Research Article

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Characterization of PVC/MWCNTs Nanocomposite: Solvent Blend

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Abstract: Polyvinyl Vinyl Chloride (PVC) multiwall carbon nanotubes (MWCNTs) nanocomposite flexible films were prepared using the solvent blend technique. Chloroform (CHCl_3) and tetrahydrofuran ($(\text{CH}_2)_4\text{O}$) were used as solvents for MWCNTs and PVC, respectively. The effect of the solvents' blend on electrical, optical and thermal properties of PVC/MWCNTs were investigated. The results of the Raman spectrum showed that all the characteristic bands of PVC polymer have a slight shift due to addition of MWCNTs. Electrical results showed that the nanocomposite samples with chloroform volume ratios of 10% and 25% had nearly the same conductivity. This is attributed to the formation of the MWCNTs network, which assisted in electrical conductivity. The I-V hysteresis curve decreases as the temperature increases and as it approaches the glass transition temperature. The non-isothermal kinetics analysis for PVC and PVC/MWCNTs were investigated by Thermogravimetry Analysis (TGA) using the model-free kinetic method. The non-isothermal measurements were carried out at five heating rates of 5 to 40°C/min. The results show that the main decomposition process has constant apparent activation energies for all samples. The use of the bi-solvent method has improved the dispersion of untreated MWCNTs, and this has been reflected on the stability of both electrical and thermal properties.

Keywords: Multiwall carbon nanotube MWCNTs, PVC, Polymer nanocomposite, solvent blend, Kinetic analysis

1 Introduction

In recent years, carbon nanotubes (CNTs) have received much attention, and they are widely used in potential applications ranging from large-scale structures in automobiles to nanometer scale electronics. Because of their remarkable properties, carbon nanotubes are used in a variety of applications individually or in nanocomposite, such as cement [1], ceramics [2] and polymer composites [3, 4]. Polymer nanocomposites based on CNTs are of great interest in both research and industrial applications. Carbon-based polymers are used in many applications, such as energy storage [5–9], chemical sensors [10–12], electromagnetic interference shielding [13–16] and electronics [17, 18]. The pristine CNTs are present in the form of bundles and ropes due to their geometry and Van der Waals and inter-tube forces, which limit its solubility in many common solvents. Therefore, the solubility of the CNTs represents one of the considerable issues in applications. This is because the reproducibility of the material's properties strongly depends on the degree of the dispersion of filler within the product. Therefore, one of the requests is to produce a stable CNT suspension for long enough to prepare homogeneous film. This also must be verified without affecting the properties of the filler or the host material. However, this problem can be solved with different techniques, such as the chemical functionalization of CNTs [19–21], ultrasonic oscillation [22–24], surfactants [22, 25, 26] and immiscible polymer blends [27–29]. Some other techniques such as non-covalent functionalization can be used to modify carbon nanotubes.

Fangwei Pan *et al.* have modified PVC/MWCNTs nanocomposite by coating MWCNTs with covalent and non-covalent N-epoxypropyl PPTA (PPTA-ECH) to prepare PPTA-ECH-MWNTs-NH₂-x and PPTA-ECH-MWNTs-x before adding as filler to reinforce PVC nanocomposite films. They found that, for PPTA-ECH-MWNTs-NH₂-x/PVC, mechanical properties were highly modified in comparison to pure PVC. Amir Abdolmaleki *et al.* [30] have investigated the physicochemical features of PVC/MWCNTs functionalized with riboflavin (RIB). Their results conclude that there is a higher mechanical and thermal behavior for 12wt%

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PVC/(RIB) MWCNTs compared to other concentrations of MWCNTs. The modification of the CNTs' dispersion in the PVC matrix using oleic acid and methyl oleate as the dispersing agent has been investigated by Katarzyna *et al.* [31]. The obtained samples demonstrated higher stability and a lower rate of agglomeration.

