



Effect of competitive adsorption between sodium gluconate and polycarboxylate superplasticizer on rheology of cement paste

Hongbo Tan ^{*}, Fubing Zou, Baoguo Ma, Yulin Guo, Xiangguo Li, Junpeng Mei

State Key Laboratory of Silicate Materials for Architectures, Wuhan University of Technology, Wuhan 430070, PR China

HIGHLIGHTS

- Effect of SG on dispersion of PCE is decided by the added dosage of SG.
- Combination between PCE and SG via Ca^{2+} has been confirmed to increase the dispersion.
- Competitive adsorption between PCE and SG would reduce the dispersion of PCE.

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ABSTRACT

Sodium gluconate (SG) is usually employed as a supplementary material in polycarboxylate superplasticizer (PCE) system to enhance the dispersion and dispersion retention. In real concrete, this enhancement cannot always be achieved as expected, and sometimes, even a negative effect can be found. This uncertainty is mainly because SG would perturb the dispersion of PCE, while the mechanism behind has not been completely understood. The objective of this study is to investigate the interaction between PCE and SG. The rheology was studied to assess the dispersion ability, and adsorption behavior was characterized with total organic carbon analyzer. The combination between PCE and SG was analyzed with conductivity, X-ray photoelectron spectrometer and dynamic light scattering. The dispersion model was then proposed to explain the main mechanism behind. The results show that two positive effects of SG on dispersion of PCE-SG system can be found: one is the formation of water film caused by adsorption of SG, and the other is more interesting, namely the combination between SG and PCE via the bridging Ca^{2+} to enhance the steric hindrance. The negative effect of SG can also be found, mainly because of the hindrance to adsorption of PCE resulting from competitive adsorption. Accordingly, the dispersion of PCE-SG depends on the predominant effect: when the dosage of SG less than 0.10%, the contribution to dispersion predominates; while the competitive adsorption effect predominates when the dosage more than 0.10%, with a decline in dispersion ability. Such results would provide guidance on the design of high efficient dispersant system in real engineering practice.

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1. Introduction

Currently, the high fluidity cement-based materials have been widely used in infrastructure construction [1–4]. Efficiently dispersing cement particles is essential for the practical application of this kind of materials. Generally, it is the incorporation of superplasticizer that ensures the high fluidity to be achieved. Polycarboxylate superplasticizer (PCE), with comb-type structure, has been accepted as the most popular superplasticizer in the world and has been developed as the main direction. The reason for this

tendency is mainly because of its high efficient dispersion and remarkable dispersion retention [5,6]. However, the commercially available PCE in real concrete is usually a multi-component system composed of polymers and retarders, rather than a single-component system. As a commonly used retarder, sodium gluconate (SG) is usually employed as a supplementary component to enhance the dispersion ability of PCE system. The generally accepted reason for this enhancement is due to the reduced consumption amount of free water and PCE by delaying the cement hydration.

In real concrete, however, the contribution of SG to the dispersion of PCE cannot always be achieved as expected, and sometimes, even a negative effect can be found. The reason for this

^{*} Corresponding author.

E-mail address: thbwhut@whut.edu.cn (H. Tan).

contribution can be reasonably revealed with the retarding effect and plasticizing effect of SG, while the opposite results cannot be explained convincingly. In fact, the main dispersion force of PCE is the steric hindrance offered by the long side chain (i.e. polyethylene oxide, PEO) of the PCE adsorbed on the surface of cement particles (S-PCE), and generally, the amount of S-PCE determines the intensity of this force, which means that more amount of S-PCE provides stronger dispersion [7,8]. Nevertheless, in superplasticizer-retarder system, the adsorption of superplasticizer would be perturbed by retarder competitively adsorbing onto the surface of cement particles, which has been confirmed in the studies about the naphthalene-based superplasticizer and amino-sulfonic acid-based superplasticizer in the presence of SG [9–11]. Competitive adsorption has also been proved in PCE system: both sulfate and phosphate can hinder the adsorption of PCE to affect the dispersion [12,13]. Additionally, it was also reported that citrate and tartrate could hinder the adsorption of PCE and influence the dispersion and dispersion retention [14]. As a consequence, the interference of SG to the dispersion and adsorption of PCE would be expected.

In this study, the interaction between PCE and SG was investigated to illustrate the mechanism behind the difference in dispersion of PCE with various dosages of SG. Specifically, the rheology was tested to assess the dispersion of PCE-SG system. Adsorption behavior of PCE and SG was analyzed with total organic carbon. The combination between PCE and SG in pore solution was analyzed with conductivity, X-ray photoelectron spectrometer and dynamic light scattering. The dispersion model was then proposed to illustrate the mechanism behind. It was anticipated that these results would provide guidance on the design of highly efficient dispersant system in real engineering practice.

2. Experimental

2.1. Materials

Cement

An ordinary Portland cement (42.5, Wuhan Yadong Cement Co., Ltd.) in accordance with the requirements of Chinese standard GB175-2007 was used. The chemical composition of cement was obtained with X-ray Fluorescence (XRF, Axios advanced, made by PANalytical B.V., Holland), and the results are shown in Table 1.

