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Electrical conductivity and shielding effectiveness of poly(trimethylene terephthalate)/multiwalled carbon nanotube composites

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Abstract Poly(trimethylene terephthalate) (PTT)/multiwalled carbon nanotube (MWCNT) composites have been fabricated to evaluate the potential of PTT composites as electromagnetic interference (EMI) shielding material. The room temperature electrical conductivity, complex permittivity, and shielding effectiveness (SE) of PTT/MWCNT composites were studied in the frequency range of 8.2–12.4 GHz (X-band). The dc conductivity (σ) of composites increased with increasing MWCNT loading and a typical percolation behavior was observed at 0.48 vol% MWCNT loading. The highest EMI SE of PTT/MWCNT composites was $\sim$23 decibel (dB) at 4.76 vol% MWCNT loading which suggest that these composites can be used as light weight EMI shielding materials. The correlation among the SE, complex permittivity, and electrical conductivity was also studied. The EMI shielding mechanism of PTT/MWCNT composites was studied by resolving the total EMI SE into absorption and reflection loss.

Introduction

With the rapid development of electrical and telecommunication industries, electromagnetic interference (EMI) has become a serious problem in modern society. EMI not only causes operational malfunction of electronic instruments, but is also hazardous to human health. Thus, EMI shielding materials are needed to protect the workspace and environment from radiation coming from electronic instruments as well as for protection of sensitive circuits. Since, many commercial and military systems like doppler, weather radar, TV picture transmission, and telephone microwave relay systems, works in the frequency range of 8.2–12.4 GHz (X-band), EMI shielding material for X band is very important for commercial and military applications [1, 2].

For an EMI shielding material it is necessary to have conductivity in the range of 10^{-4}–10^{1} S/m. Traditionally, metals in the form of thin sheets were used as EMI shielding materials. However, metals were expensive, heavy, prone to corrosion, and difficult to process. Therefore, metal-coated and metal-plated polymers were developed for shielding applications as it has the sufficient electrical conductivity. Conventional polymer composites made of stainless steel fibers, carbon fibers, metal-coated carbon fibers, and graphite were gradually developed to be used as shielding materials [3–10]. These materials have significantly high value of EMI shielding effectiveness (SE) and electrical conductivity (σ). Shui et al. [7] reported that SE value of more than 87 dB and electrical conductivity of $2 \times 10^{6}\ \Omega\ \mathrm{cm}^{-1}$ can be obtained by using 7 vol% of nickel filament in poly(ether sulphone) matrix at 1–2 GHz. Similarly Wen et al. [8] obtained 70 dB of SE and $6.2 \times 10^{2}\ \Omega\ \mathrm{cm}^{-1}$ of electrical conductivity on addition of 0.72 vol% of stainless steel fibers at 1.5 GHz in cement. Chung et al. [9, 10] reported that carbon–matrix composite with continuous carbon-fibers and flexible graphite showed exceptionally high SE of 124 and 130 dB, respectively, in the measured frequency range. The electrical conductivity of the carbon-fiber and flexible graphite composites was 2.4×10^{1} and $2.7 \times 10^{1}\ \Omega\ \mathrm{cm}^{-1}$, respectively. Inspite of having such high value of SE and conductivity, these materials are in limited use, since high concentration of filler is required to achieve adequate shielding which increases the cost and degrades polymer properties.

