



The Effect of Walled Nano-Carbon on the Physical, Thermal and Mechanical Properties of Epoxy

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(Received 2 January 2017; accepted 28 March 2017)

<https://doi.org/10.22153/kej.2017.03.006>

Abstract

The physical, the thermal and the mechanical properties of Nano-composites, that consisted of Polyprime EP epoxy that reinforced by multi-walled carbon nanotubes (MWCNTs), have been studied. Various loading ratios, 0.1, 0.5, and 1 wt. % of MWCNT have been infused into epoxy by a magnetic stirrer and then the hardener mixed with the mthat supplied with the epoxy. All sample have been cutting using CNC machine. Tensile test, three-point bending, hardness tests, lee's disk, differential scanning calorimetry, water absorption and dielectric and electrical conductivity test were utilized on unfilled, MWCNT-filled epoxy to identify the loading effect on the properties of materials. Scanning electron microscopy (SEM) was used to determine the dispersion case of nanotubes in the base matrix. The tensile test results showed an improvement in Young's modulus with higher MWCNT addition percentages. The highest value of the tensile strength was obtained at 0.5 wt.% of MWCNT with increasing ratio 66.7%. The values of the Shore D hardness tests are slightly increases as the filler percentage increased, and the maximum value was observed at MWCNT weight percent of 4.4. Images of SEM showed that the specimen of 0.1 wt.% MWCNT has less voids as compared with other specimen. Thermal conductivity and glass transition temperature increase when the loading ratio increased the maximum increasing at 1wt% with 120% and 23% respectively. The true density and electrical conductivity increase when nano carbon infused but decreasing in the dielectric constant and water absorber.

Keywords: Multi-Walled nano-Carbon tube, (MWCNT)/Epoxy composites, scanning electron microscopy

1. Introduction

The nanocomposites are kind of materials consist of dispenser inorganic nanomaterials into an organic polymer matrix, these nanomaterials improves the properties of the polymers. Adding a small quantitative of inorganic materials such as carbon nanotubes (CNTs), clay and metal nanoparticles, into the polymer matrix can considerably improving the performance of the polymer materials[1]. Numerous works were made for improving the properties of epoxy composites by the addition of fillers [2–3]. Whereas, fillers addition enhancing the modulus of elasticity and ultimate strength of epoxy, but it decreases fracture toughness. Recently, nano-fillers such as

nanoparticles, nanotubes, nanofibers, and clay have been considered as good filler for made high performance epoxy nanocomposites with improved their properties [4–6]. Carbon nanotubes are excellent nano-reinforcing filler that can be added to a variety of polymer matrices due to their properties; the ultimate tensile about 100 times higher than steel, its modulus of elasticity as high as 1 TPa, its thermal conductivity is higher than diamond, their electrical capacity is 1000 times greater than copper, and thermally stable about 2800°C in vacuum [7].

The researchers that worked on this subject showed that there are a diverse carbon nanotubes filler that could be mixed with various polymer matrices to form nanocomposites, e.g. polyamides

[8], polyimides [9], epoxy [10], polyurethane [11, 12] and polypropylene [13]. It is well documented that adding small amount of CNT (less than 1 wt.%) to a matrix system can significantly improve the thermal, electrical and mechanical properties of those composites. The epoxy/MWCNT nanocomposites is a very vital research area and attracted many researchers, e.g. Zhou *et al.* [4], Liu *et al.* [14], Sabarina than *et al.* [15], Martone *et al.* [16], Gouda *et al.* [17], and Shokrieh *et al.* [18].

Zhou *et al.* [4] studied the electrical, mechanical properties and thermal of MWCNTs/Epoxy nanocomposites. The CNTs used were 0.1, 0.2, 0.3, and 0.4 wt. %, mixed with the Epon 862 matrix via a high intensity processor of ultrasonic liquid. Good improvements were achieved, especially on electric conductivity.

Shokrieh *et al.* [18], studied a polyester/MWCNTs nanocomposites at different carbon nanotubes loading ratios (0.05wt%, 0.1wt%, and 0.5 wt. %). Sonication technique and mechanical stirring and were used to achieve well dispersion. Their results exhibited a good improvements of tensile strength and flexural strength by 6% and 20%, respectively, at the MWCNT loading ratio of 0.05 %.

The effect of concurrent presence of multi walled carbon nanotubes and nano clay on the electrical and mechanical properties of an epoxy system was investigated using ultrasonic technique was studied by Majid [19]

Jeena [20] presented result of the nanocomposites that contained epoxy and multi-walled carbon nanotubes (0% to 10 wt.%) and black carbon (0% to 15 wt%). The increasing in electrical and thermal conductivities, tensile strength, glass transition temperature and microhardness showed by using twin screw extruded.

