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Synthesis, Characterization and Study of DC Electrical Conductivity of Poly[MWCNT/Ester] Composites

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ABSTRACT

The Poly [MWCNT/ester] composites were synthesized by solution blending method from reacted MWCNT-COOH with Hydroquinone (HQ), Catechol (CA) and Ethylene glycol (EG).The obtained poly-composites were characterized by FT-IR, UV-Vis, XRD, TEM, SEM, TGA, DSC and DC electrical conductivity. The formation of Poly[MWCNT/ester] composites was confirmed. The DC electrical conductivity of poly-composites was in the range 10-6-10-4 S/cm due to the interaction between the nanotubes.

Key words: MWCNT; Polymer nanocomposites; Solution blending; Polyester; Catechol; Hydroquinone; Ethylene glycol; FTIR;SEM, TEM, XRD; UV; DSC; TGA and DC electrical conductivity.

1 INTRODUCTION

Polymer nanocomposites with CNT filler have been around almost as long as CNTs themselves, with the publication of the first report on this topic by Ajayan *et al.*[1] in 1994.The interest in this area stems from the fact that polymers offer many desirable qualities, such as toughness, space saving, low

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weight, good surface finish, flexibility, and low cost. Using CNTs as a property enhancing nanofiller for a high performance, lightweight composite is one of the lynchpins of nanocomposite research. The exceptional and unique properties of CNTs offer a significant advantage for the production of improved composites; but their utilization within a matrix depends primarily on the relationships between the matrix and nanoconstituent, specifically the particle's spatial orientation and interactions at the particle interface[2]. There are currently several methods used to process polymer/carbon nanotube composites, including melt mixing[3], in situ polymerization[4] and Solution Blending[5]. Many research efforts have been directed toward producing MWCNT/Polyester Nanocomposites. Abuilaiwi *et al.*[6]reported the functionalization of MWCNTs via the Fischer esterification method. Four functional groups: phenol, dodecylamine, 1-octadecanol and polyethylene glycol were covalently attached to MWCNTs via amidation or esterification. A similar reaction was reported by Sobkowicz *et al.*[7]. Fischer esterification of surface hydroxyl groups on multi walled carbon nanotubes. E.Y. Malikov *et al.*[8] reported that Fischer esterification approach was used to graft poly vinyl alcohol (PVA) to oxidized MWCNTs by connecting the MWCNTs to the PVA via ester groups. Other examples of esterification reactions include the grafting of poly(bisphenol-A-co-epichlorohydrin) chainstooxidized MWCNTs by a reactive blending process and the grafting of hyper-branched polyester based on 2,2-bis (methylol) propionic acid to the surfaces of MWCNTs [9],[10].

In this paper, we report on synthesis, characterization and DC electrical conductivity at room temperature of four Poly[MWCNT/Ester] Composites. These Poly[MWCNT/Ester] Composites were obtained via polymerization of the MWCNT-COOH with HQ, CA and EG. These composites have been characterized by FTIR, SEM, TEM, XRD, UV, DSC, TGA and DC electrical conductivity.

2 EXPERIMENTAL

2.1 Materials

Carboxy Multi Walled Carbon Nanotubes (MWCNT-COOH) were purchased from Timesnano (Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences) China. The diameter and length of MWCNT ranged between 8-15 nm and 50 μm respectively. Purity was over >95%, and the carboxyl group coverage over the nanotube surface of (2.56 %wt.). Catechol (CA), Hydroquinone (HQ) and Dimethylsulfoxide (DMSO) were purchased from Scharlau. N,N-Dimethylformamide (DMF 99%), Tetrahydrofuran (THF 99.9%), Ethylene glycol (EG) were purchased from Aldrich. Ethanol (96%) was purchased from Fluka and used as received without any further treatment in this study.

2.2 Instrumentation

The FTIR spectra were recorded using the KBr disc technique on a JASCO 410 FTIR Spectrophotometer (at Sana'a University, Sana'a, Yemen). The melting points were measured with an electrothermal melting point apparatus (at Sana'a University, Sana'a, Yemen). The thermal analyses (TGA and DSC) were carried out on a Mettler Toledo TGA/SDTA851e analyzer, and Mettler Toledo DSC823e analyzer, respectively, at 23 to 1000 ºC under 20 ml nitrogen per minute and a heating rate of 10 ºC per minute (at UPM AND UM Universities, Kuala Lumpur, Malaysia). UV-vis absorption spectra were measured using a Specord 200, Analytik Jana, Germany in DMF (~10-4 mol/dm3) (at

Sana'a University, Sana'a, Yemen). The X-Ray diffraction was carried out on a BrukerAxs Da Advance, Germany (at UPM AND UM Universities, Kuala Lumpur, Malaysia). Electrical conductivity measurements were taken on a Keithley Picoammeter/Voltage Source Model 6487 using a double probe locally fabricated conductivity bridge cell (at Sana'a University, Sana'a, Yemen). The Scanning Electron Microscope (SEM) was carried out on a (SEM HITACHI S-3400N) (at UPM AND UM Universities, Kuala Lumpur, Malaysia). The Transmission Electron Microscope (TEM) was carried out on a Phillips CM-12, USA, the samples were prepared by Leica ultracut UTC ultramicrotome (JEOL, Japan) with an accelerating voltage of 100 kV.

