



Effect of calcium sulfate type and dosage on properties of calcium aluminate cement-based self-leveling mortar

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HIGHLIGHTS

- Three types of calcium sulfate were utilized to study properties of self-leveling mortar.
- Anhydrite is more suited to SLM for the lowest fluidity loss and longer setting time.
- Formation of ettringite can limit the hydration of PC in ternary cementitious system.
- The quantity and morphology of ettringite depend on the type of calcium sulfate.

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ABSTRACT

In this paper, the influence of calcium sulfate type (α -hemihydrate, anhydrite and dihydrate) and amount (0%, 2%, 4% and 6%) on the performances of calcium aluminate cement-based self-leveling mortar (SLM) were investigated. The influence of calcium sulfate on fluidity and mechanical properties was investigated. Moreover, the impact of calcium sulfate on the hydration products, morphology and heat evolution of pastes hydrated up to 3 days were determined by using X-ray diffraction (XRD), Scanning electron microscope (SEM) and isothermal heat-conduction calorimeter. Test results revealed that SLM prepared with anhydrite exhibited the highest initial flow value (~ 160 mm) and setting time and the lowest fluidity loss due to the slow dissolution rate of anhydrite compared to other calcium sulfate. In comparison to control formulation, the enhancement in flexural and compressive strength for T4 (dihydrate) was the highest and was found to be 485.71% and 437.5% respectively and hence it can be concluded that dihydrate is favorable to the early strength of SLM as compared to other SLM formulations containing α -hemihydrate and anhydrite respectively. The shrinkage compensating effect became more obvious with the increase in the amount of C\$ in SLM samples. Due to the fast dissolution rate, mortar containing α -hemihydrate exhibited more expansion as compared to other calcium sulfate. SLM prepared with dihydrate showed the highest early strength due to the lowest influence on C₃S hydration while the influence of α -hemihydrate was more obvious. Hence, the hydration of PC in the ternary system can be limited by increasing calcium sulfate due to the formation of ettringite. Finally, the type of calcium sulfate has an important influence on the quantity and morphology of ettringite.

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1. Introduction

Cement-based self-leveling mortar (SLM), one kind of functional building mortar, is made by mixing cementitious materials, aggregate and mineral admixtures as well as chemical admixtures. When SLM is used, it should be excellent fluidity, pumpability and

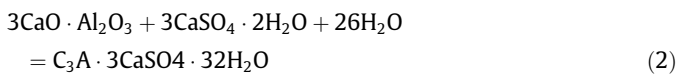
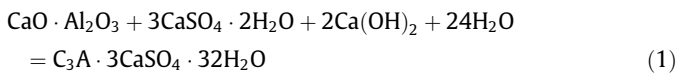
self-leveling features to form a smooth surface after slight paving. In order to meet the above-mentioned construction conditions, SLM should possess some properties such as proper setting time, greater flowability, higher early strength, good segregation resistance and stable volume change. Generally, when Portland cement (PC) is only used as cementitious material to prepare SLM, cracking and curling is reported to occur at the surface and corner of SLM due to slow hydration rate, lower early strength and severe drying shrinkage of PC [1]. Hence, calcium aluminate cement (CAC) [2,3] and calcium sulfoaluminate cement (CSC) [1,4] have been used to

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prepare self-leveling compounds to achieve higher early strength, rapid hardening and shrinkage compensating cementitious systems. In these cementitious systems, ettringite acts as the expansion source, which can compensate dry shrinkage and enhance the early strength of SLM.

Compared to CSC, the calcium aluminate cement can not generate ettringite during its hydration process. Hence, CAC is usually used with calcium sulfate (C\$) or PC as binary cementitious system (PC/CAC) and ternary cementitious system (PC/CAC/C\$). In the binary cementitious system, ettringite generated will hinder PC hydration by covering the surface of unhydrated PC grains and is detrimental to strength development of self-leveling compounds at later age. As far as the ternary cementitious system is concerned, the presence of excess calcium sulfate can promote the hydration of C₃S and enhance the strength development at later age. It is pertinent to mention that in ternary cementitious system, the cementitious components ratio has significant influence on microstructure and hydration process. The aforementioned ettringite formation reactions in these cementitious system are as follows [5]:



The amount and type of calcium sulfate play important roles in the formation of hydration products. When calcium sulfate is consumed totally and excess calcium aluminate exists, then it will react with ettringite and will result in the formation of monosulfoaluminate [6–8], which in turn, will increase the porosity of cement-based materials. However, the conversion from ettringite to monosulfoaluminate can be reduced by increasing the calcium sulfate dosage. On the other hand, the dissolution rate of calcium sulfate is also an important factor contributing to ettringite formation. Cementitious system containing hemihydrate can generate more ettringite at early hydration due to the higher dissolution rate of hemihydrate and hence which will enhance the fluidity loss and hydration heat [9,10]. Compared to hemihydrate, anhydrite has a slower dissolution rate and will reduce the formation of ettringite. This in turn would enhance the strength development of mortar under low temperature (0–10 °C) [11].

