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Research Article

Effects of Carbon-Nanotubes on the Mechanical and Electrical Properties of Epoxy Nanocomposites

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Abstract

Carbon nanotubes (CNTs) have attracted tremendous interest as a nano-filler for epoxy nanocomposites due to their excellent properties. The efficient reinforcement has been largely limited by the agglomeration of these nano-fillers within epoxy matrix. In this work, shear mixing followed by sonication is used to prepare CNT/epoxy composites. The epoxy reinforced with functionalized Multiwalled Carbon Nanotubes (CNT/epoxy) composite was prepared by mixing (0.1, 0.2, 0.3, 0.4) wt% FMWCNT with epoxy. The tensile properties, electrical conductivity and contact angle were measured. A remarkable improvement in the mechanical properties and electrical conductivity of epoxy composites is demonstrated.

Keywords: *Epoxy, Mechanical Properties, Electrical conductivity, Nanocomposites, MWCNT /epoxy*

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1. Introduction

Carbon nanotube (CNT) was first discovered by Japanese scientist S. Iijima in 1991 (S. Iijima, 1991). CNTs are one dimensional carbon materials with very high aspect ratio greater than 1000 with diameters in nanometer scale (E.T.Thostenson *et al.*, 2001).The exceptional mechanical, electrical, and thermal properties of CNTs combined with the high aspect ratio and large surface area have made them excellent candidates to substitute or complement conventional nanofillers in fabricating polymer nanocomposites (T. Yu *et al.*, 2012; T. Li *et al.*, 2011). The major difficulties encountered the processing of CNT/polymer are the cost and aggregation of CNTs in polymer matrix. CNTs tend to agglomerate due to their large aspect ratio and van der Waals forces and this will lead to dispersion difficultly of CNTs in polymer matrix (S. Bal , 2010).

There are number of researchers who have studied CNT/epoxy nanocomposites mechanical properties (M.Tasyurek *et al*, 2012 ; V. M Mahesh *et al*, 2014) and reported improvement in tensile moduli after small additions of CNTs into an epoxy resin matrix.

The main goal of this research was to study the effects of CNTs as nanofiller on the mechanical and electrical properties of the epoxy nanocomposite. The nanocomposites was prepared as Epoxy reinforced with Functionalized Multiwalled carbon nanotubes (CNT/Epoxy). Also to investigate the effects of dispersion quality on the final mechanical and electrical properties of neat epoxy and CNTs/epoxy nanocomposites which were influenced by shear mixing followed by sonication.

2. Experimental Work

2.1 Materials

Functionalized Multiwalled Carbon Nanotube (FMWCNT) was purchased from Chengdu organic chemicals CO, LTD, China. The FMWCNTs were produced by CVD with purity > 95 wt%, outside diameter 10-30 nm and length 30 µm. The epoxy material used in this research is low viscosity diglycidyl ether bisphenol A, (DGEBA) with commercial name, ML-506 (density = 1.1 g/cm^3, viscosity = 1450 centipoise). The hardener used is a polyamine hardener, HA-11. The epoxy to hardener mixing ratio is 100:15. The epoxy and the hardener were purchased from (Mokarrar Engineering Materials Co., Iran).

2.2 Preparation of Nanocomposites

The experimental work was done at the Bio-Technology laboratory/Institute for Nanoscience and Nanotechnology (INST)-Sharif University of Technology/Tehran/Iran. Two materials were prepared: Epoxy reinforced with Functionalized multiwalled carbon nanotubes (CNT/Epoxy) and neat epoxy. The epoxy reinforced with FMWCNs nanocomposite was prepared first by mixing (0.1, 0.2, 0.3, 0.4) wt% FMWCNT with epoxy. The

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nanocomposite were prepared by direct mixing of nanofiller with epoxy resin using high speed mixer (Ultra-Turrax, Germany) at 3000 rpm for 15 min. Dispersion of MWCNTs into an epoxy resin was done using ultrasonicator (UP400S, Germany) for 20 min, at 200Wpower and 25 kHz frequency in an ice-water bath to prevent excessive heating during ultrasonication process. The hardener was then added with a ratio 100:15 wt.% (resin : hardener). The mixture was then centrifuged (EB series, Centurion Scientific, Germany) for two minutes at speeds of 3,000 rpm to remove the bubble and was then poured into the mold with dimensions (200x180x3) mm and left for 24 hours to complete the curing process. The epoxy/ MWCNTs nanocomposite was then heated for three hours in an oven at a temperature of 80 °C for post-cure.

