



Experimental study of calcium sulfoaluminate cement-based self-leveling compound exposed to various temperatures and moisture conditions: Hydration mechanism and mortar properties



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ABSTRACT

The hydration mechanism and mortar properties of CSA cement-based SLC under various temperatures and moisture conditions have been investigated experimentally. The phase assemblage and hydration process were examined by XRD and DSC-TG analysis. Morphology and microstructure of hardened mortars were observed by SEM. Mortar properties including flowability, setting time, strength and dimensional change were measured. The results indicate that elevated temperatures accelerate the hydration process, leading to phase assemblage variation and heterogeneous microstructure. Despite of worse flowability and shorter setting time, strength is increased and dimensional change is reduced by increasing temperature. At each individual temperature, water curing has little influence on the long-term hydration process. It degrades the strength but is beneficial to small absolute dimensional change. Contrary to air-cured samples, all water-cured samples swell instead of shrinking. The compressive strength has a positive relation to the Aft/AFm.

1. Introduction

In the last decades, self-leveling compound (SLC) is widely used all around the world due to increasing labor cost and customer's performance demand. It is a kind of highly developed ready-to-use dry mixture which can be applied just by mixing with a defined amount of water on site [1,2]. SLC is mostly applied as thin layers of 1–20 mm thickness on different types of substrates for the purpose of renovating the uneven substrates in old buildings or creating a flat and smooth ground surface for final flooring in new buildings.

The production of commercial cementitious SLC is based on a binder system where various admixtures (organic and inorganic) and/or mineral fillers are used to guarantee specific workability [1]. Typically, the binder system is consisted of Portland cement (PC), calcium aluminate cement (CAC), and calcium sulfate ($C\bar{S}H_x$, $x = 0, 0.5$ or 2), because such ternary system can provide the principle properties like rapid setting, high early strength and dimensional stability. Many previous studies have discussed and confirmed that ettringite ($C_3A \cdot 3C\bar{S}H_{32}$, Aft), one of the major crystalline hydrates in this system, is the key parameter to achieve those properties [3–7]. Despite of the excellent performance, the large CO_2 emission and energy consumption associated with PC production have become environmental downsides

urged to be solved in the application of SLC. Besides, utilization of various chemical admixtures, as well as CAC, makes the cost of SLC relatively high.

To overcome these disadvantages, calcium sulfoaluminate cement (CSA) is regarded as a more eco-friendly and economic alternative of the traditional ternary system for SLC. Compared with PC, CSA clinker (mainly $C_4A_3\bar{S}$ named ye'elimite) requires less limestone, lower calcination temperature and better friability during manufacture, which results in less CO_2 emission and energy consumption [8–12]. Other than environmental advantages, CSA cement can also achieve outstanding properties like rapid setting, high early strength, shrinkage compensating and even self-stressing just by varying the amount of calcium sulfate addition [13–16], potentially reducing the cost of adding all sorts of chemical admixtures.

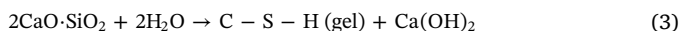
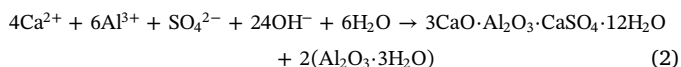
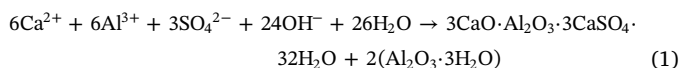
There are generally two primary mineral phases (ye'elimite and belite) presenting in CSA cement. The hydration reactions of ye'elimite can be indicated by chemical Eqs. (1) and (2) [10,13], where ye'elimite dissolves to provide calcium, aluminum and partial sulfate, while gypsum and/or anhydrite are the main source of sulfate. The hydration reaction of belite is indicated by chemical Eq. (3), where belite dissolves as the source of calcium and silicon. In the presence of calcium sulfate, Aft is generated first accompanied with poorly-crystallized or

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Table 1
Chemical and mineralogical composition of CSA clinker and anhydrite (wt%).

Chemical composition																
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Gr ₂ O ₃	Fe ₂ O ₃	SrO	ZrO ₂	WO ₃	
CSA clinker	0.04	0.73	37.5	5.8	0.11	9.7	0.05	0.22	41	1.5	0.02	3.1	0.07	0.06	0.02	
Anhydrite	0.04	3.00	0.12	1.4	/	50.1	0.02	0.05	44.8	/	/	0.04	0.44	/	/	
Mineralogical composition																
CSA clinker	C ₄ A ₃ S̄			C ₂ S			C ₄ AF			CT						
	69.50			16.65			9.42			2.55						
Anhydrite	C̄S			Dolomite												
	85.18			9.86												

amorphous aluminum hydroxide (AH₃). Once the sulfate is insufficient in the pore solution, monosulfate (C₃A·C̄S̄·H₁₂, AFm) is formed. Meanwhile, C-S-H gel precipitates due to the reaction of belite, although it hydrates much slower than ye'elimite. The other hydration product Ca(OH)₂ may not exist in the final matrix because of the low concentration of calcium with respect to the precipitation of this phase.



