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Comparative Study of Mechanical Properties of MWCNTS/Epoxy and SWCNTS/Epoxy Composites

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Abstract

The single – walled carbon nanotubes (SWCNTs) and multi – walled carbon nanotubes (MWCNTs) embedded into resin matrix with different weight concentrations ranging about (0.1, 0.3, 0.5 and 1 wt. %), the nanocomposites are synthesized by casting method. The main applications of this nanocomposites are in the sensors, actuators, radar. Mechanical tests were done for this study such as: tensile test, bending test and hardness test. Also many examinations were utilized to define the microstructure like scanning electron microscopy (SEM), X-ray diffraction and Raman spectroscopy. The results of this work showed that obviously an improvement in mechanical properties of the processed nanocomposites such as young's modulus, ultimate tensile strength, bending strength and Shore hardness. Also the micrographs of SEM demonstrated that SWCNTs and MWCNTs homogeneously dispersed into epoxy. On the other hand Raman spectra and XRD revealed that same results for SEM. Finally all the results for mechanical properties and microstructure evaluation show that SWCNTs give extremely higher values and properties than MWCNTs.

Key words: Single – Walled Carbon Nanotubes (SWCNTs), Multi – Walled Carbon Nanotubes (MWCNTs), Epoxy resin, Tensile test, Bending test, Hardness test.

الخلاصة

تم دمج انابيب كاربونية نانوية احادية الطبقة وانابيب كاربونية نانوية متعددة الطبقات مع الايبوكسي وبتركيز وزنية مختلفة تتراوح بحدود (0.1, 0.3, 0.5, 1 wt. %) وقد تم تصنيع هذه المادة النانوية بطريقة السباكة. من اهم التطبيقات لهذه المادة النانوية المركبة هي في المتحسسات، المشغلات والرادار ولهذا الدراسة اجريت العديد من الاختبارات الميكانيكية مثل: اختبار الشد، اختبار الانحناء والصلادة. كذلك تم استخدام العديد من الفحوصات لتحسين البنية المجهرية مثل الفحص بالمجهر الالكتروني الماسح، الفحص بحيود الاشعة السينية والفحص بمطياف رامان. وقد اظهرت نتائج هذا العمل ان هناك تحسن واضح في الخواص الميكانيكية للمادة النانوية المركبة مثل معامل يونك، مقاومة الشد القصوى، مقاومة الانحناء وصلادة شور. وقد اظهرت الصور المجهرية المؤخوذة بالمجهر الالكتروني الماسح ان الانابيب الكاربونية احادية الطبقة والمتعددة الطبقات قد تم تشتيتها بشكل متجانس في الايبوكسي. من ناحية اخرى اظهرت نتائج الفحص بمطياف رامان وحيود الاشعة السينية نفس النتائج المجهر الالكتروني الماسح. اخيرا قد اظهرت جميع نتائج الخواص الميكانيكية والبنية المجهرية بأن الانابيب الكاربونية احادية الطبقة تعطي نتائج افضل من نظيرتها ذات الانابيب الكاربونية متعددة الطبقات.

الكلمات المفتاحية : - انابيب كاربونية نانوية احادية الطبقة، انابيب كاربونية نانوية متعددة الطبقات، الايبوكسي، اختبار الشد، اختبار الانحناء، اختبار الصلادة.

LIST OF

m	Mass of mixture	gm
N	Order of reflection
P	Normal force	N
SWCNTs	Single wall carbon nanotubes
SEM	Scanning electron microscopy
T	Specimen thickness	mm
V	Volume of mixture	cm ³
W	Specimen width	mm
XRDA	X-ray diffraction analysis

SYMBOLS

SYMBOLES	DEFINITION	UNITS
ASTM	International Organization for Standardization
CNTs	Carbon nanotubes
CNTs-Cs	Chitosan carbon nanotubes
D	Interplaner distance	Å°
E	Young's modulus	GPa
L	Specimen length	mm
Lo	Original length	mm
MWCNTs	Multi wall carbon nanotubes

σ	True stress	MPa
ε	True strain
ΔL	The change of length	mm
λ	Wave length of X-ray	Å°
σ_b	Bending stress	MPa
Θ	Angle of incidence or reflection	Degree
ρ	Density of mixture	g/cm ³

1- Introduction

Carbon nanotubes have unique properties (physical, chemical and mechanical) which make them very important for use in numerous applications especially to fabricate nanocomposites. There are two types of carbon nanotubes: Single-walled carbon nanotubes (SWCNTs) (with one rolled sheet of graphene) and multi-walled carbon nanotubes (MWCNTs) (with many rolled sheets of graphene) (Bai and Allaoui, 2003). The utilize of carbon nanotubes into polymer matrix make them alter from insulating material to conductive material. One of an important polymer matrix is the epoxy, which possess an interesting characteristics enable it to produce advanced nanocomposites for using it in many applications ranging from aerospace to microelectronics (Qing Wang *et.al.*, 2008). CNTs are used as the main reinforcements for polymers depending on two factors: the first is the