The epoxy/ MWCNTs nanocomposite and the neat epoxy were then cut using laser cutting machine (Laser engraving & cutting machine PN6040, China) for tensile test samples in accordance with ASTM D-638) and for electrical conductivity measurements.

3. Measurements

3.1 Tensile Test

The tensile test specimens were tested using Universal testing machine HIWA 200, Korea, maximum load 5000 kgf and maximum speed 500 mm/min .

3.2 Electrical Conductivity Measurements

Electrical conductivity of the nanocomposites was measured using Agilent 4294A Precision Impedance Analyzer,in the range 5Hz to 50MHz. Electrical conductivity measurements for the prepared epoxy/MWCNTs samples were done at the Nanotechnology Lab., Ministry of Science and Technology / Baghdad , Iraq. Different voltage values were applied through the upper and the lower surface, then, the capacitance, resistance, and dielectric constant across the sample were measured.

3.3 Field Emission Scanning Electron Microscopy (FESEM)

The FESEM with spatial resolution down to 1.5 nm (Al- Razi Research Center/ Tehran – Iran) was used to characterize the epoxy nanocomposites.

4. Results and Discussion

4.1 FESEM of MWCNTS

The FESEM image of functionalized CNTs is shown in Figure 1. The image of CNTs clearly shows some catalysts particles remained from CVD preparation method. The average diameter of CNTs is about 24 nm.

4.2 Tensile Properties of CNTs/ Epoxy

The stress-strain curves of CNT/epoxy nanocomposites with CNTs (0.1, 0.2 0.3, 0.4) % are shown in Figure 2. The addition of CNTs to epoxy increases the ultimate tensile strength (the maximum stress in the stress–strain curve) and the strain to break. The ultimate tensile strength increase to 81.95 MPa at 0.4 wt% CNTs compare with 61.45 MPa for neat epoxy and the material withstands more loads before fracture, Table1. This is because the functional groups through covalent bonds act as a bridge between the CNTs and polymer chains which increase the interfacial adhesion between these two phases.

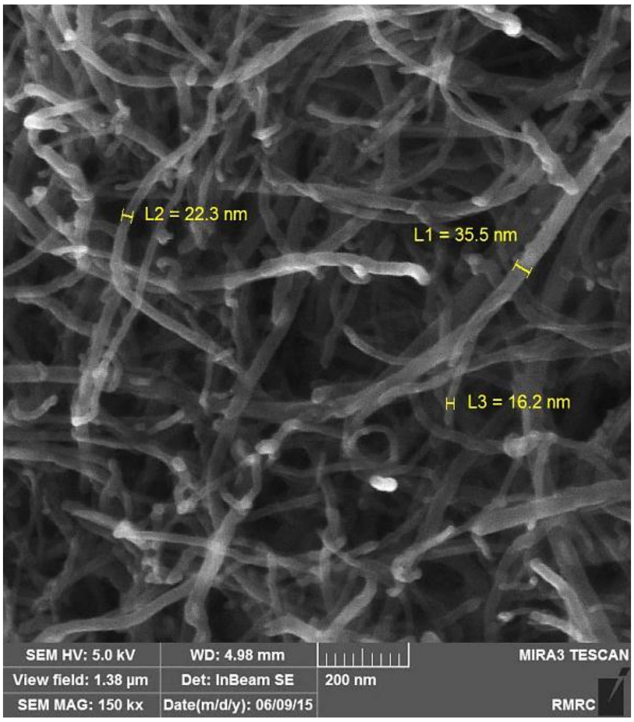


Figure 1 FESEM of CNTs

The bonds enable a stress transfer between the polymer and CNTs, which leads to improved interfacial interactions. The elastic interfacial layer between the CNTs and epoxy matrix, which is more sensitive to a large strain as reflected in the increased strain to break (J. Zhu *et al*, 2010). Therefore, the chemical functionalization of CNTs surfaces along with good dispersion of CNTs in the matrix are the key issues in preparation of good CNT/epoxy nanocomposites) (S. Bal *et al*, 2007). When the load is applied, the polymer chains will move in the loading direction with the bonded CNTs and hinder the polymer chain from slipping. Thus, more tensile load must be applied for deformation before fracture (M. S. Abed, 2014).

