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Poly[MWCNT/Imidoselenium] nanocomposite: Preparation, Characterization and DC Electrical Conductivity

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Abstract

The chemical functionalization of amino multiwalled carbon nanotubes (MWCNT-NH2) by selenium dioxide (SeO2) was used to produce Poly [MWCNT/Imidoselenium] nanocomposite. The obtained poly-composite was characterized by FT-IR, UV-Vis, XRD, TEM, SEM, TGA, DSC and DC electrical conductivity. The characteristic peaks in FT-IR and UV-Visible spectra were found shifted to higher wave numbers, and the n-π^* bands were shifted to lower wavelengths (red shift), respectively, which confirmed the formation of imidoselenium bond in the Poly[MWCNT-N=Se] nanocomposite. Morphological characterization was done by using scanning SEM, TEM indicated a good dispersion phase in the poly-nanocomposite matrix. The Poly[MWCNT-N=Se] nanocomposite showed good thermal stability. The DC electrical conductivity of poly-nanocomposite was found to be 5.34×10^{-4} S/cm in ambient temperature. This is attributed to the interaction among the nanotubes.

Keywords: MWCNT-NH_2, Polymer Nanocomposites, Functionalization, Solution Blending, Imidoselenium.

1 INTRODUCTION

The huge forceful rising trend within the space of selenium containing polymers over the past decade due to the contribution of the many research fellows throughout the globe. Selenium has competed in not solely human health, however additionally the evolution of life, it's been found to be an important elemental semiconductor and studied as reason of its specific physical properties, high photoconductivity, piezoelectricity, thermoelectricity, and nonlinear optical responses [1-9]. Among chalcogens selenium is of importance because of its applications in electronics, in photovoltaics, and as superconductors [10].

The discovery of multiwalled carbon nanotubes (MWCNTs) and the subsequent discovery of single-walled carbon nanotubes by Iijima in 1991 and 1993, respectively, have spurred the growth and development of carbon nanotube related research [11, 12].

There has been significant growth in nanocomposites research since the first incorporation of carbon nanotubes in a polymer matrix by Ajayan and colleagues in 1994 [13]. Among the different classes of conductive fillers, carbon nanotubes (CNTs) have generated a great deal of interest due to their excellent physical, mechanical and functional properties. Carbon nanotubes (CNTs), have been applied to improve the mechanical properties, electrical and thermal conductivities of polymer composites due to their unique nanostructures and outstanding properties [11, 14].

Nanotubes can be classified into single-walled carbon nanotube (SWNT) and multi-walled structures (MWNT). A single-walled carbon nanotube may be viewed as a graphene sheet that is rolled into a nanoscale tube form. A multiwalled carbon nanotube consists of additional graphene coaxial tubes around the SWNT core [15, 16]. Scientists are attracted to the unique properties of nanotubes; shape, size and outstanding physical performance. Besides that, they possess a very high tensile strength, high-rise thermal stability and high electrical conductivity electrochemical in cylindrical shape with diameter around 10 nm and length reach to 100 nm [17]. Nanotubes have found applications in many sections sensors [18], nanocomposites [19, 20], molecular devices [21] or advanced materials with electronic properties [22]. Functionalization of CNTs chemically has been reported in the literature. Functional groups are attached by covalent and non-covalent bonds with nanotubes [23-26]. General methods usually use to functionalize the MWCNTs when dispersed in solution and the MWCNTs are redeposited on surface after the modification. In general, chemically modified MWCNTs is carried out to improve the solubility of the MWCNTs in various solvents [27].

In this paper, we report the synthesis, characterization and DC electrical conductivity Poly[MWCNT/Imidoselenium] nanocomposite with a linear backbone structure. This poly-nanocomposite was prepared by chemical functionalization of amino multi-walled carbon nanotubes (MWCNT-NH_2) with selenium dioxide (SeO_2). Characterization of Poly [MWCNT/Imidoselenium] nanocomposite was performed using FTIR, UV, XRD, SEM, TEM, DSC and TGA analyses. The DC electrical conductivity of the New poly-nanocomposite and the pristine MWCNT-NH_2 was measured at room temperature. The

New Poly [MWCNT/Imidoselenium] nanocomposite was prepared and studied as an attempt to investigate the effect of MWCNT/Imidoselenium linkages on structure-property relationship of the nanocomposite.

