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EMI shielding properties of polymer blends with inclusion of graphene nano platelets

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ABSTRACT

Polymer blends of poly vinyle chloride (PVC) and polyaniline (PANI) with the inclusion of graphene nano platelets (GNP) are fabricated to enhance the EMI shielding effectiveness. The initial assessment with cyclic voltammetry has shown improved electrical conductivity both for PVC/PANI blends and PVC/PANI/GNP composites. The capacitive effect of the blends and composites is evaluated at lower frequency region (100 Hz – 5 MHz). EMI shielding measurements of PVC/PANI/GNP composites is performed in the frequency range 10 MHz–20 GHz. In case of PVC/PANI (15 wt%) blends, a maximum attenuation of ~27 dB is attained which is enhanced to ~51 dB (max.) with the inclusion of 5 wt% GNP, mainly due to the absorption phenomena. The enhanced shielding effectiveness is achieved mainly in the frequency range 11–20 GHz. The dispersion state, fillers nature and their interaction may be the main aspects for the enhanced EMI shielding effectiveness of hybrid polymeric nanocomposites.

Introduction

With an ever-evolving fields of digital electronics and telecommunications; the electromagnetic pollution (interference) is a growing concern. To avoid this problem, certain materials possessing the characteristics required for shielding have been utilized [1–8]. These characteristics mainly consist of electric and magnetic dissipation factors to attenuate the electric and magnetic parts of EM waves. One of the main parameters for electromagnetic interference (EMI) shielding is the electrical conductivity; the material should possess a certain level of electrical conductivity to be able to attenuate the EM waves [9–12]. Polymers are insulators in general, with an emerging class of conducting polymers being an exception. Conducting polymers make a straight forward choice while selecting polymeric materials for EMI shielding application [13]. It is difficult to utilize conducting polymers as stand-alone structures for EMI shielding purpose. Therefore, blending conducting polymers with the more flexible and robust thermoplastics may be the way forward. Many researchers have used the concept of blends by mixing two different polymers for EMI shielding. Blending will obviously impart the individual characteristics to the outcome. A conducting network formation is one of the main requirement if not the only one for EMI shielding blends. F. Ma et al. have synthesized PVC-PANI (89–7%) blends with additional filler incorporation of reduced graphene oxide (4%) to form a conducting network with an electrical conductivity 7.2×10^{-2} S/cm [14]. Recently, Bandeira et al. have utilized PVC-PANI blends onto steel for improved corrosion resistance by the formation of a protective oxide layer at the interface facilitated by conductive nature of PANI [15]. PVC-PANI-graphene systems may also be utilized for an additional energy storage application like supercapacitors. H. Wang et al. synthesized free-standing, thin films of PVC-graphene composite with up to 20% of PANI content. Their electrical conductivities reached up to 18 S/cm with thickness ranging between 30 and 60 µm [16]. Highly conductive pallets of surfactant coated PANI and graphene oxide were prepared by Cheng et al. with conductivity reaching up to 10 S/cm and enhanced mechanical characteristics [17]. In another work, PVC-PANI flexible electrode is formed for supercaps application achieving an electrical conductivity of ~12 S/cm. All these studies revealed that such systems form a conducting network, providing an enormous potential to be employed for various applications as discussed. Based on

