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Bond behavior and interfacial micro-characteristics of magnesium phosphate cement onto old concrete substrate

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HIGHLIGHTS

- The effect of M/P and w/c on the bonding properties of MPC pastes was studied.
- Interfacial micro-characteristics of MPC and OPC substrate were investigated.
- Bond mechanism between MPC and OPC substrate was characterized.

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ABSTRACT

In several applications of magnesium phosphate cement (MPC), one encounters a need for bonding old Portland cement (OPC) concrete with MPC. The bonding characteristics between MPC and OPC concrete substrate, were evaluated using flexural and tensile bond strength, failure modes, scanning electron microscopy (SEM) and X-ray diffraction (XRD). The experimental results show that samples having a lower magnesia to phosphate molar ratio (M/P) and water to cement mass ratio (w/c) exhibit higher bond strength and stronger affinity for OPC concrete, as well as better developed interfacial microstructure. Multiple interfacial interactions between MPC and OPC concrete are evidenced by microstructural analysis and XRD: MPC paste can infiltrate into the irregularities of the OPC substrate; etching of the surface of hardened OPC paste occurs under the pH condition of MPC paste; penetration of soluble phosphate creates the potential for the chemical reaction and filling effect at the interface.

1. Introduction

The damage of ordinary Portland cement (OPC) concrete structures will inevitably occur with the prolonging of service life. Predictably, a larger number of existing concrete structures suppose an urgent need of repairing and strengthening. In general, structure rehabilitation in different situations has different requests for repair materials or systems [1]. Sometimes the repair material may be typically required to bear the load within its first few hours for rapid repair reasons; other times, enough importance should be attached to the effectiveness and durability of reparation. In these cases, it is of great significance to make improvements by developing new materials and other aspects that should be considered in the rapid and durable repair. Among various materials available for concrete repair, magnesium phosphate cement (MPC) has been gained more and more attentions with increased researches and applications [2–7].

MPC is a new kind of cementitious material, which can form chemically bonded phosphate product by an acid–base aqueous reaction between dead burned magnesia and acid phosphate [8,9], as shown in Eqs. (1) or (2).

$$MgO_{(s)} + NH_4H_2PO_{4(s)} + 5H_2O_{(l)} \rightarrow MgNH_4PO_4 \cdot 6H_2O_{(s)} \text{ (struvite)} \quad (1)$$

$$MgO_{(s)} + KH_2PO_{4(s)} + 5H_2O_{(s)} \rightarrow MgKPO_4 \cdot 6H_2O_{(s)} \text{ (k-struvite)} \quad (2)$$

Several investigations in the literatures [2,3,8,10–14] have focused on the overall behavior of MPC paste and mortar, and showed that MPC has numerous superior properties when compared with OPC, such as fast setting time, rapid development of mechanical properties, low drying shrinkage, high bond strength and excellent wear resistance. It has been considered promising for rapid repair of highways, municipal roads, airport runways,

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bridges and military engineering [2–7,13]. Moreover, their potential use in the field of stabilization or encapsulation of hazardous wastes has been mentioned in many literatures [9,15,16].

In the field of concrete repair, except for mechanical strength, it is now generally agreed that other properties including shrinkage, bond strength and interfacial permeability of repair material, are also critical to the success of repairing [1,17,18]. Thus, whether the repair system can obtain a compacted interface and strong adhesive strength is one of the key issues that is confronted by MPC repair material. Previous studies demonstrated [4–7,13,19,20] that MPC exhibits favorable bond properties under normal conditions, mainly characterized by high bond strength and low dry shrinkage. Some researchers [5,21,22] believed that an adequate condition of concrete substrate (such as interfacial moisture, roughness and strength of old concrete) is essential for good bond strength of MPC. Apart from those factors, the bonding behaviors of MPC onto substrate concrete could be mainly influenced by different key parameters, including magnesia to phosphate molar ratio (M/P), water to cement mass ratio (w/c) and dosage of setting retarder. Hitherto, this subject has been investigated by few researchers [13,23], but only the bond strength of MPC was addressed in most cases. As in all composite materials, the interface has long been considered as a zone of weakness, where has a complex structure and may involve multiple bond mechanisms [20,24–26]. At microscale, it is the micro-characteristics of the interface that determines the bonding behavior, which eventually plays an essential role in the determination of the overall adhesion properties of the MPC paste with OPC substrate. However, scarce information is available in the literature for understanding the interfacial micro-characteristic and bond mechanism between OPC substrate concrete and MPC better. Thus, the bonding characteristics between OPC substrate and MPC is a vital issue needing to study.

