



Effect of nano-silica on hydration and conversion of calcium aluminate cement [☆]



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HIGHLIGHTS

- Nano-silica effect on hydration and conversion of calcium aluminate cement (CAC).
- Nano-silica mitigated the conversion from metastable phases to stable phases.
- Adding nano-silica maintains the long-term strength of CAC by forming strätlingite.

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ABSTRACT

This study investigated the effect of nano-silica on the hydration and conversion of calcium aluminate cement (CAC). The specimens were exposed to 60 °C to accelerate the transformation of hydrates in CAC after initial curing at 20 °C. At an early stage of curing, adding nano-silica in CAC mitigated the formation of C_3AH_6 and AH_3 . At 1 day after exposure to 60 °C, the compressive strength reduction was observed in all specimens, and it was closely related to the conversion of CAH_{10} . That is, the conversion of all specimens occurred at 28 days after exposure, which resulted in a reduction in compressive strength. However, adding nano-silica mitigated the conversion from metastable phases (CAH_{10} and C_2AH_8) to stable phases (C_3AH_6 and AH_3). As a result, the extent of compressive strength loss could be reduced by the formation of strätlingite (C_2ASH_8) in the case of 4% addition of nano-silica. In conclusion, adding nano-silica in CAC was effective regarding structural integrity by delaying the conversion of metastable hydrates, as well as maintaining the long-term strength of CAC by forming strätlingite.

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1. Introduction

Calcium aluminate cement (CAC) is of a type belonging to non-Portland cement (PC). The main difference between CAC and PC is their clinker composition. PC contains mainly C and S with a few A, whereas CAC consists of C and A, which are present as a crystalline and principal phase mono-calcium aluminate (CA, hereafter) in the CAC [1]. Although the production cost of CAC is relatively higher than that of PC, its unique properties have provided numerous special applications. In particular, CAC has a variety of advantages over PC such as sulfate resistance, fire resistance, chemical resistance, acid resistance, and high early strength development [2]. Due to these advantages, CAC has been used as a refractory material, a fast-setting compound, a carbonation-resistant material [2–5], as

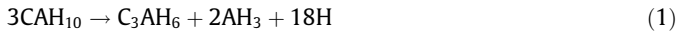
well as in the immobilization of hazardous materials [6–10]. In addition, the strength of CAC can reach as high as 150 MPa, showing it as adequate for use as a high strength material [11]. Despite these advantages, however, the phase transformation in CAC accompanies an increase in the porosity in a hardened state, causing a loss of mechanical strength [12,13]. In particular, this phenomenon is accelerated by an increase in the surrounding temperature and humidity [1]. Therefore, the use of CAC as a structural material has been limited and prohibited in many countries [14,15].

The hydration pathway of CAC is completely different from that of PC in a chemical perspective. Hexagonal calcium aluminate hydrates (CAH_{10} and C_2AH_8) and cubic hydrogarnet (katoite, C_3AH_6) are formed by hydration of CAC clinker minerals (mainly CA) [16]. Meanwhile, the hexagonal calcium aluminate hydrates are metastable, therefore they are gradually converted into stable phases (C_3AH_6 and AH_3) over time, as expressed in Eqs. (1) and (2) [17].

[☆] This paper uses standard cement chemistry abbreviations, where CaO is denoted as C; Al_2O_3 as A; SiO_2 as S; H_2O as H.

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It should be noted that the densities of stable phases are higher than that of metastable phases, meaning that the porosity of hardened cement increases as hydration proceeds, thereby causing volumetric instability [2]. In particular, the density of C_3AH_6 is the highest among the hydrates, and therefore can cause a significant reduction in the volume of hydrates, leading to a loss in strength [1].

Various means of suppressing the conversion of hydrates in CAC have been explored in numerous studies. Curing CAC at a high temperature is one method for mitigating the conversion of metastable hydrates [1]. This is due to the formation of CAH_{10} occurring by exposure to lower temperatures (below 20 °C), C_2AH_8 and AH_3 at intermediate temperatures (20–40 °C), and C_3AH_6 and AH_3 at relatively higher temperatures (40–60 °C) [1]. This implies that the formation of metastable phases, which are likely to induce unstable conversion, can be significantly reduced. Majumdar and Singh showed that the stable phases can form directly at a temperature over 50 °C, which can contribute to the maintenance of the mechanical properties of CAC [18]. Sakai et al. reported that a reduced water-to-binder ratio and a higher curing temperature can promote the formation of C_3AH_6 at an early stage of hydration [19]. As a result, the conversion of hydrates is suppressed, resulting in a sustained mechanical strength [19].

