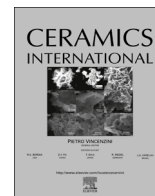




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Effect of micro-sized alumina powder on the hydration products of calcium aluminate cement at 40 °C

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ABSTRACT

In this work, the effect of different micro-sized alumina powders on the hydration products of calcium aluminate cement (CAC) during hydration at 40 °C is studied. The cement hydration at the designated times is terminated by the freeze-vacuum method. The phase development and microstructure evolution during the cement hydration are investigated by XRD and DSC, and SEM, respectively. It is found that $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ (C_3AH_6) is the dominant product of the pure CAC after hydration at 40 °C for 3.5 h. But $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$ (C_2AH_8) is the dominant hydrate and C_3AH_6 is not found in the mixtures of CAC and micro-sized alumina powder under the same condition. The results indicate that the addition of alumina powders promotes the formation of C_2AH_8 and retards the conversion of C_2AH_8 to the C_3AH_6 phase. Moreover, such phase development with alumina addition is discussed.

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1. Introduction

Calcium aluminate cement (CAC) is a widely used hydraulic binder in refractory castables [1] as CAC can endow castables with sufficient demolding strength within 24 h. It is known that the hydrate products significantly depend on the curing temperatures [2,3]. For example, $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$ (CAH_{10}) is the main hydrate at temperatures below 15 °C, whereas CAH_{10} and $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$ (C_2AH_8) are formed at temperatures between 15 °C and 35 °C [1,4,5]. As CAH_{10} and C_2AH_8 compounds are thermodynamically metastable, they have a higher solubility than the stable phases of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ (C_3AH_6) and $\text{Al}(\text{OH})_3$ (AH_3). Once stable phases start to nucleate, the metastable phases dissolve, and further convert into C_3AH_6 with the curing time and temperature. As a result, at higher temperatures above 35 °C, C_3AH_6 and AH_3 are the only CAC stable phases [6,7].

Because the conversion processes of CAH_{10} or/and C_2AH_8 to C_3AH_6 lead to the increase in the castable porosity and consequently result in the decrease of its mechanical properties [6,8], these conversion reactions should be avoided [6]. Some authors have reported that the addition of lime stone to CAC counteracts the conversion process by the formation of a lamellar hydrate $\text{C}_3\text{A} \cdot \text{CaCO}_3 \cdot 11\text{H}_2\text{O}$ [6,9]. It is also reported that the conversion reaction could be inhibited by microsilica [8], fly ash [8], granulated blast-furnace slag [10], and sodium silicate [11] due to

C_2ASH_8 ($2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$) formation in preference to C_3AH_6 . Meanwhile, other investigations have shown that the micro-sized alumina powder could also influence the hydration products of CAC [12–14]. Only CAH_{10} is detected in the pure CAC sample, CAH_{10} and C_2AH_8 are identified in a 50:50 mixture of CAC and alumina after hydration at 20 °C in the case of low surface area alumina, and C_2AH_8 and AH_3 are detected in the case of high surface area alumina powder [12]. Some researches also reveal that the addition of alumina powder to $\text{CaO} \cdot \text{Al}_2\text{O}_3$ (CA) favors the formation of C_2AH_8 between 22 °C and 25 °C [13].

It has been established that C_2AH_8 convert into C_3AH_6 with increasing temperature and the only stable hydration products of CAC at 40 °C should be AH_3 and C_3AH_6 formed via the conversion process [4]. As discussed above, the micro-sized alumina powder promotes the development of C_2AH_8 at 20–25 °C [12–14]. But little is known about the effect of micro-sized alumina powder on the phase conversion process at 40 °C. The objective of this study is to investigate the phase conversion during the hydration of CAC in the presence of micro-sized alumina powder at 40 °C. The cement pastes were cured at 40 °C for the designated times and the hydration was halted by freeze-vacuum drying method. Then the phases of the hydrated samples were analyzed by XRD and DSC, and the morphology of the samples was observed by SEM.

2. Experimental procedures

A commercial CAC (Zhengzhou Jianai Special Aluminates, China) was used in this work. The chemical composition and the

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Table 1
Chemical and mineralogical compositions of CAC.

Compositions	(%)	
Chemical compositions	Al ₂ O ₃	69.37
	CaO	28.81
	SiO ₂	0.46
	K ₂ O+Na ₂ O	0.29
Major phases	CA	51.03
	CA ₂	41.61
	C ₁₂ A ₇	0.10
	α-Al ₂ O ₃	3.76

Table 2
Characterization of the micro-sized alumina powders.

Alumina powder	S.S.A (m ² /g)	Na ₂ O content (%)	Grain size distribution		
			D10 (μm)	D50 (μm)	D90 (μm)
A1	7.48	0.10	0.47	0.99	2.60
A2	3.06	0.10	0.74	2.14	8.44

S. S. A: specific surface area by BET method.

major phase composition of the cement are listed in Table 1. Two commercial micro-sized alumina powders with different specific surface areas (A1 and A2) were used in this work. As shown in Table 2, the specific surface area of A1 is higher than that of A2.

The alumina powders were added into CAC with a CAC/alumina weight ratio of 60/40 to prepare the CAC/A1 and CAC/A2 mixtures. The pure CAC without any alumina powder was used as the reference.

The aqueous suspensions of the pure CAC and those mixtures were prepared with a water/solid ratio of 5/1. The electrical conductivity measurement was carried out by inserting a conductivity electrode (Inlab731, Mettler Toledo, Switzerland) into the aqueous suspensions at 40 °C with a relative humidity of 80%.

