



Influence of tartaric acid on early hydration and mortar performance of Portland cement-calcium aluminate cement-anhydrite binder



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HIGHLIGHTS

- Tartaric acid delays ettringite formation and suppresses early hydration of silicate.
- Inhabiting ettringite formation is critical to get satisfactory fluidity.
- Tartaric acid delays ettringite formation by poisoning nuclei growth mechanism.
- Ettringite amount is another factor suppressing early hydration of silicate.

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ABSTRACT

The influence of tartaric acid on early hydration and mortar performance of Portland cement-calcium aluminate cement-anhydrite ternary binder is investigated in the presence of polycarboxylate-based superplasticizer. Tartaric acid delays the ettringite (AFt) formation and suppresses the early hydration of silicate. Good mortar fluidity is obtained only above a critical tartaric acid dosage that can inhibit AFt formation completely. However, the silicate hydration will be suppressed significantly at the same time, resulting in poor early strength. Tartaric acid delays the AFt formation mainly by the mechanism of poisoning nuclei growth. AFt amount is another factor that suppresses the early hydration of silicate. In the ternary binder system with higher CAC and CS content, less tartaric acid can suppress the early hydration of silicate completely until AFt transforms into monosulfoaluminate (AFm) to unlock it.

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1. Introduction

Binders basing on Portland cement (PC), calcium aluminate cement (CAC) and calcium sulphate (CS) can achieve special performance such as fast setting, rapid hardening, high early strength and shrinkage compensating which cannot be gained using PC alone [1,2]. In building materials industry, CAC is an essential component of the formulation of many functional mortars. The typical applications are tile adhesive, self-levelling mortar, grouting mortar and rapid-hardening repair mortar [3].

Many researchers have reported the complex hydration process of PC/CAC/CS ternary binder. When PC is only replaced by part of CAC to form the PC/CAC binary binder, rapid setting and strength development within a few hours can be observed [4], but the

hydration of PC is delayed [5–7] due to a barrier layer consisting of mainly ettringite (AFt) covering the surface of unhydrated PC grains [6]. Adding extra CS to form PC/CAC/CS ternary binder can accelerate the C₃S hydration to improve the late strength, but the microstructure and hydration processes strongly depend on PC/CAC and CAC/CS ratios [8–10]. According to David Torrén-Martín et al. [9]: If PC is the main compound, with low CAC and CS amount, the hydration process is similar to the PC hydration; In the cases where the CAC amount is high and the CS amount is low, the AFt forms first, then the monosulfoaluminate (AFm) formation unlocks the silicate hydration; If both the CAC and CS amounts are high, the AFt formation is the first process, then the silicate hydrates, finally the remaining aluminates reacts.

Although there are still some unknowns regarding the exact hydration mechanism involved in PC/CAC/CS binder, all the studies mentioned above have revealed that the hydration process is associated with formation time, amount and later transformation

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of Aft. In addition to the PC/CAC and CAC/C \bar{S} ratios, other factors will also have an impact on the hydration process as long as they can change the formation process of Aft. These factors include temperature, humidity, raw materials compositions and chemical admixtures. Several studies have demonstrated that the sources of calcium sulphate have obvious effects on hydration and performance of the ternary binder because of their different solubility and dissolution rate [11–13], and the effect is different at different temperature [14]. Moisture curing can promote the Aft formation [12]. CA (monocalcium aluminate) is the main phase of CAC. It has been found that the hydration process and performance of both PC/CAC binary binder and PC/CAC/C \bar{S} ternary binder will change when adopting different grade CAC [15,16]. High grade CAC with higher content of CA will result in more Aft formation at same time. But until now, how different chemical admixtures alter the hydration process has rarely been reported, although lithium salts promoting the hydration of PC/CAC binary binders has been studied [17,18].

It must be emphasized that, in the typical application of the PC/CAC/C \bar{S} ternary binder such as self-levelling or grouting mortar, not only strength and expansion ratio, but also fluidity and fluidity retaining time are required. So besides superplasticizer (SP), retarder is an indispensable admixture used to control fluidity loss, which will alter the hydration process inevitably by affecting the formation of Aft. Tomohiro Emoto adopts tartaric acid as the retarder in CAC/C \bar{S} rich binder of self-levelling mortar, and citric acid or sodium citrate is used in PC rich binder [19]. G. C. Sang adopts citric acid in grouting mortar [20]. In another study on grouting mortar [21], citric acid or tartaric acid used alone cannot achieve good fluidity, but tartaric acid combined with sodium gluconate works well. However, all these researchers have not illuminated the mechanisms how the retarder functions and why the same retarder functions oppositely in different cases. J. Plank interprets the incompatibility problem between PC and citrate in self-levelling mortar from the point of competitive adsorption [22]. According to his study, citrate has stronger competitive adsorption ability to influence SP adsorption than tartaric acid, so tartaric acid performs well in self-levelling mortar than citrate. But he does not interpret the difference of hydration process resulted from tartaric acid and citrate.

