Investigation of the Effect of CNT on Spectroscopic, Thermal and Electrical Conductivity Properties of PVC-PMMA Blend

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Summary: In this study, the effect of carbon nanotubes (CNT) on the spectroscopic, thermal and electrical properties of poly(vinyl chloride)-poly(methyl methacrylate) (PVC-PMMA) nanocomposite prepared by adding 0.1, 0.5, and 1% multi-walled carbon nanotubes (MWCNT) was investigated. For this purpose, nanocomposites containing 60% PVC were prepared by the solution casting method using tetrahydrofuran (THF) as solvent. Scanning electron microscopy (SEM) imaging showed that the polymer matrix had sufficiently homogeneous distribution of CNTs and that they were in good interaction. X-ray diffraction (XRD) analyses showed that the nanocomposites had an amorphous structure with approximately 15% crystallinity and CNT addition did not change the amorphous structure. From TGA analyses, CNTs had no effect on the thermal stability of the nanocomposites. The nanocomposites decomposed in three steps, similar to PMMA-PVC blends. The first stage of decomposition started at 194 °C, the second stage at 224 °C and the third stage at 349 °C. The decomposition ended at around 474 °C with a mass loss of approximately 90%. According to the data obtained from differential scanning calorimeter (DSC) analyses, although the CNT addition increased the glass transition temperature of the nanocomposites, it did not have a major effect. The Tg of the PMMA-PVC blend and nanocomposites was determined to be between 62 and 66 °C. The electrical conductivity curve showed that the nanocomposites reached a value close to the ideal percolation curve by increasing the CNT amount, and it was evaluated that CNT addition at rates above 1% may convert the nanocomposites into semiconductors.

Keywords: Nanocomposite, PMMA-PVC blend, Spectroscopic and thermal properties, Electrical conductivity.

Introduction

Nanocomposites are advanced structures developed in cases where polymers alone are not sufficient or to expand their areas of use. Factors such as the nature of the polymer matrix, pretreatments such as compatibilization or functionalization, loading level, aspect ratio, length/diameter ratios of carbon nanotubes (CNT), and use of surface-active agents are effective in determining the properties of nanocomposites.

In a study based on viscometric measurements for the preparation of a miscible blend of PMMA and PVC, tetrahydrofuran was used as a solvent. It was revealed that these two polymers could be mixed at all ratios of PMMA/PVC, and temperature did not affect the mixture except for the 40:60 ratio [1]. Abdelghany et al. [2] performed FTIR, UV and XRD measurements on the PVC/PMMA blend prepared with THF solvent using the solution casting technique and suggested that the blend was miscible at all ratios and exhibited an amorphous structure.

Ramesh et al. [3] investigated the miscibility of blends prepared with PVC, PMMA and PEO (polyethyleneoxide). In their study, viscometric and differential scanning calorimetry (DSC) analyses showed that these two polymers were miscible. The existence of attractive forces between the polymers was evaluated according to Sun theory by determining a parameter from the viscometric data. Viscometric and thermal analyses showed that these two blends were miscible. This miscibility of PVC/PMMA and PVC/PEO blends was explained by the hydrogen bonding interaction between the chlorine atoms in PVC and the oxygen atoms in PMMA and PEO polymers. Chakrabarti et al. [4] suggested that the properties of blends such as PMMA/PVC blends could be explained by specific interactions such as H-bonding and dipole-dipole interactions between the molecules of the constituent polymers. They stated that PVC and PMMA can be mixed up to 60% by weight [5,6].

Nanocomposites are composites that have a phase formed by nanotubes, nanoparticles or nanolaminar structures. Like all composites, they are multiphasic. Their distinguishing feature is that the nano-sized phases they carry are at least in the 10-100 nm size range. Nowadays, nanocomposites have been developed to overcome the limitations of different

