



Properties of a magnesium phosphate cement-based fire-retardant coating containing glass fiber or glass fiber powder

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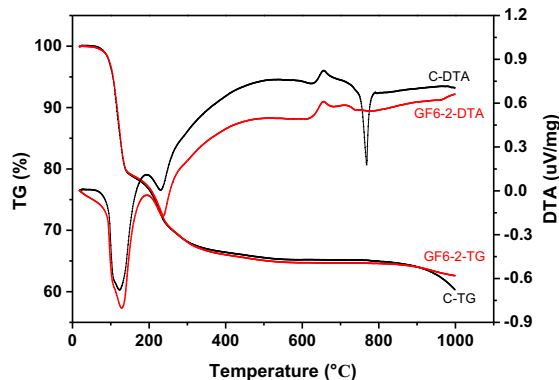
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HIGHLIGHTS

- A new inorganic fire retardant, magnesium phosphate cement (MPC) coating, is proposed.
- The fire retardancy of the new MPC fire-retardant coating was investigated.
- The effects of glass fiber and glass fiber powder on the fire retardancy of the MPC were determined.
- The workability and mechanical performances of the new MPC fire-retardant coating were studied.

GRAPHICAL ABSTRACT



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ABSTRACT

Magnesium phosphate cement (MPC) is a type of chemically-bonded ceramic and has excellent resistance to high temperatures. In this study, the properties of the MPC as a fire-retardant coating were evaluated to expand the application of the MPC. The MPC coating was prepared by blending with glass fiber (GF) or glass fiber powder (GFP) as a mineral admixture (the content was 0%, 2%, 4%, and 6%). The physical and mechanical performances of the MPC pastes were tested and their fire retardancy was investigated in detail. The results showed that the spread fluidity values of the GFP-blended MPC pastes were higher than 200 mm and the initial setting time exceeded 60 min. The bonding strength values of all MPC specimens were greater than 0.6 MPa. The tests results showed that the MPC coating had excellent fire retardancy. During a fire, the free water in the MPC paste and the chemically-bonded water in the hydrate product (struvite of potassium) of the MPC dissipated a large amount of heat, which effectively retarded the spread of the fire. At the same time, the glass fiber and glass fiber powder played an important role in preventing cracks in the MPC coating during the fire retardancy test. With the aid of micro-analyses, such as thermo-gravimetric and differential thermal analysis (TG-DTA), X-ray diffractometer analysis (XRD), and optical micrograph observations, the causes for the excellent fire retardancy of the MPC coating were determined.

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1. Introduction

Magnesium phosphate cement (MPC) is a chemically-bonded ceramic material [1–3] and its properties fall between Portland cement and sintered ceramics. MPC has many superior performances compared with Portland cement, such as high initial strength at ambient temperatures without water curing, strong bonding strength with existing Portland cement concrete substrate, etc. Therefore, MPC is an excellent repair material for concrete structure renovation in civil engineering, such as for highway pavements, airport runways, bridge decks, key municipal roads, and other concrete structures. Furthermore, MPC has many potential applications in the fields of biomedical materials, waste water treatment, and stabilization of toxic and nuclear wastes [2,4–7]. In the biomedical field, MPC is commonly used in dental and bone restorations and in 3D printing for bone replacement material [8]. However, current studies on MPC have mainly focused on the reaction and retardation mechanisms, the influencing factors on the properties of the MPC, and similar subjects [2–3,6,7,9–27]. Some studies have shown that MPC has a high-temperature resistance [28–30], but there are few reports on the use of MPC as a fire-retardant coating. Because most current fire-retardant coatings contain volatile organic components, they are expensive, toxic, and have low resistance to fire. In this study, the properties of MPC by blending with glass fiber or glass fiber powder as a novel inorganic fire-retardant coating is explored for the first time. In this study, the term fire retardant or fire retardancy means that the MPC does not burn and that there is a time delay until the coated object begins to burn.

In previous reports on the properties of MPC under high temperatures, the MPC specimens were placed into a high-temperature furnace rather than using a natural flame. There are many differences between heating the MPC inside a high-temperature furnace and firing the MPC with a natural flame, namely the thermal environment and the heating surface. These differences can produce different experimental results. Therefore, it is necessary to investigate the behaviors of the MPC with a natural flame. One U. S patent describes a fire-retardant coating based on light-burned magnesia instead of dead-burned magnesia (DBM) [31]. However, the preparation temperature is much higher for DBM than for light burned magnesia; therefore, DBM is often used as a refractory material in the metallurgical industry.