Several studies have been conducted to improve the dispersion of fillers through polymers by using a solvent mixture or so-called solvent blends. Li *et al.* [32] have investigated the effect of a solvent mixture composed of tetrahydrofuran and chloroform on the dispersion of hydroxyapatite filler in Polylactide. They found that the solvent mixture has a significant influence on the degree of dispersion of filler. They attribute these results to the difference in polarity of both solvents. Opoku *et al.* [33] have studied the effect of non-halogenated binary solvents in various ratios on conjugated polymer pre-aggregation in the solution state, morphology and the microstructure of polymer thin films cast from these solvent blends. High-performance top-gate/bottom-contact organic field effect transistor was demonstrated in this work. Zhao *et al.* [34] have investigated the effect of bi-solvent, tetrahydrofuran/isopropyl alcohol (THF/IPA) and *o*-xylene/1-phenylnaphthalene on the power conversion efficiencies (PCEs) of state-of-the-art organic solar cells (OSCs). They found an increase of PCEs to over 13%. Morgan *et al.* [35] have illustrated the effect of the solvent blend on clay exfoliation in polystyrene (PS) polymer. They observed that both the energy of sonication and solvent blending play important roles in the preparation of PS clay nanocomposites. Sen *et al.* [36] have used the solvent blending method to prepare MWCNTs/PS nanocomposite film. They examined the interaction between polystyrene and well-dispersed MWCNT using different techniques. The effects of solvent blends on the suspension stability of BaTiO₃ nanoparticles was investigated by Kim [37]. Similar dispersion levels were obtained for BaTiO₃ dispersion stability in both solvent mixtures (toluene/*n*-butanol and nonazeotropic toluene/ethanol).

In the present study, we prepared Polyvinyl Chloride (PVC) nanocomposite filled with multiwall carbon nanotubes (MWCNT) using two different solvents (Chloroform (CHCl₃) and Tetrahydrofuran (C₄H₈O)) with different volume ratios of solvents, 5%, 10% and 25%, by using the casting technique. The characterization such as surface morphology, Raman spectroscopy and thermal stability and kinetic analysis of the prepared samples was investigated.

2 Model-Free Kinetics Analysis

Some polymer decomposition curves involve several steps, with different decomposition mechanisms and then different activation energies. The apparent activation energy (E) is expected to be a function of both temperature (T) and conversion (α). According to Piloyan *et al.* [38], the kinetic model equation can be expressed as

$$\frac{d\alpha}{dt} = Af(\alpha) \exp\left(\frac{-E}{RT}\right) \quad (1)$$

Where α is the conversion, A is the pre-exponential factor, E is the activation energy (kJ/mol), R is the gas constant (kJ mol⁻¹ K⁻¹), t is the time (min) and T is the temperature (K). Hence

$$\ln\left(\frac{d\alpha}{dt}\right) = \ln(A) + \ln[f(\alpha)] - \frac{E}{RT} \quad (2)$$

$$\left[\frac{d \ln\left(\frac{d\alpha}{dt}\right)}{dT^{-1}}\right] = \left[\frac{d \ln(k(T))}{dT^{-1}}\right] + \left[\frac{df(\alpha)}{dT^{-1}}\right] = -E_a/R \quad (3)$$

In equation (3), the term $\left[\frac{df(\alpha)}{dT^{-1}}\right] = 0$ since $f(\alpha)$ does not depend on temperature. However, the present investigation does not require any information of the reaction model (model-free approach). The values of E can be estimated from $\ln(d\alpha/dt)$ vs. $1/T$ relation.

3 Materials and Experimental Work

3.1 Materials

PVC (average molecular weight 3000) was obtained from Saudi Basic Industries Corporation (SABIC). Multiwall carbon nanotubes (MWCNTs) were purchased from Chengdu Organic Chemicals Co. (China) and used as received without any physical or chemical treatment (diameter >50 nm, length 10–20 mm, and purity >95%). Chloroform and Tetrahydrofuran were purchased from Sigma Aldrich.

3.2 Sample Preparation

A fixed mass of MWCNTs and PVC (0.45wt.% MWCNTs to PVC) were dissolved separately in chloroform and THF solvents, respectively. Both solutions were aggressively mixed by magnetic stirring at room temperature for 24 hours to reach a homogenous solution. Different volume ratios (5%,

Figure 1: LSM pictures of 0.45wt.% PVC/MWCNTs samples prepared by bi-solvent method

Table 1: Samples code and solvent volumetric ratios used in sample preparation

Sample code	Chloroform	Tetrahydrofuran	Vol.% chloroform/THF
S1	1.25 ml	23.75 ml	5
S2	2.50 ml	22.50 ml	10
S3	5.00 ml	20.00 ml	25

10% and 25% of chloroform to THF) were used for the sample preparation (Table 1). The resultant mixtures were ultrasonicated for 20 minutes and then blended again with magnetic stirring at room temperature overnight. The final solution of mixtures was poured in a glass Petri dish (diameter of 5 cm). The solvent was evaporated at fume hood for 48 hours and heated inside an oven at 45°C for 2 hours.

