Preparation and hydration mechanism of aluminum formate/aluminum sulfate based alkali-free composite accelerator for sprayed concrete

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Abstract
The high alkali content of alkaline accelerators has a negative influence on the late strength and durability of concrete. Further, their corrosive nature is harmful to the skin of construction personnel, posing safety concerns. Therefore, the development of alkali-free accelerators has gained interest, especially in shotcrete-based constructions. The production process of aluminum-sulfate-based alkali-free accelerators is simple and less exothermic, with no negative impact on the later strength of concrete, aspects that are widely recognized and very important in construction. However, their high sulfate content may have a significant impact on the durability of concrete; therefore, reducing the sulfate content plays an important role in enhancing the durability of concrete. In this study, an alkali-free liquid accelerator was synthesized by replacing aluminum sulfate with aluminum formate, which effectively reduced the sulfate content. Experiments were performed on concrete samples in which aluminum sulfate was replaced with aluminum formate at different levels. The results showed that the formate and 3CaO·Al2O3 (C3A) of the alkali-free liquid accelerator prepared from aluminum formate form calcium aluminate, similar to the ettringite phase. The formate promoted the dissolution of Ca3SiO5 (C3S), thus accelerating the hydration of concrete. Meanwhile, aluminum ions hydrolyzed by aluminum formate also participated in the reaction, resulting in the dual participation of cations and anions in hydration, leading to a synergistic effect with aluminum sulfate.

1. Introduction

Cement-based materials are widely used in building and road structures, due to their excellent durability and mechanical properties. Meanwhile, cement-based materials and their additives have been extensively studied. Rafik K. studied the by-product material (cement kiln dust) from the cement manufacturing process, made it into foamed paste by adding CKD with different proportions of cement, studied the compressive strength and thermal conductivity, and found that foamed (cement kiln dust–cement) paste (FCKDC) can be used as an insulation material [1]. Zhang et al used a grading of emulsified asphalt mastic with rapid cement cracking and stony mastic asphalt to obtain a high-performance pavement material. The performance of Stony mastic cement asphalt (SMCA), continuously graded cement asphalt (CCA) and SMA was evaluated by Marshall stability value, flow value, indirect tensile strength and Cantabro loss, and it was found that the technical performance of SMCA was CCA was relatively similar, while Cantabro loss was much lower than that of CCA. Compared with SMA, the performance is higher than SMA except for the flow value [2]. He et al used a mixture of bentonite and cement (noted as BC) with a mass ratio of 2:1 as a curing agent to remediate lead-contaminated soil, and conventional cement (PC) as a comparative curing agent, and found that the mixture of bentonite and cement could effectively immobilize the heavy metal lead in the soil and avoid excessive soil strength and consequent soil hardening, which would affect redevelopment and utilization [3]. Wang et al solved the problem of low 28-day compressive strength of cement under standard curing conditions by studying the effect of activator content on
the strength of alkali-slag oil shale ash cement under standard curing conditions after 3 days of thermal excitation at 50 °C [4]. In summary, cement and the by-products produced during its production have a wide range of applications in industrial production. And shotcrete has been widely used in the construction of tunnels and mines as a cement-based material [5]. Its main characteristics, quick setting time and high early strength, are crucial for accelerating the progress of tunnel excavation. The key component that lends shotcrete these characteristics is the accelerator. There are two main types of accelerators: alkali and alkali-free [6–11].

The indexes mainly used to assess the performance of shotcrete include the freezing resistance, early strength, late strength, and condensation time. Other important factors include the role of the liquid accelerator and the injection characteristics, such as mixing dispersion, dust, and injection ejection rate. Presently, there are many types of accelerator; depending on their nature and state, they can be approximately classified into alkali powder, alkali powder-free, alkaline liquid, and alkali-free liquid. The accelerator of the rapid condenser vary, leading to distinct effects on the concrete performance. With the continuous demand for improvement in the concrete performance and stricter environmental safety standards, alkaline accelerators have been found to perform poorly in production and construction. On the contrary, liquid accelerators demonstrate excellent yet safe performance because of their high strength in the early stage and stable late-strength retention rate, making them commercially popular. Thus, an increasing number of mechanistic studies have been focusing on alkali-free accelerators as the main pro-condensation component, owing to their safety, environmental protection, and distinct condensation-promoting effect [12–14].

