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Research Article

Preparation and Performance of a New-Type Alkali-Free Liquid Accelerator for Shotcrete

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A new type of alkali-free liquid accelerator for shotcrete was prepared. Specifically, the setting time and strength and shrinkage performance of two kinds of Portland cement with the accelerator were fully investigated. Moreover, the accelerating mechanism of alkali-free liquid accelerator and the hydration process of the shotcrete with accelerator were explored. Results show that alkali-free liquid accelerator significantly shortened the setting time of cement paste, where the initial setting time of cement paste with 8 wt% of the accelerator was about 3 min and the final setting time was about 7 min. Compressive strength at 1 day of cement mortar with the accelerator could reach 23.4 MPa, which increased by 36.2% compared to the strength of cement mortar without the accelerator, and the retention rate of 28-day compressive strength reached 110%. In addition, the accelerator still shows a good accelerating effect under low temperature conditions. However, the shrinkage rate of the concrete increased with the amount of the accelerator. 5~8% content of accelerator is recommended for shotcrete in practice. XRD and SEM test results showed that the alkali-free liquid accelerator promoted the formation of ettringite crystals due to the increase of Al^{3+} and SO_4^{2-} concentration.

1. Introduction

Shotcrete has been widely used in tunnel, underground, and marine works for providing early support and preventing water seepage [1, 2]. Key performance indicators of the shotcrete are setting time and strength, which are decided by not only mixture design but also the use of the accelerator. The accelerator alters the hydration mechanisms of the cementitious material, influencing its strength development and setting time [3, 4]. Moreover, some authors reported that cement mortar added with the accelerator had same types of hydration product compared to cement mortar without the accelerator [5]. However, most existing accelerators are types of alkaline powdery accelerator which will lead to caustic harm and dust pollution and dramatically cut down the long-time strength and the inhomogeneity of concrete.

In order to solve such problems, alkali-free liquid accelerators with advantages of high efficiency, high strength, and environmental friendliness have gained more and more attention [6–9]. As early as the 1970s, the United States began to develop liquid accelerators and tried to change the accelerators from alkali-rich to alkali-free products. Sommer et al. [10] synthetized a type of alkali-free liquid accelerator containing 12% aluminum hydroxide, 0.5% complexing agent, 25% hydrofluoric acid, 7.5% amine, and 55% water, which offered the advantages of rapid increase in compressive strength compared with alkaline accelerators and reduced concrete crack due to the formation of ettringite. This accelerator can reduce the initial setting time and the final setting time of the cement paste to 6 minutes and 20 minutes, respectively; however it still has its limitation in the compatibility for different types of cement. Institutions in the United States and Europe successfully produced compound accelerators prepared with inorganic accelerators and two types of ethanol amine, nitro alcohol and acid glycol

Table 1: Properties of Portland cement.

Indexes	Standard requirement	Jidong cement	Qinling cement
Fineness/%	≤10.0	≤5.0	≤5.0
Initial setting time/min	≥45	195	205
Final setting time/min	≤390	238	250
Soundness/mm	≤5	0.8	1.2
3 d compressive strength/MPa	≥21	26.8	30.8
28 d compressive strength/MPa	≥42.5	45.3	50.8
3 d flexural strength/MPa	≥4.0	5.7	5.5
28 d flexural strength/MPa	≥6.5	7.9	8.8

derivatives, and the compound accelerators had no adverse effects on the 28-day strength but affected the early strength of the cement paste. Although early reports have shown encouraging results, some aspects of the performance of alkali-free liquid accelerator, such as the compatibility for different types of cement, sensitivity of the setting time, setting effect under different temperature, and irregular change of cement mortar's volume, are still not well understood.

The main components of various types of accelerator are greatly different; therefore, the action mechanisms are also far from the same. According to the opinion of Meiyan et al. [11], the acceleration mechanism of the setting accelerator was that the accelerator promoted the production of AFt crystal with a random orientation in the early stage of hydration. Guoqiang et al. [12] believed that a large amount of six-angle plate-shaped hydrated calcium aluminate produced by the reaction of C_3A and $Ca(OH)_2$ accelerated the condensation of cement paste, because the retardation effect of gypsum was eliminated by the accelerating agent. According to the opinions of Paglia et al. [13], $Al_2(SO_4)_3$ in accelerator promoted the formation of ettringite and connected cement particles to aid in rapid coagulation. Moreover, some researchers studied the effect of accelerator on the performance of the cement concrete. Maltese et al. [14] investigated the effect of moisture on the setting behavior of a Portland cement reacting with an alkali-free accelerator and found that the β-hemihydrate dissolution rate played an important role in the reduction in setting time of cement paste samples mixed with an alkali-free accelerator. Guo et al. [15] found that the compressive strength of cement mortar with alkali-free liquid accelerators is positively proportional to the age. Lee et al. [16] investigated the durability of mortar specimens incorporating inorganic alkali-free accelerator (AFA) exposed to external sulfate attack and pointed out that special care needs to be taken when the shotcrete with AFA is applied under sulfate-bearing environments.

