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Effect of Single Walled Carbon Nanotubes on Mechanical Properties of Nanocomposites

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ABSTRACT: The single – walled carbon nanotubes (SWCNTs) incorporated into resin matrix with different weight concentrations ranging about (0.1, 0.3, 0.5 and 1 wt. %), the nanocomposites are synthesized by a casting method. In this investigation, there are improvements of mechanical properties due to the homogeneous dispersion of SWCNTs into the epoxy matrix. SWCNTs/epoxy nanocomposites are achieved through the use of sonication technique for 10 minutes. In this work, many mechanical tests were done for the nanocomposites such as: tensile test, bending test and hardness test. Also many examinations were utilized to evaluate the microstructure of nanocomposite, like scanning electron microscopy (SEM), X-ray diffraction and Raman spectroscopy. The results of this work reveal that obviously an improvement in mechanical properties of the fabricated nanocomposites, such as young's modulus, ultimate tensile strength, bending strength and Shore hardness. Also the micrographs of SEM examination demonstrated that SWCNTs are homogeneously dispersed into epoxy. On the other hand, the results of Raman spectra and XRD showed the same results for SEM. Finally, the increment in young's modulus, ultimate tensile strength, bending strength and Shore hardness are about (2.12 GPa, 39.3 MPa, 8.28 MPa, 74.2) respectively.

KEYWORDS: single – walled carbon nanotubes (SWCNTs); nanocomposites; epoxy matrix; tensile test; bending test; hardness test

INTRODUCTION

In recent years, there are several investigations are taking interest in studying carbon nanotubes (CNTs) due to their high thermal conductivity, unique microstructure, corrosion resistance and excellent mechanical strength. The exceptional characteristics of carbon nanotubes (CNTs) make them industrially important such as nanotube transistor, solar cell, chemical sensors, lithum ion batteries, etc. The experimental and theoretical investigations commonly use two types of CNTs, single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanomaterial (MWCNTs) [3].

Carbon nanomaterials in single-walled carbon nanotubes (SWCNTs), multi-walled carbon nanotubes are considered an important material owing to their applications [4]. Carbon nanotubes (CNTs) are unique carbon materials with excellent mechanical, electrical and thermal properties. Qian et al. found the CNTs with a weight fraction of 1% lead to increase the elastic modulus of polymer about 42% and the fracture stress about 25%. He et al. studied that, comparing with a pure epoxy 0.5 wt. % of multi-walled carbon nanotubes (MWCNTs) increase the impact strength and tensile strength of composites containing epoxy as a matrix by 25.8% and 5.4% respectively [5].

The strong ability of CNTs to agglomeration due to van der waal forces which enhance a uniform dispersion of carbon nanotubes in the matrix. The casting technology has the ability to achieved excellent and homogeneous scattering of the reinforcement additive in metal or polymer [6]. Carbon nanotubes (CNTs) and their composites have been extremely studied and used as thermoelectric (TE) material for energy harvesting owing to their light-weight characteristics, mechanical flexibility and facile process ability [7].

Single walled carbon nanotubes (SWCNs) based composite have a potential applications in the powering electronics [8]. The principle objective of the present work is to estimate the microstructure and mechanical properties of polymer reinforced with the different concentrations of single walled carbon nanotubes (SWCNTs) for as-cast nanocomposites using a tensile test, bending test and hardness test.

EXPERIMENTAL PROCEDURE

Materials used

In this work, single-walled carbon nanotubes (SWCNTs) were obtained from a manufacturer / supplier: Cheap Tube Inc (USA) 3229Rte 121E,Ste 3, Cambridge port, VT 05141. Table 1 shows the characteristics of single-walled carbon nanotubes (SWCNTs).

Table 1. The characteristics of single-wall carbon nanotubes.

Property	**SWCNTs**
Diameter	< 30 nm
Length	0.5 – 2 μm
Purity	> 90 wt%
Ash	< 1.5 wt%
SSA	> 407 m^2/g
Electrical Conductivity	> 1.5*10^2 S/cm
Density	1.4 g/cm^3

The properties of polymer matrix were as follows: 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	_

Nanocomposites preparation

Synthesis of nanocomposites

The specimens of each nanocomposite SWCNTs are synthesized by using a solution casting method. At 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 × 4.3 × 1 cm to achieve total volume 43 cm^3. The mixing process was done at ordinary condition of room temperature and atmosphere pressure according to the following formula:

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

Where:

ρ: Density of mixture (g/cm^3)

M: Weight 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 SWCNTs were added at 0.1, 0.3, 0.5 and 1 wt. % respectively to the epoxy and then hardener was added. The solution was stirred in an electromagnetic stirrer type (hot plate magnetic stirrer) rotating at 1400 rpm for 15 min. Figure (1) shows the magnetic stirrer which used in this work.