2 EXPERIMENTAL

2.1 Materials

Amino Multiwalled carbon nanotubes (MWCNT-NH_2) were brought from Chengdu Organic Chemicals company limited, Chinese Academy of Sciences (Timesnano) China, with values of d = 8 to 15 nm, and l ≈50 μm, 95 % and above was the Purity. The amino group coverage over nanotubes surface of the 0.45% wt. Selenium dioxide (SeO_2) and Tetrahydrofuran (THF, 99.9%) were brought from Sigma-Aldrich. Ethanol 96% was brought from Fluka and used as received without any extra treatment in this work.

2.2 Instrumentation

JASCO 410 FTIR Spectrophotometer was used for FT-IR using the KBr disc technique. An electrothermal melting point apparatus was used for measured the melting point. The thermal analyses, DSC and TGA , were carried out on a Mettler Toledo (DSC823e) analyzer, and a Mettler Toledo (TGA/SDTA851e) analyzer, respectively, in the range 23 to 1000 ºC under 20 ml/min of N_2 and a heating rate of 10 ºC/min. UV-Visible absorption spectra were measured by a (Specord 200), Analytik-Jana, Germany, in Dimethylformamide (DMF) (~10^{-4} mol/dm^3). Bruker Axs Da Advance (Germany) was used for the X-Ray diffraction. The measurements of DC Electrical conductivity were using a Keithley (Model 6487) Picoammeter/Voltage Source, via a double probe which is locally fabricated conductivity bridge cell. The Scanning Electron Microscope was taken on a (SEM HITACHI S-3400N). The Transmission Electron Microscope (TEM) was performed on a Phillips CM-12 (USA), the materials were arranged by Leica ultracut UTC ultramicrotome (JEOL) with a 100 kV as accelerating voltage (Japan).

2.3 Preparation of Poly[MWCNT/Imidoselenium] nanocomposite

Selenium dioxide (0.005 g) was dissolved in 10 ml ethanol and then added to a mixture of MWCNT-NH_2 (0.3 g) dispersed in 15 ml THF. The reaction mixture was stirred for overnight under reflux. After cooling to ambient temperature (r.t.), then was filtered and washed several times with THF under vacuum through a 0.22 μm membrane. Finally, the filtered solid was dried in a vacuum oven at 90 °C.

3 RESULTS AND DISCUSSION

3.1 Characterization

Solution blending method was employed to prepare the Poly [MWCNT/Imidoselenium] nanocomposite. The poly-nanocomposite was synthesized by reacting MWCNT-NH_2 with SeO_2 in THF under reflux. Figure 1 shows the synthesis

of Poly[MWCNT/Imidoselenium] nanocomposite from a reaction of MWCNT-NH_2 with SeO_2 in THF solvent. Table 1 summarizes the physical properties of MWCNT-NH_2 and Poly [MWCNT/Imidoselenium] nanocomposite, namely, percentage yield, color, melting point, and solubility in polar solvents.