2.3 Preparation of Poly[MWCNT/ester] composites

The method employed to prepare the Poly-composites was solution blending. This method is the most widely used for dispersing nanotubes on the laboratory scale because it is effective and it can be used to fabricate small amounts of a sample. Solution processing begins by dispersing nanotubes in solvent, followed by mixing with the monomer that is dissolved in a suitable solvent. The poly-composite is then formed by precipitation or by evaporation of the solvent [11,12].

2.3.1 Preparation of poly-composites of MWCNT-COOH with HQ or CA

0.3 g of MWCNT-COOH was dispersed in 15 ml of DMSO and 0.2 g of (HQ or CA) was dissolved in 10 ml of ethanol and added to the MWCNT-COOH dispersion in DMSO. The mixture was then stirred at 180 °C for 24 h under reflux. After cooling to room temperature, the mixture was vacuum-filtered through a 0.22 µm membrane and was thoroughly washed several times with little amount of ethanol. The filtered solid was then dried in a vacuum oven at 90°C.

2.3.2 Preparation of poly-composite of MWCNT-COOH with EG:

0.3g of MWCNT-COOH was dispersed in 15 ml of DMF and 1 ml of EG was added to the MWCNT-COOH dispersion in DMF. The mixture was then stirred for 24 h at 90 °C under reflux. After cooling to room temperature, the mixture was vacuum-filtered through a 0.22 µm membrane and was thoroughly washed several times with ethanol. The filtered solid was then dried in a vacuum oven at 90 °C.

3 RESULTS AND DISCUSSION

3.1 Characterization

Poly[MWCNT/ester] composites were prepared by reaction MWCNT-COOH with (HQ, CA and EG) in refluxing solvent (DMSO) to give poly-composites products. The method used to prepare the Poly[MWCNT/ester] composites was solution blending. The chemical reaction of Poly[MWCNT/ester] composites is shown in Figure 1. Table 1 summarizes the physical properties (melting point, color, percentage yield and solubility) of MWCNT-COOH and Poly[MWCNT/ester] composites. Generally, these compounds showed good solubility mainly in DMSO, and either partially soluble or insoluble in the other common organic solvents.

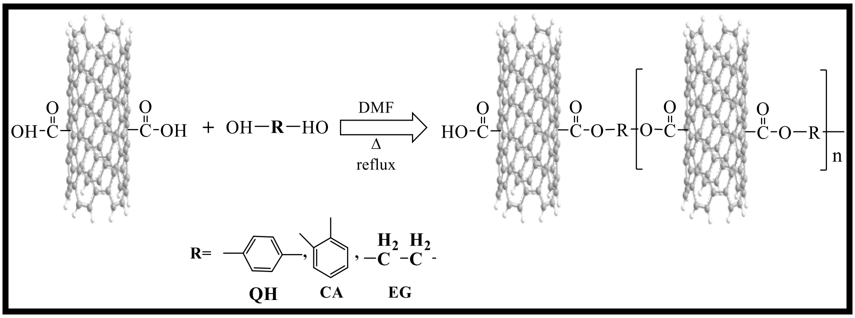

Figure 1: Synthesis of Poly[MWCNT/ester] composites.

Table 1: Physical properties of MWCNT-COOH and of Poly[MWCNT/ester] composites.

No	Symbol	%Yield	Color	M.P.	Solubility		
					DMSO	DMF	EtOH
1	MWCNT-COOH	-	Black	> 350°C	++	+	-
2	Poly[MWCNT/HQ]	98%	Black	> 350°C	++	+	-
3	Poly[MWCNT/CA]	90%	Black	> 350°C	++	+	-
4	Poly[MWCNT/EG]	95%	Black	> 350°C	++	+	-

(++;Soluble, +;Partially Soluble,-; Not Soluble.

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectroscopy (IR) is a common method for characterizing chemical bonds. Comparison of the IR spectra of MWCNT-COOH was made before and after functionalization might show whether new bonds were formed.

The FT-IR spectra of MWCNT-COOH and Poly[MWCNT/ester] composites are given in Figure 2, and the results are summarized in Table 2.