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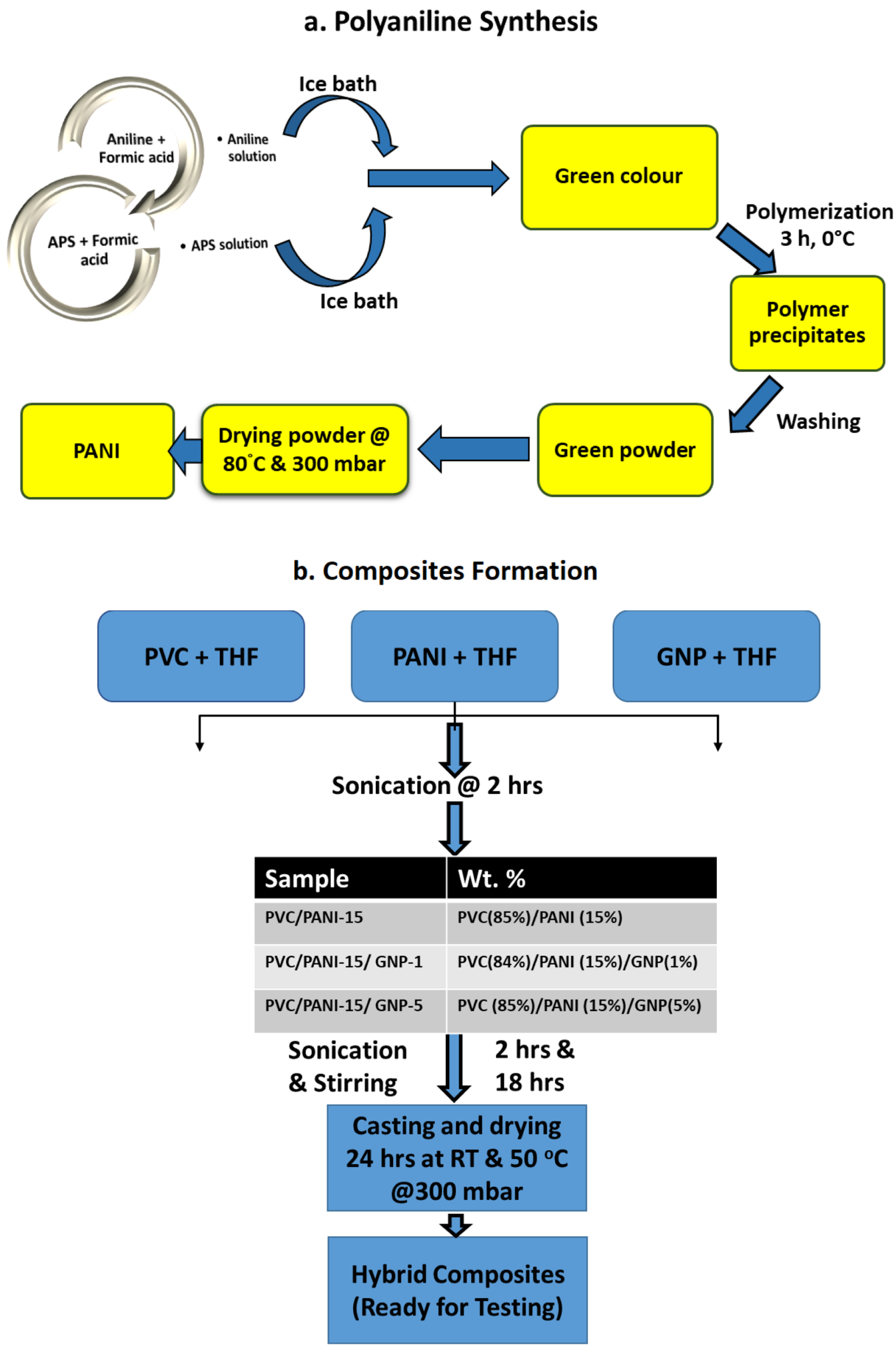

Fig. 1. Schematic diagram for (a) synthesis of PANI and (b) composites (PVC/PANI/GNP) formation.

these promising results, we have prepared a hybrid composite system of PVC matrix, PANI particles and graphene nano platelets (GNP) at various loadings. The main application is to evaluate these flexible composite films for EMI shielding applications. Dielectric characteristics in the frequency range 100 Hz–5 MHz provided a basis for EMI shielding predictions. The lower frequency range mainly deals with the magnetic attenuation while percolative systems such as PVC/PANI/GNP attenuate electric part of EM wave in GHz frequency range. Therefore, PVC/PANI/GNP composites are analysed experimentally for EMI shielding in 10 MHz–20 GHz range. Network analyser using coaxial cable method, S-parameters study revealed the attenuation more than 50 dB, thus showing the significance of hybrid composites. Cyclic voltammetry has further provided an insight to the electrical conducting network formation.