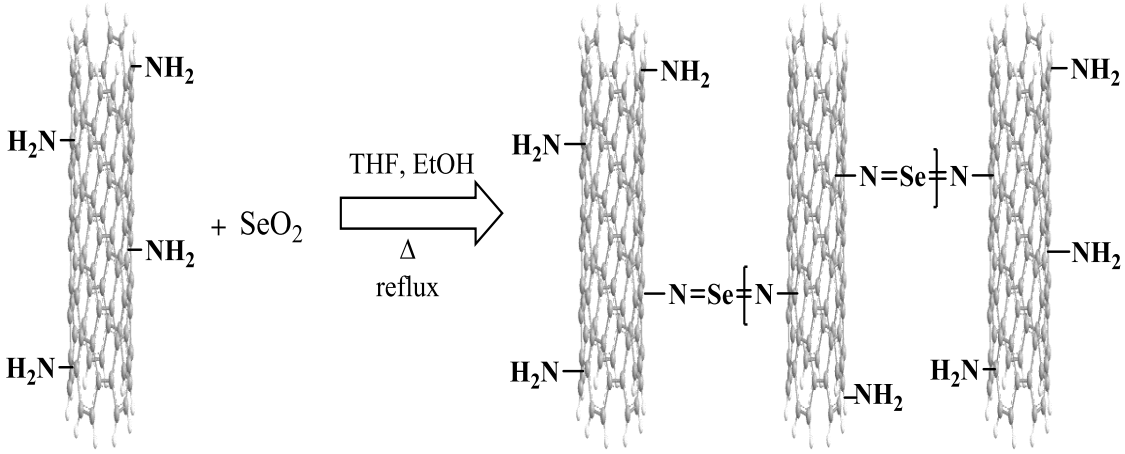

Figure 1. Synthesis of Poly[MWCNT-N=Se] nanocomposite.

Table 1. Physical properties of MWCNT-NH_2 and of Poly[MWCNT-N=Se] nanocomposite.

No	Compound	% Yield	Color	M.P.	Solubility		
					DMSO	DMF	EtOH
1	MWCNT-NH_2	-	Black	> 350°C	++	+	-
2	Poly[MWCNT-N=Se]	71 %	Black	> 350°C	++	+	-

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

3.2 IR Spectra

Figure 2 displays the FTIR spectra of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite. The IR spectrum of MWCNT-NH_2 showed an intense peak at 3448 cm^{-1} which is attributed to the presence of NH_2 group. The γ C–N bond stretching vibrations present at 1050 cm^{-1}, and the peaks at 1578-1654 cm^{-1} can attributed to the C=C bond present in the MWCNT structural backbone [28, 29].

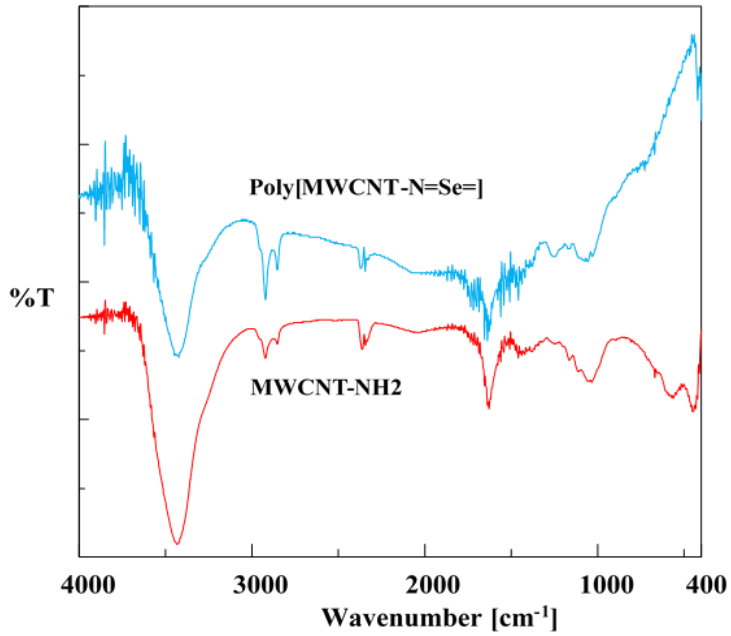

Figure 2. FT-IR spectra of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite

After functionalization of MWCNT-NH_2 with SeO_2, FTIR spectrum of the Poly[MWCNT-N=Se] nanocomposite also showed an NH_2 peak at 3448 cm^{-1}. This could be due to the presence terminal unreacted amine groups in the nanocomposite matrix. Table 2 summarizes the main FTIR absorption bands of the MWCNT-NH_2 and synthesized Poly[MWCNT-N=Se] nanocomposite.