In conclusion, how to develop an efficient and environmentally friendly alkali-free liquid accelerator for the shotcrete which can shorten the setting time of the concrete and also have no negative effects on the strength and durability of the concrete is still a very important research direction. In this paper, a new type of alkali-free liquid accelerator for shotcrete was prepared. Moreover, the compatibility of accelerator for different types of cement and water reducing agent, the sensibility under conditions of different temperatures, and the accelerating mechanism were investigated.

2. Experiment

2.1. Materials

2.1.1. Cement. Two types of Portland cement (Jidong cement and Qinling cement) were used in this experiment; the detail properties of these two types of cement are shown in Table 1. The main physical indexes of cement meet the requirements of standard JTG F30-2003 (Technical Specifications for Construction of Highway Cement Concrete Pavements). Jidong cement was produced by the Shaanxi Jidong limited liability company, and Qinling cement was produced by Shaanxi Qinling Cement Limited by Share Ltd.

2.1.2. Sand. The sand used in these experiments was clean river sand with a fineness modulus of 2.6.

2.1.3. Alkali-Free Liquid Accelerator. Five types of organic and inorganic materials apart from water have been chosen to synthesize the new type of Alkali-free liquid accelerator, which were aluminum sulfate, sodium fluoride, triethanolamine, polyacrylamide, and formic acid. All raw materials were CP grade, produced by Xi'an Chemical Factory. According to [17–19], Cl^-, SO_4^{2-}, and Al^{3+} have a significant effect on the early strength of the cement, and CO_3^{2-}, $[Al(OH)_4]^-$, SiO_3^{2-}, and F^- can shorten the setting time of the cement. Therefore, we chose the aluminum sulfate and sodium fluoride as the main components of the accelerator. Triethanolamine can increase the early strength of the concrete [20]. Polyacrylamide can improve the viscosity of cement paste, which is good for reducing the rebound of shotcrete [21]. The additive proportions of aluminum sulfate, sodium fluoride, triethanolamine, polyacrylamide, and formic acid were 5%, 0.6%, 0.2%, 0.15%, and 0.1% weight of the cement. The solid content of Alkali-free liquid accelerator was 43.7%. The alkali content was smaller than 1.0%.

2.1.4. Water Reducing Agent. Naphthalene water reducing agent and polycarboxylic acid water reducing agent were applied to study the compatibility of accelerator for water reducing agent.

Table 2: The effect of water cement ratios on setting time (20 ± 2°C).

Water cement ratio	Type of cement	Initial setting time	Final setting time
0.35	Jidong cement	1 min 30 s	3 min 20 s
	Qinling cement	2 min	4 min 30 s
0.40	Jidong cement	3 min	6 min 50 s
	Qinling cement	3 min 30 s	7 min 20 s
0.45	Jidong cement	13 min 10 s	35 min
	Qinling cement	15 min	30 min 50 s

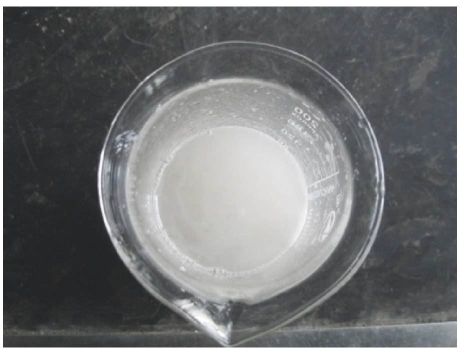


Figure 1: The new type of alkali-free liquid accelerator.

2.2. Preparation of Alkali-Free Liquid Accelerator. Aluminum sulfate was dissolved in water with high-speed stirring; dissolution time can be shortened by heating the temperature of the solution to 50–90°C during the dissolving period. Fluoride can be mixed to the solution when aluminum sulfate was stirred, which is helpful to accelerate the dissolution of aluminum sulfate, and fluoride was added to the solution after the aluminum sulfate was completely dissolved.

Mix triethanolamine and polyacrylamide with the above solution and stir it for about 20–35 minutes with high speed. Finally, acid was mixed in the solution, and the new type of alkali-free liquid accelerator was prepared as shown in Figure 1.

2.3. Tests

2.3.1. Setting Time. The test of setting time of cement paste was conducted according to the standard of JC 477-2005 (flash setting time admixtures for shotcrete).

2.3.2. Strength and Shrinkage Test. The strength and shrinkage tests of cement mortar were conducted according to the standard of JGJ/T70-2009 (standard for test method of performance on building mortar). The accelerator according to the proportion was added to the mortar after the mix of the cement and water one time.