Compared with alkali accelerators, the alkali content of alkali-free accelerators is less than 1.0 wt%, which has a negligible adverse effect on the later strength of concrete [15, 16]. In recent years, owing to the extensive research on the synthesis of alkali-free accelerators, significant progress has been made in its production [17]. There are three types of alkali-free accelerators. The first one is the hydrofluoric-acid-based alkali-free accelerator, which is usually produced using hydrofluoric acid and aluminum hydroxide as the primary raw materials. Its main characteristics are a fast setting time, low cost and early strength, poor frost resistance of concrete, and heavy pollution during production [18–20]. The second one is the fluorosilicic-acid-based alkali-free accelerator, which is usually produced using fluorosilicic acid and aluminum sulfate as the primary raw materials. Its main characteristics are a fast setting time and high early strength; however, it also demonstrates poor frost resistance, owing to the presence of fluorine ions. Additionally, as fluorosilicic acid is a hazardous chemical, the risk coefficient during production is high. The third one is the aluminum-sulfate-based alkali-free accelerator, which is mainly produced by the physical matching of aluminum sulfate and alcohol amines. Its production process is simple, the heat release during synthesis is minor, and it has no negative impact on the later strength of concrete [21, 22]. However, although these accelerators are widely applied in the construction of tunnels and mines, their high sulfate content has a significant impact on the concrete durability [23].

Cement hydration is usually divided into four stages: the initial reaction period, induction period, acceleration period, and deceleration period [24]. The initial reaction period of cement reacts sharply, and the hydration of tricalcium silicate (C₃S) dissolved in water occurs rapidly and hardens, so the first exothermic peak appears, which is very short and ends within 15 min [25–27]. After that, the reaction of the induction period is extremely slow, which is also called the resting period. It usually lasts for 1–4 h. It is the reason why the silicate cement paste can maintain plasticity within a few hours [26–28]. The induction period occurs because the rapid production of tricalcium silicate hydrate during the initial reaction period covers the surface of the cement particles and prevents further contact between water and cement. The time of initial setting is essentially equivalent to the end of the induction period [26, 27, 29]. The reaction is accelerated again during the accelerated phase, and the cement particles in the resting phase are re-exposed to contact with water molecules due to the rupture of the protective film (cement silicate hydrate). A second exothermic peak appears, and the phase ends when the peak is reached, generally in 4–8 h. At this time, the final condensation has passed and hardening begins [26, 27, 30]. The decelerator is the stage where the reaction rate decreases with time because the hydration of tricalcium silicate (C₃S) is hindered as hydrates gather around the cement particles, so hydration enters the deceleration process from the acceleration process. It lasts about 12–24 h and the hydration is gradually controlled by the diffusion rate [26, 27, 31]. The accelerator can shorten the induction and acceleration periods by reacting with 3CaO·Al₂O₃ (C₃A) and promoting the ionic dissolution of Ca₅SiO₄ (C₅S). It was observed that a large amount of ettringite is often formed on the surface of C₃S in the accelerator cement system, preventing the further hydration of C₅S [32]. The specific reaction mechanism of the aluminum-sulfate-based liquid accelerator is as follows [33]:

$$\text{Al}^{3+} + 4\text{OH}^- \rightleftharpoons \text{Al(OH)}_4^- + 4\text{OH}^- \rightleftharpoons [\text{Al(OH)}_4^-]^+$$