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engineering materials and provide useful alternatives. Nanocomposites can be classified on the basis of matrix and dispersed phase materials [7]. Saeed and Khan [8] determined that the PMMA/PVC nanocomposite prepared by the solution casting method with the addition of functionalized multi-walled carbon nanotubes (MWCNT) was fully miscible. Jager et al. [9] investigated the compatibility of polymers in PMMA/PVC blends. Their studies showed that these two polymers can be mixed at all ratios at low temperatures, while the miscibility decreases at high temperatures. Belhaneche and Bedda [10] conducted a study about the structure-property relationship of PVC-PMMA blends. For this purpose, blends with compositions varying between 0 and 100% by weight were prepared. Physicochemical characterizations of the samples were carried out by DSC and Fourier Transform infrared spectroscopy (FTIR). Analysis of thermograms showed polymer miscibility up to 60% by weight PMMA. This miscibility was suggested to be due to a special interaction of the hydrogen bond type between the carbonyl groups (C = O) of PMMA and the hydrogen from the groups of PVC (CHCl). Wang et al. [11] examined various mechanical properties of the PMMA/MC (methyl cellulose) blend they prepared using scanning electron microscopy (SEM) images and x-ray diffraction (XRD) data. They found that the brittleness of PMMA in thin films can be eliminated by increasing the MC mixture ratio.

As stated by Dixit et al. [12], a miscible polymer blend should exhibit a glass transition (Tg) temperature depending on a single composition between two pure components. In the studies, Tg was measured as 63.2 ℃ for 50:50 wt% PVC PMMA blend.

The effect of matrix type on the electrical conductivity of MWCNTs was investigated by Tohmassin et al. [13]. The researchers determined that carbonyl groups interact well with the cyclic structure of nanotubes. They reported that this interaction is also positive for morphology and electrical conductivity. The electrical conductivity provided by CNTs in polymer nanocomposites can be explained by the percolation theory. The nanomaterial concentration that initiates electrical conductivity in a nanocomposite with an insulating matrix is called the percolation threshold. The insulating matrix dominates the formation in nanocomposites below the percolation threshold [14].

Chakrabarti et al. [4] investigated the effect of blending PMMA with suitably stabilized and plasticized PVC in various ratios with reference to physical, mechanical and thermal properties. In order to find a suitable explanation for these properties, the resulting morphologies of various blends were also studied. In their study, thermogravimetric comparison of PVC/PMMA blend with pure PVC showed that the thermal stability of the PVC/PMMA blend was better than pure PVC and the thermal decomposition temperature increased with increasing ratios of PMMA at 90:10 and 70:30 PVC/PMMA ratios. In their study, the blends exhibited two phase morphology, but it was also observed that the boundary region of the phases was not very sharp. Phase homogeneity was observed at low PMMA ratios and not at increasing PMMA amounts. They suggested that the properties of blends like PMMA/PVC blend could be explained by specific interactions such as H-bonding and dipole-dipole interactions between the molecules of the constituent polymers.

Sangawar and Moharil [15] studied the electrical, thermal and optical band gaps of polypyrrole-filled PVC: PMMA thin films. From the thermogravimetric (TGA) curves, the decomposition temperature increased from 269 ℃ to 273 ℃. In their studies where they examined the electrical, thermal and optical band gaps of polypyrrole-filled PVC: PMMA thin films, the X-ray diffractograms of all samples revealed amorphous structure. Naim et al. [16] studied various properties of PVC/MWCNT nanocomposite with 0.45% fixed ratio MWCNT in chloroform/THF solvent mixtures prepared with 5, 10 and 25% ratios. They stated that PVC decomposed in two stages, and the first stage, which is the main degradation stage, can be attributed to dehydrochlorination. This stage of decomposition (first stage) occurred between 225 ℃ and 375 ℃ degrees.

Nasr et al. [17] reported that mixing PMMA with PVC using the cosolvent casting method resulted in a broad characteristic peak of PMMA at $2\theta = 16.4°$ with a shoulder at $2\theta = 22.5°$, revealing the amorphous phase of PMMA. The intensity of these characteristic peaks decreased after the addition of PVC. It was suggested that the PVC/PMMA blend shifted the Bragg peaks at $2\theta = 16.4°$ and $2\theta = 22.5°$ to Bragg peaks at 17° and 25.3°, revealing the change in crystallographic organization.

The electrical conductivity provided by CNTs in polymer nanocomposites can be explained by percolation theory. The nanomaterial concentration that initiates electrical conductivity in a nanocomposite which has an insulating matrix is called the percolation threshold. The insulating matrix dominates the formation in nanocomposites below the percolation threshold [14].