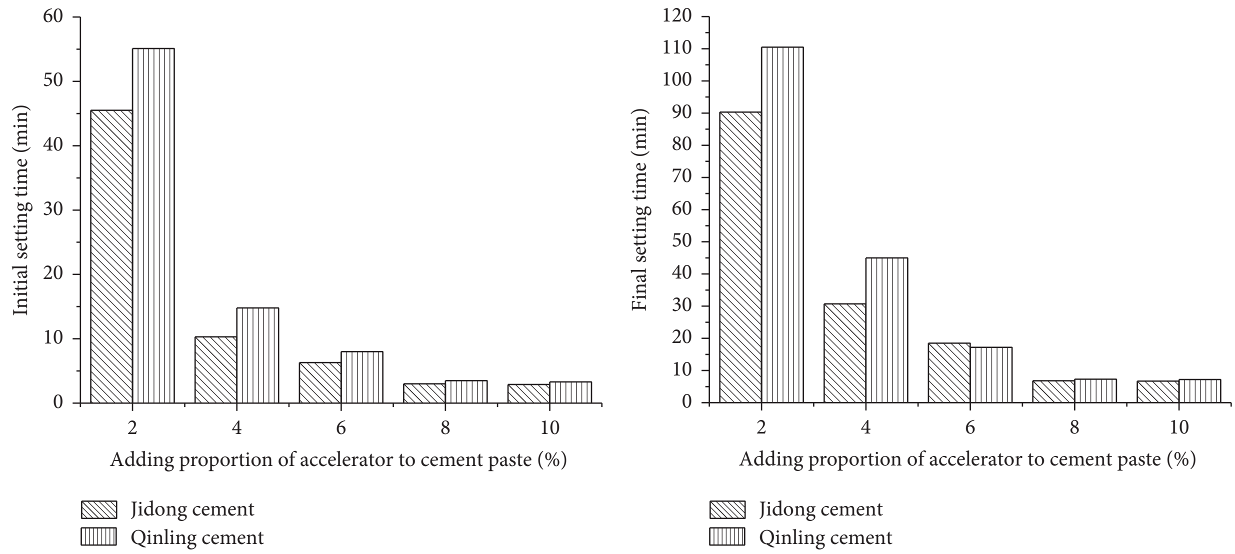
2.3.3. XRD and SEM Test. Specimens with 3~5 mm diameter size were removed from the interlayer of cement paste after setting for 7 mins, 240 mins, 1 day, and 28 days. Then the hydration of specimens was terminated by acetone, and specimens were ground to powder and sifted through 190 mesh sieve. Further, such powder was placed into a slot and tested by X-ray diffractometer (XRD7000, produced by Shimadzu, Japan; work power: 3 kW, angle range of scanning: 15~70°, scan velocity: 2~5°/min, and step size: 0.02°/step). Moreover, for the SEM test, S-4800 type field emission scanning electron microscopy (produced by Hitachi Company) was used to observe the microstructure of cement hydration products. The fracture surfaces of cement specimen were treated by desiccation and spray-gold.

3. Results and Discussions

3.1. Effect of the Content of Accelerator on Setting Time. The effect of various accelerator contents on the setting time of the cement paste with the addition of accelerator was shown in Figure 2. The test temperature was 20 ± 2°C, and the water cement ratio (w/c) was 0.4. According to Figure 2, the setting time of Jidong cement and Qinling cement paste decreased with the content of the accelerator, indicating that the accelerator had good compatibility to different types of cement. When the mixing content of accelerator was 8% of cement paste, the initial and final setting times of cement were about 3 min and 7 min. The change of setting time was not obvious beyond a content of 8% of cement paste.

3.2. Effect of Water Cement Ratio on Setting Time. Three water cement ratios of 0.35, 0.40, and 0.45 were used to study the effect of water cement ratios on the setting time of the cement. The test temperature was 20 ± 2°C. The results are shown in Table 2. According to Table 2, the accelerating effectiveness of the accelerator worsened with the increase in water to cement ratio. An excessive water cement ratio increased the setting time and the risk of shrinkage cracks. However, a low water to cement ratio causes problems such as an unstable ratio, high spring back rate, and abrasion of mechanical equipment for the use of shotcrete. Therefore, a proper water to cement ratio should be chosen according to the type of setting accelerator used in the process of shotcrete and construction situation.

