



Evaluation on the consumption and performance of polycarboxylates in cement-based materials



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HIGHLIGHTS

- The absorption and coprecipitation of 6 PCE polymers are investigated.
- Their absorbed consumption and consequent flow of superplasticized cement pastes show time dependence.
- A relation between the PCE consumption and the yield stress of the superplasticized cement pastes can be finally obtained.

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ABSTRACT

The addition of polycarboxylate based surfactants cause particles dispersion in cementitious materials and enhances their fluidity. The polymeric structures of the polycarboxylates have been formulated to meet various performance requirements such as water-reducing and consistency-maintaining. A trend on the relation between the polymeric structures and the resultant performance certainly exists, but its modeling is hardly obtained due to uncertainty on the polymeric structures as well as cement compositions. The degree of adsorption of the a polycarboxylate on cement particles is then taken to describe its performance. The rheological properties of plasticized cement pastes were evaluated and discussed to quantitatively analyze the effect of the adsorption of various polycarboxylates.

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1. Introduction

Superplasticizers, also known as high-range water-reducing admixtures, enhance the fluidity of concrete. Their water-reducing effect on the production of concrete mixtures for a given cement content allows us to improve compressive strength and the elastic modulus of concrete [1,2]. The admixture for concrete is a polymeric surfactant, and it adsorbs onto and disperses hydrating cement particles [3–5]. Various polymers for these admixtures have been developed [6,7]. The first generation was introduced as lignosulfonates. The second generation was composed of sulphonated naphthalene formaldehyde and sulphonated melamine formaldehyde, which more than doubled the improvement of the water-reducing effect. Both surfactant-based admixtures significantly improve the workability of concrete and are still widely

used [8,9]. In the last two decades, an increasing interest and application can be observed for the third-generation polymer formulated by polycarboxylate ester or ether (PCE) [10,11].

PCE is a copolymer that can be synthesized through esterification followed by polymerization [11]. The molecular structures of PCE are generally composed of a negatively charged backbone with (poly)carboxylate and grafted side chains mainly composed of polyethylene oxide (PEO) [12]. One merit of PCE is its capability to manage diverse repetitive structural units with higher molecular weight. When PCEs are added into concrete mixtures, the charged backbone adsorbs onto the surface of cement particles and the graft chains on the opposite ends extend into the water molecules in the mixtures. As a result, steric repulsion is realized, thereby hindering the agglomeration of cement particles and consequently improve the fluidity of concrete mixtures to a great extent [13,14].

Various studies have reported the relationship between the molecular structure of a PCE and its performance on the fluidity enhancement of cementitious materials [15–17]. For example,

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a lower PEO graft chain density may increase the workability of cementitious materials and the length of the PEO graft chain affects the dispersion force [10,13,18,19] and its retention time [15,16,20]. Moreover, Yoshioka et al. [17] developed a structural functioning model, based on interparticle potential energy, to describe the PCE adsorption and cement dispersion, which is dependent on the PEO graft chain length and its density. Nevertheless, the model to fully describe the structural functioning is still challengeable: Contradictory results are often found in the literature even though a longer graft chain is preferable for fluidity enhancement [15]. The model's incompleteness is reportedly explained by (1) the characteristics of PCE, which depends on the raw materials and synthesis condition, and (2) the detailed polymer structure, which is seldom characterized in a quantitative manner.

Thus, this study proposes that the rheology property of cement-based materials is explained by the interaction of PCE and cement particles rather than its polymer structure. A detailed measurement of PCE adsorption will be the first approach to understanding this adsorption even though the term "consumption" more aptly describes the PCE coprecipitation to form an organo-mineral phase [21]. Its evaluation and pattern analysis follow. Finally, a model to explain the yield stress of cement paste is presented.

2. Experimental program

2.1. PCE characteristics

A total of six commercially available PCEs, labeled by LA, LB, LC, LD, LE and LF, were tested in this study. All of them were a methoxy polyethylene glycol (MPEG) PCE synthesized with the same raw materials: polyethylene glycol (PEO) and carboxylate acid monomer. Controlling the degree of copolymerization and the PEO chain length allowed us to have six PCEs showing various performance. Their chemical structures could be analyzed by the following tests.

