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Journal Name

ARTICLE

Lightweight and solution processible thin sheets of poly (o-toluidine)-carbon fiber-novolac composite for EMI shielding

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An attempt has been made to generate lightweight and processible thin sheets of poly (o-toluidine)-carbon fiber (PoTCF) composite for electromagnetic interference shielding. PoTCF composite synthesized by oxidative emulsion polymerization was physically blended with different weight ratios of novolac resin to prepare a new class of composite materials, which were further processed to form thin sheets (thickness ~0.85 mm) by compression moulding technique. *In-situ* incorporation of carbon fiber into the polymer matrix leads to the formation of composite with improved mechanical, thermal, electrical & shielding properties, which were further optimized by varying the amount of novolac resin into the sheets. Structural and morphological studies were carried out by UV-vis, FT-IR, XRD and SEM. PoTCF composite sheets with 50 wt. % loading of novolac resin have flexural strength of 36.0 MPa and exhibit a shielding effectiveness of 24 dB in X-band (8.2- 12.4GHz) at a critical thickness of ~2.11 mm, which is more than the limit required for techno-commercial applications. Therefore, indigenously fabricated polymer based sheets would be potentially used for making durable enclosures for electronic equipments.

1. Introduction

In recent years, conducting polymers have occupied a major place and pivotal position in the field of material science.[1, 2] Intrinsically conducting polymers that represent an attractive class of materials for electromagnetic interference (EMI) shielding applications offer many advantages over the other conventional materials. Although a number of conducting polymers and their composites are known, yet looking for new shielding materials continues to be a subject of endless research. Among all conducting polymers, polyaniline (PANi) is perhaps the most promising polymer used for EMI shielding because of its desirable properties such as chemical stability, environmental stability and high conductivity at microwave frequencies.[3, 4] But the limited solubility and processibility of PANi, due to the rigid and stiff backbone of its main chain, restricts its fabrication in pure form and put a limit to the large scale applications. To overcome this problem, various routes have been adopted; two main routes which have been worked out are; derivatization of the polymer backbone[5, 6] and incorporation of a specific dopant in the polymer network.[7-9] The first route is simple one and advantageous, as the polymers of aniline derivatives exhibit greater solubility and hence good processibility. However, the lower conductivity of the PANi derivatives as compared to PANi[6] does not allow their use for shielding application, but have been used for antistatic coating,[10] sensors,[11] solar cells,[12] corrosion inhibitors[13] etc. To take the advantage of their processibility and to exploit them for shielding application their conductivity has to be improved. The conductivity of these polymers can be tailored either by doping with different protonic acids[14, 15] or by incorporation of conducting fillers to make their composites. Use of conducting fillers to make electrically conductive polymer composites, has attracted the attention of researchers for the development of new functional materials. These electrically conductive composites manifest the properties which cannot be achieved by the individual component. These materials have immense potential for their use in EMI shielding application. When the conducting polymers are taken in the form of composites with conductive fillers, their electrical as well as mechanical properties boost up.[16, 17] Numbers of articles have reported the EMI shielding properties of conducting polymer composites.[18, 19] The conductivity of these heterogeneous systems depends on many factors like the concentration of conducting fillers, their shape, size, orientation and interfacial interaction between fillers and host matrix. Previous reports have already demonstrated that polymer/conducting polymer composites filled with conductive and magnetic fillers, like carbon fiber, CNT, graphene, magnetic nanoparticles, flyash etc., have improved shielding characteristics and outperform conventional metal shielding due to their light weight, resistance to corrosion, flexibility and processing advantages.[20-24] Among these the composites based on low cost carbon fiber have demonstrated the formation of a novel composite with superior microstructure and improved mechanical as well as shielding properties.[16, 17, 25, 26] These composites are very promising for use as an effective and practical EMI shielding material owing to their light weight, low cost, easy processiblity with good thermal, mechanical & shielding properties. So these lightweight shielding materials have gained much popularity and preferred in most of the techno-commercial, aeronautical & military applications, where light weight is desirable for the means of material & energy savings. These materials have been used in various forms, like coatings, mesh wires, foils, conductive tapes, foams, films, sheets etc., for enclosures of electronic equipments to optimize EMI shielding. The aim of our work is to fabricate durable poly (o-toluidine)-carbon fiber (PoTCF) composite sheets using novolac resin as binder and find their use for EMI shielding applications. Among carbon materials as

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conductive fillers, carbon fiber is of great importance because of its proven use as shielding material.[26, 27] In the present work incorporation of carbon fiber into the polymer matrix fulfilled the desired value of conductivity for shielding. Mechanical and thermal stability of sheets are improved by both CF and novolac resin. To the best of our knowledge use of Poly (o-toluidine) composites has not been thoroughly explored for EMI shielding application till date.