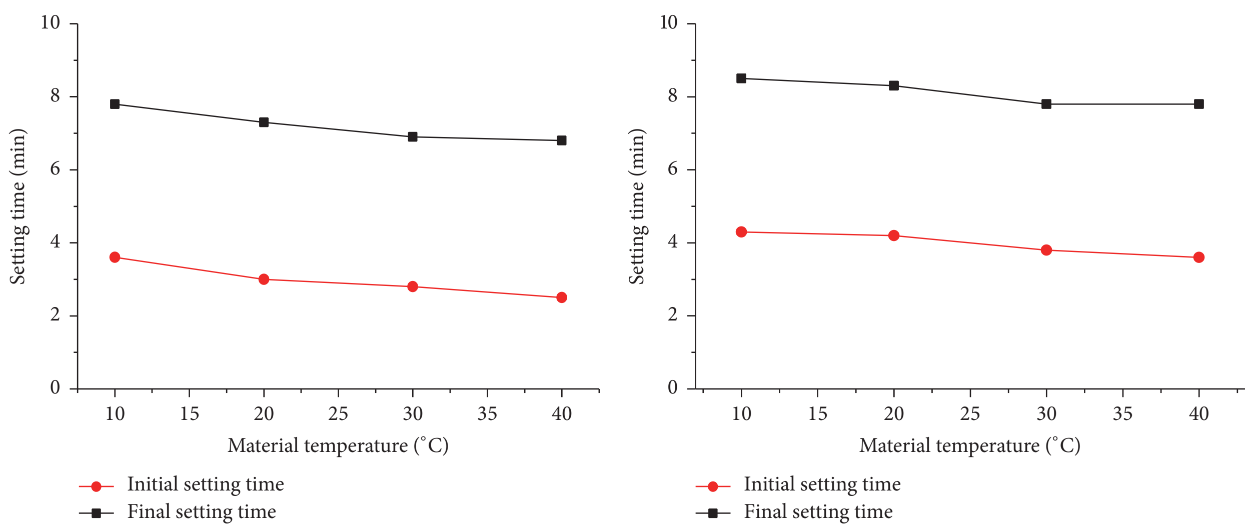
3.3. Effect of Temperature on Setting Time. Material temperature was a vital element to influence the hydration speed and setting time of cement paste. Particularly in the process of spraying concrete in winter, the temperature of construction and material temperature greatly differed by 20 ± 2°C in laboratory and a low temperature may make the setting accelerator lose its efficiency; therefore, it was necessary to explore the influence of material temperature



(a) Initial setting time

(b) Final setting time

Figure 2: The effect of the content of accelerator on setting time of cement paste (20 ± 2°C, w/c = 0.4).



(a) Jidong cement

(b) Qinling cement

Figure 3: The effect of temperature on setting time of cement paste.

on setting time of concrete with accelerator. The effect of material temperature on the setting time of Jidong and Qinling cement paste was tested, as shown in Figures 3(a) and 3(b), respectively. The water to cement ratio was 0.40.

According to Figure 3, the accelerating effect of alkali-free liquid accelerator decreased with the reducing of material temperature for both types of cement paste, but the change was not obvious. Take Jidong cement as example, the initial and final setting times were 3.6 min and 7.8 min when material temperature was 10°C, indicating that the setting accelerator had a good acceleration effect even at low temperatures. The initial and final setting times were 2.5 min and 6.8 min when material temperature was 40°C, so an appropriate content of accelerator is needed according to the construction environment.

3.4. Effect of the Content of Accelerator on Strength of Cement Mortar. Traditional alkali powdery accelerator decreased the strength of cement mortar by 20%~35%, sometimes even 50%, which restricted the use of traditional alkali powdery accelerator [22–24]. The retention rate is reference to the ratio of the strength of the concrete with accelerator to that of the concrete without accelerator. The effect of the content of accelerator on strength of cement mortar with alkali-free liquid accelerator with a water to cement ratio of 0.4 was tested, as shown in Table 3.

Table 3: The effect of content of accelerator on strength of cement mortar.

Content of accelerator/%	Type of cement	Flexural strength/MPa			Compressive strength/MPa			Retention rate of 28 d compressive strength/%
		1 d	7 d	28 d	1 d	7 d	28 d	
0	Jidong cement	1.10	3.86	5.50	15.20	30.68	39.18	100
	Qinling cement	2.65	5.85	6.16	17.18	34.80	38.75	100
6	Jidong cement	2.30	4.86	6.30	12.55	42.32	43.16	110
	Qinling cement	3.37	6.68	7.25	21.36	38.53	40.12	104
8	Jidong cement	2.80	6.52	7.10	13.75	39.60	44.45	113
	Qinling cement	3.54	7.00	7.85	23.40	42.55	42.70	110
10	Jidong cement	2.60	6.15	6.84	11.78	40.92	42.24	108
	Qinling cement	3.45	6.37	7.43	20.16	39.88	42.18	109

Table 4: The adaption of accelerator to water reducing agent.

Type of water reducing agent	Content of water reducing agent/%	Water cement ratio	Initial setting time	Final setting time
Cement paste without water reducing agent	0	0.4	3 min	6 min 50 s
Naphthalene water reducing agent	0.8	0.4	3 min 50 s	7 min 48 s
		0.35	2 min 50 s	6 min 30 s
Polycarboxylic acid water reducing agent	0.75	0.4	4 min 10 s	8 min 5 s
		0.35	2 min 38 s	6 min 10 s

As can be seen from the test results summarized in Table 3, the retention rate of compressive strength after 28 days increased rather than decreasing after mixing with alkali-free liquid accelerator. When the content of setting accelerator was 8%, the retention rates of compressive strength after 28 days of Jidong cement mortar and Qinling cement mortar were 113% and 110%, respectively. Early compressive strength after 1 d and 7 d of cement mortar with alkali-free liquid accelerator was 36% higher than cement mortar without accelerator. In addition, with the increased content of accelerator, the compressive strength after 28 d increased first and then decreased. The reason may be the fact that too much accelerator would reduce the adhesion of calcium silicate hydrates in a unit area and close contact required for condensation because of too fast of a response. The hydration cannot be finished completely, which decreased compressive strength.