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In the last two decades, carbon nanofibers (CNFs) and carbon nanotubes (CNTs) have emerged as highly effective one dimensional conductive nanofiller. Several studies have been carried out to utilize the unique properties of CNT and CNF in fabricating low cost, light weight, and highly effective polymer composites for EMI shielding applications [11–16]. As CNTs possess much better electrical and mechanical properties as well as high aspect ratio compared to CNFs, CNTs became the most preferred filler to fabricate high-performance composites [17–20]. Yang et al. [11] studied the shielding behavior of CNT/CNF/polystyrene (PS) composites and found EMI SE of ~13 dB at 10 wt% CNF loading which increases to 20 dB on further addition of only 1 wt% CNT. Sundararaj et al. [19] reported EMI SE of 35 dB for 1 mm thick plate made of multiwalled carbon nanotube (MWCNT) (7.5 vol%)/polypropylene (PP) composite. Yang et al. [20] studied the EMI shielding behavior of MWCNT/PS composites and achieved SE of ~20 dB at MWCNT loading of 7 wt%. However, several studies showed that higher CNT loading may not always lead to higher SE value e.g., Liu et al. [15] achieved only ~17 dB at 20 wt% MWCNT loading in polyurethane (PU) and Kim et al. [16] reported ~27 dB SE at 40 wt% CNT loading for MWCNT/poly(methyl methacrylate) (PMMA) composite films. These observations suggest that SE of CNT filled polymer composites depends on many factors including fabrication techniques, purity of CNT, dispersion of CNTs and its content etc. Thus, it is important to further investigate the role of CNT in fabricating lightweight, high strength, and economical EMI shielding material. To design a technically and economically feasible polymer composite, understanding the EMI shielding mechanism of polymer composite is very important. Three types of EMI shielding mechanism have been proposed, namely: reflection, absorption, and multiple reflections [3, 17, 19, 21]. Reflection is the primary shielding mechanism and it depends on the number of mobile charge carriers (electrons or holes). Absorption depends on the presence of electrical or magnetic dipoles which interacts with electromagnetic waves and the multiple reflections are the total internal reflection in the material. It is observed that, in polymer composites, shielding mechanisms are more complicated than those for homogeneous conductive barriers due to the availability of large surface area for reflection and multiple reflections. In this work, we studied the shielding mechanism of PTT/MWCNT composites by determining the contribution of reflection and absorption loss in the total SE. The correlation among electrical conductivity, tan δ, and SE was also studied.

Among the various polymer matrices used for fabricating polymer composites for EMI shielding application, polyester polymers are one of the most versatile materials because of its high strength, light weight, and ability to form fiber, film, and bulk material. Metal or intrinsically conducting polymer coated polyester poly(ethylene terepthalate) (PET) and poly(butylene terephthalate) (PBT) has been used as EMI shielding material as flexible cable jacket, laminates, fabrics, and films [22–24]. Recently CNT-reinforced PET/PBT composites have also been studied for fabricating EMI shielding material and it showed promising properties. Like PET and PBT, poly(trimethylene terephthalate) (PTT) is a newly commercialized aromatic polyester having properties in between those of PET and PBT. It has the outstanding physical properties of PET and processing characteristics of PBT, which makes PTT highly suitable material for engineering applications. Recent studies show that mechanical, thermal, and electrical properties of PTT matrix can be improved by incorporation of CNTs [25–28] but to the best of our knowledge no reports are available on the shielding behavior of PTT/CNT composites. In this article, an attempt has been made to explore the potential of PTT/MWCNT composites as EMI shielding material by systematically investigating the effect of MWCNTs on room temperature electrical conductivity, dielectric properties, and EMI shielding behavior of PTT matrix in the frequency range of 8.2–12.4 GHz.

Experimental

Materials

The MWCNTs were prepared by chemical vapor deposition using thermal decomposition of toluene in the presence of iron catalyst obtained from organo-metallic ferrocene [29]. The detailed synthesis and characterization of MWCNTs using toluene as precursor and ferrocene (8%) as catalyst have been reported elsewhere [25]. Scanning electron microscope (SEM) and transmission electron microscope (TEM) were used to determine the average length, inner and outer diameter of carbon nanotubes, which were ~20–30 μm, ~10 and ~20–40 nm, respectively. Thermo-gravimetric analysis (TGA) was used to determine the purity of CNTs which was ~88%. The neat PTT, having viscosity average molecular weight (M_v) of 80,000, was procured from Futura Polymers (Chennai, India). M_v was determined using Mark–Houwink equation $\{[\eta] = K\,(M_v)^{\alpha}\}$. K and α are Mark–Houwink constants for polymer–solvent system at 30 °C. The values of K and α were taken as 5.36×10^{-4} g/dL and 0.69, respectively, from the literature and the viscosity was determined at 30 °C using phenol: 1,1,2,2-tetrachloroethane (3:2) as solvent [30].

Sample preparation

The PTT/MWCNT composites were prepared by melt mixing using co-rotating Dutch State Mines (DSM) micro-compounder. Before compounding, PTT was vacuum dried at 100 °C for 12 h. Compounding was done at 265 °C for 5 min at a screw speed of 170 rpm. PTT/MWCNT composites were prepared with varying amounts of MWCNT ranging from 0.24 to 4.76 vol% and samples were designated as PTT-0, PTT-0.24, PTT-0.48, PTT-0.95, PTT-1.43, PTT-2.40, and PTT-4.76 having 0, 0.24, 0.48, 0.95, 1.43, 2.40, and 4.76 vol% of MWCNTs, respectively. The volume percent of MWCNT was calculated using density of MWCNT as 2.1 g/cm^3 [31]. For EMI shielding and conductivity measurements, samples were prepared by compression molding using an electrically heated Carver hydraulic press. The molding was done by pressing the pre-dried pellets at 260 °C for 5 min under a pressure of 10 kPa.

