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# Experimental investigation on the volume stability of magnesium phosphate cement with different types of mineral admixtures

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## HIGHLIGHTS

- The influences of mineral admixtures on the volume stability of MPC mortar were investigated.
- Composite admixtures containing ultra-fine fly ash can improve the volume stability of MPC mortar.
- The possible mechanisms were studied by XRD, XRD-Rietveld and ESEM analysis.

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## ABSTRACT

Magnesium phosphate cement (MPC) composited by dead burned magnesia (MgO) and potassium dihydrogen phosphate ($KH_2PO_4$, KDP) is a type of excellent repairing material, and its volume stability has great influence on its applications. This paper studied the effects of different types of mineral admixtures on the volume stability of MPC mortar by testing its volume deformation at different curing ages. The main mechanisms to explain how the mineral admixtures affect the volume stability were studied by XRD and ESEM analyses. The experimental results indicate that the influence of ultra-fine fly ash on the volume deformation is different from that of fly ash and silica fume. The volume expansion increases as the content of fly ash in mortar increases while the volume expansion decreases as the content of ultra-fine fly ash increases. For MPC-fly ash system, the use of ultra-fine fly ash can decrease the volume expansion significantly. Moreover, the addition of dipotassium hydrogen phosphate also has significant influence on the volume stability of MPC mortar. The XRD analysis indicates that the amount of sylvite in MPC mortar is the reason for the changes in the volume stability after adding mineral admixtures. The ESEM imaging confirms that the use of ultra-fine fly ash can decrease the quantity of micro cracks in the MPC mortar.



## 1. Introduction

In the past twenty years, magnesium phosphate cement (MPC) has been attracting more and more attention due to its unique advantages among those repairing materials [1–5]. MPC has a high-early strength due to the quick neutralization reaction between magnesia and phosphate [6–8]. Thus MPC possesses the characteristics of quick setting and high-early-strength. When MPC is applied to repair insecure structures, it takes only about a quarter of an hour to repair the dilapidated concrete and the structure can resume operation within only a few hours [9]. Moreover, even at the temperature of −20 °C, MPC can also set and harden rapidly on the premise of the enormous amount of heat released from the hydration reaction [10]. Compared with the traditional Portland cement, MPC has the following characteristics: (i) quick hardening property, (ii) high-bonding strength, (iii) excellent durability, (iv) good volume stability and (v) broad temperature adaptability. Therefore, MPC is considered to be one of the most widely-used repairing materials for concrete construction in the twenty-first century [11].

The retardation of MPC is one of the most important considerations in practical application. The traditional retarders include boric acid (borax) and poly sodium phosphate which have been studied since several decades ago [12,13]. Recently, some high-efficiency composite retarders prepared by boric acid and poly sodium phosphate have attracted more attentions [14]. On the basis of the results presented in the previous studies, the phosphate buffer solution prepared by potassium dihydrogen phosphate and dipotassium hydrogen phosphate could not only alleviate the negative impact of borax remarkably, but also improve the early mechanical properties to some extent.

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**Table 1**
Chemical composition of mineral admixtures (/%).

| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | $SO_3$ |
|---|---|---|---|---|---|---|---|---|
| FA | 37.9 | 20.1 | 11.2 | 2.34 | 0.83 | 0.75 | 2.37 | 1.35 |
| UFA | 59.9 | 16.3 | 7.34 | 5.08 | 0.56 | 0.43 | 1.84 | 0.70 |
| SF | 95.9 | 0.47 | 0.06 | 0.27 | 0.37 | 0.06 | 1.50 | 0.65 |

Fly ash is a type of common mineral admixture applied in Portland cement, mortar or concrete, which can improve their physical and mechanical properties and reduce the construction cost. Similarly, the application of fly ash in MPC can also improve the workability, durability and physical properties [8,9,13]. Although silica fume and ultra-fine fly ash have been widely used in Portland cement, there are very few literatures on the MPC-silica fume or MPC-ultra-fine fly ash systems. Based on the limited literatures and experiments, it could be found that the silica fume and ultra-fine fly ash can significantly improve the durability and workability of MPC [15].

