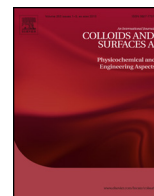




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Preparation and characterization of poly(sodium 4-styrenesulfonate)-decorated hydrophilic carbon black by one-step in situ ball milling



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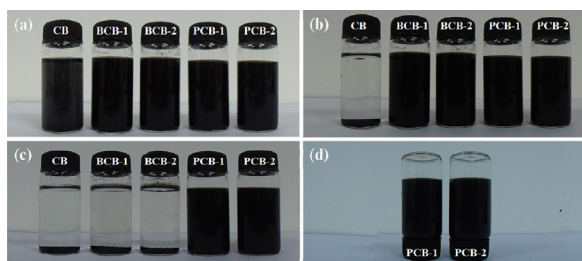
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HIGHLIGHTS

- Highly hydrophilic CB nanoparticles were prepared by one-step in situ ball milling.
- The resulting PCBs exhibited an extraordinary stability in water.
- The internal structure of CB could be seriously changed with the existence of PSS.
- Large CB aggregations could be broken into small pieces with the existence of PSS.

GRAPHICAL ABSTRACT

Natural sedimentation in water of (a) 0 day after (b) 30 min after (c) 15 days after (d) inverted bottle of PCBs. Ball milling without PSS could only make large CB aggregations loose, leading to the sedimentation of BCBs to the bottom slower than the pristine CB. With the existence of the PSS, the modified CB exhibited excellent dispersity and stability in water for at least 15 days.



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ABSTRACT

A one-step method for preparing poly(sodium 4-styrenesulfonate) (PSS)-decorated hydrophilic carbon black (CB), via in situ peeling of large CB aggregates into small pieces at room temperature, was investigated. The Raman spectroscopy results indicated that ball milling could only break large CB aggregates into small pieces but without changing its structure, while the structure of CB was significantly changed with the existence of PSS. The results of Fourier transform infrared spectroscopy, transmission electron microscope, energy dispersive X-ray spectroscopy and natural sedimentation experimental all showed that the CB was successfully coated by PSS and yielded an excellent stability in water. The backbones of PSS were coated onto the CB surface under strong π - π interactions with an encapsulation ratio of 12.7 wt%. The resulting PSS-decorated CB displayed an extraordinary stability in water with an average size of 221 nm and zeta potential of -33.8 mV, far outstripping the pristine CB and ball milled CB without PSS.

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1. Introduction

Carbon black (CB) is structurally complex particles and composed of irregularly shaped aggregates ranging in size from tens of

nanometers to several hundred nanometers [1]. CB also possesses excellent chemical stability, heat resistance, electrical conductivity, darkness, and safe features. So CB has been widely used as a reinforcing agent in the rubber industry, as filler in polymeric matrixes, as an electrode for supercapacitors and pigment in modern print technologies [2–8]. However, most of these applications require the good dispersibility of CB, and bare CB aggregates aggressively owing to its extremely large surface-area/particle-size ratio,

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which leading to the poor dispersibility of CB in most solvents, especially in aqueous solution [9], thus the practical applications of CB have been severely hindered. As the good dispersion of CB in water is required in many different application fields, the surface modification for improving its hydrophilicity by a low cost and environment-friendly method is of great significance and in urgent needs.

Until now, various methods have been reported for the surface modification and functionalization to enhance the hydrophilicity of CB, including oxide modification, graft modification, surface coating, and dispersant modification [1,10–18]. Zhu et al. fabricated dopamine encapsulated hydrophilic CB through oxide polymerization [1]. Xue et al. studied the oxidation mechanism of CB by HNO_3 [11]. Li et al. studied the effect of ultrasonic on the structure of CB, and successfully synthesized PVA-g-CB by ultrasonic method [12]. He et al. synthesized PSS-g-CB by using sodium 4-styrenesulfonate (NASS) monomer in HAAKE [13]. Paredes et al. enhanced the hydrophilicity of CB by plasma treatment [14]. Liu et al. synthesized hydrophilic DMAEMA-g-CB by grafting 2-DMAEMA from functionalized CB using atom transfer radical polymerization (ATRP) [15]. However, conventional surface modification technologies, such as oxide modification and dispersant modification have some drawbacks, like weak stirring capacities, uneven dispersion between agent and materials, and, in particular, lacking of reaction foundation [16,17]. Moreover, the preparation of hydrophilic CB by Ultrasonic, Haake and ATRP all required harsh conditions such as high-demand temperature, accurate time and precise reactant ratio control, leading to the hazardous, laborious and time consuming preparation process. Therefore, the development of an efficiency and environmental-friendly strategy for the surface hydrophilic modification of CB is of great importance.