uniform dispersion of CNTs into the polymer matrix and the second is the strong interfacial bonding between CNTs and polymer matrix. Recently, many investigations were done about CNTs/epoxy and how improve the mechanical, electrical and thermal properties (Alexandre *et.al.*, 2008). Epoxy resin has exceptional properties such as strength, stiffness, stability in dimension, high resistance to chemical environments and forming high adhesion to the incorporated fibers. For many years ago, there were many efforts had been made to improve the epoxy either by the particles of rubber or by adding fillers to improve mechanical properties such as the strength and young's modulus of the epoxy. Recently carbon nanotubes (CNTs) are used an important reinforcement for many different matrices of polymer because of it is high strength (100 higher than the steel) while the modulus of it about (1 TPa) (Zhou *et.al.*, 2008). There are many investigations published in this field, Yue Zhang and Shasha Huang (Yue Zhang and Shasha, 2006), studied the effect of chitosan grafted carbon nanotubes (CNTs-Cs) on mechanical properties of synthesis CNTs/epoxy composites and compared the results of this work with un-grafted CNTs. This work concluded that an improvement in tensile strength and impact strength of the obtained nanocomposites by concentration of CNTs about 1.5 wt %. While Taylor Tarlton et al. (Taylor *et.al.*, 2017), investigated the effect of aggregation of CNTs on electrical conductivity by using three specimens with different aggregation. The results of this work revealed that increasing the content of CNTs leads to create aggregation which in turn increasing electrical conductivity by making more paths through aggregation and encourage the electrons to transfer.

The aim of this work is to synthesis nanocomposite materials (SWCNTs/epoxy and MWCNTs/epoxy) and compare the mechanical properties between them.

2- Experimental procedure

2.1 Materials used

2.1.1 Carbon nanotubes

Two types of carbon nanotubes used in this work, single wall carbon nanotubes (SWCNTs) and multi-wall carbon nanotubes (MWCNTs) were obtained from manufacturer / supplier:

Cheap Tube Inc (USA) 3229 Rte 121E, Ste 3, Cambridge port, VT 05141. Table 1 shows the characteristics of single-wall carbon nanotubes and multi-wall carbon nanotubes.

Table 1: The characteristics of single-wall carbon nanotubes and multi-wall carbon nanotubes

property	MWCNTs	SWCNTs
Diameter	40 – 80 nm	< 30 nm
Length	1 – 12 μm	0.5 – 2 μm
Purity	> 99 wt%	> 90 wt%
Ash	0 wt%	< 1.5 wt%
SSA	> 233 m^2/g	> 407 m^2/g
Electrical Conductivity	> 10^2 S/cm	> $1.5 \cdot 10^2$ S/cm
Density	1.75 g/cm^3	1.4 g/cm^3

2.1.2 Polymer matrix

Epoxy Sikadur© 52 LP as a polymer matrix. The component of epoxy: Bisphenol A (epichlorohydrin) oxiraine[(C – 12 – 14 alkyloxy) methyl] derives. Epoxy and hardener were mixed by mixing ratio component (A: component B = 2:1 by volume). Table 2 shows the properties of epoxy and hardener.

Table 2: The properties of epoxy and hardener.

property	Epoxy	Hardener
Colour	Pale yellow	Clear color
density	1.1 – 1.15 g/cm^3	1 – 1.05 g/cm^3
viscosity	600 – 900 mPa. s	370 – 470 mP. s
Tensile strength	54 MPa	
Elongation at break	3.1 %	
Shear strength	29.6 MPa	
Modulus elasticity	1400 MPa	

2.2 Nanocomposites preparation

2.2.1 Synthesis of nanocomposites

The specimens of each nanocomposites SWCNTs/Epoxy and MWCNTs /Epoxy were synthesized by using solution casting method. At the first, the epoxy and hardener (Sika Paddle) in the ratio 2:1 wt % were mixed together for 5 min. Then, the mixture is poured in plastic mould (Teflon) with dimensions $10 \times 4.3 \times 1$ cm to achieve total volume 43 cm^3 as shown in figure (1).

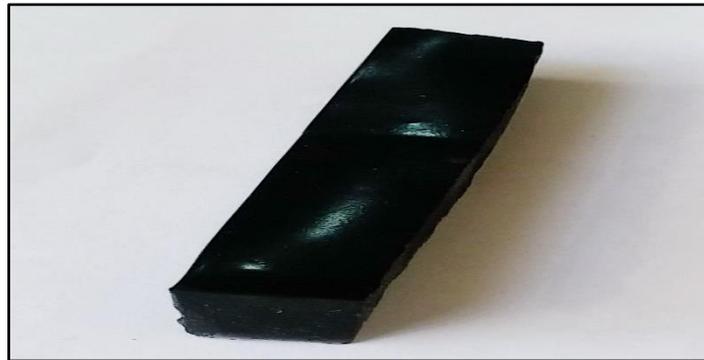


Figure (1): CNTs/epoxy specimen.

