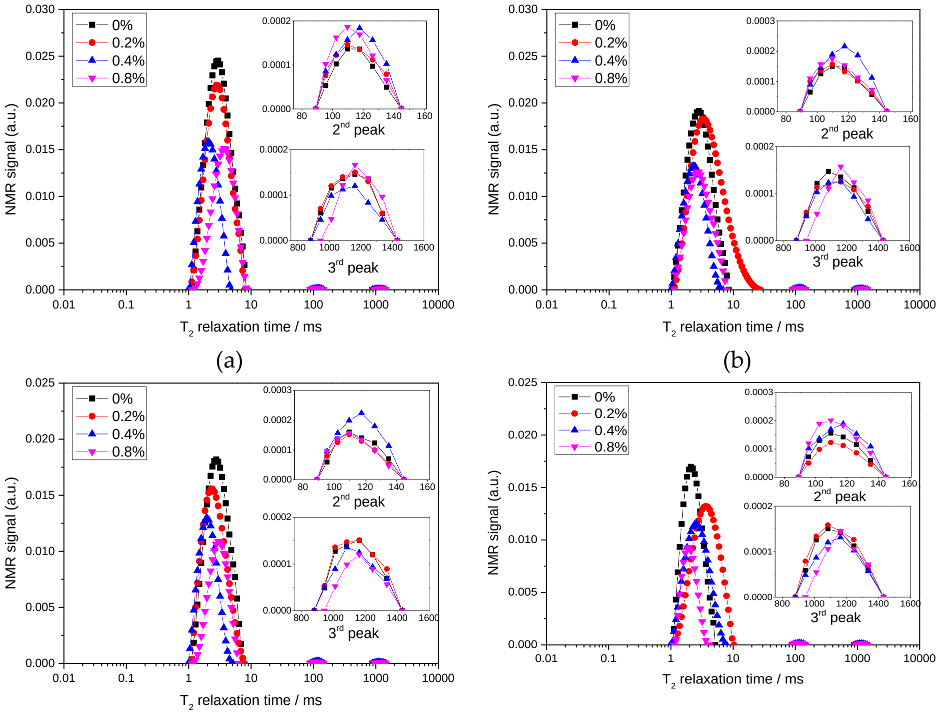
(a) (b)

Figure 6. *Cont.*



(c) (d)

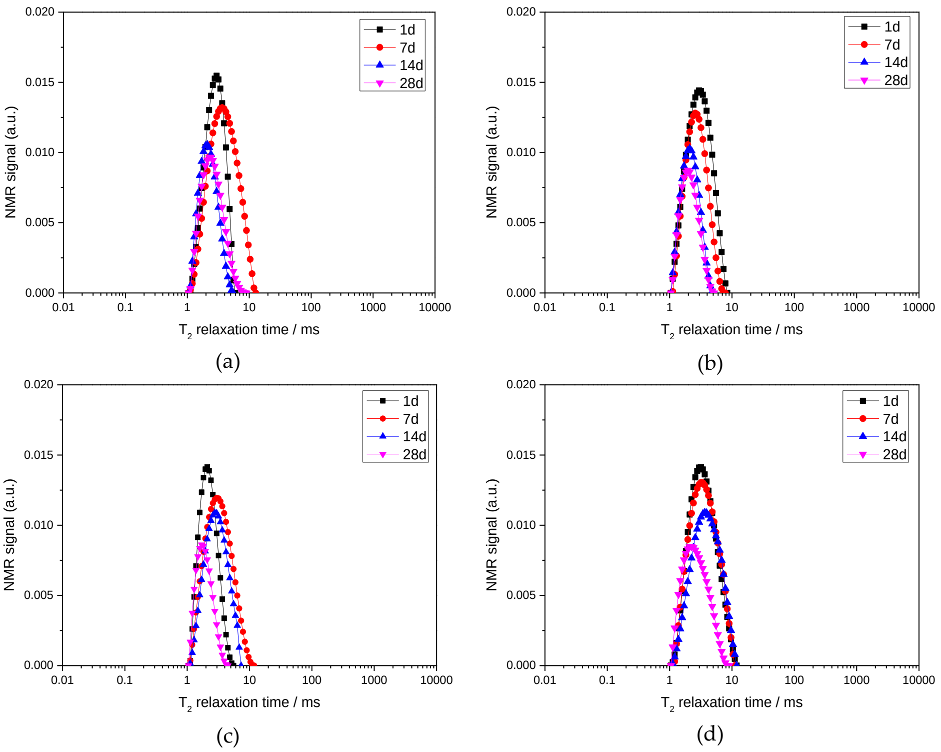
Figure 6. The short-duration T_2 relaxation time of the CPB mixture with different hydration times when polynaphtalene sulfonate dosage is: (**a**) 0%, (**b**) 0.2%, (**c**) 0.4%, and (**d**) 0.8%.



