Effect of multiwalled carbon nanotubes on electrical conductivity and magnetoconductivity of polyaniline

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Abstract
An in situ chemical polymerization method was applied in order to prepare polyaniline-multiwalled carbon nanotube (PANI-MWCNT) composites with different concentrations of MWCNT. X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, optical absorption and photoluminescence analyses of the composites were performed to investigate the structural, morphological and optical properties of the composites. Electrical transport properties of different PANI-MWCNT composites were investigated in the temperature range 77 K ≤ T ≤ 300 K in the presence and also in the absence of a magnetic field up to 1 T. The dc resistivity of the composites follows Mott’s variable range hopping theory. Two different slopes have been observed in temperature variation of resistivity, which occurs due to the presence of MWCNT in the polymer matrix. The magnetoconductivity of the samples at different temperatures is negative, which can be explained by the wavefunction shrinkage effect.

Keywords: polyaniline, multiwalled carbon nanotubes, chemical synthesis, VRH theory, magnetoconductivity

Classification numbers: 2.01, 5.10, 5.14

1. Introduction

The study of conducting polymer nanostructures such as nanorods, nanotubes, nanowires and nanofibers has become a field of popular and extensive research work due to the advantages of combining organic conductors with nanodimensional systems with interesting physicochemical properties and potentially useful applications [1–4]. Some of these properties, like mechanical, electrical and thermal properties, have been investigated by incorporating the nanomaterials into some form of matrix such as conducting polymers. Carbon nanotubes (CNTs)/conducting polymer composites have been considered as a promising advanced material for many applications such as electromagnetic shielding, organic light-emitting diodes, photovoltaic cells, energy storage devices, sensors, electrostatic dissipation and antennas [5–14]. Polyaniline (PANI) is one of the best materials among this class of polymers due to its relatively high conductivity, better stability, low cost synthesis and easier fabrication procedure. The presence of extended π-conjugation along the polymeric backbone is responsible for higher conductivity of PANI. They are attractive building blocks for the development of novel polymer-nanocomposite materials with enhanced functionality, especially if it comes to enhanced conductivity, thermal stability and reinforcement properties [15–17]. The preparation of carbon nanotubes and polymer composites was first reported by Ajayan et al [18]. Since then, there have been extensive efforts to combine carbon nanotubes and polymers to produce functional composite materials with desirable electrical and mechanical properties.
properties [19–39]. Wei et al. [27] reported the morphology and one-dimensional variable range hopping transport of PANI-multiwalled carbon nanotube (PANI-MWCNT) composites. Zengin et al. [17] observed the enhancement of room temperature conductivity of PANI-MWCNT composite films. The change in conductivity with MWCNTs content was studied by Wu et al. [28], Yan et al. [29] and Konyushenko et al. [30]. The overall temperature dependence and negative magnetoresistance of PANI-MWCNT composites was reported by Zhang and co-workers [25–31] and Long et al. [32]. Xu et al. [33] showed that owing to the incorporation of para-phenylenediamine-functionalized MWCNTs, conductivity of the nanocomposite at room temperature is increased by about two orders of magnitude over neat sulfonated PANI. According to Reddy et al. [34], the room temperature electrical conductivity of the PANI-MWCNT/Au or Ag composites was 4.8–5.0 S cm⁻¹, respectively, which was much higher than that of PANI-CNTs (0.18 S cm⁻¹) or pure PANI (2.5 × 10⁻³ S cm⁻¹). Although a unique compatibility between PANI and CNTs is shown by the recent studies, the exact nature of physicochemical properties of PANI-CNTs is still unclear. Any definite conclusion cannot be taken about their temperature and magnetic field dependent electrical conductivity. The day-by-day increasing trend in nanoelectronics and CNT-assisted polymeric devices demands a clear idea about the electrical behavior of the PANI-CNT composites. Very few of the previous reports explain the exact nature of temperature dependent electrical transport properties and magnetococonductivity of PANI-MWCNT composites, especially in the below room temperature region. In this work, in situ fabrication and characterization of HCl-doped PANI with MWCNTs has been done along with an extensive investigation of low-temperature (77 K ≤ T ≤ 300 K) conductivity and magnetococonductivity (up to 1 T) of the PANI-MWCNT composites.

