Effect of Carbon Nanotube and Surfactant on Processing, Mechanical, Electrical and EMI-Shielding of Epoxy Composites

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A B S T R A C T

Dispersing nanoparticles in a polymer matrix is intrinsically challenging due to unfavorable entropic interactions between the matrix and the nanoparticle. In this research dispersion of nanoparticles in polymer matrix was studied and the effect of dispersion on properties was investigated. The properties of polymer composite depend on the type, size, shape, concentration of nanoparticles, and interactions of polymer-nanaoparticle. The lack of compatibility between inorganic particles and polymer matrix limits the applications of produced nanocomposite. Due to incompatibility, the dispersion of nanoparticles in polymer matrix is very difficult. The epoxy/MWCNT composite was fabricated by solution mixing process. The effect of MWCNT and Amine-Functionalized surfactant, on curing process, EMI-shielding, mechanical and electrical properties of nanocomposite were investigated. Dispersion of MWCNT in solution was investigated using UV-vis spectroscopy. Curing behavior of nanocomposites at different MWCNT loadings was investigated. EMI-shielding of nanocomposite at different concentrations of MWCNT was studied and the results showed that MWCNT and ferric oxide improve absorption loss, reflection loss and shielding effectiveness. Effect of MWCNT inclusion on thermal and mechanical properties of nanocomposites was investigated. Finally, the electrical conductivity was measured using a two-point conductivity test method.

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1. INTRODUCTION

Polymer nanocomposites due to their properties have attracted tremendous attention in research centers and market [1, 2]. These materials have become important because of their wide range of applications in various fields such as lightweight constructions, especially in aircraft and wind energy industry, automotive parts, packaging, adhesives and coatings [3, 4]. Nanocomposites based on carbon nanotubes (CNTs) have received great attention due to the superior set of properties [4, 5]. Multiwall Carbon Nanotube (MWCNT) is multiple rolled up graphene sheets with diameters of almost 30 nm, have excellent thermal, and mechanical properties, as well as chemical stability. The incorporation of CNT into polymer matrix can significantly improve the properties [6-8]. CNT/epoxy

nanocomposites represent a class of materials with tailored properties including electrical conductivity, mechanical permanence, etc. [9]. The introduction of inorganic nanoparticles into organic matrix offers an effective way to improve properties such as electrical conductivity, mechanical properties, and etc. The properties of final composite depend on size, shape, concentration, and interactions of nanoparticle with the polymer matrix.

The dispersion of MWCNT in polymer matrixes is the biggest challenges for the development of polymer/CNTs composites [10]. For many industrial applications, a uniform and stable dispersion of filler are necessary [11]. The surface modification of CNT with hydrophilic or hydrophobic is an effective way to enhance the CNTs dispersion without interfering CNT properties. The surfactants carrying hydrophilic or hydrophobic moieties can effectively disperse CNTs in aqueous or organic media by forming specific

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directional π–π stacking interactions with the graphitic surfaces of nanotubes. Since small molecules or polymers can be adsorbed onto CNT surfaces by π–π interactions, the conjugated polymers [12-16] have been considered as a modifier for MWCNT surfaces [17]. The strong π–π interactions between MWCNT and conjugated polymers may drive to wrapping the polymer chain around particles [18, 19]. Surfactants adsorbed on graphite may form hemimicelle structures that sheath the nanotube surface.

Electromagnetic interference (EMI) shielding of radio frequency radiation continues to be a serious concern for humankind because of hazardous effect on human health and damages to sensitive electronic devices. Because electromagnetic radiation, particularly at high frequencies (e.g. radio waves, such as those emanating from cellular phones), interfere with electronic circuits (e.g. computers), EMI shielding has attracted many attentions in recent years [20, 21]. CNT-Epoxy nanocomposites are regarded as an materials with EMI-shielding properties. The small diameter, high aspect ratio, high conductivity, and mechanical strength of CNTs, including SWNTs and MWCNTs, make them an excellent candidate for the production of conductive composites for high-performance EMI shielding materials [20, 21]. In this paper, the effect of dispersing agent, sonication time, particle and resin concentration on the dispersion of MWCNT in epoxy resin and curing behavior was investigated. The effects of MWCNT and ferric oxide concentration on electrical conductivity and EMI-shielding properties were also investigated.