Since PTT is prone to hydrolytic degradation in the presence of moisture which can deteriorate composite properties, intrinsic viscosity measurements were carried out before and after compounding. Single point viscosity measurement [30] was used to determine the intrinsic viscosity of neat PTT before and after compounding. Measurements were performed at 30 ± 0.1 °C using phenol: 1,1,2,2-tetrachloroethane (3:2) as solvent and polymer concentration of 0.5% (w/v). No change in intrinsic viscosity of PTT was observed before and after compounding. The measured intrinsic viscosity values were between 1.29 and 1.27 dL/g which is within the experimental error limit. Thus, we can say that compounding had no effect on the molecular weight of PTT.

Characterization

Morphology of PTT/MWCNT composites was investigated using SEM (EVO-50) and TEM (JEOL 2100F) operated at accelerating voltage of 20 and 200 kV, respectively. For SEM studies, manually fractured, gold-coated samples were used whereas for TEM studies, ultra-thin sections (30–80 nm) of composites were prepared using Leica Ultamicrotome.

The dc electrical conductivity of PTT/MWCNT composites was determined using van der Pauw geometry (four-probe contact method) on Keithley SCS 4200. For this purpose, rectangular pellets of dimensions 13 × 7 × 1.5 mm^3 were prepared by compression molding. The silver paste in conjunction with copper wire on each end of sample was used to make electrical contacts. The area of point contacts was 1–1.5 mm. The two pins of four probes were connected with the current terminals and the other two pins were connected with the voltage terminals and so the resistivity/conductivity of the samples was determined consequently. Reported electrical conductivity value is the average of three samples of each composition.

The EMI shielding and dielectric measurements were carried out on an Agilent E8362B Vector Network Analyzer in a microwave range of 8.2–12.4 GHz (X-band). Rectangular pellets (2 mm thick) were inserted in 22.86 × 10.14 × 2 mm^3 copper sample holder connected between the wave-guide flanges of network analyzer. Full two-port calibration was performed along with the sample holder to neglect any loss and power redistribution due to sample holder. At least three samples of each composition were tested and for each sample 201 data points were taken within the specified frequency range.

Results and discussion

Morphological characterization

Figure 1 shows SEM and TEM images of MWCNTs. From SEM image (Fig. 1a), we can say that MWCNTs exhibit highly curved and random coiled features due to the intrinsic Van der Waals attractions between the individual nanotubes in combination with high aspect ratio and large surface area. Figure 1b show that MWCNTs are hollow cylindrical. Significantly high aspect ratio (~1,000) of these CNTs suggests that it can easily form conductive network in the polymer matrix.

Figure 2a, c, e shows SEM micrographs of fractured surface of PTT/MWCNT composites having 0.24, 0.48, and 2.40 vol% MWCNTs. The SEM micrographs suggest that, up to 2.40 vol% MWCNT loading, MWCNTs are homogeneously dispersed in the polymer matrix and most CNTs are embedded within the matrix. TEM images (Fig. 2b, d, f) also show that MWCNTs are randomly distributed in the polymer matrix and at 0.48 vol% MWCNT loading it forms interconnected network structure. Formation of interconnected network was further proved by electrical conductivity data of PTT/MWCNT composites.

Electrical conductivity of PTT/MWCNT composites

The dc conductivity at room temperature of PTT/MWCNT composites as a function of MWCNT content (vol%) is shown in Fig. 3. The conductivity of composites increased with increasing MWCNT content and exhibited a dramatic increase (approximately five orders of magnitude) at 0.48 vol% of MWCNT loading which indicates percolation phenomenon. The percolation at such a low MWCNT loading can be attributed to efficient dispersion of MWCNTs in the polymer matrix and high aspect ratio of