Table 2. The main FTIR Bands of the MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite

Compound / Bands (stretch)	MWCNT-NH_2 (cm^{-1})	Poly[MWCNT-N=Se] (cm^{-1})
N-H	3450	3448
C=C	1578	-
C-N	1050	-
N=Se=N	-	-

3.3 UV-Visible Spectroscopy

The electronic spectra of MWCNT-NH_2 and its Poly[MWCNT-N=Se] nanocomposite is shown in Figure 3, UV-visible absorption spectra for samples were recorded in

solvent such as DMF and THF, respectively. The MWCNT-NH_2 and the Poly[MWCNT-N=Se] nanocomposite showed mainly two bands in the range 200 – 800 nm. The MWCNT-NH_2 sample showed a sharp peak at λ_{max} 280 nm and a broad band at 324 nm which are attributed to π-π* and n-π^* transitions, respectively. On the other hand, the spectra of the Poly[MWCNT-N=Se] nanocomposite showed a broader band due to the π-π* transition at λ_{max} 278 nm which was found similar to the electronic transition of the MWCNT-NH_2. However, the n-π^* transition band for Poly[MWCNT-N=Se] nanocomposite showed up at a lower frequency, λ_{max} 330 nm (a red shift). The change in the shape and the position of the absorption bands confirmed the formation of imidoselenium bond in the Poly[MWCNT-N=Se] nanocomposite. Table 3 summarizes the main absorptions bands of MWCNT-NH_2 and the Poly[MWCNT-N=Se] nanocomposite.

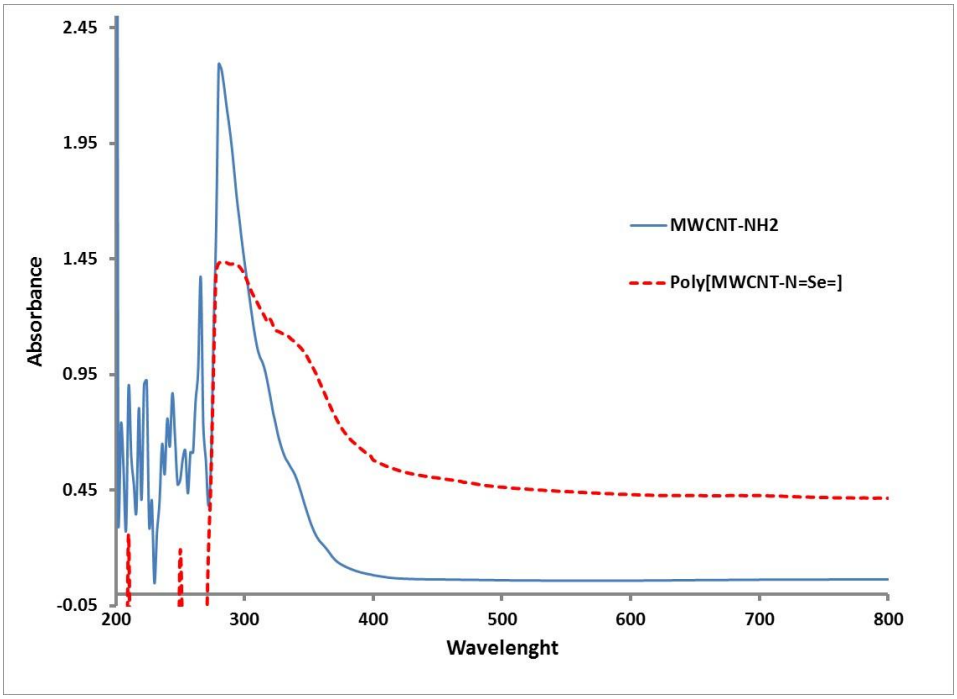

Figure 3. Electronic absorption spectra of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite.

Table 3. Electronic absorption bands of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite in DMF.

No.	compound	π-π^* λ_{max} (nm0	n-π^* λ_{max} (nm)
1	MWCNT-NH_2	280	324
2	Poly[MWCNT-N=Se]	278	330

3.4 Microscopic Characterization (TEM, SEM)

3.4.1 Transmission Electron Microscopy (TEM)

The transmission electron microscopy (TEM) allows qualitative understanding about the internal structure, spatial distribution of the various phases. Figure 4(a-d) displays TEM images of the MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite at different magnifications. In Figure 4, a and b presents TEM images of MWCNT-NH_2, while c and d presents TEM images of Poly[MWCNT-N=Se] nanocomposite. The TEM images of the Poly[MWCNT-N=Se] nanocomposite in Figure 4 (c and d) showed a relatively good dispersion phase and more entangled polymer matrix compared to that of the pristine MWCNT-NH_2. This may be due to the imidoselenium bonds in Poly[MWCNT-N=Se] nanocomposite structure, which make tight crossed links between at least two nanotubes (Figure 4(D)) [30].

