



## Effect of borax and sodium tripolyphosphate on fluidity of gypsum paste plasticized by polycarboxylate superplasticizer

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### HIGHLIGHTS

- Borax and STPP can retard the hydration of gypsum paste.
- Borax can increase the fluidity of gypsum paste with PCE, while STPP can reduce.
- Retarding effect of borax is responsible for the increase.
- Competitive adsorption and inactivation of PEO by STPP cause the decline.

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### ABSTRACT

High fluidity gypsum-based materials have great potential for applying in grouting, self-leveling floors, and wall construction. Generally, high flowability can be obtained through adding superplasticizer and some salts that have retarding effect on hydration. In this study, influence of borax and sodium tripolyphosphate (STPP) on fluidity of gypsum paste plasticized by polycarboxylate superplasticizer (PCE) was investigated with total organic carbon analyzer, inductive coupled plasma emission spectrometer, dynamic light scattering (DLS), and X-ray photoelectron spectrometer. The fluidity results show that borax can increase the fluidity of the gypsum paste plasticized by PCE, while the change trend for STPP is inverse. The setting time and conductivity measurements show that both borax and STPP retard the hydration of gypsum paste, with contribution to increasing the fluidity. Adsorption results show that both STPP and borax can hinder PCE from adsorbing onto the surface, and thus the fluidity tends to decrease. DLS and conductivity results illustrate that the reaction of borax and STPP with  $\text{Ca}^{2+}$  in the immediate vicinity of the particles exerts negative effect on steric hindrance offered by the long side chain (i.e. polyethylene oxide, PEO). Furthermore, in PCE-borax system, retarding effect plays a dominant role in increasing the fluidity. However, in PCE-STPP system, the reduced adsorption amount as a result of competitive adsorption and the inactivated PEO caused by precipitation between STPP and  $\text{Ca}^{2+}$  play a dominant role in reducing the fluidity. The findings would be expected to provide guidance on the design of superplasticizer-retarder system for gypsum paste.

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### 1. Introduction

In recent years, gypsum-based materials have been attracting great attention in both research and industrial fields [1–3], not only because of its superior performance in the highly efficient construction process, but also due to its greener manufacturing process than that of cement clinkers. In practical engineering, this kind of material has been widely used in grouting materials,

self-leveling floors, wall materials, industrial arts, architecture and 3D printing materials [4–7]. However, fast hydration leads to a very short setting time, generally with the initial setting time less than 5 min, and in this case, the workability of gypsum paste is not that easy to be controlled. According to the experience gained in Portland cement system, this problem can be solved through addition of retarder and superplasticizer; specifically, superplasticizer aims to plasticize the paste, and retarder is used for slowing down the hydration and the formation of hydrates, which has been definitely demonstrated in the literatures [8–13]. Similarly, this theory can also be applied in gypsum system.

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Polycarboxylate superplasticizer (PCE) is accepted as the most efficient dispersant in cement-based material, and it also shows excellent ability to disperse the gypsum particles [14–16]. In addition to protein-based retarder with highly efficient retarding effect on gypsum paste, these inorganic salts, such as STPP and borax, can also delay the hydration of gypsum paste. The main reason for this is that the formation of a layer resulting from adsorption prevents water from contacting particles, thus hindering the dissolution of ions and the formation of hydrates [17,18]. However, in gypsum paste, the ionized borax and phosphate in solution would perturb the adsorption of PCE, with an uncertain effect on dispersion of PCE. As reported in cement paste, an interesting phenomenon was observed that the addition of STPP into PCE system could considerably increase the dispersion ability due to the formation of double layers to increase the thickness of adsorption layer in spite of the decline in adsorption amount of PCE [19,20]. While an opposite result can also be found in the PCE-sodium gluconate system: the addition of more than 0.20 wt% sodium gluconate (SG) could reduce the fluidity, which means that SG can reduce the dispersion ability of PCE system, in spite of retarding cement hydration. The main reason for this decline is due to the fact that SG can obviously hinder PCE from adsorbing onto the surface of cement particles [21,22]. Based on discussion mentioned above, it can be summarized that not all the retarders can enhance the dispersion of superplasticizer, and dispersion ability of PCE-retarder system is affected not only by the retarding effect of retarder but also by its interference to adsorption of PCE.

In gypsum paste plasticized by PCE, the addition of borax and STPP is proposed to enhance the dispersion of PCE, as the two salts are able to delay the hydration of gypsum at the early age. However, the interference of borax and STPP to adsorption of PCE has not been clarified until now. In this study, in order to discuss this uncertain question, the effect of borax and STPP on fluidity of gypsum paste plasticized by PCE was investigated. The dispersion was evaluated with the fluidity tested by mini slump, and adsorption behavior was analyzed with a total organic carbon analyzer, an inductive coupled plasma emission spectrometer, and an X-ray photoelectron spectrometer. The interaction between these two salts and  $\text{CaSO}_4$  was studied through conductivity measurement and dynamic light scattering. Finally, a dispersion model was proposed to reveal the mechanism behind the effect of borax and STPP on plasticizing effect PCE. Such results can give experience for the design of superplasticizer-retarder system in gypsum system.

