Construction and Building Materials 151 (2017) 43–51

Contents lists available at ScienceDirect

Construction and Building Materials

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

Effect of curing regime on water resistance of magnesium–potassium phosphate cement

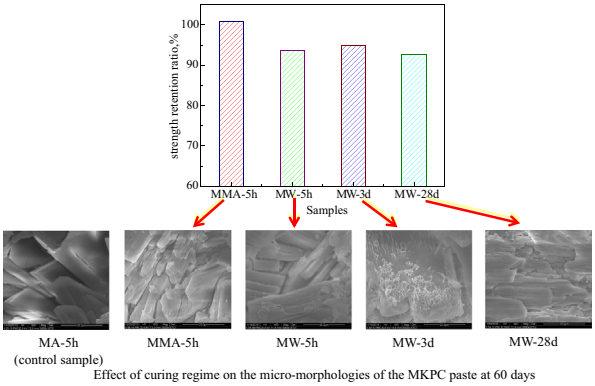
Linlin Chong [a], Jianming Yang [b,*], Caijun Shi [a,*]

[a] College of Civil Engineering, Hunan University, Changsha 410082, China
[b] College of Civil Engineering, Yancheng Institute of Technology, Yancheng 224051, China

HIGHLIGHTS

- Factors affecting the water corrosion behavior of MKPC are studied.
- The compressive strength, mass, pH value and leaching amounts were investigated.
- Both strength residual ratio and mass loss rate were used as deterioration indexes.
- Deterioration mechanisms of MKPC paste were characterized by microstructural observation.

GRAPHICAL ABSTRACT



Effect of curing regime on the micro-morphologies of the MKPC paste at 60 days

ARTICLE INFO

Article history:
Received 20 October 2016
Received in revised form 3 June 2017
Accepted 10 June 2017

Keywords:
Magnesium–potassium phosphate cement
Water attack
Strength residual ratio
Mass loss rate
pH value
Microstructure
Deterioration mechanism

ABSTRACT

In this paper, the effects of curing conditions (dry air, moist air and water), state of water (static water and flowing water) and initial air curing time (5 h, 3 d and 28 d) on the corrosion behavior of magnesium–potassium phosphate cement (MKPC) paste subjected to water attack were investigated. The strength residual ratio and mass loss rate were used as deterioration indexes of MKPC paste in water. The pH value and leaching amounts of soaking liquid were also employed to evaluate the water corrosion behavior. X-ray diffraction (XRD), scanning electron microscopy (SEM), thermo-gravimetry analysis (TG) and mercury intrusion porosimetry (MIP) were used to investigate the microstructure of MKPC paste before and after water corrosion. The experimental results showed that the water environment lead to a lower compressive strength of MKPC pastes, but the strength loss of MKPC paste under the flowing water curing was higher. Furthermore, prolonged initial air curing time could improve the water resistance of MKPC pastes, the specimens cured in air for the first 3 days then in water showed the best water resistance among all specimens immersed in water for 60 days. The deterioration mechanism of MKPC paste derived from the strength and mass loss test results was also clarified based on microstructural observation.

1. Introduction

The degradation of Portland cement concrete exposed to corrosive environments is a key factor affecting the service life and maintenance costs of concrete structures [1]. A variety of materials have been used to repair degraded concrete structures. However, conventional Portland cement-based repair materials usually need long repair cycle and show less satisfactory performance in aggressive environments [2]. Over the last few years, Magnesium phosphate cements (MPCs) that can overcome these disadvantages have been developed. MPCs are essentially acid-base cements

* Corresponding authors.
E-mail addresses: yjm_kk@163.com (J. Yang), cshi@hnu.edu.cn (C. Shi).