$$2[\text{Al(OH)}_4^-]^- + 6\text{Ca}^{2+} + 4\text{OH}^- + 3\text{SO}_4^{2-}$$
$$+ 26\text{H}_2\text{O} \rightleftharpoons \text{C}_3\text{A}_{3\text{A}} \cdot 3\text{CaSO}_4\text{A} \cdot 32\text{H}_2\text{O}$$
The reaction mechanism shows that the aluminum-sulfate-based liquid accelerator mainly promotes the formation of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ (AFt). AFt plays two roles—as an early reinforcement material or a late deterioration material [34]. This aluminum-sulfate-based quick setting agent has the characteristics of an unstable setting time and poor cement adaptability [35–37]. Therefore, to reduce the influence of aluminum-sulfate-based liquid accelerators on the concrete durability, the development of low-sulfur alkali-free liquid accelerators may be an excellent alternative [38]. Hence, this study explores the substitution of aluminum formate for some aluminum-sulfate synthetic quick-setting agents. This process mainly uses the reaction between formate and $\text{C}_3\text{A}$ (of aluminum formate) and the early strength function of formate, to produce a synergistic effect that improves the setting time and one-day compressive strength of the resultant quick-setting agent.

In this study, to reduce the sulfur content and improve the early strength of the aluminum-sulfate-based liquid accelerator, a new alkali-free accelerator was partially synthesized by selecting aluminum formate as an aluminum-containing raw material instead of aluminum sulfate. The effect of the accelerator was also evaluated in terms of the setting time, early strength, and hydration reaction.

### 2. Methods

#### 2.1. Materials

The cement used in the test was ordinary Portland cement (PO42.5). Table 1 lists the chemical composition of the cement. The reagents used in the experiment were aluminum sulfate, diethanolamine, aluminum formate, triethanolamine, glycerin, a suspending agent, and dispersant, as shown in table 2.

#### 2.2. Synthesized aluminum formate alkali-free liquid accelerator

A certain amount of aluminum formate was added to water in a 1000 ml three-port flask and stirred until it was dissolved completely. Aluminum sulfate was added to the solution and stirred for 1 h; diethanolamine and a suspending agent were added to the system and stirred continuously until dissolution. Finally, formic acid solution, with a pH of 2.1, was added and stirred continuously for 30 min.

#### 2.3. Material preparation and characterization

The setting time was determined in accordance with the Chinese standard GB/T35159-2017. To analyze the accelerator, the reference cement was chosen to be PI 42.5, and the content of the accelerator was 8% of the cement quality.

In this study, in order to exclude the influence of other concrete admixtures on the strength and setting time of cement mortar, the Chinese standard GB/T17671-1999 was used regarding the production of samples with a water–cement ratio of 0.5 and a sand ratio of 100%. The amount of quick-setting agent was determined according to the set setting time. The specimen size was $40 \times 40 \times 160$ mm, and the mold containing the
specimen was removed after curing for 24 h at the standard temperature (ambient temperature: 20 ± 2℃, relative humidity: >95%). A compressive strength test was carried out after curing to the corresponding age (one day and 28 days). In this study, three samples of each alkali-free flash setting admixture were made. Each sample was tested for setting time and compressive strength, and the average of the results obtained was used as the final result. The cement slurry samples were broken after curing to the appropriate age. Hydration was terminated using anhydrous ethanol and isopropanol for 24 h. Following filtration, the retained powder was dried in vacuum at 40 °C for 24 h. X-ray diffraction (XRD) analysis was performed on the powder after it was ground to particle sizes of <70 μm. The flake samples were then extracted from the broken surfaces for scanning electron microscopy (SEM) testing.

Figure 1. XRD of 1d hydration products.

Isothermal calorimetry was performed using a TA instrument with a TAM air bottle. The XRD test angle was in the range of 5°–80° and the scanning speed was 5° min⁻¹. SEM was used to observe the morphology of the hydration product.

3. Results and discussion

The XRD results are shown in figure 1. The intensity of the post-diffraction peak increased and that of the C₃S peak decreased with increase in the aluminum formate content, especially for 30% aluminum formate. Further, aluminum formate had a certain hydration-promoting effect. This is attributed to the penetration of aluminum formate into the hydrated layers of C₃S and C₂S, accelerating their dissolution [39–41]. The hydration products of ethephonite are lower in promoters that do not contain aluminum formate compared to the promoters that do.