In Figure 2(a), the IR spectra of MWCNT-COOH show a broad peak at 3434 cm^{-1} which can be attributed to the O-H stretching of carboxyl groups (COOH). The peak at 1542 cm^{-1} can be associated with the C=C stretching vibration of the MWCNT backbone**[13]**. The peak at 1637 cm^{-1} is related to the C=O stretching vibration of the carbonyl group acid**[14]**.

Figure 2 (b, C and d) show the IR spectra of MWCNT-COOH after functionalization with HQ, CA and EG respectively. The figure shows that the peaks at (1654, 1646 and 1653 cm^{-1}) can be attributed to the C=O stretch of the ester which is overlapped with the unreacted C=O acid. The peaks at (1024, 1026 and 1048 cm^{-1}) arise from the C−O stretching of the ester group respectively.

The broad peaks at (3433, 3432 and 3436 cm^{-1}) can be assigned to the O-H stretching of unreacted carboxyl groups, which means there is still a little amount of unreacted carboxylic acid group**[15]**.

3.3 UV/Vis Spectroscopy

UV-visible absorptions of MWCNT-COOH and its Poly[MWCNT-ester]composites are summarized in Table 3. Figure 3 illustrates the electronic spectra of MWCNT-COOH and its esters. MWCNT-COOH shows three bands π-π^* transition at λ_{max} 286,290 and 298 nm, and another two bands n-π^* transition at λ_{max} 320 and 338 nm. However, spectra of the Poly[MWCNT/HQ], Poly[MWCNT/CA] and Poly[MWCNT/EG] show a blue shift of π-π^* transition at λ_{max} (283), (282,296) and (278,282) nm respectively. But, n-π^* transition shows a red shift λ_{max} at (312,340), (322) and (348) nm respectively. This confirms the ester formation.

Table 2: Main FT-IR absorption bands of the MWCNT-COOH and Poly[MWCNT/ester] composites.

Bands Compound	O-H st.	C=Cst.	C=O st.	C-O st.
MWCNT-COOH	3434	1542	1637	1032
Poly[MWCNT/HQ]	3433	1560	1654	1024
Poly[MWCNT/CA]	3432	1578	1646	1026
Poly[MWCNT/EG]	3436	1557	1653	1048

st. = Stretching Vibration

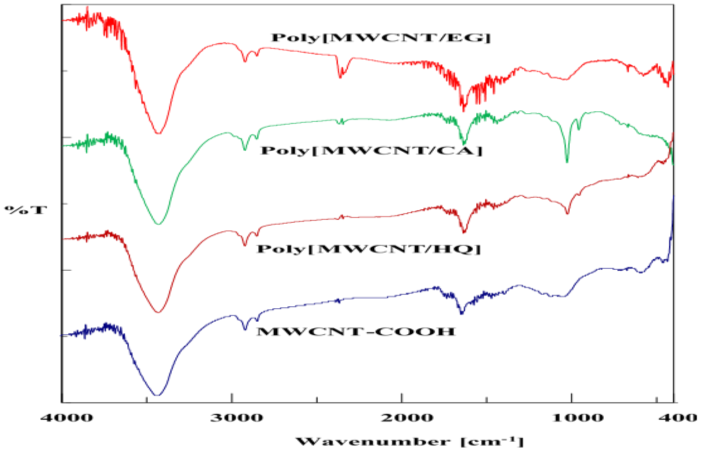

Figure 2:FTIRspectraof (a) MWCNT-COOH, (b) Poly[MWCNT/HQ], (c) Poly[MWCNT/CA] and (d) Poly[MWCNT/EG].

Table 3: UV-visible spectra of MWCNT-COOH and Poly[MWCNT/ester] composites

Compound	$\lambda_{max}\pi$-π^*	λ_{max}n-π^*
MWCNT-COOH	286,290,298	320,338
Poly[MWCNT/HQ]	283	312,340
Poly[MWCNT/CA]	282,296	322
Poly[MWCNT/EG]	278,282	348

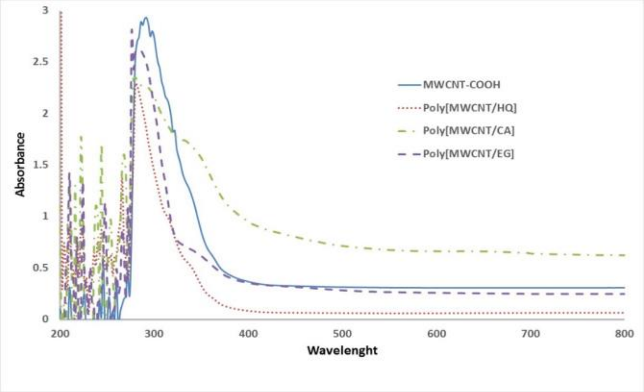

Figure 3:UV-vis spectra of MWCNT-COOH and Poly[MWCNT/ester]composites.