Fourier transform infrared (FT-IR) investigates the IR absorption spectra of each sample, where a spectral peak indicates a specific bonding of atoms. The applied spectral range was 650 to 4000 cm^{-1} at a resolution of 2 cm^{-1} . Fig. 1 shows the measured spectra of the PCEs. The peak at 1104 cm^{-1} corresponds to an ether linkage ($\text{CH}_2\text{—O—CH}_2$) in PEO, and their stretching vibrations of the semi-crystalline phase provide the band peaks at 1059, 1104 and 1146 cm^{-1} [22–24]. The vibration band of hydrophobic aliphatic groups (CH or CH_2) is stretching at 2885 cm^{-1} [23,24]. The absorbance at 1731 cm^{-1} indicates the band of ester groups (O=C—O—CH_2)

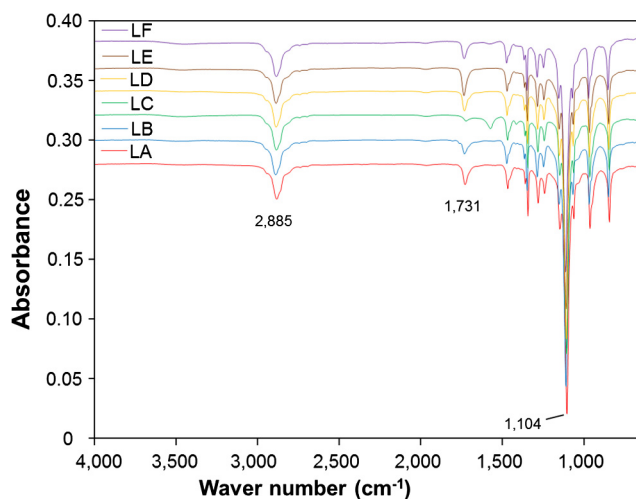


Fig. 1. FT-IR spectra of PCEs.

Table 1
Molecular weight and PDI of PCEs.

PCE	Content of PCE	Molecular weight (g/mol)		PDI
		M_w	M_n	
LA	65%	16,114	6,221	2.59
LB	76%	34,239	12,862	2.66
LC	64%	23,164	8,266	2.80
LD	82%	31,695	9,914	3.20
LE	80%	38,870	14,828	2.62
LF	52%	18,671	6670	2.80

[23,24]. The similarity of the IR absorption spectra confirms that the raw materials and principal polymerization of all PCE samples are the same.

Gel permeation chromatography (GPC) evaluates the average molecular weight of a polymer [23]. The PCEs were diluted with 10% concentration in an eluent (tetrahydrofuran, THF) and then analyzed at a flow rate of 1 mL/min. Temperature and relative humidity were controlled as 23 °C and 50%, respectively. Table 1 lists the number-average molecular weight (M_n), the weight-average molecular weight (M_w), and the polydispersity index ($\text{PDI} = M_w/M_n$) of the PCEs. A higher PDI indicates various polymers distributed in a sample while a single polymer provides $\text{PDI} = 1$ [25]. Fig. 2 shows the GPC chromatogram for the samples, where the peak of THF is beyond the upper bound of elution time (20 min). The leftmost peak designated as 1, showing a short elution time, corresponds with the synthesized PCEs. The other peaks distributed on the right side seemed to be caught by unreacted PEO macromonomers and acrylic acid [23,25]. LA and LF shows a relatively large amount of unreacted groups. Their 1st peaks were small, and consequently the calculated contents of the final product (PCE) were 65% for LA and 52% for LF%. The PCE contents were calculated by the integration of polymer peak divided by all peaks area and the results are listed in Table 1. Both are applied in field even though a large portion of unreacted groups remains. Non-adsorbed polymer together with a general PCE can contribute to its performance to enhance the fluidity [26]. Therefore, their performance, as they were, needed to be evaluated.

2.2. Sample preparation

Cement paste samples were produced by incorporating with the PCE samples. Type I Portland cement and deionized water were used. The specific density and Blaine of Portland cement were

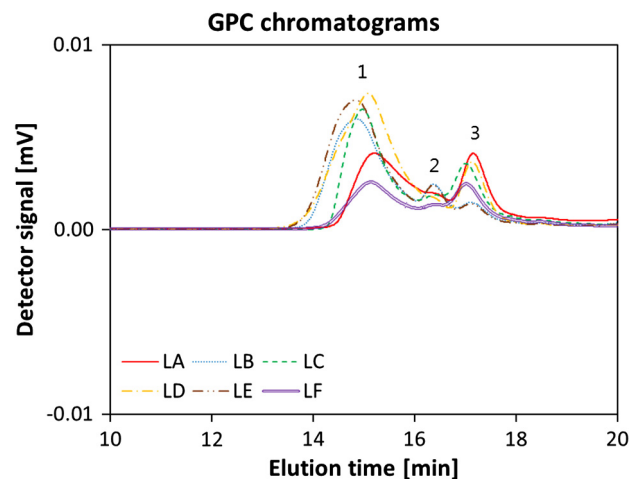


Fig. 2. GPC chromatograms for PCEs.

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