Mixing process was done at ordinary condition of room temperature and atmosphere pressure according to the following formula:

$$\rho = \frac{m}{V} (g/cm^3) \quad (1)$$

Where:

ρ : Density of mixture (g/cm^3)

m: Mass of mixture (50 g)

V: Volume of mixture (43cm^3)

The first specimen was prepared with epoxy with 33.33 g and the hardener with 16.66 g, however each of SWCNTs and MWCNTs were added at 0.1, 0.3, 0.5 and 1 wt % respectively to the epoxy and then hardener has been added. The solution was stirred in electromagnetic stirrer type (hot plate magnetic stirrer) rotating at 1400 rpm for 15 min. Figure (2) shows the magnetic stirrer which used in this work.



Figure (2): Magnetic stirrer.

After this process, the hardener has been added to the solution of epoxy and CNTs, then the mixture was stirred in same device for 5 min.

The mixture should be subjected to the sonication process for 10 min to achieve a good dispersion of CNTs in epoxy, because of the ultrasonic method has high level of energy that helps the CNTs to disperse in the epoxy through a bunch of created bubbles and collapse process (Cheol *et.al.*, 2002). This can be done by sonicator type (popular 2500ml heatable ultrasonic cleaner JP-4820 Cheap Ultrasonic Cleaner) as shown in Figure (3).



Figure (3): Sonicator.

The mixture of CNTs / epoxy was poured in a Teflon mould and cured at room temperature for 24 hr, then the specimens were post heated in an oven type (JRAD MODEL: 05) at 75C° for 1.5 hr in order to degass the air, after that the mixture was cooled inside the furnace reaching to the room temperature. Figure (4) shows the oven that used for heating process.



Figure (4): Oven type (JRAD MODEL: 05).

2.3 Examinations of nanocomposite

2.3.1 Scanning Electron Microscopy (SEM)

A Tescan Vega III (Czech Republic) SEM was used to examine the powder of SWCNTs and MWCNTs, also each of SWCNTs/Epoxy and MWCNTs/Epoxy nanocomposites. Each of the powder SWCNTs, MWCNTs and nanocomposites of the specimens of SWCNTs/Epoxy or MWCNTs/Epoxy were fixed on the stub of SEM by using a tab of silver. Small size of specimens were used for this examination, mounted on the stub of SEM by using carbon tape and coated by the gold to prevent the charging through the analysis. There are many parameters must be considered for SEM examination of nanocomposite specimens such as acceleration voltage (30 – 200Kv), size of the spot (10n – 1 μ) and working distance (4 – 11mm). There are many photomicrographs, were taken to reveal the dispersion of CNTs into epoxy, homogeneity and the possibility of forming aggregation of SWCNTs or MWCNTs.

2.3.2 Raman Spectroscopy

Raman spectroscopy was used to know Raman band shift of SWCNTs and MWCNTs powder, the spectra of Raman was collected through the Sentera Raman scope which connected to the Olympus BH-2 microscopy at 180° of geometrical scattering. Laser A CVL Melles Groit He – Ne with wave length 632 nm was used for excitation throughout the work distance length of the object which gave a spot at the surface of the specimen about 5 μ m in size.

2.3.3 X-Ray Diffraction Analysis (XRDA)

X-ray diffraction analysis was used to assess the phases of nanocomposites by calculating their crystal structure. This examination gives a high accuracy to define the arrangement of atoms in the crystal cell by scattering beam of X-ray. Bragg law enables us to determine the crystallographic structure for the specific nanocomposite by the following formula (Takada, 1980):

$$2d\sin \theta = n\lambda \quad (2)$$

Where:

N : order of reflection 1,2,3....

λ : wave length of X-ray = 1.54050 Å.

d : interplaner distance in Å.

Θ : angle of incidence or reflection of X-ray beam.

The nanocomposite specimen was held on the glass substrate with dimensions about 3×2 cm. This examination is done by XRD-6000 device, Japanese manufacturing by Shimadzu Company. The angle of Bragg range is 2θ ranging between (10°–80°) with working voltage at 40 Kv and current at 30 mA. The interplaner distance (d) was calculated using Bragg law.

2.4 Mechanical Tests

2.4.1 Tensile test

Tensile test was carried out for the nanocomposite specimens before and after adding single wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs) by using computerized universal tester by (Laryee Company) with full capacity 50 KN. The specimens of tensile test manufactured according to ASTM D638, each specimen was loaded till reaching to fracture point. The tensile strength, true strain and young's modulus were calculated by the following equations (Vorgelegt and Maciej, 2006):

$$\sigma = F/A \quad (MPa) \quad (3)$$

$$\varepsilon = \Delta L/L_0 \quad (4)$$

$$E = \sigma/\varepsilon \quad (GPa) \quad (5)$$

Where:

E: Young's modulus

σ, ε : True stress, true strain respectively

$\Delta L, L_0$: The change of length and original length respectively

2.4.2 Bending test

Bending test was performed by using bending device type (Microcomputer Controlled Electronic Universal Machine). The specimen of SWCNTs/epoxy and MWCNTs/epoxy with dimensions 9×1×1 cm were mounted at each edge of bending machine. The specimen was loaded using programmable system which controlled by computer according to ASTM d790 where the force and deflection recorded simultaneously. The general formula for bending stress for the specimen (Sinval Adalberto Rodrigues Junior *et.al.*, 2007):

$$\sigma_b = \frac{3PL}{2wt^2} \quad (MPa) \quad (6)$$

Where:

