

# Preparation of nanoscale carbon black dispersion using hyper-branched poly(styrene-*alt*-maleic anhydride)

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## ABSTRACT

Hyper-branched poly(styrene-*alt*-maleic anhydride) (BPSMA) was used as a dispersant to disperse carbon black (CB) in aqueous media. The dispersing ability of BPSMA for CB was estimated by measuring the particle size, zeta potential, stability to temperature and centrifugal stability of BPSMA-dispersed CB dispersions. The experimental results showed that BPSMA prepared with a molar ratio of 4-vinylphenyl methanethiol (VPMT), St and MA at 6:47:47 and a mass ratio of BPO and (VPMT + St + MA) at 1:50 exhibited optimal dispersing ability when the CB dispersion was at pH 8, and a mass ratio of BPSMA and CB was about 1:5. Compared to linear poly(styrene-*alt*-maleic anhydride) (LPSMA), BPSMA prepared CB pigment dispersion with a smaller particle size, a more narrow particle size distribution and higher stabilities.

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## 1. Introduction

Pigments are an important group of colorants that have been extensively used in coating, printing and paint industries due to many advantages in photosensitivity, color strength and transparency, etc. [1]. Pigments are commonly prepared for application in aqueous dispersions with the addition of dispersants because of their little water solubility. Conventional pigment dispersions generally have large particle sizes, wide particle size distributions and poor stabilities which limit their applications [2,3]. It is thus desirable to find a feasible method to overcome these drawbacks.

Dispersing pigments with aid of polymeric dispersants can greatly improve dispersion stabilities [4–6]. In aqueous media, polymeric dispersants are adsorbed onto pigment surface via anchor groups to build voluminous shells or intensify charges around pigment surface, thereby preventing flocculation and coagulation of the pigments. However, dispersing performance of polymeric dispersants may be limited due to some of their defects. For example, a polymeric dispersant, commonly having a low molecular weight, can be easily desorbed from pigment surface

at a high temperature and tends to tangle together for long-term storage, resulting in poor dispersion stabilities. An alternative approach to high performance pigment dispersion is to use the hyper-branched polymers with low viscosity, high solubility and numerous terminal functional groups [7–15]. Such hyper-branched polymers can overcome defects of conventional polymeric dispersants by building a more voluminous shell to provide enough repulsive forces among the pigment particles [16–19]. The use of hyper-branched polymers in aqueous pigment dispersions has recently been attracting increasing attention [20–23]. However, no studies, to the best of our knowledge, have been reported to date to use a hyper-branched poly(styrene-*alt*-maleic anhydride) (BPSMA) in aqueous carbon black dispersions.

In this study, we extensively investigated the effects of BPSMA structure and dispersing conditions on properties of carbon black dispersion, and also compared the dispersing ability with the linear poly(styrene-*alt*-maleic anhydride) (LPSMA) for carbon black pigment dispersion.

## 2. Materials and methods

### 2.1. Materials

CB pigment with a water content of 3% was purchased from Daxiang Co. Ltd. (China) and dried before using. 4-Vinylphenyl methanethiol (VPMT) was synthesized in our laboratory. Styrene (St) and maleic anhydride (MA) had inhibitor removed using an inhibitor-removal column (Aldrich). Benzoyl peroxide (BPO) was

Abbreviations: BPSMA, hyper-branched poly(styrene-*alt*-maleic anhydride); CB, carbon black; LPSMA, linear poly(styrene-*alt*-maleic anhydride); VPMT, 4-vinylphenyl methanethiol; PS, particle size;  $S_T$ , stability to temperature; CS, centrifugal stability; CS, centrifugal speed.

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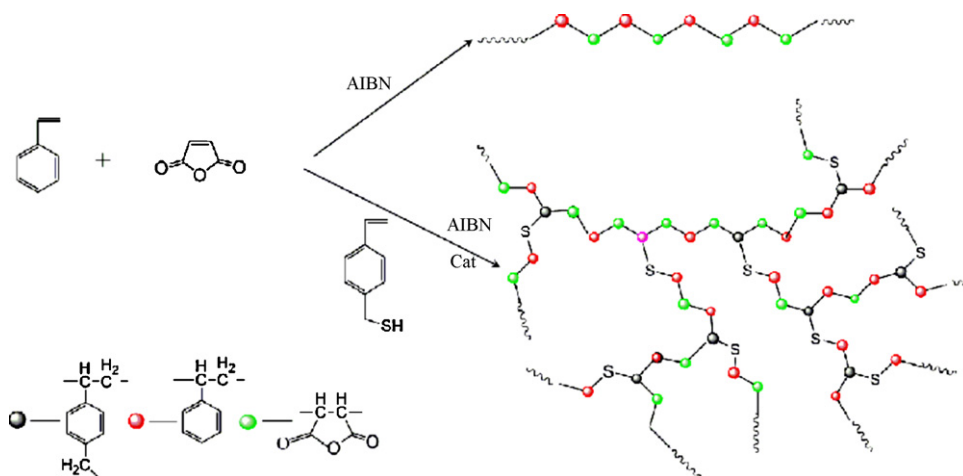


Fig. 1. Polymerization of St and MA with the addition of VPMT as a chain transfer agent.

purchased from Shanghai Chemical Reagent Co. Ltd. of China, and recrystallized from ethanol before using. 2-Butanone (AR grade) and sodium hydroxide (AR grade) were purchased from Shanghai Chemical Reagent Co. Ltd. of China. Distilled water was used in all the experiments.