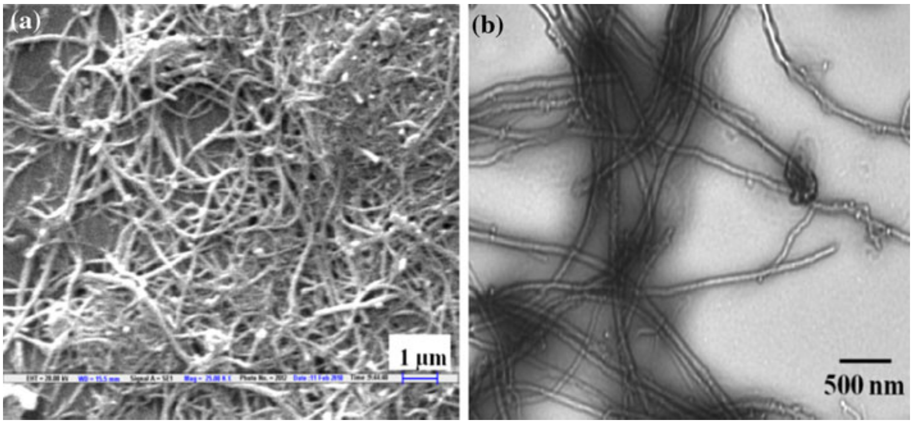

Fig. 1 SEM (**a**) and TEM (**b**) images of MWCNTs

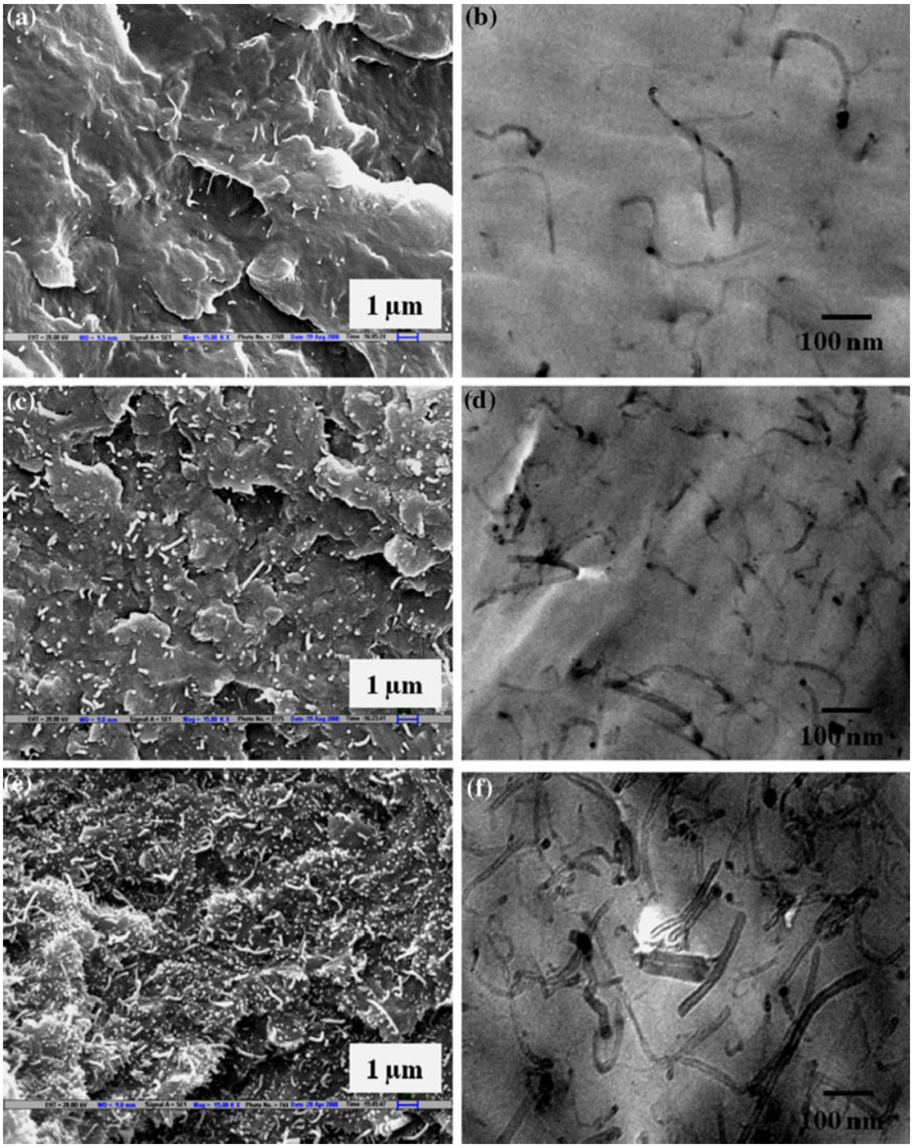

Fig. 2 SEM and TEM images of PTT composites **a**, **b** PTT-0.24, **c**, **d** PTT-0.48, and **e**, **f** PTT-2.40