In recent years, the hydration properties of binary or ternary cementitious systems, which are important for preparation and engineering application of SLM were studied in detail. Zhang et al. [12] used tartaric acid to control the setting time of ternary cementitious system containing PC, CAC and anhydrite. The authors found that tartaric acid can restrain the hydration of calcium silicate and delay the formation of ettringite, which in turn, prolonged the setting time of system and improved the flowability of mortar. Xu et al. [13] evaluated the effect of different curing temperature on hydration process of ternary cementitious system (PC/CAC/C\$) and found that with the rise in the curing temperature, the transformation from ettringite (AFt) to calcium monosulfoaluminate hydrated (AFm) was accelerated. Moreover, the rise in temperature, influenced the porosity distribution of paste. Kastiukas et al. [14] investigated the influence of citric acid and lactic acid on properties of binary cementitious system (PC/CAC). The authors found that a low amount of citric acid reduced the strength of binary system while the lactic acid was found to improve the early strength development. Bizzozero et al. [15] investigated the influence of limestone in a ternary cementitious system containing CAC and calcium sulfate and found that the role of limestone is depended on the amount of calcium sulfate in the system.

Although many researchers have investigated the hydration mechanism and material properties of ternary cementitious system (PC/CAC/C\$), however, as per authors knowledge, the investigations related to different types of calcium sulfate on the properties and hydration process of SLM are scarce. Hence, in this research, three types of calcium sulfate (α -hemihydrate, anhydrite and dihydrate) were employed to prepare calcium aluminate based SLM. The influence of calcium sulfate on fluidity and mechanical properties was investigated. Moreover, the impact of calcium sulfate on the hydration products, morphology and heat evolution of pastes hydrated up to 3 days were determined by using X-ray diffraction (XRD), Scanning electron microscope (SEM) and isothermal heat-conduction calorimeter to evaluate the mechanism of three types of calcium sulfate as well as to provide theoretical basis for preparation and construction of self-leveling compounds.

2. Experimental

2.1. Materials

The Portland cement (type CEMIII52.5) and calcium aluminate cement (Ternal CC) were obtained from Jiangnan-xiaoyetian Cement Co. and Kerneos Co. respectively. Three types of calcium sulfate (α -hemihydrate, anhydrite and dihydrate) having specific surface area of 370, 648, 516 m²/kg respectively were obtained Tianjing. The chemical composition of the cements and calcium sulfates determined by X-ray fluorescence (XRF) is shown in Table 1. The fine aggregates were composed of coarse quartz sand (40–60 mesh) and fine quartz sand (60–120 mesh) while fine limestone (400 mesh) was used as mineral filler. Finally, the chemical admixtures used in this research contained re-dispersible polymer powder, polycarboxylate-based superplasticizer (SP) dry powder, retarder, anti-form agent and cellulose ether.

2.2. Mix proposition and sample preparation

Three types of calcium sulfate (α -hemihydrate, anhydrite and dihydrate) abbreviated as A, N and T, respectively were used for making SLM. In total, ten formulations with different calcium sulfate amount (0%, 2%, 4% and 6%) and constant CAC amount were used (C, A2, A4, A6, N2, N4, N6, T2, T4, and T6). The details of SLM mix formulations are given in Table 2 in which all the components were calculated by mass percent. It is known that excess amount of calcium aluminate cement delays the acceleration period of the PC dominated ternary cementitious system hydration [6], which in turn, delays the hydration of silicates resulting in a severe influence on the strength development of SLM. Hence, the amount of CAC was fixed as 4%, which was around 10% wt. of the ternary cementitious binder. The dosage of polycarboxylate superplasticizer, redispersible emulsion powder, tartaric acid, hydroxypropyl methyl cellulose ether (HPMC) and antifoamer was fixed as 0.2 wt%, 1.2 wt%, 0.06 wt%, 0.02 wt% and 0.12 wt% of dry mortar powder (cementitious materials, sand and filler) respectively. Therefore the dosages of all chemical admixtures were fixed as 1.60 wt% of dry powder. The dosage of aggregates, filler and chemical admixtures were fixed while the water to total powder ratio was kept as 0.185.

According to the above mix formulations, all materials were (except water) mixed at a slow speed for 30 s. Then water was added and the mixture was mixed at slow speed for another 60 s. Thereafter, the SLM was mixed at higher speed for another 60 s. The fluidity and setting time of SLM were measured immediately after mixing. For other tests, mortar was poured into molds having size of 40 × 40 × 160 mm and cured at 22 ± 1 °C and 95% relative humidity (RH). The SLM samples were demolded after 24 h.

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