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Low electrical percolation threshold and PL quenching in solution-blended MWNT–MEH PPV nanocomposites

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Pristine multiwall carbon nanotubes (MWNT) (synthesised using CVD approach) and poly[2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene vinylene] (MEH PPV) based composites were prepared using a solution blending approach by employing various nanotube weight fractions. The prepared composites have been characterised using SEM, AFM, PL spectroscopy, UV-Vis studies and *I*–*V* characterisation. Increase in MWNT concentration has been found to quench the PL spectra of the composites suggesting photoinduced electron transfer from polymer to MWNT. The increase in MWNT concentration also increases the absorption of the composites. PL quenching and increase in absorption are desirable attributes for the design of photovoltaic systems. Also, the electrical conductivities of the composites can be described by the scaling law based on percolation theory and based upon the scaling law, a low electrical percolation threshold value (0.5 wt%) has been obtained for this composite system. The value of *t* (critical exponent) based on percolation theory is found to be 1.11. The low value of *t* is attributed to the aggregation and bundling of nanotubes in the prepared composites, as is evident from SEM and AFM micrographs. The turn-on voltage is also found to be reduced in the case of polymer–nanotube composite system as compared to the pristine polymer system. Also, it has been observed that at higher weight percentages, the MWNTs form an immensely dense network and act as nanometric heat sinks, thus preventing the build up of large thermal effects, caused by the increased current in the pixels at higher voltages. Analysis of these optical and electrical properties is important before utilising the composite in organic electronics applications, in order to obtain more scientifically correct and repeatable results with fabricated devices.

Keywords: multiwall carbon nanotubes; MEH PPV; nanocomposite; percolation threshold; PL quenching

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1. Introduction

Composites of conducting polymers with carbon nanotubes (CNTs) [1–3] have attracted considerable attention in the past few years because of their potential applications in polymer light emitting diodes [4,5], photovoltaic cells and photodiodes [6], field effect transistors [7], optical limiting devices [8], supercapacitors [9], sensors [10], etc. In fact, the extensive and exhaustive applications of CNTs in the field of polymer composites has perhaps exhilarated the scientific community to consider nanotubes as a part of the polymer family [11] rather than as a novel nanotechnology product. In the wide cosmos of conducting polymers, poly[2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene vinylene] (MEH PPV) occupies a unique place for itself because of its good electrical and optical properties which enable its wide application in organic light emitting diodes (OLEDs) and photovoltaic devices. MEH PPV possesses high absorbance in the visible region [12] and high hole mobility [13]. The composites of CNTs with MEH PPV have been synthesised and utilised in OLEDs [14], solar cells [15], optical limiting devices [16], etc. However, systematic studies relating morphological, optical and electrical properties of the multiwall carbon nanotube (MWNT)–MEH PPV composite with electrical percolation threshold value have been lacking in literature. Percolation threshold value of a composite system is an important parameter, because above the percolation threshold value in a composite system, a connected network is formed throughout the host matrix, resulting in a jump in the electrical conductivity. For economic reasons, the achievement of extremely low percolation threshold value is important. Also, extremely low percolation threshold value signifies use of less amount of filler material, thus maintaining the intrinsic properties of the host material in the case of a composite system. It is beneficial to design the devices by utilising filler concentration above the percolation threshold value in the composite system. We have prepared pristine MWNTs (synthesised using CVD approach) and MEH PPV based composites using a solution blending approach by employing various nanotube weight fractions. The use of MWNTs is particularly important here, considering the fact that owing to their larger diameter and more complex multilayered structure, MWNTs are essentially metallic in nature having a small but finite density of states at the Fermi level, much like graphite [17]. Hence, separation of MWNTs into semiconducting and metallic types before utilisation in an application is not required. The presence of two types of tubes in the case of single-wall CNTs affects the electronic properties of the composite, and separation of the two types of tubes in a 100% efficient manner is still a puzzle for the scientific community. The synthesised composites have been characterised using SEM, AFM, PL spectroscopy, UV-Vis studies and I – V characterisation. Increase in MWNT concentration has been found to quench the PL spectra of the composites suggesting photoinduced electron transfer from polymer to MWNT. The increase in MWNT concentration also increases the absorption of the composites. PL quenching and increase in absorption are desirable attributes for the design of photovoltaic systems. Also, the electrical conductivities of the composites can be described by the scaling law based on percolation theory and, based upon the scaling law, a low electrical percolation threshold value (0.5 wt%) has been obtained for this composite system. The value of t (critical exponent) based on the percolation theory is found to be 1.11. The low value of t is attributed to the aggregation and bundling of nanotubes in the prepared composites, as is evident from SEM and AFM micrographs. The turn-on voltage is also found to be reduced in case of polymer–nanotube composite system as compared to the pristine

polymer system. Also, it has been observed that at higher weight percentages, the MWNTs form an immensely dense network and act as nanometric heat sinks, thus preventing the build up of large thermal effects, caused by the increased current in the pixels at higher voltages. Analysis of these optical and electrical properties is important before utilising the composite in organic electronics applications, in order to obtain more scientifically correct and repeatable results with fabricated devices.