The Young's modulus of the CNT/epoxy nanocomposites also increases with increasing CNTs% as shown in Figure 3. At 0.4 wt% CNTs addition the Young's modulus increases drastically compare with that of neat epoxy. The strong bonding between CNTs and the epoxy matrix due to functional group restricts the mobility of the main chain of the epoxy resin leading to an increase of the Young's modulus of the CNT/epoxy nanocomposites (J. Zhu *et al*, 2010) .

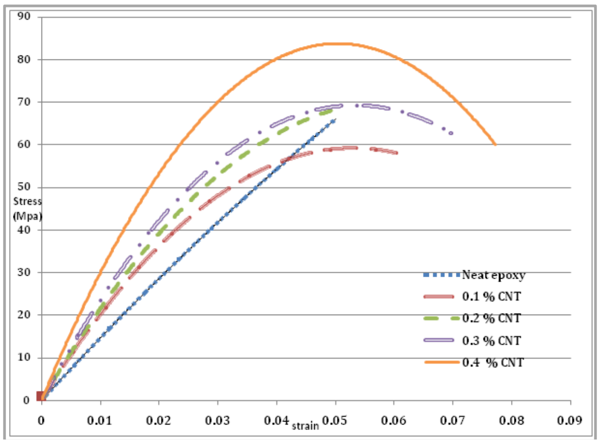


Figure 2 Stress–strain curves of CNT/epoxy

Table 1 Average Tensile Strength of CNT/Epoxy

No	Material	Average UTS (MPa)
1	Neat epoxy	61.45
2	0.1 % CNT	61.56
3	0.2 % CNT	72.44
4	0.3 % CNT	75.74
5	0.4 % CNT	81.95

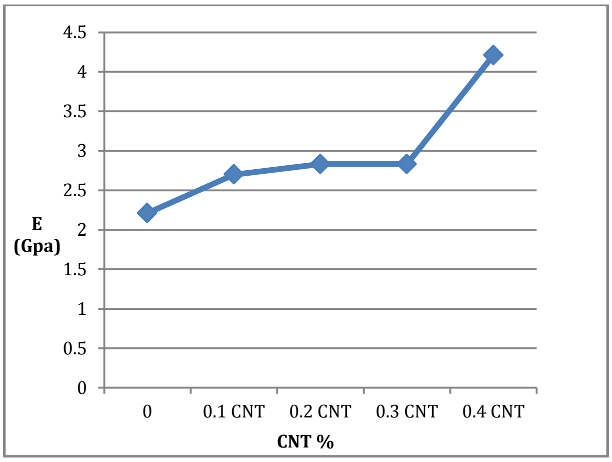


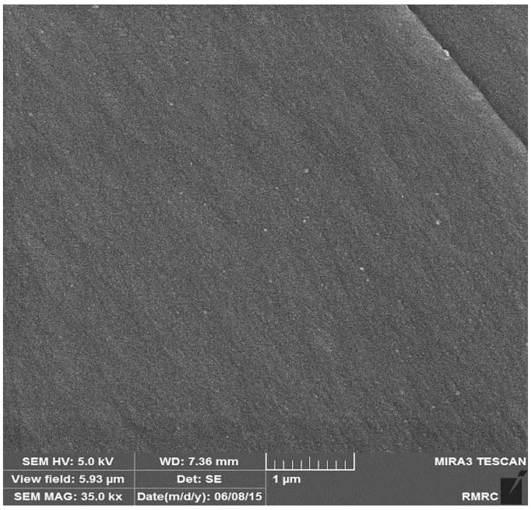
Figure 3 Young modulus of nanocomposites vs. CNTs percentage