The purpose of the present paper was to study the effect of adding Multi-Walled Carbon Nanotubes (MWCNTs) on the properties of epoxy/MWCNTs nanocomposites. The MWCNTs's weight ratios are (0.05, 0.1, and 0.5 wt. %) were studied, all samples were prepared by using a magnetic stirrer and cutting by CNC machine. Tensile tests, flexural tests, hardness tests (Shore D), Thermal Conductivity Test, differential scanning calorimetry test, water absorption and electric and dielectric test, were performed to evaluate mechanical, thermal and physical performances. Scanning electron microscopy (SEM) was used to investigate the dispersion of nano carbon tubes in the epoxy matrix.

2. Experimental work

2.1. Materials Processing

The epoxy used was Polyprime EP manufactured by Henkel Polybit Industries Ltd, provided with a hardener that used to achieve curing. The mixing percentage was 1/3 by weight of the epoxy resin. The density was 1.03 g/cc, initial cure at 25°C and 50% RH is 6-8 hours and application temperature is 10 to 35 °C. The multi-walled nano carbon tubes (MWCNT) that used here (purity > 90%) was manufactured by the Cheap Tubes Inc. (USA), as shown in Figure 1. The tube diameter ranges from 10 to 30 nm, the tube length ranges from 10 to 30 μm.

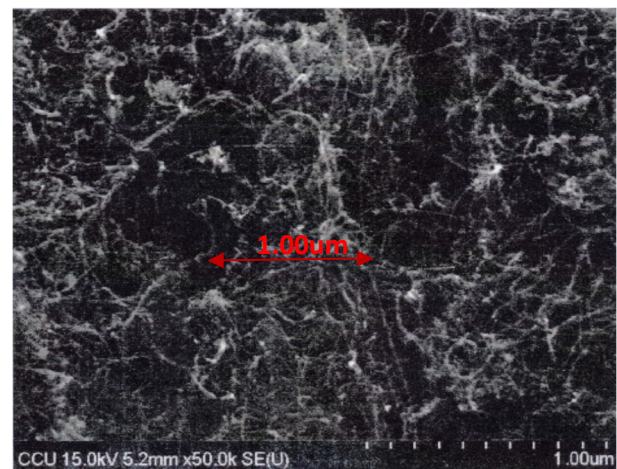


Fig.1. TEM image of MWCNTs.

Three series of composites were prepared with different content of MWCNTs as 0.1, 0.5 and 1 wt. % by magnetic stirrer shown in Fig.2. A magnetic stirrer that used in this work employs a rotating magnetic field or a set of stationary electromagnets to make a stir bar that immersed in a liquid, to spin rapidly. A stir bar mixing a solution on a combined hot-plate magnetic-stirrer device. The mixing of epoxy and hardener initially made highly interactive, volatile vapor bubbles. Therefore, the epoxy resin, in the magnetic stirrer, was preheated to 60°C to reduce its viscosity, thus reducing the chance of voids initiations and to provide good mixing with nano-carbon. Then the MWCNTs were added and mixed via stirrer bar for about 15 min to obtain homogeneity, then the hardener was added while the mixing process continue and mixing with them by wood rod for 5 min approximately. After the bubbles were totally removed, the mixture was transferred to glass molds with overhead paper to prevent adhesive for 24 hours at room temperature.



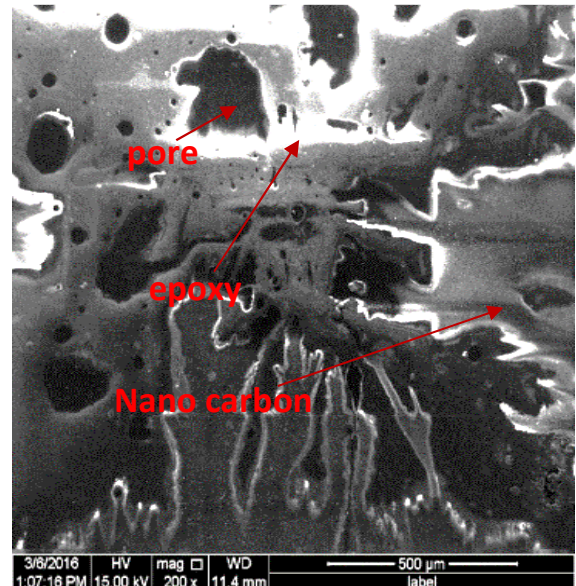
Fig.2. Magnetic stirrer.