2. Sample preparation and experimental techniques

PANI, cetyl trimethylammonium bromide (CTAB), MWCNTs (Nanocyl 7001), ammonium peroxysulphate (APS), ethanol and acetone were used as received from the market and purified. Double distilled water and aniline were used in this investigation. Composites of polyaniline with MWCNTs were synthesized by in situ chemical oxidative polymerization using CTAB as a surfactant. Then 1.136 g CTAB and 60 mg MWCNTs were added in 300 ml 1 M HCl and the mixture was sonicated for 4 h to obtain a well-dispersed suspension which was then kept at 0–5 °C in the refrigerator. A precooled 1.2 ml aniline and 125 ml 1 M HCl containing 2.7 g APS were added sequentially to the MWCNT-CTAB suspension taken in an ice chamber with continuous magnetic stirring for 1 h and then left standing in the refrigerator at 0–5 °C for 24 h. A black precipitate was obtained on filtration. The solid mass was washed with ethanol and acetone repeatedly to remove oligomers and unreacted monomer then washed with double distilled water several times and dried at room temperature in a dynamic vacuum for 24 h. For comparison purposes we synthesized a total of five samples with 0, 1, 2, 3 and 4% MWCNTs. Samples were numbered as PC0, PC1, PC2, PC3 and PC4 for 0, 1, 2, 3 and 4% MWCNTs, respectively.

The morphology of the samples was obtained by using a scanning electron microscope (SEM, Hitachi S-3000N). The phase identification was performed using x-ray diffractometer (XRD) with nickel filter Cu-Kα radiation (λ = 1.5414 Å) in 20 range from 10° to 80°. Fourier transform infrared (FTIR) spectra were recorded on a NEXUS, Nicolet FTIR spectrometer in the range 400–2000 cm⁻¹ with KBr pellets. The optical absorption of the samples was taken by a double beam spectrophotometer (UV-3010) using dimethylsulphoxide (DMSO) as a solvent. Photoluminescence (PL) spectrum of the samples was obtained using F-2500 FL spectrophotometer with DMSO as a solvent. The excitation wavelength for the photoluminescence study of the sample was 270 nm. The electrical conductivity of the samples was measured by a standard four probe method after good contact was ensured with highly conductive graphite adhesive (Electrodag 5513, Acheson, Williston, VT) and fine copper wires as the connecting wires. The dc conductivity was measured with an 8 1/2-digit Agilent 3458A multimeter. The temperature dependence of the conductivity was studied with a liquid nitrogen cryostat. For the control and measurement of the temperature, an ITC 5025 Oxford temperature controller was used. To measure the dc response, pellets of 1 cm in diameter of the samples were made by pressing the powder under a hydraulic pressure of 500 MPa. The magnetococonductivity was measured in the same manner by the variation of the transverse magnetic field (B < 1 T) with an electromagnet.

3. Results and discussion

Figures 1(a)–(d) represent the scanning electron microscopy (SEM) images of PC1, MWCNTs, PC4 and PC0, respectively. Rod-like morphology of PANI-MWCNTs is seen from the SEM images. Columnar and pellet-type growth of the rod are observed. It is also observed that the diameter of the PANI-MWCNT composite has been increased in comparison to MWCNTs after polymerization and this suggests that the aniline monomer is uniformly polymerized on the surface of the MWCNTs and forms columnar and pellet-type grows. The diameter of the rod is 80–90 nm and length is in the range 2–3 μm. So it can be suggested that polyaniline is coated over the MWCNTs.