4.3 FESEM of CNT/ Epoxy Fracture Surface

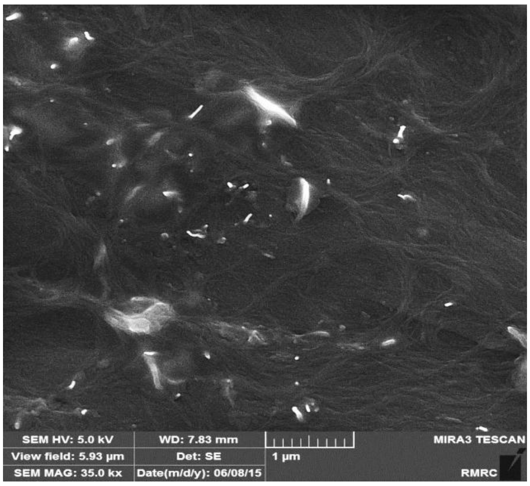
FESEM micrographs of fracture surface of CNT/epoxy nanocomposites and neat epoxy produced by shear mixing are shown in Figure 4. The fracture surface of the neat epoxy system displays long cracks with a relatively smooth surface, typical of brittle fracture behavior (Figure4a). After shear mixing and sonication the CNTs are and well dispersed in the epoxy and no agglomeration, Figure 4b-4d. Better dispersion of CNTs improves mechanical properties of the nanocomposites.

A good dispersion not only makes more filler surface area available, but also prevents aggregation of the CNTs action as stress concentrators as well as slippage of CNTs when the load is applied (L. Liu and H.D.Wagner, 2005). Since CNTs has an extremely high surface area, only a few polymer molecules can penetrate between them while covered with matrix. A good CNT/matrix interfacial bonding and a perceptible reinforcement of the matrix with the CNTs can improve the fracture strength of the composite by ensuring a shear stress transfer to the reinforcement (S. Bal *et al*, and S.Samal,2007) .

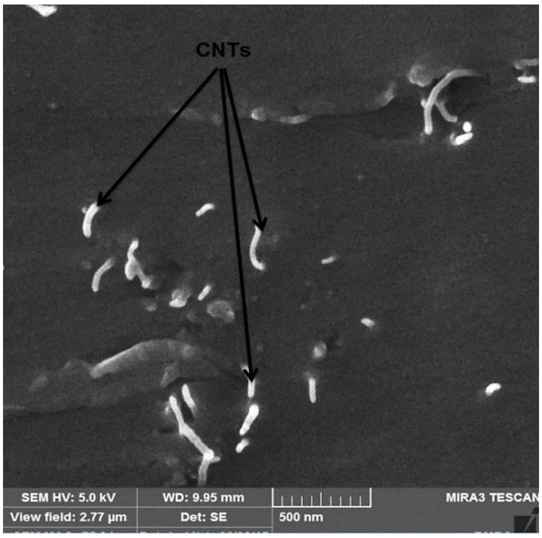
The fracture surfaces of CNTs/epoxy composites, Figures 4 b-4d, show ductile mode. This is because CNTs are very flexible and can absorb a tremendous amount of energy; or CNTs can change the crack propagation pattern and this will help in slowing down the crack formation rate; and the good interfacial bonding between the tubes and the resin matrix can enhance toughness (K. D. S. Davey, 2005).



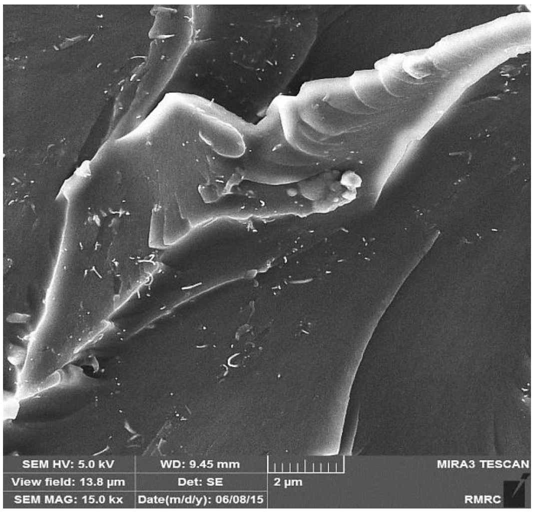
(a) Neat Epoxy



(b) 0.1% CNT



(c) 0.3% CNT



(d) 0.4 % CNT

Figure 4 FESEM images of the fracture surface of epoxy composite (a) Neat epoxy; (b) 0.1% CNT; (c) 0.3 % CNT; (d) 0.4% CNT

4.4 Electrical Conductivity of CNT /Epoxy

Electrical conductivity of the nanocomposites was measured using Agilent 4294A Precision Impedance Analyzer, in the range 5Hz to 50MHz. The electrical conductivity of CNT/epoxy nanocomposite at different CNTs % is shown in Figure 5.