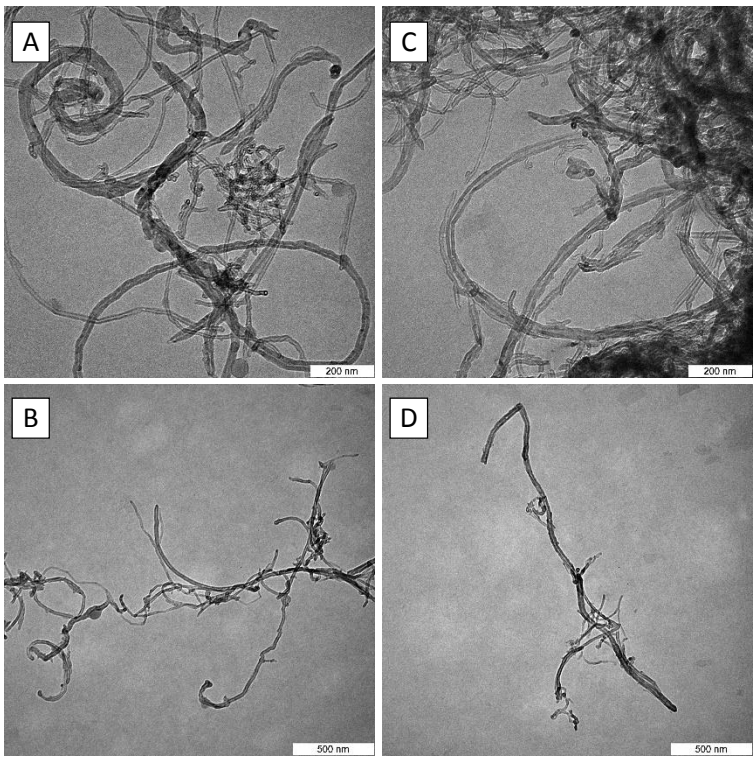

Figure 4. TEM images of MWCNT-NH_2: (a; ×50000, b; ×100000) and Poly[MWCNT-N=Se]nanocomposite: (c; ×50000, d; ×100000).

3.4.2 Scanning Electron Microscopy (SEM)

The morphology of the samples was examined by the scanning electron microscopy, SEM. Figure 5(a-h) shows SEM images of the surface morphology and dispersion of

the MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite with change magnifications. It can be seen clearly from the SEM images that the surface morphology of Poly[MWCNT-N=Se] nanocomposite is remarkably different compared to that of MWCNT-NH_2 [31]. MWCNT-NH_2 has a larger mass than the Poly[MWCNT-N=Se] nanocomposite.