Figure 2 shows XRD patterns of polyaniline (PC0) and its composites with MWCNTs. Polyaniline exhibits three broad peaks at 2θ angles 15°, 20° and 25°, which are similar to those of the polyaniline reported by other groups [28–35]. These peaks may arise due to regular repetition of aniline. Composites of polyaniline with MWCNTs show similar crystalline behavior of polyaniline. Peaks in the composite are sharper and peak intensity is also increased in the composites in comparison with those of polyaniline. This may be due to the incorporation of MWCNTs and ordering of polyaniline along the MWCNT axis [24]. In our previous work [36], we have reported that MWCNTs have some peaks at 25°, 44° and 51°. The peak at 25° is the merged peak of polyaniline and MWCNTs. However, the peaks at 44° and 51° are absent in the diffractogram of PANI-MWCNT composites. This
Figure 1. SEM images of (a) PC1, (b) MWCNTs, (c) PC4 and (d) PC0.

Figure 2. X-ray diffraction patterns of PANI and different PANI-MWCNT composites.

Figure 3. FTIR spectra of PANI and different PANI-MWCNT composites.

suggests that the polymers are coated over the MWCNTs, due to which these peak intensities are very weak compared to the PANI peak.

Figure 3 represents FTIR spectra of polyaniline (PC0) and its composites (PC2 and PC4) with MWCNTs. Bands at 1690, 1620, 1450, 1376, 1240, 926, 820 and 685 cm\(^{-1}\) are characteristic of polyaniline [37-41]. The bands at 1690, 1610 and 1450 cm\(^{-1}\) are assigned to C=C and C=N stretching modes of vibration of quinoid (\(-\text{N}=\text{Q}=\text{N}-\) where Q = Quinoid ring) and benzenoid units of polyaniline. The band at 1376 cm\(^{-1}\) is for the C–N stretching of mode of benzenoid unit. Bands at 1240 and 820 cm\(^{-1}\) are assigned for C–C and C–H stretching for benzenoid unit of polyaniline and the band at 685 cm\(^{-1}\) is assigned to out of plane C–H vibration. Therefore, peak assignment indicates the produced product is polyaniline [37-41]. From the FTIR spectra of PC2 and PC4 it is observed that MWCNT interacts with different reaction sites of polyaniline causing both red and blue shift of the band from 1376, 1240 to 1370, 1222 cm\(^{-1}\), respectively, and from 1690 to 1730 cm\(^{-1}\). The reason behind the irregular blue or red shift may be due to the change in bond strength of different bonds of polyaniline in the presence of MWCNTs. This FTIR result suggests that N–H, C–H, C–C bond becomes weaker and C=\(=\)C, C–N bond becomes stronger in the composite [42].
Figure 4. TGA curves of two representative samples PC0 and PC4.

Figure 5. UV-Vis absorption spectra of PANI (right inset) and different PANI-MWCNT composites. Left inset shows the band gap of PANI and different PANI-MWCNT composites.

for the formation of a bond with MWCNTs. All other composites show a similar result.

Thermograms of polyaniline (PC0) and its composite PC4 with MWCNTs are presented in figure 4. In PC0 weight loss starts for the loss of water molecules from polyaniline at around 25°C and ends at around 100°C. About 15–17% of weight loss occurs due to the removal of water molecules. A very small weight loss is obtained up to 400°C and thereafter a rapid weight loss is obtained for the degradation of the polyaniline chain. A similar type of result is obtained for polyaniline elsewhere [43]. In the case of PC4, a steady but slow decrease in weight is observed up to 650°C and thereafter a rapid weight loss is obtained. This indicates better thermal stability of the composite than of polyaniline. This increase in thermal stability may be due to the dominance of benzenoid structure over its quinoid structure of polyaniline in the composite.

Optical absorption spectra of the samples are given in figure 5. It is found that there are two main absorption bands at 330–335 nm and 600–625 nm in polyaniline (in the inset of figure 5). These bands are obtained due to π→π* transition in benzenoid rings and exciton absorption of quinoid rings (n→π*), respectively [44]. In nanocomposites, there occurs a blue shift of the absorption bands. The first band shifts to 310–320 nm and the second band shifts to 564 nm. It is observed that the MWCNTs we have used for our investigation have no such optical absorption in this wave length region. It is also reported that the UV-Vis peak has been observed for pristine CNTs at 260 nm [45], so the observed bands for our composites may be due to polyaniline only. This blue shift may be due to the increase in interaction energy for interaction of polyaniline with MWCNTs (as stated in the FTIR spectrum) or the decrease in wavelength. Optical absorption can be determined using equation $\alpha \nu = A(\nu - E_g)^n$, where $E_g$, $\alpha$, $\nu$ and $A$ are the band gap, absorption coefficient, frequency and constant, respectively, and $n$ can have values of 0.5, 1.5, 2 and 3 depending on the mode of transition [46]. In our experiment $n = 0.5$ offers the best fit for optical absorption data of the sample. The value of $n$ supports the allowed direct band transition of the nanocomposites.