2. EXPERIMENTAL

2. 1. Materials and Equipment   The epoxy resin, Epon 828 (Shell) was used as the matrix. Xylene (Merck) was used as the solvent and Amine hardener (Keramide 115) at a weight ratio of (1:1) was used as the curing agent. The hydroxyl functionalized multi-walled carbon nanotubes, abbreviated as MWCNT-OH with the diameter of 5-10 nm, the length of 30 μm and purity of >95% purchased from Nanotech, China. DISPERBYK-163 (BYK Additives & Instruments) was used as the dispersing agent. Sonication processes were carried out with a horn sonicator at 70% power amplitude, a 0.5-second pulse on and 0.5-second pulse off.

The ultrasonication of the MWCNT mixtures was performed using probe KE76 (Bandelin HD 3200 processor Germany) at different sonication times. The MWCNT dispersion was characterized in the visible wavelength range of 300-700 nm using a UV-vis spectrophotometer (UV-1650 PC, Shimadzu, Japan). Samples were taken regularly during the ultrasonication process and carefully diluted for 15 times with xylene. The baseline correction was equilibrated using a pure

solution of dispersant. The electrical resistivity of nanocomposite samples was measured using a standard two-point test method (Keithley electrometer 610c, USA) at room temperature. The samples were prepared by curing nanocomposite at room temperature for 48 hours. The curing behavior of the MWCNTs/epoxy nanocomposites was determined using a differential scanning calorimeter (Netzsch DSC 200 F3, Germany) under a nitrogen atmosphere. The DSC curves were obtained at the heating rate from 50°C to 200°C at 10°C/min. The samples containing 0.1, 1 and 10 wt% of CNT were placed in DSC pans immediately after proper mixing with hardener.

2. 2 Preparation of the MWCNT/Epoxy Nanocomposites   0.1 g MWCNT was dissolved in 50 mL xylene, and the molar ratio of dispersant to MWCNT was chosen 1:1. The prepared solution was stirred for 30 min at 300 rpm and then sonicated at different sonication times under mild condition. The flask was placed in an ice water bath during sonication in order to stabilize the temperature. Using shear mixing of 100 RPM for one hour at room temperature MWCNT dispersion was incorporated into the epoxy resin. The organic solvent and the bubbles were removed using a vacuum. In order to study the effect of nanoparticles, different samples were prepared by the inclusion of 0.5, 1, 2, 3 and 4 wt% of MWCNT. The curing agent was added to an equal weight percent with resin and stirred to prepare a homogenous mixture. Final samples were taken into a Teflon pan at 80℃ for 45 min to complete the curing reactions. For the samples that were prepared without dispersing agent, the MWCNT dispersion was immediately mixed with epoxy resin for 10 min in a sonication bath. The sample sheets were prepared after curing nanocomposite at room temperature for 48 hours.

3. RESULTS AND DISCUSSION

3. 1. The Effect of Surfactant Concentration on Dispersion Stability of MWCNT   Figure 1 shows the UV-vis spectroscopy of dispersant-treated MWCNT dispersions at different sonication times. The results of UV visible spectra show the effective dispersion of MWCNTs at longer sonication times [22]. As can be seen, as sonication time increases from 2 to 20 min, absorption enhanced correspondingly. The UV-visible spectra showed maximum absorption at 300 nm following a steady reduction as wavelengths increase. The longer ultra-sonication time may dissociate MWCNT bundles with possible breakage or damage. The MWCNT breakage leads to a shorter length of the nanotubes, structure damaging and hence reduction of important properties of nanotube such as electrical properties [23].
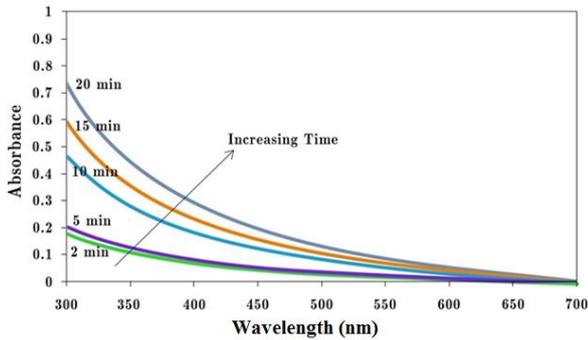
Figure 1. UV-vis spectra of dispersant-treated MWCNT dispersions in different sonication time