The electrical conductivity of composite increases with increasing CNTs%. The electrical conductivity of CNT/epoxy nanocomposites depends on many factors including aspect ratio, type of CNTs, wt% of CNTs and surface functionalization. The addition of CNTs will lead to the formation of 3-D conductive network within the matrix , hence electron can tunnel from one filler to another , and in doing so it disables the high resistance offered by insulating polymer matrix(V. Choudhary and A. Gupta, 2011). The charge carriers increase by increasing CNTs% and can move directly from one CNTs to another. Increasing the amount of filler leads to overlapping interaction zones of carbon nanotubes inside the epoxy matrix of nanocomposites and this enhances the electrical conductivity and the area of interfacial surface (A.B. Alsafee *et al*,2014).

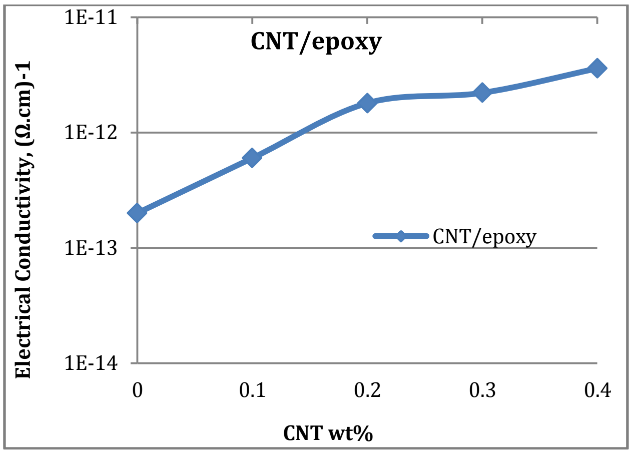


Figure 5 Electrical conductivity of epoxy nanocomposite

The formation of a conductive network in CNTs /epoxy nanocomposite requires a very high CNT content, no matter how the CNT dispersion state is. This result is also seen for other epoxy with short carbon fibers or carbon black (J. Li *et al*, 2007).

4.5 Contact Angle of CNT/Epoxy Nanocomposite

The static contact angles of water droplets on the CNT–epoxy nanocomposites with different CNTs % and neat epoxy plate surfaces were measured by an optical microscope as shown in Figure 6. The contact angle of CNT/epoxy increases with CNTs% is shown in Figure 7. This indicates that the wettability of CNT/epoxy nanocomposites surface decreases with increasing CNTs.

The decrease in the wettability behavior of CNT/epoxy surface might be related to how CNTs is dispersed in the epoxy. The contact angle of the CNT/epoxy nanocomposites was greater than that of the neat epoxy, implying a higher hydrophobicity for CNT–epoxy nanocomposites than that for the neat epoxy (S.H.Kim, 2008). CNT/ epoxy nanocomposites showed a higher surface energy due to the CNTs nanostructures (J. Park *et al*, 2009). It is well known that the surface functional groups of CNTs can play an important role in improving the surface free energy and also the adhesion between epoxy matrix and fiber. This improves the mechanical properties of the nanocomposites (Y. K. Choi *et al*,2005).

4.6 AFM of Epoxy Nanocomposite

AFM image shows that CNTs are mostly as individual ones rather than in bundles indicating that the CNTs are successfully dispersed in epoxy matrix and no big agglomerates were found as shown in Figure 8.

