



The effect of water and gypsum content on strätlingite formation in calcium sulfoaluminate-belite cement pastes

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HIGHLIGHTS

- Properties of CSAB cement were investigated with different water and gypsum content.
- Thermodynamic modeling based on QXRD results was conducted to explore the hydration.
- Increasing water promoted hydration, but lowered the strength development of pastes.
- Gypsum played a role in controlling the hydration of ye'elmitite as well as belite.
- Strätlingite formation depended on a water content and a kinetic effect of hydration.

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ABSTRACT

The main objective of this study was to investigate the phase and strength development of calcium sulfoaluminate-belite (CSAB) cement pastes with different amounts of gypsum and water. Thermodynamic modeling and a series of experiments including X-ray diffraction (XRD), thermogravimetric analysis, isothermal calorimetry, and compressive strength tests were performed. Decreasing the mixing water increased the strength of CSAB pastes, but decreased the degree of hydration. Interpretation of the early age XRD results and thermodynamic modeling suggested the formation of a meta-stable phase from the hydration of belite, possibly C-(A)-S-H, which transformed into strätlingite at later ages only in the samples with high water content, likely due to easier diffusion of ions at higher w/c. Furthermore, the XRD results and thermodynamic modeling confirmed that the amount of gypsum controls the hydration of ye'elimitite as well as belite in the CSAB cements.

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1. Introduction

Ordinary Portland cement (OPC), one of the most widely used industrial products, is manufactured by firing limestone and clay at high temperature. The manufacturing process makes the production of OPC responsible for 2% of primary energy consumption and 5% of industrial energy consumption globally [1]. Furthermore, owing to the direct stoichiometric release of CO₂ and fuel consumption from the process, the manufacture of OPC contributes approximately 5% of the global anthropogenic CO₂ emissions. Due to these environmental issues, the cement and concrete research fields have been actively developing more sustainable cements with lower CO₂ emissions [2].

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Among the numerous sustainable cements being developed, calcium sulfoaluminate (CSA) cements have been receiving increasing attention. Although being initially developed in the 1960s as an expansive binder [3], CSA cements have been the subject of intense research by industry and academia during the last decades as a sustainable and high performance cementitious binder [4]. The environmental benefits of CSA cement compared to OPC can be classified as (1) a relatively low firing temperature (1250–1350 °C), (2) a lower CaO content resulting in less CO₂ emissions during calcination, and (3) a more friable clinker which requires less energy to pulverize [4,5].

The primary properties of CSA cement are mainly achieved based on the hydration of ye'elimitite (C₄A₃S̄, hereafter cement chemistry notation will be used; C = CaO, S = SiO₂, A = Al₂O₃, S̄ = SO₃, H = H₂O, F = Fe₂O₃, M = MgO, and T = TiO₂). The cement produces monosulfate (C₄A₃H₁₂) and/or ettringite (C₆A₃H₃₂) with

Table 1
Chemical and mineralogical compositions of CSAB clinker.

XRF		QXRD	
Oxide	Weight (%)	Phases	Weight (%)
CaO	45.0	C ₄ A ₃ S	69.4
Al ₂ O ₃	33.7	β-C ₂ S	20.0
SiO ₂	8.1	C ₁₂ A ₇	4.2
SO ₃	8.0	CT	3.8
Fe ₂ O ₃	2.2	C ₂ AS	1.5
TiO ₂	1.4	M	1.0
MgO	1.4		
K ₂ O	0.2		
SrO	0.1		
ZrO ₂	0.1		

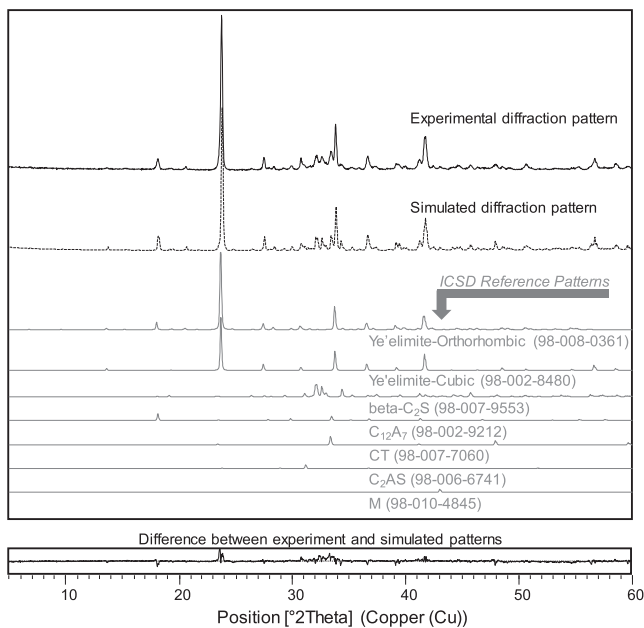


Fig. 1. Rietveld refinement result of raw CSAB clinker. The numbers in parentheses indicate the PDF numbers of the ICSD reference patterns.

aluminum hydroxide as the main binding materials depending on the calcium sulfate amount. In comparison to OPC, the hydration of CSA cements is faster (i.e., most of the hydration heat occurs within 24 h) due to the rapid hydration of ye'elimite [6–9].