MWCNT which remained unaffected during compounding. The high aspect ratio of MWCNT is very important in the formation of interconnecting network. Kovalchuk et al. [32] observed that for CNTs having aspect ratio ~25 and ~10, percolation could not be achieved even at 3.5 wt% CNTs loading. Relatively high value of percolation

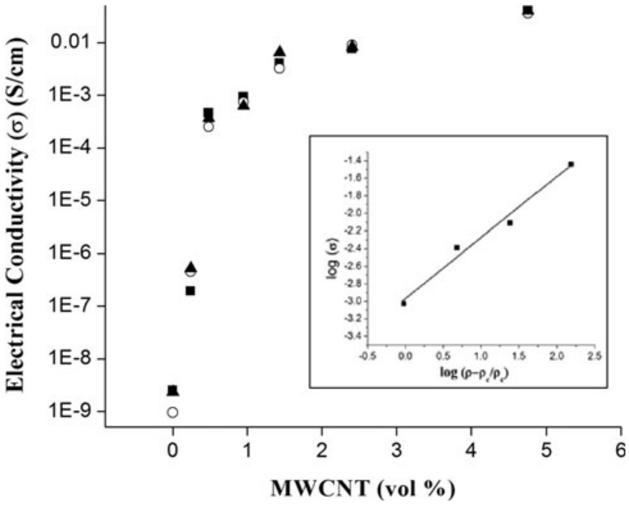

Fig. 3 Plot of dc electrical conductivity (σ) versus volume fraction of MWCNT in PTT/MWCNT composites measured at room temperature (*Inset* shows the log–log plot of σ versus $(\rho - \rho_c)/\rho_c$ of same composites)

threshold concentration reported for carbon black (CB) and CNF composites [33, 34] can be attributed to low aspect ratio of these fillers. The measured percolation threshold concentration (0.48 vol%) in this study is in the range of typical reported values for CNT/polymer composites [35].

According to the classical percolation theory, the conductivity of a conductor–insulator composite obeys the power law relationship, as shown by Eq. 1, around the percolation threshold concentration [36].

$$\sigma(v - v_c)^{\beta}, \qquad (1)$$

where σ is the conductivity of composite, v is the volume fraction of MWCNT, v_c is the volume fraction at percolation threshold, and β is the critical exponent, which is related to the system dimensions.

The log (σ) versus log $(\rho - \rho_c)/\rho_c$ plot is shown in the inset of Fig. 3; from the plot, we can say that conductivity of PTT/MWCNT composite agrees well with the percolation behavior predicted by Eq. 1. The least square fit line of the conductivity data in the log–log plot gave $\beta = 2.9$ at $\rho_c = 0.48$ vol%. The theoretical values of critical exponent (β) for a three dimensional percolating systems varies from 1.6 to 2.0 [36], but the experimental values for different polymer/carbon nanotube composites ranged from 1.3 to 5.3 [37–39]. The value of critical exponent ($\beta = 2.9$) obtained in this study is in good agreement with the reported values. After 0.48 vol% MWCNT loading i.e., percolation threshold, further addition of MWCNTs showed no significant change in electrical conductivity of PTT matrix. The maximum electrical conductivity of PTT/MWCNT composite at 4.76 vol% MWCNT loading was around 4×10^{-2} S/cm which is in the range of electrical conductivity (10^{-6}–10^{3} S/cm) reported for various CNT/polymer composites [35]. The electrical conductivity obtained in case of polymer composites filled with different conductive fillers like metals (copper, nickel, zinc, iron, silicon, silver, and aluminum powder), CB, carbon fiber, graphite, and CNF are in the range of 10^{-2}–10^{2} [40], 10^{-10}–10^{-2} [33], 10^{-12}–10^{1} [10, 41] 10^{-8}–10^{1} [9, 42], and 10^{-8}–10^{2} [34], respectively. The electrical conductivity of polymer composites depends on a number of factors such as nature, size, dispersion, percentage loading, and aspect ratio of filler. Among these variables, aspect ratio of fillers is very important in imparting conductivity to insulating polymer matrix. As CNTs have much higher aspect ratio compared to these conductive fillers, comparable electrical conductivity can be obtained at much lower weight fraction of filler. The maximum electrical conductivity obtained in this study is much higher than required electrical conductivity for EMI shielding material. The conductivity value is comparable to the value reported for PE/PP–CNT composites [43, 44] and higher than those reported for PET/PC–CNT composites [45, 46] at similar filler loading.