3.3 Optical Characterization

Raman spectroscopy was performed to investigate the interactions' nature between PVC and MWCNTs in the resultant films. The optical properties were determined with UV-vis Shimadzu (METTLER TOLEDO, USA) spectrophotometry.

3.4 Electrical Characterization

Electrical measurements were carried out using the 4200-SCS Semiconductor Characterization System (KEITHLEY Co.) USA. Samples were shaped into square pieces with an area (A) of about 2.0 cm² and a thickness (d) of 0.1 to 0.12 mm and placed between two copper electrodes.

3.5 Thermal Characterization

Decomposition kinetics for PVA and PVC/MWCNTs were carried out non-isothermally by TGA, using TA instrument "TGA Q50". All measurements were carried out with a flow of nitrogen gas (60 mL/min) and sample weight of about 13 mg for each run. Five non-isothermal runs were achieved for each sample at linear heating rates of 5, 10, 20, 30 and 40°C/min.

4 Results and Discussion

4.1 Laser Scanning Microscopy

Laser scanning microscopy (LSM) was used to investigate the rate of agglomeration at the sample surface. It is known that MWCNTs have the ability to float near the surface and form agglomeration due to Van der Waals force and electrostatic force. The LSM pictures for the three samples are represented in Figure 1. The agglomeration rate decreased by increasing the chloroform volume ratios, as shown in Figure 1. It is clear that the adsorption of chloroform by MWCNTs reduced the agglomeration formation, which can be attributed to the difference in the dielectric constant and viscosity level of both solvents.

4.2 Raman Spectroscopy

Normalized Raman spectra of PVC and PVC/MWCNTs nanocomposites were prepared using different solvent ratios (10 and 20% vol.% of chloroform), as shown in Figure 2. The spectra showed the main characteristic band of PVC (listed in Table 2), and two characteristic bands (D band and G band) for MWCNTs are also presented. As mentioned in many references [23], the G band is due to the sp²-bond vibration emitted by the carbon atoms in the

Table 2: The main peaks of PVC in Raman spectrum

Raman Shift (cm ⁻¹)	vibration modes	Raman Shift (cm ⁻¹)	vibration modes
361	C-Cl in trans HCIC = CHCl in-plane bending mode	636.5	C-Cl in Cl ₂ C = CHCl stretching mode
695	C-Cl in H ₂ C = CHCl symmetric stretching mode	961	C-Cl in H ₂ C = CHCl asymmetric stretching mode IR
1100	C-O stretching	1179	CH ₂ twist
1330	CH bend, out of phase IR + CH ₂ deformation	1431	CH ₂ , bend, in phase IR
1100	C-O stretching	1255	CH-rocking vibration

Figure 2: Normalized Raman spectra of PVC and PVC/MWCNTs nanocomposite

two-dimensional hexagonal network. While D-band, is attributed to the dispersion of hybrid carbon *sp*³ defects in the hexagonal structure of the MWCNTs wall.

In Figure 2, C-Cl vibration of PVC presented at 694 and 635 nm. Also, Alkyl (CH_n) and D band indicated in 1431 and 1330 cm⁻¹, respectively. G band of MWCNTs is located at 1590 cm⁻¹. It can be observed that there is a little shift of PVC characteristic bands which indicating that, there is a weak interaction occurred between carbon nanotubes and host polymer chains. This means that the solvent mixture method disperse the carbon nanotubes by isolation and capsulation technique, such as the surfactant, and latterly by chemical functionalization or reaction.

4.3 Electrical Characterization

Figure 3 shows the IV characteristic curves at different temperatures for the pure PVC sample. It is obvious that the current values gradually increase as the temperature increases, while there is no significant increase after the poly-

Figure 3: I-V Characteristic curves of pure PVC at different temperatures

mer approaches its glass transition temperature (about 85°C) [39]. The maximum current at 100°C is less than 1 μA.

