



Study on the Adsorption Effects of Polycarboxylic Acids with Different Molecular Structures on the Surface of Desert Sand

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Abstract. Polycarboxylate superplasticizers (PCE) with different molecular structures were prepared through radical polymerization using monomers with varying molecular weights, and their molecular structures were characterized by gel permeation chromatography. The adsorption properties of PCE on the surface of desert sand were studied using a total organic carbon analyzer, and mortar fluidity tests were conducted. The results showed that PCE with a molecular weight in the range of 30,000 to 40,000 Da exhibited the best fluidity effect in desert sand mortar.

Keywords: Desert sand, Polycarboxylate, Adsorption.

1 Introduction

With the rapid development of desert sand concrete, studies have found that desert sands from different regions have varying impacts on the performance of concrete, and there are also very few suitable water-reducing agents that can be applied^[1-3].

Wu et al^[4]. conducted mortar performance tests using Maowusu desert sand and found that different polycarboxylate water-reducing agents had a significant impact on the fluidity of desert sand mortar. Zhang et al^[5]. found that PCE (Polycarboxylate Ether) effectively improved the flowability of desert sand concrete. Hui^[6] discovered that when the dosage of the initiator was 1.0% and the dosage of the chain transfer agent was 0.4%, the PCEs demonstrated good adaptability when used in desert sand concrete. However, none of these studies analyzed the internal mechanism of how the PCE interacts with the surface of desert sand.

This paper focuses on the significant issue of excessive dosage of PCEs caused by the use of desert sand, and conducts research on the adsorption mechanism. By synthesizing and preparing a series of PCEs with different molecular weights and side chain lengths, and measuring the adsorption capacity on the surface of desert sand, the internal mechanism of desert sand adsorbing PCEs is studied. This provides a basis for the development of water-reducing agents suitable for desert sand concrete.

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2 Experiments

2.1 Raw Materials

The study used desert sand from Yulin, Shaanxi Province, China, and P.I 42.5 reference cement, China United Cement Corporation.

Raw Materials for Synthesis: Ethylene Oxide Polyglycol Ether (EPEG): Industrial grade, purity $\geq 99\%$, manufactured by Jahwa Chemical Co., Ltd.; Acrylic Acid (AA): Industrial grade, purity $\geq 99\%$, made by Jiangsu Runhan Chemical Co., Ltd.; L-Ascorbic Acid (Vc), analytical reagent, purity 99.7%, Sinopharm Chemical Reagent Co., Ltd.; Hydrogen Peroxide (H_2O_2): Analytical reagent, mass concentration 30%, Shanghai Macklin Biochemical Co., Ltd.; Mercaptoethanol (T4), industrial grade, purity $\geq 99\%$, Daicel Corporation, Japan; Sodium Hydroxide ($NaOH_2$): Analytical reagent, purity $\geq 98\%$, Shanghai Macklin Biochemical Co., Ltd.; Tap Water (W).

2.2 Synthesis

Add 180g of EPEG, 0.45g of Vc, and 195g of water into a four-necked flask. Prepare 0.25g of H_2O_2 and T4 into Solution 1, and 17.7g of AA into Solution 2. Place the four-necked flask in iced water and wait until the mixture inside reaches the reaction temperature ($10\sim 15^\circ C$). Simultaneously, start to add Solution 1 and Solution 2 dropwise over a period of 3 hours. Maintain the temperature for an additional hour, and then allow it to cool to room temperature. Neutralize the mixture with a 32% $NaOH_2$ solution until the pH reaches 6-7, yielding a polycarboxylic acid water reducer mother liquor with 53% solids concentration.

2.3 Test Method

Gel Permeation Chromatography (GPC). The molecular weight, molecular weight distribution, intrinsic viscosity, and hydrodynamic radius of the samples were tested using a Waters 1515/2414 Gel Permeation Chromatography (GPC) system. The mobile phase was a 0.1 mol/L aqueous solution of $NaNO_3$, with a flow rate of 0.8 ml/min.

Adsorption. To prepare the water reducer solution, randomly add 10g of desert sand to 50mL of the water reducer solution. After thoroughly mixing, pour an appropriate amount of the liquid into a centrifuge tube. Use a centrifuge to separate the filtrate. Collect the supernatant and test it using an Elementar TOC-VCPH Total Organic Carbon Analyzer. Calculate the adsorption amount of the PCE on the cement particles using Equation (1).

$$\Gamma = \frac{(c_0 \times V_1 - c_1 \times V_1)}{m \times 1000} \quad (1)$$

Mortar Fluidity. The mortar fluidity was tested according to GB/T 8077-2024 "Test Methods for Uniformity of Concrete Admixtures."