http://dx.doi.org/10.1016/j.conbuildmat.2017.06.056
0950-0618/

formed by the hydration reaction between magnesium oxide (MgO) and phosphate at room temperature [3]. However, traditional MPCs with ammonium dihydrogen phosphate, limits its application due to the release of ammonia gas during hydration. A new MPC system, magnesium potassium phosphate cements (MKPC) prepared with MgO and potassium dihydrogen phosphate (KH_2PO_4) has been developed lately. The main reaction product is magnesium potassium phosphate hexahydrate or k-struvite ($MgKPO_4{\cdot}6H_2O$, MKP) [4], according to the following reaction [5]:

$$MgO(s) + KH_2PO_4(s) + 5H_2O \rightarrow MgKPO_4 \cdot 6H_2O(s) \quad (1)$$

MKPC has many superior properties, such as quick setting, high early age and long-term strengths, good bonding with old concrete as well as better durability [6,7]. As a rapid hardening high strength cement, it has been drawing more attention, especially for the rapid repairs of damaged concrete structures, roads, bridges, decks airfields and any other type of application [8–10].

However, the mechanical properties of MPC paste can be significantly weakened when immersed in, or even in contact with water. This can limit its applications in moist environments. Sarkar [11] found that the maximum strength loss of MPC mortar was about 20% cured for 28 days of natural curing then 90 days of water immersion. The strength loss decreased with the increase of intial natural curing time. Seehra et al. [8] reported that the residual compressive strength of MPC sand mortar was 83% after 30 cycles (30 days) immersion in potable water. Paceagiu et al. [12] and Li et al. [13] also examined the mechanical behavior of MPC motars under different curing conditions, and concluded that moist air curing and water curing decreased the strength of hardened MKPC paste. Most researches were concentrated mainly on the macroscopic properties of MPC after water corrosion. There is little information about the difference of microstructure before and after water corrosion to reflect the deterioration mechanism of MKPC pastes. Li et al. [14] analyzed the degradation product after soaked in water and salt solutions with the help of XRD and SEM, and proved that loss of MKP and losse microstructures of MPC are the major cause of strength loss after soaking in different solutions. Zheng et al. [15] have also studied the microstructure changes of MKPC paste, but they were mainly focused on the improvement effects mechanism of the combination of fly ash and silica fume on the water resistance of MKPC paste. This paper investigated the effects of curing condition, state of water and initial air curing time on the short-term water corrosion behavior of MKPC pastes. Both the residual strength and mass loss ratios were used to evaluate the deterioration of MKPC pastes under variable curing environments. The pH value and leaching amounts of soaking liquid were also measured to analyze the water corrosion behavior. In addition, the microstructure was characterized by X-ray diffraction (XRD), thermo-gravimetry analysis (TG), scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP). The causes of strength degradation on pastes exposed to water attack were then clarified. This would offer scientific information on the water corrosion mechanism, help durability design and enlarge applications of magnesium phosphate cement.

2. Materials and methods

2.1. Materials

The raw materials used in this study included dead-burnt Magnesia oxide (MgO; particle size distribution: d10 = 7.52 um, d50 = 38.1 um, d90 = 90.68 um), Industrial grade potassium dihydrogen phosphate (KH_2PO_4, KDP; particle size distribution: d10 = 22.77 um, d50 = 77.6 um, d90 = 246.3 um) and tap water. The chemical composition of MgO is given in Table 1. The composite retarder (CR) [16] is composed of borax ($Na_2B_4O_7{\cdot}10H_2O$, NB), chloride salts (Cl) and disodium hydrogen phosphate ($Na_2HPO_4{\cdot}12H_2O$, NHP) were used as setting retardants. The NHP could decrease the initial temperature and temperature rise rate by absorbing lots of heat when dissolved and transformed in the paste, improve the pH value of paste, these effectively reduce the dissolution rate of reactants and slow down the hydration reaction rate [17].

