



# Improvement in fluidity loss of magnesia phosphate cement by incorporating polycarboxylate superplasticizer

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## HIGHLIGHTS

- PCE is ineffective to plasticize MPC paste.
- PCE can improve the fluidity retention of MPC paste.
- PCE can delay the hydration of MPC paste.
- PCE most likely enhances the stability of magnesium-based borate layer.

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## ABSTRACT

Effect of polycarboxylate superplasticizer (PCE) on fluidity of magnesia phosphate cement (MPC) paste was investigated. The fluidity and fluidity loss were assessed with mini slump. Hydration process of MPC in the presence of PCE was analyzed with setting time, hydration heat, X-ray diffraction (XRD), scanning electron microscope (SEM), and conductivity. Adsorption behavior of PCE and borax in MgO suspension was studied with total organic carbon analyzer, inductive coupled plasma emission spectrometer, and X-ray photoelectron spectroscopy. The results show that PCE has almost no effect on plasticizing MPC paste, but can obviously increase the 30 min fluidity. The ineffectiveness of dispersion of PCE is because borax in MPC can significantly obstruct adsorption of PCE as well as invalid PCE adsorbed on the surface of MgO particles. The improvement in 30 min fluidity is due to the retarding effect of PCE on hydration of MPC, which can be illustrated from the results of setting time, hydration heat, XRD and SEM. The main reason for retardation is because PCE can enhance the stability of magnesium-based borate layer and can also hinder the formation and growth of struvite by adsorption. Such results would be expected to provide guidance on the design of new type of retarder for MPC and also offer useful experience for controlling the fluidity of MPC paste over time in practical engineering.

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## 1. Introduction

Magnesia phosphate cement (MPC) has been widely used in quick repair and reinforcement of structure area, such as rapid repair of damaged roads, bridges and runways [1–5], and also employed for solidification of waste and biomedical application areas [6,7]. The main reason for these special uses is attributed to its remarkable advantages, such as near-neutral pH, low water

demand, ultra-fast setting, ultra-early strength, high bond strength and good volume stability [8–12].

The hardening process of MPCs involves an acid-based reaction between magnesia (MgO) and some compounds containing phosphate ions [13–16]. And the most popular phosphates used are mono-ammonium dihydrogen phosphate ( $\text{NH}_4\text{H}_2\text{PO}_4$ , MDP) [17,18] and potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ , KDP) [19–24]. Many mechanisms have been proposed in the past several decades [25,26]. Taking MgO-MDP for example, the mechanism can be described as follows:

At the very beginning, because of excellent solubility of MDP, the ionized  $\text{H}^+$  can react with MgO, accelerating the release of  $\text{Mg}^{2+}$  into solution [27], and  $\text{Mg}^{2+}$  can react with water molecules

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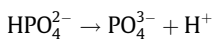
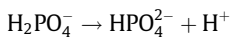
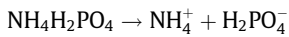
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**Table 1**  
Chemical composition of MgO.

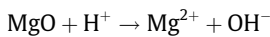
	Loss	MgO	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MnO
wt%	0.80	94.87	0.26	3.17	0.17	0.53	1.65	0.03

to form the compounds, which can substitute the water molecules in the process of wetting MgO. At the same time, the ionized PO<sub>4</sub><sup>3-</sup> and NH<sub>4</sub><sup>+</sup> ions can react with Mg<sup>2+</sup> to develop a struvite network [28].

Dissolution of MDP can be described as follows:

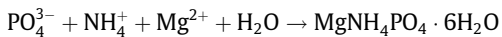


Dissolution of MgO can be described as follow:



In this process, ionized Mg<sup>2+</sup> can be combined with water molecules to form the complexes.

Formation of struvite is shown as follow:



The formation of struvite is very fast, resulting in considerably fast setting. Generally, this can be effectively delayed by the addition of retarder [29–31]. With addition of borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O), the ionized borax can react with Mg<sup>2+</sup> to form a magnesium-based borate layer on the surface of MgO particles [30,32]. It is because of this layer that the dissolution of MgO can be obstructed and the contact of PO<sub>4</sub><sup>3-</sup> and NH<sub>4</sub><sup>+</sup> with Mg<sup>2+</sup> can also be hindered. In this case, the formation of struvite can be delayed. Nevertheless, with time going on, the layer would be broken, and then the release of Mg<sup>2+</sup> would be accelerated to hasten the formation of struvite. Based on discussion above, the stability of the magnesium-based borate layer is of great importance for controlling the hydration at the very early age.