Figure 1. Magnetic stirrer

After this process, the hardener was added to the solution of epoxy and SWCNTs, then the mixture was stirred in the same device for 5 min. The mixture should be subject to the sonication process for 10 min to achieve a good dispersion of SWCNTs in epoxy, because of the ultrasonic method has high level of energy that helps the CNTs to disperse in the epoxy through many of created bubbles and collapse process. This can be done by a sonicator type (popular 2500ml heatable ultrasonic cleaner JP-4820 Cheap Ultrasonic Cleaner) as shown in Figure (2).

Figure 2. Sonicator

The mixture of SWCNTs / 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 75 C° for 1.5 hr in order to degas the air, after that, the mixture was cooled inside the furnace reaching the room temperature. Figure (3) shows the oven that was used for the heating process.

Figure 3. Oven type (JRAD MODEL: 05)

Examinations of nanocomposite

Scanning Electron Microscopy (SEM)

A Tescan Vega III (Czech Republic) SEM was used to examine the powder of SWCNTs and SWCNTs/Epoxy nanocomposites. Each of SWCNTs powder and SWCNTs/Epoxy 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 gold to prevent the charging through the analysis. There are many parameters that must be considered for SEM examination of nanocomposite specimens, such as acceleration voltage (30 – 200Kv), size of the spot (10n – 1μ) and working distance (4 − 11mm). There were many photomicrographs taken to reveal the dispersion of SWCNTs into epoxy, homogeneity and the possibility of forming aggregation of SWCNTs.

Raman Spectroscopy

Raman spectroscopy was used to know Raman band shift of SWCNTs powder, the spectra of Raman were 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 μm in size.

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 beams of X-ray. Bragg law enables us to determine the crystallographic structure for the specific nanocomposite by the following formula:

$$2dSin\,\theta = n\lambda \quad \ldots\ldots\ldots\ldots (2)$$

Where:

n: order of reflection 1,2,3….

λ : wave length of X-ray = 1.54050 A°.

d: interplaner distance in A°.

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 was done by a 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.

Mechanical Tests

Tensile test

A tensile test was carried out for the nanocomposite specimens before and after adding single wall carbon nanotubes (SWCNTs) 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 until reaching to fracture point. The tensile strength, true strain and young's modulus were calculated by the following equations:

$\sigma = F/A$ (MPa) ---------- (3)
$\varepsilon = \Delta L/L_o$ ---------- (4)
$E = \sigma/\varepsilon$ (GPa) ---------- (5)
Where:
E: Young's modulus
σ, ε : True stress, true strain respectively
$\Delta L, L_o$: The change of length and original length respectively

Bending test

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

$\sigma b = \frac{3PL}{2w\,t^2}$ (MPa) ---------- (6)

Where:

σb: bending stress (MPa)

P: Normal force (N)

L: Specimen length (mm)

w: specimen width (mm)

t: specimen thickness (mm)

Hardness test

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

RESULTS AND DISCUSSION

Scanning electron microscopy

SEM device shows an image of fabricated nanocomposites of SWCNTs/epoxy as shown in Figure (4). Figure (4) represents aggregation of SWCNTs with concentration (1 wt. %), increasing the concentration of SWCNTs means a good dispersion will occur especially at 1 wt. %. The homogeneous dispersion creates many paths which facilitate 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 [9].

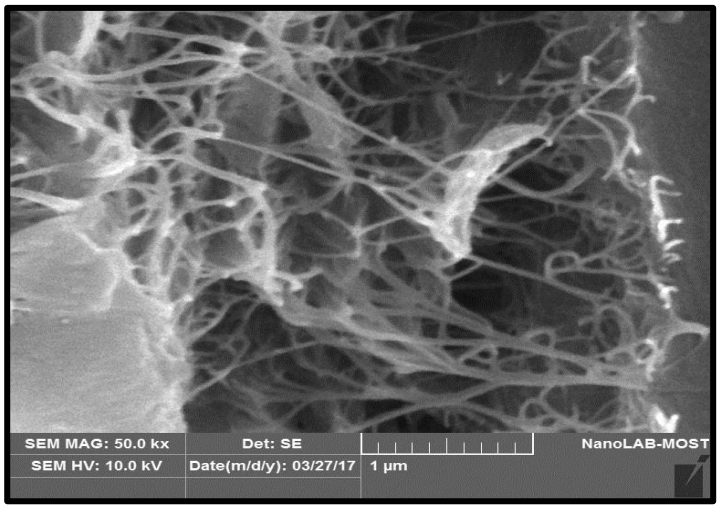

Figure 4. Scanning electron microscopy (SEM) image of SWCNTs (1 wt. %) /epoxy

Finally, the image of SWCNTs/epoxy shows the alignment of SWCNTs into epoxy, meaning that a good distribution of CNTs in this epoxy matrix has occurred and this agrees with the results Raman spectroscopy [10].

