



Influence of curing temperatures on the hydration of calcium aluminate cement/Portland cement/calcium sulfate blends

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ABSTRACT

In this paper, the effects of curing temperature on the hydration of calcium aluminate cement (CAC) dominated ternary binders (studied CAC: Portland cement: calcium sulfate mass ratio were 22.5: 51.7: 25.8) were estimated at 0, 10, 20 and 40 °C, respectively. Both α -hemihydrate and natural anhydrite were employed as the main source of sulfate. The impacts of temperature on the phase assemblages, morphology and pore structure of pastes hydrated up to 3 days were determined by using X-ray diffraction (XRD), backscattered electron imaging (BEI) and mercury intrusion porosimetry (MIP). Results reveal that the main hydration products are firmly related to calcium sulphoaluminate based phases. Increasing temperature would result in a faster conversion from ettringite to plate-like monosulfate for both calcium sulfate doped systems. When the temperature increases to 40 °C, an extraordinary formation of strätlingite (C_2ASH_8) and aluminium hydroxide is observed in anhydrite doped pastes. Additionally, increased temperature exerts different effects on the pore structure, i.e. the critical pore diameter shifts to finer one for pastes prepared with α -hemihydrate, but changes to coarser one for those made with anhydrite. From the mechanical point of view, increased temperature accelerates the 1-day strength development prominently, while exerts marginal influence on the development of 3-day strength.

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1. Introduction

It is well accepted that the strength development of mortars based on Portland cement (PC) is easily guaranteed at 20 °C. However, when temperature decreases to 5 °C, it is very difficult to achieve sufficient strength, especially at early hydration stage. Technically, calcium aluminate cements (CAC) which is more reactive compared with PC seems to be a choice for low temperatures condition, but the high cost and final strength reduction strongly limits its application in structural constructions [1]. Thus CAC based blended cements become another alternative solution.

Due to the excessive formation of massive pellet-like calcium aluminate hydrates [2–4], the compressive strength of PC-CAC blends decreases significantly with the addition of CAC in most cases. To modify the phase assemblages of calcium aluminate based cements, calcium sulfate (anhydrite, hemihydrate or gypsum) is normally added to achieve a thermodynamically stable hydrates

[5,6]. In this way, massive ettringite forms quickly in the matrix. This enables ternary binders containing CAC, PC and calcium sulfate to exhibit excellent and controllable performances, such as certain workability, rapid setting and hardening, shrinkage compensation and etc. Also owing to these aspects, the ternary binders are widely applied for those non-structural finishing operations in buildings [6–9], despite the relatively higher costs with respect to plain PC [10]. Additionally, given that CAC based formulations develop more homogeneous enrichments than PC rich systems [11], they are highly preferred in self-leveling mortar, high tech tile adhesives and rapid repairing materials [7,12].

It has been well confirmed that curing temperature plays an important role on the cement hydration kinetics [13,14]. Varying temperature leads to the changes in phase assemblages and also the spatial distribution of hydration products. On the one hand, the dissolution behaviors of raw materials such as calcium sulfate varies significantly with temperature [15]. Higher temperature increases the solubility of calcium sulfate and ettringite and thus leads to an increase of sulfate ion concentrations in the aqueous phase [13,14,16]. C-S-H phases in Portland cement are denser and

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exhibit a heterogeneous distribution at increased temperature, which would result in a reduced hydration degree at later stage, coarser porosity and decreased final later strengths [14,15,17–19]. On the other hand, the formed metastable calcium aluminate hydrate (CAH_{10} , C_4AH_{13} or C_2AH_8) converts to C_3AH_6 for the neat CAC at higher temperature. This phase transition is always accompanied by an increase in porosity and also a prominent decrease in strength [6,20,21]. In practice, besides of the impacts from ambient environmental temperature and applied heat in precast concrete production, the exothermic chemical reaction between cement and water also generates massive heat at very early hydration stage. It was found that, even for 1.7 liters of calcium sulfoaluminate cement paste which cured at 22 °C, the inner temperature is able to increase by 5 °C within 1 day [22]. All those heat effects consequently lead to microstructural transformations within cement pastes or concrete [23].

As discussed and confirmed in previous works, ettringite dominates the early hydration for both CAC and PC based ternary systems [5,7,8,11,24], which is highly different from the aforementioned hydration of plain PC or CAC paste. Above 70 °C, the formed ettringite is thermodynamically less stable than monosulphate (AFm) [14,20], the conversion from ettringite to AFm tends to happen. This is also a known prerequisite for concrete deterioration caused by delayed ettringite formation (DEF). Consequently, controlling temperature in CAC dominated ternary binders might be even more important than neat PC or CAC cements.

Despite the wealth of engineering information available in functional ternary binders, the precious investigations mainly focus on the physical properties and only few are available regarding its hydration mechanisms at 20–25 °C [7,12,25–27]. Results from our previous study [28], both lower temperature (0, 5 and 10 °C) and types of calcium sulfate play important roles on the hydration of Portland cement dominated ternary systems, especially on the formation and stability of ettringite. Given that the CAC based ternary systems contain much higher quantities of aluminate (in the form of monocalcium aluminate [10]), there is an increased potential of promoting ettringite formation. Therefore, this work mainly focuses on the CAC based ternary systems hydrated at 0, 10, 20 and 40 °C, and separately blended with anhydrite or hemihydrate (exhibiting significantly different dissolution characteristics). It works as part of a comprehensive study on the impact of curing temperature on the microstructure development of ternary systems, enabling a much deeper understanding of the chemistry and phase composition, and hence overall performance of these functional materials in service.

