

Carbon nanofiber/polyethylene nanocomposite: Processing behavior, microstructure and electrical properties



Mohammed H. Al-Saleh^{a,*}, Genaro A. Gelves^b, Uttandaraman Sundararaj^b

^a Department of Chemical Engineering, Jordan University of Science and Technology, P.O. Box 3030, Irbid 22110, Jordan

^b Department of Chemical and Petroleum Engineering, Schulich School of Engineering, University of Calgary, 2500 University Drive N.W., Calgary, Alberta, Canada T2N 1N4

ARTICLE INFO

Article history:

Received 24 March 2013

Accepted 13 May 2013

Available online 28 May 2013

Keywords:

Composite material

Microstructure

Polymers

Electrical conductivity

ABSTRACT

Electrically conductive polymer nanocomposite of high density polyethylene (HDPE) filled with carbon nanofibers (CNFs) were prepared by melt compounding in a batch mixer. The nanocomposite processing behavior was studied by monitoring the mixing torque vs. time as function of filler content. Scanning electron microscopy and optical microscopy were used to investigate the nanocomposite dispersion of nanofiller and the adhesion between the nanofiller and polymer matrix. The electrical and electromagnetic interference (EMI) shielding behaviors of the nanocomposite were reported as function of nanofillers concentration, and an empirical correlation related the EMI SE to the nanocomposite's electrical resistivity was developed. Good level of CNF dispersion was evident despite the poor adhesion exhibited between the nanofibers and the HDPE matrix. At 1.5 vol% CNF loading, the nanocomposite exhibited an electrical volume resistivity of $10^5 \Omega\cdot\text{cm}$. EMI shielding effectiveness was found to increase with increase in nanofiller concentration. In the 0.1–1.5 GHz frequency range, 2 mm thick plate made of 5 vol% CNF/HDPE nanocomposite exhibits an EMI shielding effectiveness of 20 dB.

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1. Introduction

Electrically conductive polymer composites are typically produced by blending conductive fillers with insulating polymer matrices. A critical concentration of filler known as the electrical percolation threshold is needed to create conductive networks within the polymer matrix. At this critical concentration, the composite conductivity increases by several orders of magnitude. Conductive composite materials have several applications such as protection of electronics from electrostatic discharge (ESD) and preventing electromagnetic interference (EMI) to maintain functionality and compatibility of electronics [1–3]. The light weight, cost and adaptability to application needs are some of the advantages that conductive composites have over metals for EMI shielding applications.

ESD is a serious problem for electronic devices. Electronics are susceptible to ESD damage during manufacturing, assembling and transportation. In order to protect electronics from ESD, packaging materials should have a surface resistivity in range of 10^6 – $10^9 \Omega/\text{sq}$. In addition to protection from ESD, electronic devices should be shielded to prevent outgoing and incoming EMI. The shielding effectiveness (SE) of a material is the ratio of the incident power to the transmitted power. It is usually expressed in decibels (dB) according to the following equation:

$$SE = 10 \log \frac{P_1}{P_2} \quad (1)$$

where P_1 and P_2 are the incident power and transmitted power, respectively. Shielding effectiveness of 20 and 30 dB means that 99 and 99.9%, respectively, of the EM waves have been attenuated. For most applications a SE greater than 20 dB is required. For automotive and computer industries, 30 dB is considered an adequate SE for 50% of the applications [4].

Many options are available for shielding of EMI, including: metal sheets, conductive paints, electro-less plating and CPCs. For example, film/foil laminate and metal liners provide good shielding at lower cost but they have limited design flexibility [5]. Metal coated or plated polymers are widely used for EMI shielding. However, conductive coatings can delaminate, the coating requires several expensive steps of fabrication, and coated materials are very difficult to recycle. On the other hand, conductive composites have greater design flexibility than coatings, but they suffer from the very high filler loadings required to bring the composite material to competitive shielding level. Thus, a composite with low filler loading is required to overcome this problem. Technically, this is possible by using filler with high electrical conductivity and high aspect ratio.

Conductive nanostructured polymeric materials are promising alternatives for the currently used technologies for ESD and EMI. Because of their high aspect ratio and intrinsic conductivity, conductive nanofillers can create nanocomposites for ESD and EMI applications at very low filler loading. The focus in this paper is

* Corresponding author. Tel.: +962 2 7201000x22415; fax: +962 2 7204074.

E-mail address: mhsaleh@just.edu.jo (M.H. Al-Saleh).

on carbon nanofiber (CNF) based nanocomposites. Previous work showed that CNF can enhance electrical, mechanical, and thermal properties of polymer matrix nanocomposites at relatively low loadings [6–8]. For example, only 0.5 vol% CNF was required to achieve electrical percolation threshold in polypropylene nanocomposite [9]. Adding 1 wt% CNF enhanced the thermal conductivity of CNF/epoxy nanocomposite by 45% [10]. Thermal stability of many polymers including PE [11], epoxy [12], PP [13,14], PS [15], PC [16,17], and PMMA [18,19] were improved by CNF addition. Addition of 8 vol% CNF was reported to enhance Young's modulus and tensile strength of neat PP by 100% and 80%, respectively [20]. In this work, CNF/high density polyethylene (HDPE) nanocomposites were prepared by melt compounding in a laboratory batch mixer. The study aims to investigate the processing behavior, microstructure, electrical percolation and EMI shielding properties of the nanocomposites as function of CNF concentration. The processing behavior was studied by analyzing the mixer torque vs. time curves as function of CNF addition and concentration. Scanning electron microscopy and optical microscopy were used to study the microstructure of the obtained nanocomposites in terms of adhesion between the nanofiller and polymer matrix and level of nanofiller dispersion. The electrical percolation and EMI SE behaviors were discussed and the relation between EMI SE and electrical conductivity was investigated.

