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A comprehensive study on the relationship between mechanical properties and microstructural development of calcium sulfoaluminate cement based self-leveling underlayments

Linglin Xu [a,b], Nan Li [a], Ru Wang [a,b], Peiming Wang [a,b,*]

[a] School of Materials Science and Engineering, Tongji University, Shanghai 201804, China
[b] Key Laboratory of Advanced Civil Engineering Materials (Tongji University), Ministry of Education, Shanghai 201804, PR China

HIGHLIGHTS

- Both mechanical properties and hydration of CSA based SLU was evaluated.
- The formation of hemicarbonate was hindered by the addition of anhydrite.
- Ettringite formed at different hydration stage plays different roles on properties.

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ABSTRACT

In this study, the mechanical properties of calcium sulfoaluminate cement based self-leveling underlayments (SLU) prepared with various amount of anhydrite were evaluated through setting times, flowability, compressive strengths and linear shrinkage tests. Meanwhile, to optimize the workability and cost, a high percent of limestone powder was added in all the blends. Influences of CSA cement clinker variety and anhydrite dosage on the hydration of SUL were investigated in terms of the phase assemblage and microstructural development between 1 h and 56 d. Results show that the main hydrates of the control SUL samples made with neat CSA cement clinker and limestone powder are hemicarbonate, monosulfate, aluminium hydroxide and minor amounts of ettringite; massive ettringite and aluminium hydroxide are observed once anhydrite is further added. The SLU containing neat CSA1 cement clinker exhibits higher compressive strengths and drying shrinkage compared with those of neat CSA2 cement clinker. But such differences among different CSA cement clinkers based SLU are getting narrower with increasing anhydrite dosage. Those SLU samples made with 30% anhydrite yield the highest strength but the lowest drying shrinkage. The formation of ettringite at early term mainly contributes to strength development, while the formation of ettringite at later hydration stage favors to decrease the late-term drying shrinkage.

1. Introduction

Since its invention in the 1970s, self-leveling underlayments (SLU) has been frequently used on a concrete floor in various fields, such as new-building construction and repair work [1,2]. It gains lots of popularity due to its particular properties in fresh state (high flow ability, self-smoothing and homogeneousness) and hardened state (rapid hardening, early strength, shrinkage compensation, smooth surface and good durability) [3], which also contributes to the saving of production time and labor. To fulfill all the requirements of self-leveling and low thickness, a commercial SLU contains at least 10 components at the expenses of cost [3,4], associating with high dosage of special cement, chemical admixtures and high volumes of fine powders [4].

For the requirement of fast curing and high final strength, SLU is typically based on a blended binder system containing calcium aluminate cement (CAC), Portland cement and calcium sulfate. Regardless of the mix proportion, the hydration of those CAC based SLU mainly links to the formation of ettringite and aluminium hydroxide [2,5–9] (as shown in Eq. (1)). When the calcium sulfate in the system is exhausted, ettringite dissolves and monosulfate precipitates instead (cement nomenclature will be used hereafter: C = CaO, S = SiO_2, A = Al_2O_3, T = TiO_2, $\bar{S}$ = SO_3, F = Fe_2O_3 and H = H_2O).

* Corresponding author.
E-mail address: tjwpm@126.com (P. Wang).

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$$3CA + 3C\bar{S}H_2 + 32H \rightarrow C_6A\bar{S}_3H_{32} + 2AH_3(gel) \quad (1)$$

One solution to reduce the cost of SLU is using comparative cheaper but more eco-friendly cement. Designed by the China Building Materials Academy in the 1970s, the low-energy calcium sulfoaluminate (CSA) cement represents an eco-friendly alternative to Portland cement with respect to lower levels of CO_2 emission and energy consumption [10]. The main phase in CSA cement is ye'elimite ($C_4A_3\bar{S}$)), which can be produced with less limestone and about 200 °C lower than that required for the sintering of alite (C_3S) [11]. The CSA clinker has better grindability [12], thus lower energy is needed. Additionally, the manufacture of CSA cement can fully utilize industrial by-products, such as slag, phosphogypsum, red mud and even municipal solid waste incineration fly ash [13–17]. All those advantages enables that CSA cement has a 35% CO_2 footprint lower than Portland cement, and the cost for one ton CSA cement only accounts for 25–50% of CAC. Therefore, the CSA cement can be employed as an alternative binder to CAC for SLU [18,19].

