# Construction and Building Materials 105 (2016) 384-390

Contents lists available at ScienceDirect

# **Construction and Building Materials**

journal homepage: www.elsevier.com/locate/conbuildmat

# Effects of fly ash and quartz sand on water-resistance and salt-resistance of magnesium phosphate cement



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# HIGHLIGHTS

• Strength loss of MPC soaked in different solutions for 1 year is studied.

• The effects of fly ash and guartz on strength loss of soaked MPC.

• Trends of strength loss of MPC soaked in different solutions.

#### ARTICLE INFO

Article history: Received 28 February 2015 Received in revised form 29 November 2015 Accepted 22 December 2015 Available online 28 December 2015

Keywords: Magnesium phosphate cement Salt solution Water resistance Compressive strength Mechanism

# ABSTRACT

The loss of strength of MPC in water, Na<sub>2</sub>SO<sub>4</sub> solution, and NaCl solution are studied. The samples after soaking in water and solutions are analyzed by XRD and SEM. The results show that, for the same type of the samples soaked in different solutions for a given duration, the effects of solution on strength are in decreasing order: water > NaCl solution > Na<sub>2</sub>SO<sub>4</sub> solution. For the different samples soaked in the same solution for a given duration, the strength of samples containing fly ash is larger than that of samples in absence of fly ash, and the strength of sample containing quartz sand is larger than that of samples in absence of quartz sand.

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## 1. Introduction

The aging-related deterioration is very common in the life cycle of concrete structures, thus the selection of proper repair materials has significant importance [1]. In some cases, fast repair is needed for deteriorated structures to regain the daily service in a short time, such as repair of airfield runways and pavement [2]. In different types of cementitious patch repair materials, MPC attracts considerable attention, because of the short setting time and high early strength [3–5].

MPC was first developed in 1939 by Prosen [6] as refractory materials for the application in casting dental alloys. In previous studies, the primary raw materials were dead burned MgO [7] and ammonium phosphate salt-diammonium hydrogen phosphates ((NH<sub>4</sub>)<sub>2</sub> HPO<sub>4</sub>) [8] or ammonium dihydrogen phosphate (NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>) [9]. The major problem of ammonium magnesium

\* Corresponding author. E-mail address: Jiaqi.li@berkeley.edu (J. Li). phosphate cements is the release of ammonia during the hydration, which impedes its wide application [10]. Thereafter, potassium dihydrogen phosphate was used to replace ammonium phosphate [11], and this type of MPC was studied [12]. Subsequently, the addition of fly ash, ground blast furnace slag and low-grade magnesium oxide by-product was proposed, which improved the performance and properties of MPC or/and reduced its cost [13-22].

MPC has several noticeable advantages, such as short setting time, high early strength, and high bond strength. Thus, it has been widely used as patch repair material in concrete structures [15]. The setting time and mechanical properties of MPC have been studied by many researchers [16,23-25]. The effects of phosphorous/magnesium ratio, water/solid ratio, and curing age on the microstructure and thermal conductivity were investigated [26,27]. A few studies have investigated the durability of MPC [28,29], including water-resistance. However, few research concerns salt-resistance of MPC. Sarkar [14] presented that the largest loss of compressive strength in water soaking for 90 days was



about 20%, after the curing age of 28 days. Seehra et al. [13] proposed that the residual strength of MPC in long-term water soaking was 87% of compressive strength at 28 days. Gai et al. [30] concluded that the addition of silica and cellulose effectively improved the water-resistance, but reduced its strengths. Shi et al. [31] investigated the effects of waterglass on water-resistance of MPC. Li et al. [32] researched that compound additives and modified stearic acid-styrene acrylic acids copolymer were capable of improving the water-resistance of MPC. Yang et al. [28] described that, for the MPC materials soaked in water or 3% NaCl solution for 3 days, the strength loss occurs, but it is not large enough to affect the application of MPC. In this work, the properties and micro-scale variation of MPC soaked in water and salt solution were studied, the effects of different admixtures on strength of MPC after soaking were investigated, and the causes of the results were analyzed by micro-scale tests.

#### 2. Raw materials and methods

#### 2.1. Raw materials

Magnesia in the form of pale yellow powder was directly calcined from magnesite at 1600 °C. The physical properties and chemical compositions of calcined MgO and fly ash are given in Table 1. Potassium dihydrogen phosphate was in the form of white crystalline powder, which contained  $\rm KH_2PO_4$  of 98%. The content of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>· 10H<sub>2</sub>O in borax was 99.5%. The quartz sand used in this study was ISO standard sand. Specific surface area was analyzed using an automatic specific surface area tester (F-Sorb2400, Gapp, China).