2. Experimental details

2.1. Materials and procedure

The carbon nanofiller used in this work is low heat treated (LHT)-vapor grown carbon nanofiber (VGCNF) (Pyrograf III™ Applied Sciences, Inc., OH-USA). LHT-VGCNF is about 4.2 μm in length and 110 nm in diameter [21]. The polyethylene (HDPE-19G, MFI of 1.2 g/10 min (190 °C/2.16 kg), specific gravity of 0.962) was kindly provided by Nova Chemicals. The CNF/HDPE nanocomposites were prepared by melt compounding in a Haake internal batch mixer (Haake Rheomix series 600 batch mixer, Thermo Scientific, Germany). Prior to compounding, the HDPE pellets and CNF powder were placed in a vacuum oven at 70 °C and 130 °C, respectively for about 16 h. The melt mixing conditions were: mixing speed 50 rpm, mixing temperature 180 °C and mixing time 9 min (3 min pure polymer melting, 2 min CNF feeding and 4 min additional mixing). Circular disks for electrical resistivity and EMI SE measurements, were prepared by compression molding using Carver compression molder (Carver Inc., Wabash-IN, USA) and had dimensions of 2.0 mm thickness and 133 mm diameter. The compression molding conditions were as follows: temperature 200 °C, time 4 min and pressure 18.5 MPa. For each formulation, at least two specimens were prepared and characterized.

2.2. Characterization

2.2.1. Morphology

Scanning electron microscopy (SEM) and optical microscopy (OM) were used to investigate the structure of the nanocomposites. For SEM characterization, the nanocomposites were examined using JEOL 6301F (Field Emission Scanning Electron Microscope). SEM specimens were fractured in liquid nitrogen and coated with gold prior to imaging. For the optical microscopy characterization, ZEISS AXIO Scope A1 microscope was used. The OM specimens were thin sections of about 1 μm cut at –80 °C using Leica cryoultramicrotome (EM UC6/FC6).

2.2.2. Electrical resistivity measurements

Two different set-ups were used to measure the electrical resistivity of the CNF/HDPE nanocomposites. For samples with electrical resistivity higher than $10^6 \Omega\cdot\text{cm}$, the characterization was conducted according to ASTM: D257 standard using a Keithley 6517A electrometer connected to Keithley 8009 test fixture (Keithley Instruments, USA). For samples with lower electrical resistivity, measurements were conducted using a Loresta GP resistivity meter (MCP-T610 model, Mitsubishi Chemical Co., Japan) connected with a four-pin probe (MCP-TP08P model, Mitsubishi Chemical Co., Japan). The inter-pin spacing is 5 mm and the pin diameter is 2 mm.

2.2.3. Shielding effectiveness

EMI SE of the CNF/HDPE nanocomposites was conducted according to the ASTM: D4935-99 standard for planar materials. The set-up consists of RF network analyzer (HP 8752C, Hewlett-Packard, USA), EMI shielding test fixture (EM-2017A, Electro-Metrics Corp, Johnstown-NY, USA), two coaxial cables and two 10 dB attenuators. The set-up dynamic range is 80 dB. More information about the set-up can be found elsewhere [22]. For each formulation, a minimum of two load specimens were tested in the frequency range of 0.1–1.5 GHz. The input power used for all tests was 1 mW.

3. Results and discussion

3.1. Processing and microstructure

Fig. 1 depicts the mixing torque vs. time curves of CNF/HDPE nanocomposites as function of filler content. Three major processing zones can be observed in the figure. The first zone, from $t = 0$ min to $t = 3.0$ min, is the pure polymer melting zone. The second zone, from $t = 3.0$ min to $t = 5.0$ min, is the CNF feeding zone. In this operation window, CNF was gradually fed to the mixture in order to avoid any sudden increase in torque and to avoid overflow problems especially for nanocomposites with high nanofiller content. The last zone, from $t = 5.0$ to $t = 9.0$ min, is the nanocomposite compounding zone. The increase in mixing torque with the increase in CNF loading is evident. However, this level of increase is insignificant. For example, at the end of compounding, the mixing torque of the 5 vol% and 10 vol% nanocomposites were 10.7 N·m and 13.4 N·m, respectively. Moreover, for the 1 vol% nanocomposite, the addition of nanofiller has negligible effect on the mixing torque. For this nanocomposite the recorded mixing torque was 9.5 N·m before the addition of CNF and 9.4 N·m at the end of the compounding process.

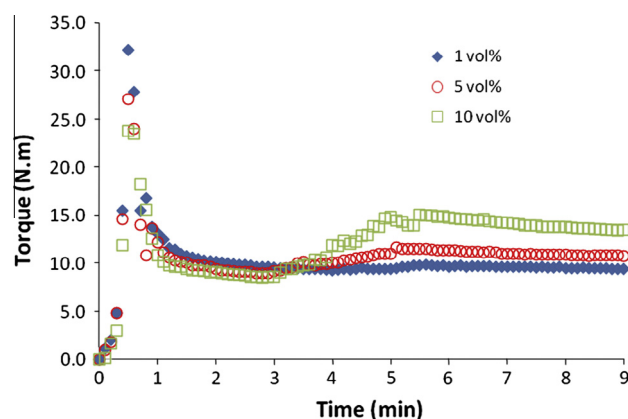


Fig. 1. Processing behavior curves of CNF/HDPE nanocomposites.

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