

Fabrication and properties of polycarbonate composites with polycarbonate grafted multi-walled carbon nanotubes by reactive extrusion



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ABSTRACT

Bisphenol-A polycarbonate (PC) and multi-walled carbon nanotubes (MWCNTs) functionalized with hydroxyl groups were melt mixed in a twin extruder to produce PC composites containing PC-grafted MWCNTs (PC-g-MWCNTs) by reacting hydroxyl groups on MWCNTs with the carbonate groups in PC. Formation of PC-g-MWCNTs by reactive extrusion was explored as were their resulting properties including interfacial adhesion energies between PC and MWCNTs, dispersion of MWCNTs in the PC matrix and mechanical and electrical properties of PC/MWCNT composites. The interfacial adhesion energy of the PC/PC-g-MWCNT composite was higher than that of the PC/pristine MWCNT composite; as a result, the PC/PC-g-MWCNT composite exhibited a higher level of dispersion of MWCNTs in the PC matrix and better adhesion at the interface between PC and MWCNTs than the PC/pristine MWCNT composite. For a fixed MWCNT content in the composite, the mechanical strength of the PC/PC-g-MWCNT composite was higher than those of the corresponding PC/pristine MWCNT composite.

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1. Introduction

Carbon nanotubes (CNTs) are a one-dimensional material that possesses excellent mechanical strength and electrical and thermal conductivity [1–7]. Polymer/CNT composites have been the subject of many recent papers [7–17]. This interest is mainly driven by the promise of strongly enhanced physical properties. To achieve such properties in composites requires high levels of dispersion of the CNTs, which is possible when there is good affinity between the polymer and the CNT. Since CNTs are entangled due to the strong inter-tube van der Waals forces, which occur as they are synthesized, the inherent properties of CNTs cannot be exploited without eliminating this CNT entanglement in the polymer matrix. Much effort has been made to safely disentangle CNTs and disperse them in the polymer matrix [10–17].

Bisphenol-A polycarbonate (PC) has experienced substantial growth in its use since its introduction in the late 1950s [18]. The main reason for this growing market is its unique combination of properties including high heat distortion resistance and impact strength, fire retardant properties, transparency, and dimensional

stability. Even though PC is especially suitable for the housings of electrical devices, its applications are often limited because of its low stiffness, strength, and electrical conductivity. In principle, such properties can be improved by adding small amounts of CNTs to the PC matrix [12–17,19–27]. It was shown that CNTs are very effective at increasing mechanical properties and electrical conductivity of PC composites at low loading levels without significantly increasing the melt viscosity [19–27]. The production of composites filled with CNTs requires the ability to effectively disperse the nanotubes throughout the polymer.

Surface treatments of CNTs are commonly used to enhance dispersion of CNTs in a polymer matrix and improve interfacial adhesion between CNTs and polymer matrices [11–17,28]. One of the most widely used methods is the covalent introduction of functional groups on the CNT surface. PC composites with functionalized CNTs were produced to enhance the stiffness and strength of PC [12–17]. PC composites containing multi-walled carbon nanotubes (MWCNTs) functionalized with ozone were fabricated, and then their properties were examined [14]. Eitan et al. prepared MWCNTs functionalized with an organic molecule containing a terminal epoxide group, and then PC composite films with these MWCNTs were fabricated by solution casting to examine the load transfer mechanism at the interface [16]. PC composites with MWCNTs encapsulated with poly(methyl methacrylate) were

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studied to enhance the electrical conductivity of PC [17]. Choi et al. prepared poly(styrene-co-acrylonitrile) grafted MWCNTs by an atom transfer polymerization technique, and then PC/MWCNT composites were prepared by melt mixing [15].

Even though PC composites containing well-dispersed MWCNTs and exhibiting improved mechanical and electrical properties can be produced by modification of MWCNTs with functional groups, a new, simple route is still required to improve mechanical and electrical properties of PC composites to commercially produce PC/MWCNT composites. In this study, PC composites with PC grafted MWCNTs (PC-g-MWCNTs) were fabricated from PC and MWCNTs functionalized with hydroxyl groups (MWCNTs-OH) by reactive extrusion. It is expected that a grafting reaction will take place between the carbonate groups of PC and hydroxyl groups of MWCNTs-OH during melt extrusion. Formation of PC-g-MWCNTs was therefore conducted by melt extrusion, and the interfacial adhesion energies between PC and MWCNTs and the mechanical and electrical properties of PC/MWCNT composites were explored.

2. Materials and procedure

2.1. Materials

Commercially-available PC (grade: LUPOY GP-1000LP) was supplied by LG Chemicals (Seoul, Korea). According to the supplier, the molecular weight as determined by gel permeation chromatography (GPC) using polystyrene standards was $\bar{M}_w = 31,000$ g/mol ($\bar{M}_n = 14,000$ g/mol). MWCNTs (grade: CM250) grown by chemical vapor deposition were purchased from Hanwha Nanotech Co. (Seoul, Korea). The diameter of the MWCNTs was 10–15 nm, and the average bundle length and the average length of MWCNTs were 100 μm and 1.6 μm , respectively. Hydrogen peroxide (H_2O_2), used as a coupling agent to form hydroxyl groups on the MWCNT surface, was purchased from Aldrich Chemicals (Milwaukee, WI, USA). The tetrahydrofuran (THF) used as a PC solvent was also purchased from Aldrich chemicals. PC was dehydrated in a vacuum at 100 $^\circ\text{C}$ for 24 h before use.