2.2. Specimen preparation

The mass ratio of MgO to KDP was kept at three according to the previous work from the authors [18]. Composite retarder (CR) of 14.7% MgO in weight (NB:2%, NHP:8%, Cl:4.2%) was added, the water-to-solid ratio (W/S) used was 0.11. The solid components, including KDP and CR, were first dry-mixed for 1.5 min. Two thirds of water were then added and mixed at a low speed for 1 min. Subsequently, the MgO and rest water were added and mixed at a low speed for 1.5 min and another 2 min at a high speed. The paste was cast into 30 × 30 × 30 mm steel moulds and then compacted. All samples were cured in the cement molding room at 20 ± 2 °C and relative humidity (RH) 50 ± 5% with plastic film covered for 5 h, then demoulded and further cured in different conditions up to testing ages.

2.3. Curing regimes

Samples were exposed to different curing conditions as follows:

(a) Curing regime A (reference): specimens were cured in lab at 20 ± 2 °C and 65 ± 5% RH until testing ages of 3(6,31),7(10,35),15(18,43),28(31,56),45(48,60),60(63,88) days and denoted as MA-5 h(MA-3d and MA-28d).
(b) Curing regime B: specimens were cured in a sealed glass tank at 20 ± 2 °C and 98% RH until testing ages of 3,7,15,28,45,60 days and denoted as MMA-5h.
(c) Curing regime C: specimens were cured in water at 20 ± 2 °C until testing ages of 3,7,15,28,45,60 days and denoted as MW-5h.
(d) Curing regime D: specimens preconditioned in curing regime A for 3 and 28 d, then cured in water at 20 ± 2 °C until testing ages of 3,7,15,28,45,60 days and denoted as MW-3d and MW-28d, respectively.

2.4. Experimental methods

2.4.1. Water resistance test

Strength of the samples was assessed by using a controls AutoTest machine at load rate of 1.0 MPa/min according to the Chinese National Standard GB/T17671-1999 [19]. A minimum of four samples at each ages were prepared for obtaining the reliable mean compressive strength. The strength residual ratio [18,20] was used to determine the strength deterioration of the hardened MKPC paste, which can be calculated as follows:

$$W_n = \frac{R_{cn}}{R_c} \times 100\% \quad (1)$$

where W_n (%) is the strength residual ratio after immersing in water for n days; R_{cn} is the average compressive strength of wet specimens (the wet samples were taken out from water and the compressive strength of them were measured at once each time after completing their prolonged water immersion.) after immersion in water for n days; R_c is the average compressive strength of dry specimens cured under the same days in air condition. The mass variation of hardened MKPC pastes after water soak was another indicator to characterize the water resistance. Six paste cylinders samples with size of 30 mm in diameter and 30 mm in heigh were saturated using a vacuum feed water device for 24 h and the initial mass was recorded. The specimens were taken out from water and washed with flowing tap water to remove the impurities and precipitates from the surface, then dried by using a towel before weigh. The cumulative mass variation at each testing age (ML_t) can be calculated according to Eq. (2).

$$ML_t = \left(\frac{M_t - M_0}{M_0}\right) \times 100\% \quad (2)$$

where ML_t (%) is the mass variation ratio of the specimens during water corrosion; M_0 is the initial saturated-surface dry weight of the specimens before water corrosion; M_t is the saturated-surface dry weight of the specimens after water corrosion at time t.

2.4.2. Water immersion test

Two cylinder specimens with dimension of 30 mm in diameter and 30 mm in heigh were immersed into a plastic-made sealed bottle till testing ages. Deionized water was employed as soaking liquid at a liquid-to-solid ratio (L/S) of 5 $dm^3{\cdot}kg^{-1}$. An approximately 50 ml well-stirred solution was sampled and dried in an oven at 60 °C until constant mass. The mass of residual material in solution was recorded to evaluate the corrosion degree of MKPC paste. The pH value variation of the solutions was measured by a pH-meter with temperature compensation before the MKPC paste samples immersed and after they were removed.

2.4.3. Microstructural analyses

Samples were collected from the fracture surface of the paste specimens after compression strength testing. They were immersed in anhydrous alcohol for about 2–3 days, then vacuum-dried at 60 °C for 24 h. Parts of them were subsequently