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Effect of the combination of fly ash and silica fume on water resistance of Magnesium–Potassium Phosphate Cement

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HIGHLIGHTS

- Investigates water resistance of Magnesium–Potassium Phosphate Cement (MKPC).
- Revealed the mechanism of fly ash and silica fume combinations on the MKPC water resistance.
- Provided the optimal combination of fly ash and silica fume on the water resistance of MKPC.
- The physical and chemical effects on the water resistance of MKPC is proposed.

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ABSTRACT

The effect of the combination of fly ash and silica fume on the water resistance of Magnesium–Potassium Phosphate Cement (MKPC) was investigated, and the improvement mechanism was discussed based on the micro-analysis of XRD, FSEM and pore structure. The results indicate that, the physical effect of the combination of fly ash and silica fume on MKPC is dominated compared with the chemical effect because of the optimization of pore structure. The combination of fly ash and silica fume leads to higher density and later-age compressive strength of MKPCs compared to those without fly ash and silica fume under air curing and water curing, respectively. The strength retention ratios (namely water resistance) of MKPCs with the combination of fly ash and silica fume at 56 days are found to be significantly higher than those without fly ash and silica fume.

1. Introduction

Magnesium Phosphate Cement (MPC) is a new kind of environmental protection cementitious material, which has the properties similar to chemically bonded ceramics. It has become one of the focuses of civil engineering materials specialty in recent years due to its advantages of quick setting, early strength, good volume stability and high bonding strength [1–5]. MPC is attracting for the applications in the rapid repairs of road pavement [6–7] and in the stabilization of harmful or radioactive nuclear waste [8]. MPC includes magnesium–ammonium phosphate cement (MAPC) and Magnesium–Potassium Phosphate Cement (MKPC). With the same excellent mechanical properties, MKPC is gradually attracting more attentions, because it overcomes the problem of releasing the awful ammonia, which happens during the hydration reaction and the molding of MAPC. Researches [2,9–14] showed that the compressive strength of MPC decreased greatly when cured in water environment for a long time. The shortcoming of poor water resistance is bound to affect the durability of MPC, and consequently its application in practical engineering projects will be affected.

In order to improve the water resistance of MPC, the common method is to improve the pore structure, and form the dense matrix as much as possible. Mao et al. [12] have studied the effect of fly ash on the water resistance of MAPC. The results showed that the compressive strength of MAPC with 30% fly ash cured in water for 28 days was equivalent to that without fly ash cured in air curing condition. Huang et al. [15] have also studied the effect of fly ash on the water resistance of MAPC. The results showed that, the water resistance of MAPC with 30% fly ash was improved and its compressive strength increased by about 40% compared with that without fly ash under water curing. Chen et al. [16] have studied the effect of fly ash and silica fume (not the combination of fly

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ash and silica fume) on the water resistance of MKPC, respectively. The results showed that both fly ash and silica fume could improve the water resistance of MKPC, and the improvement effect of silica fume was superior to that of fly ash. Authors previous work has studied the effect of fly ash and silica fume on the water resistance of MKPC [17] and obtained results similar to the study of Chen et al. [16]. Considering the high price of silica fume in China and the difference in fineness of fly ash and silica fume, the better admixture gradation can be obtained by mixing fly ash with silica fume, and subsequently the water resistance of MKPC may be greatly improved (due to combined action of fly ash and silica fume).

Based on the macro and micro tests, the improvement effects of the combination of fly ash and silica fume on the water resistance of MKPC is investigated, and its effect mechanism is explored in this paper.

2. Raw materials and test methods

2.1. Raw materials and mix proportion

Magnesia powder (MgO or M), originally obtained from Haicheng city, Liaoning province, China, was calcinated in the laboratory under 900 °C for 1.5 h, and the MgO content was 92.55%. The admixtures used were fly ash (FA) and silica fume (SF) with mean particle size of 12.504 μm and 0.612 μm, respectively. In addition, industrial grade potassium dihydrogen phosphate (KH_2PO_4 or P) and borax ($Na_2B_4O_7{\cdot}10H_2O$ or B) were used in this study. The purity of KH_2PO_4 and $Na_2B_4O_7{\cdot}10H_2O$ was more than 98% and more than 95%, respectively. KH_2PO_4 was used as the acid compound. Borax was used as the retarder. Borax can decrease the reaction rate by forming oxide films on the surface of MgO. The chemical compositions of the raw materials are listed in Table 1.

Considering that the price of silica fume is much higher than that of fly ash in China and the maximum mixing amount of fly ash can reach 20% (in the test of only mixing fly ash), the mix proportion of the combinatorial mixing test was obtained by varying the content of fly ash and silica fume (Table 2). The purpose was to reduce the cost of MKPC and improve its water resistance by optimizing the gradation of the combination of fly ash and silica fume. The mix proportion of MKPC without fly ash and silica fume (Group 0) had, weight ratio of KH_2PO_4 to MgO (P/M) of 1/3, water to M + P (W/C), where C means M + P, of 0.16, and borax to MgO (B/M) of 0.02, as shown in Table 2. Fly ash and silica fume were added by replacing part of P + M of MKPC, and the mix proportions of MKPC with the combination of fly ash and silica fume were listed in Table 2. When part of P + M of MKPC was replaced by fly ash and silica fume, C denotes M + P + FA + SF in Table 2.