2. Experimental

2.1 Materials

Ortho-toluidine (o-methyl aniline) (Merck, India), β-naphthalene-2-sulphonic acid (β-NSA, Himedia, India) and ammonium peroxydisulfate (APS, Merck, India) were used in the present study. Ortho-toluidine was freshly distilled before use; other chemicals were used as received. The novolac resin (Pheno-Organic, India) was used as a binder and PAN based carbon fiber (CF, Toray-T-300-Japan) were used as reinforcement material to improve mechanical strength. Aqueous solutions were prepared using double distilled water having specific resistivity of 10^6 Ω-cm.

2.2 Synthesis of PoTCF composite & fabrication of thin sheets

PoTCF composite was prepared by chemical oxidative emulsion polymerization of o-toluidine in the presence of CF as filler and NSA as surfactant as well as dopant. The detailed synthesis process is same as that of our previous report given for the synthesis of polyaniline-carbon fiber composites.[17] In addition, poly (o-toluidine) (PoT) without CF was also synthesized by the same route for comparative study.

Further, thin sheets of PoTCF were fabricated using solution based processing method. Novolac resin was used as a binder and the concentration of resin was varied (25, 50 and 75 wt. % of PoTCF composite) to optimize the thermal, mechanical & electrical properties of sheets. The fabrication procedure for PoTCF sheets is as follows:- first of all, measured amount of PoTCF was dispersed in ethanol by means of ultrasonication for two hours and thereafter, adequate amount of novolac resin (25, 50 and 75 wt. %) was added, keeping the PoTCF plus phenolic resin wt. % as 100. The solution was then sonicated for two hours, homogenized for 30 min and then poured into a specially designed vessel attached with vacuum filtration (see supporting material, fig. S1). These obtained sheets were then pressed in a compression mould at 100°C and contact pressure. Further, curing of sheets was carried out at 150°C for 2 hours. The compression moulded sheets were then ejected out from the mould after cooling up to room temperature. PoTCF thin sheets with 25, 50 and 75wt. % loading of novolac resin so prepared were abbreviated as PoTCF25, PoTCF50 and PoTCF75.

2.3 Characterization

SEM (Zeiss EVO MA-10) is used to study the morphology of PoTCF composites and sheets. Structural analysis was done by X-ray diffraction (XRD) studies, carried out on a D8 advance X-ray diffractometer (Bruker) using CuKα radiation (λ= 1.540598 A^0) in scattering range (2θ) of 3°- 60° with a scan rate of $0.02^{\circ}sec^{-1}$ and slit width of 1 mm. Thermogravimetric analysis (TGA) of the samples was carried out by Mettler Toledo TGA 851^e. Fourier transform infrared (FTIR) analysis was carried out on Nicolet 5700 in the wavenumber range of 3500-500 cm^{-1}. UV-vis absorption spectra were recorded on Shimadzu 1601 spectrophotometer in the wavelength range of 200-1100 nm (see supporting data information, fig. S2). Flexural strength of PoTCF sheets was measured by three point bending test based on ASTM D790 [28] using an INSTRON Universal Testing Machine (model 4411) at a crosshead speed of 0.5 mm/min. Room temperature conductivity was measured by four-probe technique using Keithley programmable current source (model 6221) and nanovoltmeter (model 2182 A). PoTCF sheets with dimension 13×7 mm were cut and four ohmic contacts were made on each end using silver paste. Shielding measurement was performed on an Agilent E8362B Vector network analyzer in microwave range of 8.2 to 12.4 GHz (X-band). Measurements were carried out using 15.8×7.9×6 mm^3 copper sample holder connected between the waveguide flanges of network analyzer.

3. Result and Discussion

3.1 Scanning electron microscopy

Fig. 1a shows SEM image of PoT, Fig. 1b displays the low resolution optical image of PoTCF50 sheet and Fig. 1c shows the high resolution optical micrograph of PoTCF50 sheet.