Complex permittivity of PTT/MWCNT composites

To investigate the dielectric behavior of PTT/MWCNT composites, we measured the room temperature complex permittivity of the composites in the frequency range of 8.2–12.4 GHz. Figure 4 shows the plot of complex permittivity versus frequency for composites having varying amounts of MWCNTs. The real and imaginary part of permittivity for composites having 0–0.48 vol% MWCNT were independent of frequency in the measured frequency range, whereas in case of samples having higher amounts of MWCNT i.e., 0.95–4.76 vol%, the real and imaginary part of permittivity decreased as the frequency increases from 8.2 to 12.4 GHz (Fig. 4).

Both real and imaginary parts of complex permittivity increased with increasing MWCNT loading. According to the theory of complex permittivity, when the electromagnetic radiation is incident on a shielding material, the electric field induces two types of electrical currents within the material, i.e., the conduction and displacement current, the former arising from free electrons and give rise to electric loss and the latter coming from the bound charges, i.e., polarization effect, which mainly involves the lattice defects. Accordingly, an increase of the real part of complex permittivity can be ascribed to dielectric relaxation and space charge polarization effect and an increase of imaginary parts of complex permittivity can be attributed to the enhanced electrical conductivity of the composites. It is also noted that the real and imaginary parts of complex permittivity exhibit a dramatic increase when the MWCNT loading was higher than 2.40 vol%. The maximum values

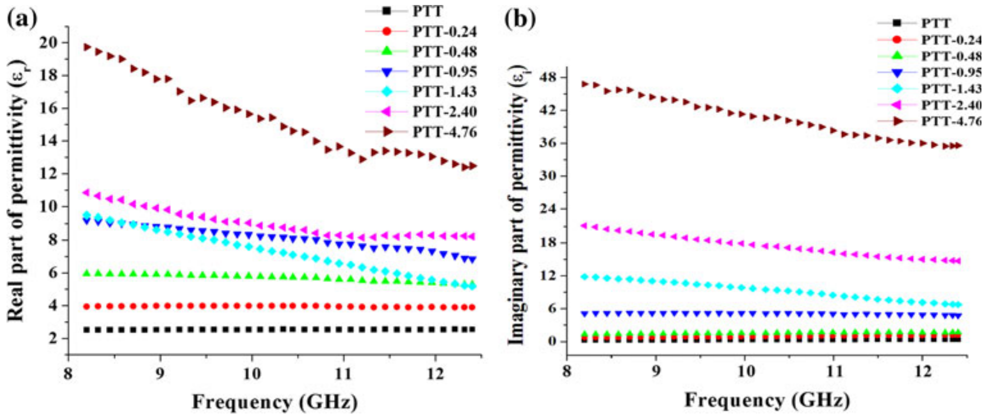

Fig. 4 Plot of real (ε_r) and imaginary (ε_i) part of permittivity versus frequency in PTT/MWCNT composites

of the real and imaginary parts of the complex permittivity for the composite containing 4.76 vol% MWCNT were 20 and 47, respectively, in the measured frequency range. These results show that PTT/MWCNT composites can be used as EMI shielding material in the measured frequency range.

The plot of tan δ versus frequency (8.2–12.4 GHz) for PTT/MWCNT composites at different MWCNT loading is shown in Fig. 5. In case of PTT/MWCNT composites, tan δ increases gradually with increasing amount of MWCNTs, whereas frequency showed no effect except for sample containing 4.76 vol% MWCNTs. The loss factor or the imaginary part of the permittivity reflects the energy loss due to charge conduction which depends on the conductivity of material. So, it was observed that as the conductivity of material increased upon addition of CNT, tan δ also increased. For the composite containing 4.76 vol% of MWCNTs, tan δ increased from 0.08 to 2.8. Thus, the increase in tan δ upon incorporation of MWCNTs can be attributed to phase transition of material i.e., conversion from insulator to conductor [11, 47, 48].