Figure 6. Photoluminescence spectra of PANI and different PANI-MWCNT composites.

PL spectra of polyaniline (PC0) and its composites (PC1, PC2, PC3 and PC4) are given in figure 6. It is found that the intensity of PL increases with an increase in MWCNTs content. Exciton wavelength is taken as 270 nm for our investigation. The emission peak is centered at 345 nm which is obtained due to the π→π* transition of the benzenoid unit of polyaniline [47]. In contrast, quinonoid unit of polyaniline quenches the emission because of intrachain energy dissipation [47]. As nanocomposite is more crystalline, which is evident from its XRD pattern, so the benzenoid and quinonoid units have a more orderly arrangement without
clustering of quinonoid units. Singlet exciton forms due to the higher extent of π-conjugation with a more orderly arrangement of benzenoid and quinonoid units in nanocomposite. It decays to the ground state with the emission of light. Formation of singlet exciton increases with an increase in the conjugation length of the polymer chain because the delocalization length of the singlet exciton in conjugated polymers is comparable to its conjugation length. The triplet exciton of the conjugated polymer cannot produce PL emission, but singlet excitons are mostly responsible for PL emission because conjugated polymers cannot produce the spin flip which is necessary for an optical transition. Hence, one should expect higher PL emission from the nanocomposite which has a higher extent of π-conjugation.

To investigate the dc transport mechanism of the PANI-MWCNT composites, the dc resistivity of the samples was measured as a function of temperature within a range 77 K ≤ T ≤ 300 K. The room temperature conductivity (σ300K) of the different samples is shown in figure 7 which shows an enhancement of conductivity with an increase in the concentration of MWCNTs in the PANI matrix. Again, the resistivity ratio (ρr) of the pure PANI is 1.89 and MWCNTs is 1.30, whereas for the composite samples, it varies between 2.19 and 15.26. Thus, the resistivity ratio also increases with the introduction of MWCNTs in PANI. Hence, it may be concluded that the incorporation of highly conducting MWCNTs in the insulating PANI matrix increases the conductivity of the samples to a significant degree. PANI is considered as a good electron donor whereas MWCNTs are relatively good electron acceptors. So there is some interaction between the quinoid rings of PANI and MWCNTs resulting in a charge transfer process between the two components. For the presence of a large π-conjugated structure, the localization length in MWCNTs is around 10 nm and hence has a high conductivity. In the case of crystalline and amorphous PANI, the localization length is only 2 nm resulting in poor conductivity. Thus, there is a strong coupling between the MWCNTs and the polymer chains that increases the average localization length and hence an increase in conductivity for the composite samples compared to pure PANI.

The variation of resistivity as a function of temperature of PANI, PANI-MWCNT composites and MWCNTs is shown in figures 8(a) and (b), respectively. The decrease in resistivity with increasing temperature (semiconducting nature) has been observed for all the investigated samples. Generally, the temperature dependence of resistivity of a disordered semiconducting system can be explained in terms of Mott’s variable range hopping (VRH) model, according to which, the resistivity can be expressed as

\[
\rho(T) = \rho_0 \exp\left(\frac{T_{\text{Mott}}}{T}\right)^\gamma,
\]

where the VRH exponent \( \gamma \) determines the dimensionality of the conducting medium by the relation \( \gamma = 1/(1 + d) \). For three, two and one-dimensional systems, the possible values of \( \gamma \) are 1/4, 1/3 and 1/2, respectively; \( \rho_0 \) is the resistivity at infinite temperature; \( T_{\text{Mott}} \) is the Mott characteristic temperature depending on the electronic structure, and the energy distribution of the localized states and can be written as