It has been well documented that various temperatures and moisture have significant influence on not only the phase assemblages but the microstructure as well, which can lead to quite different performances of SLC [17–21]. As reported in Ref. [21], the availability of mixing water in the different layers of the SLC influences the maximum content of Aft which can potentially be formed within the hardened SLC. Besides, Aft is more likely to convert to AFm at elevated temperatures [18–19,22]. The latter is a plate-like phase contributing little to the strength and dimensional stability but much to the protection of chloride attack [23]. The transformation is also affected by the activity of moisture: at very low RHs, the nucleation of AFm is hindered due to the absence of an aqueous phase, and Aft may decomposed to metaettringite (which is amorphous with a water content of 9 to 13 H₂O per mol of Al₂O₃ [19,21,24]. Furthermore, when the temperature increases to above 40 °C, strätlingite [18] and siliceous hydrogarnet [25], more thermodynamically stable phases, may precipitate, leading to a potentially improved resistance to drying shrinkage or swelling [26]. In addition to hydrates, microstructure also changes at exposure to increasing temperature. As shown by E. Gallucci et al. [27], in neat Portland cement, the microstructures are much coarser with elevated temperatures (5 °C, 20 °C, 40 °C and 60 °C). The hydration products distribute less homogeneously in the matrix and tend to be concentrated around the original locations of the anhydrous grains, especially at 60 °C. Such effect of elevated temperatures is also confirmed in the CSA cement paste [28]. Further study by L.L. Xu et al. [18] illustrates that the critical pore diameter, which is defined as the diameter of the pore that completes the first interconnected pore pathway in the pores network of the concrete or the cement matrix, shifts to coarser one in a PC/CAC/C̄S̄ system but to finer one in a PC/CAC/C̄SH_{0.5} system at elevated temperatures. Despite of the above literature, most of researches are focused on the hydration and properties of PC/CAC/C̄SH_x ternary systems. Little is known on the hydration mechanism and performance of CSA cement-based SLC, especially at the conditions of different temperatures and moisture conditions.

Therefore, in order to contribute to understanding the effect of various temperatures and moisture on CSA cement-based SLC, a comprehensive experimental study concerning about hydration mechanism as well as mortar properties was carried out. In terms of phase

assemblages and hydration process, X-ray diffraction (XRD) and differential scanning calorimetry-thermogravimetric (DSC-TG) analysis were used to do the identification and semi-quantitative determination. Morphology and microstructure of hardened mortars was observed by scanning electron microscope (secondary electronic imaging and backscattered electronic imaging mode). As for mortar properties, flowability, setting time, flexural and compressive strength and dimensional variation were measured. Finally, an effort was made to correlate mortar properties with hydration mechanism.

2. Experimental

2.1. Materials

The experiments were carried out on a binary binder system (the total content was about 26.6% in the dry mixture) consisting of a commercial CSA clinker and natural anhydrite, where the mass ratio of CSA clinker and anhydrite was fixed at 70:30. The chemical composition of them was measured by X-ray fluorescence (XRF) and given in Table 1. Combining with analysis derived from XRD, the main mineral composition of them were carried out by stoichiometric calculations based on the XRF results and summarized in Table 1 as well.

Limestone powder (LSP) was used as mineral filler, of which the mineral phase was calcite and considered reactive in such system [29,30]. The particle size distribution of CSA clinker, anhydrite and LSP are shown in Fig. 1. The particle size of LSP is more homogenous and appears smaller than those of CSA clinker and anhydrite. The LSP to binder ratio was 1:1 by mass.

Sand with size of 0.1–0.3 mm was used as aggregates. The mass ratio between the binder and sand was 1:1.76. Accelerator, retarder and superplasticizer were added to control the flowability and initial setting time at room temperature (around 140 ± 10 mm and 50–100 min,

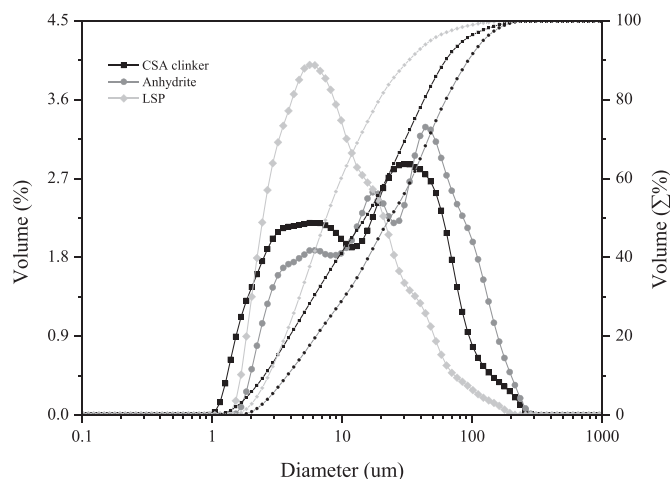


Fig. 1. Particle size distribution of CSA clinker, anhydrite and LSP.

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