3.4 Microscopy Characterization (TEM, SEM)

3.4.1 Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) is often used to observe the length and diameter of carbon nanotubes. Figure 4 (a-h) displays TEM microphotographs of the MWCNT-COOH and Poly[MWCNT/ester] composites at different magnifications. Figure 4 (a and b) shows TEM images of MWCNT-COOH. It shows that MWCNT-COOH formed an entangled structure with an average diameter of 8-15 nm and their average length is approximately equal to 50μ mas announced by the supplier (Timesnano). In addition, a small spot shape was observed which might be ascribed to –COOH group.

As shown in Figure 4 (c-f) the TEM of MWCNT-COOH after polymerization HQ, CA and EG, respectively. Poly[MWCNT/ester] composites relatively exhibit good dispersion and less entangled except EG. The most entangled, showed on Poly[MWCNT/EG], may be due to EG is an aliphatic compound has only two carbons in backbone, which make a tight crossed connecting between the nanotubes.

The images clearly show that the spot shape for –COOH groups disappeared in Poly[MWCNT/ester] composites.

3.4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy was used also to confirm the possible morphological changes on functioned MWCNT. Figure 5 (a-P) presents SEM microphotographs of the surface morphology and dispersion of the MWCNT-COOH and Poly[MWCNT/ester] composites at different magnifications. Figure 5 (a-d) shows that the group of MWCNT-COOH forms large agglomeration, random and curled structure, and possesses high aspect ratio; this may be because of the hydrogen bonds between the nanotubes. Figure 5 (e-l) shows MWCNT-COOH after polymerization with HQ and CA; many walls were broken and appear to be thicker compared to the MWCNT-COOH. In addition, it is noted that the conglomerates have become smaller and the ester bonds exist between MWCNT with HQ and CA, which reduced the hydrogen bonding.

On the other hand, Figure 5 (m-p) shows that the SEM of Poly[MWCNT/EG] forms large random masses, and possesses high aspect ratio, suggesting that covalent bonding of ester exists between the MWCNT and EG, while EG is an aliphatic compound that has a small size, and which will form a tight crossed link between the nanotubes.

The images clearly show that the surface morphology of Poly[MWCNT/ester] composites is significantly different in contrast with MWCNT-COOH.

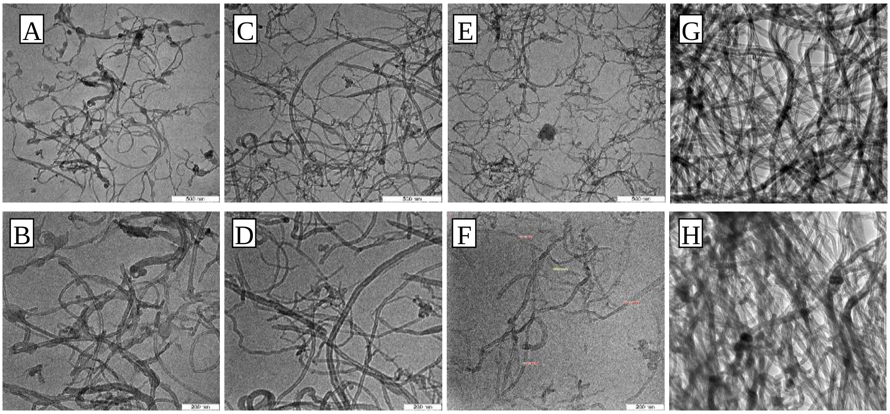

Figure 4: TEM microphotograph of MWCNT-COOH and Poly[MWCNT/ester] composites: MWCNT-COOH (a) ×50 000; (b) ×100 000, Poly[MWCNT/HQ] (c) ×50 000; (d) ×100 000, Poly[MWCNT/CA] (e) ×50 000, (f) ×100 000 and Poly[MWCNT/EG] (g) low magnification, (H) high magnification.

3.5 X-ray Diffraction

X–Ray diffraction (XRD) is used to identify materials by determining their crystallographic structure, and in this way it is a characterization tool on the scale of nanometers, and even angstroms, thus, providing a high degree of accuracy.

X-ray diffractions of the MWCNT-COOH and Poly[MWCNT/ester] composites are shown in Figure 6. The diffraction patterns having large diffraction peaks resulting from the nanoscale, suggest enhanced crystallinity. The sharp and strong diffraction peaks of MWCNT-COOH at $2\theta=26.6^\circ$and 45.45° correspond to the hexagonal graphite structure support [16] in Figure 6(a). In Figure 6 (b and c) it would appear that the intensity of the Poly[MWCNT/HQ] is sharper than that of the Poly[MWCNT/CA]. This behavior may indicate that the crystallinityof Poly[MWCNT/HQ] is higher than Poly[MWCNT/CA] due to angle strain. The pattern of Poly[MWCNT/EG] is the highest crystalline due to straight-chain.

