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Preparation of modified carbon black with nano-scale size and enhanced stability in organic solvent by solid state method

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Abstract

To improve the dispersion stability of carbon black (CB) in organic solvent, a hinder phenyl antioxidant Irganox 1330 (1330) was chosen to modify the CB by a facile solid state method, which was based on the blending of CB and 1330 in an internal mixer. The majority of the modified CB particles are nanosized, as proven by dynamic light scattering and transmission electron microscopy. The modified CB nanoparticles were highly stable in acetone. Time-of-flight secondary ion mass spectroscopy was used to detect the presence of 1330 fragments on the CB surface and understand the preparation mechanism of the modified CB.

Keywords: Carbon black; Hinder phenyl antioxidant; Dispersion; Solid state method; Dynamic light scattering

1. Introduction

CB consists essentially of elemental carbon in the form of nearly spherical particles, which are typically 13–100 nm in diameter. Such spherical particles, also named primary particles, fuse together to form aggregates having size in the range of 200–1000 nm and further to micro-size agglomerates. The unique aggregate morphology of CB resulted in its poor dispersion in solvents or polymer matrix when CB was used for coatings [1], plastics [2], inks [3], inkjets [4,5] and toners [6].

To solve this key problem, lots of effective strategies including surfactant adsorption [7–9], substrate induced coagulation [10], oxidation [11,12] and polymer grafting [13,14] or encapsulating [15,16] have been developed for improving the dispersibility of CB. However, most of the above approaches were conducted in liquid phase, leading to an inconvenient processing. Moreover, the modified CB usually could not be dispersed in nano-scale size, resulting in its application often as micron material.

In the present work, solid state method was used to prepare the real nano-scale dispersed CB particles by blending a hinder phenyl antioxidant Irganox 1330 (1330) and CB in an internal mixer. By adopting the thermal and mechanical effects of the general mixing machine and the radical trapping nature of CB [17], the large agglomerates of CB were broken down into smaller aggregates and the decomposed 1330 fragments radicals were trapped by CB, which finally formed 1330 fragments modified CB. The resulting 1330 fragments modified CB could be dispersed stably in organic solvent in nano-scale size. It should be noted that solid state method is more advantageous due to its inherent simplicity and solvent-free reaction conditions. In our previous research [18], nature rubber grafted CB has been obtained successfully by the same method.

2. Experimental

2.1. Materials and preparation

CB used was Mogul-L provided by Cabot Corp. and its mean particle size was 24 nm. Irganox 1330 (1330) whose melting range was 240–245 °C was provided by Ciba Specialty Chemicals. The molecular structure of 1330 was shown in Fig. 1.

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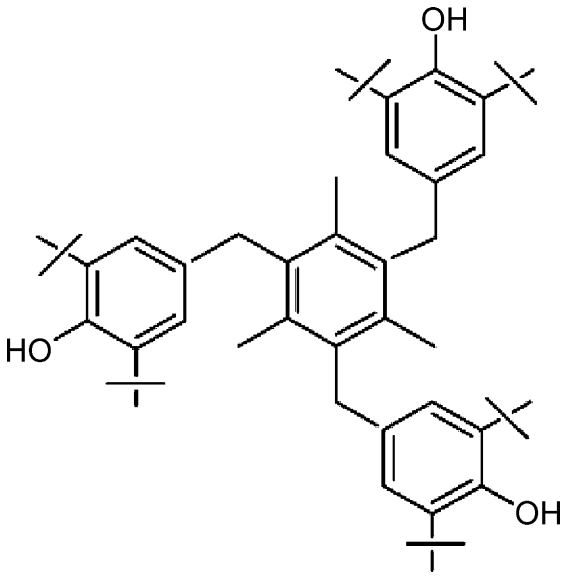

Fig. 1. Molecular structure of Irganox 1330.

