

Highly conductive graphene oxide/polyaniline hybrid polymer nanocomposites with simultaneously improved mechanical properties



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ARTICLE INFO

Article history:

Received 27 August 2015

Received in revised form 9 December 2015

Accepted 14 December 2015

Available online 19 December 2015

Keywords:

A. Graphene

A. Polymer-matrix composites (PMCs)

B. Electrical properties

B. Mechanical properties

ABSTRACT

Graphene oxide (GO) was added to a polymer composites system consisting of surfactant-wrapped/doped polyaniline (PANI) and divinylbenzene (DVB). The nanocomposites were fabricated by a simple blending, ultrasonic dispersion and curing process. The new composites show higher conductivity (0.02–9.8 S/cm) than the other reported polymer system filled with PANI (10^{-9} – 10^{-1} S/cm). With only 0.45 wt% loading of GO, at least 29% enhancement in electric conductivity and 29.8% increase in bending modulus of the composites were gained. Besides, thermal stability of the composites was also improved. UV–Vis spectroscopy, X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) revealed that addition of GO improves the dispersion of PANI in the polymer composite, which is the key to realize high conductivity.

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1. Introduction

Conductive polymer composites are at the forward position of materials science research because of the large number of applications that have been developed utilizing their interesting and unique features integrating the good mechanical, electrical [1], and thermal properties [2]. Polyaniline (PANI) is one of the most promising conducting polymers for industrial application mainly for its easy preparation, low cost, excellent electrical, optical, magnetic properties and environmental stability [3–5]. However, due to poor mechanical properties and poor process performance, blending PANI with another polymer matrix has been shown to be a good strategy to develop conducting structure composites [6–8]. For example, epoxy resin/PANI–DBSA (n-Dodecylbenzenesulfonic acid) composite with the electrical conductivity in the range for electrostatic discharge (ESD) applications (10^{-9} – 10^{-2} S/cm) have been reported by Tsotra and Friedrich [9], and Massoumi et al. [10] Afzal et al. [11] used a solution blending technique to synthesize polyvinyl chloride/PANI–DBSA blends with the highest tensile strength of 37.7 MPa when adding 20% of polyaniline emeraldine base. Our

group has also reported a simplified one-step method to prepare PANI/DVB conductive composites, where doping of PANI and curing of the composite take place simultaneously [8].

Graphene oxide (GO) is an attractive nanomaterial because of its low cost, mass production and solution processability. It is established that graphene can help to get a better dispersion of other additives in the polymer matrix by synergetic effects due to its extremely high aspect ratio and surface area [12–14], while GO has the similar structure. Good dispersion of the components leads to the formation of efficient networks for strain, electrical and heat transfer in the composite materials [15–17]. On the other hand, as an oxidized derivative of graphene, GO has a similar structure as graphene except for diverse chemical functional groups on the surface: carbonyl, epoxide, carboxylate and alcohols [18], which can reduce restacking, enhance interfacial interactions between the dispersed GO and the polymer matrix. As a result, GO has been used as multiscale reinforcement fillers to fabricate polymer composites. For example, Hussain reported multiscale polymer composites by using aramid fibers and GO nanosheets, where the tensile strength of the material improved significantly [19].

In this paper, we investigated the effect of GO added to a polymer composites system consisting of surfactant-wrapped/doped polyaniline (PANI) and divinylbenzene (DVB), where simultaneous improved conductivity and mechanical properties were obtained

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in the new nanocomposites. The nanocomposites are prepared by using DVB as solution-phase premixed with GO of different concentrations, which was then mixed with the part-doping PANI to form an all-organic composite and then followed by curing at higher temperature. The morphology of nanocomposites, dispersity and doping process were studied by SEM, UV–Vis and XRD.