σ_b : bending stress (MPa)

P: Normal force (N)

L: Specimen length (mm)

w: specimen width (mm)

t: specimen thickness (mm)

2.4.3 Hardness test

In this work, Shore Hardness was used to measure the hardness of neat epoxy as a matrix and SWCNTs/Epoxy, MWCNTs/Epoxy nanocomposites by using shore D scale (Durometer) device type (Bareiss). The Shore D scale test provides an experimental hardness values that not related to any fundamental characteristics. Shore hardness, using either Shore A or D scales, where Shore A scale for soft materials such as rubbers while Shore D scale for harder ones. However Shore D scale is the convenient method for testing the epoxy and nanocomposites. The dimensions of the specimen was about 3 cm in length, 1.5 cm in width and 1 cm in thickness. Four readings for each specimens were recorded directly from the hardness device.

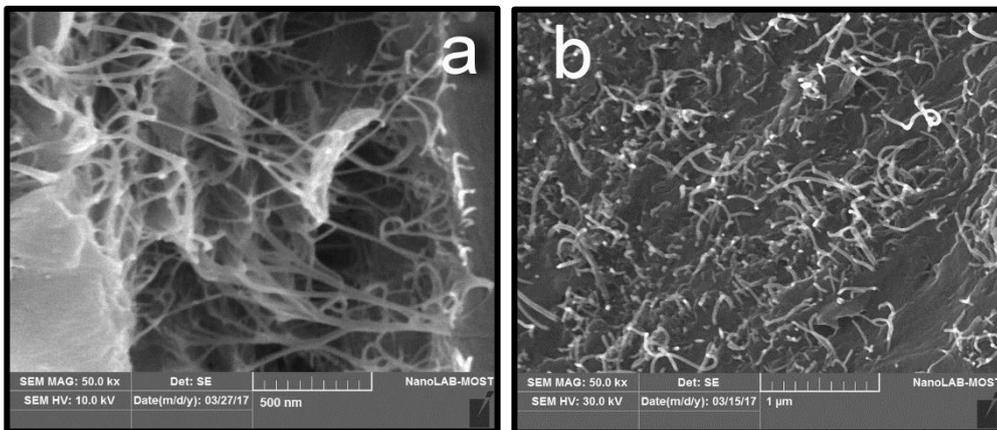
3- Results and discussion

3.1 Scanning electron microscopy

SEM device shows images of fabricated nanocomposites of SWCNTs/epoxy and MWCNTs/epoxy as shown in Figure 5. Figure (5) represents aggregation of CNTs with small concentrations (0.1, 0.3, 0.5 and 1 wt. %), increasing the concentration of SWCNTs means a good dispersion will occurred especially at 1 wt %.

While Figure (5–b) illustrates the uniform dispersion of MWCNTs into epoxy, the homogeneous dispersion enables to create many paths which are facilitated the electron to move and transfer it through the network more easily and then enhance the electrical, mechanical, thermal and electromagnetic properties of the processing nanocomposites (Erik and Tsu – Wei, 2006).

Figure (5): a) SWCNTs/epoxy nanocomposites, (b) MWCNTs/epoxy nanocomposites.



Finally, the images of SWCNTs/epoxy and MWCNTs/epoxy show the alignment of SWCNTs and MWCNTs into epoxy, this means that a good dispersion of each CNTs in this epoxy matrix and this agreement with the results Raman spectroscopy (Qing *et.al.*, 2008).

3.2 X-ray diffraction

The X-ray diffraction results were revealed some information about nanomaterial such as purity, distribution of chirality, interlayer space and diameter. Figure (6) shows a mainly XRD pattern peaks of pure SWCNTs and MWCNTs at 2θ which almost ranged between (25.6° - 26°), from this peaks pattern we can define that the reinforcement material is CNTs.