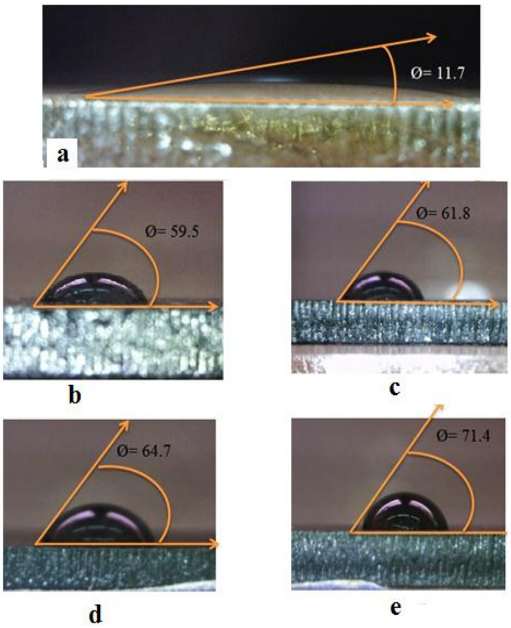


Figure 6 Contact angle of nanocomposite, (a) Neat epoxy;(b) 0.1% CNT;(c)0.2% CNT; (d)0.3% CNT;(e)0.4% CNT.

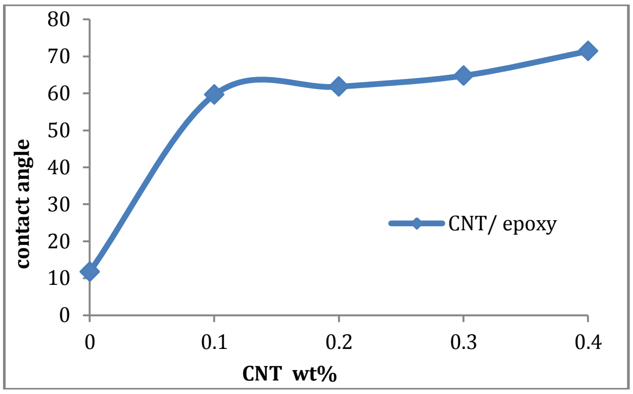


Figure 7 Contact angle vs. CNT wt%

Yellow spots in the phase image indicate relatively harder sections (CNTs) while the darker regions show softer material epoxy. The CNTs in this region are fairly uniform throughout the matrix which increases the ability of the CNTs to relieve the stress from its surroundings.

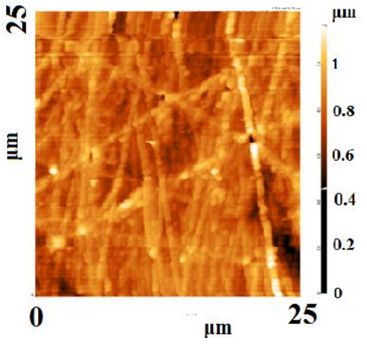


Figure 8 AFM Image of CNT/Epoxy

4.7 Water absorption of CNT/Epoxy

Water absorption on CNT/epoxy nanocomposite surfaces decreases with increasing CNTs % as shown in Figures 9. It was found that the maximum water uptake decreases gradually with increasing nano-filler (W. Liu *et al*, 2005). The presence of high aspect ratio nano-fillers can create a tortuous pathway for water molecules to diffuse into the composites (H. Zhao and R. K. Y. Li, 2008). In general, the presence of CNTs is found to decrease the water uptake of nanocomposites compared to neat epoxy due to the excellent barrier properties of these nano-fillers.

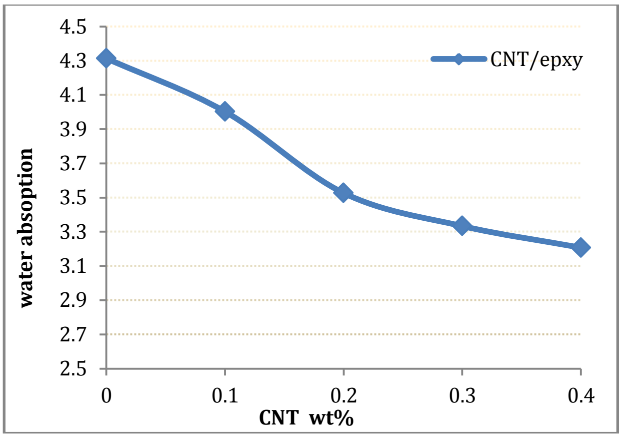


Figure 9 Water absorption for CNT/epoxy

Conclusions

CNT/epoxy composites were prepare by shear mixing followed by sonication. The results showed that the mechanical and electrical properties of composites were significantly improved. The contact angle of CNT /epoxy is increasing with CNt wt%. Water absorption for CNT/epoxy is decreasing with increasing CNT wt% .

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