2.2. Scanning Electron Microscope

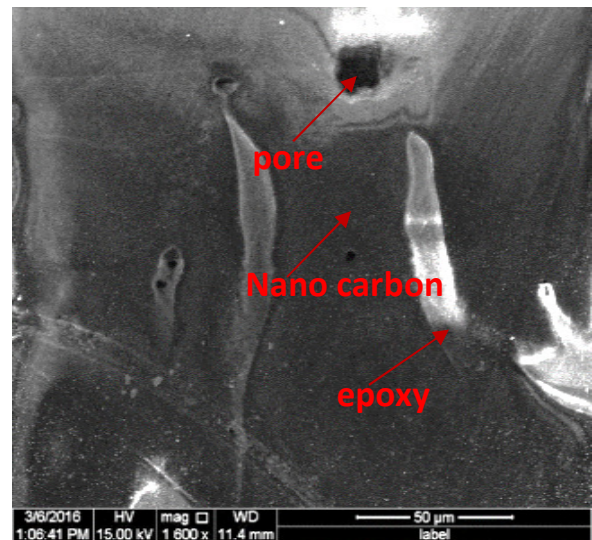
A scanning electron microscope (SEM) is shown in Fig. 3. For scanning the epoxy/MWCNTs composites SEM images at different magnifications are shown in Figure 4 for different MWCNTs ratios. It is shown from this figure that the nanocomposite with 0.1 wt.% carbon nanotubes has less voids. Micro agglomeration particles and some voids, where pores are located at the agglomerates junctions. The dark and black regions correspond to the pores and Nano carbon particles respectively, while the lighter areas are for epoxy phase. Furthermore, it is clear that by increasing nano carbon, the porosity decreased because the agglomeration increase and denser materials obtained. If there were a crack, its propagation occurs near the voids in the microstructure.



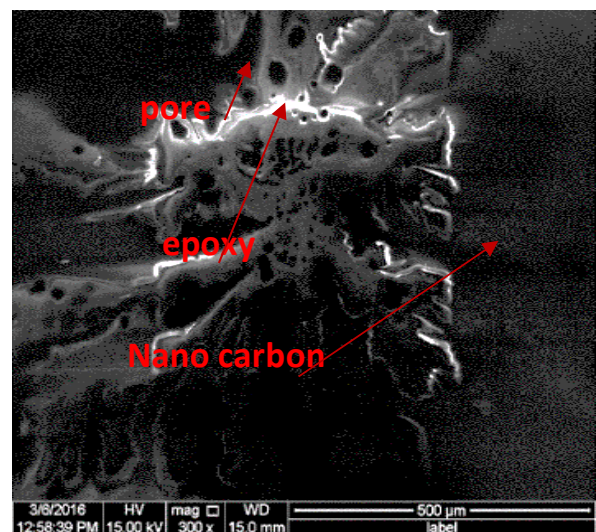
Fig. 3. SEM microscope (Inspect S50) used.



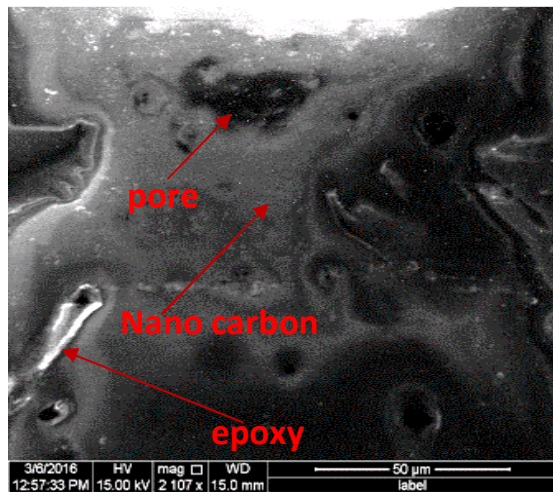
(a)



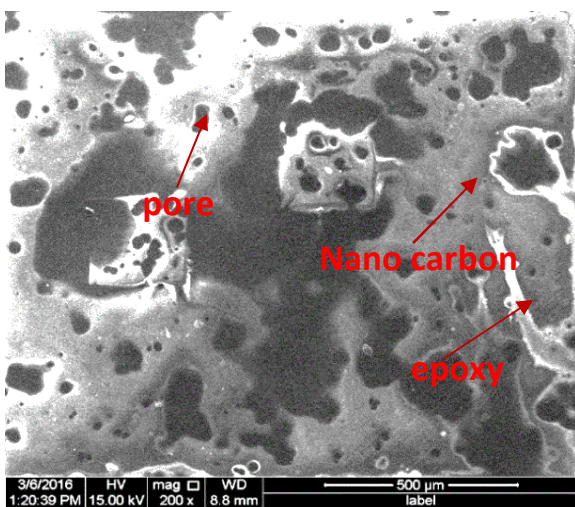
(a)