3 Results and Discussion

3.1 Structural Characterization

GPC Testing. The polycarboxylate superplasticizers used in the study, which have different molecular weights, underwent GPC testing, and their results are tabulated in Table 1.

Table 1. Measurements from GPC tests on PCE with different molecular weights

Sample	T4/g	Structural Characterization			
		M _n	M _w	Conversion Rate/%	PDI
PCE-1	1.10	11599	26976	96.31	2.326
PCE-2	0.70	11343	38237	92.20	3.371
PCE-3	0.50	29090	56518	90.74	1.943
PCE-4	0.30	43410	98484	93.09	2.269

Table 1 reveals that, throughout the synthesis, the weight-average molecular weight (M_w) undergoes a gradual increase as the concentration of the chain transfer agent, mercaptoethanol (T4), diminishes.

Water-reducing agents with various side chain lengths underwent GPC testing, and the resultant data are provided in Table 2.

Table 2. GPC test results of PCE with different side chain lengths

Sample	XD22	Structural Characterization			
		M _n	M _w	Conversion Rate/%	PDI
PCE-5	1200	8149	26073	99.2	3.200
PCE-6	2400	6015	23912	98.3	3.975
PCE-7	3000	13077	31208	99.4	2.386
PCE-8	5000	32951	56396	68.87	1.712
PCE-9	6000	30378	47051	76.6	1.549

As can be seen from Table 4, the synthesized PCE agents show a gradual increase in weight-average M_w as the selected XD22 molecular weight increases, ranging from 26,000 to 57,000. This is in line with the experimental design requirements.

3.2 Adsorption Characteristics

PCE Exhibiting Various Molecular Weight Configurations. In order to investigate how the molecular weight structures of PCE affect its adsorption capabilities, we

conducted tests on the initial adsorption capacity at different PCE concentrations, and the findings are illustrated in Figure 1.

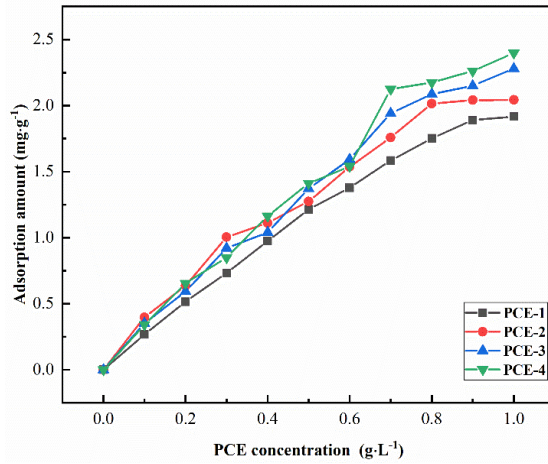


Fig. 1. Effect of Concentration on PCE Adsorption in Desert Sand Mortar Systems

Figure 1 demonstrates that the adsorption capacity of PCE rises as the concentration increases, with the rate of this increase progressively decelerating as the concentration continues to climb. For PCE-1, the adsorption capacity on the surface of desert sand reaches saturation when the concentration exceeds 0.9 mg·L⁻¹; whereas for PCE-4, the adsorption capacity attains saturation when the concentration reaches 1.0 milligrams per liter.

The flowability changes of desert mortar and high-efficiency water reducing agents PCE-1 to PCE-4 were tested by adding an equal amount of PCE. The result is shown in Figure 2.

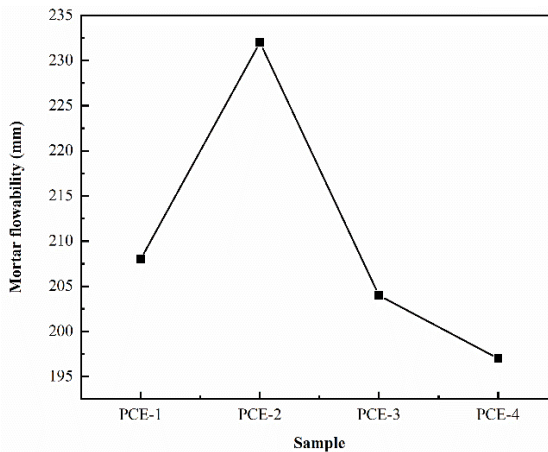


Fig. 2. Effect of PCE's Molecular Weight Structures on Mortar's Fluid Properties

Observing Figure 2, it is evident that when an equal quantity of PCE is applied, as the adsorption amount of PCE with varying molecular weights onto desert sand particle surfaces rises, the mortar's fluidity exhibits a trend of initial increase followed by decrease. The order of fluidity, from highest to lowest, is PCE-2 > PCE-1 > PCE-3 > PCE-4. Notably, PCE-2 demonstrates the highest initial fluidity, which can be attributed to the enhanced steric hindrance force resulting from the adsorption of polycarboxylic acid molecules onto the particle surface.

