

Synthesis and electrorheology of multi-walled carbon nanotube/polyaniline nanoparticles

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Abstract

Nanoparticles with multi-walled carbon nanotube (MWNT) and polyaniline (PANI) were synthesized via an oxidative dispersion polymerization using poly(vinyl alcohol) (PVA) as a polymeric stabilizer and HCl as a dopant. Prior to the synthesis, the MWNT was treated with strong acidic solutions via a reflux process to eliminate impurities including metallic catalysts. These MWNT/PANI nanoparticles were well dispersed in water because the hydrophilic PVA was stably adsorbed onto the nanoparticle surface. These MWNT/PANI nanoparticles were used as suspended particles for an electrorheological (ER) suspension, and the rheological properties for this ER fluid including yield stress and shear viscosity under several different applied electric field strengths were examined via a rotational rheometer equipped with a high DC voltage generator.

Keywords : multi-walled carbon nanotube, polyaniline, electrorheology

1. Introduction

Carbon nanotubes (CNTs) have been widely studied as fillers for polymeric composites due to their extremely high strength, stiffness and flexibility [1, 2]. CNTs have a large aspect ratio (nanometer scale diameter vs. micrometer scale length) with low densities. These large aspect ratios can enhance their electrical conductivity [3, 4]. A single-walled carbon nanotube (SWNT) is known to be much stiffer than steel in mechanical strength and the MWNTs have the modulus of 200–4,000 GPa [2]. Due to these remarkable properties, CNTs can be considered as a potential filling material in polymeric nanocomposites.

When compared with composites containing spherical conducting particles, CNT composites show a high electrical conductivity at a low CNT concentration due to a low percolation threshold [4]. Furthermore, nanoscopic dimension can ensure the transparency. Applications of such polymer composites require well-dispersed CNTs with good affinity between CNTs and polymers. Since they have large surface areas and possess strong van der Waals interparticle forces, which result in aggregation,

it is extremely difficult to disperse CNTs in a polymeric matrix.

We prepared MWNT/PANI composites via an oxidative dispersion polymerization. A surfactant PVA with hydrophilic OH group under ultrasonication was used for dispersing MWNT in water. We then characterized their electrorheological (ER) properties, in which an ER fluid is generally a colloidal suspension of conducting particles in an insulating oil with a low dielectric constant and a low viscosity [5, 6]. They show a reversible transition from a liquid-like state to a solid-like state under an applied electric field. The dramatic increase in viscosity and rapid response time originate from the substantial alteration of the suspension structure, especially the field-induced formation of fibrous aggregation aligned with the applied electric field. The well-known electrostatic polarization model incorporates the field-induced polarization of the dispersed phase particles relative to the continuous phase, in which the driving force of the particle fibrillation originates mainly from the electrostatic interaction among the particles. Various conducting polymers such as PANI [7, 8], poly(acene quinone) radicals [9], polyphenylenediamine

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[10], poly(*p*-phenylene) [11], and PANI-inorganic composites [12, 13] are known as potential ER materials. These materials possess either branched polar groups such as amine, hydroxyl and amino-cyano, or semi-conducting repeated groups. ER fluids have been applied in various electromechanical devices such as engine mounts and shock absorbers because of their superior design properties; short response time, low power consumption, smoothness of operation, and simple mechanics [14].

The applied electric field induces formation of chainlike structures of particles in surrounding fluid in the order of milliseconds. As a result, the apparent viscosity of the ER fluid increases tremendously when an electric field is applied.