3. 2. Effect of MWCNT on Curing Process   Effect of MWCNT on curing behavior of epoxy resin was studied by DSC analysis of epoxy resins containing different concentrations of MWCNT. As shown in Figure 2, all samples show an exothermic peak near 125°C. As MWCNT concentration increases, the heat flow amount increases. The onset temperature (Ti), peak temperature (Tp) and the heat of curing (ΔH) for pristine epoxy resin (sample A), an epoxy resin containing 0.1 wt% MWCNT (sample B) and an epoxy resin containing 1 wt% MWCNT (sample C) are tabulated in Table 1. Figure 2 represents as MWCNT concentration increases Ti and Tp decrease. Generally CNT has a higher amount of heat capacity compared with polymer and due to this reason, increasing CNT amount shifts heat flow peak to higher values. With the increase of the MWCNT content, the initial curing temperature, exothermic peak temperature and heat of curing first increase and subsequently decrease. The initial increase of the MWCNT content results in the increase of the activation energy in the cure process. Due to the high thermal conductivity of CNTs, the curing heat transferred rapidly to different parts of the sample resulting in increase in reaction rate and faster curing initiation.
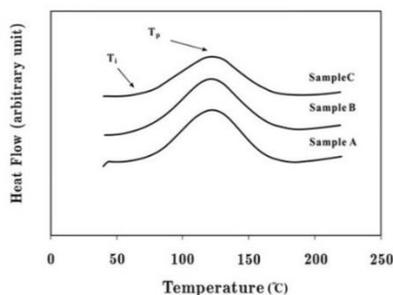
At higher concentrations of MWCNT 1wt%, the amine groups on the surface of MWCNTs play the role of curing agents and promote the primary amine-epoxide reaction. When the MWCNT content continuously increases, these amine groups will efficiently impair the retardation effect on the cure reactions caused by MWCNTs. Accordingly, the activation energy of the studied systems decreases [24].

3. 3 Effect of MWCNT and Ferric Oxide on EMI Shielding   EMI shielding was measured using Vector Network Analyzer in the range of 8-12 (GHz). Ferric oxide (Fe_3O_4) with 30nm particle size was used to increase magnetic properties of Epoxy/MWCNT nanocomposites. Ferric oxide improves EMI shielding effect due to high magnetic permeability properties.

Figure 3 represents the absorption loss of electromagnetic waves for MWCNT composites. As can be seen, the inclusion of 4 wt% MWCNT into the epoxy resin increases absorption loss in the range of 11-12 GHz. Ferric oxide further increases the absorption loss. As shown in Figure 3, the inclusion of 40 wt% and 60 wt% ferric oxide in system respectively increases the absorption loss 3 times and 5 times at 12 GHz compared with pristine epoxy. This effect is attributed to the magnetic and dielectric absorption capacity of ferric oxide and MWCNT. Electromagnetic waves lose their energy by collision to magnetic nanoparticles in the composite.

The reflection loss of electromagnetic waves for MWCNT composites is shown in Figure 7.

TABLE 1. The initial curing temperature (Ti), the peak temperature (Tp) and the heat of curing for the samples A, B and C

Sample	T_i (°C)	T_p (°C)	ΔH (J/mol)
A	76.7	122.5	134.8
B	73.1	121.8	145.4
C	73.1	120.2	1135.7

Figure 2. DSC thermograms for (A) epoxy resin (B) epoxy resin containing 0.1 wt% MWCNT and (C) epoxy resin containing 1 wt% MWCNT
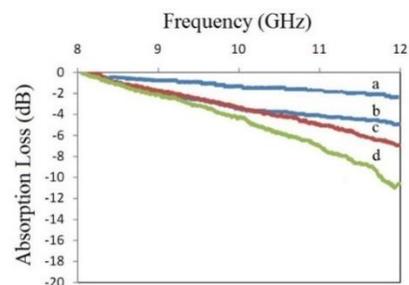

Figure 3. Effect of MWCNT and Ferric oxide inclusion on absorption loss a) Pristine epoxy nanocomposite, b) 4 wt% of MWCNT, c) 4 wt% of MWCNT and 40 wt% Ferric oxide, d) 4 wt% of MWCNT and 60 wt% Ferric oxide

As can be seen, addition of 4 wt% MWCNT to the epoxy improves reflection loss considerably. MWCNT has high electrical conductivity and acts as an electrical circuit which traps electromagnetic energy and causes energy loss in the form of heat. The inclusion of Ferric oxide further improves reflection loss. As shown in Figure 4, addition of 40 and 60 wt% Ferric oxide to epoxy, respectively improves the reflection loss to -15 and -17 at a range of 11-12 GHz.