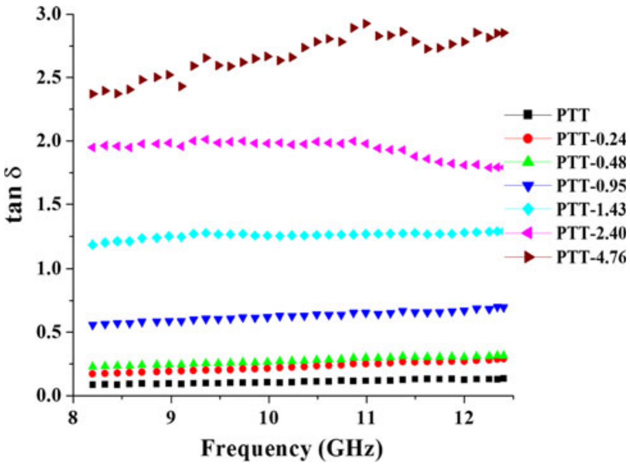

Fig. 5 Plot of tan δ versus frequency in PTT/MWCNT composites

EMI SE of PTT/MWCNT composites

The incident electromagnetic radiation on a shield can be resolved into three part, namely, absorption (A), reflection (R), and transmittance (T), whose sum ($A + R + T$) is equal to 1. Hence, the total EMI SE (SE_{total}) is the sum of contributions from absorption loss (SE_A), reflection loss (SE_R), and multiple reflection (SE_M), i.e., $SE_{total} = SE_A + SE_R + SE_M$. The SE_{total} of shielding material can be written as $SE_{total} = -10 \log T$, where T is the transmittance value which can be measured from the ratio of P_t to P_0 i.e., $T = (P_t/P_0)$ [49]. The P_t and P_0 are transmitted and incident power, respectively. Considering the effective absorbance (A_{eff}), which is defined as $A_{eff} = (1 - R - T)/(1 - R)$ with respect to the power of the incident electromagnetic wave inside the shield, the SE due to reflectance and effective absorbance can be described as $SE_R = -10 \log (1 - R)$, $SE_A = -10 \log T/(1 - R)$. Using these equations the total SE was resolved into absorption and reflection loss.

The effect of MWCNT content and frequency on EMI SE is shown in Fig. 6a. It was observed that the EMI SE for the PTT/MWCNT composites is almost independent of frequency in the measured frequency range; however, EMI SE increased with increasing MWCNT content. In this study, maximum EMI SE value of ~23 dB was obtained at 4.76 vol% MWCNT loading. Although very high SE value (62 dB) [50] have been reported for CNT composites at much higher CNT content (50 wt%), our data is comparable or higher than many reported EMI SE value for polymer/CNT composites containing equivalent CNT loading [15, 16, 20, 51]. The higher EMI SE obtained in this study can be attributed to efficient processing method and high aspect ratio of CNTs. Finally, we can say that the target value of the EMI SE needed for commercial applications, ~20 dB, could be obtained at 4.76 vol% MWCNT loading in PTT matrix.

The effect of MWCNT content on SE_A and SE_R is shown in Fig. 6b. We observed that with increasing

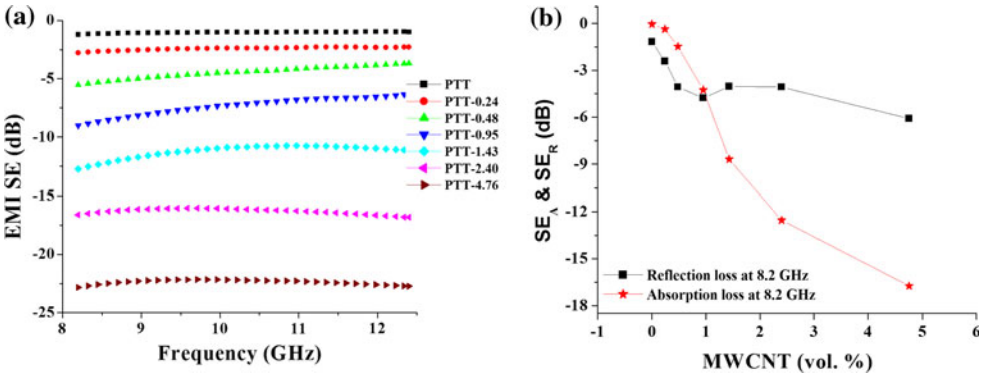

Fig. 6 **a** EMI SE of PTT/MWCNT composites in the frequency range from 8.2 to 12.4 GHz and **b** contribution of reflection and absorption loss to EMI SE in PTT/MWCNT composites

Table 1 Results of electrical conductivity and reflection/absorption contribution to the total SE