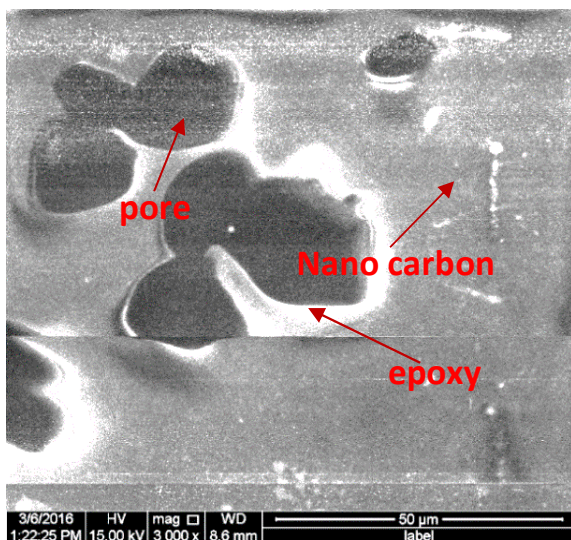
(b)



(b)



(c)



(c)

Fig. 4. SEM pictures of epoxy/MWCNTs nanocomposites at different weight ratio of MWCNTs; (a) with 0.1 wt.% MWCNTs, (b) with 0.5 wt.% MWCNTs, and (c) with 1 wt.% MWCNTs.

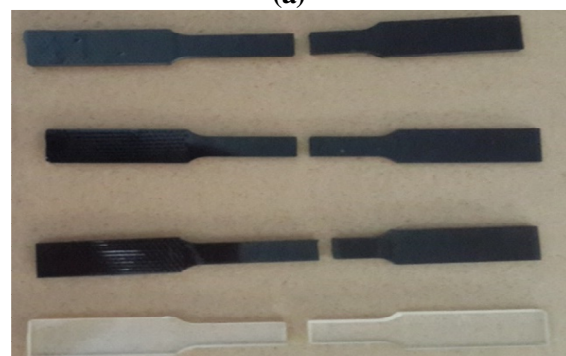
2.3 Mechanical Tests

2.3.1. Tensile Test

Tensile tests specimens were obtained using CNC machine, Knuth Rapimill 700. Four series of specimens were prepared, three of them have MWCNTs ratio of 0.1, 0.5, and 1 wt.%, respectively, and the fourth one is a neat epoxy, the specimens' dimensions were achieved according to ASTM D638, as in Figure 5. The tensile tests were performed using a servo-hydraulic Tinius Olsen H50KT. All tests were performed at room temperature and crosshead speed of 5.0 mm/min. The tensile stress-strain curve can provide data on toughness, ultimate tensile strength, maximum elongation, and Young's modulus. The tests result for the four specimens is listed in Table 1. Stress-strain curve from tensile test are shown in Fig.(6). The observed improvements may be attributed to the excellent dispersion of MWNT in resin phase and the better interphase or interfacial bonding between resin and carbon fibers.



(a)



(b)

Fig. 5. Tensile test specimens according to ASTM D638, (a) before tests, (b) fractured specimens.

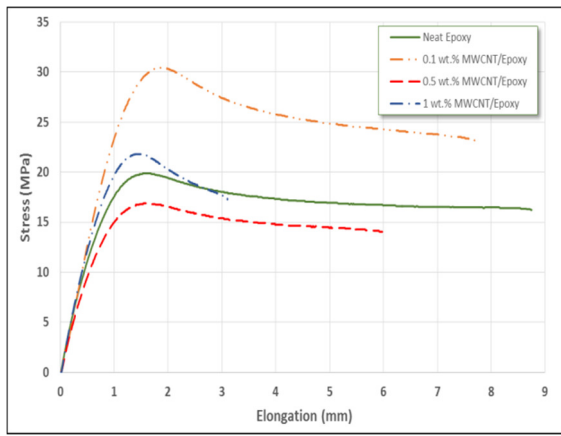


Fig. 6. Stress-Strain behavior of MWCNT/Epoxy nanocomposites with different MWCNT ratios.

It is shown from table 1 that the Young's modulus slightly increases by 7% as the filler addition of the MWCNTs increased maximum increasing young modulus is 26% when the wt% of nano-carbon is 1% depends on clusters size of nano-carbon and their distribution in the matrix. The ultimate strength results showed a maximum value of 30.448 MPa at 0.1 wt.% MWCNT addition, at this ratio a less voids were observed from the SEM images (Figure 4) performed in the present work. The maximum elongations obtained from the experimental results were decreased as the MWCNT values were increased because the formation of attractive polar forces, Vander-Waals bonding the nanotubes with chains leads to increase restriction between chains of epoxy and chains of nano-tubes/epoxy, will decrease free volume space and decreases the length of epoxy chains performed decreasing ultimate strength.

Table 1, Mechanical Property of Epoxy /MWCNT Nanocomposites.