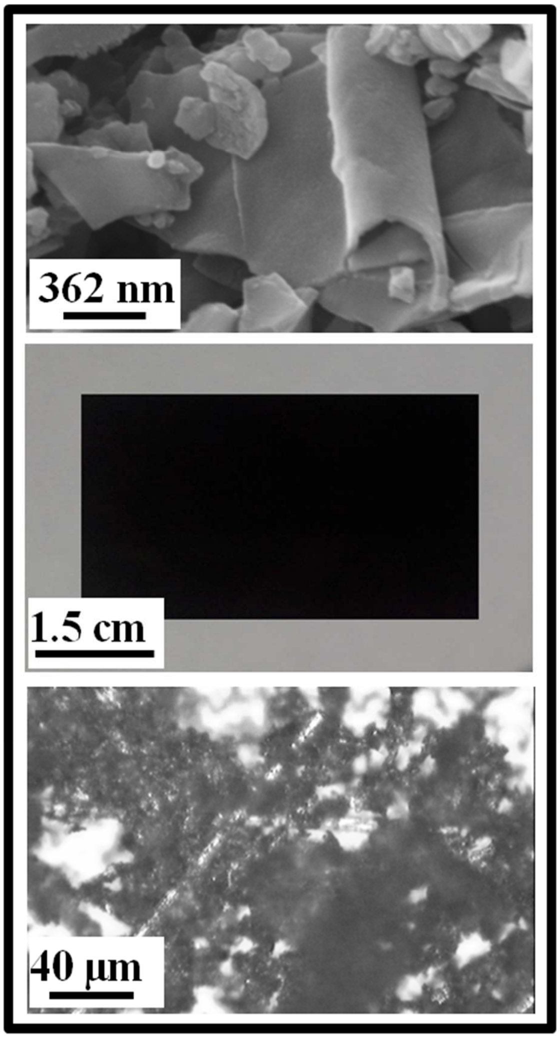

Fig. 1 (a) SEM image of PoT, (b) Low resolution optical image of PoTCF50 sheet and (c) High resolution optical micrograph of PoTCF50 sheet.

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SEM image of NSA doped PoT was found to have tubular morphology, which is similar to the previous reports.[29] Morphology of the polymers is affected by the nature and concentration of dopant as well as polymerization methods. Wan et al. has shown that the lower concentration of NSA favoured the formation of complete tubules of PoT NSA, whereas a high concentration of NSA was required to form the complete PANi NSA tubules, moreover the size of tubules could be controlled by changing the concentration of NSA.[29] Fig. 1a displays the formation of PoT tube, which confirms that the tubes of PoT prepared in NSA medium are hollow and porous in nature. Fig. 1b and 1c show the low and high resolution optical micrographs of PoTCF sheet. The PoTCF composite prepared by *in-situ* polymerization leads to uniform coating of PoT particles on the surface of CF[17]. The interaction between PoT particles and CF facilitates the charge transfer between the two components thereby increasing the conductivity of PoTCF composite. The homogenous coating of PoT on CF does not cause any deformation in the longitudinal appearance of CF as shown in Fig. 1c. Therefore PoT is physically attached to CF surface thereby improving the mechanical strength of polymer.

3.2 X-ray diffraction

Crystallinity and orientation of conducting polymers have been of much interest, because more highly ordered systems can display a metallic- like conductive state. The XRD patterns provide significant information in relation to the nature and structure of the sample. The X-ray diffraction patterns for PoT, PoTCF, PoTCF25, PoTCF50 and PoTCF75 were shown in Fig. 2a, which show the semi crystalline nature of PoT and PoTCF. In the XRD curve of PoT, a sharp peak at 2θ = 8.5° (d = 10.36A°) was observed, which was assigned as the repeat unit of polyemeraldine chain, indicating that the introduction of the $–CH_3$ group does not change the repeat unit of the polymeric chain of PANi. In addition, the sharp peaks at 2θ = 6.74°, 18.78°, 19.55° and 23.74° corresponding to d = 13.09, 4.72, 4.54 and 3.74 A°, ascribed to the periodicity parallel and perpendicular to the polymer chain, were also observed.[29] The XRD curve of PoTCF is similar to PoT, due to the coating of PoT molecules on CF, except slight change in the peaks between 2θ = 24°-27°. All the main peaks of CF are suppressed in the PoTCF which confirms that CF has uniform and thick polymer coating on its surface. In the XRD curves of PoTCF25, PoTCF50 and PoTCF75 sheets the crystallinity decreases because of the presence of phenolic resin which is amorphous in nature but the signatures of PoT peaks are visible in all curves. Also, with the increase in the loading of phenolic resin some new peaks start to appear that may be because of the presence of resin in the composite.