The pastes of the pure CAC and mixtures were prepared with a water/solid ratio of 0.37. Then the pastes after curing at 40 °C for designed times were frozen at -40 °C for 1.5 h to halt the hydration process. The frozen pastes were immediately dried in a vacuum box under a pressure of 60 Pa at 20 °C for 24 h. Then the phase compositions and morphology of the dried cement pastes were characterized by XRD (D8 Focus, Bruker, Germany) and SEM (ZEISS-FESEM MERLIN Compact, Germany), respectively. Differential scanning calorimetry (DSC, NETZSCH STA 449, Germany) of the dried cement pastes was performed at a heating rate of 10 °C/min in nitrogen flow.

3. Results and discussion

Fig. 1 shows the electrical conductivity of the aqueous suspensions of the pure CAC and mixtures at 40 °C. Initially, the conductivity of the cements with and without alumina powder increases, indicating that the addition of alumina powder in the cement does not affect the dissolution process of the cement. It is also seen in Fig. 1 that the drop in conductivity attributed to the onset of hydrate precipitation occurs at around 45 min, 10 min and 40 min for the aqueous suspensions of CAC, CAC/A1 and CAC/A2 respectively. These results indicate that addition of alumina powder accelerates the hydration of the cement.

Fig. 1 also shows that the decrease in electrical conductivity of the aqueous suspension with addition of A1 happens much earlier (10 min) than that with addition of A2 (40 min), which is probably due to the higher specific surface area of A1 (7.48 m²/g) than that

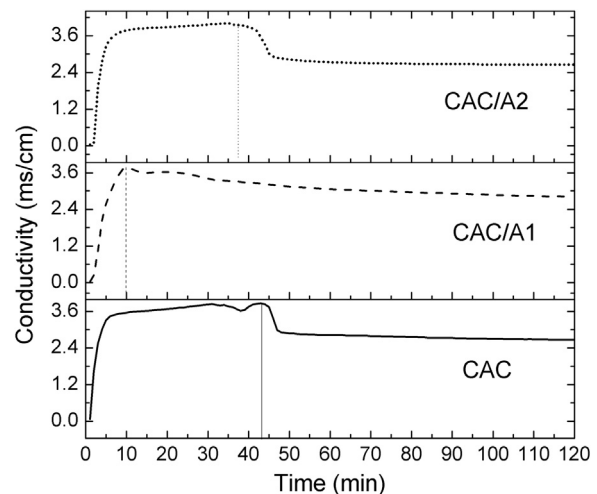


Fig. 1. Conductivity of CAC and mixtures (CAC/A1 and CAC/A2) at 40 °C.

of A2 (3.06 m²/g) (Table 2). This is most likely caused by the preferential nucleation of the hydration products on the surface of alumina particles. The alumina with the higher specific surface area in the mixture is more beneficial to form the nucleation of the hydration products, thus speeds up the hydration of the cement to a higher degree [14,15].

The XRD patterns of the samples of the pure CAC and mixtures after curing at 40 °C for 0.5 h are shown in Fig. 2. Hydrated products are not detected in the pure CAC paste after the halted hydration. In contrast, C₂AH₈ and AH₃ are present in the mixture samples, demonstrating that alumina powders favor the formation of C₂AH₈ in CAC after 0.5 h of hydration at 40 °C. Moreover, the peak of C₂AH₈ at 8.17° in CAC/A1 sample is higher than that in CAC/A2, implying that more C₂AH₈ was formed in the former sample during hydration. This observation confirms that the alumina powder with a higher specific surface area (Table 2) accelerates the hydration of CAC to a higher degree than the alumina powder with a lower specific surface area [15].

Fig. 3 presents XRD patterns of the samples of the pure CAC and mixtures after curing at 40 °C for 3.5 h. It is seen that C₃AH₆ and AH₃ are the major hydrates in the pure CAC paste. In comparison, C₂AH₈ and AH₃ are the main hydrates and no C₃AH₆ is detected in the mixtures, further confirming that the presence of alumina powders favor the formation of C₂AH₈.

It is reported that the stable crystalline hydrates at 40 °C are only C₃AH₆ and AH₃ [5,16,17], and C₃AH₆ can be produced only via conversion from C₂AH₈ [17,18]. As seen in Fig. 3, C₃AH₆ and AH₃, as expected, are present in the CAC sample. However, C₂AH₈ peak at

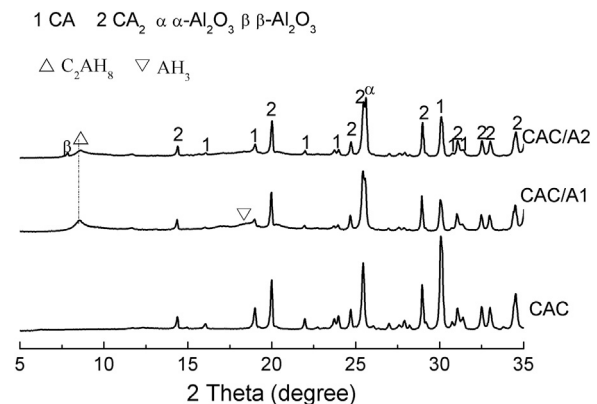


Fig. 2. X-ray diffraction patterns of CAC and mixtures (CAC/A1 and CAC/A2) hydration for 0.5 h at 40 °C.

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