The shielding effectiveness of samples in the range of 8-12 GHz was investigated. Figure 5 represents, the inclusion of MWCNT and ferric oxide which improves shielding effectiveness. As can be seen in Figure 8 curve a, Pristine epoxy nanocomposite has the lowest shielding effectiveness. Addition of 4 wt% of MWCNT (Figure 5 curve b) improves shielding effectiveness considerably. Loading 40 wt% (Figure 5 curve c) improves shielding effectiveness more, and 60 wt% of ferric oxide (Figure 5 curve d) shows highest shielding effectiveness. This effect is attributed to the magnetic and dielectric absorption loss of ferric oxide and MWCNT in the system.
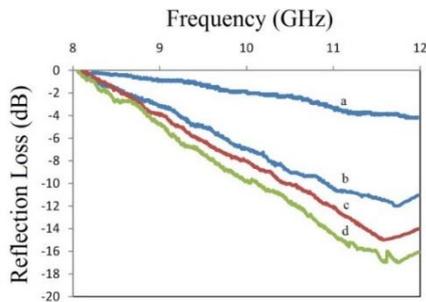
3. 4 Effect of MWCNT and Ferric Oxide on Mechanical Properties

Figure 6 represents the results from Dynamic Mechanical Thermal Analysis (DMTA) test for epoxy nanocomposites containing MWCNT and ferric oxide. As can be seen in Figure 6 addition of MWCNT improves the modulus. Increasing MWCNT concentration from 1 to 4 wt% improves modulus in both glass transition and a rubbery plateau region. As shown in Figure 7 loss factor peak from 28°C for reference sample changes to 35°C, 52°C and 63°C for samples containing 1 wt% MWCNT, 4 wt% MWCNT and 40 wt% ferric oxide, respectively. Nanoparticles have higher modulus compared with epoxy matrix and they usually improve the system modulus. Also, MWCNT and ferric oxides can improve modulus by increasing curing temperature. These particles due to high thermal conductivity, improve curing process. Increasing MWCNT concentration increases cross-link density and as a result, polymer chains movability decrease which reduces modulus loss in Figure 7. As shown, the inclusion of particles reduces the loss factor. Particles reduce the chains mobility and subsequently polymer chains cannot glide on each other upon applying tension. Therefore, it was shown that in the presence of MWCNT and ferric oxide, energy lost decrease and modulus increase.

Figure 4. Effect of MWCNT and Ferric oxide inclusion on reflection loss. a) A pristine epoxy nanocomposite, b) 4 wt% of MWCNT, c) 4 wt% of MWCNT and 40 wt% Ferric oxide, d) 4 wt% of MWCNT and 60 wt% Ferric oxide

Figure 6. Effect of MWCNT and Ferric oxide on Modulus, a) Pristine epoxy nanocomposite, b) 1 wt% of MWCNT, c) 4 wt% of MWCNT, d) 4 wt% of MWCNT and 40 wt% Ferric oxide
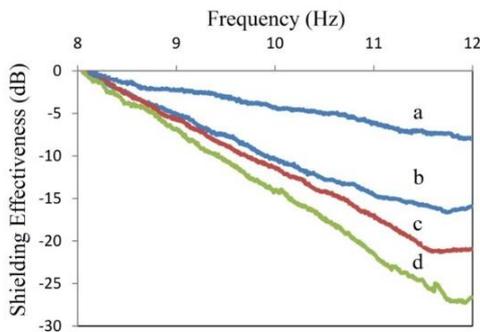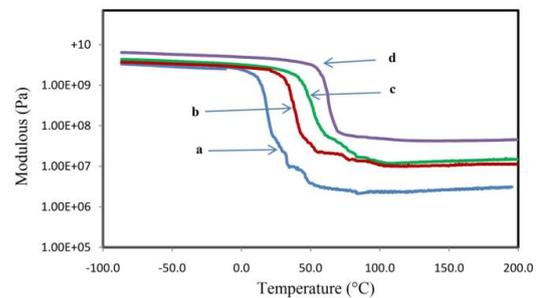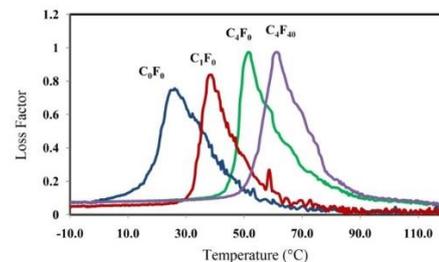

Figure 5. Effect of MWCNT and Ferric oxide inclusion on shielding effectiveness. a) A pristine epoxy nanocomposite, b) 4 wt% of MWCNT, c) 4 wt% of MWCNT and 40 wt% Ferric oxide, d) 4 wt% of MWCNT and 60 wt% Ferric oxide

Figure 7. Effect of MWCNT and Ferric oxide on loss factor, a) Pristine epoxy nanocomposite, b) 4 wt% of MWCNT, c) 4 wt% of MWCNT and 40 wt% Ferric oxide, d) 4 wt% of MWCNT and 60 wt% Ferric oxide