2.2. Test methods

Specimens for the compressive strength test were formed in the molds of 40 × 40 × 160 mm. Firstly, the raw materials (M, P, B, FA, SF) were stirred for 3 min without water in a mixer. Secondly, after the water was added, the mixer began to work for 30 s in low speed and then 90 s in high speed. Thirdly, the mixture was put into the mold of 40 × 40 × 160 mm rapidly, and then shook it for 120 s on a vibration table to form the dense MKPC paste. Finally, the specimens were demolded 1 h after casting and were cured in air (20 ± 2 °C with a relative humidity of 70%) and water (20 ± 2 °C, the specimens were submerged into water) curing conditions respectively for different ages. The compressive strengths of the specimens at 3 days, 7 days, 28 days and 56 days were tested according to Method of Testing Cements-Determination of Strength (GB/T17671-1999) [18].

MKPC was a kind of early strength cement. The compressive strength of MKPC at 1 h could reach 50% of the compressive strength at 56 days under air curing. So the MKPC specimens were demolded at 1 h after casting. Based on the existing evaluation indexes for water resistance [10,19–20], the strength retention ratio (K) was taken as evaluation index for MKPC. Firstly the specimens were demolded 1 h after casting, and then cured in air and water curing, respectively. Secondly, the compressive strengths of the specimens were tested under two curing conditions (namely air curing and water curing), and the specimens cured in water were taken out 3 h before testing. The ratio of the compressive strength cured in water to that cured in air (the strength retention ratio, K) at certain ages (3 days, 7 days, 28 days, and 56 days) were used to evaluate the water resistance of MKPC. The higher the strength retention ratio was, the better the water resistance of MKPC. The 'K' of the specimen can be calculated by Eq. (1):

$$K = f/F \times 100\% \tag{1}$$

where K denotes the strength retention ratio of the specimen at a certain curing age, f denotes the unconfined compressive strength of the specimen at a certain water curing age (MPa) and F denotes the unconfined compressive strength of the specimen at a certain air curing age (MPa).

The samples for micro tests were taken from the broken specimens after the compressive strength test. The pore structures of the samples were measured with PoreMaster GT-60 mercury porosimeter. The hydration product morphologies and chemical components of the samples were analyzed by Nova Nano SEM 230 Field emission scanning electron microscope (FSEM) and X-ray diffraction (XRD), respectively.

Table 2
Mix proportion of MKPC.

Group	P/M	W/C	B/M (%)	FA (%)	SF (%)
0	1/3	0.16	2	0	0
1	1/3	0.16	2	5	5
2	1/3	0.16	2	5	10
3	1/3	0.16	2	5	15
4	1/3	0.16	2	10	5
5	1/3	0.16	2	10	10
6	1/3	0.16	2	10	15
7	1/3	0.16	2	15	5
8	1/3	0.16	2	15	10
9	1/3	0.16	2	15	15
10	1/3	0.16	2	20	5
11	1/3	0.16	2	20	10
12	1/3	0.16	2	20	15

3. Results and discussion

3.1. Macroscopic experimental results and discussion

3.1.1. Compressive strength

Figs. 1–4 show the effects of the dosages of silica fume on the compressive strengths (Rc) of MKPC under two curing conditions when the content of fly ash is 5%, 10%, 15%, 20% respectively. The compressive strengths of MKPC show a trend of first increase and then decrease with the increase of silica fume dosages from 5% to 15%. Silica fume has large surface area and would absorb mixing water to reduce the flowability and so some of the strength loss for 15% of silica fume maybe due to the poor uniformity of the material. Fig. 5 shows that the compressive strength of Group 0 (MKPC without fly ash and silica fume) under air curing was higher than that of Group 0 under water curing. Also the compressive strength of Group 0 under water curing decreased quickly with the increase of curing age, showing the poor water resistance (Fig. 5). For different contents of fly ash, the compressive strengths of MKPC with 10% silica fume were the highest, which exceeded the compressive strengths of Group 0 under both air and water curing conditions at the curing ages of 28 days and 56 days (Figs. 1–4).

When the content of fly ash was 5% (Fig. 1), the compressive strength of MKPC with 10% silica fume cured in air curing condition for 56 days reached 83.02 MPa, showing an increase of 10.87 MPa compared with that of Group 0 (Fig. 5). The highest compressive strength of MKPC cured in water condition for 56 days

Table 1
Chemical composition of raw materials (%).

Material	MgO	SiO_2	CaO	Al_2O_3	Fe_2O_3	Na_2O	K_2O	SO_3	LOI
M	92.55	2.68	2.54	0.60	0.96	–	0.015	0.061	0.27
FA	1.47	50.02	7.52	24.96	4.46	0.78	1.08	1.06	4.01
SF	0.05	97.57	0.03	0.06	0.02	–	0.78	–	2.26