Experimental

Materials and Reagents

PMMA, low molecular weight PVC, 6-13 nm × 2.5-20 µm, >98% CNTs and 99.9% purity tetrahydrofuran (THF) were supplied by Sigma-Aldrich. Dathan MS-MP8 was used as magnetic stirrer and Lab Companion Ultrasonic Cleaner was used as ultrasonic bath. Sigma 300 FESEM electron microscope was used for SEM imaging, and RIGAKU ULTIMA IV X-Ray Diffraction Spectrometer was used for XRD analysis. In XRD analysis, samples were placed on the device with scanning angle: 3-90°, scanning speed: 10°/min, wavelength: Cu Kα (λ=1.5405 Å), the desired angle was set to 3-90° and the analysis was started. The analysis was concluded by giving the intensity spectrum graph corresponding to the 2θ angle. In TGA/DTG measurements, analyses were performed on the Hitachi STA 7300 TGA/DTA device in the range of 20-500 °C in a nitrogen environment and at a heating rate of 10 °C/min. A Perkin Elmer Diamond DSC device was used. DSC analyses were performed in the air environment at a heating rate of 10 °C/min in the range of 0-100 °C. DSC graphs were drawn using the Origin Pro 8.0 program based on data sets. The alternating polarity method was used for electrical conductivity measurements with a Keithly Model 6517A Electrometer. The electrical conductivities of the blend and nanocomposites were calculated with the following equation. Percolation theory is expressed with an equation given below

$$\sigma \approx \sigma_o (P - P\_c)t$$

In this equation, σ_0 is a constant, P symbolizes the weight fraction of CNT, Pc shows the weight fraction of CNT that corresponds to the percolation threshold, and t is an exponential factor depending on the conductivity of doping material, resistance of contact, and web topology [18].

Preparation of PVC/PMMA Blends

Nanocomposites were prepared by the solution casting method of Nasr et al. [17]. Four separate polymer blends were prepared from 1 g PVC/PMMA solid mixtures in the ratio of 60:40 (600 mg PVC/400 mg PMMA).

Preparation of CNT-Doped PVC/PMMA Nanocomposites

Three separate 1 g PVC/PMMA blends were dissolved in 30 mL of THF by stirring on a magnetic stirrer for two hours. CNT proportions corresponding to 0.1, 0.5 and 1% were dispersed in 5 mL of THF each in an ultrasonic bath at a temperature range of 24-30 °C for half an hour.

After the CNT dispersions were carefully added to the polymer blend solutions, the new mixtures were homogenized in an ultrasonic bath for two hours. The composite dispersions taken from the ultrasonic bath were mixed in a magnetic stirrer for one hour and then carefully poured into petri dishes. The nanocomposite dispersions poured into petri dishes were dried at room temperature for 24 hours and nanocomposite films were obtained. Sample codes of the obtained blend and nanocomposites are given in Table 1

Table-1: Sample codes of blend and nanocomposites.

CNT (%)	**%0 CNT**	**%0.1 CNT**	**%0.5 CNT**	**%1 CNT**
Numune Kodu	**PVC60-0**	**PVC60-0.1**	**PVC60-0.5**	**PVC60-1**

Results and Discussion

Polymeric nanocomposites, which are obtained by dispersing nanoscale fillers in a polymer matrix in order to improve mechanical, thermal and electrical conductivity properties, expand the areas of application for polymeric materials. In the investigation of the properties of PVC-PMMA blend containing 60% PVC and PVC-PMMA nanocomposites with 0.1, 0.5 and 1% multiwalled carbon nanotube additives, SEM, XRD, TGA, DTA, DTG, DSC and electrical conductivity analyses were performed. The presence of multiwalled carbon nanotubes and the effect of different ratios on the polymer blend were investigated. The SEM images obtained in the PVC/PMMA blend studies conducted by Chuayjuljit et al. [19] appear to be consistent with the images obtained in this study. In Fig. 1a, the composite has a relatively smooth surface and there is no phase separation indicating the good miscibility of PVC and PMMA. As shown in Fig. 1b-c, as the amount of CNT increased, the morphologies of the sample dramatically changed and CNT was homogenously disturbed throughout the composite. However, when the amount of CNT was 1.0%, as shown in Fig. 1d, CNT aggregated on the composite, most probably due to the interaction between CNT being dominant and as a result, CNT was not homogenously distributed throughout the composite.

