



Effect of borax on rheology of calcium sulphoaluminate cement paste in the presence of polycarboxylate superplasticizer



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HIGHLIGHTS

- Dispersion of PCE-borax depends on the added dosage of borax.
- Precipitation with Ca^{2+} results in adsorption of borax.
- Borate layer is responsible for retarding and plasticizing effect of borax.
- Competitive adsorption between PCE and borax would take place.

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ABSTRACT

Sulphoaluminate cement (SAC) is increasingly attracting the attention of academic and industry researchers, not only because of its fast setting and high early strength, but also due to the low CO_2 emissions and low preparation temperature. The fast setting also results in poor workability. Generally, this problem can be improved by incorporation of retarders. However, it is often ignored that these retarders may obviously affect the plasticizing effect of superplasticizer by competitively adsorbing onto the surface of the cement particles. In this study, the effect of borax on rheology of SAC paste with polycarboxylate superplasticizer (PCE) has been investigated to gain deeper insight into competitive adsorption. The rheology was characterized with mini slump and rotor rheometer. Adsorption behavior was studied with total organic carbon analyzer and inductive coupled plasma emission spectrometer. The surface performance of cement grains was discussed with X-ray photoelectron spectrometer and scanning electron microscopy. Dispersion model was then proposed to illustrate the mechanism behind the difference in rheology. The results show that borax can be precipitated with Ca^{2+} in the immediate vicinity of cement particles to adsorb onto the surface of cement grains and form a calcium-based borate layer on the surface, which is responsible for the retarding effect and plasticizing effect of borax. Competitive adsorption would take place between PCE and borax, and this would tend to reduce adsorption amount of PCE and reduce the dispersion. The dispersion of PCE-borax system in SAC depends on the dosage of borax: a small amount of borax would increase the dispersion, mainly because the retarding effect and plasticizing effect of borax predominate; while a large amount of borax would reduce the dispersion, because the competitive adsorption effect becomes predominant. It is expected that these results would provide the useful experience for the application of PCE and borax in high fluidity SAC paste in real engineering practice.

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1. Introduction

In recent year, great progress has been gained in calcium sulphoaluminate-based cements. One of this kind of cement, known as sulphoaluminate cement (SAC) mainly composed of calcium sulphoaluminate ($\text{C}_4\text{A}_3\bar{\text{S}}, 4\text{CaO}\cdot 3\text{Al}_2\text{O}_3\cdot \text{SO}_3$) and dicalcium silicate ($\beta\text{-C}_2\text{S}, \text{Ca}_2\text{SiO}_4$), has been widely used in fast construction engineering, fast repair engineering [1], permeability resistance

engineering [2,3], sub-zero temperature engineering, and waste disposal [4]. One reason for these is attributed to its high early strength and fast setting. However, more important thing is that SAC are much greener than ordinary Portland cement (OPC), because of the lower CO_2 emissions and lower preparation temperature, which have significantly attracted the attention of academic and industry researchers. OPC is fired in kilns at temperatures of around 1450 °C, as reported, whereas calcium sulphoaluminate-based cements only need to be fired at temperatures of around 1250 °C, with 200 °C lower than that of OPC [5,6]. Additionally, the resulting SAC clinker is softer than OPC clinker, requiring less

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energy to grind. Furthermore, regardless of the process used or the fuel efficiency, every 1000 kg of calcium trisilicate (C_3S) produced from limestone results in 579 kg of CO_2 gas emission from the chemical reaction, but that for calcium sulphoaluminate is only 216 kg of CO_2 , with a reduction of about 62% relative to OPC [7]. Similarity to OPC, SAC can also consume the industrial by-products or waste materials in preparation and application process [8,9]. For these reasons discussed above, calcium sulphoaluminate-based cements has been considered as a probable alternative to OPC [10].

It is well known that the short setting time and high early strength of SAC paste is attributed to the fast hydration of C_4A_3S at very early time. This special performance also brings about the problem of poor workability of the SAC paste plasticized by superplasticizer, especially by polycarboxylate superplasticizer (PCE). PCE has been reported as the most efficient superplasticizer in the world, which means that high water reducing ratio can be obtained with a small amount of PCE. Usually, the added dosage of PCE is about 0.12–0.20 wt% of cement, which is less than one-third dosage of the traditional water reducers, such as naphthalene-based superplasticizer and lignosulfonate-based water reducer. In that case, slight consumption of PCE resulting from the cement hydration may obviously affect the fluidity of the paste, which means that PCE would be more sensitive than the traditional water reducers in SAC paste. Generally, this sensitivity can be improved with the incorporation of retarders, because these retarders can effectively delay the hydration of SAC to cut down the consumption of free water and PCE [11,12]. That is to say that in superplasticizer-retarder system, superplasticizer provides the main dispersion force, and retarder delays the hydration of cement minerals and the formation of hydration products to enhance efficiency of superplasticizer. Based on discussion above, significant improvement in fluidity of SAC paste with PCE would be expected in the presence of borax, because borax is an effective retarder for SAC in real engineering practice [12].