However, it is worth noting that borax or boric cannot obviously improve the fluidity loss of MPC paste, despite the delay of setting process can be obtained, which means that excellent fluidity retention of MPC paste cannot be gained with the increasing dosage of borax. Another problem is that excessive amount of borax in MPC can exert obviously negative effect on mechanical performance. In this study, an attempt to retard the hydration of MPC by incorporating polycarboxylate superplasticizer (PCE) was made, and the effect of PCE on fluidity and fluidity loss of MPC paste was discussed. The fluidity and fluidity loss were assessed with mini slump. Effect of PCE on hydration of MPC was analyzed with setting time, hydration heat, X-ray diffraction, and scanning electron microscope. The dissolving process of MPC was characterized with conductivity and pH value. Adsorption behavior of PCE and borax was studied with total organic carbon analyzer, inductive coupled plasma emission spectrometer, and X-ray photoelectron spectroscopy. Finally, a model was proposed to illustrate the mechanism behind. Such results were expected to provide guidance on the design of new type of retarder for MPC and also offer useful experience for controlling the fluidity of MPC paste in practical engineering.

## 2. Experimental

### 2.1. Materials

The chemical composition of magnesia (MgO) was obtained with X-ray fluorescence (Axios advanced, made by PANalytical B.V., Holland), and the results are shown in Table 1. A reagent-grade struvite was used in this study. The particle size distribution of both MgO and struvite was characterized with laser particle size analyzer (LPSA, Mastersizer 2000, made by Marvin, UK), and the results are shown in Fig. 1. MgO and struvite were also characterized with XRD, and the results are shown in Fig. 2.

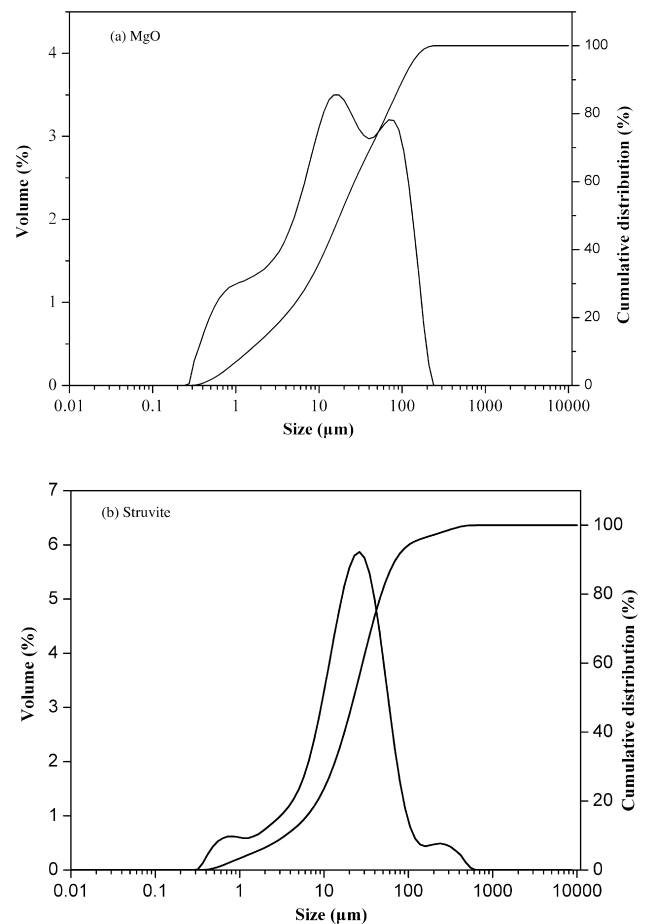
A reagent-grade borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O) and ammonium dihydrogen phosphate (MDP, NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>) were used in this study. Magnesia phosphate cement (MPC) used in this study was prepared with weight ratio of MgO: MDP: borax = 320: 80: 48. The crystal water in borax was not considered here, while the crystal water was considered when the borax solution was prepared.

A commercially available polycarboxylate-based superplasticizer (PCE) was used, and the basic performance and molecular structure, obtained from the company, are shown in Fig. 3 and Table 2. Additionally, PCE used in the experiments were recorded as solid content.

### 2.2. Test methods

#### 2.2.1. Fluidity and fluidity loss

MPC paste with different dosages of PCE (0–0.36 wt% of MgO) was prepared with a water/MPC weight ratio of 0.18:1 (water: 80.64 g; MPC: 448 g), and MgO paste with PCE-borax (PCE: 0.14 wt% of MgO; borax: 0–0.40 wt%) was prepared with a water/MgO weight ratio of 0.20 (water: 60 g; MgO: 300 g), in accordance



**Fig. 1.** The particle size distribution of MgO and struvite.

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