	Young's Modulus (GPa)	Ultimate Strength (MPa)	Maximum Elongation (%)
Neat Epoxy	6.813	19.884	3.508
Epoxy with 0.1 wt% MWCNT	7.299	30.448	3.128
Epoxy with 0.5 wt% MWCNT	8.220	16.910	2.450
Epoxy with 1.0 wt% MWCNT	8.564	20.996	1.248

2.3.2. Bending Test

Flexural tests were performed according to ASTM D790 under a three-point bending. The tests were done using a servo-hydraulic testing machine, WDW-200 with speed of 5.0 mm/min. The results of the flexural tests are shown in Figure 7 the maximum improvement in the flexural strength was obtained at the MWCNT ratio of 0.5 wt. % with has the maximum increasing percentage is 66.67% from pure epoxy. When the weight ratio of carbon increase to 1% the flexural strength decrease by 23.3% because the agglomerate increase that produced stress concentration point leads to the creating the fracture source.

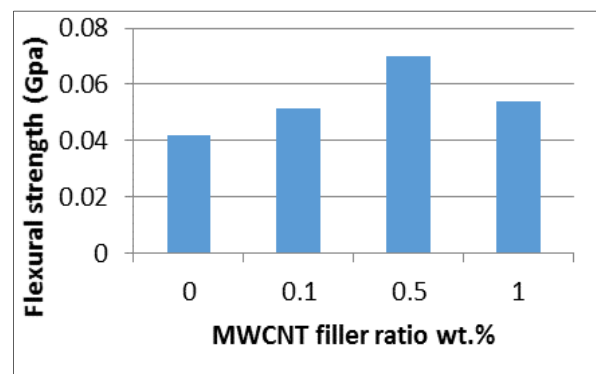
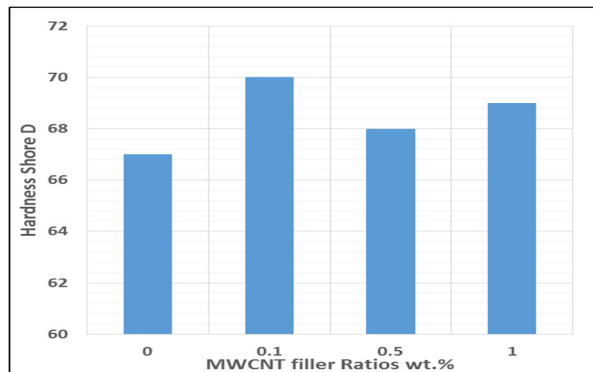


Fig.7. Flexural strength of MWCNT/epoxy nanocomposites in various loading ratios.

2.3.3. Hardness

The hardness of material is an important mechanical property because it relates how much the material will inelastic deformed when a surface load is applied. In the present paper, the Shore D hardness is used for measuring the hardness of the Epoxy/MWCNT nanocomposites, the apparatus used is known as a Durometer. The tests were performed according to ASTM D2240 at room temperature and measured three times at different locations. The average value of three time testing have been recorded as the hardness value. The device that used for measuring the hardness of the present paper is shown in Figure fig.8.b, the results of the hardness tests are shown in Fig. 8.a. The hardness test results revealed a relatively slight increase in the hardness test values, because adding the filler nano-particles will raise the materials hardness due to increasing in material resistance against the plastic deformation. The maximum one was obtained at MWCNT ratio of 0.1 wt. %.

Increased hardness values due to stacking, which decrease the polymer's molecules movement, which lead to rise the resistance of material to scratch, cut and improving the resistance to the plastic deformation. Hardness of material was depended on the kind of force that interconnected between particles in the material.



(a)



(b)

Fig. 8. (a) Hardness Shore D results for the nanocomposites with different MWCNT Ratios, (b) Hardness Shore D devise used for this purpose.

2.4 Thermal Test

2.4.1. Thermal conductivity test

Coefficient of thermal conductivity of all specimens was measured using Lee's disk method. Very often composite materials results in anisotropic media and their thermal conductivity change along the axes because of the presences of reinforcing fibers embedded in the matrix.

Fig.9. represents the test apparatus (Lee's disk apparatus) with tested composite specimen and some accessories to measure the temperature of both sides of the composite specimen.

The heater is switch on with (V = 6 Volts and I = 2 Amp.) to heat the brass disks (2,3). And the

temperatures were recorded every (5 minutes) until reach to the equilibrium temperature of all disks.