2. Experimental

The MWNTs were prepared using the CVD technique [18]. A solution of ferrocene in toluene (in an appropriate ratio) is injected into a two-stage furnace whose reaction zone is maintained at 750°C. Argon is used as the carrier gas and its flow rate is adjusted in such a manner that the maximum quantity of precursor is consumed in the reaction zone. The first stage of the furnace is maintained at around 200°C to ensure that the solution is vaporised as soon as it is injected (the vapourisation temperature of ferrocene and toluene are 175°C and 110°C, respectively). MWNTs grow predominantly in the centre of the reaction tube, which are then scraped out, after cooling the reaction tube to room temperature. The as-prepared MWNTs are mixed with MEH PPV polymer in toluene solution in various weight percentages (0, 0.5, 2.0, 5.0, 15.0 and 25.0%) and sonicated for 2 h. MEH PPV (Product No. 541443-1G) and poly(3,4-ethylenedioxythiophene):poly(styrenesulphonic) acid (PEDOT:PSS) (product no. 483095-250G) were purchased from Sigma Aldrich. PEDOT:PSS was filtered using a durapore PVDF membrane filter with a pore size of 0.45 µm. ITO-coated glass substrates (25 mm × 25 mm, $R_s = 20 \Omega/\square$) having ITO thickness of 120 nm were procured from Vin Karola, USA. These were patterned and then cleaned sequentially using a non-ionic detergent solution, distilled water, acetone, trichloroethylene and propanol, respectively. After baking in a vacuum oven for 30 min at 120°C, a PEDOT:PSS layer is applied to these substrates using a spin coating unit (SCU 2007 A, Apex Instruments Co.), at 2000 rpm for 2 min. The thickness of the PEDOT:PSS layer is 100 ± 5 nm, as measured using an ellipsometer (J. A. Woollam Co., M-2000U). The PEDOT:PSS layer has been applied as a buffer layer. It improves the quality of the ITO electrode by minimising the surface roughness of ITO and reducing the resistance between the polymer layer and the ITO. The PEDOT:PSS coated substrates are baked in vacuum oven at 120°C for 2 h. After that, the substrates are cooled down to room temperature and MEH PPV–MWNT nanocomposite solutions with varying concentrations of MWNTs (0, 0.5, 2.0, 5.0, 15.0 and 25.0%) are applied to different substrates using a spin coating unit (SCU 2007 A, Apex Instruments Co.), at 2000 rpm for 2 min. The thickness of the MEH PPV–MWNT nanocomposite layer is 200 ± 10 nm, as measured using an ellipsometer (J. A. Woollam Co. M-2000U). The nanocomposite solution coated substrates are baked in a vacuum oven at 150°C for 2 h and then allowed to cool down to room temperature. Subsequently, aluminium electrodes are deposited by vacuum evaporation technique. A schematic diagram which illustrates the structure is shown in Figure 1.

The as-synthesised MWNTs were examined using scanning electron microscope (Carl Zeiss, EVO[®] MA 10). The spin-coated MWNT–MEH PPV composite films on ITO substrates were examined using a scanning electron microscope (Carl Zeiss, EVO[®] MA 10) and an atomic force microscope (SPM Solver P47H-PRO) in semicontact mode. HRTEM studies were carried out using Tecnai G2F-30 STWIN 300kV FEG HRTEM.

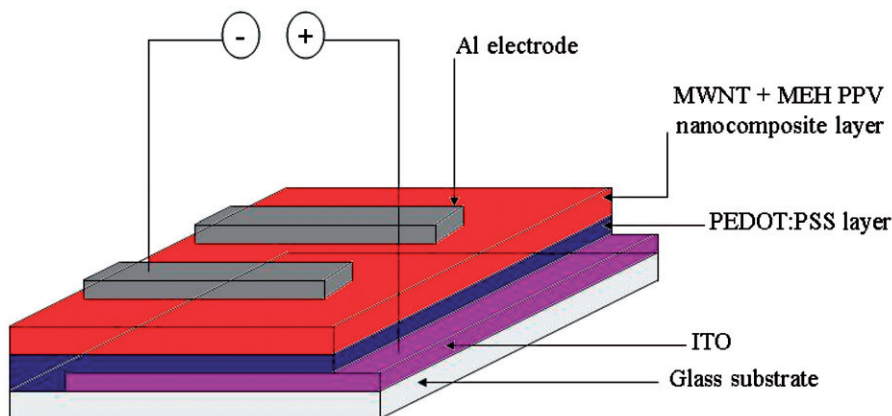


Figure 1. Schematic sandwich geometry employed.