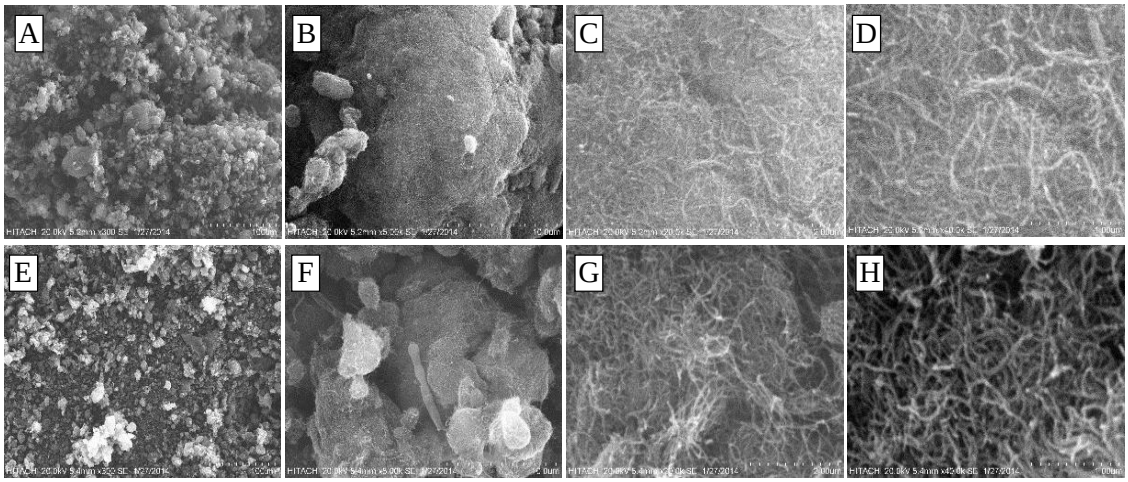

Figure 5. SEM images of MWCNT-NH_2: (a; ×300, b; ×5000, c; ×20 000 and d; ×40 000) and Poly[MWCNT-N=Se]nanocomposite: : (e; ×300, f; ×5000, g; ×20 000 and h; ×40 000).

3.5 X-Ray Diffraction (XRD)

X-ray diffraction was performed to study the crystalline structures of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite. Diffraction pattern of MWCNT-NH_2 was characterized by a peak (strong) at $2\theta = 25.9^{\circ}$ which is attributed to the graphite-like structure. It can be seen from Figure 6 that crystallinity increases upon addition of SeO_2 to MWCNT-NH_2 [32-34].

3.6 Thermal properties

Figures 7 and 8 show the TGA, DTG and DSC curves on record for the MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite, respectively. The degradation processes of MWNT-NH_2 the poly-nanocomposites are summarized in Tables 4 and 5, respectively.

The degradation process of Poly[MWNT-N=Se] nanocomposite and MWNT-NH_2 exhibits one-step in the range (819-1000 ºC), at T_{DTG} 954 ºC, and in the range (860-915 ºC), at T_{DTG} 915 ºC, respectively. This might be attributed to the presence of the strong imidoselenium bonds in Poly[MWCNT-N=Se] nanocomposite backbone. Moreover, the wt % loss decreases from 8.3 to 2.3 % for Poly[MWNT-N=Se] nanocomposite and MWNT-NH_2, respectively. This indicates that the order of thermal stability of the Poly[MWCNT-N=Se] nanocomposite is greater than that of MWCNT-NH_2 [35-37].

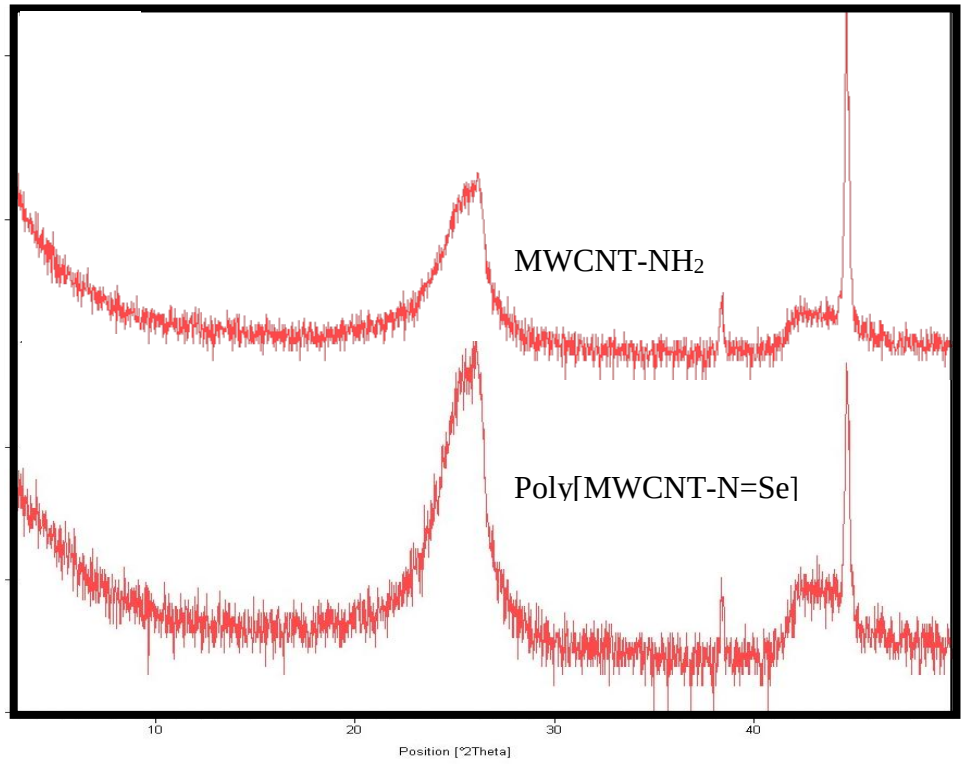

Figure 6. X-ray diffractograms of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite.