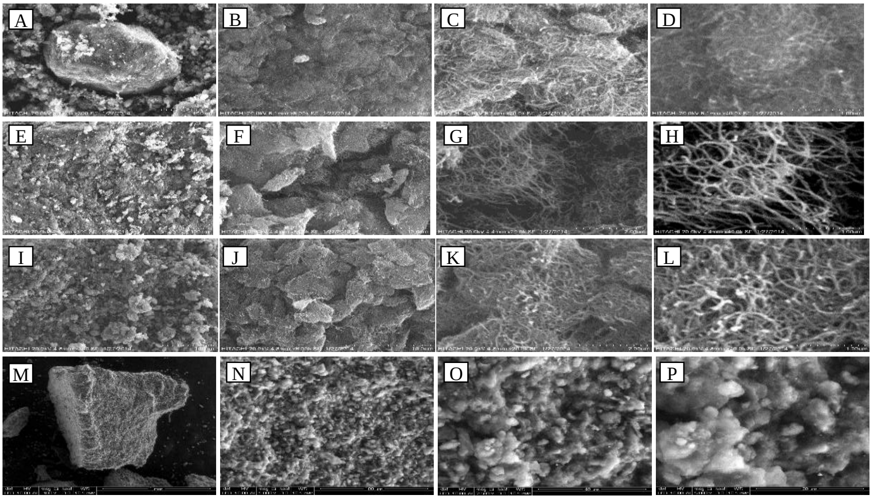

Figure 5: SEM microphotograph of MWCNT-COOH and Poly[MWCNT/ester] composites: (a) MWCNT-COOH (×300), (b) MWCNT-COOH (×5000),(c) MWCNT-COOH (×20 000), (d) MWCNT-COOH (×40 000) (e) Poly[MWCNT/HQ] (×300) (f) Poly[MWCNT/HQ] (×5000), (g) Poly[MWCNT/HQ] (×20 000), (h) Poly[MWCNT/HQ] (×40 000), (i) Poly[MWCNT/CA] (×300), (j) Poly[MWCNT/CA] (×5000), (k) Poly[MWCNT/CA] (×20 000), (f) Poly[MWCNT/CA] (×40 000), (m) Poly[MWCNT/EG] (×100), (n) Poly[MWCNT/EG] (×1000), (o) Poly[MWCNT/EG] (×2500), (P) Poly[MWCNT/EG] (×5000).

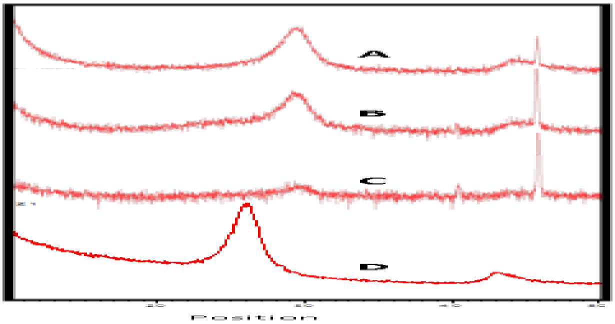

Figure 6: X-ray diffraction of MWCNT-COOH and Poly[MWCNT/ester] composites: (a) MWCNT-COOH, (b) Poly[MWCNT/HQ], (c) Poly[MWCNT/CA] and (d) Poly[MWCNT/EG].

3.6 Thermal properties: [Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC)]

The TGA (TG and DTG) and DSC curves recorded for the MWCNT-COOH and Poly[MWCNT/ester]composites are given in Figures 7 and 8. These curves show the characterization and comparison of the thermal decomposition behaviour of the MWCNT-COOH and their poly-composites at 10 °C min^{-1} of the heating rate under nitrogen. In general, the curves show consecutive steps for almost uninterrupted weight losses in the sequential decomposition of these four compounds over the experimental temperature range (25-1000 °C). Therefore, the T_{DTG} peaks are determined for all steps in the decomposition sequence with a maximum rate of weight loss in their DTG curve, and the peaks T_{DSC} are observed only for some steps in the DSC curves.

The thermal decomposition process of these four compounds may be describe as follows: In general, it was observed that the MWCNT-COOH was more stable than their poly-composites; and the order of thermal stability is MWCNT-COOH, Poly[MWCNT/HQ], Poly[MWCNT/CA] and Poly[MWCNT/EG].