3.5. Adaption of Accelerator to Water Reducing Agent. Water reducing agent is usually mixed in concrete construction to reduce the water to cement ratio and improve the strength and durability of concrete while maintaining the same flowability. In order to maintain similar consistency in this experiment, the water to cement ratios of Jidong cement paste with and without water reducing agent were 0.35 and 0.40, respectively. The effect of the water reducing agent on the setting time of cement paste was measured, and the results are shown in Table 4. It can be seen from the experimental results that if the water to cement ratio was constant, the free water increased with the increase in water reducing agent because of a water reducing effect, which had a negative effect on quick setting. But if water to cement ratio was reduced properly, not only was the acceleration effect of setting accelerator enhanced but also the flowing property of cement was well kept, which was beneficial to make sprayed concrete with high strength.

3.6. Effect of Accelerator on the Volume Change of Cement Mortar. The volume change of cement mortar refers to the expansion and shrinkage of mortar under the influence of outside temperature, as well as the self-shrinkage caused by the hydration of cement under the influence of separation from outside temperatures. The effect of accelerator on the volume change of cement mortar was analyzed and the results are shown in Figure 4.

According to Figure 4, shrinkage of cement mortar mixed with the new type of alkali-free liquid accelerator increased obviously with increased mixing amount, consistent with previous researches [7, 11, 15]. The main reason may be the fact that high dosages of accelerators will make the matrix set too quickly, which increases the volume of voids and defects in the matrix. Porosity makes hardened cement mortar prone to drying shrinkage. In addition, early formation of ettringite cannot keep pace with the formation of C-S-H, and swelling is reduced by plasticity paste, so it cannot be reflected in the total volume change of mortar. It is well known that overly large volume shrinkage can easily cause cracking in concrete; therefore, it is necessary to carry out reasonable and effective maintenance of sprayed concrete mixed with accelerator, or little swelling agent can be mixed into counteract volume shrinkage caused by the accelerator. Moreover, in practice, the compacting function by high-speed shotcrete jet stream will

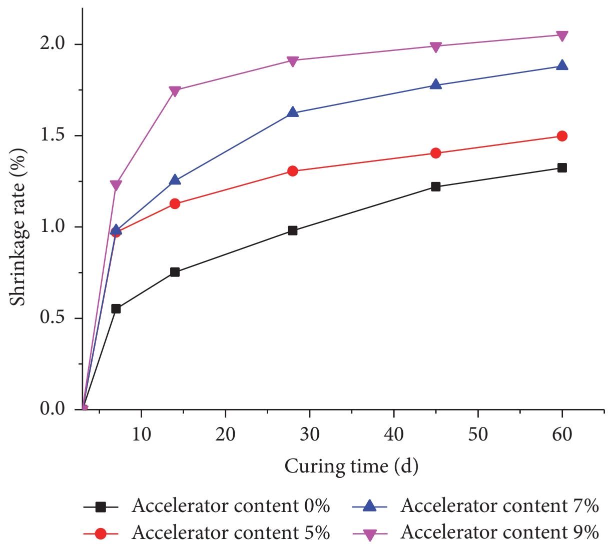


Figure 4: The effect of accelerator on shrinkage rate.

make the matrix denser and reduce the shrinkage. To balance the setting time and shrinkage, 5~8% content of accelerator is recommended for shotcrete in practice.

3.7. Analysis of XRD Results for Hydration Samples. The reference hydrated sample and the hydrated sample with the addition of 8% of the new type of alkali-free liquid accelerator at each age were tested by XRD, as shown in Figures 5 and 6, respectively.