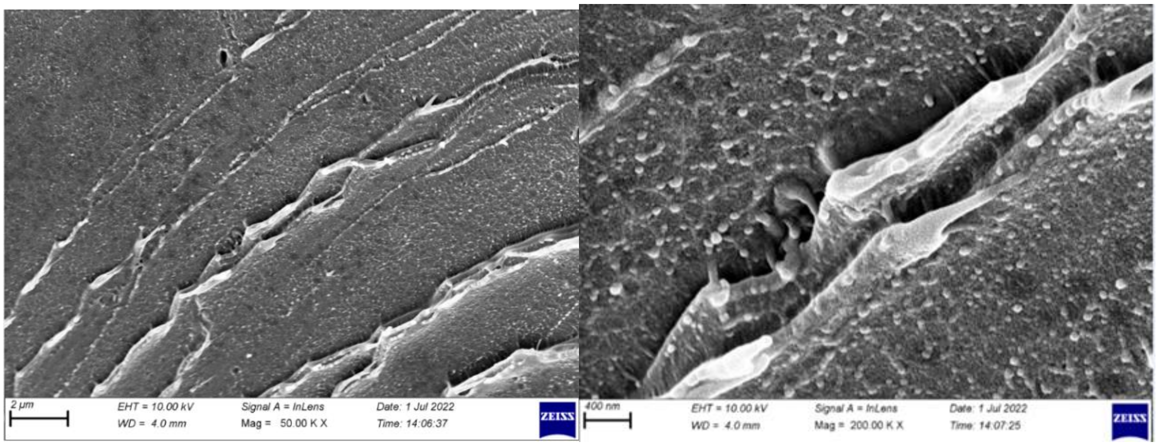

Fig. 1a: SEM Images of PVC60- 0 SEM with different magnifications (50.000x-200.000x).

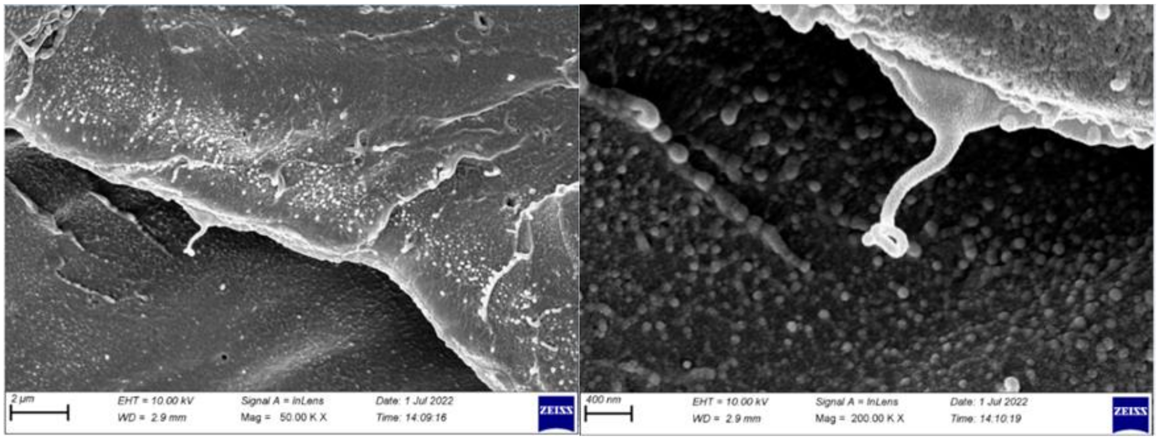

Fig. 1b: SEM Images of PVC60- 0.1 SEM with different magnifications (50.000x-200.000x).