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 (5) shows a mainly XRD pattern peaks of pure SWCNTs at 2θ which almost ranged between (25.6°- 26°). From this peaks pattern we can define that the reinforcement material is CNTs. By adding SWCNTs 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 increases as shown in Figure (6).

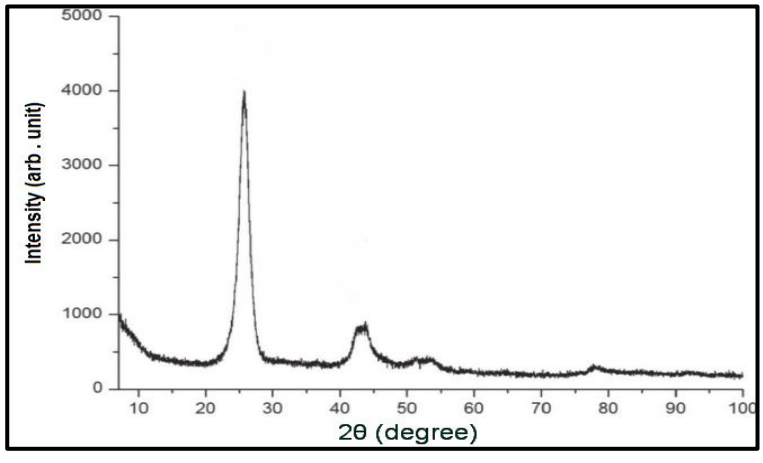

Figure 5. XRD pattern of pure sample of SWCNTs.

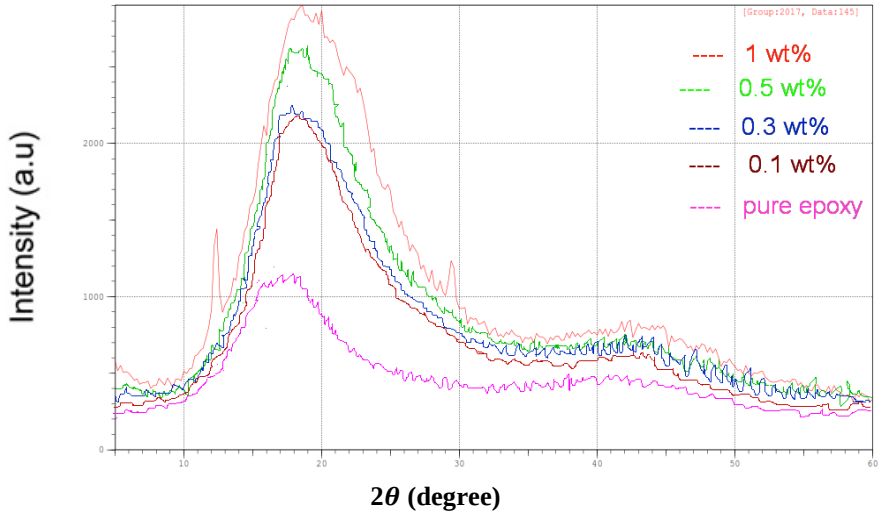

Figure 6. XRD pattern of SWCNTs/epoxy nanocomposites

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

Raman spectroscopy

Raman spectrum is considered one of the most effective techniques that apply to the analysis of an extensive fundamental characteristic of carbon nanotube structures. Also, it ensures a little time (fast), non- destructive evaluation and no need to prepare the specimens. Figure (7) shows SWCNTs Raman spectroscopic, it can be seen clearly that the G band for SWCNTs located at 1589.79 cm^{-1} 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 means good purified material, and that is compatible with the results of this work [11].