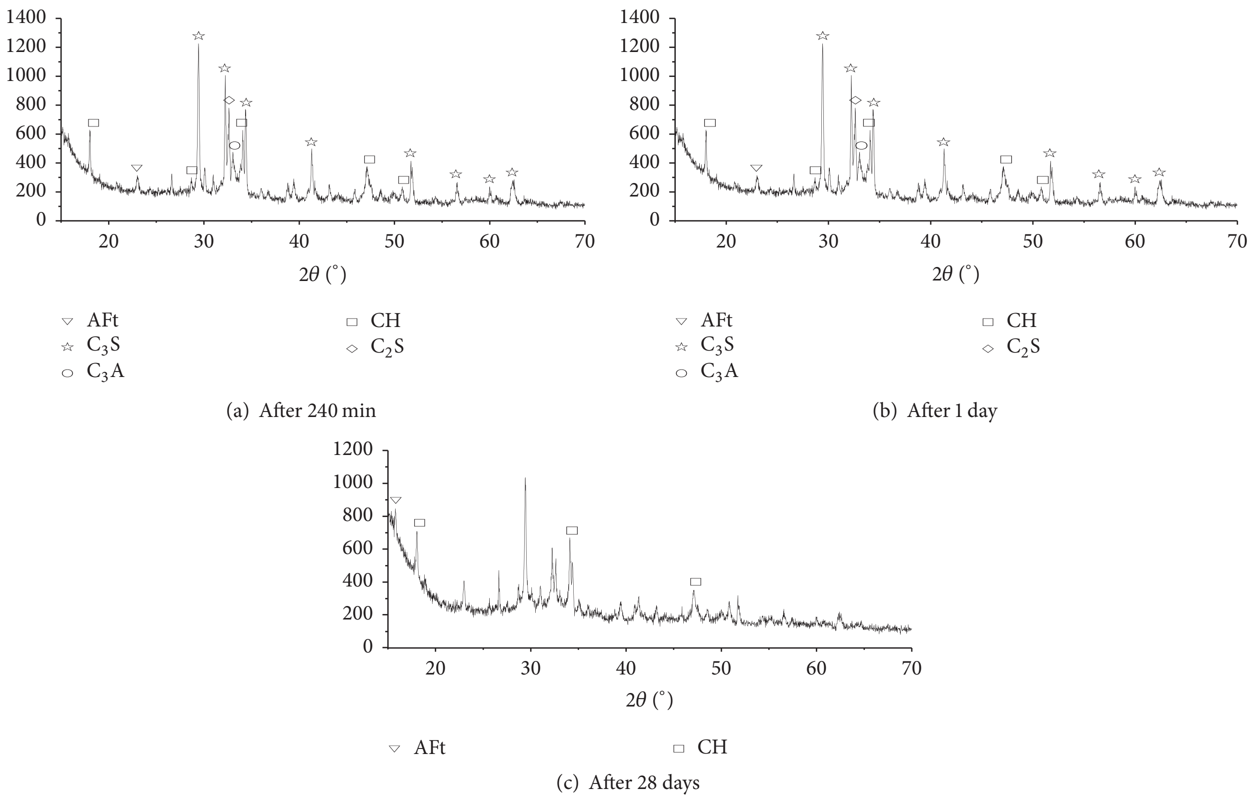
Figure 5 presents the XRD patterns of the reference hydration samples at each age. Ettringite was not detected in the reference hydrated sample at the age of 240 min (Figure 5(a)). The characteristic peaks of ettringite began to appear in this sample at the age of 1 day (Figure 5(b)); however, the peak value was relatively low and not obvious, indicating the beginning of an ettringite formation, but the amount was limited. In addition, there were unhydrated C_3A, C_3S, and C_2S, as well as $Ca(OH)_2$ produced by the hydration of C_3S, among which the characteristic peak of $Ca(OH)_2$ was most obvious. It was observed that the main mineral hydration of the sample was $Ca(OH)_2$ and ettringite at the age of 28 days (Figure 5(c)).

Figure 6 presents the XRD patterns of the hydrated sample with the accelerator at each age. It was found that the samples had obvious characteristic peak diffraction of ettringite at the age of 7 min, as shown in Figure 6(a), which indicated that a considerable amount of ettringite had been formed at this time. In Figure 6(b), the characteristic diffraction peaks of ettringite at the age of 240 min were more obvious compared to the hydration after 7 min, indicating that the amount of ettringite crystals formed increased gradually with hydration time. The formation of ettringite in the early curing age indicated that the hydration speed of cement was accelerated by the accelerator. As a result, the setting time and the strength of concrete reduced and increased, respectively. Moreover, it may be the reason accounting for the increase of shrinkage in the early curing age. The main mineral in the hydrated sample with the accelerator after 1 day was the same as the reference hydrated sample, but the diffraction peak of $Ca(OH)_2$ was relatively low, as shown in Figure 6(c). On one hand, this is because a large amount of Ca^{2+} released by C_3S hydration was consumed during the formation of ettringite. On the other hand, F^- in the accelerator also reacts with Ca^{2+} to consume Ca^{2+} released by C_3S hydration. The decrease of Ca^{2+} concentration further promoted the hydration of C_3S and played a role in accelerating the coagulation of cement, as well as improving the early strength of cement. As time proceeded (28 days), the diffraction peaks of ettringite of the hydrated sample with the accelerator were more obvious than the reference hydrated sample; moreover, the diffraction peak of $Ca(OH)_2$ was relatively low (Figure 6(d)). This is also due to the reaction between $SO_4{}^{2-}$ and F^- in the accelerator and Ca^{2+}, and $Ca(OH)_2$ was consumed as mentioned earlier. In general, we found that the addition of the accelerating agent changed the amount of cement hydration products but did not change the type of hydration products by comparing Figures 5(c) and 6(d).