The Poly[MWCNT/HQ] starts with two rapid and consecutive steps of mass losses (steps l and 2) followed by a slow bleed of mass loss (step 3 and step 4). The Poly[MWCNT/CA] decomposes rapidly in only two steps, one rapid step of mass loss (step l) followed by a slow bleed of mass loss (step 2). The Poly[MWCNT/EG] starts with two rapid and consecutive steps of mass losses (steps l and 2) followed by a very fast step of mass loss to evaporate all the material at 600 °C (step 3). On the other hand, the MWCNT-COOH decomposes only by a slow bleed of mass loss and rapidly in all steps (steps 1, 2, 3 and 4). These are summarized in Tables 4 and 5.

In conclusion, we noticed the highest thermal stability of MWCNT-COOH; such results are also related to the intra-hydrogen bond between carboxylic groups. The carboxyl group at the surface of MWCNTs grew to form a hexagon hydrogen bond net. This could enhance and increase the thermal stability of the material that is why decomposition temperature rose. The poly-composites that were prepared would make the COOH concentration disappear or reduce; furthermore, the terminal and unreacted carboxyl's group can create a little bit of H-bonded and decomposition temperature also obviously increased[17].

For comparison, in addition, we can say that the molecular weight and the staking of tubes factors play a key role in the thermal stability in the poly-ester composites; the order of thermal stability is MWCNT-COOH >Poly[MWCNT/HQ] >Poly[MWCNT/CA] > Poly[MWCNT/EG].

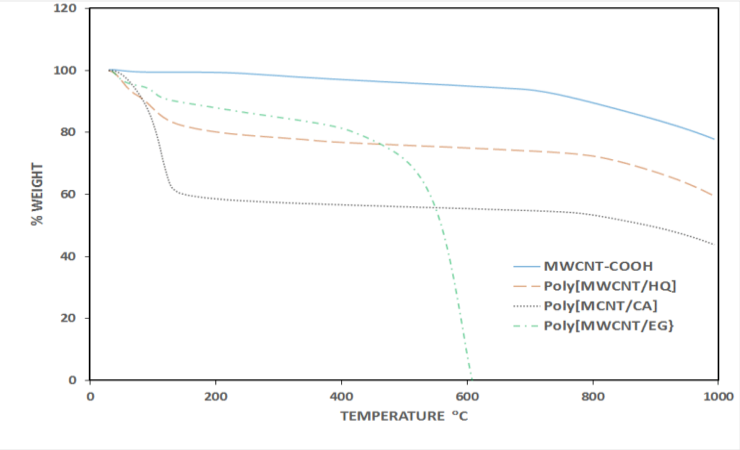

Figure 7: TGA curves of MWCNT-COOH and Poly[MWCNT/Ester] composites.

Table 4: %Weight loss at different temperature of the MWCNT-COOH and Poly[MWCNT/ester] composites.

Compound	% Wt Loss at					
	100°C	300°C	500°C	700°C	900°C	1000°C
MWCNT-COOH	0.57	1.30	1.63	2.10	10.9	6.02
Poly[MWCNT/HQ]	11.50	9.97	2.99	2.04	7.60	6.73
Poly[MWCNT/CA]	22.50	18.97	2.53	0.50	6.90	4.97
Poly[MWCNT/EG]	5.14	9.31	15.05		-	-

The differential scanning calorimetry (DSC) curves obtained for MWCNT-COOH and Poly[MWCNT/ester] composites are presented in Figures 8. The DSC curves of the four compounds show several peaks: endothermic peaks and exothermic peaks. All these peaks were associated with peaks in TGA in the same range, which corresponds to decomposition of the compound, Table 5. On the other hand, some peaks were not associated with peaks in TGA. This may be because of physical transformation, as glass transition.

Glass Transition Temperatures (T_g) of the MWCNT-COOH and Poly[MWCNT/ester] composites are shown in Table 6. It can be noticed that there are high variations in T_g for four compounds; these variations may be because of the ability to make Hydrogen bond. The highest T_g is for Poly[MWCNT/CA], whereas the lowest T_g is for Poly[MWCNT/HQ]. The Hydrogen bond could increase the cross-link density of the composites, thus increasing their glass transition temperatures.

Table 5: TGA and DSC results of the MWCNT-COOH and Poly[MWCNT/ester] composites.