3.3 FTIR spectra

Fig. 2b shows the FTIR spectra of PoT, PoTCF and PoTCF25 samples. The characteristic bands of PoT doped with NSA are similar as observed previously.[15] A characteristic band at 3220 cm^{-1} is due to N-H stretching of amine group, the peak at ~2963 cm^{-1} can be assigned to the stretching vibration of the methyl ($-CH_3$) group. The two bands appearing at 1580-1590 cm^{-1} and ~1508 cm^{-1} correspond to the stretching vibration of the quinoid and benzenoid ring, respectively. The characteristic peak at 1315 cm^{-1} is attributed to C-N stretching in Q-B-Q sequence, the peaks at 948 and 813 cm^{-1}were attributed to in plane C-H vibration and out of plane C-H vibration of quinoid rings, whereas a strong band characteristically appears at 1168 cm^{-1}, which has been explained as electronic or vibrational band of nitrogen quinine, exhibits the degree of electron delocalization in conductive PoT, responsible for electrical conductivity.[30, 31] The presence of $-SO_3$ group is confirmed by the presence of band at 1022 cm^{-1} in all the spectra.[32] On comparing spectra of PoT with PoTCF & PoTCF25 samples, it is observed that there is appearance of no new bands except slight shifting of some bands only. This indicates that there is only some ionic interaction of novolac with PoT & CF, and no net chemical reaction.

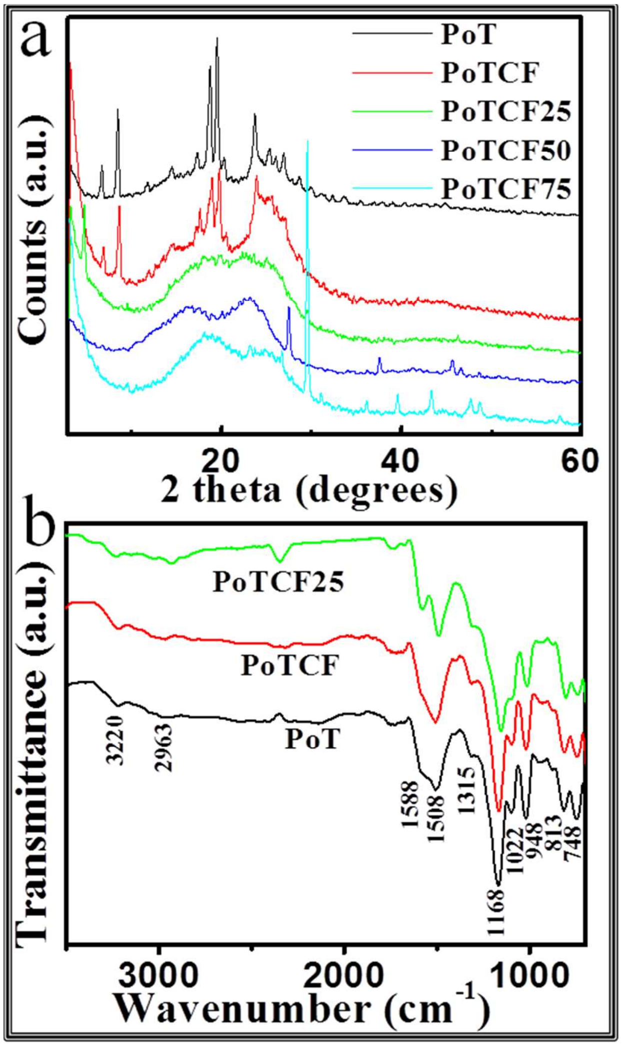

Fig. 2 (a) XRD spectra of PoT, PoTCF sheets and (b) FTIR spectra of PoT, PoTCF and PoTCF25 composites.

3.4 Thermogravimetry

The TGA curves of the synthesized PoT, PoTCF composite & PoTCF sheets are displayed in Fig. 3a. All the samples show similar thermal degradation and undergo a three stage weight loss pattern. The first stage weight loss up to 115 °C corresponds to the loss of water molecules present in the polymer matrix. The second stage loss, from 285 to 440 °C is associated with the loss of dopant molecules from the polymer matrix. The weight loss after 440 °C is due to the complete degradation and decomposition of the polymer backbone.[33] On comparing the thermograms of PoT with

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thermograms of PoTCF composite and PoTCF sheets (Fig. 3a), it is found that, in case of PoTCF sheets, the loss of dopant continues after 440 °C. Also the third stage weight loss in case of PoTCF & PoTCF sheets are less conspicuous as compared to the third stage weight loss in case of PoT. Weight of residue left at 800 °C for PoT, PoTCF, PoTCF25, PoTCF50 & PoTCF75 are 40%, 45%, 49%, 51% & 54% respectively. All these findings confirm that the thermal stability increases by incorporation of CF into polymer matrix and blending of PoTCF composite with novolac resin, which further increases with increase in the wt. % of novolac resin into the composite.

