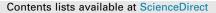
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Effect of hydroxypropyl-methyl cellulose ether on rheology of cement paste plasticized by polycarboxylate superplasticizer



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HIGHLIGHTS

- Agglomeration and even precipitation of HPMC in pore solution would take place.
- HPMC with greater molecular weight is more easily agglomerated in pore solution.
- Negative effect of HPMC on dispersion of PCE was confirmed.
- HPMC with greater molecular weight results in stronger negative effect.

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ABSTRACT

Generally, bleeding and segregation can be solved with addition of hydroxypropyl-methyl cellulose ether (HPMC) in real concrete. However, the negative effect of this on dispersion of polycarboxylate superplasticizer (PCE) can always be found. In order to deeply understand this negative effect, the rheology of cement paste in the presence of PCE and HPMC was investigated, and the mechanism behind the interaction between PCE and HPMC was revealed with total organic carbon analyzer, X-ray photoelectron spectrometer and dynamic light scattering. The results show that addition of HPMC can obviously increase the plastic viscosity and yield stress of cement paste plasticized by PCE, showing negative effect on fluidity. One reason for this negative effect is that HPMC can be combined with Ca²⁺ in pore solution, resulting in agglomeration and even precipitation, which can consume and invalid PCE; another reason is that HPMC can perturb the adsorption of PCE to exert dispersion by competitive adsorption. By contrast, much stronger negative effect can be observed with greater molecular weight of HPMC. It is inferred that proper molecular weight of HPMC can balance the negative effect of HPMC on dispersion of PCE and the viscosity-enhancing effect of HPMC to improve bleeding and segregation. Such results were expected to provide guidance on the use of HPMC to optimize workability of cement-based materials plasticized by PCE in real engineering practice.

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1. Introduction

High fluidity cement-based materials have been widely employed in civil engineering, such as self-compacting concrete, self-leveling mortar, and grouting materials [1–4]. The widely concerned issue for this kind of materials is that the high fluidity and high stability should be achieved at the same time. This means that high flowability without any bleeding and segregation should be obtained. Generally, high fluidity can be gained with addition of

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https://doi.org/10.1016/j.conbuildmat.2017.11.010 0950-0618/© 2017 Elsevier Ltd. All rights reserved. superplasticizer, while non-bleeding and non-segregation can be made with the addition of viscosity-enhancing admixture. This experience has been widely accepted in real engineering practice.

Cellulose ether has been widely used in self-leveling indoor floor mortar and self-compacting concrete, with intention to improve bleeding and segregation obviously. The reason reported in the literatures is that cellulose ether can increase the viscosity of liquid phase [5–8]. To be more precise, hydroxyl groups (–OH) and ether groups (R–O–R) can be combined with water molecules to form the hydroxyl bond. In this case, free water may be wrapped inside the chain of cellulose ether or be fixed outside the structure [9,10]. Furthermore, molecular weight is one of the factors affecting its viscosity-enhancing effect, as reported, and generally, higher molecular weight would result in stronger effect, but no specific explanation was given in the literature [7]. In addition, Ca^{2+} in pore solution should be considered. pH value of the cement paste can reach more than 12 in the first few minutes, as reported, and the release of the Ca^{2+} into solution almost takes place at the same time with the increasing pH value [11–13]. These Ca^{2+} may be combined with ether groups known as complexation [14–18], resulting in the change of conformation size of cellulose ether. This may noticeably affect the viscosity-enhancing effect of cellulose ether. However, the complexation, which is often ignored, has not been clarified until now.

Polycarboxylate superplasticizer (PCE) has been accepted as the most efficient water-reducing agent in the world, and with addition of this kind of superplasticizer, the cement-based material can be plasticized effectively, which means that the high fluidity can be easily achieved [19–21]. However, based on practical experience, high fluidity may result in poor stability of concrete, and this means that bleeding and segregation can easily take place. This problem can be observed widely, showing considerably negative effect on durability of concrete. Many attempts have been made to solve this issue, including modification of molecular structure of PCE and incorporation of chemicals, and the latter (i.e. incorporation of chemicals) seems more acceptable in real engineering practice. Hydroxypropyl-methyl cellulose ether (HPMC), a commonly used viscosity-enhancing admixture, has been always used to increase viscosity to solve this problem.

However, in cement paste plasticized by PCE, the addition of HPMC can sometimes improve the viscosity of cement paste, whilst sometimes the opposite is true. Another problem can also be found that a little more amount of HPMC increases viscosity so greatly that the fluidity is reduced to be unacceptable, obviously increasing the added dosage of PCE to reach the fluidity demand. One possible reason for this is due to the interference of HPMC to dispersion of PCE. As discussed in the literatures, the dispersing efficiency of PCE depends on both the amount of PCE adsorbed on the surface of cement particles and the activity of the long side chain (i.e. polyethylene oxide, PEO, which offers the steric hindrance as the main dispersion force) [22–24]. It can be inferred that the addition of HPMC may noticeably interfere the adsorption of PCE or the function of PEO. Furthermore, ether bonds in HPMC can also result in adsorption. In this case, competitive adsorption between HPMC and PCE would take place [25-28], and mutual influence on adsorption should be observed.