To measure the thermal conductivity using the Lee's Disk method is in the form of a disk whose thickness d_s is small relative to its radius (r) with ($d_1 = d_2 = d_3 = 12.25$ mm). (T_1, T_2, T_3) is the temperature of the brass disk. Using a thin sample means that the system will reach thermal equilibrium more quickly. And the thermal conductivity can be calculated experimentally by using the following equation, [23]:

$$K * \left[\frac{T_2 - T_1}{d_s} \right] = e * \left[T_1 + \frac{2}{r} * \left(d_2 + \frac{1}{2} * d_s \right) * T_1 + \frac{1}{r} * d_s * T_2 \right] \dots(1)$$

And (e) can be evaluated from the following equation, [23]:

$$I * V = \pi * r^2 * e * (T_1 + T_2) + 2 * \pi * r * e * \left[d_1 * T_1 + \frac{1}{2} * d_s * (T_1 + T_2) + d_2 * T_2 + d_3 * T_3 \right] \dots(2)$$

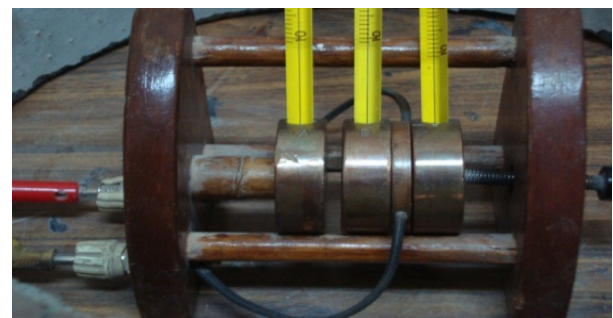


Fig. 9. Lee's disk apparatus.

From fig. 10. The thermal conductivity coefficient increase when the weight ratio of nanocarbon is (1%) the maximum percentage of increasing is 120% because the nanocarbon tube has a very good thermal conductivity that cause increasing the thermal conductivity of polymer matrix.

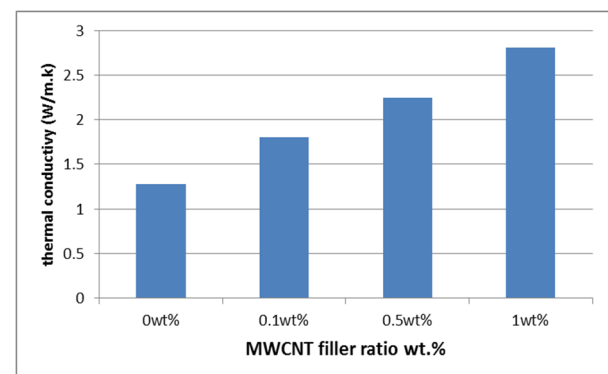


Fig. 10. Thermal Conductivity coefficient results.

2.4.2. Differential scanning calorimetry (DSC)

Differential scanning calorimetry is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample is measured as a function of temperature.

Glass transition temperature, T_g , were obtained by using DSC (Model STA PT-1000 linseis, Temperature Range: 0°C to 600°C) showed in fig. (11). A 20mg sample was used with range of temperature is from 23°C to 300°C with heating rate 5°C/min. To obtain the curing heat flow of the nano-composite. Fig (12) shows the value of glass transition temperature which increasing with increasenanano-carbon content in the composite. T_g is the point at with the material goes from a hard brittle state to a soft rubbery state which increased with increase in MWCNT expect when weight ratio is 0.5% the T_g is decrease with respect to 0.1% wt with percentage 8.1% because the dispersion of carbon nano tube. thea maximum of increasing was achieved with 1wt% loading is 23% with respect to pure matrix. The addition of nano carbon tube enhanced the thermal stability of thematerial, whichmay be because the decrease in the mobility of the chains of the epoxy around the nanofillers by strong interactions.



Fig. 11. Differential scanning calorimetry device.

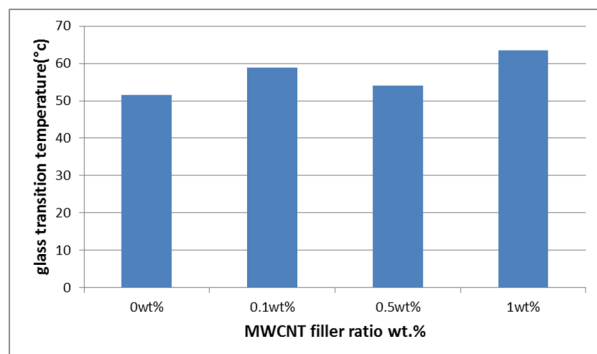


Fig. 12. Glass transition temperatures, T_g , results for the nanocomposites with different MWCNT Ratios.

2.5. Physical Test

2.5.1 Water Absorption

It is well known that the water absorption is the physical property upon that depends on the water enters the open pore channel [21]. The mechanism of water absorption is explained to be the direct uptake and flow of water by capillary and transport along the reinforcement-matrix interface. Water absorption percentage is calculated using [20] :(Archimedes base)

$$W.A \% (\text{the water absorption of the spacement}) = \frac{W_3 - W_1}{W_1} * 100 \quad \dots(3)$$

The water absorption of all prepared composites can be seen the specimen (pure epoxy) have higher water absorption than specimen (with nano filler), the decreasing water absorption percentage with increasing weight ratio of nanocarbon because the present nano carbon enclosed the porosity. The water absorption attacked the nono particle-matrix interface, causing de-bonding of the nano carbon and the matrix. The results shown in fig. 14.