In the studies of grouting mortar, we find similar phenomenon as the literature [21] describes. In most cases, adding tartaric acid at enough dosage can satisfy the fluidity requirement. But in all cases, combining sodium gluconate with tartaric acid will always be beneficial to both fluidity and 1-day strength, especially when variety of PC or CAC exists. In order to understand the mechanisms behind these, a subject is established to study the synergic effect of sodium gluconate and tartaric acid on early hydration of PC/CAC/C \bar{S} ternary binder. As the first step of the subject, the effect of tartaric acid on early hydration and mortar performance of PC/CAC/C \bar{S} ternary binder is investigated in this paper.

2. Materials and methods

2.1. Materials

Portland cement type II52.5R and calcium aluminate cement CA50 used in this study were obtained from JinYang Cement Co. and ChangChen Cement Co. respectively, and a commercial natural anhydrite was from Nanjing. The chemical compositions of the cements determined by X-ray fluorescence (XRF) are shown in Table 1.

Table 1
Chemical compositions of cements (wt%).

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	TiO ₂	SO ₃	K ₂ O	Na ₂ O
PC	65.42	21.57	5.02	3.35	0.77	0.25	2.72	0.41	0.17
CAC	34.62	8.27	50.71	1.92	0.84	2.76	–	–	–

L+ tartaric acid was from ChangMao Biological Chemistry Co. and polycarboxylate-based superplasticizer (SP) in dry powder form was from XinBang Co. The river sand was smaller than 1.25 mm.

2.2. Methods

Two kinds of ternary binders with different PC/CAC ratio and constant CAC/C \bar{S} ratio were adopted, which were typical binders of grouting mortar in china for different expansion rate request. The formulations of binders, mix proportions of the samples and relative measurements are listed in Table 2.

The mortar samples were prepared and tested according to the Chinese Industry Standard JC/T 986-2005. For the strength test, mortar was poured into 40 × 40 × 160 mm steel mould without vibration, which was demolded after 24 h and cured at 22 ± 1 °C and a relative humidity of 95%. Flexural and compressive strength were measured after 1, 3 and 28 days. The fluidity was measured using a truncated cone shaped mould with a height of 60 mm, a top inner diameter of 70 mm and a bottom inner diameter of 100 mm. Measurement started immediately after mixing. The spread width in two perpendicular directions on a glass plate was measured after the mortar did not spread. The average was taken as the initial fluidity. The mortar was collected and remixed and measured again after 30 min.

The setting times of pastes were determined according to ASTM C191 using a Vicat apparatus.

An isothermal heat-conduction calorimeter with 8 channels (TAM air C80, Thermometric, Sweden) was used to measure the early hydration heat evolution of the binders. The paste was prepared mixing the binder and water for 3 min in the vial and after that, it was introduced into the calorimeter sample compartment. The experimental temperature was 20 ± 0.1 °C. Data logging was continued for about 72 h.

The samples for XRD and DSC analysis were also prepared according to ASTM C191. At certain hydration time, small pieces of the hydrated pastes were soaked in alcohol for 24 h to stop hydration. The samples were then dried at 40 °C, ground to <63 μm and used for XRD and DSC analysis. XRD analysis was performed on a Bruker D8 system using CuK radiation in the range of 5°–20–60°2θ, with step of 0.04°/s. DSC analysis was carried out on a NETZSCH/STA449F3JUPITER system, sample was heated under N₂ over a temperature range of 30–650 °C at a rate of 20 °C/min.

3. Results

3.1. Mortar performance

3.1.1. Compressive strength

Fig. 1(a) and (b) show the compressive strength of mortar for these two ternary binders. Tartaric acid has less influence on 28d compressive strength for both mortars, and the mortar with higher CAC content has lower strength. Lower dosages of tartaric acid have less influence on the 1d and 3d compressive strength, and no obvious difference between two mortars can be observed. When the tartaric acid dosage is higher than a critical value (0.12% in Fig. 1(a) and 0.06% in Fig. 1(b)), the 1d and 3d compressive strength of both mortars are decreased obviously, but the mortar with higher CAC content has higher strength.

3.1.2. Fluidity

As seen in Fig. 2(a) and (b), without tartaric acid, the mortar with higher CAC content has no initial fluidity at all (bottom inner diameter of the mould is 100 mm). Tartaric acid significantly increases the initial fluidity of both mortars especially for that with high CAC content, and both mortars have the same saturated tartaric acid dosage of 0.12%, above which, the initial flow will not increase further.

Both mortars get good 30 min fluidity only when the tartaric acid dosage reaches a critical value (0.18% in Fig. 1(a) and 0.24% in Fig. 1(b)). The higher CAC is contained, the more tartaric acid dosage is needed to control the fluidity loss. But as shown in Fig. 1, an obvious decrease of 1d and 3d compressive strength is inevitable at this case.

3.1.3. Setting time

At lower dosage, tartaric acid prolongs the initial setting time and final setting time a little for both ternary binders (Fig. 3(a) and (b)). When the tartaric acid dosage >0.12% for 92.5/7.5/3.75

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