The characteristic curves for PVC/MWCNTs nanocomposite samples (S2 and S3) at different temperatures (60 and 90°C) are represented in Figure 4. It is clear that both samples S2 and S3 have nearly the same electrical behavior, which indicates that the dispersion of MWCNTs is the same for both samples. Noticeably, the current level for PVC/MWCNTs is higher than that of the pure PVC sample. This is attributed to the uniformly dispersed MWCNTs for both S2 and S3 composite samples. By considering sample S2 as a representative sample for the prepared nanocomposite, one can note that the values of the electric current increase with temperature to more than 10 μA at 100°C (refer to Figure 5).

It is noted that the symmetry in the shape of the I-V curve around the y-axis (zero voltage) increases as the temperature increases for both pure and nanocomposite

Figure 4: I-V Characteristic curves for S2 and S3 nanocomposites at 60°C and 90°C

Figure 6: Cyclic I-V curves for S2 (10 vol.% chloroform) nanocomposites at 70°C and 100°C

Figure 5: I-V Characteristic curves for S2 (10 vol.% chloroform) nanocomposites at different temperatures

Figure 7: $\ln(I)$ versus $1000/T$ for S2(10 vol.% chloroform) nanocomposite

samples (as it approaches the glass transition of the samples). Measurements were carried out by applying $-200V$ and voltage increases gradually in steps to reach $+200V$. The curve asymmetry around y-axis (below T_g) may be attributed to opposite surface charge accumulated near the electrodes. Near T_g , the chain mobility increases due to chains segmental motion, which prevents such surface charge from accumulating.

Figure 6 illustrates the current-voltage cycles at 70°C and 100°C for sample S2. It is clear from the figure that, above the glass transition temperature, the hysteresis area was finished. This was attributed to the increases of chain mobility (previously discussed). The trajectory of electrical hysteresis, shown in Figure 6, takes the form of butterfly wings, and it is a well-known phenomena in many prop-

erties [40–43]. This butterfly shape is produced due to the presence of delay or lag between the applied driving parameter and sample response.

The activation energy for the nanocomposite sample was predicted by drawing the relation between $\ln(I)$ and $1000/T$ at a constant voltage value. In the present work, we selected 50V and the Arrhenius relation represented in Figure 7. The calculated activation energy was found to be 0.163eV for the nanocomposite, which is less than the one that corresponds to pure PVC (about 0.4eV).

Figure 8: a) TGA and b) DTGA curve for PVC and PVC/MWCNTs nanocomposite (S2 and S3)

4.4 Thermal Stability

PVC decomposed through two stages: the first stage, which is the main decomposition stage, is attributed to the dehydrochlorination process. The formation of the conjugated polyene structure followed to dehydrochlorination process and is represented as a shoulder in the first decomposition stage. The second stage is consistent with the thermal cracking of the carbonaceous conjugated polyene. Figure 8 illustrates the first stage of decomposition (TGA and its first derivative DTGA curve) for pure and nanocomposite samples at a heating rate of 10°C/min and under N₂ gas. This stage (first stage) of decomposition occurs in the temperature range between 225°C and 375°C degrees.

Figure 9: Variation of conversion (α) with temperature during first decomposition stage for pure PVC and PVC/MWCNTs nanocomposite samples at different heating rates

Figure 10: Variation of $\ln(da/dt)$ with $1000/T$ during first decomposition stage for pure PVC and PVC/MWCNTs nanocomposite samples at different heating rates

It is clear that the addition of MWCNTs with this weight ratio (0.45wt%) improves the thermal stability of PVC. The DTGA peak position indicates a shift of about 7 degrees for PVC/MWCNTs composite samples.

In this temperature range (for the main decomposition stage), it was observed that all samples lost approximately 62.5% of their weight. This result shows that the bi-solvent method succeeded in distributing the MWCNTs distinctly with a little interaction with the host polymer. The increase in the thermal stability of the nanocomposite samples at this stage can be attributed to the role of MWCNTs in absorbing HCl and thus contributing to reducing the number of polymer chains that may be decomposed by the action of HCl [44]. In addition, the presence of homogeneously distributed MWCNTs between the polymer chains acts to isolate these chains, which leads to a delay in PVC decomposition by HCl [44].