As interest in the use of CSA cement increased, researchers tried to utilize CSA cement in a variety of engineering applications. Glasser and Zhang [10] suggested that CSA cement could be a high-performance binder for concrete production due to its high mechanical properties and durability. They suggested that a high

self-desiccation of CSA cement could mitigate the penetration of harmful external ions, resulting in high durability. Péra and Ambroise [11] investigated the possible application of CSA cement to self-leveling mortar using its rapid strength development characteristics. Khalil et al. [12] tried to utilize the cement as a setting control agent for 3D-printable mortar. They concluded that only a small amount of CSA cement with OPC could obtain suitable properties for 3-D printing. Zhang et al. [13] used a finely ground CSA cement for a grouting application. Furthermore, due to the ionic substitution capability of ettringite and AFm phases, CSA cements are also of interest in hazardous waste encapsulation or immobilization [14,15].

However, the hydration of CSA cement is a complex phenomenon, and there is much variability in clinker phases. Thus, various experiments along with thermodynamic modeling were performed for CSA-based systems to understand their complex hydration behaviors [8,9,16–19]. From a mineralogical point of view, hydration mechanisms of two polymorphs of ye'elimite (i.e. cubic and orthorhombic) were compared [20]. Recent fundamental studies extended the understanding on mechanical and thermodynamic properties of ye'elimite [21,22]. The effects of mineral admixtures on CSA systems have also been studied with respect to expansion and hydration characteristics [9,19,23,24]. The strength development and volumetric stability of CSA cement particularly depend on the types and amount of calcium sulfate incorporated (i.e. intentionally added or formed in the clinker) [18,23,25–27]. Winnefeld et al. [18] found that the use of gypsum with anhydrite-blended CSA cement could modify the hydration kinetics of CSA cements.

Calcium sulfoaluminate-belite (CSAB) cement possesses main components of ye'elimite, belite, and calcium sulfate with other minor phases which can vary depending on the sources of the raw materials [4,10,28]. Ye'elimite hydration dominates the early age property development of CSAB cements, and belite hydration occurs later and further densifies the microstructure. While OPC is interground with a few percent of calcium sulfate, CSAB cements typically contain 16–25 wt% calcium sulfate [6,10]. Calcium sulfate alters the hydration kinetics of ye'elimite and, therefore, affects strength development and setting time. Calcium sulfate content is also a principal factor controlling volumetric expansion [4,8,29]. Likewise, the water-to-cement ratio (w/c) also controls the hydration kinetics of CSAB cement and determines the available space for hydration products to form and grow [25]. As with OPC, the w/c significantly affects the overall macroscopic mechanical properties of hardened CSAB cement [7,10].

Belite hydration in CSAB-based system is generally promoted after calcium sulfate depletion and produces a crystalline calcium aluminosilicate hydrate i.e. strätlingite (C₂ASH₈) [29,30]. However, in other previous studies [9,18,31,32], no strätlingite formation was identified in XRD analysis while their thermodynamic models reported the strätlingite formation, causing a mismatch between

Table 2
Mixture proportions by mass for the CSAB pastes.

Label	CSAB cement		Water	w/c	Molar ratio of gypsum/ye'elimite (M)
	CSAB clinker	Gypsum			
CSAB_g0.15_wc0.4	87.0	13.0	40	0.4	0.8
CSAB_g0.15_wc0.6	87.0	13.0	60	0.6	0.8
CSAB_g0.15_wc0.8	87.0	13.0	80	0.8	0.8
CSAB_g0.20_wc0.4	83.3	16.7	40	0.4	1.1
CSAB_g0.20_wc0.6	83.3	16.7	60	0.6	1.1
CSAB_g0.20_wc0.8	83.3	16.7	80	0.8	1.1
CSAB_g0.25_wc0.4	80.0	20.0	40	0.4	1.4
CSAB_g0.25_wc0.6	80.0	20.0	60	0.6	1.4
CSAB_g0.25_wc0.8	80.0	20.0	80	0.8	1.4

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