Sample designation	CNTs (vol%)	Electrical conductivity (S/cm)	Reflection loss (dB) at 8.2 GHz	Absorption loss (dB) at 8.2 GHz	Total EMI SE (dB)
PTT	0	1.5×10^{-9}	1.16 ± 0.2	0.10 ± 0.04	1.2 ± 0.2
PTT-0.24	0.24	1.9×10^{-7}	2.60 ± 0.23	0.51 ± 0.16	3.0 ± 0.5
PTT-0.48	0.48	4.6×10^{-4}	3.88 ± 0.17	1.26 ± 0.19	5.2 ± 0.4
PTT-0.95	0.95	9.3×10^{-4}	4.44 ± 0.32	5.08 ± 0.83	9.3 ± 0.7
PTT-1.43	1.43	4.0×10^{-3}	4.16 ± 0.16	7.96 ± 0.64	12.0 ± 0.7
PTT-2.40	2.40	7.6×10^{-3}	4.33 ± 0.27	12.6 ± 0.20	17.0 ± 0.2
PTT-4.76	4.76	4.5×10^{-2}	6.09 ± 0.5	16.0 ± 0.67	22.0 ± 0.6

amounts of MWCNT, both SE_A and SE_R increased but rate of increase of SE_A was higher than that of SE_R, e.g., at 0.24 vol% MWCNT loading, absorption contribution was only 16% which increased to 73% at 4.76 vol% MWCNT loading. On the basis of these results, we can infer that the primary shielding mechanism of PTT/MWCNT composites is absorption rather than reflection in the measured frequency range. Significant increase in tan δ value (more than 20 times on 4.76 vol% MWCNT loading) also confirms microwave absorbing property of these composites.

The SE of a conductive composite is related to its electrical conductivity as shown in Table 1 and Fig. 7. The results show that on increasing MWCNT content, both electrical conductivity and EMI SE of PTT/MWCNT composites increased. The SE_R depends on the conductivity of the material. At 0.48 vol% MWCNT loading, significant increase in electrical conductivity was accompanied with similar increase in SE_R. The reason for this behavior was the formation of interconnected network at this concentration i.e., the insulating PTT matrix became conductive at and above this concentration which lead to increase in SE_R. As there was no significant increase in electrical conductivity on further addition of MWCNT, SE_R did not show any significant change. However, the behavior of SE_A is completely different from SE_R on increasing MWCNT loading. The SE_A kept on increasing with increasing MWCNTs, even after percolation concentration, as it depends on the polarization and charge transfer which increases on increasing CNT loading. With increasing amount of MWCNTs, number of percolating networks increased which interacted with incident radiation and lead to the higher SE_A. Thus, we can say that, PTT/MWCNT composite is absorption-based EMI SE material and shielding efficiency strongly depends on MWCNT content, electrical conductivity, and dielectric properties of the material.

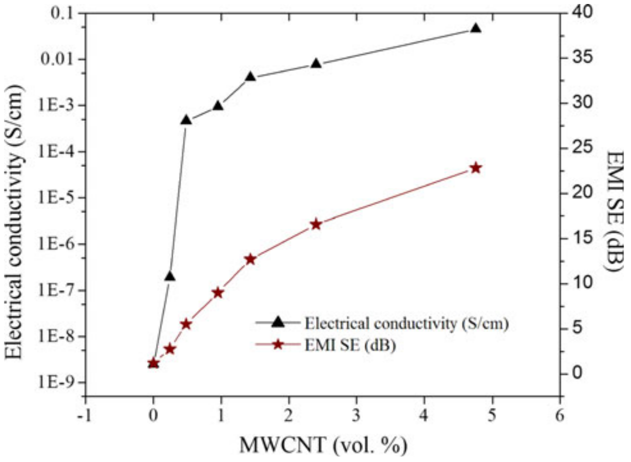

Fig. 7 Effect of MWCNT content on EMI SE and electrical conductivity (σ) of PTT

Conclusions

In this study, well-dispersed PTT/MWCNT composites with varying amounts of MWCNTs (0.24–4.76 vol%) were prepared by melt compounding. The electrical conductivity, permittivity, and EMI SE were found to increase with increasing MWCNT loading. Electrical percolation was observed at 0.48 vol% MWCNT loading which indicates the formation of interconnected networks of MWCNTs in the polymer matrix. The EMI shielding behavior of the composite was measured in the frequency range of 8.2–12.4 GHz and the maximum SE ~23 dB was obtained at 4.76 vol% MWCNT loading. The study on the shielding mechanism of PTT/MWCNT composite suggests that absorption loss is the dominant shielding mechanism which depends on the amount of MWCNT loading. Electrical conductivity, dielectric properties, and MWCNT loading plays significant role in increasing the EMI SE of the PTT/MWCNT composite. Finally, we can say that PTT/MWCNT composite has sufficient electrical conductivity and EMI SE at 4.76 vol% MWCNT loading to be used as a microwave absorbing material.

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