



Hydration of quaternary phase-gypsum system



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HIGHLIGHTS

- The main hydrates are AFt, AFm, hydrated calcium aluminate and amorphous AH₃.
- The hydration rate of Q phases is accelerated by gypsum.
- The AFm platelets are smaller for the samples with higher gypsum.
- The hydrates in Q phase-gypsum samples are closely intermixed.
- A monolithic region with no apparent porosity around grains is observed.

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ABSTRACT

Since the quaternary (Q) phase (Ca₂₀Al₂₆Mg₃Si₃O₆₈) shows a potential to improve early strength and reduce the shrinkage of Portland cement paste, understanding the hydration of Q phase with and without sulfate is of great importance. In this work, the hydration of Q phase-gypsum systems were investigated in terms of the kinetics, phases evolution and microstructure development. The test results show that the hydration kinetics, microstructure and composition of hydration products depends on the gypsum addition. The observation of heat evolution indicates that the hydration rate is accelerated by gypsum, and formation of ettringite (AFt) and monosulfoaluminate (AFm) is probably the controlling factor at early age. The main hydration products are AFt, AFm, calcium aluminate hydrate and AH₃ phase. And the calcium aluminate hydrate and AH₃ phase will continue to form as the hydration proceeds. The compressive strength increases significantly with increasing gypsum content, which is due to the increasing hydration degree of Q phase-gypsum samples. Microstructural study shows that the formation of AFm phases occurs in the space between the Q phase grains but also within the “inner” regions around the Q phase grains. The AFm platelets become small and the “inner” regions around grains disappear in the samples with high gypsum. In addition, the AFt or AFm is closely intermixed with AH₃ and calcium aluminate hydrate, microcracks occur in samples with high gypsum content due to the formation of AFt.

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1. Introduction

Q phase (Ca₂₀Al₂₆Mg₃Si₃O₆₈) is one of phases in the quaternary system, alternatively referred to pleochroite phase or “Hanic phase” [1]. Pleochroite earns its name from its extremely strong pleochroism in optical microscopy in extra thin sections [2]. The investigation on structure of Q phase can be dated back to the work of Parker and Ryder [3], while the present structure description was given by Hanic et al. [1]. In fact, Q phase is extremely impor-

tant in many technological applications such as ceramics, blast-furnace slags and cement et al. Many researchers investigated the composition and structure of Q phase [4–7]. Among them, the kinetics and mechanism of formation of Q phase was investigated by Wang and Kapralik [5,4]; The formation mechanism of Q phase prepared with fly ash were studied, and the Q phase showed a rapid hydration [6]. In addition, the formation and microstructure of Q phase with Fe²⁺ substitution at sites of Mg²⁺ and partial Fe³⁺ substitution at sites of Al³⁺ was investigated by Sourie et al. [8], and a new kind of Q phase was synthesized. The hydration of Q phase was also studied, and the test results showed that the hydrates of Q phase were close to those of the high alumina cements [9]. However, the Q phase obtained a high early strength, nearly as high as high alumina cements, but the strength development was slower [10], a final setting time of 6.5 h had been

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reported [3]. Meng et al. reported that the mortar containing Q phase showed a continuous increasing compressive strength, and a high compressive strength value of 60.94 MPa could be obtained at the age of 28 d [6]. Kaprilik investigated the compressive strength of Q phase-water system at room temperature, a significantly high early strength (27.93 MPa) obtained at 1 day [9].

Because Q phase possesses advantages of high early strength, relatively stable hydrates and high chemical resistance [9,11], it shows a potential to improve the early strength of Portland cement paste. Also, previous study showed that mortar prepared with cement containing Q phase clinker and Portland clinker showed a high early compressive strength [12]. However, gypsum is usually added to regulate the rapid reaction of C_3A and preserves the workability of Portland cement paste during the first few hours [13,14], and the presence of gypsum would also extremely influence the hydration of high alumina cement [11] as well as Q phase. Then, investigation on the hydration of Q phase with gypsum is important to promote the usage of Q phase in Portland cement. In addition, according to the model of Powers and Brownard updated by Brouwers, the Portland cement paste undergoes shrinkage during hydration [15–17]. The reason is that the volume of hydration products is less than that of consumed water and cement [18]. This leads to obvious shrinkage and even cracks of cement paste [19,20]. As Q phase is close to high aluminate cement [9], together with sulfate phase, shows a potential to be used as expansive admixture [18,20]. The hydration of Q phase in presence of gypsum is probably the key factor that influences its expansion in Portland cement paste or concrete. Hence, understanding the basic hydration of Q phase-gypsum system is crucial for the application of Q phase in Portland cement paste or concrete, in order to acquire shrinkage neutral cement.