Fig. 13. Sensitive Libra used (division 0.001g) with Hang system.

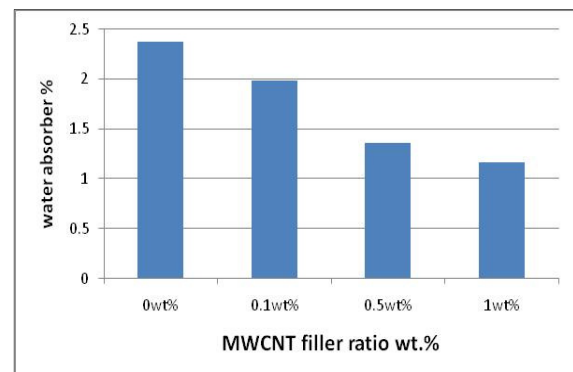


Fig. 14. result of water absorber for the nanocomposites with different MWCNT Ratios.

2.5.2 Electrical and dielectrical Test

Epoxy polymer has nature of insulating can cause garnering of electrostatic charge on their surface, causing premature degradation to the electronic structures. Electrical and dielectrical constants of the samples were found according to ASTM D257 using LCR-8110G/8105G Resistivity Test Fixture shown fig.15. The specimens were adhesive with tape of copper on two faces and two wires one for each face for better contact. The electrical properties of the nano carbon composites material depends on the shape and size of the nano carbon tube, the properties of epoxy matrix, method of manufacturing, dispersion of the nano carbon in matrix and interaction between component. The electrical percolation threshold is the minimal weight ratio of the nano materials so that a remaining conductive network exists in the material. Above this weight ratio, the electrical conductivity of the composite is very high. Under the electrical percolation threshold, the composite material behaves as an insulator.[22] Electrical conductivity is the ability of material to conduct the electrical current.

$$G = \sigma \cdot A / l \quad \dots(4)$$

A dielectric material is an electrical insulator that can be polarized by an applied electric field is the reciprocal of electrical conductivity. When a dielectric is placed in an electric field, electric charges do not flow through the material as they do in a conductor, but only slightly shift from their average equilibrium positions causing dielectric polarization.

$$C = \epsilon_r \epsilon_0 A / d \quad \dots(5)$$

In nanoscale capacitors, however, the electrostatic potentials experienced by electrons are determined by the number and locations of all electrons that contribute to the electronic properties of the device. In such devices, the number of electrons may be very small, however, the resulting spatial distribution of equipotential surfaces within the device are exceedingly complex. The results shown in figs. (16,17) the electrical conductivity is increase when the loading ratio increased the maximum value of thermal conductivity obtained at wt is 1% because the percolation threshold were obtained i.e., the conductive network were built in the based (matrix) and electrons can be conducted also, the dielectric constant decrease when the loading ratio increase the pure epoxy gave the maximum dielectric constant.



Fig. 15. LCR-8110G/8105G Resistivity Test Fixture.

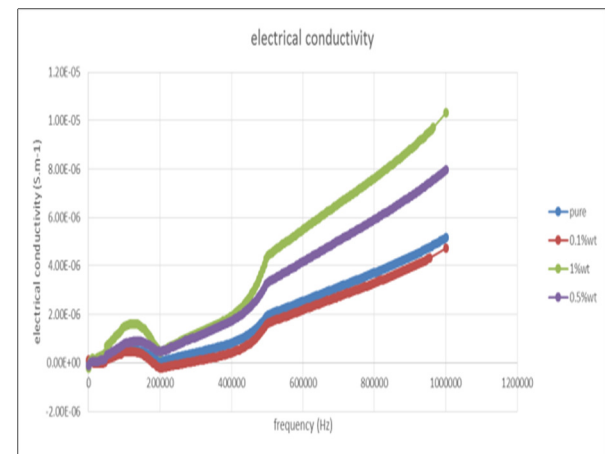


Fig. 16. Electrical conductivity's results for the nanocomposites with different MWCNT Ratios.