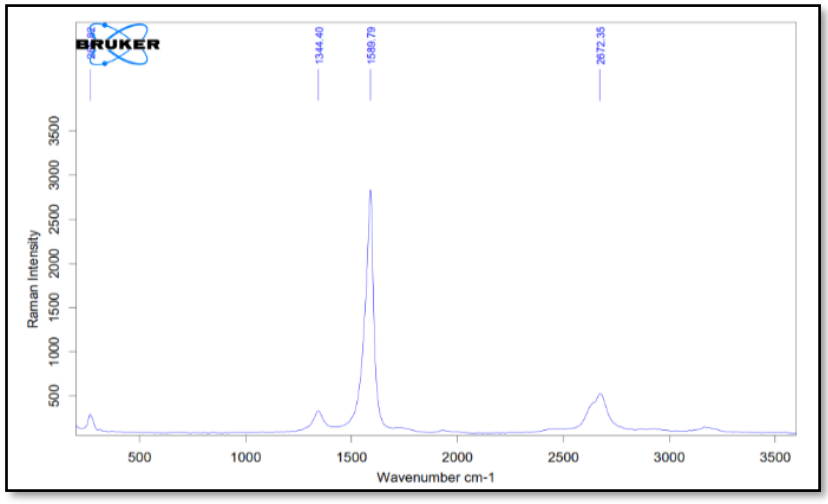

Figure 7. Raman spectra of SWCNTs

Mechanical properties of nanocomposites

Tensile properties of nanocomposites

A tensile test was conducted according to (ASTM D638), where mechanical properties are improved with the addition of SWCNTs because of the exceptional properties of them. Some of these properties are Young modulus, stiffness, hardness and impact strength. Figure (8) shows stress–strain curves with different concentrations of SWCNTs 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 SWCNTs and epoxy. However CNTs/epoxy give an improvement in mechanical properties and the stress will transfer from SWCNTs to the epoxy at the interfacial surfaces through the tensile test and this is agreed with [12]. Table 3 summarized the mechanical properties of the nanocomposites SWCNTs/epoxy.

Table 3. Summarized the mechanical properties of the SWCNTs/epoxy nanocomposit

Concentration	SWCNTs/epoxy		
Wt. % CNTs	δy(MPa)	$\delta uT(MPa)$	E(GPa)
0 % CNTs	26	27.68	1.285
0.1 % CNTs	23.85	34.11	1.35
0.3 % CNTs	25.2	35.22	1.481
0.5 % CNTs	26.3	36.38	1.661
1 % CNTs	28.6	39.3	2.12

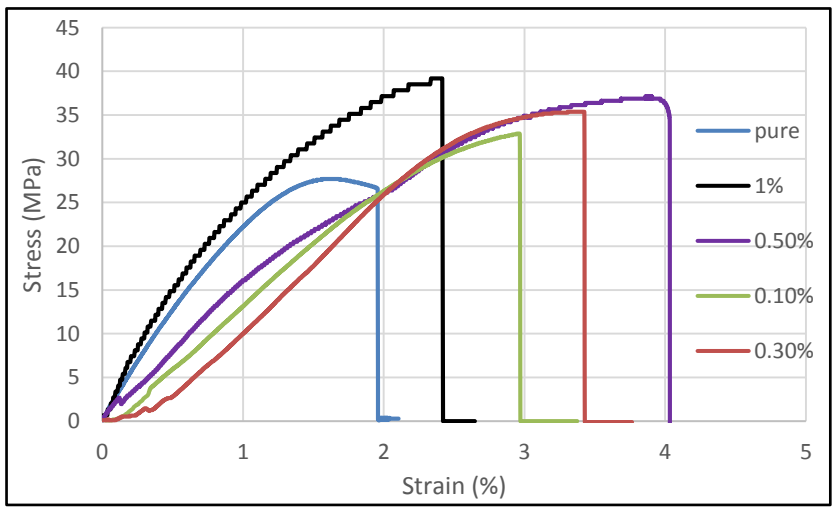

Figure 8. Tensile Stress-Strain of SWCNTs/Epoxy composites

Bending properties of nanocomposites

Bending tests were done according to (ASTM D790) with three mounted points. The tests were performed by applying a hydraulic machine supplied with a system recording the data. Bending tests were done at room temperature for all specimens of SWCNTs with different concentrations. Stress strain curves for this test are shown in Figure (9), these curves show that the bending strength increases along with the increasing content of CNTs, and the highest bending strength was obtained at 1 wt. % of SWCNTs. However, the improvement in bending strength perhaps attributed to the mechanical properties of SWCNTs. Meanwhile, SWCNTs causes a good dispersion and gives high bending strength.