PCE Featuring Varying Side Chain Lengths. The initial adsorption capacity for these PCE samples, across various concentrations, was examined, and Figure 3 displays the findings.

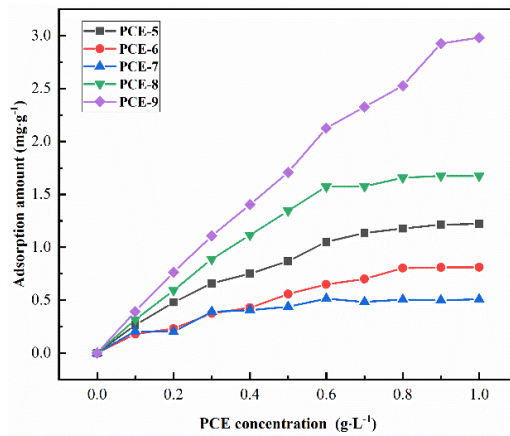


Fig. 3. Assessment of PCE Adsorption Capacity across Different Concentrations in Desert Sand Mortar

Observing Figure 3, when the PCE concentration is set at $1.0 \text{ mg}\cdot\text{L}^{-1}$, the adsorption capacity follows the order of PCE-9 > PCE-8 > PCE-5 > PCE-6 > PCE-7.

The flowability changes of desert mortar and water reducing agents PCE-5 to PCE-9 were tested separately. The data from the experiment is illustrated in Figure 4.

Figure 4 demonstrates that, with equal quantities of PCE, mortar fluidity varies, initially increasing and then decreasing, with changes in the adsorption of PCE with differing side chain lengths on desert sand surfaces. The mortar fluidity sequence, in descending order, is PCE-7 > PCE-6 > PCE-5 > PCE-8 > PCE-9. PCE-5, PCE-6, and PCE-7, all featuring longer side chains, exhibit mortar fluidity values above 200 mm, hinting at a higher adsorption tendency on desert sand surfaces. Through electrostatic adsorption, they can better adsorb on the surface of cement particles by complexing with Ca^{2+} on the cement surface, resulting in a larger steric hindrance and thus improving the dispersion ability^[7].

It is recommended that in practical applications of desert sand concrete, polycarboxylate superplasticizer concentrates with smaller molecular weights and shorter side chain lengths should be selected to adjust the properties of the desert sand concrete.

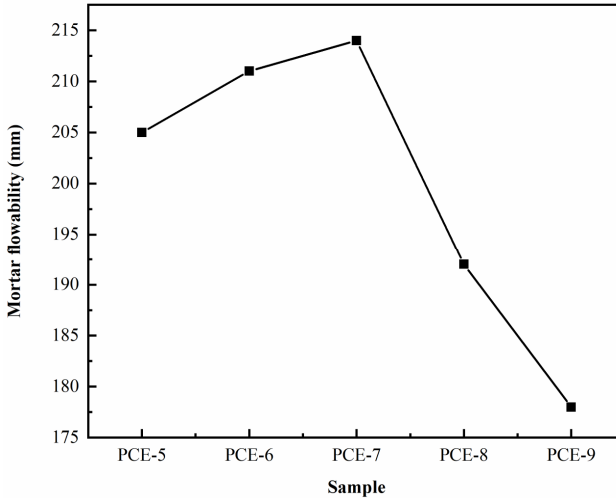


Fig. 4. Fluidity of Mortar Containing PCE with Varying Side Chain Lengths

4 Conclusion

Based on the investigation of the microstructure, adsorption characteristics, and dispersion properties of PCEs with varying molecular weight structures and side chain lengths, the following conclusions are derived:

As the concentration of PCE increases, the adsorption capacity enhances, but the rate of this increase gradually diminishes as the concentration further rises.

Within the range of 30,000 to 40,000 molecular weight for PCE, it achieves the best fluidity effect in desert sand mortar.

In practical applications of desert sand concrete, selecting a polycarboxylate superplasticizer base with a smaller molecular weight and shorter side chain length tends to yield better results.

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