(c) (d)

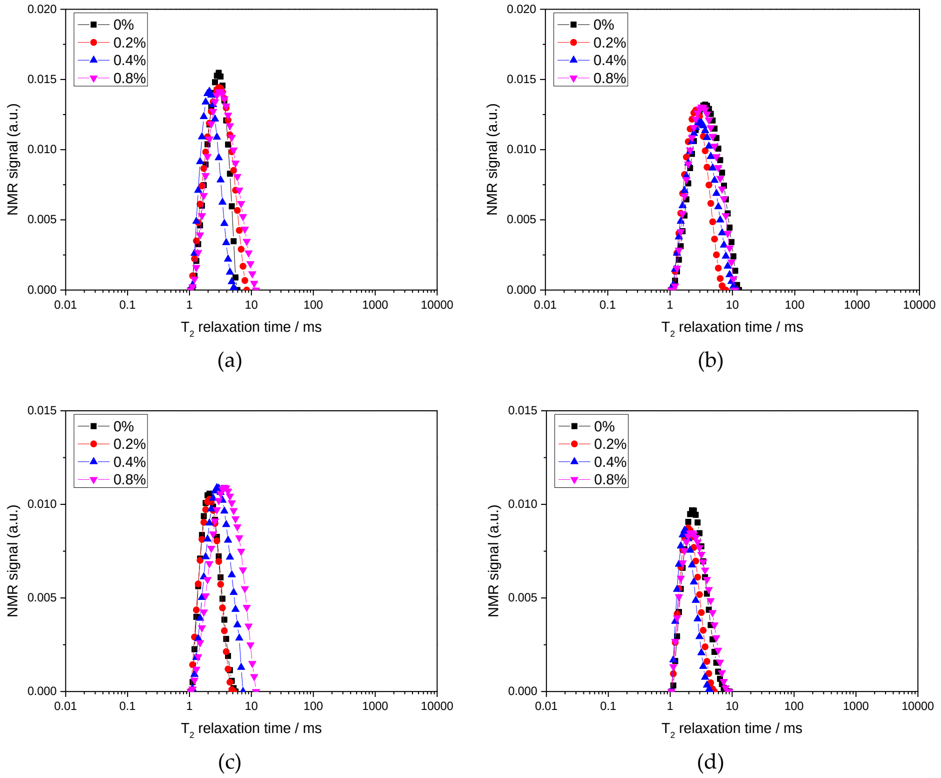
Figure 7. The short-duration T_2 relaxation time of CPB mixture with different polynaphtalene sulfonate dosages when the hydration time is: (**a**) 10 min, (**b**) 1 h, (**c**) 2 h, and (**d**) 8 h.

The T_2 relaxation time of long-duration (1 d–28 d) hydration is shown in Figures 8 and 9. Figure 8 presents that, similar to Figure 6, the peak value of the 1st peak decreased with an increase in curing time, which demonstrated that the amount of bonded water in the gel phase decreased. In addition, there is only one peak of the long-duration T_2 relaxation time curve, identifying that during the hydration time 1 d–28 d, there is almost no pore water and free water in the CPB specimens. On the other hand, as presented in Figure 9, during long-duration hydration, the peak values of the 1st peak showed no conclusive trend with the increased dosage of polynaphtalene sulfonate. This result further demonstrated that the polynaphtalene sulfonate has a stronger influence on the short-duration hydration than the long-duration hydration. This has been explained in the next part.



(a) (b) (c) (d)

Figure 8. The long-duration T_2 relaxation time of CPB mixture with different hydration time when the polynaphtalene sulfonate dosage is: (**a**) 0, (**b**) 0.2%, (**c**) 0.4%, and (**d**) 0.8%.