Raman spectra were recorded using Ar ion laser with an excitation wavelength of 514.5 nm and analysed using a Renishaw Raman spectrometer (Invia Reflex) equipped with a charge-coupled device in a backscattering geometry. The optical absorption spectra of the composite films were recorded using Perkin-Elmer Lambda 25 UV-Vis spectrophotometer. PL was measured using a home-assembled system consisting of a two-stage monochromator, a photomultiplier tube (PMT) with a lock-in amplifier for PL detection and an Ar⁺ ion laser operating at 405 nm and 5 mW (corresponding to 0.125 W/cm²) for excitation in all the measurements. The thickness of the films was measured using an ellipsometer (J. A. Woollam Co., M-2000U). *I-V* measurements were carried out using a Keithley 2400 programmable voltage-current digital source meter.

3. Results and discussion

The SEM micrograph of synthesised MWNTs (Figure 2) shows the presence of MWNTs with length in the range of hundreds of nanometre and diameter in the range of tens of nanometers (30–50 nm). The HRTEM micrograph (Figure 3) confirms the multiwall structure of nanotubes and shows the inner diameter of MWNTs less than 10 nm and outer diameter in the range of tens of nanometres (30–50 nm). The Raman spectrum (Figure 4) shows the presence of a sharp peak at 1580 cm⁻¹ (G band) corresponding to the high frequency E_{2g} first-order mode and a peak at 1352 cm⁻¹ (D band) corresponding to the disorder-induced mode. Thus the Raman spectrum also confirm the formation of MWNTs. MEH PPV polymer (Figure 5) is a derivative of PPV in which two of the hydrogen atoms on each phenylene ring are replaced by O-CH₃ and O-CH₂CH(C₂H₅)C₄H₉. Figure 6 is a digital photograph of the MWNT-MEH PPV nanocomposite solutions with varying weight percentage of MWNTs (0%, 0.5%, 2.0%, 5.0%, 15.0% and 25.0%). It can be seen that the 0% solution is a clear reddish orange solution of the pristine polymer whereas as the weight percentage of MWNTs in the solution increases, the solution changes colour from reddish orange to brown to dark brown. These nanocomposite solutions were found to be stable over a period of more than four months, i.e. the MWNTs were suspended indefinitely in the solution and did not precipitate out from the solution.

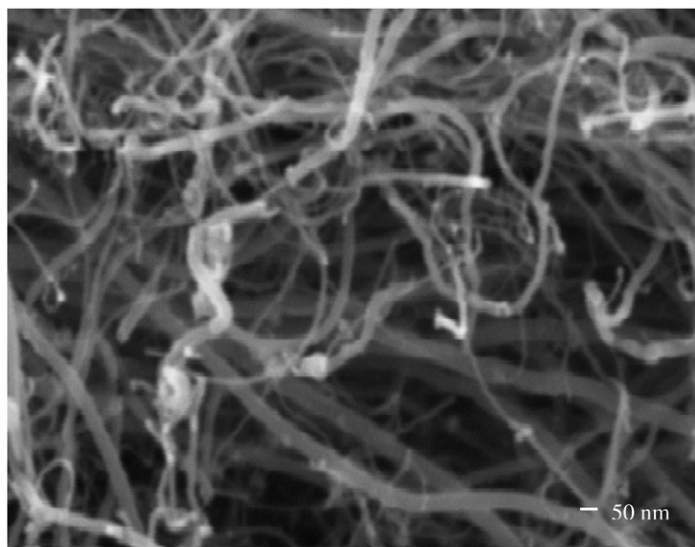


Figure 2. SEM micrograph of synthesised MWNTs.

3.1. Morphological characterisation

The SEM micrographs of the composite solutions spin coated on ITO-coated glass substrates were observed in order to know how the nanotubes are organised and dispersed in the polymer matrix, and how the increase in weight percentage of MWNTs in the nanocomposite solutions affects their distribution and alignment in films.

Considering the morphology of MWNTs in polymer solution, on ITO-coated glass substrates, to be representative of the level of dispersion of nanotubes in the polymer matrix, it can be observed from the SEM micrographs (Figure 7) that the nanotubes are dispersed relatively uniformly in the low-concentration solutions and they have a tendency to agglomerate in higher concentration solutions. It can be inferred that at higher concentrations, the attractive forces between CNTs appear in a more dominating manner and hence, agglomeration of nanotubes is observed. Also, from the SEM micrographs, it can be observed that nanotubes are quite long. This is a beneficial feature for the nanocomposites because longer nanotubes exhibit much lower percolation thresholds in a composite system as compared to shorter nanotubes [18]. Also, it can be observed from the SEM micrographs that the nanotubes are randomly distributed and not aligned in films. This is expected since we had not taken any measures to align the nanotubes in the film, although, alignment of nanotubes in films has also been found to lower the percolation threshold in composite films [19].

