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Electrically conductive and strong cellulose-based composite fibers reinforced with multiwalled carbon nanotube containing multiple hydrogen bonding moiety

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ABSTRACT

Highly conductive and strong composite fibers based on cellulose and multiwalled carbon nanotube with multiple hydrogen bonding moiety (MWCNT-MHB) were manufactured via a facile organic solvent-based solution spinning. The microstructure, molecular interaction, mechanical and electrical properties of the composite fibers were investigated as a function of MWCNT-MHB content (0.5–30.0 wt%). The FT-IR spectra of the composite fibers revealed the existence of the specific interaction between cellulose backbone and MWCNT-MHB via hydrogen bonding. The MWCNT-MHB was dispersed uniformly in the cellulose fiber matrix, which was confirmed by SEM images of the cross-section and longitudinal surfaces for the composite fibers. Accordingly, the composite fibers with different MWCNT-MHB contents exhibited high performance in tensile mechanical properties such as tensile strength of 156–278 MPa, failure strain of 3.7–7.0%, and initial modulus of 11.3–13.9 GPa. In addition, the electrical conductivity of the composite fibers increased significantly from ~10^{-8} S/cm to ~10^{0} S/cm with the increase of the MWCNT-MHB loading from 0.5 wt% to 30.0 wt%, particularly at a certain MWCNT content between 0.5 and 1.0 wt%. The composite fiber with 30.0 wt% MWCNT-MHB, which has high electrical conductivity of 2.7 S/cm and tensile modulus of ~12.5 GPa, could be thus used for electrically conductive wire and electric heating element for smart textiles.

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1. Introduction

Owing to the growing environmental concerns over reliable availability of petroleum-based polymeric materials, the industrial and academic efforts have been recently directed to design and produce fibers, films, plastics, and composites based on renewable natural resources [1–3]. In addition, the advances toward electronic materials and devices based on green polymer materials and composites have been evident by achieving new promising concept to optoelectronics, smart textiles, photovoltaic cells, energy storage devices, etc [4–6].

As the most well-known renewable natural resource, cellulose, a long chain polymer of linearly linked sugar molecules, is the main component of plant cell walls, and the basic building material for textile fibers, papers, and composites for diverse industrial applications [7–9]. The polar hydroxyl groups of the cellulose chain form many hydrogen bonds with other hydroxyl groups on adjacent chains, bundling the chains together. The chains also pack regularly in places to form hard, stable crystalline regions that give the bundled chains even more stability and strength. To expand the applications of natural cellulose to advanced fields including electrostatic dissipation/electromagnetic interference shielding materials [10,11], supercapacitors [12], actuators [13], and sensors [14,15], carbon nanotubes (CNTs) have been widely adopted as one-dimensional reinforcing nanofillers for cellulose-based composite films and papers, due to their outstanding carrier mobility of 1000–4000 cm^2/V, Young's modulus of 0.27–1.25 TPa, electrical conductivity of 10^4–10^5 S/cm, thermal conductivity of 3000–6600 W/mK, and thermal stability up to ~700 °C in air [16,17]. CNT/cellulose composite fibers have been also manufactured, and their mechanical and electrical

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properties have been investigated [8,18]. Zhang et al. reported that multiwalled carbon nanotube (MWCNT)-reinforced regenerated cellulose composite fibers, which were prepared with ionic acid and dry-jet wet-spinning, have enhanced mechanical properties and thermal stability owing to the good interaction between cellulose and MWCNTs [8]. The composite fiber containing 4 wt% MWCNT exhibited the highest electrical conductivity of 8.3×10^{-3} S/cm. However, for the composite fibers containing >4 wt% MWCNT, higher electrical conductivity could not be attained due to the alignment and severe aggregation of MWCNTs. Lu et al. also manufactured cellulose/MWCNT composite fibers under different draw ratio by dry-wet spinning, and investigated their structure and properties [18]. It was found that more MWCNT content and lower draw ratio could improve the electrical conductance of the composite fiber and that the composite fiber containing 5 wt% MWCNT had a volume conductivity of 8.8×10^{-4} S/cm. However, the electrical conductivity of the cellulose composite fibers in above literature was not high enough to be applied for the eco-friendly smart textiles including conductive wires and electric heating elements. On the other hand, Han et al. have recently manufactured dispersant-free conducting pastes based on organic solvents with CNTs and graphene nanoplatelets, which are chemically modified to have quadruple hydrogen bonding motif [19]. By using MWCNT containing quadruple hydrogen bonding sites as reinforcing nanomaterials, highly electrically conducting poly(vinyl alcohol)-based composite fibers could be obtained [20].

In the present study, we have manufactured a series of cellulose-based composite fibers reinforced with MWCNT containing multiple hydrogen bonding moiety (MWCNT-MHB) via a facile and efficient wet-spinning process using organic solvent system of dimethylacetamide and lithium chloride (DMAc/LiCl), and have investigated their molecular interaction, microstructures, mechanical and electrical properties. The potential availability of the MWCNT-MHB/cellulose composite fibers as electrically conductive wires and electric heating elements for smart textiles have been also discussed.