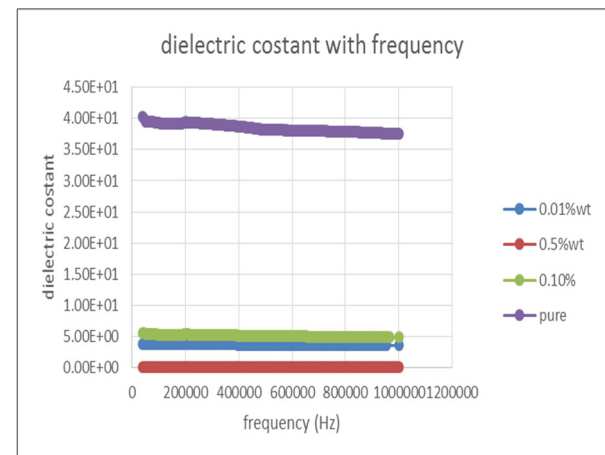


Fig. 17. Dielectrically constant's results for the nanocomposites with different MWCNT Ratios.

3. Conclusion

In this paper, the mechanical, thermal and physical properties of MWCNT/Epoxy nanocomposites at different MWCNTs weight ratios (0.1, 0.5, and 1 wt. %) were investigated.

The MWCNTs were infused into epoxy through a magnetic stirrer and then mixed with the hardener that supplied with the epoxy. It is shown that the dispersion of multi-walled nanotubes in the epoxy is greatly influences the results of mechanical tests. SEM images were used to distinguish the dispersion state of nanotubes in the matrix. The trend of results shows that adding low weight fractions of MWCNT improves tensile and flexural properties of the nanocomposites that the maximum improvement in the flexural strength was obtained at the MWCNT ratio of 0.5 wt. % with has the maximum increasing percentage is 66.67%. However, more increase in MWCNT weight fraction may causes reduction in mechanical properties. Improvements in Young's modulus were observed with a higher MWCNT weight percent. The best amounts of filler for the best properties for tensile strength and flexural strength occur at 0.1 and 0.5 wt. %, respectively. The maximum elongations values were decreased with the increases of MWCNTs weight ratios. The hardness test results did not change significantly. The decrease in mechanical properties were attributed to poor dispersions of the carbon nanotubes in the composite. Thermal conductivity and glass transition temperature increase when the loading ratio increased the maximum increasing at 1wt% with 120% and 23% respectively. The electrical conductivity increase when nano carbon infused but decreasing in the dielectric constant and water absorber.

Notation

A	the cross-sectional area (m^2)
C	the capacitance, in farads
D	the separation between the plates, in meters;
G	electrical conductance (Siemens)
L	the length of the conductor (m),
W_1	dry sample weight (g).
W_2	weight of the sample, a commentator and submerged with water (g).
W_3	weight of the sample is saturated with water (g).

Greek letters

ϵ_r	the dielectric constant,
ϵ_0	the electric constant ($\epsilon_0 \approx 8.854 \times 10^{-12} \text{ F} \cdot \text{m}^{-1}$);
σ	the electrical conductivity measured in Siemens per meter ($\text{S} \cdot \text{m}^{-1}$).

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تأثير اضافة كاربون نانوي متعدد الجدران على الخواص الفيزيائية والحرارية والميكانيكية الى الراتنج الايبوكسي

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الخلاصة

تم دراسة الخصائص الفيزيائية والحرارية والميكانيكية للمركبات النانوية المتكونة من كاربون نانوي متعدد الجدران ومادة الايبوكسي. تمت عملية الخلط بواسطة خلاط مغناطيسي بإضافة نسب وزنية مختلفة للكاربون والنانوي (0.1%, 0.5%, 1%) الى الايبوكسي، ومن ثم تمت إضافة المصلد. تم إجراء اختبارات الشد والانحناء والصلادة والموصلية الحرارية والمسعر الحراري التبايني وامتصاص الماء والعزل والتوصيل الكهربائي لجميع العينات المصنعة (التي تحتوي على الكاربون النانوي) ولك المصنوعة من الايبوكسي بدون اية إضافة. تم أخذ المجهر الإلكتروني لدراسة حالة التشتت للكاربون النانوي في المركب النانوي. أظهرت النتائج للاختبارات الميكانيكية هنا لك حسنا لمعامل المرونة مع زيادة نسبة الكاربون. تم الحصول على الاجهاد الأقصى (في اختبار الانحناء) عند نسبة 0.5% بنسبة زيادة 66.7%، وزيادة بسيطة بحدود 4.4% في الصلادة. أظهر صور المجهر الإلكتروني أقل نسبة للفجوات. تم ملاحظة عند النسبة الوزنية للكاربون النانوي البالغة 0.1% بالمقارنة مع النسب الأخرى. معامل التوصيل الحراري ودرجة حرارة التحول الزجاجي زاد عند زيادة نسبة الكاربون النانوي المضاف. يصل نسبة الزيادة عند النسبة الوزنية 1% الى 120% و 23% على التوالي. في حين أن الموصلية الكهربائية زاد عند زيادة نسبة الكاربون النانوي على عكس ثابت العزل الكهربائي وقابلية امتصاص الماء. وفقد عند زيادة النسبة.