As seen in figure 2, different aluminum formate additions result in different amounts of ettringite and C-S-H in the hydration product. The amount of ettringite produced by AR without aluminum formate was less, and C-S-H was not apparent. On comparing the one-day hydration products of the accelerator with aluminum formate, the most prominent was 30% aluminum formate, and its AFt was more needle-like. However, C-S-H is cluster-like, which indicates that the hydration products are abundant, and the matrix structure is dense, indicative of their compressive strength. The effect of formate radicals and the aluminum of aluminum formate can be explained as follows.

Figure 3 shows the effect of aluminum formate substituting aluminum sulfate on the initial and final setting times. It can be seen from figure 3(a) that the initial setting time is shortened as the aluminum formate content is increased. The initial setting time without aluminum formate was 195 s, whereas that with 30% aluminum formate was 147 s. Thus, the addition of aluminum formate can greatly improve the initial setting time of the accelerator. The main reason for this is the synergistic effect of formate in the system, resulting in an increase in the ettringite content. It has also been proven in relevant literature that formate can promote the hydration of cement. Figure 3(b) shows that the effect of aluminum formate on the final setting time is similar to that on the initial setting time. Aluminum formate has a positive effect on the setting time of the alkali-free liquid accelerator. This may be because the formate easily forms an AFt-like substance with C₃A in calcium-rich
cement systems, with a structure of $\text{C}_3\text{A} \cdot \text{Ca} \cdot (\text{HCOO})_2 \cdot n\text{H}_2\text{O}$, which belongs to the calcium aluminate phase. Meanwhile, the dissolution of $\text{C}_3\text{S}$ and $\text{C}_2\text{S}$ is promoted because of the higher diffusion rate of the formate ions [39, 42]. Therefore, the coagulation time of the sample was effectively reduced by adding 30% aluminum formate.

Figure 4 shows the compressive strength of mortar with different aluminum formate substitution amounts. When the maintenance time was one day, the compressive strength of the mortar first increased and then decreased with an increase in the aluminum formate content. The strength of the mortar reached its highest value (9.5 MPa) when the aluminum formate content was 30%, which is 26.7% higher than that of the promoter without aluminum formate. When the curing time was 28 days and the aluminum formate content was 20%, the compressive strength was 43.4 MPa, which is approximately 4% higher than that of the accelerator without aluminum formate. The compressive strength data reveal that aluminum formate mainly affects its early strength, which may be related to the early strength of formate. Aluminum formate hydrolyzes formate, which permeates into the interface of $\text{C}_3\text{S}$ and $\text{C}_2\text{S}$ as hydration progresses, dissolving them and increasing the degree of hydration. Therefore, 30% aluminum formate can effectively improve the early strength of the sample, which is consistent with the results shown in Figure 3.

**Figure 2.** SEM of 1d hydration products: (a) 0% aluminum formate; (b) 10% aluminum formate; (c) 20% aluminum formate; (d) 40% aluminum formate; (e) 50% aluminum formate.

**Figure 3.** Effect of the dosage of aluminum formate on setting time: (a) Initial setting time; (b) Final setting time.
Figure 5 shows that the first peak in the cement hydration process is produced by C₃A hydration. The 30% aluminum formate takes the shortest time to appear at the first peak (figure 5(a)), which is consistent with the setting time. As the hydration reaction progresses, the second peak appears in 9–16 h, and the heat release is the largest for 30% aluminum formate (figure 5(b)). This period mainly represents the stage of one-day strength growth. Combined with figure 4, we can infer that the addition of 30% formic acid accelerates the reaction of C₃S, forming a structural phase similar to that of AFt with C₃A. Concurrently, aluminum ions in aluminum formate dissociate and participate in the hydration reaction, causing a superposition of formate and aluminum ions. The strength data shown in figure 4 also support this conclusion.