2. Experimental details

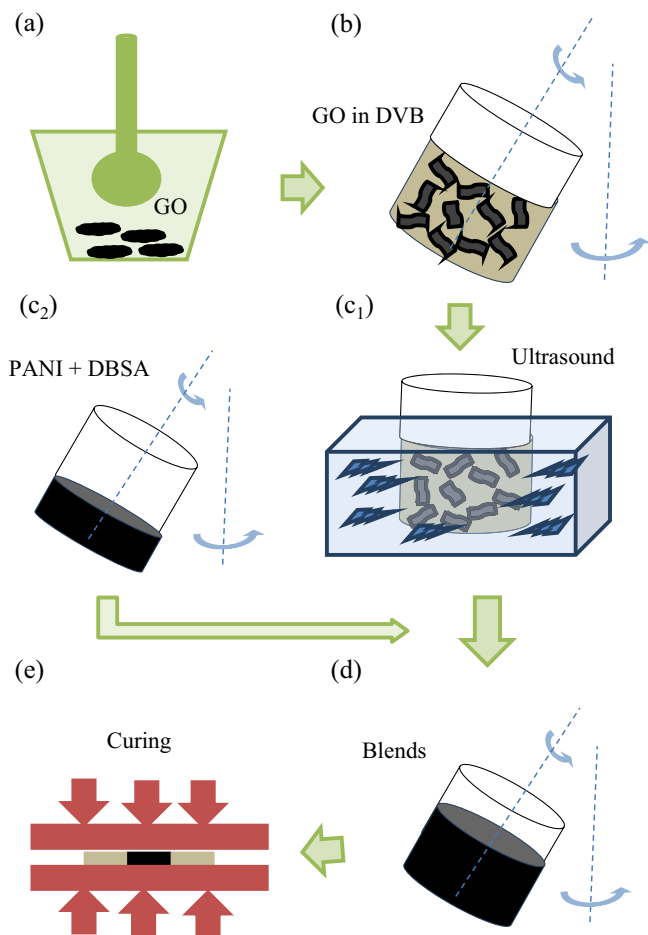
2.1. Materials

PANI Powder, supplied by Regulus Co. Ltd., Tokyo, Japan; DBSA, Kanto Chemical Co. Inc., Tokyo, Japan; DVB, supplied by Sigma-Aldrich Co., St. Louis, USA. PANI in emeraldine base form was used, and then doped with DBSA to make emeraldine salt. The conductivity of the prepared emeraldine salt was in the range of 10–20 S/cm.

GO was synthesized based on Hummers method [20]. The detailed process was reported elsewhere [21].

2.2. Processing of the composites

The process for preparing DVB nanocomposites is shown in Scheme 1. The composites were prepared by the following 3 main steps. Firstly, fine-grinded GO was dispersed into DVB solution by a



Scheme 1. Schematic illustration of the preparation process for DVB composites with PANI and GO (a) grinding GO into small particles; (b) centrifugal mixing GO in DVB; (c₁) dispersing GO in DVB by sonication; (c₂) centrifugal mixing of PANI with DBSA; (d) final centrifugal mixing; (e) curing under hot-press machine. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

centrifugal mixer and a sonicator (Scheme 1a–c₁). In parallel, PANI and DBSA were mixed thoroughly in a centrifugal mixer to obtain a homogeneous paste (Scheme 1c₂), which were then added to the GO/DVB solution. The suspension was again mixed thoroughly (Scheme 1d) and then poured into the mold for curing in a hot-press machine (Scheme 1e).

2.2.1. Preparation of PANI–DBSA doping paste

PANI was kept in an oven for 2 h at 40 °C to eliminate moisture content. Dried PANI and DBSA were mixed to form the PANI–DBSA paste by physical mixing in the ratio of 30:70 by weight percentage which is equivalent to the molar ratio of 1:0.69 of PANI:DBSA [8]. The mix paste of PANI–DBSA was made by a centrifugal mixer with speed of 2000 rpm for 5 min 3 times.

2.2.2. Preparation of DVB–GO solution

GO was dispersed into DVB by inconsecutive sonication for 10 h in the ice bath to get good disperse suspension. The content of the suspension is 0.15%, 0.3%, 0.45%, 0.6%. The dispersion stability against Van der Waals aggregation of GO in DVB was monitored at 10 min, 12 h and 24 h (Fig. 1). GO stayed homogenous in the DVB suspension at least for 24 h during which no obvious settlement was observed.

2.2.3. Preparation of PANI–DBSA/DVB–GO

The paste of PANI–DBSA and DVB–GO was then mixed by a centrifugal mixer and poured into a mold, cured using a Hot-press machine (Toyoseiki Minitest press.10). Samples were put into the Hot-press machine at 120 °C for 2 h [8]. Samples of different dimensions were cut for various measurements and at least 3 parallel samples for every test were prepared and measured.

2.3. Characterizations

UV–Vis absorption spectra of PANI–DBSA, PANI–DBSA/DVB, PANI–DBSA/DVB–GO films were obtained using a U-4100 spectrophotometer. Spectra were recorded from 250 to 1100 nm. Wide-angle X-ray scattering (WAXS) measurements were performed on a MicroMax007, RAXIS-IV ++ with copper X-ray tube (Cu K α , wavelength $\lambda = 0.15418$ nm). The data were collected in the step-by-step mode of 0.038° from 5° to 45° in 2 θ . Thermogravimetric analysis was performed with a SDT Q600 V20.9 Build 20 instrument under a 100 mL min⁻¹ N₂ at the heating rate of 10 °C min⁻¹. Atomic Force Microscopy (AFM) measurements were performed to characterize the thickness and dimensions of the as prepared nanosheet using a Scanning Probe Microscope (Veeco Instruments, Nanoscope Multimode IIIa, USA) operating in contact mode. FT-IR spectra were investigated using FT-IR analyzer (NICOLET 6700, Thermo Scientific Corporation). The electrical conductivity (DC measurement) of the polymer composites was measured using LCR meter (3522-50LCR HiTESTER, Hioki E.E. Corporation, Ueda, Japan) by four-probe method. DOTITE conductive adhesive paste (supplied by Fujikura Kasei Co. Ltd. Tokyo, Japan) and aluminum tape have been used to measure the conductivity. We applied constant voltage of 1 V and then the drop of current and voltage was measured using highly precise ammeter and voltmeter respectively to calculate the resistance of the samples using Ohm's law ($R = V/I$). The resistance R , of uniform cross section can be computed as $R = \rho l/A$, while $\rho = 1/\sigma$, where l is the length of the conductor, A is the cross-section area of the conductor, σ (sigma) is the electrical conductivity and ρ (rho) is the electrical resistivity of the material. Resistivity and conductivity are reciprocals.

The flexural properties of the polymer composites were investigated according to ASTM D790, using Universal Testing Machine (Instron-5582) by three-point bending method. High quality of scanning electron microscopy (SEM) pictures were obtained on a

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