3.8. Analysis of SEM Results for Hydration Samples. The hydration products of cement paste with and without the addition of 8% of the new type of alkali-free liquid accelerator at each age were observed by SEM to reconfirm the formation of ettringite in the early hydration stage; the results are shown in Figure 7.

According to Figure 7, for the cement paste without accelerator, ettringite was not observed in the hydration specimen at the age of 240 min. After 1 day, a small amount of threadiness gel and a number of nonhydrated cement particles covered with ettringite on the surface were observed. Then it was found that a large amount of fibrous and reticular C-S-H gel which was filled with $Ca(OH)_2$ crystal appeared in the reference sample after 28 days. However, it was observed that ettringite in a short bar outline formed in the hardened cement specimen with addition of the accelerator in the final setting time of 8 min. Due to a large number of ettringite crystals generated within a short period of time, the crystals overlapped each other, resulting in an accelerated setting of the cement paste. At the hydration time of 1 day, $Ca(OH)_2$ crystals began to appear in the sample; moreover, it was noted that the gap between the cement particles was small and the structure became compact at this time. As time proceeded (28 days), the cement structure was more compact due to the formation of a large number of ettringite crystals.

In summary, the mechanism of the new type of alkali-free liquid accelerator should be noted that a large number of ettringite crystals deposited in the early stage of cement hydration due to the increase of Al^{3+} and $SO_4{}^{2-}$ concentration in cement paste after the addition of the accelerator. More importantly, these crystals overlapped each other, forming a space frame structure, resulting in the rapid condensation of cement paste.

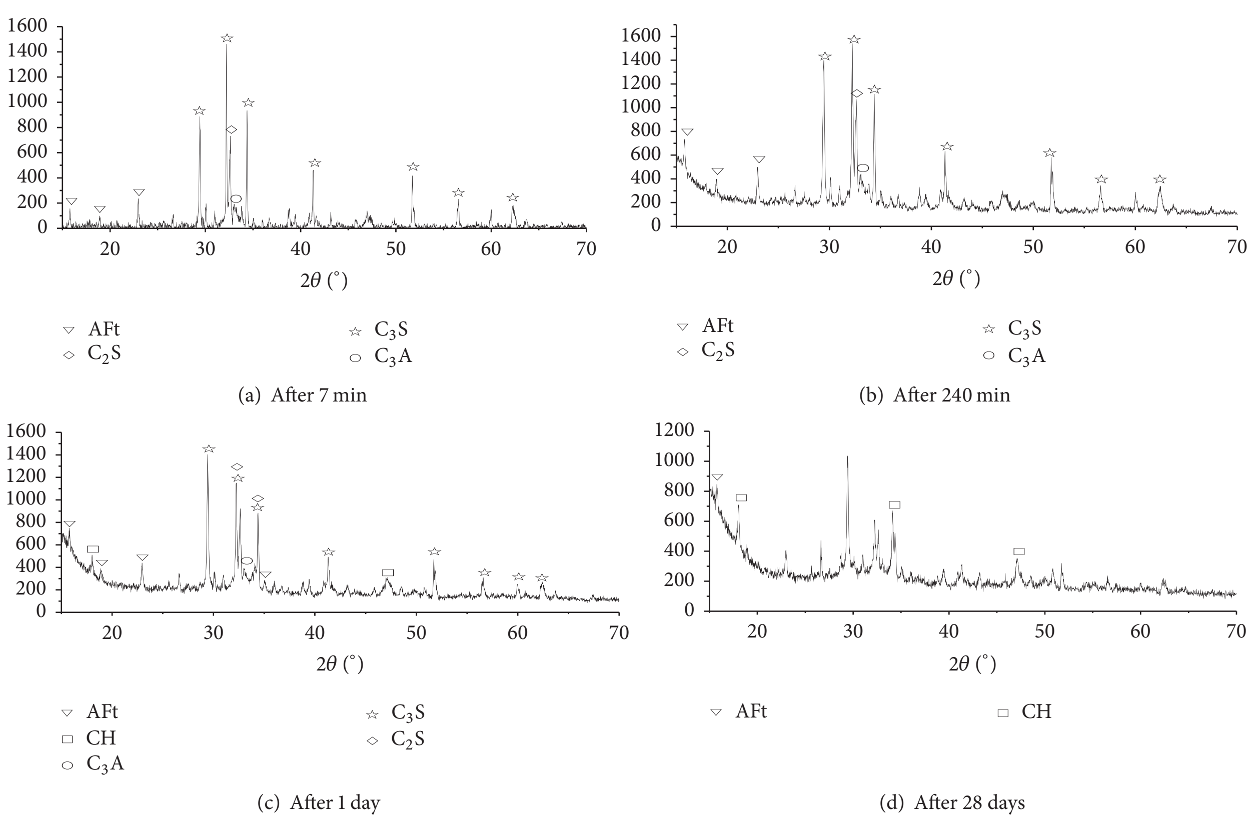


(a) After 240 min

(b) After 1 day

(c) After 28 days

Figure 5: The XRD diffractograms of the hydrated cement samples without the accelerator.