\[
T_{\text{Mott}} = \frac{16}{\hbar k_B N(E_F) L_{\text{loc}}},
\]

where \( k_B \) is the Boltzmann constant, \( N(E_F) \) is the density of states at the Fermi level and \( L_{\text{loc}} \) is the localization length. To have an idea of the proper charge transfer mechanism,
the temperature dependence of dc resistivity of all the investigated samples has been analyzed by equation (1). A graph of ln[ρ(T)] with $T^{-1/2}$ (not shown in the text) gives a nonlinear variation indicating that one-dimensional (1D) transport is not the dominating charge transport mechanism for the investigated samples. Then, a graph has been plotted between ln[ρ(T)] with $T^{-1/4}$ for all the samples which shows a linear behavior for PC0 and MWCNTs throughout the entire range of temperatures ($77 \text{ K} \leq T \leq 300 \text{ K}$). For the composite samples, there is a deviation from the linear dependence at lower temperature ($T < 105 \text{ K}$) but at higher temperature, linear temperature dependence is observed. Thus, three-dimensional (3D) charge transport mechanism is suitable for explaining the temperature dependence of dc resistivity of the investigated samples. $T_{\text{Mot}}$ has been calculated from the slopes of these variations in figure 8. The value of $T_{\text{Mot}}$ is strongly influenced by the disorder present in the sample, which in turn, is expressed in terms of the resistivity ratio. The value of disorder increases with an increase in the resistivity ratio. The higher value of $\rho_1$ (= 15.26) for PC1 indicates the presence of more disorder in that sample. So, a higher value of $T_{\text{Mot}}$ is obtained in the case of PC1. Similar observations were reported in many previous studies [12, 53]. According to Long et al [32] and Wang et al [54], PANI and PANI-CNT composites follow quasi-1D transport mechanism. The results obtained in the present investigation contradict the previous results, as 3D transport is the dominating charge transport mechanism in the present case. For an isolated conducting polymer chain, 1D transport mechanism can be observed due to strong interchain coupling in polymer composites. When the conducting polymer chains are non-isolated, the electronic wavefunctions extend in three dimensions due to the presence of conducting islands between the insulating polymer matrix. SEM images of the composite samples reveal the existence of non-isolated polymer chains and the PANI matrix encapsulates MWCNTs. Therefore, the charge transport mechanism of the present samples can be explained by a 3D VRH model that is supported by the experimental data. A deviation from linearity in the ln[ρ(T)] with $T^{-1/4}$ curve in the lower temperature range has been observed in PANI-MWCNT composites (figure 8), which suggests the presence of more than one conduction mechanism. In such a system, the nanotubes are separated by a thin layer of PANI, which acts as a potential barrier to internanotube hopping. At lower temperature, interaction between PANI and MWCNTs may not be prominent, due to which barrier height is high and internanotube conduction is reduced. However, at higher temperature, the barrier height between the nanotubes may decrease and conduction among the nanotubes may occur. As a result, a deviation from the linear behavior of ln[ρ_0(T)] versus $1/T^{-1/4}$ plot has been observed at low temperature.

To explore the magnetotransport property of the PANI-MWCNT composites, the magnetic field dependent conductivity of the different samples has been measured within a temperature range 77–300 K and under a transverse magnetic field up to 1 T. The variation of magnetoconductivity with magnetic field strength at $T = 300 \text{ K}$ for different samples is shown in figure 9. All the samples show negative magnetoconductivity i.e. their conductivity decreases with increasing magnetic field strength. The maximum percentage changes of conductivity $\frac{\sigma(B,T) - \sigma(0,T)}{\sigma(0,T)} \times 100\%$ under a magnetic field of 0.8 T at 300 K are $-0.3\%$ for PC0, $-0.31\%$ for PC1, $-0.38\%$ for PC2, $-0.53\%$ for PC3 and $-0.61\%$ for PC4, respectively. The percentage change of magnetoconductivity increases with an increase in MWCNT content in the PANI matrix. In general, the dc magnetoconductivity can be analyzed in terms of a simple phenomenological model that consists of two simultaneously acting hopping processes—the wavefunction shrinkage model [55, 56] and the forward interference model (quantum interference effect) [57–59]. In the wavefunction shrinkage model, the average hopping length reduces due to the contraction of wavefunction of electrons under the influence of a magnetic field. As a result, the conductivity decreases with an increasing magnetic field. Under a small magnetic field, the magnetoconductivity ratio can be expressed as [55]