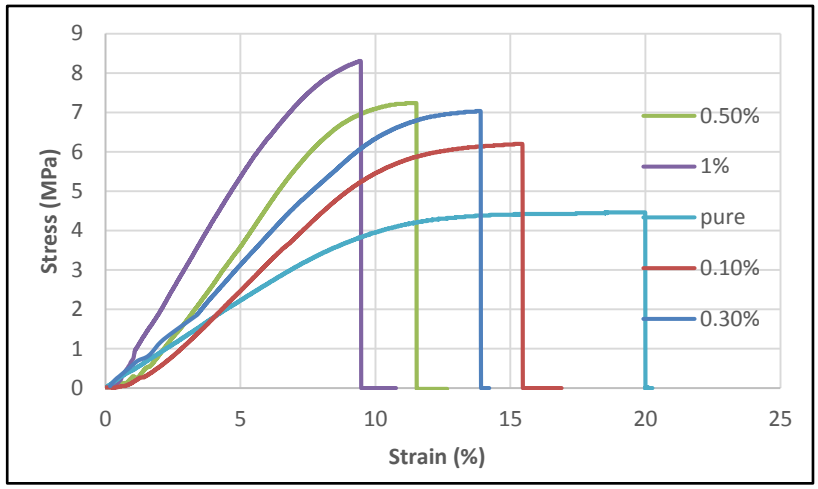

Figure 9. Bending Stress-Strain of SWCNTs/Epoxy composit

In the stress strain curves for this test, the beginning of the relationships was linear and later the strain enlarged significantly. This is most clear because of SWCNTs are elastically buckling, then the strain still increases with increasing of the stress. This test exhibited that SWCNTs/epoxy give high values of bending strength, which enable them to produce a novel nanocomposites utilized for many applications needed to absorb strain energy [13].

Concentration	SWCNTs/epoxy	
Wt. % CNTs	Bending strength (MPa)	Bending modulus (GPa)
0 % CNTS	4.46	0.39
0.1 % CNTS	6.2	0.606
0.3 % CNTS	7.1	0.724
0.5 % CNTS	7.3	0.884
1 % CNTs	8.28	1.09

Table 4. Summarizes the bending properties of the SWCNTs/epoxy nanocomposite.

Hardness properties of CNTs/epoxy

Figure (10) shows the relationships for SWCNTs/epoxy at different percentages of SWCNTs. These relationships demonstrated that SWCNTs/epoxy give high hardness. This is attributed to the same reasons which are mentioned previously for tensile test and bending test.

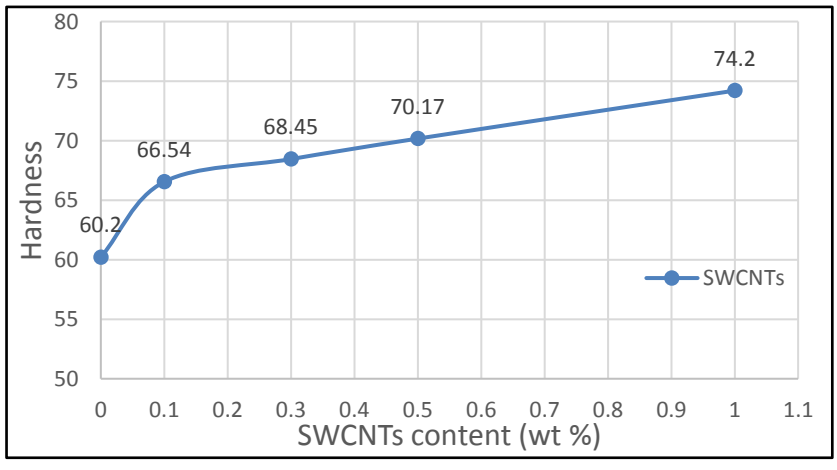

Figure 10. Hardness of SWCNTs/Epoxy Composites

CONCLUSIONS

In summary, this investigation demonstrates for the feasibility of fabricating SWCNTs / epoxy nanocomposites by casting process with enhanced mechanical properties. The conclusions of the present work are summarized as the follows:

1. The photomicrographs of SEM show the uniformly distribution of SWCNTs in to epoxy.
2. For XRD analysis, the pattern peaks of pure SWCNTs at 2θ ranged between 25.6^0- 26^0. So, the addition of SWCNTs with deferent concentration will shifted all peaks toward more degree values of 2θ.
3. The G band of Raman spectroscopy for SWCNTs is located at 1582.26 Cm^{-1}.
4. Improving the mechanical properties (of 1wt.% SWCNTs) such as young modulus, ultimate tensile strength, bending strength and hardness as 2-12 GPa, 8.28MPa and 74.2 respectively.

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