



# Effects of sulphaaluminate cement on the strength and water stability of magnesium potassium phosphate cement



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## HIGHLIGHTS

- Water-hardening SAC was used to modify the water stability of MKPC.
- Both compressive strength and water stability were improved by SAC.
- Reaction products and morphology were affected by SAC significantly.

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## ABSTRACT

To improve the poor water stability of magnesium potassium phosphate cement (MKPC), the hydraulic sulphaaluminate cement (SAC) was used. In this study, the effects of SAC and phosphate content on the strength and water stability of MKPC were studied. Furthermore, X-ray diffraction and scanning electron microscopy-energy dispersive spectroscopy were used to analyze the products of the reaction. The results show that the compressive strength and water stability were improved by partially substituting SAC for MgO. The highest compressive strength was obtained in the SAC content of 20–30 wt% and the increasing amount of SAC improved the water stability of samples. More phosphate could increase the compressive strength under air-cured condition but significantly decrease the water stability of samples. Mass amorphous hydration products generated by the hydration of SAC, having the cohesive effect on the residual unreacted MgO and K-struvite crystals, increasing the density of the hardened paste.

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## 1. Introduction

Maintenance and repair of concrete structures have been very important for the concrete industry. At present, sulphaaluminate cement (SAC), aluminate cement, geopolymers, phosphate cements, and emulsified asphalt are frequently-used rapid repair materials [1–5]. Among these materials, magnesium phosphate cement (MPC) has been used for rapid repair of airport runways, highways, industrial floors, and bridge decks, etc. owing to its excellent properties [6]. It is a new type of chemical bonding air-hardening cement with high early strength, considerable stiffness and bonding strength, favorable durability, and promising temperature adaptation [7–11]. The outstanding properties of MPC are owing to its reaction characteristic and products. MPC is generally prepared from magnesium oxide (MgO), ammonium dihydrogen phosphate ( $\text{NH}_4\text{H}_2\text{PO}_4$ ) and retarder, in which major reaction occurs between magnesium oxide and ammonium dihydrogen

phosphate. Struvite ( $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ ) is the main reaction product, usually accompanied by schertelite ( $(\text{NH}_4)_2\text{Mg}(\text{HPO}_4)_2 \cdot 4\text{H}_2\text{O}$ ) and dittmarite ( $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ ) [10,12–14]. However, the release of ammonia gas during the reaction limits its application. Hence, the ammonium dihydrogen phosphate was replaced by potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ), by which produced is called magnesium potassium phosphate cement (MKPC). The main reaction product of MKPC is K-struvite ( $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ ), which has a similar structure and physicochemical properties with struvite [15,16].

However, poor stability is the biggest disadvantage of air-hardening materials, the same to MPC and MKPC. In 1990s, Sarkar et al. found that the strength of MPC decreased almost 20% after immersing in water [17]. In addition, the durability of MPC under different conditions was studied by Seehra et al., got the results that the residual strength of MPC further decreased by immersing in water and was only 83% [18]. Furthermore, the MKPC samples cured in water for 28 days showed a significant loss of strength, and the value decreased from 65.3% to 12.0% as the ratio of n ( $\text{MgO}$ ):n( $\text{KH}_2\text{PO}_4$ ) increased from 2:1 to 8:1 [19]. Hence,

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application of MKPC is seriously limited by the poor stability. It was found that the poor water resistance of MPC was mainly because of the dissolution of struvite, which mostly contribute to the strength [19].

Significant amount of work has been done in the last two decades to improve the water stability of MKPC [20–27]. The results could be summarized into three types as follows: First, adding a dosage of substance, which could itself fill the pores or react with the ingredients of MKPC to produce a fine particle or gel to fill pores, thus increase the density of hardened paste. This was achieved by adding fly ash, which was fine enough to fill in the pores of hardened paste, improving the water stability of MKPC [20–23]. The water resistance was also improved to some extent by silicasol and cellulose, for generating a gel-like substance, enhanced the density [24]. Moreover, Fan et al. partially substituted MgO with  $\text{Al}_2\text{O}_3$ , improving the water stability of MKPC for generating a geopolymer-like material, in which the Mg atoms in the  $(\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O})_n$  framework were partially replaced by Al atoms [25]. Second, another insoluble hydration product was promoted by adding substance, such as waterglass, avoiding the dissolution of struvite in MKPC paste under water curing condition [26]. Third, coating waterproof agent is also a way to prevent water entering inside the hardened paste; however, this is not suitable for wearing structures such as pavements, it was easy to grind off [27]. However, an economic and convenient method especially suitable for construct is urgently required.

A material, which could not only improve water stability but also decrease cost is needed. SAC is a traditional high early strength and fast setting cement, comprising  $4\text{CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot \text{SO}_3$  ( $\text{C}_4\text{A}_3\bar{\text{S}}$ ),  $\text{Ca}_2\text{SiO}_4$ ,  $\text{C}_2(\text{A},\text{F})$  (ferrite), and gypsum [28–31]. The high early strength of SAC is because of the hydration of  $\text{C}_4\text{A}_3\bar{\text{S}}$ , generating a high strength adhesive crystal called ettringite ( $\text{Ca}_6[\text{Al}(\text{OH})_6]_2(\text{SO}_4)_3 \cdot 26\text{H}_2\text{O}$ ,  $\text{C}_6\text{A}\bar{\text{S}}_3\text{H}_{32}$ , Aft) [32,33]. In addition, SAC is a type of hydraulic materials, may contributing to the water residual strength of MKPC if blended with it.

