The Influence of Water Reducing Agents on Early Hydration Property of Ferrite Aluminate Cement Paste

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Article

The Ferrite Aluminate Cement (FAC) could rapidly lose fluidity or workability due to its excessive hydration rate, and greatly reduce the construction performance. Chemical admixtures are commonly used to provide the workability of cement-based materials. In this study, to ensure required fluidity of FAC, chemically different water reducing agents are incorporated into the FAC pastes. The experiments are performed with aliphatic water reducing agent (AP), polycarboxylic acid water reducing agent (PC) and melamine water reducing agent (MA), respectively. Influence of the water reducing agents on fluidity, setting time, hydration process, hydration product and zeta potential of the fresh cement pastes is investigated. The results show that PC has a better dispersion capacity compared to AP and MA. Besides decreasing water dosage, PC also acts as a retarder, significantly increasing the setting times, delaying the hydration rate and leading to less ettringite in the hydration process of FAC particles. The water reducing agents molecules are adsorbed on the surface of positively charged minerals and hydration products, however, for PC, steric hindrance from the long side chain of PC plays a critical role in dispersing cement particles, whereas AP and MA acting through an electrostatic repulsion force.

Keywords: chemical admixture; water reducing agent; ferrite aluminate cement; cement hydration

1. Introduction

As the most important building material, Portland cement has been widely used in engineering construction worldwide. However, in the manufacturing process of Portland cement clinker, the decarbonation and calcination process produce approximately 5–8% of global anthropogenic CO\(_2\) emissions [1–5]. One of the ways to reduce CO\(_2\) emission from Portland cement production is to replace Portland cement with Calcium sulfoaluminate. Compared with Portland cement clinker, the CO\(_2\) emissions are relatively lower owing to the lower production temperature and lime saturation factor of Calcium sulfoaluminate clinker [6–9].

Calcium sulfoaluminate cement is often used for emergency repairing including bridge decks, airport runways, municipal roads and so on, due to its better performances than PC, such as rapid setting, negative hardening temperature, high early strength, small volume expansion and anti-permeability [10–12]. Therefore, Calcium sulfoaluminate cement is considered as an alternative binder to PC [13]. However, despite the increasing interests and excellent advantages of this cement, the depletion of alumina-bearing raw materials such as bauxite is also the challenging issue of the Calcium sulfoaluminate-based materials, which limits its industrial-scale production and wider application [8,14]. Therefore, lowering the consumption of aluminum material and seeking alternative alumina sources are inevitable and essential requirements in the Calcium sulfoaluminate clinker production. In recent years, even though red mud and fly ash, which are Al-rich industrial wastes, have been successfully used to produce Calcium sulfoaluminate clinker in the laboratory, drawback of
these industrial wastes is their low Al₂O₃ content, owing to the high content of SiO₂ [15–19]. These industrial wastes still can not meet the requirement of aluminum materials in Calcium sulfoaluminate raw meal [14]. To reduce the demand for bauxite, another strategy is to partially substitute the aluminum material in Calcium sulfoaluminate cement with ferrite material [20–22]. The ferrite aluminate cement (FAC) is prepared by partially replacing Al₂O₃ in Calcium sulfoaluminate clinker with Fe₂O₃, and the substitution level depends on the F/A ratio of the raw mixtures [23]. The FAC paste will rapidly lose fluidity or workability due to its excessive hydration rate, and greatly reduce its construction performance. Water reducing agents is a kind of important chemical admixture for Portland cement-based materials [24–27]. They can increase the workability of fresh concrete without additional water, or reduce the amount of water for a given fluidity [28–31]. At present, very few researches concerning the effect of water reducing agents on the early performance of FAC paste are reported, and how the water reducing agents regulate the hydration process of FAC paste is still not clear. In the present work, the impact of water reducing agents on the early properties of FAC is investigated by evaluating the fluidity, setting time, hydration process, hydration product and zeta potential of fresh pastes. The results data of chemically different water reducing agents in FAC can provide a fundamental understanding for the industrial application.