In this study, in order to deeply understand the mutual influence between PCE and HPMC, the rheology of cement paste with these two kinds of chemicals was investigated. The yield stress and plastic viscosity were calculated from Bingham model, and the zeta potential and adsorption were studied to analyze the surface performance of cement particles. Interaction between PCE and HPMC was analyzed with conductivity, dynamic light scattering, and X-ray photoelectron spectrometer. Finally, a dispersion model was proposed to illustrate the mechanism. Such results would provide useful experience for the application of PCE-HPMC system, and also give some suggestion on optimizing the workability of fresh concrete in real engineering practice.

2. Materials and test methods

2.1. Materials

2.1.1. Cement

An ordinary Portland cement (42.5, Wuhan Yadong Cement Co., Ltd.) in accordance with the requirements of GB175-2007 Chinese standard was used. The content of CaSO₄ was 4.2%, and the specific surface area was 350 m²/kg.

The chemical composition of cement was obtained with X-ray Fluorescence (Axios advanced, made by PANalytical B.V., Holland), and the results are shown in Table 1.

2.1.2. Polycarboxylate superplasticizer (PCE)

A commercially available PCE was used in this study. The basic performance and molecular structure obtained from the company are shown in Table 2 and Fig. 1.

2.1.3. Hydroxypropyl-methyl cellulose ether (HPMC)

Two kinds of commercially available HPMC, with the difference in molecular weight, were used in this study. These two are non-ionic cellulose ethers. The basic performance and molecular structure obtained from the company are shown in Table 3 and Fig. 2.

2.2. Test methods

2.2.1. Rheological measurements

The cement pastes with addition of HPMC (0.0–0.20 wt% of cement) were prepared with a water/cement ratio of 0.50 by weight, and the cement pastes with PCE-HPMC system (PCE: 0.08 wt%; HPMC: 0.0–0.20 wt%) were prepared with a water/cement ratio of 0.29 by weight. These chemicals were mixed with water in advance. 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 Rheology 2000 V2.8 software, and the yield stress and plastic viscosity were calculated according to the Bingham fluid model.

2.2.2. Adsorption amount

The carbon content of PCE and HPMC solution (0.40 g/L, 0.80 g/L, 1.2 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. 3. Based on these results, the concentration of HPMC or PCE in solution can be obtained with TOC value.

Cement (1.0 g) was mixed with PCE and HPMC solution (20 mL; PCE 0–2.0 g/L; HPMC 0–2.0 g/L), respectively. After that, these suspensions were stirred for 5 min, respectively, and then separated by centrifugation at 4000 r/min for 5 min. TOC was used to test the carbon content of the upper supernatant. Based on the results shown in Fig. 3, the concentration of PCE and HPMC in upper supernatant (i.e. residual concentration) was obtained. Adsorption amount of PCE and HPMC (mg/g-cement) was calculated as follows:

Adsorption amount = $V (C_0 - C)/m$

where, C_0 is the initial concentration (g/L) of PCE and HPMC 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).

Measurements were generally repeated three times to avoid deviation, and the average was the result. All operations were made at 25 $^\circ$ C.

2.2.3. Zeta potential

Cement (1.0 g) was mixed with the solution of HPMC (20.0 g, 0-2.0 g/L) and HPMC-PCE (20.0 g; 0-2.0 g/L HPMC; 1.0 g/LPCE), respectively, and stirred for 5 min. The suspension (0.5 g) was added into deionized water (45.0 g) and mixed, and the zeta potential was then tested with the instrument (ZetaSizer Nano-ZS, made by Malvern instrument Ltd., UK). The testing process should be finished within 10 min.

2.2.4. Conductivity measurement

PCE solution (4.0 g/L), HPMC solution (1.0 g/L), and calcium hydroxide (CH, 0.5 g/L) were prepared in advance. With the addition of CH solution, conductivity of the solutions (100 g) or the reference (i.e. deionized water, 100 g) was tested with an electrical conductivity meter (Seven Compact S230, made by Mettler Toledo, Switzerland), respectively. The different change tendency could demonstrate the reaction between Ca^{2+} and chemicals in solution.

2.2.5. Combination of Ca^{2+} with chemicals

Solution of CH (10.0 g/L), PCE-CH (10.0 g/L PCE, 1.0 g/L CH), and HPMC-CH (10.0 g/L PCE, 1.0 g/L CH) was prepared, respectively. These solutions were dried in a vacuum drier at about 105 °C. And then, the solid was characterized with X-ray photoelectron spectrometer (XPS, Escalab 250Xi, made by Thermo Fisher Scientific, USA). The data was processed with XPS peak 4.0, and extra peaks, except the peak belonging to CH, were added to fit the observed curve. The difference in binding energy of Ca2p could indicate the formation of new types of calcium bond. Additionally, the ratio of these calcium bonds can be calculated from the ratio of each divided peak area.

2.2.6. Conformation of PCE and HPMC in solution

Solutions of HPMC (2.0 g/L), PCE (2.0 g/L), and PCE-HPMC (2.0 g/L PCE, 2.0 g/LHPMC) in the presence and absence of CH (1.0 g/L) were prepared. Size distribution of these solutions was characterized with dynamic light scattering (DLS, Zetasizer Download English Version:

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