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Effects of polyethlene oxide chains on the performance of polycarboxylate-type water-reducers

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Abstract

A series of polycarboxylic acid-based copolymers with block and graft groups of polyethlene oxide (PEO) chains was synthesized; effects of the different PEO chains on the fluidity, Zeta potential and adsorption in cement paste and performances of the copolymer in concrete were discussed. It was proved that properties of the copolymer were affected by the length and density of PEO graft and block chains, and that copolymers with some block PEO chains at a certain length and moral percent had good performances in the water-reducing capability and fluid-retaining ability. Experimental results indicated that this kind of copolymers could be used as a high-range water-reducer because of the effects of electrostatic repulsive force and steric hindrance; one of its applications was to produce high-flowing concrete by incorporating with mineral admixtures, such as fly ash, etc.

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Keywords: High-range water-reducers; Polycarboxylate type; Concrete; Mineral admixture

1. Introduction

Many new types of polycarboxylic water-reducers (called PCs) and methods of their application have been developed and studied [1-4]; they have drawn more and more attention in the construction industry recently because of their novel applications in concrete, such as high-volume fly ash concrete, superplasticized fiber-reinforced concrete and shotcrete, high-strength and high-performance concrete, etc. PCs can interact with cement particles physically and chemically, although some PCs have already been used to solve a variety of concrete problems because of their higher water-reduction ratio and their less slump loss; effects of PEO chains in the molecular structure on the performance are still not very clear [5-8]. Studies on this aspect may be useful in designing the molecular structure of this new and important class.

Observations have indicated that the absolute Zeta potential of ordinary Portland cement particles with NSF or MSF (naiphthylene- or melamin-type superplasticizers) must be more than 20 mV; in contrast, the PCs yield much smaller values for the Zeta potential, less than 10 mV [3,7,8]. Uchikawa et al. [2,7] considered that electrostatic forces play a major role in the dispersion mechanism for PNS water-reducer while steric hindrance is critical for a copolymer of acrylic acid with acrylic ester water-reducer. Yoshioka et al. [8,9] developed a model to describe the adsorption behavior of these water-reducers, calculated the total interparticle potential energy account for long-range Van der waals, electrostatic, and steric interactions, and concluded that the repulsive potential that resulted from electrostatic interactions was negligible; the steric hindrance plays a dominant role compared to electric repulsion in the deflocculating of cement pastes than previously believed.

PCs are copolymers of which chemical structures have the potential to be modified [5]. In our study, a polycarboxylic water-reducer (called MPC hereafter) containing block and graft groups of polyethlene oxide (PEO) was manufactured synthetically. The density of PEO block and graft chains in the molecular structure had been adjusted and a series of polycarboxylic acid-based copolymers, MPCs, were obtained. Effects of PEO chains on the results of fluidity, Zeta potential, and adsorption in cement pastes

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were discussed; one of the copolymers was then used to prepare high-flowing concrete.

2. Experimental

2.1. Synthesis of MPCs

The copolymers with the given PEO chains in length were synthesized; the synthetic process was carried out in four steps. The first step was esterifying the polyethylene glycol (PEG) of various molecule weights. In this study, chemicals of PEG1000, PEG2000, and PEG4000 were used, which contain a molecule weight of 1000, 2000, and 4000, at a length of n = 23, 46, and 92 for PEO chains (the adding moles of ethylene oxide), respectively. The PEG1000 and PEG2000, which were esterified with (meth)acrylic acid into PEG mono (meth)acrylic acid esters, took the macromonomers as PEO1 and PEO2; the PEG4000 was esterified with maleic anhydride and changed into a PEG diester, the macromonomers as the PEO3. The harvest ratio of esterification might be controlled by changing the reaction conditions, such as reaction temperature, reaction time, quantity of acid and catalyzer, etc. The second step was to blend various compounds with the vinyl radical at a certain moral ratio, which included monomers containing carboxylic acid groups, sulfonic acid groups, and various PEO block and graft groups. The third step was to synthesize in aqueous system; the vinyl solution and initiator radical solution were dropped, respectively, for about 3 and 4 h, and the reaction temperature kept at a range from 70 to 75 °C. In the final step, the polymer solution was neutralized by alkali solution of 30% sodium hydroxide and ethylene diamine, so the



Fig. 1. The possible chemical structure of MPCs.

| Table 1 | | | | | | |
|-------------|-------------|------|-----------|--------|-----|---------------|
| Molar ratio | of monomers | with | different | groups | and | concentration |

| Series number | Monome | Monomer molar ratio (mol %) | | | | | | | |
|------------------|-------------------------------|-----------------------------|------|------|------|-------------|--|--|--|
| | -SO ₃ ⁻ | -COO ⁻ | PEO1 | PEO2 | PEO3 | content (%) | | | |
| MPC-1 | 10 | 70 | 20 | 0 | 0 | 19.7 | | | |
| MPC-2 | 10 | 70 | 0 | 20 | 0 | 20.1 | | | |
| MPC-3 | 10 | 70 | 18 | 0 | 2 | 23.2 | | | |
| MPC-4 | 10 | 70 | 0 | 18 | 2 | 23.9 | | | |
| MPC-5 | 10 | 70 | 10 | 10 | 0 | 20.3 | | | |
| MPC-6 | 10 | 70 | 9 | 9 | 2 | 23.5 | | | |
| MPC-7 | 10 | 70 | 8.25 | 8.25 | 2.5 | 25.6 | | | |
| MPC-8 | 10 | 70 | 8 | 8 | 4 | 27.0 | | | |
| MPC-9 | 10 | 70 | 7 | 7 | 6 | 29.1 | | | |

alkali level could be lower and reduce the risk of alkalisilica reactivity.

By adjusting the ratio of these PEOs as the copolymer was synthesized, a series of MPCs with different characteristics could be obtained. As the macromonomers of PEO3 were almost PEG diester of maleic acid, the PEO3 chains might be in the backbone of molecular structure; thus, macromonomers of PEO1, PEO2, and PEO3 were therefore grafted or blocked in the molecular structure. Because it is very difficult to estimate the actual structures, one of the possible chemical structures was shown in Fig. 1. In our study, MPCs were synthesized at different monomer moral ratios in Table 1. The total PEO chains was kept at about a monomer ratio of 15.5%, the moral percent of PEO3 chains in PEOs was from 0% to 30%; concentrations were also shown. More details about the synthetic process of the water-reducer could be seen in Ref. [10].

2.2. Property tests on cement paste

2.2.1. Flow test of cement paste (minislump)

The fresh cement paste with MPC was prepared at a water/binder ratio of 0.29 at 25 °C. The cement materials included an ordinary Portland cement and a fly ash; their chemical compositions were shown in Table 2. The admixture was added into the mixing water at a given content of 0.3% (weight percent of solid content to cement). The sample of cement paste was carefully sealed in a container during test intervals of 5, 30, 60, and 90 min after being mixed with water. The flow value was measured by using a minislump cone (60 mm high, top diameter 30 mm, and bottom diameter 60 mm), and the mixing and measurement procedure was carried out according to the Chinese standard GB/T 8077-2000: methods for testing uniformity of concrete admixture.

2.2.2. Zeta potential test

The Zeta potential of cement particle was tested within 3 min after the cement paste had been made. Pastes were prepared just as the test of minislump. The slurries were diluted with distilled water at a paste/solution ratio of 0.02, dispersed for about 30 s by an ultrasonic cell disruptor, took

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