2. Experimental Section

2.1. Materials

Water Reducing Agent. Aliphatic water reducing agent (AP) and polycarboxylic acid water reducing agent (PC), melamine water reducing agent (MA), were used to investigate the compatibility with ferrite aluminate cement (FAC), respectively. The AP (powder) and PC (solution, solid content: 50.5%), MA (powder) were purchased from BASF Advanced Chemicals Co., Ltd., Shanghai, China.

Cement. The FAC used in the experiment was obtained from Yicheng Anda Special Cement Co., Ltd., Yicheng, Hubei, China. And its chemical composition is shown in Table 1.

Table 1. The Chemical Composition of FAC.

| Chemical Composition | CaO  | SiO₂ | Fe₂O₃ | Al₂O₃ | MgO  | SO₃  | Other |
|----------------------|------|------|-------|-------|------|------|-------|
| Weight (%)           | 44.53| 6.58 | 7.10  | 18.60 | 2.39 | 14.34| 6.46  |

2.2. Characterization and Test

X-ray Diffraction (XRD): After curing to the test age, the hydration of FAC pastes was stopped by submerging the crushed pastes in anhydrous ethanol for 24 h. Then, the sample was ground in agate mortar. Crystal phases were characterized by XRD (Supernova, Japan, Cu Kα) in the range from 5° to 60°, with operating voltage of 40 kV, working current of 40 mA, and step size of 0.02°.

Fluidity: 300 g of FAC, water reducing agent and water were mixed by stirring at low speed for 2 min in a mixer. After that, the FAC mixture was allowed to rest for 15 s and then stirred at high speed for 2 min. After mixing, the mixture was transferred into a mini-slump cone open at both ends (upper diameter of 36 mm, bottom diameter of 60 mm, and height of 60 mm) to measure the fluidity. For all samples, the ratio of water to FAC (Water/Cement) was 0.29.

Setting Time: After fluidity test, the pastes were placed into a standard curing cabinet to study the effect of water reducing agents on the setting time of FAC, and Vicat apparatus was used according to Chinese National Standard GB/T 1346-2011. The temperature was 20 ± 1 °C, and the relative humidity was 95 ± 1% in the standard curing cabinet.

Calorimetric Measurements: An isothermal calorimetry (TAM Air, TA instruments, USA) was performed to determine how water reducing agents influence FAC hydration kinetics with Water/Cement ratio of 0.29. The tests of hydration exothermic rate and hydration heat during the first 30 h were executed at 25 °C. After initial FAC and water reducing
agent solution contact in ampoule, the ampoule (11.61 g FAC paste) was closed and placed into the instrument as soon as possible. After 30 h, the measurement was stopped, and the hydration exothermic rate and hydration heat was obtained. Furthermore, a reference sample without water reducing agent was simultaneously prepared and measured.

Zeta potential: Zeta potential of FAC pastes with different water reducing agents was determined by a zeta-potential instrument (NanoZS90, Malvern Instruments, Ltd., Malvern, UK) with a fixed. The Water/Cement ratio was fixed at 50. The FAC was added to water containing a certain amount of water reducing agents, and the suspension was poured into the sample cell. Then, zeta potential tests were conducted.

3. Results and Discussion

3.1. Minerals of FAC Particles

It could be seen from Table 1 and Figure 1 that a FAC particle contained cement minerals and gypsum in interstitial phase. The cement minerals contained calcium sulfoaluminate (C₄A₃S) and belite (C₂S), and Al was partially replaced by Fe (C₄A₃₋xFₓS) in C₄A₃S.

3.2. Structure of Water Reducing Agents

AP was an anionic surfactant with chain-like molecular structure, containing carboxylate, sulfonate and hydroxyl active groups. The second employed polymer was comb-shaped PC, which consisted of main backbone with carboxylate groups and side chains. The third polymer-based water reducing agent used was MA, which was a water-soluble melamine sulfonate formaldehyde polymer with strong anions. The molecular structure of AP, PC and MA were presented in Figure 2, respectively.