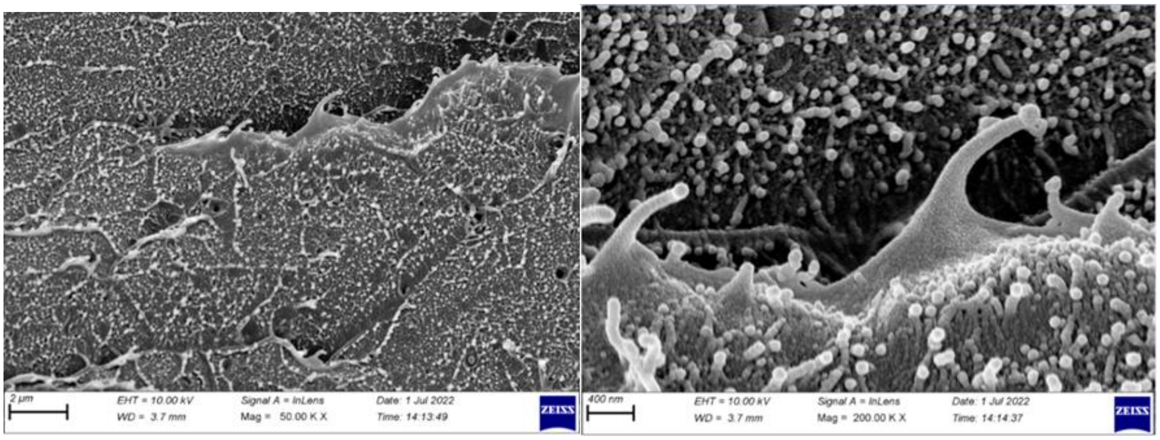

Fig. 1c: SEM images of PVC60- 0.5 SEM with different magnifications (50.000x-200.000x).

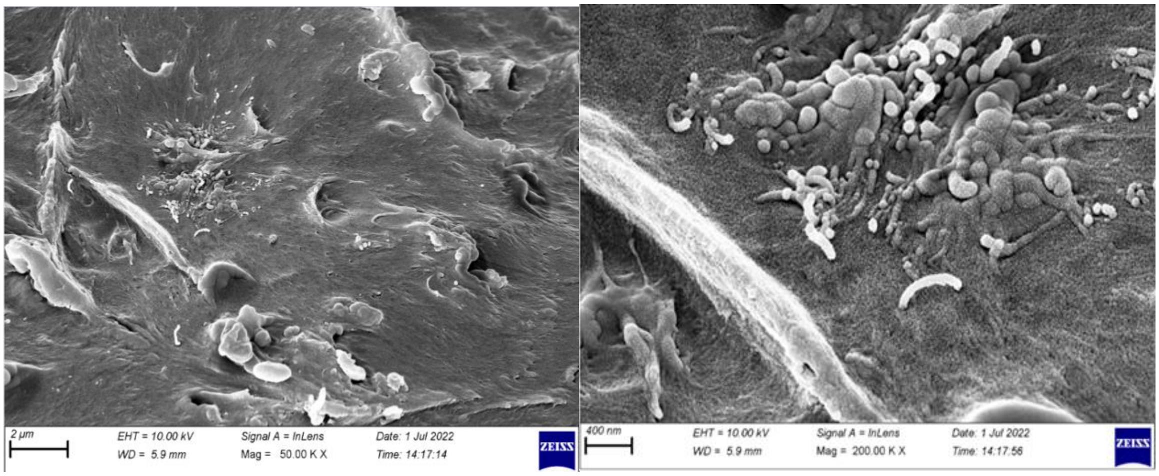

Fig. 1d: SEM Images of PVC60-1 SEM with different magnifications (50.000x-200.000x).

In XRD analysis, the general structure was amorphous (Fig. 2). This observation is supported by Sangawar and Moharil [15] and El Gamal and El Sayed [20]. The peaks obtained are compatible with the 2θ values of the blend prepared by Nasr et al. [17]. The signals for the PVC/PMMA blend with CNT additive were preserved, although they partially shifted in some regions (Fig. 2).