Cement-based materials with nano-materials have recently gained significant attention in the academic and industrial communities. The incorporation of nano-silica in PC can induce an accelerated hydration effect, due to its high specific surface area [20], and provide a seed of nucleation site for the precipitation of C-S-H gels via pozzolanic reactions [21,22]. In particular, only a small amount of nano-silica is needed to significantly reduce the setting time and to enhance the early age strength and durability [23–25]. Moreover, the effect of nano-silica on the hydration characteristic and the mechanical property of PC has been extensively explored in previous studies, and in-depth knowledge has been gained in the past few years. Nevertheless, the incorporation of nano-silica in CAC has never been attempted, and more importantly, the information for understanding such an effect is currently unavailable. This study, therefore, investigated the effect of nano-silica on the hydration and conversion of CAC. Specimens with various dosages of nano-silica were fabricated and initially cured at 20 °C. The specimens were thereafter immersed in water at 60 °C to accelerate the conversion of hydrates and to draw a correlation between nano-silica incorporation and the conversion process.

2. Experimental procedure

2.1. Materials and specimens preparation

CAC (manufactured by Kerneos Inc.) and nano-silica (manufactured by OCI Inc., SiO_2 with 99.5% purity) were used in this study. The chemical composition of the CAC is given in Table 1, and the morphology of nano-silica is shown in Fig. 1. Mortar specimens were produced with a constant weight ratio of water:CAC: fine aggregate as 0.4:1:1. The effect of nano-silica on the conversion of CAC hydrates was investigated by replacing CAC with various

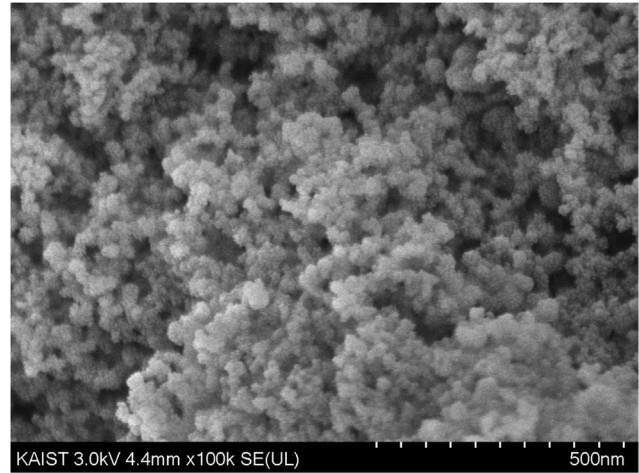


Fig. 1. SEM image of nano-silica used in this study.

dosages of nano-silica (0, 1, 2, and 4% relative to the weight of CAC). The mix proportion used in this study is summarized in Table 2. Nano-silica can be added to cement paste in a powder or colloidal form. The nano-silica in a colloidal form inevitably contains water, which would increase the water-to-cement ratio and hence affects the hydration characteristic of CAC. Therefore, the nano-silica in a powder form was used in this study to keep the water-to-cement ratio constant throughout the mixtures.

The following procedure was conducted for fabricating specimens. Dry mixtures of CAC, sand, and nano-silica were stirred to ensure homogeneity. Water was added to the dry mixture and mixed for five minutes. The workability of the mixtures varied according to the dosage of nano-silica (i.e., lower workability at a higher dosage of nano-silica). In the present study, the water-to-cement ratio was kept at 0.4, where the mix with the highest dosage of nano-silica was in the workable state. The fresh mixture was poured into a 50 × 50 × 50 mm³ mold. Simultaneously, paste specimens with an identical water-to-cement ratio were fabricated without any fine aggregates. The mortar and paste specimens were sealed in a plastic wrap to prevent moisture from evaporating and were cured at 20 °C. Since the formation of CAH_{10} occurs at a temperature higher than 20 °C in a moisture condition [26], the curing temperature was set at 20 °C to ensure the formation of metastable hydrate. All specimens were demolded at 1 day after casting and were further cured at 20 °C. At 7 days, the specimens were immersed in water at 60 °C for an additional 28 days to accelerate the ageing of CAC, as proposed by Sakai et al. [19]. The time at which the specimens were immersed in warm water is hereafter referred to as “before/after exposure”.

2.2. Test methods

X-ray diffractometry (XRD) and thermogravimetry (TG) analyses were conducted to investigate the influence of nano-silica on the conversion, which is highly associated with the compressive strength loss in CAC. The samples for the analyses were immersed in acetone and desiccated for 48 h to remove capillary water, and then crushed and sieved to pass 100 μm. The XRD analysis was conducted using Rigaku D/MAX-2500 with Cu-K α radiation and

Table 1
Chemical composition of CAC used in this study.

	Al_2O_3	CaO	SiO_2	Fe_2O_3	TiO_2	MgO	SO_3	Na_2O
wt%	50.77	38.39	4.83	1.82	2.04	0.40	0.24	0.63

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