This study is expanding the possibility of using MPC as a fire-retardant coating. At the same time, the excellent fire retardancy of the DBM is advantageous for this application. In this study, an MPC paste was used as an inorganic coating and was mixed with glass fiber or glass fiber powder. There are no volatile organic components in the fire-retardant coating. The workability and mechanical performances of the MPC were measured, including the spread fluidity, setting time, and bonding strength. Furthermore, the fire retardant behaviors were studied in detail. The micro-structure of the MPC coating was evaluated using X-ray diffractometer

analysis (XRD) and thermo-gravimetric and differential thermal analysis (TG-DTA). The XRD and TG-DTA results are expected to elucidate the fire retardant property and the mechanisms of the new fire-retardant coating.

2. Experimental procedures

2.1. Raw materials and preparation of the freshly-mixed MPC paste

The raw materials for preparing the fire-retardant coatings were dead-burned magnesia (DBM), mono-potassium phosphate (MPP), a composite retarder (which was prepared by our group), tap water, glass fiber (GF), and glass fiber powder (GFP). The composite retarder consisted of 19% KCl and 81% borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) by weight. The chemical compositions of the DBM were determined by X-ray fluorescence spectrometry (XRF) (S4 EXPLORER, Bruker AXS GmbH, Germany). The chemical compositions of the DBM were 97.97% MgO and 1.24% CaO by weight and the average particle size of the DBM was 45 μm . The MPP, KCl, and borax were all industrial grade and the effective content of the KH_2PO_4 was 98%. Prior to the test, the three chemicals were milled to be smaller than 1 mm by using a sealed high-speed pulverizer. Glass fibers with lengths of 3 mm and 6 mm were used. The softening temperature of the GF and GFP ranged from 680 °C to 900 °C and the melting temperature ranged from 1050 °C to 1150 °C. The diameter of the GFs ranged from 13 μm to 15 μm . In addition, the glass fiber was also milled into powder (GFP) and the average particle size of the powder was 150 μm .

In the setting time test, the magnesia-to-phosphate molar ratio (M/P ratio) ranged from 2.0 to 4.0 with an interval of 0.2 and the initial setting time was about 20 min when the M/P ratio was 2.4; however, the initial setting time was about 10 min for the other M/P ratios; therefore, the M/P ratio of 2.4 was selected. The MPC coating pastes were prepared according to the proportions shown in Table 1. Because borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) contains water, it can increase the water content of the MPC mixtures and result in an increase in the spread fluidity and setting time for mixtures of the same proportion. Therefore, the water content used in the MPC mixture includes the water in the borax. According to the setting time test, the water-to-solids (the DBM and MPP) weight ratio was 0.19, namely, the water-to-DBM weight ratio (Water/DBM ratio) was 0.68.

2.2. Testing methods

2.2.1. Tests of the spread fluidity and setting time

The various raw materials for creating the MPC coatings (Table 1) were weighed and mixed for 30 s using a cement mixer (the rotating speed was 140 ± 5 r/min.). After that, tap water was added and the paste was mixed for 60 s (the rotating speed was 285 ± 10 r/min.). At last, the freshly-mixed MPC paste was ready.

For spread fluidity of the freshly-mixed MPC paste, its test was performed according to the standard GBT 2419-2005 [32]. A mini steel slump meter (the upper and lower bottom inner radius are 18 mm and 30 mm, the height is 60 mm, the wall thickness is 6 mm), a glass plate (larger than 500 mm \times 500 mm), and a ruler were used. During the testing, the glass plate and the mini steel slump meter were first wiped with a wet cloth to avoid absorbing water from the freshly-mixed MPC paste and the mini steel slump meter was placed in the middle of the glass plate. Then, the freshly-mixed MPC paste was poured into the mini steel slump meter and it was lifted up immediately. The MPC paste spreads in a circular shape and the resulting diameter represents the spread fluidity of the freshly-mixed MPC paste. The diameter of the circular shape was measured. The spread fluidity test setup is shown in Fig. 1.

Because the difference in time between the initial setting and the final setting of the MPC is very small, only the time of the initial setting was measured in this study. The initial setting time was measured using a Vicat meter and according to the standard GB 1346-2011 [33]. The temperature was 20 ± 2 °C and the relative humidity was $60 \pm 5\%$ during the tests of the spread fluidity and initial setting time.

Table 1
Material proportions of the MPC mixtures (weight ratio).

MPC mixtures	MPP/DBM ratio	Retarder/DBM ratio	Water/DBM ratio	3-mm GF/DBM ratio	6-mm GF/DBM ratio	GFP/DBM ratio
C	1.42	0.10	0.68	—	—	—
GF3-1	1.42	0.10	0.68	0.02	—	—
GF3-2	1.42	0.10	0.68	0.04	—	—
GF6-1	1.42	0.10	0.68	—	0.02	—
GF6-2	1.42	0.10	0.68	—	0.04	—
GF6-3	1.42	0.10	0.68	—	0.06	—
GFP-1	1.42	0.10	0.68	—	—	0.02
GFP-2	1.42	0.10	0.68	—	—	0.04
GFP-3	1.42	0.10	0.68	—	—	0.06

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