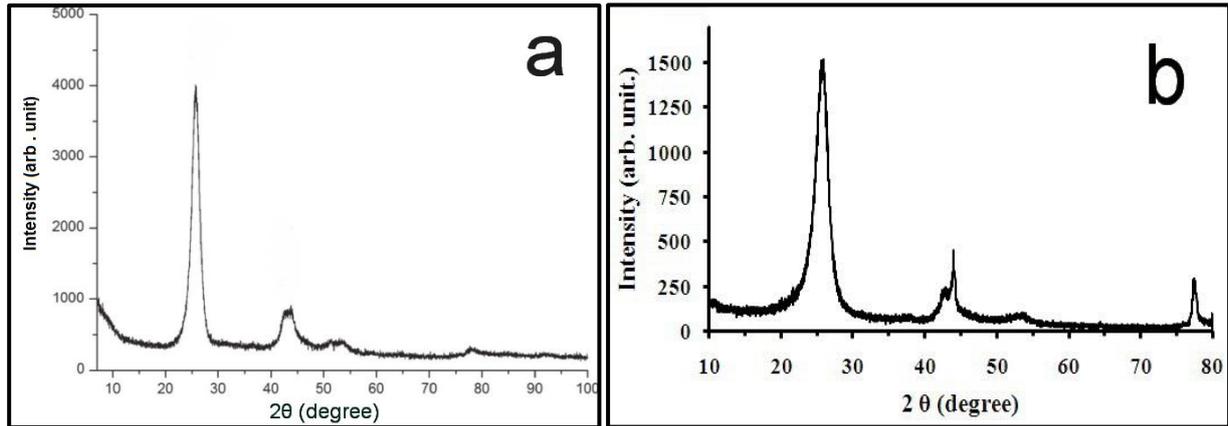
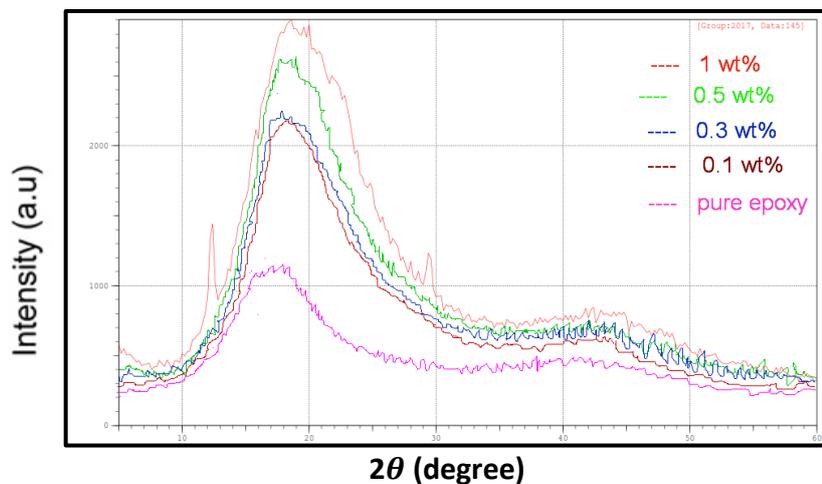
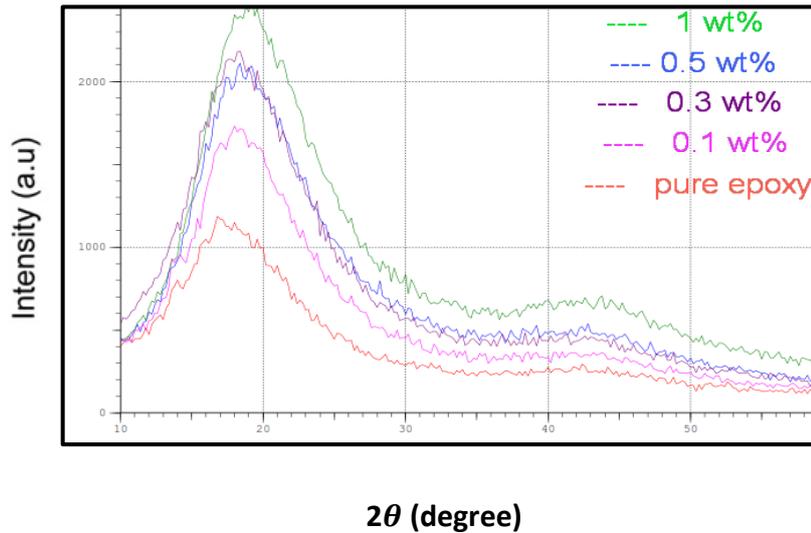


Figure (6): XRD pattern of pure sample of a) SWCNTs, b) MWCNTs.

By using Bragg law according eq (2), the interplaner space is found at (3.430 and 3.485 A°) for MWCNTs and SWCNTs respectively, which are a little larger than graphite interplaner space distance (3.353 A°). The most important property of XRD pattern of multiwall carbon nanotubes is approach to graphite characteristics, this is attributed to their innate nature. For the samples of pure epoxy we can observe an amorphous peak located at $2\theta = 17.1^\circ$. By adding SWCNTs or MWCNTs with different weight fractions into epoxy, all peaks were shifted toward more degree values of 2θ reaching to (19°). So the increasing of 2θ will continue as long as weight fraction of CNT increasing as shown in Figure (7).



(a)



