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Effect of calcium aluminate cement on water resistance and high-temperature resistance of magnesium-potassium phosphate cement

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HIGHLIGHTS

- Both strength and water resistance of MKPC were improved by CAC.
- CAC-MKPC exhibited better compressive strength after exposure high temperature.
- Revealed the improvement mechanism of CAC on the MKPC.

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ABSTRACT

The effects of calcium aluminate cement (CAC) on strength, water resistance and high-temperature resistance of magnesium-potassium phosphate cement (MKPC) was investigated. The CAC substituted with 20, 30 and 40 wt% of MgO in MKPC. It resulted in higher compressive strength and better water resistance. This improvement is originated from K-struvite crystals coated by the amorphous and hydrous phases from the CAC. Improved high-temperature resistance of CAC-MKPC was observed as well. Spinel $MgAl_2O_4$ formed in CAC-modified MKPC after the sintering.

1. Introduction

Magnesium phosphate cement (MPC) is a type of chemically bonded ceramics, which has advantages over conventional Portland-based cements, such as short setting time, high-early strength, low drying shrinkage, good volume stability and fine bonding strength [1–5]. Therefore, it is widely used as a repair material for civil engineering [6–9]. Magnesium-ammonium phosphate cement (MAPC) and Magnesium-potassium phosphate cement (MKPC) are two different kids of MPC [10]. Both have excellent mechanical properties, yet MKPC is more popular because it does not have strong ammonia smell during the hydration reaction. The acid-base reaction between magnesia and potassium dihydrogen phosphate (KH_2PO_4) can be written as [3,11,12]:

$$MgO + KH_2PO_4 + 5H_2O \rightarrow MgKPO_4 \cdot 6H_2O \tag{1}$$

Currently, MKPC is widely used for airport runways, concrete pavements and bridge decks [13,14]. However, the instability of MKPC in water limits its applications in humid and moist environments. Researchers [15,16] showed that the mechanical properties of MKPC deteriorate greatly after exposure to water for a long time due to its poor water resistance. To improve the water resistance of MKPC, many researches focused on better structure and denser matrix [17–19]. Addition of waterglass, fly ash or silica fume improve residual strength ratio of MKPC [20–23] as well as its microstructure during. Calcium sulphoaluminate cement (CSA) was also used to improve water resistance [24,25]. By itself, CSA has the ability to hydrate in water, harden and gain mechanical strength. Thus, we believed that the addition of hydraulic material can improve water resistance of MKPC. We also believed that similar effect can be achieved when MKPC is mixed with calcium aluminate cement (CAC).

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CAC is not only a hydraulic cement, but also a refractory material [26,27]. CAC-bonded refractory castables are used in steelmaking applications such as the linings of steel ladles, continuous casting tundishes and degasser snorkels since spinel is beneficial to thermal shock and erosion resistance improvements [28–30]. By combining MKPC and CAC we might also achieve improved high–temperature and desired water resistance of MKPC making it suitable for more applications in the high temperature field. The effect of CAC in the MKPC blended cement might be more complex and needs to be thoroughly studied. On one hand, CAC might continue its hydration in water promoting a dense microstructure of the CAC–MKPC blend. On the other hand, at high temperature addition of CAC might improve the internal structure of MKPC as CAC-hydrates can react with MKPC at high temperature and making the composite more resistant to the thermal shock and destruction.

In this study, the compressive strength, water stability and high–temperature resistance of the CA–MKPC blended cement by changing CAC content, P/M values and different temperatures. Besides, the reaction process, reaction products and morphology of microstructures were systematically studied using X-ray diffraction (XRD) and Scanning Electron Microscope coupled with the Energy Dispersive Spectrometer (SEM-EDS).