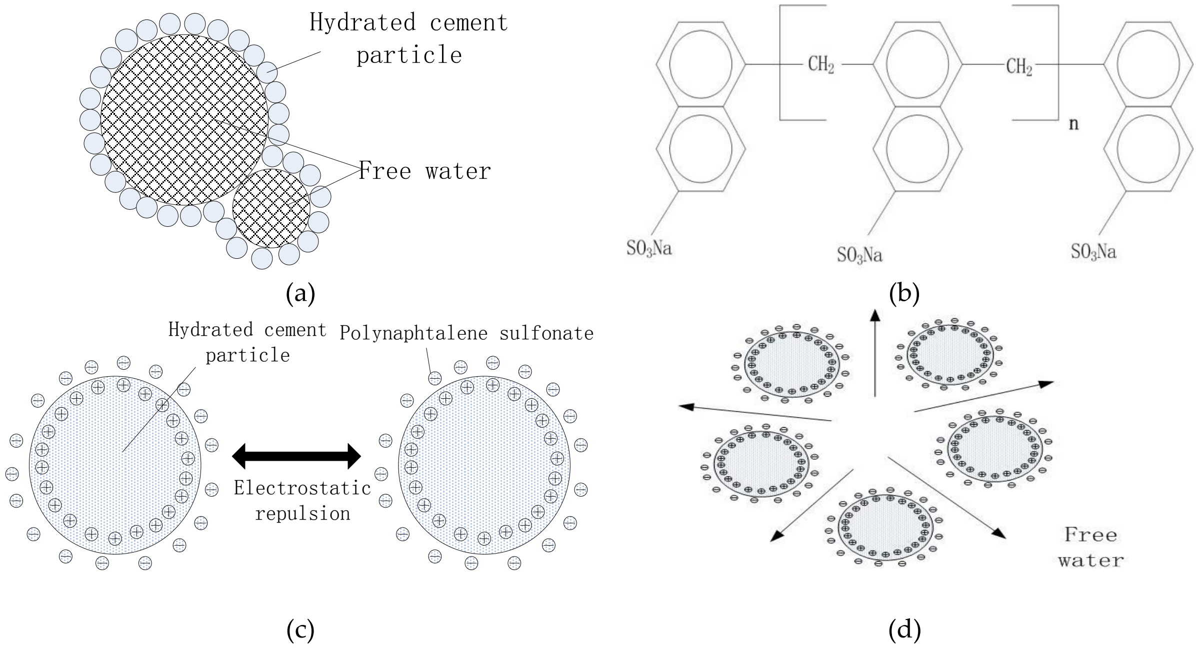


(a) (b) (c) (d)

Figure 9. The long-duration T_2 relaxation time of CPB mixture with different polynaphtalene sulfonate dosages when the hydration time is: (**a**) 1 d, (**b**) 7 d, (**c**) 14 d, and (**d**) 28 d.

4. Physico-Chemical Mechanism of Superplasticizer in the Mixture

When the CPB materials were mixed with water, the cement particles envelop the water molecules to form a flocculent structure, leading to the free water content decreasing. Thus, the fluidity of the CPB mixture decreased. However, when polynaphtalene sulfonate was added to the mixture, the fluidity increased. The chemical mechanism is demonstrated in Figure 10. As shown in Figure 10a, without using any additive, free water is surrounded by cement particles. By adding polynaphtalene sulfonate to the mixture, the radical group $-SO_3Na$ (see Figure 10b) in polynaphtalene sulfonate hydrolysed into $-SO_3^-$, which is an ion with a negative charge. $-SO_3^-$ ions then were attracted by the cement particles due to the attractive force between negative and positive charges. Thus, the cement particles were surrounded with $-SO_3^-$, leading to the separation of cement particles due to the electrostatic repulsion (see Figure 10c). In this regard, the flocculent structure will be destroyed, resulting in the increase of fluidity. On the other hand, the free water enveloped by the flocculent structure will be released as a result of the destruction of the flocculent structure (see Figure 10d). The amount of water that contributes to the mixture fluidity increased. Therefore, with the increase of dosage of polynaphtalene sulfonate the fluidity of CPB increases. In addition, the water molecules attached by the cement particles formed hydrated films (see Figure 11a), which increased the lubrication between cement particles. This is another reason for the fluidity increase when polynaphtalene sulfonate was added to the mixture.



(a) (b)

(c) (d)

Figure 10. Physico-chemical mechanism of the effect of polynaphtalene sulfonate on the consistency of CPB: (**a**) flocculent structure, (**b**) chemical structure of polynaphtalene sulfonate, (**c**) electrostatic repulsion effect and (**d**) destruction of the flocculent structure.