Therefore, in order to increase the early strength and decrease the shrinkage of concrete by using Q phase, understanding of the hydration mechanism of Q phase with gypsum is very important. However, current researches in this field are mainly focus on the synthesis, structure and firing kinetics of Q phase [1,3,4]. There is still limited information regarding the hydration behavior of Q phase with and without gypsum in the present. The mechanisms controlling the hydration rate or microstructural development are still unclear. In this work, reaction kinetics of Q phase-gypsum system was investigated at 20 °C. Effects of gypsum dosage on the mechanical properties of Q phase-gypsum samples were considered. And the hydrates formed as a function of gypsum content were analyzed by XRD, IR and thermal analysis etc. Finally, the microstructural development of Q phase-gypsum system was investigated

2. Experiment

2.1. Materials and sample preparation

2.1.1. Q phase synthesis

Q phase was synthesized by sintering compacted powder of $CaCO_3$, Al_2O_3 , SiO_2 and MgO at 1350 °C for 4 h preceded by a stage of 3 h at 1000 °C to ensure complete decarbonation of $CaCO_3$. The pellets were fired two times to ensure complete reaction of reactants. After firing, the pellets were grounded and sieved, and the powder less than 75 μm was selected. The tested density is 2.98 g/cm^3 , and its specific surface area (Blaine value) is 321 m^2/kg . The XRD analysis and images of the microstructure of Q phase are shown in Fig. 1. The quantitative analysis showed traces of C_2AS and $C_{12}A_7$ (about 2.9%).

2.1.2. Q phase-gypsum sample preparation

Gypsum powder (The density is 2.31 g/cm^3 , and its specific surface area (Blaine value) is 335 m^2/kg) was added into the Q phase powder. The gypsum dosage between 0% of total samples and 30% were investigated, which is named Q0, Q1, Q2 and Q3 respectively. These samples were dry mixed in a blender mixer for 24 h and then stored in a drying vessel. The Q phase-gypsum pastes were prepared at room temperature, and the mixing time was 5 min. And a water to solid ratio of 0.5 was used. The water used is clean tap water.

2.2. Testing methods

2.2.1. Mechanical properties

Cubic Q phase-gypsum samples in size of 40 mm \times 40 mm \times 40 mm were prepared for compressive strength test. The compressive strength was tested at 3 d, 7 d and 28 d. Three samples were tested to calculate the average strength. The compressive strength test was conducted in a compression testing machine at a loading rate of 2.40 kN/s.

2.2.2. Calorimetry

The hydration of Q phase-gypsum paste was followed by a C80 isothermal calorimetry. The normalized heat flow and the total heat evolutions of Q phase with different gypsum contents were determined. The heat release of these pastes was recorded continuously until 3 d.

2.2.3. Thermal analysis

A Power-Compensation Differential Scanning Calorimeter was used to obtain the thermos-gravimetric (TG) and differential thermos-gravimetric (DTG) curves of Q phase-gypsum pastes. The thermal tests were conducted at a heating rate of 10 °C/min from 40 °C to 1000 °C under nitrogen atmosphere.

2.2.4. Scanning electron microscopy (SEM) and backscattered electron (BSE) imaging

A FEI QUANTA FEG 450 ESEM fitted with a BSE detector was used. The microscope with an accelerating voltage of 15 kV and a Bruker AXS XFlash Detector for energy dispersive X-ray analysis were used. The hydration was stopped by soaking the paste in ethanol. After soaking in ethanol for 24 h, these samples were in vacuum dryer at room temperature for 24 h. Once dried, the samples were prepared for BSE-SEM observation and EDS test. Before testing, the samples were stored in a drying vessel.

2.2.5. X-ray diffraction (XRD)

The X-ray diffraction patterns were collected with D8 Advance X-ray diffractometer equipped with $Cu K\alpha$ radiation ($\lambda = 1.5405 \text{ \AA}$) at 35 kV and 30 mA. The 2-theta values range from 5° to 75°.

2.2.6. Infrared spectrometry (IR)

Infrared spectroscopy (IR) was performed with Bio-Rad FTS 6000 FTIR by using KBr pellets. The spectra were recorded in the range of 4000–400 cm^{-1} .

3. Results and discussion

3.1. Mechanical properties

The compressive strength of Q phase-gypsum samples at the age of 3 d, 7 d and 28 d are shown in Fig. 2. It can be observed that the compressive strength increases with increasing gypsum addition. The pastes containing 20% and 30% gypsum have much higher strength than reference samples without gypsum. This phenomenon is different from that of the hydrated samples mixing by C_3A , C_2A or CA with gypsum [21,22]. But it also can be observed that the compressive strength of sample containing 10% gypsum and reference sample are similar, which could indicate that a minimum gypsum content is necessary to obtain a higher strength.

Nevertheless, for the sample with 30% gypsum, the 3 d and 28 d compressive strength are 46.7 and 58.8 MPa, respectively. In general, all pastes, with and without gypsum, show high strength at the age of 3 d, after which the compressive strength still increases steadily in the following days. Usually, the differences in the compressive strength of pastes mainly depend on their microstructure. It can be concluded that the microstructure of samples with 20% and 30% gypsum are more compact than that of reference sample, and the Q phase shows a potential to improve the strength of Portland cement pastes, especially at early age.

3.2. Kinetics analysis of the reaction of Q phase-gypsum system

Fig. 3 shows the heat evolution curves of Q phase-gypsum system. Based on the evolution curves, the hydration process of Q phase-gypsum system can be divided into 2 stages. It can be observed that the last obvious exothermal peak characterizing the beginning of second stage, and the peak occurs at later time with

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