Ball milling, which could activate the CB in the near surface region where solids come into contact with each other [18–20], is an effective surface modification method that could provide better results than conventional methods by ultrafine grinding. Furthermore, due to the changeable Gibbs free energy of reactions triggered by the large active particles, the chemical activation parameters and sites of polymer composites can be modified by the ball milling processing. Therefore, ball milling has been applied to a variety of organic syntheses with high-speed reactions and large-scale productions by controlling the parameters such as ball milling time and rotating speed [21,22].

In the previous study, the one step ball milling method was used to synthesis polymer-functionalized graphene nanocomposites [20], and here we report the preparation of PSS-decorated highly hydrophilic CB by this method. The as-prepared CB particles were coated by PSS under the binding force of strong π - π interactions [23–25]. The PSS chains, which are rich of highly hydrophilic sulfonic acid groups, would coat onto the CB surface and thus prevented the CB nanoparticles from aggregating and led to an extraordinary stability than the original CB and ball milled CB without PSS. This paper provides an environmental-friendly method for preparing excellent hydrophilic CB with terminal sulfonic acid groups.

2. Experimental

2.1. Materials

CB, in the form of Mogul-L, was obtained from Cabot Co. PSS was purchased from Nanjing Searchbio Tech. Co. Ltd., with the molecular weight of 200,000. Deionized water was also used in this study. All the materials were used without further purification.

2.2. Preparation of PSS-decorated CB

CB was dried for 24 h at 105 °C under vacuum before using. A planetary ball milling (QM-1SP2, Nanjing University instrument plant) was used in this study. Firstly, the grinding balls with good proportion were filled into the grinding bowl, which took up at least one-third of its volume to reduce the wear between grinding balls and bowl. Then put 5.0 g CB, 5.0 g PSS into the grinding bowl. To ensure that the samples were effectively grounded, 200 ml deionized water was filled but not past three quarters of the bowl volume. The closed grinding bowl was then placed into the planetary ball mill and secured using the “safe lock”. The counter weight was adjusted based on the weight of the bowl and samples to reduce the vibration of the milling operation [26]. Finally, the modified CB was separated from aqueous solution by ultra-centrifuge (SORVALL RC6+ Centrifuge, American) at 20,000 rpm for 30 min. As comparison, ball milled CB without PSS was also prepared to study the mechanism of ball milling on the evolution of CB structure. To simplify the following discussion, the ball milled CB without PSS treated for 18 and 36 h were abbreviated as BCB-1 and BCB-2, respectively. Similarly, the ball milled CB with PSS treated for 18 and 36 h were named as PCB-1 and PCB-2, respectively.

2.3. Characterizations

Raman spectra were recorded using an inVia + Reflex from Renishaw, UK. A He-Ne laser at an excitation wavelength of 633 nm was used to accurately profile the contribution of D bands (~1340), G bands (~1600) and A bands (1550) of CB [11]. Generally, the D band is attribute to the presence of defects (sp^3 carbons, vacancies, foreign atoms, etc.), while the G band is considered to be an in-plane bond-stretching motion of sp^2 -hybridised C atoms [27,28], and the A band is ascribed to amorphous carbon [29]. A linear background was subtracted. Then, the Lorentzian-shaped D and G bands and the broad, Gaussian-shaped A band were used to fit the spectra [29]. The ratio of G and D bands can be used to approximate the microcrystalline size (L_a) of the CB particles [11,30].

Fourier transform infrared spectroscopy (FTIR) spectra were obtained by a Nicolet 5700 spectrometer under ambient conditions. The spectra were obtained in the wave number range from 600 to 4000 cm^{-1} .

The elements composition and surface morphologies of CB particles were observed by energy dispersive X-ray spectroscopy (EDS) (QUANTAX 400-30, Bruker AXS GmbH) and transmission electron microscope (TEM) (JEM-2010, Hitachi, Japan), respectively. The CB samples were ultrasonically dispersed in distilled water, and then a drop of the solution was deposited on a copper screen for TEM characterization.

The particle size distribution and zeta potential of CB was measured by a laser particle analyzer (Zetasize Nano-ZEN3600, Malvern, Great Britain). The CB samples were dispersed in distilled water with ultrasonic for 30 min, and then the dispersions (0.005 wt% CB) were laid up in the laser particle analyzer.

The thermal gravimetric analysis (TGA) of CB was carried out with TGA analyzer (STA449 F3 Jupiter, NETZSCH, German). Each sample weighing about 10 mg was heated from room temperature to 800 °C at a rate of 10 °C min^{-1} in nitrogen atmosphere.

3. Results and discussion

Raman spectroscopy, which is an useful method to determine the structure of carbon materials, was used to study the evolution of CB structure during ball milling process [31]. Fig. 1 shows the Raman spectra acquired from CB, BCBs (BCB-1 and BCB-2) and PCBs (PCB-1 and PCB-2) samples. Each spectrum corresponds to

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