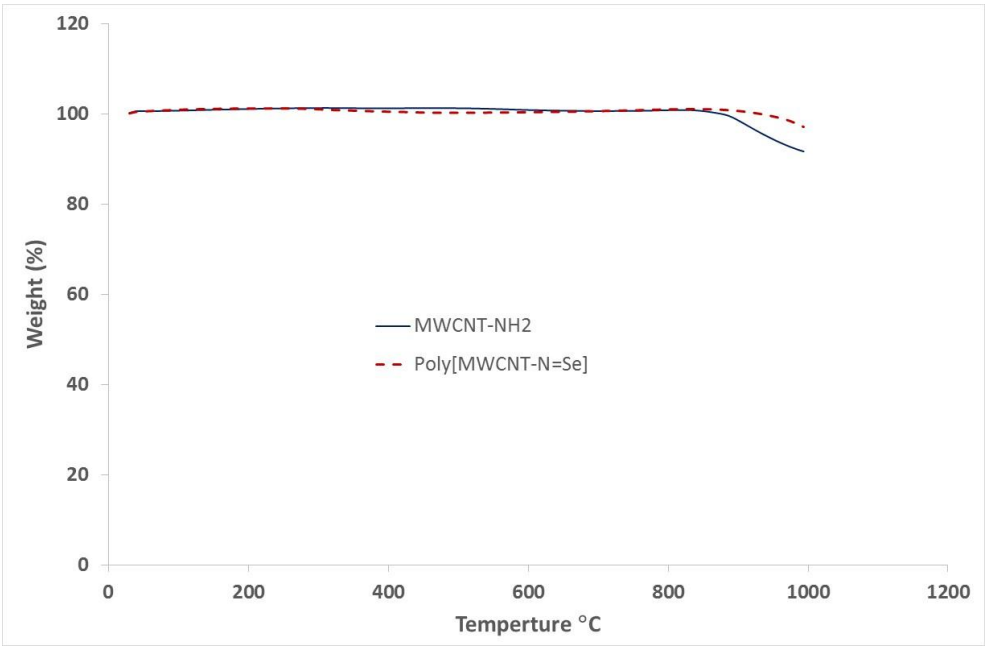

Figure 7. TGA thermograms of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite.

Table 4. Thermal characteristics of materials.

Compound	Step	TGA				DSC		Res. %
		Wt. Loss %	T_i/°C	T_f/°C	T_{DTG}	T_{DCS}	Peak	
MWCNT-NH_2	1st	8.3	860	994	915	-	91	91.7
Poly[MWCNT-N=Se]	1st	2.3	819	1000	954	-	-	97.7

Table 5. % Weight loss at different temperatures for MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite.

Compound	% Wt Loss at					
	100°C	300°C	500°C	700°C	900°C	1000°C
MWCNT-NH_2	0	0	0	0	2	7
Poly[MWCNT-N=Se]	0	0	0	0	0	2.80

3.7 Electrical Characterization of Materials

The frequency dependences of the DC conductivity of the materials (MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite) at room temperature is shown in Figure 9. The DC electrical conductivity values of the Poly[MWCNT-N=Se] nanocomposite and MWCNT-NH_2 were equal to 2,29 E-04 and 5.34 E-04 S/cm, respectively. A two-fold increase in DC electrical conductivity of Poly[MWCNT-N=Se] nanocomposite was observed which may be due to the imidoselenium links between the nanotubes which led to the higher molecular weight of the Se-complex and good stacking ability of the nanotubes in the composite [38, 39].