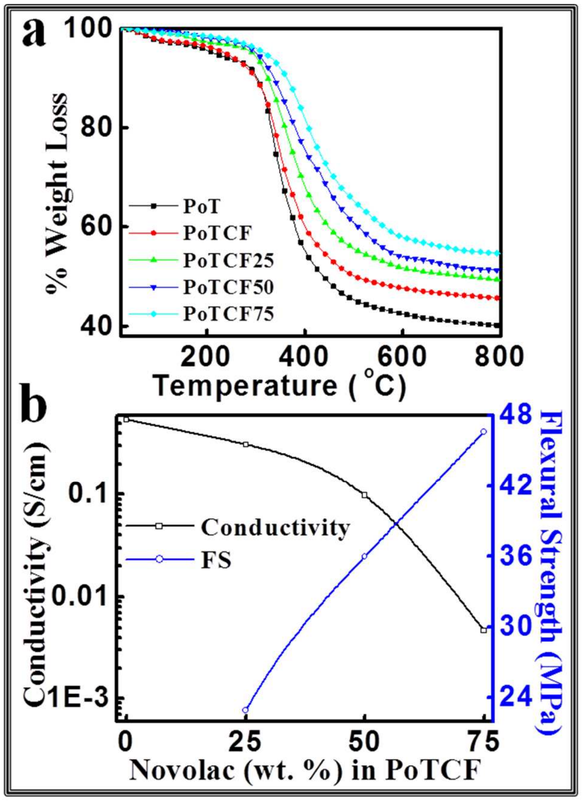

Fig. 3 (a) TGA curves of PoT, PoTCF and PoTCF sheets and (b) Variation of conductivity and flexural strength as a function of wt. % of novolac resin in PoTCF sheets.

3.5 Conductivity & Flexural strength

Table 1 shows the room temperature conductivities of PoTCF composite and sheet samples, measured by four probe method. Generally, conducting PANi with substituents on their frameworks showed lower conductivities compared with those of the unsubstituted PANi. Leclerc et al. proposed that the presence of bulky substituents in the polymer chain can induce some nonplanar conformations that decrease the conjugation along the backbone.[34] The methyl group on the phenyl ring in PoT can be expected to increase the torsional angle between adjacent rings to relieve steric strain and then lead to the lower conductivity of PoT as compared to unsubstituted PANi.[35] The conductivity of blank PoT is of the order of 2.62×10^{-3} S/cm which is of the same order as of previous reports,[15] while upon addition of CF the conductivity increases to 5.4×10^{-1} S/cm (see supporting data, table S1). When PoTCF composite powder was transformed into sheets by blending with novolac resin, the conductivity was found to decrease (Fig. 3b), but this decrease in conductivity is small up to low loading of the resin (25 wt. %). It could be suggested that the thermal curing during moulding of sheets affects the chain alignment of the polymer, which leads to the increase of conjugation length and that maintains moderate conductivity of the sheets, in spite of addition of insulating polymer into PoTCF composite. The decrease in conductivity is noticeable, when the amount of resin is increased beyond 25 wt. %. An expected high decrease in conductivity found at 75 wt. % loading of resin (4.68×10^{-4} S/cm), because the higher amount of insulating resin hamper the free flow of electrons in the composite sheets. PoTCF50 also shows fair conductivity because up to 50 wt. % loading of resin the formation of conducting network is favoured. But, at 75 wt. % loading of resin the electrical conductivity and EMI shielding properties have been adversely affected due to insulating nature of resin so the study is restricted beyond this limit. The conductivity of PoTCF composite powder samples having different amount of novolac resin is less than their respective sheet samples (see supporting data), this is due to the increase in connectivity after moulding of sheets and also the excess of novolac resin squeezes out from the sheets during the thermal curing of sheets.

Flexural strength of the composite sheets was measured by three point bending test based on ASTM D790[28] and shown in Table 1. These values show that PoTCF composite sheets have sufficient strength to be used as external enclosure for electronic equipments for EMI shielding. It is observed that flexural strength increases with increase in the amount of novolac resin in PoTCF composite (Fig. 3b). This improvement in the flexural strength of sheets is due to strong binding nature of novolac resin. With this, CF used as filler in composite sheets is also responsible for the strength. It has been reported by some research groups that incorporation of carbon fiber and CNT's in the conjugated polymer matrix improves the electrical and mechanical properties.[36]

Table 1 Flexural strength, electrical conductivity & shielding effectiveness of the PoTCF sheets.