$$\ln \left[ \frac{\sigma(B,T)}{\sigma(0,T)} \right] = -t_1 \frac{e^2 L_{\text{loc}}^4}{h^2} \left( \frac{T_{\text{Mot}}}{T} \right)^{\frac{3}{4}} B^2,$$

where $t_1 = 5/2016$. It was observed that due to the small localization length of PANI, the average hopping length $R_{\text{Hopp}} = (3/8) (T/T_{\text{Mot}})^{1/4} L_{\text{loc}}$ is small and wavefunction shrinkage effect is observed in PANI [41]. However, a large positive magnetoconductivity for CNT films or pellets at weak magnetic field due to their large $L_{\text{loc}}$ and $R_{\text{Hopp}}$ is found in the literature [60, 61]. Therefore, the sign and magnitude of magnetoconductivity of the PANI-MWCNT composites are changed due to the competition of these two (wavefunction shrinkage and quantum interference) effects. The observed decrement of magnetoconductivity of the investigated samples indicates the predominance of the wavefunction shrinkage effect over the quantum interference effect, so the measured data has been analyzed according
to the wavefunction shrinkage model. Figure 9 shows the linear variation of $\ln[\sigma (B, T)/\sigma (0, T)]$ with $B^2$ for different samples at room temperature. The points in the graph represent the experimental data and the curves represent the theoretical best fits according to the wavefunction shrinkage model. It is evident from figure 9 that the experimental data are fitted reasonably well with the presumed theoretical model. Localization length has been calculated from the slopes of the graphs of figure 9 using equation (3) and its variation with resistivity ratio is shown in the inset of figure 9. There is a decrease in localization length with increasing $\rho$. In general, the disorder present in the samples is expressed in terms of resistivity ratio. The higher the resistivity ratio, the more disorder is present. Again, for higher disorder, the electronic wavefunctions are localized in a smaller region resulting in a decrease in localization length and hence, localization length decreases with increasing $\rho$. Thus, the disorder present in the sample has a strong influence on the localization length. The variation of magnetoconductivity of PC4 with magnetic field is shown in figure 10 at different but constant temperatures. Negative magnetoconductivity is observed at different temperatures. In figure 10 the points are the experimental data and the curve is the theoretical best fit with wavefunction shrinkage model. The data are reasonably well fitted with the theoretical curves showing that the magnetotransport at different temperatures is in accordance with the wavefunction shrinkage effect. The average hopping length at different temperatures has been calculated from the known values of $L_{100}$ (obtained from the slopes of the graphs in figure 10) and $T_{Met}$. The variation of average hopping length with temperature is shown in the inset of figure 10 which shows a rise in $R_{Hopp}$ with increasing temperature.

4. Conclusions

The overall conductivity of the PANI-MWCNT composites enhances the pure PANI and also increases with an increase of MWCNTs content in PANI. The 3D VRH model is followed by all of the investigated samples. A plot of $\ln[\rho (T)]$ versus $T^{-1/4}$ shows linear temperature dependence throughout the entire temperature range for PANI but a deviation from linearity has been observed for PANI-MWCNT samples at lower temperatures. The room temperature magnetoconductivity of the samples are negative due to small average hopping length, which can be interpreted by wavefunction shrinkage effect. The average hopping length increases from 100 to 300 K although negative magnetoconductivity is observed at different temperatures.

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Figure 10. Variation of the dc magnetoconductivity with perpendicular magnetic field of the PC4 sample at different temperatures. The solid lines are fitted to equation (3). Inset shows the variation of average hopping length ($R_{Hopp}$) with temperature.
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