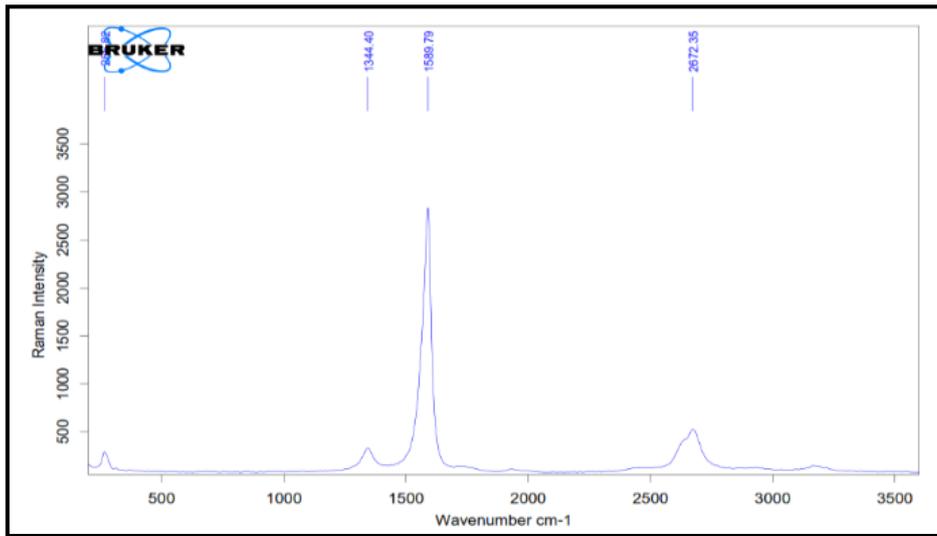
(b)

Figure (7): XRD pattern of a) SWCNTs/epoxy nanocomposites, b) MWCNTs/epoxy nanocomposites.

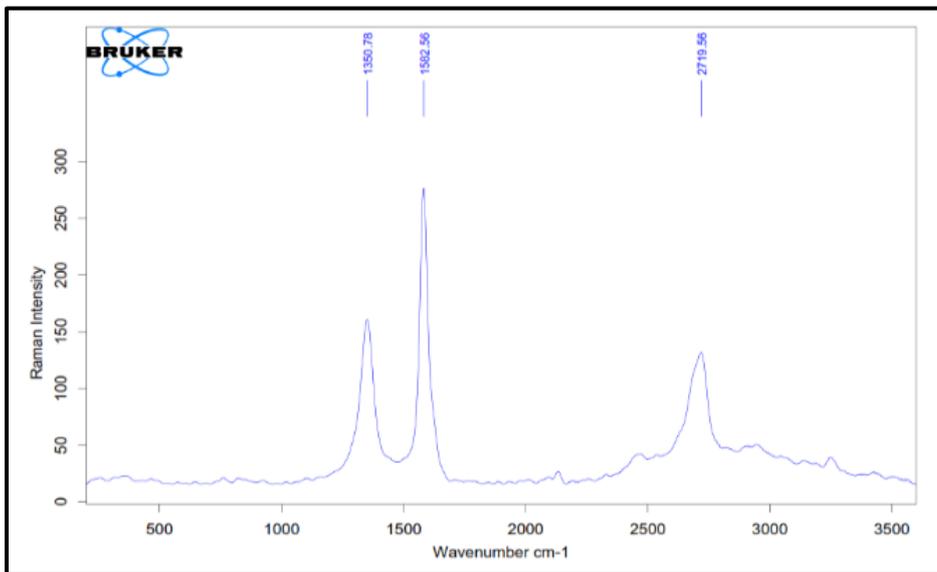
Unfortunately, the XRD method has a slightly benefit to distinguish between nanostructural details of SWCNTs and MWCNTs because of non-crystalline atoms arrangement of carbon and their special vibrations. So the spectroscopy is more compatible with carbon atomic structure.

3.3 Raman spectroscopy

Raman spectrum is considered one of the most effective techniques that apply to analysis an extensive fundamental characteristics of carbon nanotube structures. Also, it's ensure a little time (fast), non- destructive evaluation and no need to preparation the specimens. Figure (8) shows SWCNTs and MWCNTs Raman spectroscopic, it can be seen clearly that the G band for SWCNTs located at 1589.79 cm^{-1} and 1582.56 cm^{-1} for MWCNTs which are virtually consistent with the theoretical standards position and intensity of CNTs. also it can be noted the difference between them especially in the intensity of them in spite of their similarity in vibration structure which are related with carbon lattice vibration. The G band is widely used as a measure of quality of carbon nanotubes, where the more narrowing of G band mean good purified material and that is compatible with the work results of (Joe Hodkiewicz, 2010) which referred that Raman spectrum of SWCNTs and MWCNTs bear a lot of similarity to graphene, so Raman microscopy considered a valuable tool in the characterization of CNTs and it can get a closer look by using the G band which is indicator as parameter of quality. While the D band refers to disorder or defects of carbon structure which appear prominently at 1350.72 cm^{-1} for MWCNTs, and less prominent for SWCNTs at 1344.4 cm^{-1} .



(a)



(b)

Figure (8): Raman spectra for (a) SWCNTs, (b) MWCNTs.