## 2. Materials and test methods

### 2.1. Materials

#### 2.1.1. Gypsum

Gypsum used in this study is a by-product of phosphorus industry. And the main component is  $\beta\text{-2CaSO}_4\cdot\text{H}_2\text{O}$ . The gypsum was characterized with X-ray Fluorescence (Axios advanced, made by PANalytical B.V., Holland) and X-ray diffraction (XRD, D/Max-RB, Rigaku Inc, Japan), and the results are shown in Table 1 and Fig. 1.

#### 2.1.2. Chemicals

The reagent-grade borax ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ , the crystal water was considered in the experiments) and STPP ( $\text{Na}_5\text{P}_3\text{O}_{10}$ ,  $\geq 99.0\%$ ), made by Sinopharm Chemical reagent Co., Ltd., were used.

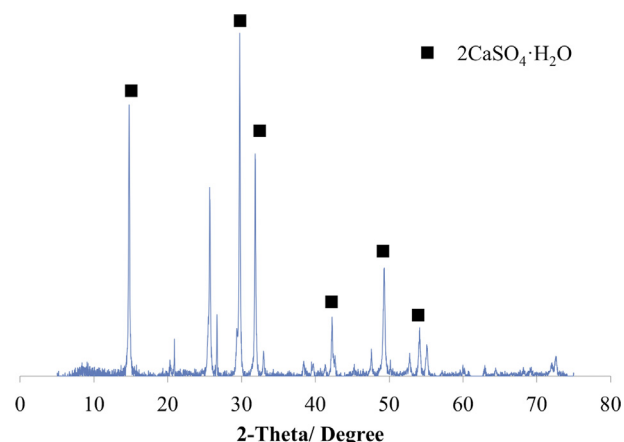


Fig. 1. XRD pattern of gypsum used.

A commercially available PCE was used in this study. The basic performance and molecular structure, provided by the company, are shown in Table 2 and Fig. 2. And the added dosage was recorded as solid amount.

### 2.2. Test methods

#### 2.2.1. Fluidity of gypsum paste

The gypsum pastes were prepared with water/gypsum ratio (W/G) of 0.70 by weight. The additives, namely PCE-borax (PCE: 0.20 wt%; borax: 0–0.30 wt%) system and PCE-STPP (PCE: 0.20 wt%; STPP: 0–0.30 wt%) system, were dissolved in water in advance. It is noticed that the same dosage of PCE (0.20 wt% of gypsum) was used. Fluidity was measured with a cylinder (50 mm inner diameter and 100 mm height) in accordance with the Chinese standard of GB/T 17669, 4-1999. About 50 s after the paste was poured into the cylinder, the cylinder was lifted vertically and the average diameter in two vertical directions was defined as the fluidity value. Each test was measured twice and the average value was considered as the final result.

All the operations were carried out at 20 °C.

#### 2.2.2. Setting time

The paste was likewise prepared according to the procedures shown in Section 2.2.1. And then, the setting time was examined in accordance with the Chinese standard of GB/T 17669, 4-1999. The mold was fully filled with the paste. Then, the steel needle was used to evaluate the setting time. The measurements were repeated every 10 s.

#### 2.2.3. Adsorption behavior

PCE (0.20 g/L, 0.60 g/L, 1.00 g/L, 2.00 g/L), borax (0.40 g/L, 0.80 g/L, 1.20 g/L) and STPP (0.40 g/L, 0.80 g/L, 1.20 g/L) solutions were prepared in advance. The carbon content was examined with total organic carbon analyzer (TOC, Liquid TOC II, made by Elementar, Germany), and boron content and phosphorus content were measured with inductive coupled plasma emission spectrometer (ICP, Optima 4300 DV, made by Perkin Elmer Ltd., USA). The results are shown in Fig. 3. A good linear relationship between element content and concentration of solution has been found, which

Table 1  
Chemical compositions of gypsum.

	$\text{Na}_2\text{O}$	$\text{MgO}$	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{P}_2\text{O}_5$	$\text{SO}_3$	$\text{K}_2\text{O}$	$\text{CaO}$	$\text{TiO}_2$	$\text{Fe}_2\text{O}_3$	$\text{SrO}$	$\text{BaO}$	$\text{F}$	LOSS
wt%	0.13	0.16	0.45	5.14	1.02	48.29	0.10	35.1	0.07	0.28	0.06	0.04	0.70	8.47

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