Materials and characterization methods

The materials used in current work are Aniline (Sigma-Aldrich), Ammonium persulfate-APS (DAU JUNG Korea), Formic Acid (Merck Schuchardt, Germany), Tetrahydrofuran-THF (Fisher chemicals UK),

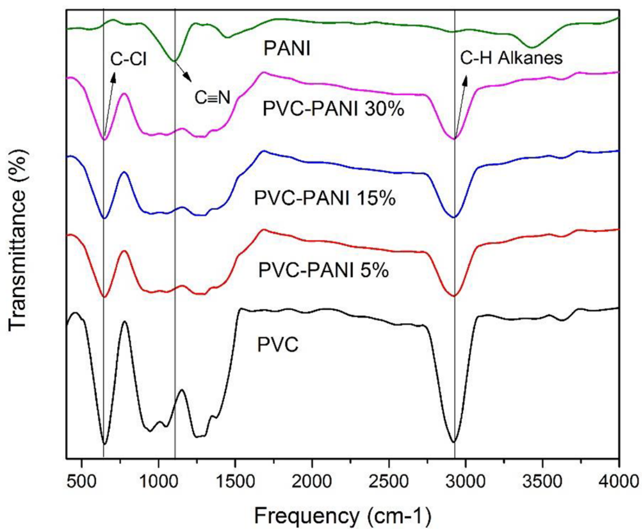

Fig. 2. FTIR spectrum of PVC, PANI and PVC/PANI blends.

Graphene and polyvinyl chloride (PVC). Polyaniline can be prepared with chemical oxidative polarization [18–21]. In this work two separate solutions of aniline (4 ml) and APS (9.76 g) have been prepared in formic acid (100 ml) at 50 °C [20] by vigorous stirring. Both these solutions are placed in an ice bath to lower their temperature (0 °C). Afterwards APS solution is added to the aniline solution in dropwise (30 min) manner. As the APS solution is added, the dark greenish colour starts to appear. The solution is then placed for 3 hrs at 0 °C, with continuous stirring, to polymerize. The polymer precipitates are formed as formic acid is evaporated at 80 °C and dark greenish polymer powder is leftover at the bottom of the beaker. After washing with ethanol and water, the polymer powder is dried in a vacuum oven at 80 °C and 300 mbar pressure. The solution processing method is used for the preparation of PVC/PANI/GNP composites. PVC, PANI and GNP are dissolved in THF separately and sonicated for 2 hrs. A range of composites, formed with PANI concentration fixed at 15% (PVC/PANI-15) while GNP concentration is varied from 0 to 5% and two concentrations 1 & 5% have been reported here (PVC/PAN-15/GNP-1 & PVC/PANI-15/GNP-5). The composite dispersions are sonicated for 2 hrs and stirred for 18 hrs. The samples were casted in glass petridish, dried first at room temperature (24 hrs) and then at 300 mbar pressure @50 °C in a vacuum oven (24 hrs). The films obtained are about 110–130 µm thick. The schematic diagram for the preparation of PANI, blends and hybrid composites is presented in Fig. 1. Polymers (PVC and PANI) and blend (PVC/PANI) are evaluated using Perkin Elmer FTIR spectrophotometer in frequency range 400–4000 cm^{-1} to attain FTIR spectrum shown in Fig. 2. The peak appearing at 650 cm^{-1} indicates C-Cl stretching, representing the PVC structure (black line). On the other hand PANI characteristic peaks (green line) are shown at 1100 (corresponds to −C−N bonds) and 3500 cm^{-1} (correspond to N−H stretch). The above mentioned peaks are present for the respective material in blends for all the three PANI concentrations. This configuration of peaks indicate that PVC and PANI are mixed well physically and both have different phases [22]. The composites formation is assessed with the well-established X-ray diffraction (XRD) technique using Cu Kα radiations in STOE theta-theta diffractometer. The characteristic peaks for PVC, PANI and GNP are clearly indicated individually shown in Fig. 3(a). The characteristic peak of amorphous PVC is shown around 22°. In the case of PANI, the two peaks appear around 17° and 24° respectively, confirming the semi crystalline nature. The characteristic graphitic peak for graphene of (0 0 2) plane is observed at ∼26°. Interestingly, PANI dominates the blend as it incorporates the semi crystalline nature to it. Graphene addition to the blend may strengthen the conducting network formation as the peak (0 0 2) plane appears at its designated place along with the PANI. The graphene nano platelets clearly dominate the XRD pattern in case of PVC/PANI-15/GNP-5 composite (Fig. 3(b)).