Sample	Phenolic Resin (wt. %)	CF (wt. %)	Flexural Strength (MPa)	Conductivity (S/cm)	Shielding Effectiveness (dB)	Thickness of Sheet (mm)
PoTCF25	25	20	22.9	3.10×10^{-1}	9.1	0.81
PoTCF50	50	20	36.0	9.80×10^{-2}	16.0	0.85
PoTCF50*	50	20	–	–	18.8	1.26
PoTCF50*	50	20	–	–	23.9	2.11
PoTCF75	75	20	46.6	4.68×10^{-4}	8.1	0.90

*Samples having higher thickness

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3.5 EMI Shielding

The EMI shielding effectiveness (SE) of a material is defined as the ratio of transmitted power to incident power and given by

$$SE_T\,(dB) = 10\log(P_T/P_I) = 20\log(E_T/E_I) = 20\log(H_T/H_I)$$

Where, P_I (E_I or H_I) and P_T (E_T or H_T) are the power (electric or magnetic field) of incident and transmitted EM waves respectively.

To investigate the microwave attenuation performance of the PoTCF composite sheets the scattering parameters (S_{11} & $S_{21)}$ were measured by vector network analyzer to calculate the absorption coefficient (A), reflection coefficient (R), transmittance coefficient (T) and absorption efficiency of the sheets. The reflection and transmission coefficient were calculated using scattering parameters as, $R = |E_R/E_I|^2 = |S_{11}|^2 = |S_{22}|^2$ and $T = |E_T/E_I|^2 = |S_{21}|^2 = |S_{12}|^2$. The absorption coefficient and effective absorbance (A_{eff}) was calculated as $A = (1 - R - T)$; $A_{eff} = (1-R-T)/(1-R)$ respectively.[37] Fig. 4a & 4b show the calculated transmission coefficient and absorption efficiency values of the PoTCF sheets having thickness ~0.85 mm in the frequency range of 8.2-12.4 GHz. The transmission coefficient (T) is minimum (0.03) for PoTCF50 sheet. For all PoTCF sheets the value of T is less than 0.22 as shown in Fig. 4a which is because of the higher value of reflection and absorption coefficients, this proposed that more wave energy is attenuated by the PoTCF sheets leading to decrease in the T value. Also, the absorption efficiency of the PoTCF50 sheets was 87 and it reaches upto 99.9 at a thickness of 2.11 mm. The total SE_T is the sum of SE due to reflection (SE_R) and SE due to absorption (SE_A) and given by $SE_T = SE_R + SE_A$, SE_R & SE_A were calculated using equations

$$SE_R = -10\log(1-R) \text{ and } SE_A = -10\log(1-A_{eff}) = -10\log(T/1-R).^{21,\,23}$$

Fig. 5a shows the variation of SE_R and SE_A of PoTCF sheets with frequency in the 8.2 to 12.4 GHz frequency range. It has been observed from the figure that the total shielding effectiveness (SE_T) value is equally shared with SE_R and SE_A. From the experimental measurements, the shielding effectiveness values due to absorption (SE_A) is found to be 3.1, 8.7 & 3.3 dB for PoTCF25, PoTCF50 and PoTCF75 respectively, while the shielding effectiveness due to reflection (SE_R) is 6.0, 7.2 & 4.8 dB for PoTCF25, PoTCF50 and PoTCF75 respectively.