Figure 11 demonstrates the strength gain under the effect of polynaphtalene sulfonate. It can be seen that, when polynaphtalene sulfonate was added to the mixture, more water molecules were attracted by the hydrophilic radical $-SO_3^-$ attached on the surface of cement particles (see Figure 11a), leading to the increase of the hydration extent of CPB. In this regard, the strength of CPB increased. On the other hand, as presented in Figure 11b, the addition of polynaphtalene sulfonate destroyed the flocculent structure, making the cement particles more dispersed. Thus, the cement particles can enter the void spaces between tailing particles more easily. Therefore, the polynaphtalene sulfonate reduces the pore diameter of the backfill [46] and increases the long-term strength.

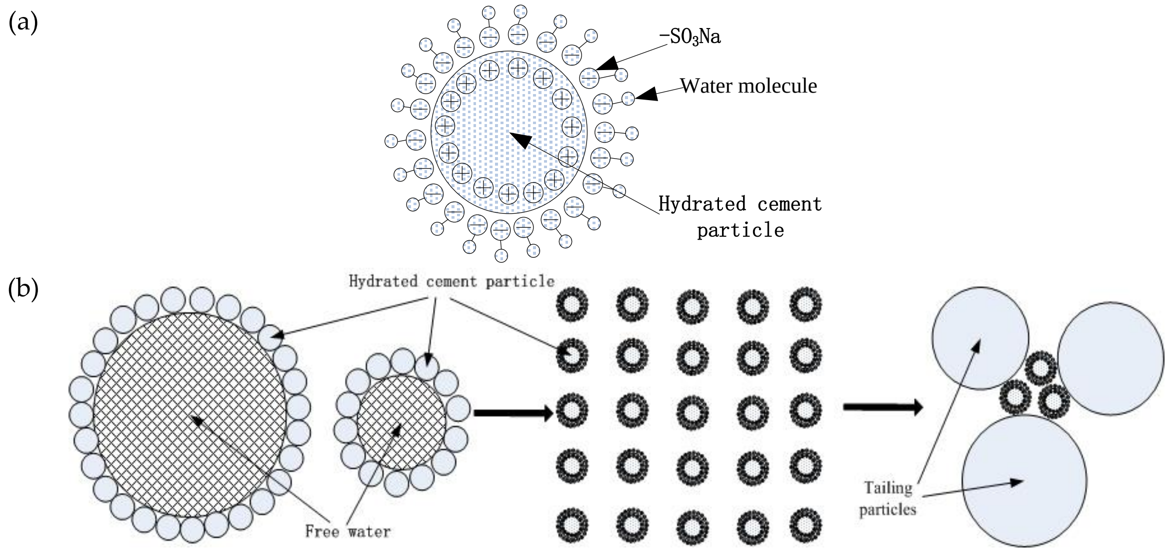


Figure 11. Physico-chemical mechanism of the effect of polynaphtalene sulfonate on the strength of CPB: (**a**) adsorption effect and (**b**) filling effect.

5. Conclusions

In order to investigate effects of superplasticizer on the hydration, consistency, and strength of cement paste backfill, a polynaphtalene sulfonate is used as a superplasticizer and mixed with ordinary Portland cement, fly ash, full tailings, and discharged water from the ore concentrator to generate CPB materials. An NMR system is adopted to investigate the hydration of CPB. The fluidity and strength evolution, as well as the physico-chemical mechanism of the effect of polynaphtalene sulfonate on the fluidity and strength, are also presented. The following conclusions can be drawn:

(a) The polynaphtalene sulfonate has a positive effect on the fluidity of CPB mixture. With an increase in the dosage of polynaphtalene sulfonate, the slump of CPB mixture increased.

(b) The superplasticizer has a negligible effect on the 1 d strength of CPB, while it increases the 7 d, 14 d, and 28 d strength of CPB significantly. With the increased dosage of superplasticizer, the strength also increased.

(c) During the short-duration hydration of CPB, the amount of bonded water in the gel phase decreases with the increase of the hydration time, while the amount of pore water and free water do not change significantly. However, during long-duration hydration, no pore water and free water were detected via the NMR test. It was also found that polynaphtalene sulfonate has a stronger effect on the short-duration hydration than the long-duration hydration. Due to the fact that the CPB properties are strongly affected by the hydration of the CPB mixture, more attention should be paid to the hydration of the CPB mixture with different admixtures. In addition, new methods should be developed to investigate the microscopic mechanism of CPB hydration.

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