Volume stability of repairing materials is an important property index in the field of rapid-repairing. After the repairing operation, the high dry shrinkage of the repairing materials may lead to some cracks in the new interface [16,17]. Therefore, the volume stability (shrinkage or expansion) of MPC at different curing ages should be investigated [18–24]. The chemical shrinkage would be occurred during the process of MPC hydration, which is similar to Portland cement. For example, the reaction between magnesium oxide and potassium dihydrogen phosphate occurred in MPC would lead to the volume shrinkage of about 2.09% [18]. Actually, the chemical shrinkage of MPC is always much smaller than 2.09% due to the excessive dead burned magnesium powder and a small amount of unhydrated phosphate. In addition, the volume stability of MPC can be influenced by multiple factors, such as the molar ratio of magnesium oxide to potassium dihydrogen phosphate, the retarder content, water to binder ratio, the binder-sand ratio and magnesium oxide activity [19–21]. So far, the previous studies mainly focused on the effect of fly ash on the mechanical and physical properties of MPC. Therefore, the effects of mineral admixtures on the volume stability of MPC should be studied further more.

In this study, MPC mortar modified by the application of dipotassium hydrogen phosphate was employed. The influences of fly ash, ultra-fine fly ash and silica fume on the volume stability of MPC mortar were investigated by testing the volume deformation at different curing ages. And the mineral admixtures were incorporated with single addition of mineral admixture or composite addition of mineral admixtures. Finally, the possible mechanism for the influences of mineral admixtures on the volume stability was detected through XRD and ESEM analysis.

## 2. Experimental details

### 2.1. Raw materials

MPC is prepared by dead burned magnesium oxide, phosphate and borax at a predetermined proportion (M/P = 2.5, B/M = 8%, W/C = 0.12). The dead burned magnesia with a purity of about 90% and the specific surface of 267.0 $m^2/kg$, was obtained from Taizhou, P.R.C. Industrial grade potassium dihydrogen phosphate was used and partial substituted dipotassium hydrogen phosphate was employed to prolong the setting time. Fly ash (FA), ultra-fine fly ash (UFA) and silica fume (SF) are the main mineral admixtures. The specific surfaces of FA, UFA and SF are 465 $m^2/kg$, 1237 $m^2/kg$ and 15282 $m^2/kg$. The chemical composition of mineral admixtures is shown in Table 1. Common river sand and tap water were used in the experiments. The microstructures of mineral admixtures are shown in Fig. 1.

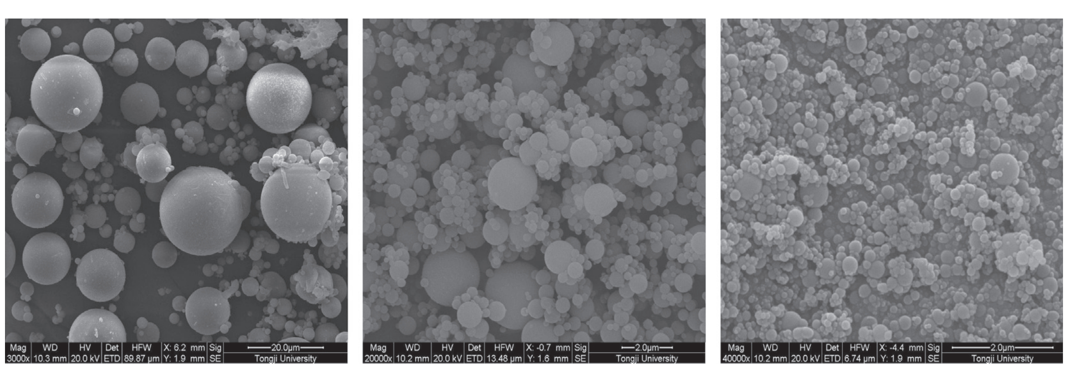
### 2.2. Specimen preparation

In this study, the mass ratio of cementitious materials to sand and water to cementitious materials are fixed at 1.5 and 0.15, respectively. The mix proportion of MPC mortar is illustrated in Table 2. The preparation of MPC mortar separates into two steps. Firstly, MPC, mineral admixture, water and sand were added into the 15 L epicyclic mixer for low-speed mixing for 30 s. And then, a homogeneous mixture was obtained by continue mixing in high speed for 90 s. The fresh mortar were filled into the standard mould 40 mm × 40 mm × 160 mm and then vibrated by concrete vibrator. The specimens were demoulded after 30 min and then maintained at 50 ± 5% RH and 20 ± 2 °C.

### 2.3. Testing method

The volume deformation of MPC mortar is tested and calculated by the following equation.

$$\varepsilon_{age} = \frac{L_{age} - L_0}{L_0 - L_d} \times 10^{-6} \quad (1)$$



(a) Fly ash (b) Ultra-fine fly ash (c) Silica fume

**Fig. 1.** The microtopography of the mineral admixtures, (a) fly ash, (b) ultra-fine fly ash and (c) silica fume.