Figure 8 shows the AFM micrograph for MWNT–MEH PPV composite with 25 wt% MWNTs. From the micrograph, MWNT bundles can be observed (indicated by arrows) and from the scale bar showing the height along the z -axis, it can be inferred that the height of these bundles is around 145 nm, which shows that a polymer layer has wrapped around the nanotubes. However, the exact thickness of the polymer layer cannot be determined since it is difficult to determine individually, the contribution to thickness, because of bundling of nanotubes as well as coating by the polymer.

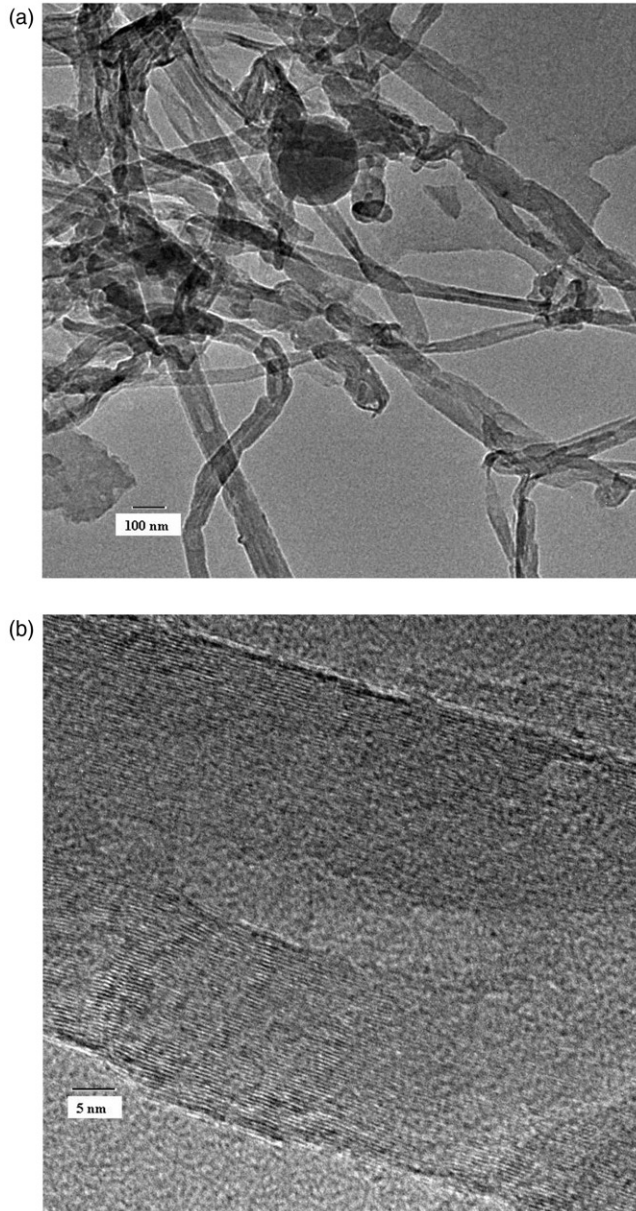


Figure 3. (a) TEM and (b) HRTEM micrograph of synthesised MWNTs.

3.2. Optical characterisation

From the optical absorption spectra for the MWNT–MEH PPV nanocomposites with varying MWNT concentration (Figure 9), it can be observed that with the increase in the MWNT concentration in the nanocomposite, the absorption increases. The absorption by MWNTs is responsible for this observed behaviour [6]. The spectra for 0%

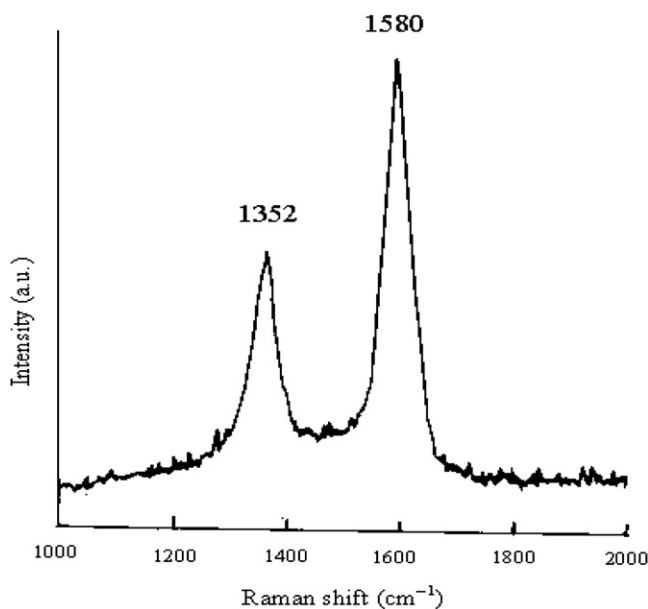


Figure 4. Raman spectrum of synthesised MWNTs.