The dispersion ability of superplasticizer would be influenced by retarder competitively adsorbing onto the surface of the cement particles, as proved by other researchers, and this phenomenon is referred to as competitive adsorption [13,14]. For example, the presence of sodium gluconate in PCE can inhibit the adsorption process of PCE but enhance the dispersion ability of the superplasticizer system [15]. While different mechanism can be found in PCE-sodium tripolyphosphate system: the presence of the phosphate reduces the adsorption amount of PCE but forms a thicker adsorption layer to enhance the dispersion ability [16]. However, the effect of borax on the fluidity of SAC paste with PCE has not been completely understood until now. The objective of this study is, therefore, to investigate the effect of borax on dispersion of PCE and illustrate the mechanism behind competitive adsorption. The rheology of the SAC paste in the presence of borax and PCE was studied with mini slump and rotor rheometer. Adsorption behavior of PCE and borax as well as the surface performance of cement grains was investigated. Dispersion model was then proposed to illustrate the mechanism behind the difference in rheology. It is anticipated that the results would provide experience for the application of PCE in SAC paste in real engineering practice.

2. Experimental

2.1. Materials

2.1.1. Cement

Commercially available sulphoaluminate cement (SAC), in accordance with Chinese standard GB 20472-2006, was used. The chemical composition of the cement was obtained by XRF (Axios advanced, made by PANalytical B.V., Holland), as shown in Table 1.

2.1.2. Chemical additives

The reagent grade borax ($\geq 99.0\%$), made by Sinopharm Chemical reagent Co., Ltd., was used. A commercially available PCE was used in this study. The basic performance and molecular structure, supplied by the company, are shown in Table 2 and Fig. 1.

2.2. Test methods

2.2.1. Rheological measurements

Mini slump

The cement paste was prepared with a water/cement weight ratio of 0.29:1 ($W/C = 0.29$; water: 87 g; cement 300 g) in accordance with the requirements of Chinese standard GB 8076-2008. PCE (0.16 wt% of cement) was added into water in advance. Borax (0–1.40 wt% of cement) was mixed with SAC firstly, and then the PCE solution was added into the mixture. A flow cone (height 60 mm, top diameter 36 mm and bottom diameter 60 mm) specified in Chinese standard GB/T 8077-2012 was used to test the fluidity of the cement paste. The flow cone was filled with a sample on a glass plate. After the cone was slowly removed from the sample, the maximum diameter of the spread sample and the maximum width perpendicular to that diameter were measured. The average of these two values was defined as the fluidity value. After the measurement of the initial fluidity, the paste was measured again after 45 min and the result was named as 45 min fluidity.

Rheological properties

The cement pastes with PCE-borax (PCE: 0.08 wt% of cement; borax: 0–1.80 wt% of cement) were prepared with the same process as what is shown in mini slump test. While the cement paste with borax only (0–1.80 wt% of cement) was prepared with a different water/cement weight ratio of 0.44:1 ($W/C = 0.44$; water: 132 g; cement 300 g). Rotor rheometer (R/S-SST, rotor: CC45, made by Brookfield, USA) was used to evaluate the rheology of the cement paste in the presence of borax or PCE-borax. The cup of the rheometer was filled with the cement paste and the measurement sequence was then conducted. In order to bring the cement paste to a reference structural state, it was firstly pre-sheared at a shear rate equal to 120 s^{-1} for 30 s. An evenly increasing shear rate was then directly applied from $0\text{--}100\text{ s}^{-1}$ within 120 s. After that, an evenly decreasing shear rate was applied from $100\text{--}0\text{ s}^{-1}$ within 60 s. The data was processed with Rheo 2000 V2.8 software. Each flow curve was fitted with the Bingham model in order to calculate the value of yield stress and plastic viscosity.

All operations were conducted at $25\text{ }^\circ\text{C}$.

2.2.2. Adsorption behavior

Firstly, PCE solutions (0.2 g/L, 0.6 g/L, 1.0 g/L, and 1.6 g/L) and borax solutions (0.4 g/L, 0.8 g/L, 1.2 g/L, and 1.6 g/L) were prepared in advance. The carbon content of PCE solution was tested with total organic carbon analyzer (TOC, Liquid TOC II, made by Elementar, Germany), and the boron content of borax solution was measured with inductive coupled plasma emission spectrometer (ICP, Optima 4300 DV, made by Perkin Elmer Ltd., USA). The results are shown in Fig. 2. Based on these results, the concentration of PCE or borax in solution can be calculated with TOC or ICP results.

Secondly, borax solutions (0–16.0 g/L) and PCE-borax solutions (PCE: 1.0 g/L; borax: 0–20.0 g/L) were prepared in advance, respectively. The solutions (20.0 g) were mixed with cement (1.0 g), and then stirred for 5 min. The suspension was separated by centrifugation at 3000 r/min for 4 min. The carbon content, the boron content, and concentration of Ca^{2+} in the upper supernatant were measured with TOC and ICP. Based on those results shown in Fig. 2, the concentration of PCE and borax in upper supernatant was gained. Additionally, the solid was dried in a vacuum drier at $25\text{ }^\circ\text{C}$ and prepared for XPS and SEM measurement.

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