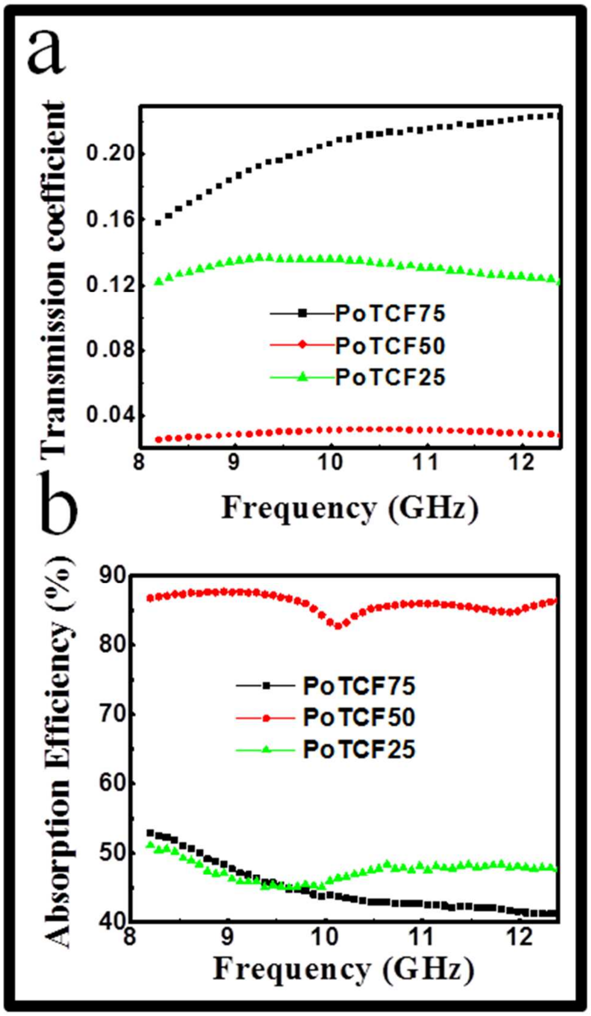

Fig. 4 (a) Transmission coefficient and (b) Absorption efficiency of PoTCF sheets at a thickness of ~0.85 mm.

Table 2 Electromagnetic shielding effectiveness of different conducting polymer-based composites

Polymer Matrix	Filler	Filler Wt. %	Frequency Range (GHz)	Total Shielding Effectiveness (dB)	Refrence
Polyaniline	Carbon Black	30	2-18	40	Wu *et al.*[38]
Polyaniline	CF	20	8.2-12.4	35	Kumar *et al.*[16]
Polyaniline	Graphene	33	2-18	34.2	Bingqing *et al*[22]
Polyaniline	SWCNT	25	2-18	31.5	Bingqing *et al.*[22]
Polyaniline	Fe_3O_4	66	12.418	11.5	Singh *et al.*[39]
Polyaniline	CF	20	8.2-12.4	31.9	Joon *et al.*[17]
Polyaniline	Fly	75	12.4-18	32	Singh *et al.*[40]
Polypyrrole	CF	25	12.4-18	23	Varshney *et al.*[25]
Poly-o-toluidine	CF	20	8.2-12.4	24	Present study

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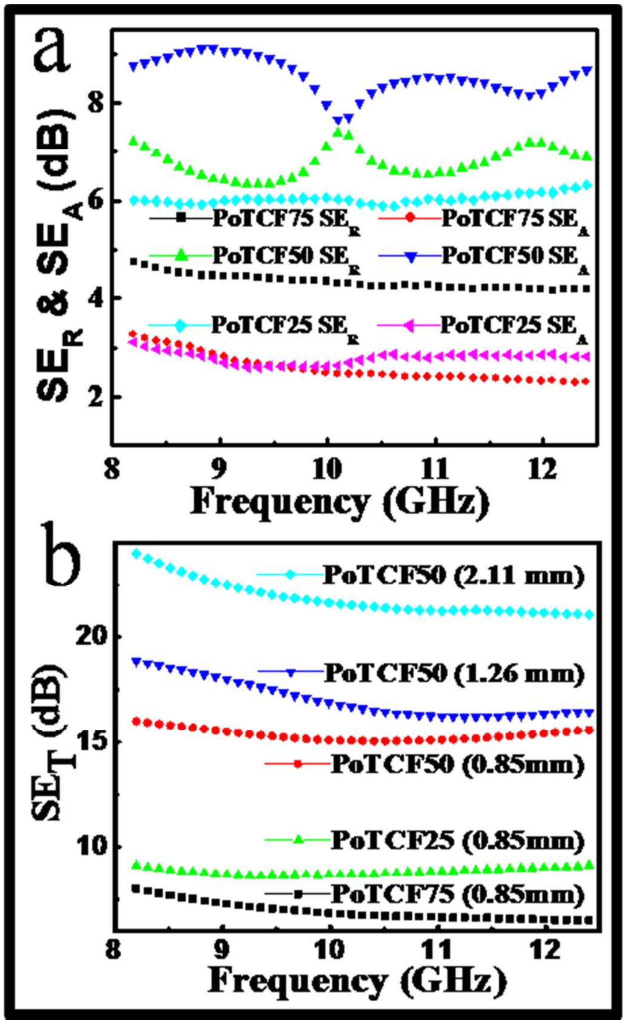

Fig. 5 Variation of shielding effectiveness with frequency in 8.2-12.4 GHz range, showing effect of novolac concentration, (a) SE$_A$ & SE$_R$ of different sheets, (b) SE$_T$ of different sheets & SE$_T$ of PoTCF50 sheets with different thickness.