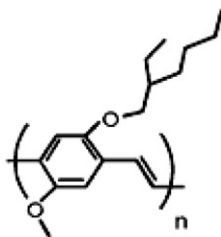


Figure 5. Structure of MEH PPV.

and 0.5% were overlapping. Similarly, the spectra for 15% and 25% were found to be overlapping. The PL spectra for the nanocomposites of MEH PPV with MWNTs (Figure 10) show that with increase in MWNT concentration, the PL spectra are quenched. The line shape of PL spectra of MWNT-MEH PPV from the composite is the same as that of pure MEH PPV directly dissolved in toluene, reflecting that the conformation of MEH PPV is not changed during the formation of nanocomposite. Therefore, the PL quenching is only related to the presence of MWNTs in MEH PPV. This PL quenching takes place by charge carrier separation, arising from polymer-to-MWNT photoinduced electron transfer leading to exciton dissociation near the MWNT-polymer junction [20]. Thus, addition of MWNTs to the MEH PPV polymer matrix, introduces internal polymer-MWNT junctions within the polymer matrix. The high electric field at these junctions splits up the excitons and the charge transfer then follows by the transport of electrons through the

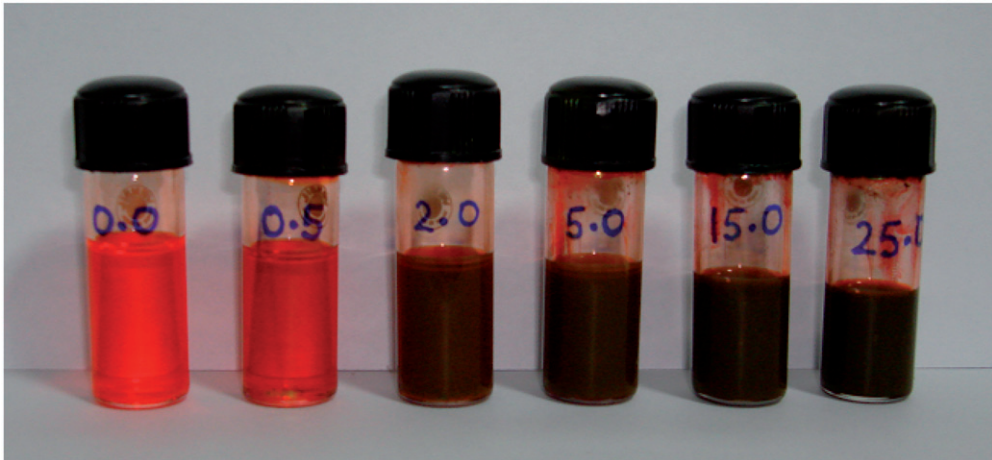


Figure 6. Digital photograph of the MWNT–MEH PPV nanocomposite solutions with varying weight percentage of MWNTs (0, 0.5, 2.0, 5.0, 15.0 and 25.0%).

nanotube length to the Al contact and holes through the polymer to the ITO contact [21]. This leads to the observation of the PL quenching phenomena in MEH PPV–MWNT nanocomposite system.

3.3. Electrical characterisation

I – V characteristics of the MWNT–MEH PPV nanocomposite systems were measured using the schematic sandwich geometry, as shown in Figure 1. From the I – V characteristics (Figure 11) for the schematic sandwich geometry structure shown in Figure 1, we can observe that as the concentration of MWNTs in the nanocomposite system increases, the threshold voltage reduces considerably (from 1.46 V for 0% MWNT case to 0.96 V for the 25% MWNT case). Also, current increases when MWNTs are incorporated in the system as compared to the pristine case. This is expected considering the high electrical conductivity of CNTs. Thus, we can infer that resistance of the composite is reduced with increase in MWNT concentration. The reduction in resistance leads to an increase in the current density of the device at the same drive voltage, which may contribute to the lower turn-on voltage of the sandwich geometry structure. A mathematical fit to the experimental data, of the form of $\sigma = C(f - f_c)^t$ (where $f > f_c$) provides the percolation threshold, $f_c = 0.5$ wt% and critical exponent, $t = 1.11$ (Figure 12). The critical exponent's value is smaller than the universal value for a three-dimensional percolating system ($t = 1.94$) [22]. As reported by Kilbride et al. [23], values of t around 1.3 have been observed in polyaniline–PMMA [24,25] or carbon black–polyethylene [26] composites. Recently, t value of 0.97 [27] has been reported for pristine MWNTs (synthesised by CVD approach) and poly(bisphenol A carbonate) (PC) composites prepared by solution blending approach. Percolation corresponds to the formation of a CNT network that allows electron transport by tunnelling or electron hopping along