In this study, the strength and water stability of MPC blended with SAC by changing the weight ratio of phosphate to magnesium oxide (P/M) and the content of SAC were investigated. Besides, the reaction process, reaction products, and morphology of production were examined by X-ray Diffraction (XRD) and scanning electronic microscopic-energy dispersive spectrometer (SEM-EDS) to explain the mechanism of this novel group.

## 2. Experimental

### 2.1. Raw materials

Magnesium potassium phosphate cement mortars were prepared from a mixture of MgO,  $\text{KH}_2\text{PO}_4$ , borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ), silica sand and water. To obtain dead burned magnesium oxide, MgO with a purity of 99 wt% was calcined at  $1500^\circ\text{C}$  for 10 h and then ground to particles with a specific surface of  $230\text{--}250\text{ m}^2/\text{kg}$ .  $\text{KH}_2\text{PO}_4$  and borax used in the present study were in the chemical grade and were provided by Aladdin of Shanghai, China. Besides, sulphoaluminate cement with a specific surface of  $250\text{ m}^2/\text{kg}$  was used to modify the properties of MKPC. The chemical composition of SAC is listed in Table 1.

**Table 1**  
Chemical composition of SAC.

Components	CaO	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	MgO	$\text{SO}_3$	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$	Loss
Contents (wt%)	40.57	8.42	34.78	3.12	2.32	9.96	0.22	0.05	0.56

### 2.2. Mix proportions

The mix proportions are listed in Table 2. MgO was partially replaced by SAC at 0, 20, 30 and 40 wt% contents. The weight ratio of  $\text{KH}_2\text{PO}_4/(\text{MgO} + \text{SAC})$  (P/M) was 1/3, 1/4, 1/5, and 1/6. For extending the setting time of MKPC paste, 4 wt% borax was used. Besides, the weight ratio of sand to powders, consisted of MgO, SAC and  $\text{KH}_2\text{PO}_4$ , was 1:1, and the weight ratio of water to powders was 0.16.

### 2.3. Curing and testing

The samples for compressive strength testing ( $20 \times 20 \times 20\text{ mm}^3$ ) were prepared according to the mix proportions as described. After dry-mixing MgO,  $\text{KH}_2\text{PO}_4$ , borax, and SAC powders for 1 min in a vertical-axis planetary mixer, silica sand, and water were added to the mixer and then mixed for 2 min. Then, the prepared mixtures were cast and cured in air-cured condition in temperature range  $20 \pm 2^\circ\text{C}$  and the relative humidity of  $50 \pm 10\%$ . The samples were demolded after 30 min.

The compressive strength of the samples was measured using a hydraulic universal testing machine. Each proportion was measured as a set of three samples. The samples after air-curing for 1 h, 1 day, 7 days, and 28 days were tested for compressive strength. For testing water resistance, the samples after air curing for 7 days were immersed in water. After 7 and 28 days (7 + 7 d and 7 + 28 d) in water, the compressive strength was measured. Before testing, the samples immersed in water were taken out of the water and dried for 4 h. Besides, the MKPC pastes without sand were prepared for examine setting time, XRD and SEM-EDS. The setting time was measured with the vicat apparatus. Because of the fast setting of MKPC, the interval time between the initial setting and final setting is very short. Hence the setting time was measured every 15 s and the initial setting time value was chosen as the setting time of MKPC. For microstructure analysis, the reaction of MKPC paste was stopped by adding ethanol at 1 h, 1 day, 7 days, 28 days, 7 + 7 days, and 7 + 28 days. After washing and filtering with ethanol three times, the samples were dried in vacuum at  $40^\circ\text{C}$  for 48 h. XRD analysis was performed using an X-ray diffractometer (Model D/MAX-3C, Rigaku, Japan) with Cu  $\text{K}\alpha$  radiation for testing the reaction products of the MKPC binders. The microstructure of the reaction products was examined by SEM-EDS (Model/S-4800, Hitachi, Japan).

In addition, to demonstrate whether the Aft, which is the main hydration product of SAC, can formation in the MKPC environment or not, pH testing was used to measure the pH environment of the MKPC blended with SAC system. Pure MKPC paste and MKPC blended with SAC paste diluted three times by deionized water as the testing samples, and pH meter (Model/DZ-2, Beijing, China) was used to measure the pH value.

## 3. Results and discussion

### 3.1. Effect of SAC on the setting property of MKPC

The setting time of the cement prepared with varied P/M and different SAC contents are listed in Table 3. The setting time of the pure MKPC paste shorted with decreasing P/M ratio. Soudée et al. hold the idea that the reaction between magnesium oxide

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