2. Experimental

2.1. Materials

The dead burned magnesia (MgO) powder with 230–250 m^2/kg surface area and purity of >98% was calcined at 1500 °C for 10 h. Potassium dihydrogen phosphate (KH_2PO_4 or P) and sodium tripolyphosphate ($Na_5P_3O_{10}$ or STP), both with the purity of >99%, were used. Water reducer, ISO-standard sand and calcium aluminate cement (CAC) with 330–350 m^2/kg surface area were used as well. Chemical composition of CAC is listed in Table 1. The performance of CAC was tested according to the universal ASTM C191 and Chinese GB17671-1991 standards, and the results are listed in Table 2.

2.2. Mix proportion and curing

The amounts of CAC that substituted MgO in the CAC-MKPC composite were 20, 30 and 40 wt%. The CAC-MKPC pastes or mortars were prepared with the different mass ratios of KH_2PO_4 to (MgO + CAC). These ratios are called P/M values and they were 1/3, 1/4, 1/5 or 1/6. STP content (STP/M) was 2 wt%. To maintain the same flow, additional water was added to CAC-MKPC composite, therefore different water to cement (W/C) ratios were used. Cement (C) consisted of MgO, CAC and KH_2PO_4. When P/M was 1/4, the W/C were 0.208, 0.212 and 0.216 with the CAC contents 20, 30 and 40 wt%, respectively. For the MKPC mortar, STP content was 2 wt% and W/C was 0.200. For CAC mortar, the W/C was 0.300. The weight ratio of sand to cement (S/C) was 1/1 for all mortar samples. The detailed mix proportions are listed in Table 3. All CAC–MKPC and MKPC samples were cured at room temperature (20 ± 1 °C) and relative humidity (50 ± 5% HR).

2.3. Testing methods

2.3.1. Setting time and compressive strength

The setting time of fresh paste was tested by Vicat Needle tester based on the ASTM C191 standard. A batch of six $20 \times 20 \times 20\ cm^3$ mortar samples was made for compressive strength testing. The prepared mixtures were cast and cured at their corresponding curing schedules, followed by the compressive strength tests at 1 h, 1 day and 7 days with the MTS servo hydraulic testing machine at 1 mm/min.

2.3.2. Water resistance

Water resistance is reflected by the compressive strength retention coefficient after immersion in water. Compressive strength of MKPC was reported to be stable after air curing for 7 days [19]. To shorten the test cycles, the compressive strength of samples cured in air for 7 days was used as a standard. After the initial sample preparation, samples were immersed in water for 28 days. The compressive strength retention coefficient was then calculated using Eq. (2):

$$K = f/F \tag{2}$$

where K denotes the compressive strength retention coefficient, f is the compressive strength in MPa, and F is the standard compressive strength in MPa.

2.3.3. High-temperature resistance

High-temperature resistance is reflected by the residual compressive strength after the heat treatment. Prior to the heat-resistance measurements, all samples were cured at designed curing schedules for 28 days. Then samples were heated in MC-T 2100 F-electric furnace with the heating rate of 10 °C/min up to the target temperatures: 400, 600, 800, 1000 and 1200 °C. For each temperature, samples were held at 400, 600, 800, 1000 and 1200 °C for 3 h during the tests measurements,

2.3.4. XRD analysis

The pastes prepared for XRD analysis were cured at the same condition as mortar samples. XRD analysis was performed by X-ray diffractometer (Model D/MAX-3C, Rikagu, Japan) with Cu Kα radiation. Sample were ground into powder and then soaked in ethanol to stop hydration.

2.3.5. SEM-EDS analysis

The microstructure was examined by scanning electron microscope coupled with the energy dispersive spectrometer (Model S-4800, Hitachi, Japan). Two types of samples were prepared: with the W/C ratio of 5:1 and the normal W/C ration, which was equal to the original mortars. Samples were also immersed in ethanol to stop further hydration.

3. Results and discussion

3.1. Setting time

The effect of the P/M ratio and CAC content on the setting time of the MKPC paste are demonstrated in Fig. 1. Reaction of MKPC is typically fast and the addition of CAC slows it down slightly. After

Table 1
Chemical composition of CAC (wt%).

CaO	Al_2O_3	SiO_2	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	Loss
34.25	52.41	7.81	2.42	0.02	0.05	0.24	0.13	0.21