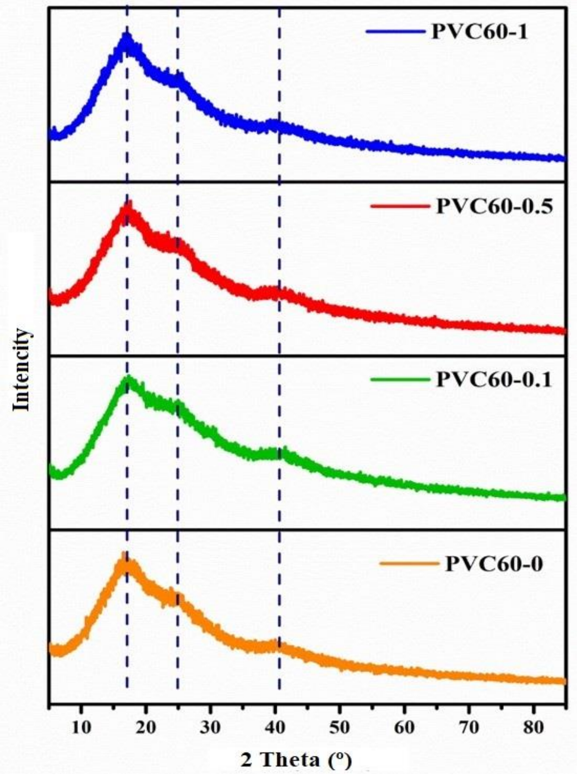

Fig. 2: XRD patterns of the synthesized blend and nanocomposites with different ratio of CNT.

CNT addition generally showed decreasing crystallinity at 0.1% and 0.5% rates, and crystallinity increased again at 1% CNT (Table 2). This situation can be attributed to the electrostatic interaction and van der Waals forces or micromechanical interlocking between CNT and the polymer matrix when there is no chemical bond (since CNT used in the study is not functionalized, this possibility can be neglected) [21]. As seen in Table 1, CNT addition did not cause a significant change in the amorphous structure. In the study of El Sayed [22], the Tg values were found to be 61.0, 59.0 and 59.3 °C with MWCNT added to the PMMA/PVC blend at 0.25%, 0.5% and 1%, respectively. These values show that CNT addition did not create a systematic difference in the PMMA/PVC/MWCNT nanocomposite.

Table-2: 2θ and crystallinity values of the synthesized blend and nanocomposites with different ratio of CNT.

	2 θ	Crystallinity %
PVC60-0	16.52 23.86 40	15.69
PVC60-0.1	17.34 24.82 40.02	14.97
PVC60-0.5	17.6 24.5 40.32	15.11
PVC60-1	16.98 25 39.22	15.70

DSC analysis showed that CNT doping increased Tg values slightly (Fig.3). Considering the weak interaction between the polymer matrix and CNT, the small increases in Tg can be attributed to the weak interaction between the polymer matrix and CNT and the decrease in the free volume of the CNT. Despite this, no correlation was found between Tg values of samples. The highest Tg was about 66.31 ºC in the samples. Tg results according to DSC and DTA analyses are shown in Table-2. The glass transition temperature data obtained from DTA plots (Fig4) are compatible with the glass transition temperature data obtained from DSC analyses (Table-3).

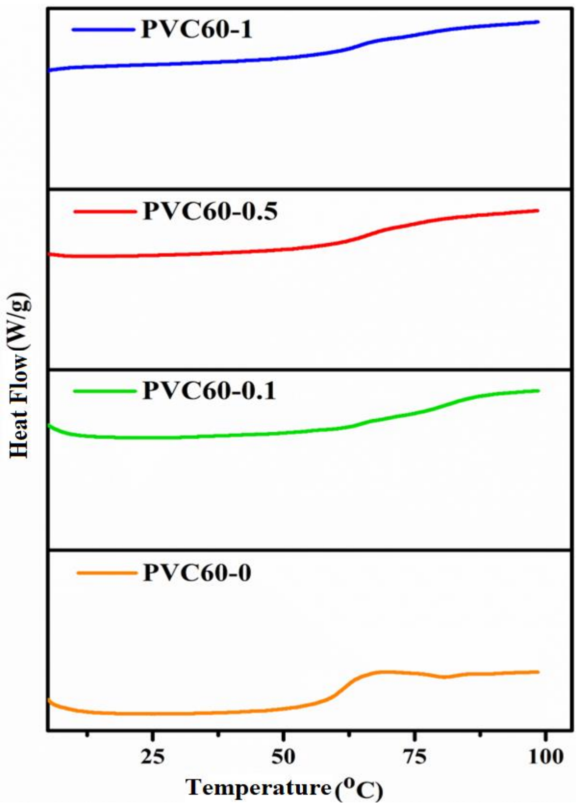

Fig. 3: DSC curves of the synthesized blend and nanocomposites with different ratio of CNT.