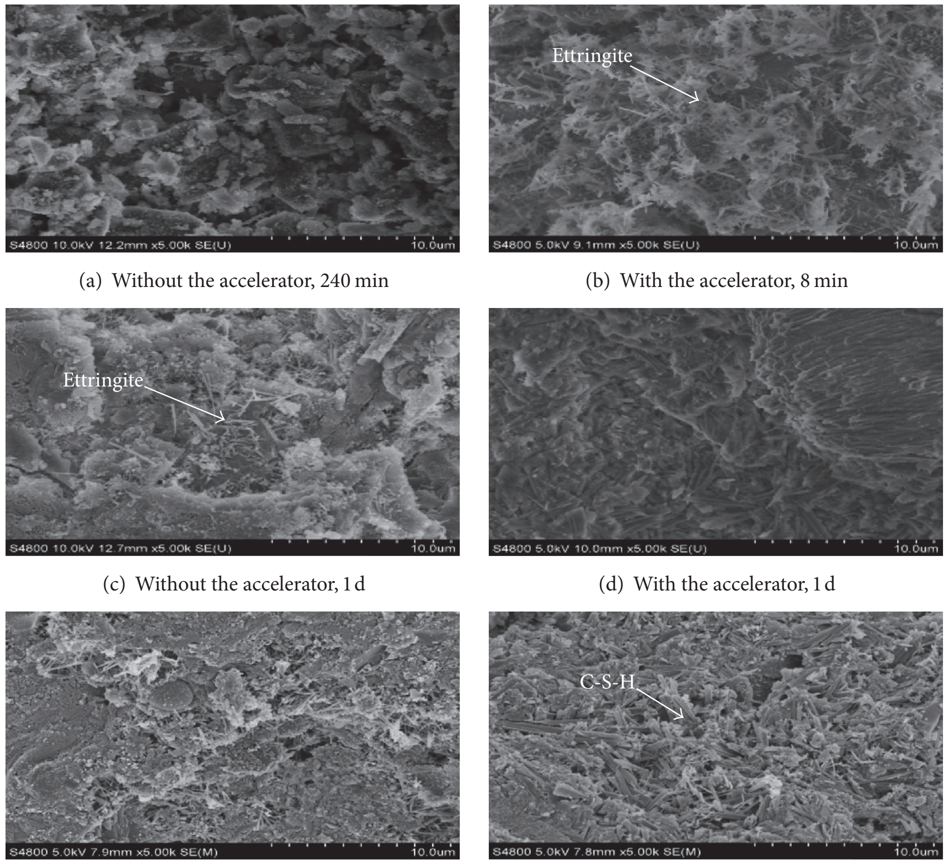
(a) After 7 min

(b) After 240 min

(c) After 1 day

(d) After 28 days

Figure 6: The XRD diffractograms of the hydrated cement samples with the accelerator.



(a) Without the accelerator, 240 min
(b) With the accelerator, 8 min
(c) Without the accelerator, 1 d
(d) With the accelerator, 1 d
(e) Without the accelerator, 28 d
(f) With the accelerator, 28 d

Figure 7: SEM images of hardened cement paste specimens with and without the accelerator.

4. Conclusion

We conclude the following:

(a) A new type of alkali-free liquid accelerator prepared by aluminum sulfate, sodium fluoride, triethanolamine, polyacrylamide, and formic acid in this paper has shown good accelerating effects. When the adding content of accelerator was 8 wt% of cement paste, the initial and final setting times of cement paste were about 3 min and 7 min for two types of Portland cement (Jidong cement and Qinling cement).

(b) Compressive strength at 1 day of cement mortar with the 8 wt% accelerator could reach up to 23.4 MPa, which increased by 36.2% compared to the strength of cement mortar without accelerator. The retention rate of compressive strength of cement mortar at 28 days can reach as high as 110%.

(c) The accelerator was not sensitive to the change of material temperature, and it still has a good effect of promoting coagulation even at low temperatures (10°C).

(d) The shrinkage of cement mortar increased with the adding content of alkali-free liquid accelerator. The main reason may be the fact that excessive accelerators make the cement matrix set too quickly, which increases the volume of voids and defects in the matrix. To balance the setting time and shrinkage, 5~8% content of accelerator is recommended for shotcrete in practice.

(e) XRD and SEM test results showed that the accelerating mechanism was that the alkali-free liquid accelerator promoted the formation of ettringite crystals by increasing the Al^{3+} and SO_4^{2-} concentration of the matrix.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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