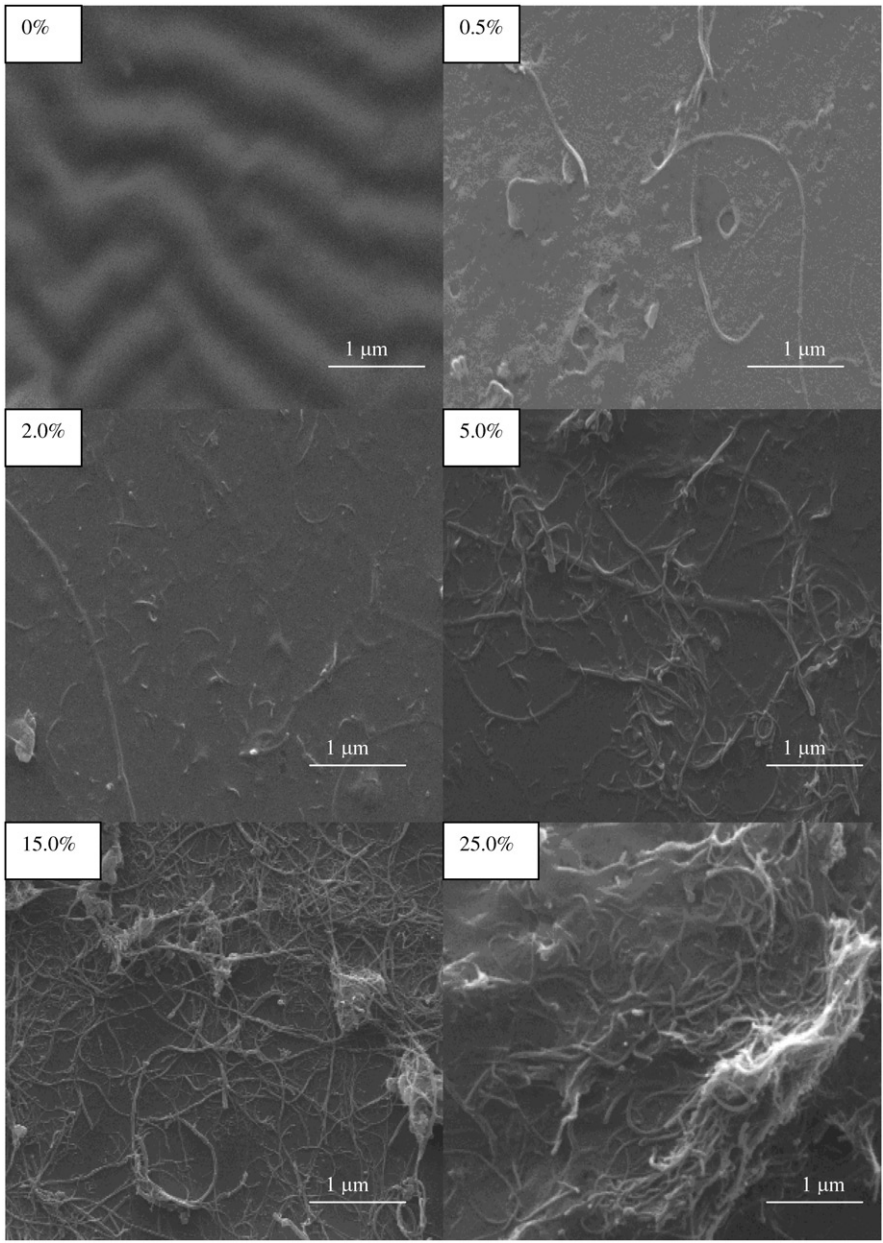


Figure 7. SEM micrographs for the nanocomposite samples spin coated on ITO-coated glass substrates with varying weight percentage of MWNTs (0, 0.5, 2.0, 5.0, 15.0 and 25.0%).

CNT interconnects [28–30]. The low value of percolation threshold (0.5 wt%) obtained is a useful characteristic with regard to the nanocomposite system. Hence, a continuous conductive network is formed at very low filler (MWNT) concentration, because of the high aspect ratio of the nanotubes used in this study. The lower value of t can be attributed

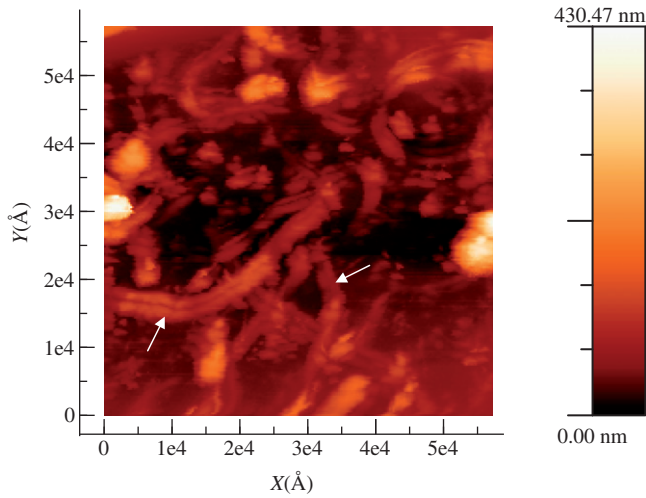


Figure 8. AFM micrograph for MWNT–MEH PPV composite with 25 wt% MWNTs.