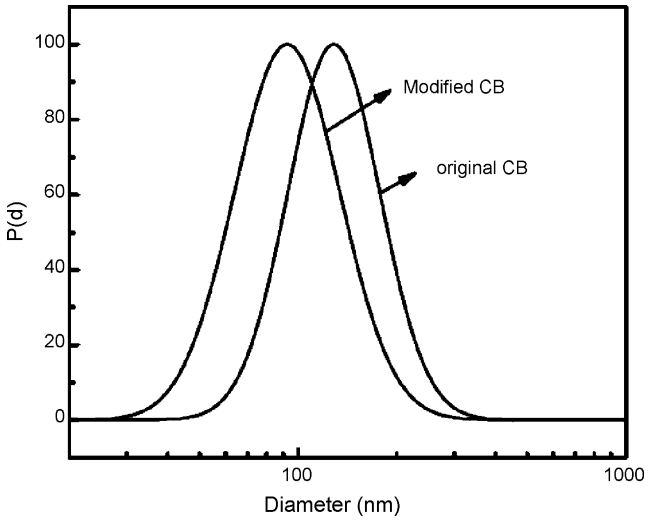

Fig. 2. The particle size distributions of original CB and modified CB.

Modified CB were prepared in an internal mixer (Rheomix600p, Haake, Germany). Firstly, CB blended with 1330 was treated for 30 min in Haake at starting temperature of 230 °C. Two counter-rotating σ-type rollers ran at a speed of 60 rpm. Then the products were filtered and extracted with acetone to remove the free and physical adsorbed 1330.

2.2. Characterizations

2.2.1. Dynamic light scattering (DLS)

DLS was used to measure the particle size of CB dispersed in acetone. The experiments were carried out by NICOMP^{TM} 380 ZLS. A He, Ne, Ar ion laser was light source and the wavelength of the laser light was 639 nm. The detection angle was 90° and the temperature was set at 23 °C. Further, by using a diluted carbon solvent dispersions (20 mg L^{-1}), the multiple scattering effects were avoided and the autocorrelation function was measured correctly [19].

2.2.2. Transmission electron microscopy (TEM)

TEM was used to investigate the morphology and particle size of the CB powder. It was carried out on a JEOL 2000FX microscope operating at 80 and 200 kV, respectively. The samples for TEM characterization were prepared by transferring a small amount of the CB acetone dispersions onto a 200-mesh copper grid covered with a Formvar^{TM} (polyvinyl formal) film and the excess suspension was removed by filter paper and dried by infrared lamp. Then, the sample was vacuumized and analyzed.

2.2.3. Time-of-flight secondary ion mass spectroscopy (ToF-SIMS)

ToF-SIMS was performed on a Physical Electronics TFS-2100 (TRIFTα) instrument. Mass spectra were acquired using

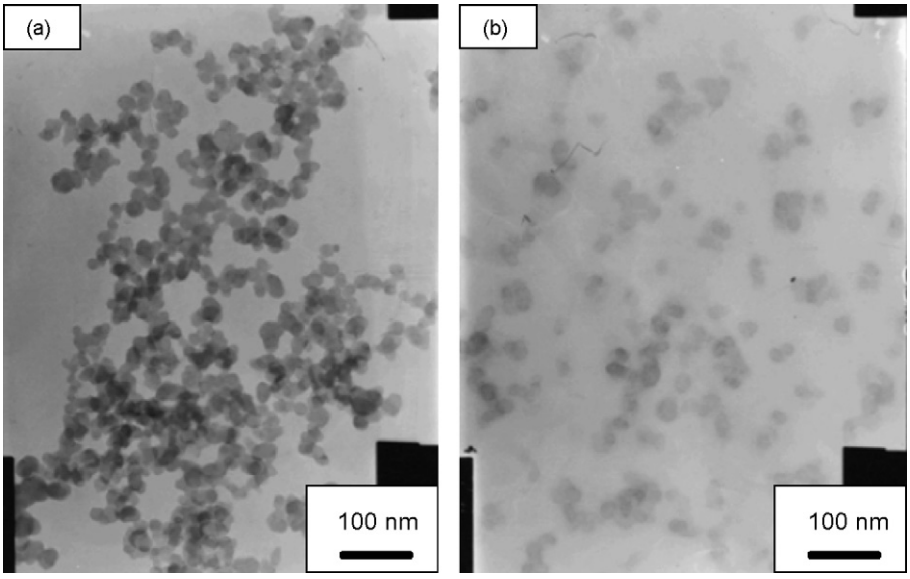

Fig. 3. TEM images of (a) original CB and (b) modified CB.