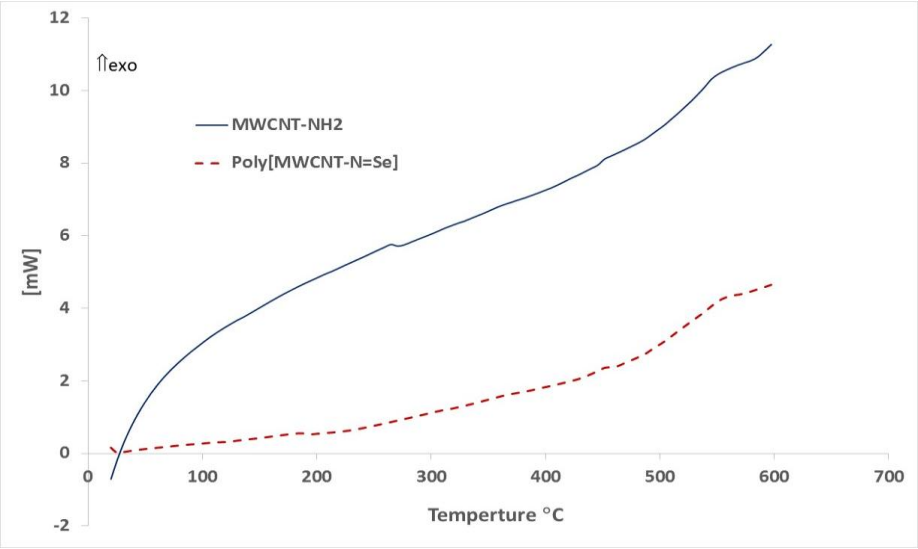

Figure 8. DSC thermograms of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite.

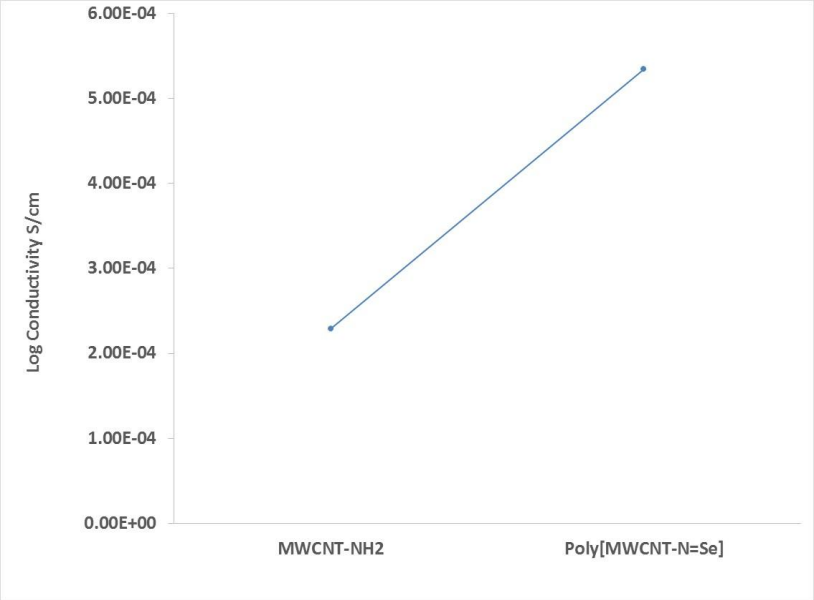

Figure 9. The DC electrical conductivity of MWCNT-NH_2 and Poly[MWCNT-N=Se] nanocomposite at room temperature.

4 CONCLUSIONS

Poly[MWCNT/Imidoselenium] nanocomposite was synthesized by reaction of MWCNT-NH_2 with SeO_2 via a simple solution blending method. The obtained poly-nanocomposite was characterized by FT-IR, TEM, SEM, UV-Vis, XRD, TGA, DSC, and DC electrical conductivity. The Poly[MWCNT/Imidoselenium] nanocomposite showed an increased crystallinity, higher thermal stability and higher DC electrical conductivity compared to MWCNT-NH_2, due to the linear structure of the poly-nanocomposite which in turn allowed stacking of the nanotubes in the polymer matrix.

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