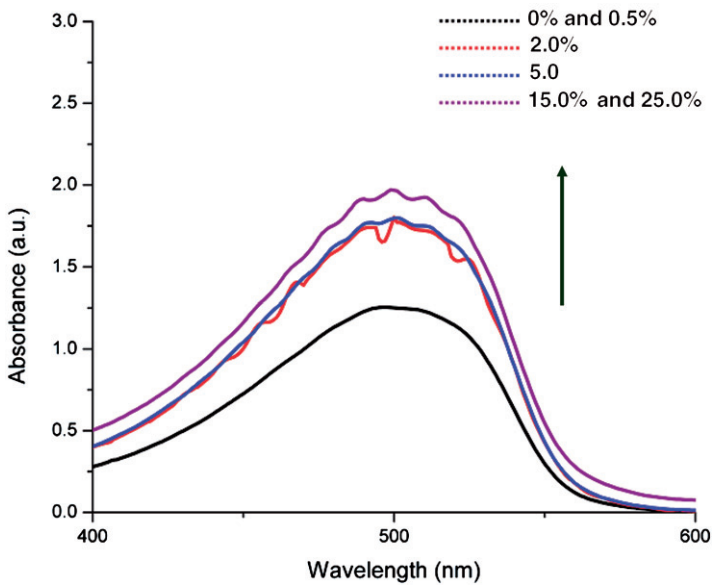


Figure 9. Optical absorption spectra of MWNT–MEH PPV nanocomposites with varying MWNT concentration.

to the aggregation and bundling of CNTs during the preparation of the samples [31], as it is evident from the SEM and AFM micrographs.

One more phenomenon which was observed was that for the 15 and 25.0 wt% MWNT-based composites, application of higher voltages (15 V and above) did not lead to shorting of the pixels in the sandwich geometry structure, whereas for the lower MWNT wt%

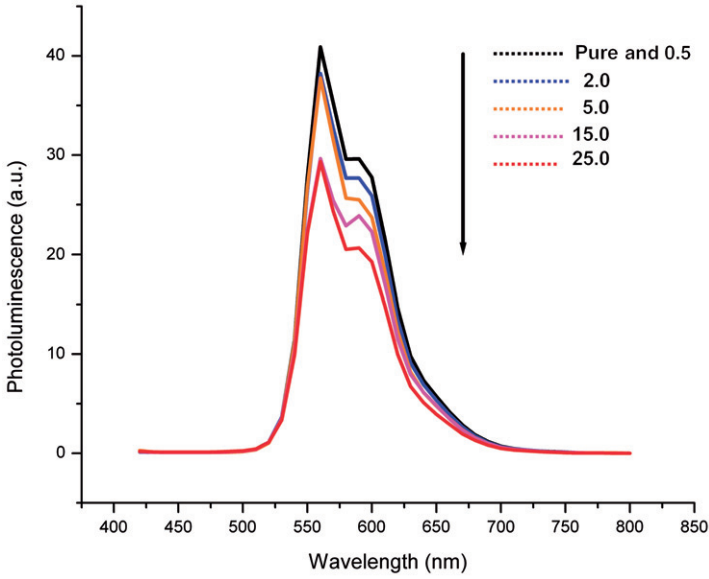


Figure 10. PL quenching of the polymer MEH PPV with increase in MWNT concentration.

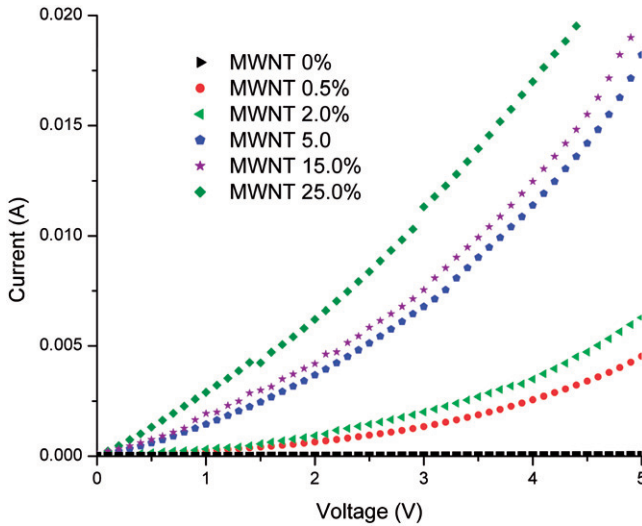


Figure 11. I - V characteristics for the schematic sandwich geometry structure shown in Figure 1.