#### 2.2. Mix proportion

The mix design of MPC sample is shown in Table 2. Where, P/M is the molar ratio of KH<sub>2</sub>PO<sub>4</sub> to MgO. The dosage of borax is solid dosage and calculated by weight of MgO. Fly ash is used to replace the raw materials of binder, MgO and potassium dihydrogen phosphate, by mass. Sand ratio denotes the mass ratio of quartz sand to total raw materials of binder, namely fly ash, MgO and potassium dihydrogen phosphate.

## 2.3. Methods

Potassium dihydrogen phosphate, fly ash, borax, quartz sand and water were mixed in given proportions, and stirred for 2 min. Then magnesia powder was added to the mixer to obtain MPC paste. The paste was cast and placed in molds. The samples were demolded after 30 min. And the curing conditions were  $20 \pm 2$  °C, RH (50 ± 5)% and 28 days. The concentration of Na<sub>2</sub>SO<sub>4</sub> solution and NaCl solution was 1 M. After curing of 28 days, three groups of samples were soaked into pure water, 1 M Na<sub>2</sub>SO<sub>4</sub> solution and 1 M NaCl solution, respectively. Compressive strengths were tested at 1, 3, 6, and 12 months. Before each test, the samples were stored in ambient temperature and ambient relative humidity for 1 day to remove the moisture on sample surfaces. XRD and SEM were tested after soaking of 12 months.

Based on ASTM C191 – 13 Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle, the setting time of MPC was obtained. According to ASTM C109 – 13, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens), the compressive strengths of MPC samples were tested. Identification of the crystalline phases was performed using an X-ray diffractometer (D8 Advance, Bruker, Germany) with CuK $\alpha$  radiation ( $\lambda = 1.5418$  Å). The scans were collected from 5° to 75° 20 at a 1°/min scan rate. The scale factor, sample displacement, 20 shift, and cell parameter were refined for quantification. The crystallographic information files utilized for pericalse, struvite-K, mullite, quartz, sodium chloride and thenardite were PDF# 45-946, 75-1076, 15-776, 11-252, 5-628, and 37-1465. Degradation products were examined by imaging the surfaces in a scanning electron microscope (Quanta200, Fei, Japan).

#### Table 2

Mix proportions of MPC.

No.	P/M	Borax content (%)	Fly ash replacement	Sand ratio	Water/cement ratio
1	1/4.5	5	0	0	0.12
2	1/4.5	5	20%	0	0.12
3	1/4.5	5	0	1/1.5	0.12
4	1/4.5	5	20%	1/1.5	0.12

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Compressive strength and setting time.

No.	Sample					
	Setting time/min	Compressive strength/ MPa				
		3d	28d			
1	10	35.2	47.4			
2	15	34.8	54.8			
3	10	30.4	45.2			
4	15	30.2	53.4			



Fig. 1. Strength variation of samples soaked in water.

# 3. Results and discussion

# 3.1. Compressive strength and setting time

Table 3 indicates that (1) Setting time of each sample was very short, setting time of fly ash incorporated sample was relatively longer, and quartz sand does not alter the setting time of MPC (2). The compressive strength of each sample at 3 days developed very fast, and obtained a relatively high strength at 28 days. The compressive strength of Sample 3 at 3 days was 67% of strength at 28 days. The compressive strength of fly ash incorporated sample at 3 days was lower than that of sample in absence of fly ash, while its compressive strength at 28 days was higher than MPC sample in absence of fly ash. The compressive strength of Sample 2 at 3 days was 1.1% lower than that of Sample 1. And its

Table 1
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Physical properties and chemical compositions of calcined MgO and fly ash.

Samples	MgO/%	CaO/%	SiO <sub>2</sub> /%	Al <sub>2</sub> O <sub>3</sub> /%	Fe <sub>2</sub> O <sub>3</sub> /%	Loss/%	Density/g/cm <sup>3</sup>	Bulk density/g/cm <sup>3</sup>	Specific surface area/cm <sup>2</sup> /g
Magnesia	91.7	1.6	4	1.4	1.3	-	3.46	1.67	805.9
Fly ash	1.7	6.2	45.3	25.4	11.4	7.5	2.31	0.81	4013

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