2.2. Preparation and characterization of PC-g-MWCNT

The synthetic procedure for forming PC-g-MWCNTs is shown in Fig. 1. To produce MWCNTs functionalized with hydroxyl groups, pristine MWCNTs were reacted in a H_2O_2 solution, as shown in Fig. 1 [29–31]. The pristine MWCNTs (0.5 g) were dispersed in an

aqueous solution of H_2O_2 (concentration: 30 vol.%, 500 ml) under sonication at 30 $^\circ\text{C}$. After reacting at 60 $^\circ\text{C}$ for 24 h, the resulting mixture was diluted with deionized water (1000 ml) and then filtered through a nylon membrane (pore size: 450 nm). The resulting MWCNTs were washed with deionized water (5×200 ml) and dried for 24 h in a vacuum oven at room temperature (hereafter referred to as “MWCNT-OH”).

PC/PC-g-MWCNT composites were prepared from PC and MWCNTs-OH by reactive extrusion in a twin extruder (BA-11, L/D ratio = 40, Bau Technology, Seoul, Korea). The temperatures of the feeding zone, melting zone, mixing zone, and exit die of the twin extruder were 270, 280, 290, and 300 $^\circ\text{C}$, respectively. The material feed rate and the extrusion speed were held constant at 10 g/min and 300 rpm, respectively. Melt-mixed composites were immediately quenched in a water bath after extrusion. To obtain PC-g-MWCNTs formed by a reaction between the hydroxyl groups on MWCNTs and the carbonate groups in PC during extrusion and to remove the unreacted PC, the composite (3 g) was dissolved in THF (200 ml), and then PC-g-MWCNTs were collected using a centrifuge. The collected PC-g-MWCNTs were again dispersed in THF (200 ml), and PC-g-MWCNTs were again collected using a centrifuge. This procedure was repeated five times to remove unreacted PC. The resulting product was dried for 12 h in a vacuum oven at 100 $^\circ\text{C}$.

The molecular structure of PC-g-MWCNTs was confirmed by Fourier transform infrared (FT-IR, Magna 750, Nicolet, WI, USA) analyses and X-ray photoelectron spectroscopy (XPS, VG Microtech, ESCA2000, UK) using a spectrometer with a Mg K α X-ray source (1253.6 eV) and a hemispherical analyzer. IR spectra were collected over 30 scans in the 4000–500 cm^{-1} region using attenuated total reflection (ATR) mode at a resolution of 4 cm^{-1} . The XPS spectra were obtained in high-resolution mode with a 20 eV pass energy and a 0.1 eV step size. All binding energies were calibrated to carbon (C1s) at 284.5 eV. For curve fitting, the widths of the Gaussian peaks were kept constant in each spectrum. Field emission scanning electron microscopy (FE-SEM, model: Sigma, Carl Zeiss, Germany) and high resolution transmission electron microscopy (HR-TEM, model: JEM 2000EXII, JEOL, Japan) were employed to investigate the morphologies of the pristine MWNTs, MWCNT-OH, PC-g-MWCNT and composites. Thermogravimetric analysis (TGA, model: TGA-2050, TA Instruments, USA) of the samples was carried out to determine the amount of PC grafted on the MWCNTs. TGA analyses were performed under nitrogen at a heating rate of 10 $^\circ\text{C}/\text{min}$. The specimens for the TGA experiments were dried in a vacuum oven at 80 $^\circ\text{C}$ for 1 day.

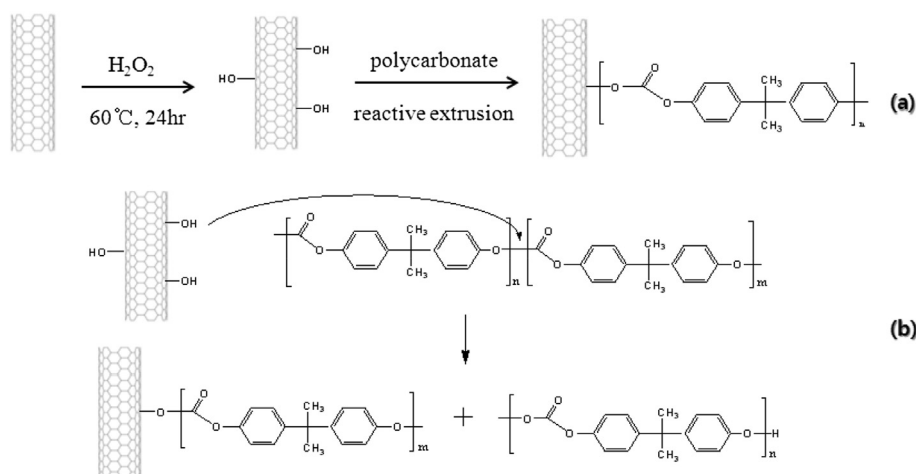


Fig. 1. (a) Synthetic route for PC-g-MWCNT by reactive extrusion and (b) reaction mechanism of MWCNT-OH and PC.

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