The morphology of composite films is evaluated using SEM (JEOL-instrument JSM-6490A). The samples are prepared in a liquid nitrogen environment and broken for edge assessment. The various micrographs show the presence of PANI particles and GNP in the composites (Fig. 4(a–d)). It is evident from the SEM images that a network has been formed with both PANI and GNP fillers inside, which may improve the electrical conductivity.

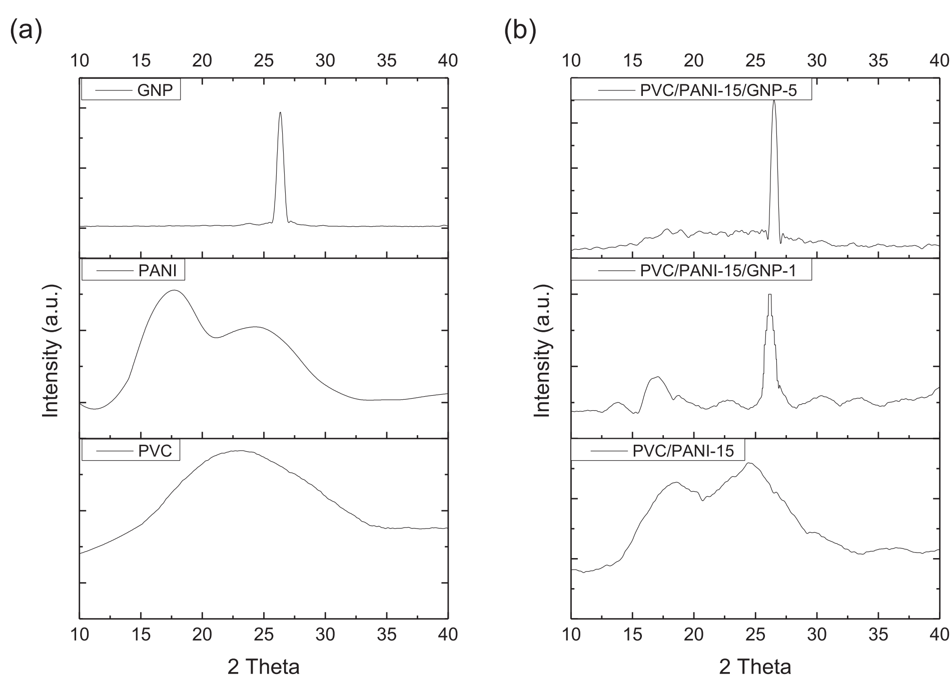

Fig. 3. XRD pattern of (a) PVC, PANI, GNP and (b) PVC/PANI-15 blend & PVC/PANI/GNP composites.