Compound	Step	TGA				DSC		Res. %
		Wt. Loss %	T_i/°C	T_f/°C	T_{DTG}	T_{DCS}	Peak	
MWCNT-COOH	1st	0.78	33.46	149.99	52.54	31, 55	endo	99.22
	2nd	5.30	148.89	673.29	298.22	-	-	93.92
	3rd	8.19	673.23	880.34	753.55	*	*	85.73
	4th	8.30	880.17	1015.74	948.50	*	*	77.43
Poly[MWCNT/HQ]	1st	8.33	34.62	72.32	55.12	26, 66	endo	91.67
	2nd	12.19	80.03	134.61	101.80	130	endo	79.48
	3rd	2.93	141.71	514.56	355.94	360	endo	76.55
	4th	16.34	790.26	914.29	838.26	*	*	60.21
Poly[MWCNT/CA]	1St	44.99	39.41	197.19	59.55, 118.62	26, 84, 142	endo	55.01
	2nd	11.55	696.77	975.56	816.43	*	*	43.46
Poly[MWCNT/EG]	1St	5.13	36.24	80.19	48	30	exo	5.13
	2nd	4.58	82.19	128.93	104	129	endo	4,58
	3rd	90.29	465.00	631.66	664	*	*	0.00

endo; endothermic peak, exo; exothermic peak, *, out of the DSC range.

Table 6: Glass Transition Temperatures (T_g) for MWCNT-COOH and Poly[MWCNT/ester] composites.

Sample	T_g (°C)		T_m (°C)	
	Onset	Midpoint	Onset	Midpoint
MWCNT-COOH	232.49°C	233.65 °C	*	*
Poly[MWCNT/HQ]	153.01°C	160.25 °C	*	*
Poly[MWCNT/CA]	314.33°C	315.09 °C	*	*
	369.28°C	371.72 °C	*	*
Poly[MWCNT/EG]	152.00 °C	158.00 °C	165.7	167.4

* Out of range

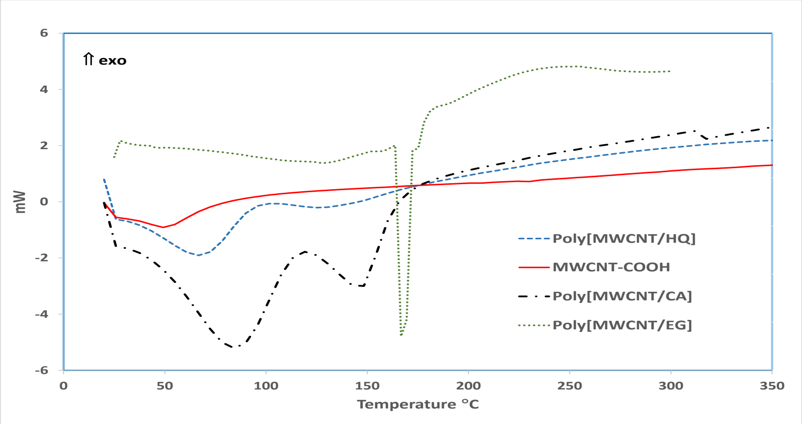

Figure 8: DSC curves of MWCNT-COOH and Poly[MWCNT/ester] composites

3.7 DC Electrical conductivity

The Poly[MWCNT/ester] composites consist of long chain-like molecules bonded by strong covalent and hydrogen bonds along the chains and only weak Van der Waals forces between the chains. If each repeat unit is considered a separate molecule, then the molecular orbitals, which are degenerated on each unit, will overlap to form a series of extended electronic states: the energy bands. Thus, the bonding and anti-bonding molecular unit orbitals lead to bulk valence and conduction bands, respectively. Consequently, the electronic properties of the π-electron system of the conjugated polymer or polymeric materials can be discussed in terms of band model with a characteristic bulk energy gap between the bulk valence band (B.V.B) and the bulk conduction band (B.C.B). The highest occupied molecular orbital (HOMO) is at the upper edge of the valence band. The edge of the conduction band is the lowest unoccupied molecular orbital (LUMO) supposedly at 0 K. Hence, polymers or polymeric is considered as organic semiconductors and the energy band theory can be used to characterize their electronic states and properties[18, ,19].

It is generally agreed that the mechanism of conductivity in the π-conjugated polymeric materials is based on the motion of charge defects within the conjugated framework. The charge carriers, either positive p-type or negative n-type, are the products of oxidizing or reducing the material, respectively. The following overview describes these processes in the context of p-type carriers although the concepts are equally applicable to n-type carriers[20,21].

The infinite conjugation chain on either side can migrate in either direction without affecting the energy of the backbone, provided that there is no significant energy barrier to the process.

Figure 9 shows DC electrical conductivity of MWCNT-COOH and Poly [MWCNT/ester] composites at room temperature. There is no so much enhancement of DC conductivity after functionalization because MWCNT-COOH has already a long chain with conjugation. The order of DC electrical conductivity is MWCNT-COOH/HQ > MWCNT-COOH/CA >MWCNT-COOH/EG > MWCNT-COOH ((5.5838E-06, 4.47656E-06, 4.06701E-06 and 1.7181E-06 S/cm), respectively). In addition, we can say that the co-planarity factor plays a key role in the DC electrical conductivity of Poly[MWCNT/ester] composites. However, the long chain with conjugation does not make a good factor to improve the DC electrical conductivity; and it is not significant.