The traditional mechanism of promoting the setting of cement emphasizes too much on the role of C₃A hydration on cement setting, while the mass of C₃A generally accounts for only about 7% of the clinker mass of silicate cement, the mass of C₃S can account for 60%, the traditional mechanism of promoting the setting of cement obviously ignores the role of quick-setting agents to promote the hydration of calcium silicate. The main
The reason for the rapid hydration of cement by alkali-free liquid quick-setting agent is that C₃S rapidly hydrates to produce a large amount of C-S-H gel and Ca(OH)₂, while a large amount of heat of hydration raises the temperature of cement slurry, and a large amount of free water is combined to make the slurry lose fluidity, including the hydration products formed by the reaction between quick-setting agent and cement components. Combining the setting time, compressive strength, and XRD and SEM results, the mechanism of cooperation of aluminum formate with aluminum sulfate can be summarized as shown in figure 6. By promoting the production of large amounts of AFt, which cross-links into a mesh structure between the cement particles, rapid setting is achieved. Meanwhile, in the calcium-rich cement system, formate easily forms an AFt-like substance with C₃A, with a structure of C₃A·Ca(HCOO)₂·nH₂O. This substance belongs to the calcium aluminate phase and can effectively reduce the final setting time. The high diffusion rate of formate ions promotes the dissolution of C₂S and C₃S, which can accelerate the hydration of C₃S in cement, leading to the rapid setting of cement and improvement in its early strength [43]. Aluminum ions from aluminum formate hydrolysis are also involved in the reaction, causing a dual participation of cations and anions in hydration and forming a synergistic effect with aluminum sulfate [10, 44], as shown in the following reaction equations [33]:

\[
\begin{align*}
Al^{3+} + 4OH^- &\leftrightarrow Al(OH)_3 + 4OH^- \leftrightarrow [Al(OH)_4]^- \\
2[Al(OH)_4]^- + 6Ca^{2+} + 4OH^- + 3SO_4^{2-} &+ 26H_2O \leftrightarrow C3AA \cdot 3CaSO_4A \cdot 32H_2O \\
C3A + (HCOO)_2Ca + H_2O &\rightarrow CA_3Ca(HCO_2)_2 \cdot XH_2O
\end{align*}
\]

(5)

The introduction of aluminum formate improves the comprehensive performance of alkali-free liquid quick-setting agent, especially shortens the setting time and improves the 1d compressive strength, mainly because: the complexation effect of the aluminum salt of possible organic acids makes aluminum ions more active, and the role of formic acid itself in alkali-free liquid quick-setting agent also has the role of promoting the setting and improving the early strength, the reason is that it can promote the hydration of C₃S from; the introduction of aluminum salts in the cement system. The introduction of aluminum salts in the cement system can increase the concentration of aluminum-containing groups, which may have the effect of consuming gypsum dihydrate, so that C₃A can play a role in promoting coagulation, and it is also possible that the aluminum salts generate Al(OH)₃ in the alkaline environment of the cement slurry, which further reacts with the alkali in the cement to generate meta-aluminate, which is equivalent to the component of alkali quick-setting agent, and also shows part of the role of alkali quick-setting agent. Alkali-free liquid quick-setting agent late strength performance is very good, mainly because alkali-free liquid quick-setting agent can consume part of the alkali in the cement (their own acidic), but also to promote the role of C₂S and C₃S hydration. Comprehensive view of the alkali-free liquid quick-setting agent mechanism is more complex, is a multi-factor interaction, and ultimately achieve the promotion of coagulation, super early strength and late strength without inversion shrinkage. Replacing a part of aluminum sulfate with aluminum formate to form an accelerator can produce a synergistic effect which can accelerate the hydration of cement effectively.

4. Conclusion

We found that with an increase in the substitution amount of aluminum formate, the initial and final setting times are shortened and the compressive strength is increased, with 30% substitution providing the best results. The main reason for this is the superposition effect of formate and aluminum ions in aluminum formate, which can promote
the dissolution of C₃S and participate in the hydration of C₃A to form a phase similar to that of AFt. Therefore, the partial replacement of aluminum sulfate with aluminum formate is a feasible option for the synthesis of an alkali-free liquid accelerator, which provides a scientific basis for the development of sulfur-free liquid accelerators.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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