



Facile fabrication of poly (tetrafluoroethylene)/graphene nanocomposite via electrostatic self-assembly approach



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ABSTRACT

Dispersion of graphene nanosheets (GNs) in a polymer matrix as mono layers was an important step towards fabricating high performance polymer/GNs nanocomposites. However, the insoluble and infusible properties of poly (tetrafluoroethylene) (PTFE) made the incorporation of nanofillers in PTFE matrix difficult. In this paper, a novel method based on poly (tetrafluoroethylene) (PTFE) latex that assured the fine dispersion of GNs in PTFE matrix was developed. PTFE latex was first modified by polyethylenimine (PEI) to create the positive charges on the surface of the PTFE particles, and then assembled with negatively charged graphene oxide sheets directly in water through electrostatic interaction, followed with chemical reduction, cold briquetting and hot sintering. Nanocomposites with controllable content and uniformly distributed GNs in PTFE matrix were prepared. The electrostatic coupling interactions improved the dispersion of GNs and facilitated the formation of filler networks in the PTFE matrix. Both the mechanical and wear performance of PTFE/GNs nanocomposites were greatly improved. PTFE/GNs nanocomposites also exhibited excellent electrical properties with a percolation threshold as low as 0.5 wt% and an electrical conductivity of 1.4 S/m at only 2 wt% graphene loadings. The new method agrees well with the latex technical process in PTFE bulk industrial manufacture and paves the way for an environmentally benign process for the bulk production of high quality polymer–graphene nanocomposites.

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1. Introduction

Graphene, a two dimensional macromolecular sheet of carbon atoms with a honeycomb structure, has attracted tremendous attention in the research community recent years owing to its outstanding thermal, mechanical, and electrical properties [1–3]. Graphene sheets (GNs) are considered as an ideal two-dimensional reinforcing component for versatile composite materials. When incorporated appropriately, these GNs with atomic thickness can significantly improve physical properties of the host polymers at extremely small loading [4–8]. As known from classic composites science, the fine dispersion of graphene in the matrix is one of the most important and challenging tasks towards maximizing the transfer of the intrinsic graphene attributes into the composites. A variety of chemical and physical approaches have been introduced to avoid the aggregation of GNs in polymer matrices.

Several methods have been proposed, including melt-mixing, electrostatic self-assembly, the co-coagulation method, and in situ polymerization [5,9–13]. While, challenges remain mainly from the difficult dispersion of the GNs in polymeric media or the complicated preparation methods, which require large amounts of organic solvents.

Polytetrafluoroethylene (PTFE) is widely used as a kind of special engineering plastic due to its self-lubricating capacity, high chemical inertness and excellent thermal stability. However the low hardness, poor mechanical properties, high wear rates and severe creep deformation of PTFE have hindered its applications. Many scholars have carried out a great deal of work to modify and reinforce PTFE [14,15] to improve the overall performance of PTFE for expanding its applied fields by adding micron or nano size fillers [16–20], such as graphite, molybdenum disulfide, SiC, SiO₂, Al₂O₃, ZnO, and MWCNT. However, the extremely high molecular weight of PTFE leads to a melting viscosity about six orders of magnitude higher than that of most thermoplastic polymers, so that melt-processing is not applicable for PTFE fabrication. Instead, powder metallurgy is used for manufacturing block items from virgin PTFE powders produced by emulsion or suspension

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polymerization, which makes the dispersion of nanofillers in PTFE even more difficult.

In this paper, we developed an environmentally friendly approach to prepare fine dispersed PTFE/graphene composites (PTFE/GNs) by a simple electrostatic self-assembly process where a well-dispersed GO dispersion having a negative charge was mixed with modified positively charged PTFE latex. The hybrids of PTFE–GN obtained via this process were used as the initial building block of PTFE/GNs composites, in which, GNs were incorporated onto the surface of PTFE microspheres by this surprisingly simple and effective process. As a result, the distribution of GNs within the final composite materials was highly controllable. The well dispersed GNs led to significantly improved mechanical and electrical conductivity at low GNs loading. The electrostatic method starting from commercial available PTFE latex was particularly compatible with the processing procedure of modern industrial manufacture of PTFE products. This work demonstrated an environmentally friendly and effective way to obtain graphene-based material by utilizing the strong interaction between modified PTFE latex and graphene sheets.

2. Experimental section

2.1. Material

The commercial aqueous PTFE dispersion (20 wt% solid content) was supplied by Zhonghao Chenguang Research Institute of Chemical Industry. The 200 nm sized PTFE particles were well dispersed in water with the help of 0.2 wt% of perfluorooctanoic acid (PFOA). Polyethyleneimine (PEI) was purchased from Aldrich. Graphite powders were purchased from Qingdao Black Dragon graphite Co., Ltd. Potassium permanganate (KMnO_4), sulfuric acid (H_2SO_4 98%), hydrogen peroxide (H_2O_2) and sodium nitrate (NaNO_3), sodium hydroxide (NaOH), hydriodic acid (HI 38%) were purchased from Kermel Chemical reagent plant (Chengdu, China), all reagents were used as received.