4.5 Model-Free Kinetic Analysis

Using equation (2) and neglecting $\ln f(\alpha)$, the relation between $\ln(da/dt)$ versus $1000/T$ can be used to predict both thermal activation energy (E) and the pre-exponent factor (A) under such conditions. This was carried out for the main reaction stage, and by comparing the kinetic parameters, one can investigate the effect of dispersion of MWCNTs on the thermal behavior of the samples. Figure 9 shows the conversion - temperature curves ($\alpha(T)$) for the first decomposition stage of pure and MWCNTs nanocomposite samples at different heating rates, 5, 10, 20, 30 and 40 °C/min. $\ln(da/dt)$ data were calculated after converting temperature to time and then numerically differentiating the $\alpha(t)$ curves.

Figure 10 represents the dependence of $\ln(da/dt)$ on $1000/T$ at different heating rates. Assuming that the activation energy of decomposition does not depend heavily on the value of the conversion factor, we can consider the peak points ($\ln(da/dt)_{peak}$ and $1/T_{peak}$) and apply the Arrhenius equation to calculate the apparent activation energy (as represented in Figure 11).

The value of the apparent decomposition activation energy for the pure sample represents the greatest value compared to other nanocomposite samples. Note that there is no significant change in E_a between nanocomposite samples (S2 and S3). The maximum change in activation energy between pure and nanocomposite samples does not exceed 14%. This confirms that the dispersion of MWCNTs was mainly due to the presence of the solvent mixture but not due to interaction between MWCNTs and the host polymer. The dispersion of MWCNTs can be at-

Figure 11: $\ln(d\alpha/dt)_{\text{peak}}$ versus $1000/T_{\text{peak}}$ (1st decomposition stage) for PVC and PVC/MWCNTs nanocomposite samples

Table 3: Apparent activation energy (E_a), for pure and nanocomposite samples, at different values of conversion (α)

Conversion	Activation Energy (kJ/mol)		
	S0 (PVC)	S2 (10 vol.% CF)	S3 (25 vol.% CF)
0.2	84	78	89
0.3	82	77	85
0.4	91	73	75
0.5	94	71	72
0.6	99	74	71
0.7	-	79	76
Average	90	75	78

tributed to the surfactant-like behavior of the solvents' mixture. Both solvents have nearly the same polarity and are miscible in each other but vary in degree of viscosity and dielectric values. All of these factors result in a good dispersion of dissolved carbon nanotubes in chloroform through the polymer solution based on THF solvent mixed with the volume fraction of chloroform (less or equal 25 vol.%).

The apparent activation energy (E_a) at specific values of conversion (α) can be calculated from the slope of the linear relation between $d(\ln(\alpha))/dt$ vs $1000/T$. The predicted values at different values of conversion are represented in Table 3. The values of E_a confirm the above results and pure PVC has the greatest value of E_a , while both nanocomposite samples have nearly the same activation energy.

5 Conclusion

Solvent blends or the solvent mixture technique was used to enhance the dispersion of untreated MWCNTs in PVC host polymer. Tetrahydrofuran and chloroform were used as solvents for PVC and MWCNTs, respectively, in different volume ratios (5%, 10% and 20% of the chloroform solvent volume ratio). Aggregations of MWCNTs were found for the sample prepared with a percentage of 5 vol.% chloroform. The other two samples (10% and 20 vol.% of chloroform) showed a marked improvement in the dispersion state, and this was reflected on both electrical and thermal properties. The results of Raman spectroscopy showed that there was no strong interaction between MWCNTs and PVC, and all the characteristic bands of PVC and MWCNTs were detected. I-V characteristic curves showed an increase in the electrical current of the nanocomposite samples with respect to the pure sample. The formation of MWCNT net-

works represents the main reason for the higher conductivity and lower activation energy of the nanocomposite samples. The values of the activation energies of pure PVC and its MWCNT nanocomposite samples were calculated by using the model-free method. The values of E_a confirm the above results, and pure PVC has the greatest value of E_a , while both nanocomposite samples have nearly the same activation energy. The results showed that the dispersion of the untreated MWCNTs has improved, so this method can be used in the preparation of thin films without any treatment (chemical or physical) to the nature of CNTs.

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