3. 5. Electrical Conductivity of MWCNT/Epoxy Nanocomposites   Figure 8 shows the effect of dispersant and MWCNT content on the electrical conductivity of MWCNT/epoxy nanocomposites. As shown, the electrical conductivity of samples improves as the MWCNT loading increases. The percolation threshold defines the concentration that the nanocomposites conduct the electrical conductivity. At the percolation threshold, the average interval between the particles decreases enough to conduct current and to start the electrical conductivity. In both sample series, the electrical percolation threshold was measured at about 0.5 wt% MWCNT loading. The lower electrical conductivity for the dispersant-treated MWCNT nanocomposite may be attributed to the adsorption and screening effect of dispersant. It may be concluded that the surfactant adsorbed on the MWCNT surfaces and hence cover the surface charges so that it leads to decreasing the conductivity [25, 26].
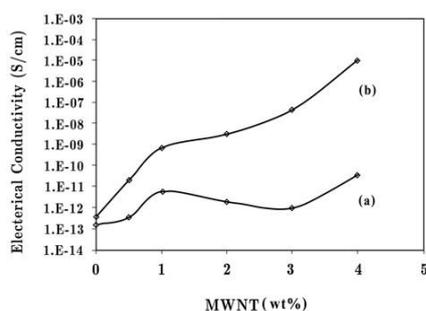

Figure 8. The variations of electrical conductivity of the MWCNT/epoxy nanocomposites (a) with dispersant-treated MWCNT and (b) without dispersant

4. CONCLUSIONS

The UV-vis spectroscopy was used to monitor the effect of surfactant concentration and sonication time on the dispersion of MWCNT. The visual inspection of MWCNT dispersion showed that combining the dispersant concentration and sonication time can improve nanotube dispersion and stability at least for 48 hours. The effect of epoxy resin concentration during subsequent high shear mixing on MWCNT dispersion was studied to get more effective MWCNT dispersion. The EMI-shielding of nanocomposite at different concentrations of MWCNT was studied, and the results showed MWCNT and ferric oxide improve absorption loss, reflection loss and shielding effectiveness greatly. Results of thermal and mechanical tests of nanocomposites showed as MWCNT and ferric oxide concentration increases, mechanical properties improved. Electrical conductivity tests showed that the electrical percolation threshold of the nanocomposites is about 0.5 wt% MWCNT loading. In comparison with

dispersant-treated MWCNT nanocomposites, the electrical conductivity was higher in MWCNT/epoxy nanocomposites without dispersant .

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چکیده

پراکنش نانوذرات در یک ماتریس پلیمری بدلیل برهمکنش های آنتروپیک نامطلوب بین ماتریس و نانوذرات یک کار به شدت چالش برانگیز است. در این تحقیق پراکنش نانوذرات در ماتریس پلیمری مطالعه شد و اثر پراکنش روی خواص کامپوزیت بررسی شد. خواص کامپوزیت پلیمری به نوع، سایز، شکل و غلظت نانوذرات و نیز به برهم کنش پلیمر-نانوذره بستگی دارد. عدم وجود سازگاری بین نانوذرات معدنی و ماتریس پلیمری کاربردهای نانوکامپوزیت تولیدشده را محدود میکند. به دلیل این عدم سازگاری، پراکنش نانوذرات در ماتریس پلیمری امری دشوار است. نانوکامپوزیت اپوکسی/نانولوله کربن چند دیواره با فرایند اختلاط محلولی تهیه شد. اثر نانولوله چند دیواره و سورفکتانت آمینی روی شرایط پخت، شیلدینگ الکترومغناطیسی، خواص مکانیکی و الکتریکی نانوکامپوزیت بررسی شد. پراکنش نانولوله کربن چند دیواره در محلول با استفاده از UV-vis spectroscopy بررسی شد. رفتار پخت نانوکامپوزیت در مقادیر غلظت مختلف نانولوله کربن چنددیواره مطالعه گردید. شیلدینگ الکترومغناطیسی نانوکامپوزیت در مقادیر غلظتهای مختلف نانولوله کربن چنددیواره مطالعه شد و نتایج نشان داد که نانولوله کربن چنددیواره و اکسیدآهن موجب افزایش اتلاف جذبی، اتلاف انعکاسی و ضریب تاثیر شیلدینگ میگردد. اثر نانولوله کربن چنددیواره روس خواص حرارتی، و مکانیکی نانوکامپوزیت مطالعه شد. نهایتاً، رسانایی الکتریکی با استفاده از روش دونقطه ای اندازه گیری شد.

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