2.2. Preparation of PTFE/graphene nanocomposites

GO was prepared from natural graphite by the classical Hummers' method. Exfoliation of GO was achieved by sonication for 2 h in aqueous solvent; centrifugation was used to remove non-exfoliated GO sheets. The as-synthesized GO (10 mg/mL) was diluted with deionized water to 1.0 mg/mL and sonicated in an ultrasonic bath for 10 min to create a homogeneous GO dispersion.

Positively charged PTFE particles were prepared by addition of 200 mL of PEI solution (2 mg/mL) to 1000 mL of a 20 wt% dispersion of PTFE latex (zeta potential -20.7 mV). The mixture was left for 30 min, then repeatedly centrifuged (3000 rpm, 20 min) and washed to remove excess PEI polymer, redispersing the PTFE particles in deionized water, which led to a positively charged stable PTFE latex (zeta potential $+25.6$ mV). Prior to use, the PEI-coated PTFE particles were sonicated for 10 min in an ultrasonic bath to disassemble any large aggregates. GO-wrapped PTFE particles were prepared by adding the PEI-coated PTFE particles (typically 5 wt% aqueous solution) to GO dispersion (1.0 mg/ml) and mixed at high speed for 30 min. A yellow precipitate was immediately formed, and the coagulated sample was collected through filtration, then washed in deionized water three times to remove residually isolated and unattached GO sheets and dried overnight. Subsequently, the PTFE–GO hybrid was in-situ reduced with hydriodic acid at 80°C for 8 h. The obtained black PTFE–GN hybrids were filtered, washed, air-dried and then vacuum-dried at 150°C overnight. Samples of hybrids containing 0.25, 0.5, 1, 1.5 and 2 wt% GNs were prepared via the same method. PTFE/

GNs nanocomposites were fabricated from the PTFE–GN hybrids through cold briquetting and hot sintering technologies. The hybrids were compressed into billets at 25°C and 25 MPa for 5 min, and then free sintered in air at 380°C for 5 min.

2.3. Characterization

Zeta potentials of the graphene oxide dispersion and PTFE latex were measured using a laser electrophoresis zeta potential analyzer (Etasizer 3000HSA Malvern Instruments). Raman spectra of GO, RGO and PTFE–GN hybrid samples were obtained using a multi-channel confocal micro spectrometer with a laser wavelength of 535 nm.

The morphology, microstructure of PTFE latex particles, PTFE–GO, PTFE–GN hybrid powder and the fresh-fractured cross-section of the PTFE/GNs nanocomposites surfaces were investigated by SEM instrument (SEM, JSM-5900LV). High-resolution TEM was also carried out to further elucidate the morphology and structure of PTFE/GN nanocomposites by TEM, Tecnai F20 S-TWIN measurements.

Tensile tests were conducted on Instron testing machine, with 20 mm/min cross-head rate, and carried out at 23°C . The results were subjected to standard statistical treatment; the data presented were average values for a series of at least five measurements with standard deviation not exceeding 10%. Tensile modulus was estimated from the initial linear part of the stress–strain curve limited by 3% relative elongation.

Friction and wear tests were conducted in dry friction mode choosing thrust-washer arrangement as the friction pair at the specific load of 1 MPa and relative velocity of 1 m/s on 2070 CMT-1 friction machine. The steel roller measuring 40 mm in diameter and 12 mm wide made from 40X steel hardened to 46–48 HRC and machined to $R_a \leq 0.16 \mu\text{m}$ was used as a counterface. Wear rates based on mass losses were determined by weighing on analytical balances with an accuracy of 0.5×10^{-5} g and referred to the steady state wear. Friction coefficients were calculated from the friction moments and corresponded to the steady friction stage. The temperature in the friction zone was measured with a thermocouple which probe was located on the roller surface next to the junction of the specimen and counterface.

The electrical conductivity was measured using a standard four-probe method. A series of parallel contact lines 1 cm in length and with a 1 cm interval were drawn with conductive-silver paint (Fluka) on the composite material, and all conductivity measurements were performed at room temperature, conductivity data represented the average value of 10 consecutive measurements.

3. Results and discussion

3.1. Self-assembly of PTFE latex and GO through electrostatic interaction

When the PTFE colloids were added to the homogeneous dispersed GO aqueous dispersion, adsorption of the GO rapidly occurred on the surface of the PTFE microspheres, thereby inducing the co-coagulation of the two species. As illustrated in Fig. 1, the PTFE/GO was prepared by the electrostatic interaction driven self-assembly strategy. After the dilute suspension of GO poured into aqueous PTFE latex under mild mechanical shaking, a spontaneous change of dispersion from respective colloidal states of GO and PTFE latex to irreversible yellow agglomerates occurred (Fig. 2b), indicating the formation of GO–PTFE particles assembly. The appearance of the simultaneous sedimentation of GO nanosheets with PTFE latex implied the strong interactions between the GO and PTFE latex particles. The electrostatic interaction which

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