## 2.2. Synthesis of BPSMA

BPSMA was synthesized according to Ref. [24]. Fig. 1 shows a scheme of polymerization of St and MA with the addition of VPMT as a chain transfer agent. Desired amounts of St, MA and VPMT were dissolved in 2-butanone to give a monomer solution. A desired amount of BPO was dissolved in 2-butanone to give an initiator solution. One third of monomer solution and one third of initiator solution were added to a four-neck flask equipped with a mechanical stirrer, a condenser and a thermometer. The mixture was heated to reflux for 1 h. The remaining monomer and initiator solutions were then added drop-wise to the four-neck flask, allowing the reaction to continue at reflux for 4 h. The product was precipitated by the addition of an adequate amount of ethanol at room temperature. The precipitated product was washed with ethanol for 3 times and then dried in a vacuum oven at 30 °C.

## 2.3. Synthesis of LPSMA

The reaction solution was prepared by dissolving 46.3 g of St, 43.7 g of MA and 1.8 g of BPO in 208.2 g of 2-butanone, these amounts being optimized as reported in a previous study [25]. The polymerization for LPSMA was carried out under the same conditions except that VPMT was excluded from the reaction solution. The product was precipitated by the addition of an adequate amount of ethanol at room temperature. The precipitated product was washed with ethanol for 3 times and then dried in a vacuum oven at 30 °C.

## 2.4. Preparation of CB pigment dispersions

A desired amount of LPSMA or BPSMA was dissolved in water at 70 °C. The pH of the polymer solution was adjusted to a target value using 0.1 mol/L sodium hydroxide. 2.5 g of CB pigment was added to the solution by balancing a total weight at 50 g. The mixture was stirred for 15 min, and then treated with ultrasonic waves (input power 1100 W) for 30 min.

## 2.5. Characterization of CB pigment dispersions

The morphology of particles in CB pigment dispersions was observed under a Hitachi H-7000 transmission electron microscope (TEM). The particle size (PS) and zeta potential ( $\zeta$ ) of CB pigment dispersion (diluted 100 times with distilled water) were determined by dynamic light scattering (DLS) using a Malvern Zetasizer Nano ZS90 instrument at an angle of 90°.

The stability to temperature ( $S_T$ ) of CB pigment dispersion was estimated in terms of relative change in particle size of a dispersion sample for 24 h storage at a desired temperature ( $T$ ) as given in Eq. (1):

$$S_T = \left| 1 - \frac{|PS_T - PS_0|}{PS_0} \right| \times 100\% \quad (1)$$

where  $PS_0$  and  $PS_T$  are the particle size of the CB pigment dispersion before and after 24 h storage, respectively.

The centrifugal stability ( $S_C$ ) of CB pigment dispersion was estimated in terms of relative change in absorbance ( $A$ ) of a dispersion sample after centrifugation process, i.e. the dispersion was centrifuged at a centrifugal speed ( $CS$ ) for 30 min and 0.03 g super dispersion was diluted 2000 times with distilled water. Spectrophotometry was performed on a UNICO UV-2000 spectrophotometer to determine the absorbance at the wavelength for the maximum absorbance (540 nm) before and after the centrifugation process, respectively, i.e.  $A_0$  and  $A_{CS}$ . The centrifugal stability was then calculated by Eq. (2):

$$S_C = \frac{A_{CS}}{A_0} \times 100\% \quad (2)$$

## 3. Results and discussion

### 3.1. Preparation of nanoscale CB pigment dispersion by BPSMA

Fig. 2 shows the effects of the VPMT amount used in polymerization on the dispersing ability of BPSMA for CB dispersions. BPSMA synthesized using an amount of VPMT at 6% exhibited the best dispersing ability. The appearance of the optimal amount of VPMT may be attributed to its relationship to the degree of branching in BPSMA. As shown in Fig. 1, an increase in the amount of VPMT for polymerization leads to a higher degree of branching in BPSMA. The branch chains in BPSMA play an important role in improving its dispersing ability for CB dispersion by enhancing the electrostatic repulsive forces on CB surface so as to result in a smaller particle size, a larger zeta potential, and higher stabilities to temperature and centrifugal forces. However, a higher amount of VPMT

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