This paper is aiming at studying the interfacial characteristics between the OPC substrate and MPC paste in attempt of providing insights into the kinetics of bond formation. The effect of some key parameters on the bond behavior of MPC was evaluated according to the bond strength and failure modes, and the interfacial microstructures between the OPC substrate and MPC pastes prepared with different formulations was also studied. The micro-characteristics including morphologies, elements distribution and potential reaction products at the interface were investigated systematically by means of scanning electron microscope with energy dispersive X-ray spectroscope (SEM/EDS), and powder X-ray diffractometer. These aspects are of crucial importance for a better understanding of the development of the bond properties in view of effective and durable repair.

2. Experimental programme

2.1. Materials

The 1700 °C burned magnesium oxide (MgO) was sourced from Guolian Refractory Plant, Chongqing, China, with a purity of 95%. The chemical composition and physical properties of magnesia are provided in Table 1. Industrial grade ammonium dihydrogen phosphate ($NH_4H_2PO_4$ or ADP) and potassium dihydrogen phosphate (KH_2PO_4 or KDP) used in current research, had 99% purity and were obtained from Xincheng Phosphate Plant, Chengdu, China. In addition, pentahydrate borax ($Na_2B_4O_7{\cdot}5H_2O$ or borax) with a purity of 99% was supplied by Huanuo Chemicals Company Ltd., Wuhan, China, and used as a setting retarder. Phosphates and borax were subjected to a grinding process of 60 s with high-speed disintegrator prior to use, respectively.

2.2. Mix design

To study different possible interfacial properties and morphologies of new MPC with old OPC substrate, MPC paste was prepared by mixing magnesia, phosphate, borax and potable water according to the designed formulations as shown in Table 2. In all series, the borax to magnesia weight percentage of 12% was employed to ensure that a workable paste can set beyond 20 min. M/P in this paper refers to the molar ratio of magnesia and phosphate. The mass ratio of water to cement is expressed as w/c. All solid reagents were pre-mixed for 60 s in a planetary mixer at low speed. Water was then added to the mix, and was stirred slowly for 90 s. Finally, after 150 s of rapid stir, MPC slurry was obtained, and then poured into the specified mould to form repair sample. All the samples were cured in room at a temperature of 23 ± 1 °C and a relative humidity of 50 ± 5% until testing.

2.3. Analytical methods

Setting time was determined by using Vicat needle according to ASTM standard C191 [27]. One of the objectives of this paper is to study the bond behavior of MPC repair sample, which was commonly evaluated by measuring its joint strength [13,20]. In this study, two kinds of specimens were casted and employed to investigate bond behaviors between the OPC substrate and MPC paste, as shown in Table 3. The first specimen for flexural bond test was a prism of 40 × 40 × 160 mm^3 made by half OPC substrate and half of MPC paste. The mix proportion of the substrate OPC concrete was based on a 28-day flexural strength of 9.6 MPa and compressive strength of 61.8 MPa, consisting of P.O 42.5 ordinary Portland

Table 1
Physical and chemical properties of the magnesia.

Mass fraction of the major oxides (wt%)							Specific surface area (m^2/kg)	Density (g/cm^3)
MgO	Al_2O_3	CaO	SiO_2	Fe_2O_3	P_2O_5	TiO_2		
95.25	0.25	2.14	1.53	0.60	0.10	0.06	228	3.45

Table 2
Mixture proportion and setting time of MPC pastes.

Series[a]	P[b]	M/P	w/c	Components by weight ratio				t_{IS}[c]	t_{FS}[c]
				MgO	P	Borax	Water		
A6W13	ADP	6	0.13	1	0.481	0.12	0.208	35	42
A10W13	ADP	10	0.13	1	0.287	0.12	0.183	29	35
A14W13	ADP	14	0.13	1	0.205	0.12	0.172	24	28
A10W17	ADP	10	0.17	1	0.287	0.12	0.239	30	36
A10W21	ADP	10	0.21	1	0.287	0.12	0.295	27	32
K10W13	KDP	10	0.13	1	0.340	0.12	0.190	25	31

[a] Expression of A6W13 indicates a paste containing ADP and with the M/P of 6 and the w/c of 0.13.
[b] P is short for ADP or KDP.
[c] t_{IS} and t_{FS} is the initial and final setting time of MPC paste, respectively.