2. Experimental

2.1. Materials

The Portland cement (PC, CEM I 42.5) and Calcium aluminate cement (CAC, Ternal White) used in this study were obtained from Xinfu cement Co. and Kerneos cement Co, respectively. α -hemihydrate (H) and natural anhydrite (A) were employed as the main sources of calcium sulfate. It was originated from Henan Yongtai gypsum Co. and Anhui Hengtai nonmetallic material Co. Additionally, the Blaine values of the PC, CAC, A and H were 344, 400, 387 and 412 kg/m^2 , respectively. The CAC used in this work was mainly composed of calcium aluminate ($\text{CaO}\cdot\text{Al}_2\text{O}_3$, denoted as CA) and calcium di-aluminate ($\text{CaO}\cdot 2\text{Al}_2\text{O}_3$, denoted as CA_2). The chemical compositions of the raw materials which determined by X-ray fluorescence (XRF) are listed in Table 1.

Since both calcium sulfate source [24,29] and CAC/calcium sulfate ratio [30] exert significantly influence on the hydration of

Table 1
Chemical compositions of raw materials (wt./%).

	SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	TiO_2	LOI
PC	21.50	64.20	4.14	2.40	2.57	2.89	0.84	0.67	0.32	1.67
CAC	0.34	31.20	67.10	0.10	–	0.06	–	0.34	–	0.9
A	1.72	39.44	0.35	0.16	1.80	51.96	–	–	0.02	4.15
H	1.03	38.50	0.17	0.04	0.10	53.40	0.14	–	0.01	6.08

ternary binders, the mass ratio of PC: CAC: calcium sulfate in this work was fixed at 22.5: 51.7: 25.8. All specimens were prepared at 20 ± 1 °C with a water to binder ratio of 0.5. According to the introduced abbreviation for calcium sulfate used, as well as the curing temperature (0, 10, 20 and 40 °C), the samples were labeled as follows: A0, A10, A20 and A40; H0, H10, H20 and H40 (indicating A = anhydrite, H = hemihydrate and respective numbers for temperature).

2.2. Testing programs

Water, cement and sand were stored at the respective temperatures (0, 10, 20 and 40 °C) for 2 days before mixing. Mortar samples (40 mm \times 40 mm \times 160 mm) for compressive strength tests were prepared and measured at designed testing age according to ISO 679: 1989. The mixing protocol was: the dry powders were firstly blended for 30 s at low speed, then water was added and mixed for 30 s at low speed; finally, the sand is added and mixed for 30 s at low speed followed by a break of 15 s and an extra 60 s of blending at high speed. These samples were demoulded after 24 h of curing and three specimens were randomly selected for mechanical testing after periods of 1, 3, 28 days. The average values of each set were taken as compressive strengths. The tests were undertaken at a loading speed of 2.4 kN/s. All the specimens were rapidly prepared at 20 °C and then moved to 0, 10, 20 and 40 °C chamber with a variation of ± 1 °C respectively and the relative humidity was $60 \pm 10\%$.

In order to characterize the hydration process, the heat release of the pastes was monitored by means of isothermal heat conduction calorimetry at 20 °C (ToniCal Trio, Toni Technik, Berlin, Germany). The water to binder ratio was fixed at 0.5, which was the same as mortars. Measurements were carried out over 72 h.

In parallel, the paste specimens were firstly cast in polystyrene cylinders (35 mm \varnothing \times 50 mm) for X-ray diffraction (XRD), secondary electron imaging (SEI), backscattered electron imaging (BEI) and mercury intrusion porosimetry (MIP) measurements. After mixing and preparing, all these cylinders were immediately cured in 0, 10, 20 and 40 °C, respectively. The secondary electron imaging samples were freshly fractured from the cylinders without any further procedure after 0.5 h of hydration. And several slices of 2–3 mm thickness were cut from the cylinders for BEI analysis after 3 days, which were immersed in acetone to stop hydration. Then the slices were stored in desiccators under vacuum and over silica gel for 2 days to remove the acetone, which is able to prevent carbonation and removes possible moisture as well. Two slices were crushed and grounded into fine powder (about 400 m^2/kg Blaine surface areas) for XRD analysis.

XRD data were recorded with Rigaku-D/max2550VB3+. The scanning was performed between 5° and 75° with a 2θ increment 2° per min, and a Cu $K\alpha$ radiation ($\lambda = 1.541$ Å). In addition, an accelerating voltage of 40 kV and a current of 100 mA were employed.

In order to well capture the microstructure of very early age pastes, the microstructure of natural fracture surface was analyzed by high-resolution Environmental Scanning Electron Microscope equipped with a Field Emission Gun (ESEM-FEG, XL30, FEI/Philips)

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