From the aspect of hydration, although differing in mineral compositions with CAC based SLU, the hydration of calcium sulfate blended CSA cement also produces ettringite and aluminium hydroxide as the hydration product (Eq. (2)). Once the sulfate in pore solution is depleted and there is enough free water available, monosulfate forms instead according to Eq. (3).

$$C_4A_3\bar{S} + 2C\bar{S}H_2 + 34H \rightarrow C_6A\bar{S}_3H_{32} + 2AH_3\ (gel) \quad (2)$$

$$C_4A_3\bar{S} + 18H \rightarrow C_4A\bar{S}H_{12} + 2AH_3\ (gel) \quad (3)$$

Thus the formation and conversion of ettringite in CSA based SLU is also highly dependent on the reactivity and the amount of calcium sulfate [20–22]. It was found that the initial setting time of CSA cement mortars prepared with heimihydrate was too short to produce homogeneous microstructure, which had a dramatic effect upon the mechanical strength [20,21]. And compared with the gypsum or flue-gas desulfurization gypsum blended CSA cement paste, a denser and more ordered structure can be formed in the presence of anhydrite [21,22]. Hence, based on the cost, environment and performance, considerable experimental activities on CSA cement utilizing anhydrite as calcium source are well documented [21,23–25]. The amount of anhydrite can directly modify the ettringite to monosulfate mass ratio and the water content to complete hydration [20]. However, there is still many mysteries on the hydration process and technological performances of CSA cement based SLU [26].

Another option to reduce the expenses of SLU is using mineral additions like limestone powder. Besides the well accepted filler and nuclearation effects, limestone powder can also play chemical roles on the hydration of CSA cement. In the presence of limestone, less monosulfate is formed, while hemicarbonate and monocarbonate and more ettringite are present (as shown in Eq. (4)) [27]. This indirect stabilization of ettringite results in a higher total volume of solids and a higher compressive strength [27–30].

$$6C_4A_3\bar{S} + C\bar{S} + 135H \rightarrow 2C_4A\bar{C}_{0.5}H_{12} + 2C_6A\bar{S}_3H_{32} + 14AH_3\ (gel) + 5CH \quad (4)$$

These inspired our interest to carry out a study on the utilization of CSA cement as main binder with various amounts of anhydrite for the preparation of SLU. For a wider application of obtained conclusions, two kinds of CSA cement clinkers were applied. Meanwhile, extremely large amount of limestone powder was applied for properties and cost optimization. The phase assemblages and morphology of fractured surface of CSA cement based SLU were systematically explored with XRD, DSC-TG and SEM between 1 h and 56 d, at fixed water to binder ratio. And the strengths development and dimensional stability of the SLU pastes were evaluated in an effort to correlate the overall performance with microstructure development.

2. Experimental

2.1. Raw materials

Two varieties of CSA cement clinkers (named as CSA1 and CSA2 respectively) and natural anhydrite were provided as the main binder. The chemical compositions of those binders which determined by X-ray fluorescence (XRF) are listed in Table 1. On the basis of the chemical compositions, the mineral composition is calculated and the result is also shown in Table 1. It can be clearly seen that CSA1 contains higher amount of ye'elimite but lower amount of C_2S.

Limestone powder (LS) was introduced in the mixture to enhance workability and reduce total cost. The particle size distribution of CSA cement clinkers and LS was analyzed by laser diffraction, and the result is illustrated in Fig. 1. As can be seen from this

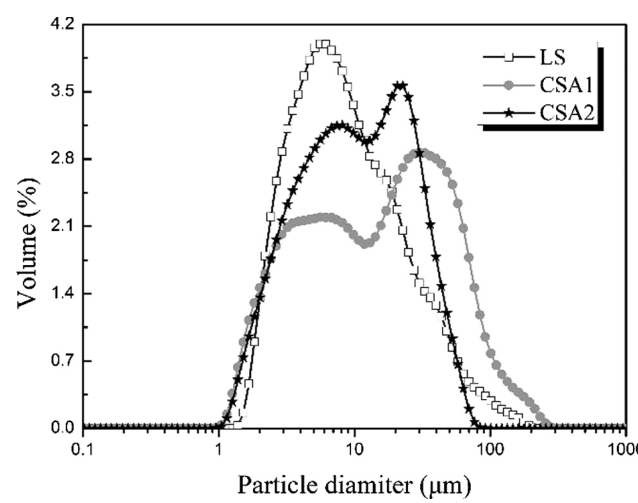

Fig. 1. Differential particle size distributions of the applied constituents.

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

Oxide	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	Gr_2O_3	Fe_2O_3	SrO	ZrO_2
CSA1	0.04	0.73	37.50	5.80	0.11	9.70	0.05	0.22	41.00	1.50	0.02	3.10	0.07	0.06
CSA2	0.05	1.30	35.20	8.60	0.11	7.80	0.05	0.19	42.90	1.40	0.04	2.20	0.09	0.05
Anhydrite	0.04	3.00	0.12	1.40	–	50.10	0.02	0.05	44.80	–	–	0.04	0.44	–

Minerals	$C_4A_3\bar{S}$	C_2S	C_4AF	CT
CSA1	69.50	16.65	9.42	2.55
CSA2	65.60	24.68	6.69	2.38