![Figure 1: FAC minerals.](image1)

![Figure 2: Molecular structure of three water reducing agents.](image2)
3.3. Flow Test of FAC

In measuring process of dispersing properties for the water reducing agents, proportions of water reducing agents added into the samples were investigated for a given fluidity (180 mm, 240 mm, 270 mm, respectively). Figure 3 exhibited the fraction variation of water reducing agents with fluidity of FAC paste. The FAC paste almost could not flow when the water reducing agents were not added, and the fluidity value was too small to be recorded. It could be seen from Figure 3 that addition of the water reducing agents indeed improved the fluidity of FAC paste. When the fluidity was 180 mm, dosages of the water reducing agents were 5‰ (AP), 0.84‰ (PC) and 4‰ (MA), respectively. An increased dosage was observed for higher fluidity. The dosages increased to 5.5‰ (AP), 1.5‰ (PC), 5‰ (MA) and 7‰ (AP), 2.5‰ (PC), 6.5‰ (MA), when fluidity increased to 240 mm and 270 mm, respectively. For a given fluidity (180 mm, 240 mm and 270 mm), dosage of the water reducing agents was in the following order: AP > MA > PC. In other words, compared with AP and MA, PC had a better dispersion capacity.

![Figure 3](image)

**Figure 3.** Flow test of FAC pastes.

3.4. Setting Time of FAC

Setting time of the FAC pastes mixed with different water reducing agents was presented in Figure 4. The figure indicated that the initial and final setting times of blank FAC paste (without water reducing agents) were 1.05 h and 1.41 h, respectively. It could be clearly seen that both initial and final setting times of FAC pastes increased with the water reducing agents added. The setting time of AP modified FAC pastes increased slightly when the AP water reducing agent dosage increased from 0‰ to 7‰. For a given fluidity (180 mm, 240 mm and 270 mm), setting time of the FAC pastes mixed with MA water reducing agent was longer than that with AP. However, initial and final setting times of the pastes increased significantly with increasing dosage of the PC water reducing agent. When the content of PC water reducing agent was 2.5‰, initial and final setting times of the FAC paste were 480% (6.08 h) and 400% (7.16 h) higher than those of blank FAC paste.

3.5. Early Hydration Kinetics of FAC

Hydration kinetic comparisons of the FAC pastes with different water reducing agents at the fluidity of 240 mm was conducted. Hydration exothermic rate and accumulative heat of these FAC pastes with ongoing hydration process were shown in Figures 5 and 6, respectively. As shown in Figure 5, the hydration process of FAC particles could be divided into five stages: the initial reaction period firstly after adding water, then transited to the induction period, then entered the acceleration period, finally gradually shifted into the deceleration period and the stable period, which was similar to that of Portland cement. However, unlike Portland cement, multiple consecutive exothermic peaks were presented due to the formation of ettringite in the hydration process of FAC particles.
Figure 4. The effect of water reducing agents on setting times of the FAC pastes.

Figure 5. Heat flow curves for FAC pastes from 0 to 30 h.

Figure 6. Cumulative heat curves for FAC pastes from 0 to 30 h.

As Figures 5 and 6 illustrated, a change in the hydration induction period of FAC pastes was observed for three types of water reducing agents used. Presence of these water reducing agents retarded occurrence of the heat evolution peak. Time for dissolution of FAC particles was increased by 1.5 h for paste mixed with AP, 4.7 h for PC, and 1.8 h for MA. Duration of the induction period for PC modified paste was prolonged significantly. Obviously, in comparison with the reference paste, the hydration heat released from PC modified paste before 6 h lowered down greatly, due to its longer setting time. However, the released hydration heat of AP modified paste and MA modified paste before 6 h was not affected. These results were in accordance with the setting times. After 24 h, cumulative heat for the reference paste, and AP, PC, MA modified pastes was 181.68 J/g, 181.49 J/g,
184.83 J/g and 173.14 j/g, separately. It indicated that the water reducing agents did not affect hydration degree of FAC particles within 24 h except for MA.