2. Experimental

We purified the MWNT (Iljin Nanotec Co., Korea), synthesized by a thermal chemical vapor deposition method, to eliminate impurities such as metallic catalysts. Purity of the pristine MWNT was 97 %. It was treated in 3M HNO₃ at 60 °C for 12 h, followed by a reflux process in 5 M HCl at 120 °C for 6 h with stirring [15, 16]. Purity of the after-treated MWNT was measured, using thermogravimetric analysis (TGA, Polymer Lab., TGA1000, UK), to be 99 %. PVA was dissolved in deionized water. Then the acid-treated MWNT (0.1 wt% for aniline), aniline monomer and dopant HCl were added in PVA solution. Since MWNTs by themselves are difficult to be dispersed uniformly, we used a surfactant PVA to disperse hydrophobic MWNT into water. Ultrasonication and stirring via ultrasonic generator (Kyungill Ultrasonic Co., Korea), with a nominal frequency of 28 kHz and power of 600 W, for 5 h at 25 °C were applied to break up MWNT aggregates. The size of dispersed MWNTs in the solution was measured by light scattering method (Otsuka, ELS-8000, Japan).

The deionized water containing ammonium peroxydisulfate was added later through dropping, with continuous stirring and ultrasonication for 24 h at 20 °C. The MWNT/PANI composites were separated three times by centrifugation at 5000 rpm for 0.5 h. An ER fluid was prepared by suspending MWNT/PANI particles in an insulating silicone oil, and its rheological properties were examined under an applied DC electric field. The flow curves of MWNT/PANI nanocomposites based ER fluid were measured by a rotational rheometer (Physica, MCR 300, Germany) equipped with a high DC voltage generator.

3. Results and Discussion

Figure 1 shows a photograph of MWNT dispersed in an aniline monomer and HCl with three different PVA concentrations. Each vial contains the same amount of CNT 0.1wt%. The concentration of PVA in deionized water is

0.5, 1, and 5 wt% from left to right. PVA was dissolved in water at 90 °C and ultrasonicated for 5 h to disperse MWNTs into the solution by surfactant PVA as the hydrophobic part of PVA contacts with MWNTs and aniline monomers, while the hydrophilic part contacts with water.

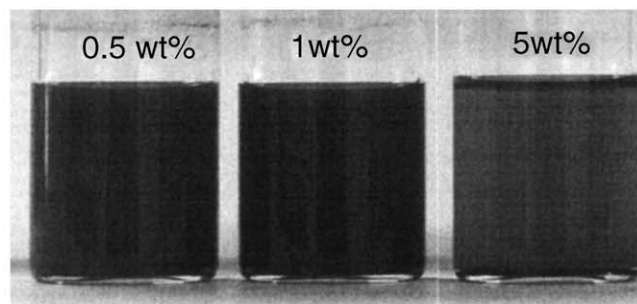


Figure 1. Visualization of MWNT in the solution (PVA concentration of 0.5, 1 and 5 wt%)

One month later, the degree of dispersion and sedimentation of MWNTs in PVA solution were examined. The solutions with low PVA concentration (0.5 and 1 wt%) after one month, possess a black color, indicating that individual MWNTs were uniformly dispersed in the solution. The sedimentation of right vial (5 wt%) was formed via interconnection of the MWNTs while the upper of the solution appears to be transparent. The sedimentation of MWNTs is due to the high viscosity of the solution at high PVA concentration. The size distributions of dispersed MWNTs in each solution were measured by light scattering and presented using a number conversion distribution (Fig. 2).

The size distributions of 0.5 and 1 wt% PVA solutions (with an opaque black appearance) are narrower than that of 5 wt% PVA solution. Although semitransparent PVA solution (5 wt%) has a similar size distribution as that of 0.5 and 1 wt%, there exists another CNT cluster of larger size (above 1000 nm) in 5 wt% solution (binodal distribution).

We found that the concentration of PVA affects MWNT dispersion in the solution. Therefore, the most monodisperse PVA solution (1 wt%) was selected for further investigation.

The flow curve of MWNT/PANI suspension in insulating silicone oil (ER fluid) under four different DC electric field strengths is shown in Fig. 3. Before applying the shear, a DC electric field was maintained for 3 min to make an equilibrium columnar structure. The measurements were performed at 25 °C. At a low shear rate, the shear stress remained almost constant (plateau region) as the shear rate increased to certain critical value.

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