3.4 Mechanical properties of nanocomposites

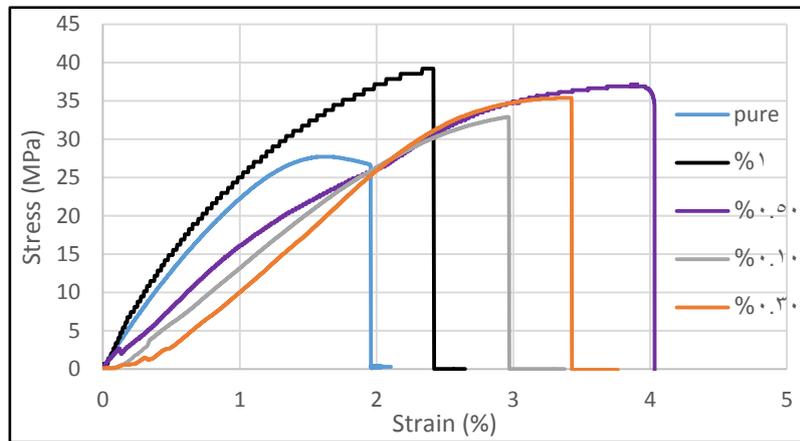
3.4.1 Tensile properties of nanocomposites

Tensile test was done according to (ASTM D638), where mechanical properties are improved with the addition of MWCNTs and SWCNTs because of the exceptional properties of them. Some of these properties are Young modulus, stiffness, hardness and impact strength. Figure (9) shows stress – strain curves with different concentrations of SWCNTs and MWCNTs incorporated into epoxy, the mechanical properties of nanocomposites will increase with increasing the weight fractions of reinforced CNTs. This is due to uniform dispersion of CNTs into epoxy and good interfacial reaction between CNTs and epoxy. However, CNTs/epoxy give an improvement in mechanical properties and the stress will

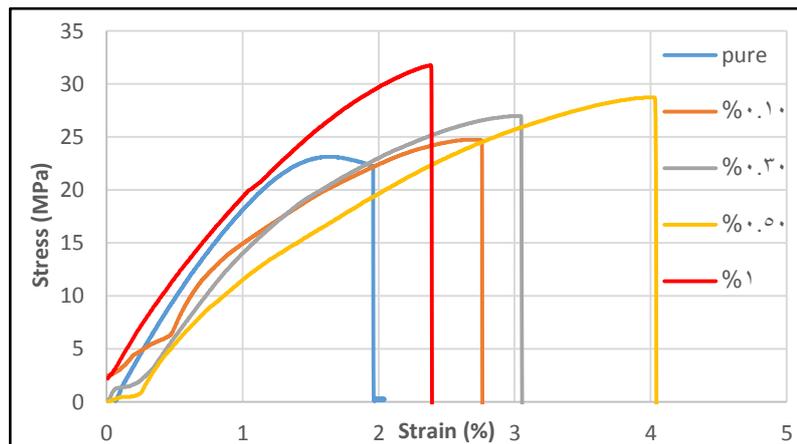
transfer from CNTs to the epoxy at the interfacial surfaces through the tensile test and this is agreed with (Saho NG, 2010). Table (3) summarized the mechanical properties of the nanocomposites SWCNTs/epoxy and MWCNTs/epoxy respectively.

Table 3: summarized the mechanical properties of the nanocomposites SWCNTs/epoxy and MWCNTs/epoxy respectively

Wt % CNTs	SWCNTs/epoxy			MWCNTs/epoxy		
	σ_y (MPa)	$\sigma_{u.T.s}$ (MPa)	E(GPa)	σ_y (MPa)	$\sigma_{u.T.s}$ (MPa)	E(GPa)
0 % CNTs	26	27.68	1.285	26	27.68	1.285
0.1 % CNTs	23.85	34.11	1.35	18.841	24.46	1.302
0.3 % CNTs	25.2	35.22	1.481	19.98	26.97	1.4
0.5 % CNTs	26.3	36.38	1.661	20.77	28.6	1.526
1 % CNTs	28.6	39.3	2.12	22.59	31.56	1.81



(a)

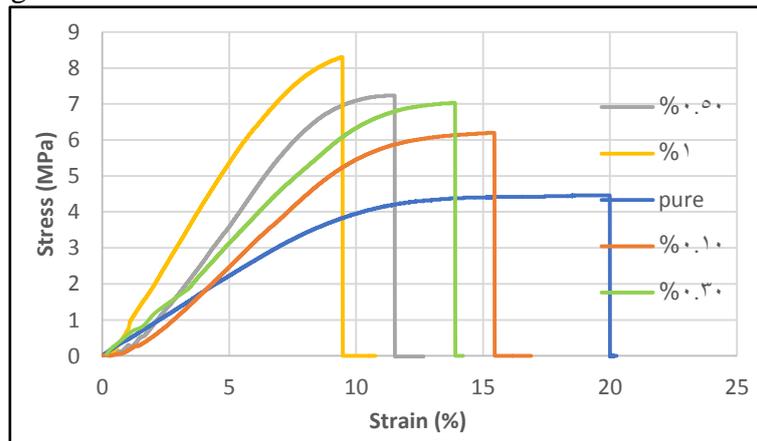


(b)

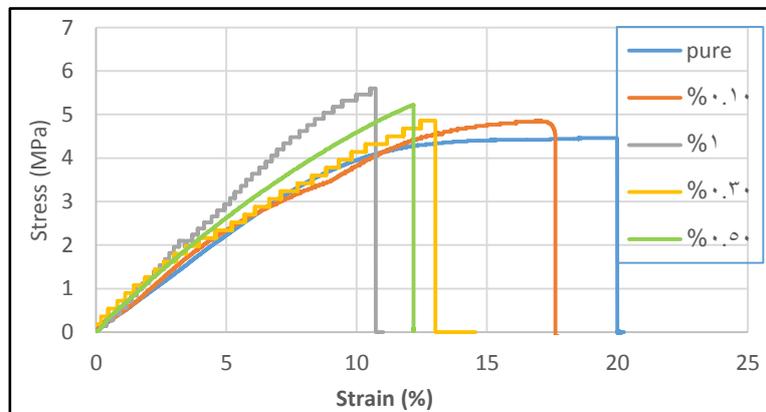
Figure (9): Tensile Stress-Strain of; (a) SWCNTs/Epoxy composites, (b) MWCNTs/Epoxy.