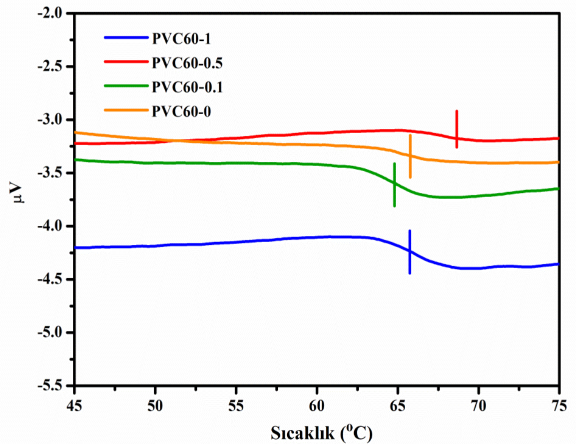

Fig. 4: Tg values obtained from DTA curves of blends and nanocomposites with different ratio of CNT.

Table-3: Tg values of the synthesized blend and nanocomposites with different ratio of CNT.

Sample Code	Tg (°C) (Obtained from DSC)	Tg (°C) (Obtained from DTA)
PVC60-0	62.58	65.72
PVC60-0.1	64.14	64.64
PVC60-0.5	66.31	67.58
PVC60-1	64.26	65.82

As stated by Dixit et al. [12], a miscible polymer blend should exhibit a single composition-dependent glass transition temperature between the two pure components. The observation of a single glass transition temperature for the blend and its nanocomposites in DSC analysis showed that PVC and PMMA are fully miscible. Belhaneche-Bensemra et al. [23] observed a single Tg value in miscible systems in their studies with PVC-PMMA blends, while two different Tg values were observed in blends containing more than 50% PMMA. Dixit et al. [12] measured Tg as 63.2 °C for a 50:50 PVC-PMMA blend by weight. The glass transition temperature of the PVC-PMMA blend containing 60% PVC by weight was measured as 62.58 °C by DSC, and this value is consistent with the literature.

A three-stage mass loss was observed in the TGA analysis (Fig. 5). The first-stage mass loss is thought to be due to solvent, impurities, and dehydration of trapped moisture. This observation is supported by El Sayed [20] and Ramesh et al. [24]. Two major degradation curves were observed in TGA analysis. The first of these curves is thought to be mostly due to dehydrochlorination that occurs due to the degradation of PVC. The second stage mass loss was observed between 224-349 °C in PVC-PMMA blends and nanocomposites (Table 4). In the study conducted by Naim et al. [16], the first decomposition occurred between 225 °C and 375 °C. In the third step, the mass loss reached a maximum at 473 °C. This step is considered to be the stage where the polymer matrix disperses into dimers, trimers and oligomers. In DTG analyses, two separate endothermic peaks were observed around 300 °C and 400 °C (Fig. 6). These peaks coincide with the temperatures at which the first and second mass losses occur in the TGA analyses. The results obtained from TGA showed that CNT addition did not cause any significant change in the decomposition temperatures and mass losses of the PVC-PMMA blend. There is no effect on thermal stability due to weak interactions, such as electrostatic and van der Waals, between the polymer matrix and CNT. Functionalization of CNT can be used to provide homogenization and to form hydrogen bonds with the polymer matrix. The increase in thermal stability of functionalized CNT compared to pure CNT can be attributed to this second reason.

Table-4: TGA data of the synthesized blend and nanocomposites with different ratio of CNT.

Numune Kodu	T_1 (°C)	Weight Loss %	T_2 (°C)	Weight Loss %	T_3 (°C)	Weight Loss %	T_f (°C)	Maximum Weight Loss %
PVC60-0	194.23	5.08	349.16	47.77	442.48	84.87	473.25	90.84
PVC60-0.1	194.14	6.11	349.39	48.75	442.76	85.23	473.46	90.89
PVC60-0.5	194.64	5.28	350.17	47.25	443.46	82.48	474.19	88.28
PVC60-1	194.12	5.68	349.50	48.12	442.96	83.43	473.77	89.81

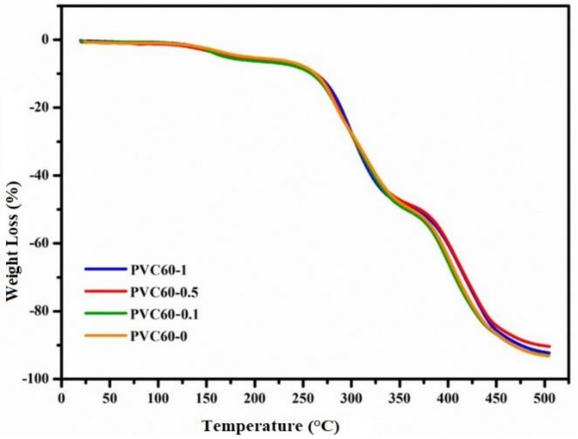

Fig. 5: TGA curves of the synthesized blend and nanocomposites with different ratio of CNT.