Additives

A single component PCE (commercially available) was used in this study. PCE neutralized with sodium hydroxide (NaOH) was marked as PCE (pH = 6.8), and that neutralized with calcium hydroxide (Ca(OH)₂, CH) was marked as PCE-Ca (pH = 6.8). The basic performance of PCE (obtained from the specifications supplied by the company) is shown in Table 2. An industrial-grade sodium gluconate (SG, white powder, ≥98.0%) and the reagent-grade calcium gluconate (CG, white powder, ≥99.9%) were used. The SG solution (2.0 wt%) was prepared for the experiment. The added dosage of PCE and SG was recorded as the solid amount.

The molecular structures of PCE (Information of the molecular structure was obtained from the company) and SG are shown in Fig. 1.

2.2. Test methods

2.2.1. Fluidity of cement paste

SG (0–0.30 wt% of cement) and PCE (0.13wt% of cement) were added into water in advance. The cement paste was then prepared with a water/cement weight ratio of 0.29:1 (water: 87 g; cement 300 g), in accordance with the requirements of Chinese standard GB 8076-2008. A flow cone (height 60 mm, top diameter 36 mm and bottom diameter 60 mm) specified in Chinese standard GB/T 8077-2012 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.

2.2.2. Rheological measurements

With the same process above, the cement pastes with SG (0.0–0.50 wt% of cement) were prepared with a water/cement weight ratio of 0.40:1, and the cement pastes with PCE-SG system (PCE: 0.10wt%, SG: 0.0–0.50 wt%) were prepared with a water/cement weight ratio of 0.29:1. The rheology of the paste was evaluated with Rotor rheometer (R/S-SST, rotor: CC45, made by Brookfield, USA). The cup of the rheometer was filled with the cement paste and the measurement sequence was 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 increasing shear rate was then directly applied from 0 to 100 s⁻¹ within 120 s. After that, a decreasing shear rate was applied from 100 to 0 s⁻¹ within 60 s. The data was processed with Rheo 2000 V2.8 software, and the yield stress and plastic viscosity were calculated according to the Bingham fluid model.

2.2.3. Competitive adsorption between SG and PCE

The carbon content of PCE or SG solution (0.20 g/L, 0.60 g/L, 1.0 g/L, 1.6 g/L, 2.0 g/L) was measured with total organic carbon analyzer (TOC, Liquid TOC II, made by Elementar, Germany). The results are shown in Fig. 2. Based on these results, the concentration of SG or PCE can be obtained with TOC results.

2.2.3.1. Adsorption amount of single system. Single system of PCE (0.2–2.4 g/L) or SG (0.2–2.4 g/L) was prepared, respectively. Cement (1.0 g) was mixed with the solutions (20 g), and then stirred for 5 min. After that, the mixture was separated by centrifugation at 3000 r/min. The carbon content in upper supernatant was measured with TOC. Based on the results shown in Fig. 2, the residual concentration of PCE or SG in upper supernatant could be obtained. The adsorption amount of PCE or SG (mg/g-cement) was calculated as follows:

$$\text{Adsorption amount} = V(C_0 - C)/m$$

where C₀ is the initial concentration (g/L) of PCE before adsorption; C is the residual concentration (g/L) after adsorption; V is volume of the solution (mL); m is the mass of the cement (g).

2.2.3.2. Competitive adsorption between SG and PCE. Theoretically, with TOC results, it is impossible to directly obtain the accurate amount of PCE or SG in PCE-SG solution, because both SG and PCE contain carbon, and the TOC result is the total carbon content including PCE and SG. However, the competitive adsorption can be speculated as follow:

The PCE-SG solutions (0.2–2.0 g/L PCE and 1.0 g/L SG; 1.0 g/L PCE and 0.2–2.0 g/L SG) were prepared, respectively. Cement (1.0 g) was then mixed with the solution (20.0 g) and processed with the same steps as above. The carbon content in upper supernatant was measured with TOC. These results were marked as the measured results. If no competitive adsorption between SG and PCE, the measured results would be almost equal to the summation of the carbon content in the upper supernatant of the single system of PCE and SG with the same dosage after adsorption (these results were marked as the expected results). If the measured results were greater than the expected results, PCE and SG would hinder each other from adsorbing onto the surface of the cement particles, which means that the competitive adsorption between these two additives would take place.

2.2.4. Conductivity measurement

PCE solution (10.0 g/L), SG solution (1.0 g/L), CH (0.5 g/L) and CG solution (3.0 g/L) were prepared in advance. Conductivity of the solution (100 g) or the reference (deionized water, 100 g) with the increasing dosage of CH or CG was carried out with an electrical conductivity meter (Seven Compact S230, made by Mettler Toledo, Switzerland). The different increasing tendency from the reference could demonstrate the reaction between Ca²⁺ and additives in solution.

2.2.5. Conformation of PCE and SG in solution

The solution of SG (0.30%), PCE (0.30%), and PCE-SG (0.30% PCE and 0.30% SG) in the presence and absence of CH (0.01%) were prepared. Size distribution of these solutions was characterized with dynamic light scattering (DLS, Zetasizer Nano, made by Malvern instrument Ltd., UK). The difference in size distribution could illustrate the conformation of organic in solution, as further evidence to prove the combination between organics and Ca²⁺.

Table 1
Chemical composition of cement.

		Loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	CaO	MgO	K ₂ O	Na ₂ O
Cement	wt%	3.82	24.08	4.72	2.46	2.31	58.24	1.95	1.02	0.27

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