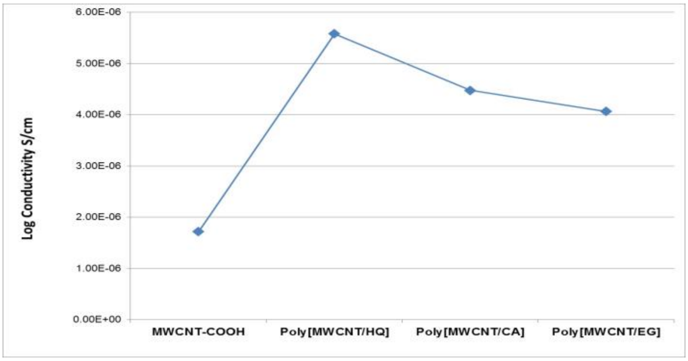

Figure 9: DC electrical conductivity of MWCNT-COOH and Poly[MWCNT/ester] composites at room temperature.

4 CONCLUSIONS

In summary, the Poly [MWCNT/ester] composites were synthesized by solution blending method from reacted MWCNT-COOH with (HQ, CA and EG).The obtained poly- composites were characterized by FT-IR, UV-Vis, XRD, TEM, SEM, TGA, DSC and DC electrical conductivity. The analysis of the FT-IR and UV-V provided an evidence for the formation of the poly-composites. X-ray diffraction confirms the crystallinity of the MWCNT-COOH and the poly [MWCNT/ester] composites. The formation of poly-composites on The MWCNT-COOH was confirmed by TEM and SEM. The MWCNT-COOH and the poly [MWCNT/ester] showed improvement in the thermal stability. The MWCNT-COOH were more stable than their poly-composites because of the intra-hydrogen bond between carboxylic groups. The thermal stability of the poly [MWCNT/ester] composites on the basis of the terminal and unreacted carboxyl group can create a little bit of H-bonded. The DC electrical conductivity of poly-composites increased slightly due to MWCNT-COOH which already has a long chain with conjugation. The long chain with conjugation does not constitute a good factor to improve the DC electrical conductivity of poly-composites.

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تحضير و تشخيص ودراسة التوصيل الكهربي المستمر لخلائط بولي [MWCNT/Ester]

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ملخص

تمتلك أنابيب الكربون النانوية خصائص كهربائية وميكانيكية وكهروميكانيكة ممتازة. عند إضافة أنابيب الكربون النانوية الى البوليمر لتكوين خلائط موصله كهربياً، تزداد التوصيلية الكهربية مع إضافة كميه قليله جداً من أنابيب الكربون النانوية. تهدف هذه البحث الى تحضير خلائط بوليمريه بتفاعل كربوكسي-متعدد أنابيب الكربون النانوية (MWCNT-COOH) مع مركبات ثنائي الهيدروكسيل وهي (الهيدروكينون والكاتيكول والإيثلين جلايكول) لتحضير خلائط متعدد الإستر. التحضير لهذ الخلائط تم بواسطة تقنية خلط السوائل. هذه الطريقة تتكون من ثلاث خطوات، عن طريق إذابة أنابيب الكربون النانوية في مذيب مناسب كذلك إذابة المركبات الأخرى في نفس المذيب، ثم يتم إضافته الى أنابيب الكربون النانوية حتى تتداخل المركبات مع أنابيب الكربون النانوية. في الخطوة الأخيرة، تترسب هذه الخلائط بواسطة تبخير المذيب.

هناك طرق عدة أُستخدمت لتشخيص الخلائط الجديدة المحضرة مثل (الأشعة تحت الحمراء والأشعة الفوق البنفسجية وانحراف اشعة إكس والميكروسكوب الإلكتروني النفاذ والميكروسكوب الإلكتروني الماسح والتحليل الوزني الحراري والماسح الكالومتري التفاضلي وكذلك قياس التوصيل الكهربي للتيار المستمر). نتائج ال FTIR وUV اثبتت تكوين الخلائط البوليميرية عن طريق تكوين روابط جديده. XRD اثبتت بلورية هذه الخلائط البوليميرية. SEM, TEM أكدت تكوين هذه المركبات. دراسة الخصائص الحرارية لهذه الخلائط اثبتت تحسين الإستقرارية الحرارية. بالنسبة للتوصيلية الكهربائي لهذه الخلائط تزداد قليلاً بسبب ان متعدد أنابيب الكربون النانوية لها سلسلة طويله في وجود اقترآن. زيادة السلاسل مع الاقتران لم تلعب دور هام في زيادة التوصيلية الكهربائية لهذه الخلائط. جميع النتائج السابقة اثبتت تحضير هذه الخلائط البوليمريه.

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