3.4.2 Bending properties of nanocomposites

Bending tests were done according to (ASTM D790) with three mounted points. The tests were performed by applying hydraulic machine supplied with a system recording the data. Bending tests were done at the room temperature for all specimens of SWCNTs/epoxy and MWCNTs/epoxy with different concentrations. Stress strain curves for this test are shown in Figure (10), these curves show that the bending strength increases with increasing the content of CNTs and the highest bending strength was obtained at 1 wt % for each SWCNTs and MWCNTs and for SWCNTs/epoxy higher than MWCNTs/epoxy. The explanation of this behavior is when the applied load exceeds the elastic deformation of matrix, CNTs have the ability to afford elastic deformation stresses more than the host matrix but with less elongation.



(a)



(b)

Figure (10): Bending Stress-Strain of; (a) SWCNTs/Epoxy composites, (b) MWCNTs/Epoxy.

However the improvement in bending strength perhaps attributed to the mechanical properties of SWCNTs higher than for MWCNTs. Meanwhile SWCNTs causes a good dispersion and give high bending strength comparing with the MWCNTs. Table (4) summarized the bending properties of the nanocomposites SWCNTs/epoxy and MWCNTs/epoxy. So the results of bending test are agreed with (Hiroaki *et.al.*, 2005).

Table 4: The bending properties of the nanocomposites SWCNTs/epoxy and MWCNTs/epoxy.

Wt % CNTs	SWCNTs/epoxy		MWCNTs/epoxy	
	Bending strength (MPa)	Bending modulus (GPa)	Bending strength (MPa)	Bending modulus (GPa)
0 % CNTS	4.46	0.39	4.46	0.39
0.1 % CNTS	6.2	0.606	4.7	0.471
0.3 % CNTS	7.1	0.724	4.86	0.501
0.5 % CNTS	7.3	0.884	5.22	0.551
1 % CNTs	8.28	1.09	5.6	0.657

3.4.3 Hardness properties of CNTs/epoxy

Figure (11) shows the relationships for SWCNTs/epoxy and MWCNTs/epoxy at different percentages of CNTs. These relationships demonstrated that SWCNTs/epoxy give higher hardness than MWCNTs/epoxy. This is attributed to due to the nanostructural network which are formed into matrix, uniform dispersion of CNTs into epoxy and good interfacial reaction between CNTs and epoxy.

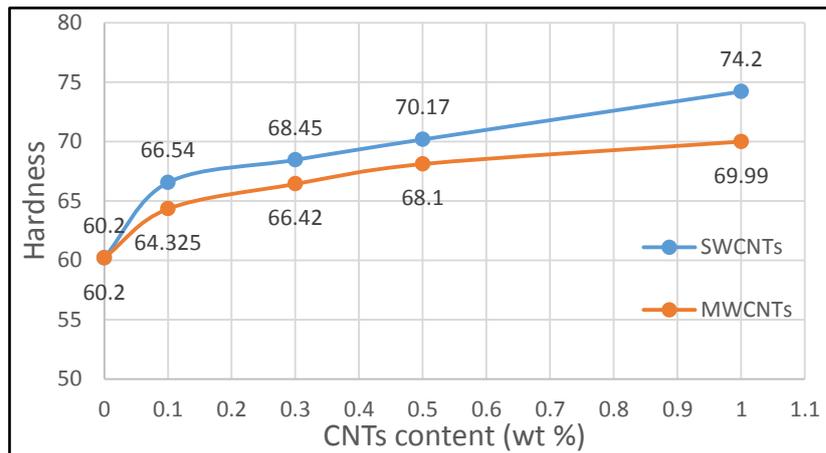


Figure (11): Hardness of CNTs/Epoxy Composites.

3.5 Conclusions:

- 1- The photomicrographs of SEM show the uniform dispersion of MWCNTs and SWCNTs into epoxy separately.
- 2- For XRD, the increasing of 2θ is concerned with increasing of weigh fractions of MWCNTs or SWCNTs.
- 3- The G band of Raman Spectroscopy is located at 1589.79 cm^{-1} and 1582.56 cm^{-1} for MWCNTs and SWCNTs respectively.
- 4- Improving the mechanical properties such as tensile strength, bending and hardness with increasing of weight fractions of MWCNTs or SWCNTs, but SWCNTs showed a clear better result than MWCNTs.

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