composites as well as pristine polymer based sandwich geometry structures, application of higher voltages (15 V and above) lead to shorting of the pixels in the sandwich geometry structures, as the pixels were not able to tolerate the excessive current flow at higher voltages. This may be explained as follows. For the same amount of composite solution in all the cases, the films in case of 15.0 and 25.0 wt% MWNT composite based sandwich

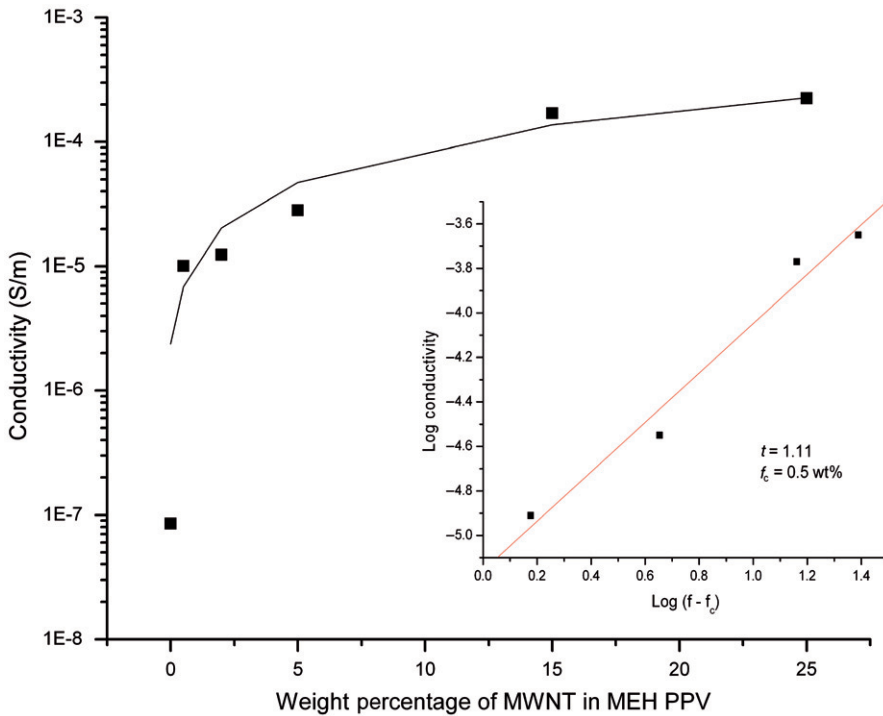


Figure 12. Variation of electrical conductivity as a function of MWNT weight percentage in MWNT–MEH PPV composites.

geometry structures were comparatively thicker and more rough as compared to the films for pristine polymer based structure as well as films for lower wt% of MWNTs. At higher weight percentages, the MWNTs form an immensely dense network and in fact act as nanometric heat sinks, as explained by Curran et al. [3] and prevent the build up of large thermal effects, caused by the increased current in the pixels at higher voltages. Hence, the pixel is able to tolerate higher current without getting destroyed. This use of nanotubes as nanometric heat sinks is very important in certain device applications.

4. Conclusion

Composites based on pristine MWNTs (synthesised using CVD approach) and the polymer MEH PPV have been prepared using a solution blending approach by employing various nanotube weight fractions. The composites have been found to exhibit PL quenching and increase in absorption with increase in concentration of MWNTs. Both these attributes are desirable with respect to the design of a photovoltaic system based on this composite. Moreover, the ease of preparation (solution blending approach) makes this composite system attractive for fabrication of flexible devices using the roll-to-roll approach or the inkjet printing technology. Although there have been attempts at designing photovoltaic devices using CNT–MEH PPV composites, studies related to

determination of electrical percolation threshold for this system have been lacking. Before utilising the composite for designing a photovoltaic device or any other electronic application, it is important to know the electrical percolation threshold for the system, so that the optimum amount of filler material can be utilised, without compromising the intrinsic properties of the host material and also for economic reasons. We have determined the electrical percolation threshold for this composite system to be 0.5 wt%. The value of t (critical exponent) for this composite system based on percolation theory has been found to be 1.11. This low value of t is attributed to the aggregation and bundling of nanotubes during composite preparation, as it is evident from SEM and AFM micrographs. Measures to decrease the aggregation and bundling of nanotubes in the composite system as well as to align the nanotubes can be expected to further lower the electrical percolation threshold value for this composite system, making it even more attractive for photovoltaic device applications. Recently, it has been reported that a longer mixing process [27] can significantly increase the electrical conductivity of a CNT–polymer composite prepared using a solution blending approach and managing the dimensionality is the trade-off between high conductivities and well-dispersed nanotubes. Perhaps, a similar approach in case of our nanocomposite system can be expected to yield a higher t value and show more increase in electrical conductivity of the composite system. PL quenching, increase in absorption, low electrical percolation threshold and exhibition of nanometric heat sink property are some of the desirable characteristics of the designed nanocomposite system, which we have been able to obtain using a solution blending approach.

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