3.6. XRD Analysis

As water reducing agents were observed to have a significant retarding effect on the hydration kinetics of FAC (as illustrated in Figures 5 and 6), the effect of water reducing agents on phase development at the fluidity of 240 mm was further investigated through XRD studies. XRD patterns for the samples containing water reducing agents hydrated for different curing ages were illustrated in Figure 7. It could be seen from Figure 7 that at the initial stage of hydration (within 5 h), in contrast to FAC material, a large amount of Ye’elimite and gypsum were dissolved in the blank paste, and a small amount of ettringite formed, of which the reaction mechanism was shown in Equation (1). After 11 h, peak intensity of the hydration product increased, indicating that more ettringite formed. While, because Al(OH)_3 and Fe(OH)_3 gels were poorly crystallized or amorphous, they were not detected in the pastes. It was observed that the incorporation of PC significantly lowered the characteristic peak of ettringite compared with the blank FAC sample at the ages of 5 h and 11 h, which is consistent with the setting time and hydration heat tests. However, for AP and MA modified pastes, there was an increasing trend for the diffraction peak intensity of ettringite, because of their less effect on the setting time.

$$\text{C}_4\text{A}_3-x\text{F}_x\text{s} + 2\text{CS}_2\text{H}_2 + 34\text{H} \rightarrow \text{C}_6\text{A}_{1-y}\text{F}_y\text{sH}_{32} + (x - y)\text{FH}_3 + (2 - x + y)\text{AH}_3$$  \hspace{1cm} (1)

![Figure 7. XRD patterns of samples hydrated for 5 h and 11 h (1-ettringite, 2-Ye’elimite, and 3-gypsum).](image)

3.7. Zeta Potential of the Cement Suspension

In order to analyze the interaction mechanisms between FAC particle and water reducing agents, zeta potential of various FAC pastes was tested. Effects of the water reducing agents on the zeta potential of the FAC pastes were presented in Table 2. As we could see from Table 2, the zeta potential of FAC particles had a positive value (+0.53 mv) due to partial dissolution of C_4A_3S, C_4A_3−xF_3s and formation of ettringite. When the water reducing agents were added, these negatively charged molecules were adsorbed on surface of the positively charged C_4A_3S, C_4A_3−xF_3s and ettringite, and then zeta potentials of all the FAC samples were negative. In the meantime, adsorption of water reducing agents reduced the interactions between the FAC particles, so that the whole paste system became more stable.
Table 2. Effect of Water Reducing Agents on Zeta Potential of the Cement Suspension.

| Types   | Dosage (%) | Zeta Potential (mv) |
|---------|------------|---------------------|
| FAC particle | 0          | +0.53               |
|          | 5          | −15.50              |
|          | 5.5        | −16.20              |
|          | 7          | −17.40              |
| AP      | 0.84       | −0.74               |
|          | 1.5        | −0.90               |
|          | 2.5        | −1.28               |
| PC      | 4          | −12.10              |
|          | 5          | −13.60              |
|          | 6.5        | −13.90              |
| MA      |            |                     |

With PC added, zeta potentials (between −0.74 mV and −1.28 mV) of the FAC pastes slightly decreased. However, when MA was added, it induced lower potentials between −12.1 mV and −13.9 mV significantly. The effect of AP was similar to that of MA. Zeta potential values (between −15.50 mV and −17.40 mV) of the AP modified FAC pastes were slightly lower than that of MA modified FAC pastes. Therefore, for AP and MA, the strong decline of zeta potential to more negative values indicated that electrostatic repulsion was the main force for dispersion. While the slightly decreased zeta potential of the PC modified FAC particles indicated that steric repulsion caused by the PC side chains was the key factor for dispersion (Figure 8).

Figure 8. Schematic illustration for the adsorption of water reducing agents on FAC particle surfaces: (a) PC water reducing agent, (b) AP and MA water reducing agent.

4. Conclusions

In this paper, three types of water reducing agents as dispersants were incorporated into ferrite aluminolate cement to increase its fluidity. The effect of water reducing agents on the early hydration of ferrite aluminolate cement was studied using isothermal calorimetry, XRD, and zeta potential analysis. The results showed that PC was much more efficient for increasing fluidity of the FAC pastes than AP and MA. However, in comparison with AP and MA, PC water reducing agent increased significantly setting times of the pastes, and delayed immensely hydration rate and formation of hydration product ettringite in the hydration process of FAC particles. The action mechanism of PC water reducing agent was confirmed to be mainly steric resistance, whereas AP and MA acted through an electrostatic repulsion mechanism.
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