2. Experimental details

2.1. Materials

Cellulose powder with particle size of 50 μm (Type 50, crystallinity of ~91%) was supplied from Sigma–Aldrich., Ltd. Pristine MWCNT (CM-250, diameter of 10–15 nm, length of ~100 μm) was purchased from Hanwha Chemical Co., Ltd. N,N-dimethylacetamide (DMAc, 99.5%, Samchun Pure Chemical Co., Ltd.) and lithium chloride (LiCl, anhydrous, 98.2%, Samchun Pure Chemical Co., Ltd.) were used for the dissolution of the cellulose powder. 2-Amino-4-hydroxy-6-methylpyrimidine, hexamethylene diisocyanate and 1,1,1,3,3,3-hexafluoro-2-propanol were purchased from Sigma–Aldrich Co. All the materials and chemicals were used as received.

2.2. Preparation of MWCNT-MHB

To manufacture MWCNT-MHB, carboxylic acid-functionalized MWCNTs (MWCNT-COOH) were obtained by treating the pristine MWCNTs with a mixture of sulfuric acid and nitric acid (3:1 by volume fraction) at 50 °C for 24 h. After that, MWCNT-COOHs were filtrated and washed by distilled water, which was repeated until pH 7. MWCNT-COOHs were then dried in a vacuum oven at 70 °C for 24 h. A solution of 2-amino-4-hydroxy-6-methylpyrimidine (1 eq) in hexamethylene diisocyanates (10 eq) was heated at 100 °C in a N_2 atmosphere. After 16 h, the reaction mixture was precipitated using n-hexane. Filtration and washing of the residue with n-hexane and subsequent drying of the solid gave the pure product. A solution of MWCNT-COOH in DMF was dispersed by bath sonication. Hexyl-NCO was added to the solution at 100 °C under N_2 atmosphere. The weight ratio of the MWCNT-COOH and hexyl-NCO was 1:0.25. After 18 h, the reaction mixture was filtrated and washed by DMF and chloroform. Then, MWCNT-MHBs were dried in a vacuum oven at 70 °C for 24 h.

2.3. Preparation of the composite fibers

Regenerated cellulose/MWCNT-MHB composite fibers were manufactured by a facile wet-spinning, as can be seen in the schematic illustration of Fig. 1. First, a transparent cellulose solution was prepared by dissolving cellulose powder (5.0 wt%) into DMAc/LiCl (86/9 by weight ratio) and heating at 110 °C for 24 h. Second, predetermined amounts of MWCNT-MHB (0.0–30.0 wt% of cellulose powder) were added in DMAc to obtain 2.5–3.0 wt% solutions. Third, each MWCNT-MHB solution was mixed with cellulose solution and stirred for 30 min at 50 °C. Subsequently, the mixed solutions were sonicated for 2 h with a bath-type ultrasonicator (50–60 Hz), stirred for 40 h at room temperature, and sonicated again for 1 h. Forth, the cellulose/MWCNT-MHB solution dopes were spun into a water bath at room temperature at a speed of 0.3 mL/min through a spinneret needle with an inner diameter of 0.41 mm. The take-up speed of wet-spun fibers was controlled to be ~125 cm/min. After wet-spinning and take-up, the composite fibers were dried at 60 °C for 24 h. Finally, the cellulose/MWCNT-MHB composite fibers were immersed in a water bath for 12 h and then dried in a vacuum oven at 70 °C for 12 h to remove any residual solvents remaining in the fibers. The MWCNT-MHB contents in the composite fibers were controlled to be 0.5, 1.0, 3.0, 5.0, 10.0, 20.0, and 30.0 wt%. The final composite fibers were thus named as MM/C-x, where x denotes the MWCNT-MHB content in the composite fibers by weight percent (wt%). For comparison, a neat cellulose fiber was also prepared at the same procedure.

2.4. Characterization

The structural features of the pristine MWCNT and MWCNT-MHB were investigated by a high resolution Raman Spectrometer (LabRAM HR-800 UV–Visible–NIR, Horiba Jobin Yvon). The molecular structure of the neat cellulose and its composite fibers with different MWCNT-MHB contents was identified by using a microscope infrared spectrometer (iN10, Nicolet). The dispersion state of the MWCNT-MHB in the composite fibers was characterized with aid of a cold type field emission scanning electron microscope (SEM, S-4800, Hitachi). The tensile mechanical properties of the composite fibers were examined by using a universal tensile machine (INSTRON, 4467) with an extension rate of 8 mm/min at room temperature. For each fiber sample, five different tests were carried out and the results were averaged. The electrical properties of the composite fibers were investigated by obtaining current–voltage (I–V) and electric power-voltage (P–V) curves with multiple electrometers (2400, 6517B, Keithley Instruments Inc.). For the electrical experiments, the electrode distance of the composite fibers was set to be 10.0 mm. The electric heating behavior of the composite fibers was characterized with an infrared camera (SE/A325, FLIR Systems) and a sourcemeter (2400, Keithley Instruments Inc.). The diameter of the composite fibers used for the mechanical and electrical tests was in the range of 93 ± 4.4 μm.

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