Fig. 5b shows the total shielding effectiveness SE$_T$ of PoTCF25, PoTCF50 and PoTCF75 which is found to be 9.1, 16.0, 8.1 dB respectively at a matching thickness of ~0.85 mm. It is clear from the graph that SE$_T$ is found to be maximum for PoTCF50. At 50 wt. % loading of novolac resin strong fiber-matrix interface interaction is created. The conductive PoTCF composite sheets have charge storage capacities capable of absorbing electromagnetic waves by electric polarization. Fig. 5b also shows the variation of SE$_T$ with frequency, for PoTCF50 sheets at varying thickness of 0.85 mm, 1.26 mm and 2.11 mm, which shows that by varying the thickness of the composite sheets the total shielding effectiveness SE$_T$ increases from 16 to 24 dB. Thus we can increase the value of total shielding effectiveness (SE$_T$) by increasing the thickness of sheets to bring it to value sufficient for techno-commercial as well as military applications. Apart from thickness of the sample, shielding effectiveness depends on many other factors like the penetration depth (See supporting material data) polymer matrix type, concentration of conductive filler, the fabrication method and processing conditions. Although the shielding performance of composite materials cannot be compared on the basis of literature data because EMI shielding varies with many uncontrolled factors, yet the synthesized light weight PoTCF composite having satisfactory value of SE is compared (Table 2) with some of the recently reported conducting polymer composites. On the bases of experimental measurements and comparison with literature data, it is now clear that poly (o-toluidine) with CF, which has not been explored for shielding applications, may have the potential to be used as a convenient material for EMI shielding.

4. Conclusion

PoTCF thin sheets were prepared by low cost facile method. By *in-situ* incorporation of CF into PoT matrix, the thermal, mechanical, electrical & shielding properties of these sheets were improved and further optimized by using varying amount of novolac resin as a binder. Optimum amount of novolac resin increases the thermal and mechanical properties of PoTCF sheets without affecting the shielding properties significantly. It is found that these sheets have improved thermal stability and electrical conductivity was found to be between 3.1×10^{-1} to 4.68×10^{-4} S/cm. PoTCF50 sheets is found to have maximum shielding effectiveness (24 dB at a thickness of 2.11 mm) with flexural strength of 36.0 MPa. From the obtained data it is concluded that PoTCF sheets, due to their lightweight, good processibility and sufficient electromagnetic shielding effectiveness at thin matching thickness, can be added as an economical material into the list of electromagnetic wave absorbers for military as well as other techno-commercial applications.

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Lightweight and solution processible thin sheets of poly (o-toluidine)-carbon fiber-novolac composite for EMI shielding

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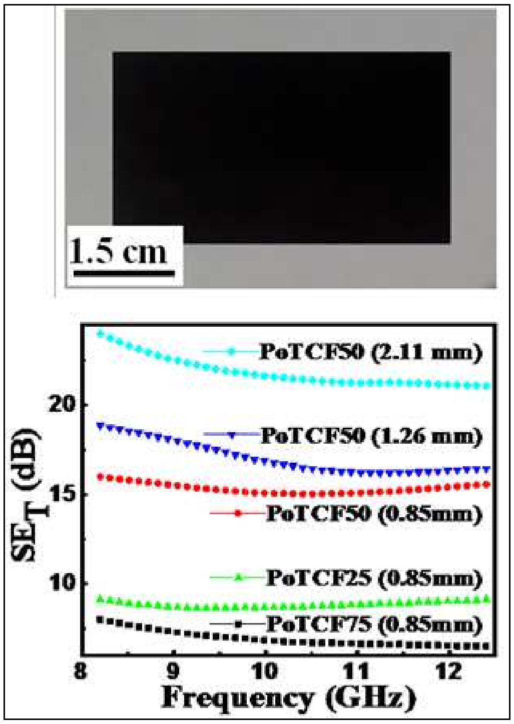
Graphical Abstract

Demonstration of lightweight poly (o-toluidine)-carbon fiber (PoTCF) composite thin sheets were fabricated using compression moulding technique for electromagnetic interference shielding. The presence carbon fiber improves the strength thermal, electrical & shielding properties of sheets. These thin sheets exhibit flexural strength of 36.0 MPa and a shielding effectiveness of 24 dB in X-band (8.2- 12.4GHz) at a critical thickness of ~2.11 mm, which is more than the limit required for techno-commercial applications.



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