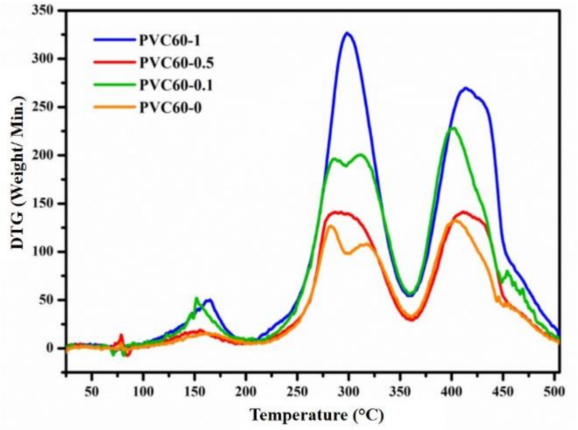

Fig. 6: DTG curves of the synthesized blend and nanocomposites with different ratio of CNT.

In the electrical conductivity measurements of PVC-PMMA blend containing 60% PVC and nanocomposites with CNT added, the addition of CNT increased the electrical conductivity. The values for electrical conductivity are given in Table 5. It was observed that 0.1% CNT added had very little effect on the electrical conductivity. Especially 0.5% and 1% CNT additions significantly increased the electrical conductivity. As can be understood from the percolation curve shown in Fig.7, more than 1% CNT addition can make the nanocomposites semiconductors. In the study by El Sayed [22], electrical measurements of PVC/PMMA/MWCNT nanocomposites reached conductivity values of 1.39 × 10-6 S/cm at 0.5% MWCNT concentration and 9.25 × 10-6 S/cm at 1% MWCNT concentration. The effect of matrix type on the electrical conductivity of MWCNT was investigated by Tohmassin et al. [13]. The researchers determined that carbonyl groups interact well with the cyclic structure of nanotubes. They reported that this interaction also positively impacted their morphology and electrical conductivity. A homogeneous distribution is essential to ensure that the MWCNT form an interconnected filler network [25]. From the conductivity curves obtained in this study, SEM images also support that CNT are sufficiently homogeneously distributed to form a conductive filler network

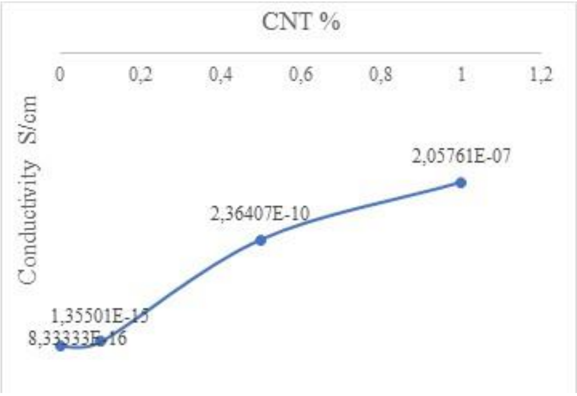

Fig. 7: Electrical conductivities of the synthesized blend and nancomposites with different ratio of CNT.

Table-5: Electrical conductivities of the synthesized blend and nancomposites with different ratio of CNT.

Table-5: Electrical conductivities of the samples.

Sample Code	Conductivity (S/cm)
PVC60- 0	8.33×10^{-16}
PVC60- 0.1	1.35×10^{-15}
PVC60- 0.5	2.36×10^{-10}
PVC60- 1	2.05×10^{-7}

Conclusion

Spectroscopic properties, thermal behaviors and electrical conductivities of nanocomposites obtained by adding MWCNT to PVC-PMMA blend were investigated. As a result of the investigations, morphological changes did not create a significant difference to spectroscopic properties and thermal behaviors. In addition to preserving the properties of nanocomposites, their electrical conductivities increased significantly and more than 1% CNT addition could convert the nanocomposites into semiconductors.

Acknowledgements

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