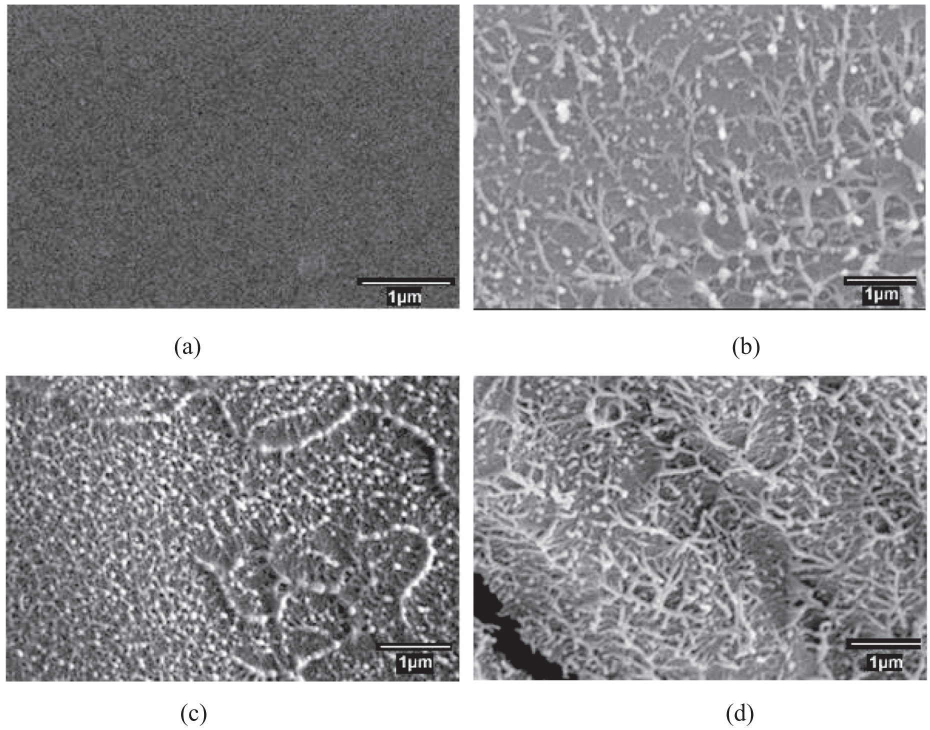

Fig. 4. Scanning electron micrographs of (a) PVC only (b) PVC/PANI-15 (c) PVC/PANI-15/GNP-1 (d) PVC/PANI-15/GNP-5.

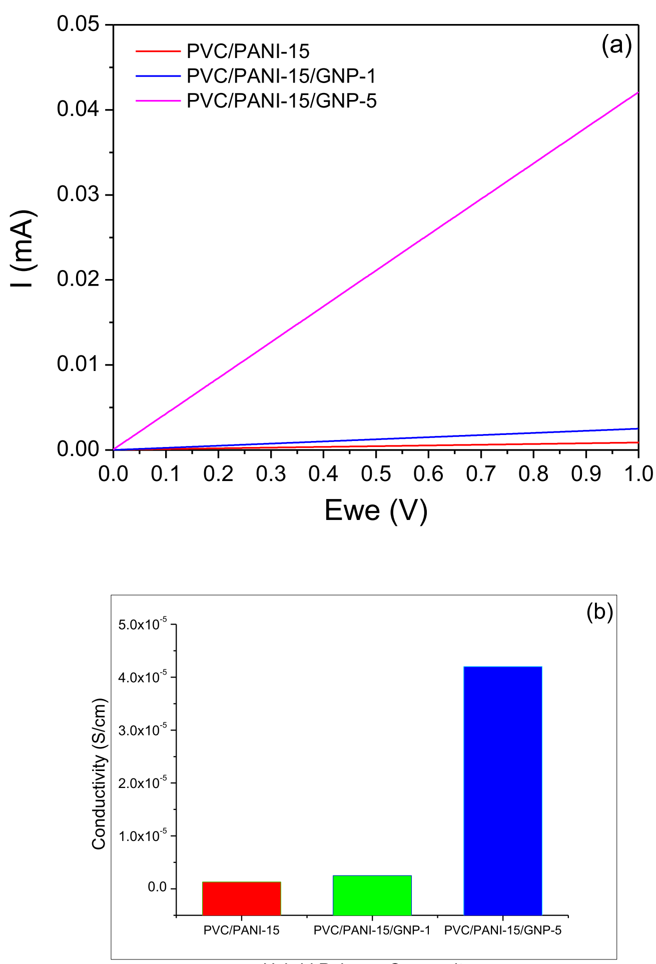

Fig. 5. (a) IV curves and (b) DC electrical conductivity of hybrid polymer composites.

Results and discussion

The electrical conducting network formation forms a basis for the EMI shielding effectiveness. Here in this work, initially the electrical conductivity has been evaluated with the I-V characteristics of the composites measured with BioLogic VSP electrochemical workstation. A three-electrode cell is utilized with a Saturated Calomel Electrode as the reference electrode, a platinum wire as the counter electrode and the composite film (1×1 cm^2) as the working electrode in 1 M solution of Sodium Sulfate (Na_2SO_4) electrolyte. The I-V plots, shown in Fig. 5(a), are the source for the electrical conductivity measurements of the composites using the basic equations such as $R = \frac{V}{I}$; $\rho = R\frac{A}{l}$ and $\sigma = \frac{1}{\rho}$. In the mentioned equations, ρ is the resistivity of the material, σ is the conductivity, R is the resistance measured from the I-V plot, A is the cross-sectional area and l is the length of the film. The electrical conductivity of the PVC/PANI-15 is low while with PVC/PANI-15/GNP-1, it reaches to 2.5×10^{-6} S/cm. For PVC/PANI-15/GNP-5, the electrical conductivity is maximum (4.2×10^{-5} S/cm) as predicted with the increase of graphene content [23]. The graphical presentation for electrical conductivity is shown in Fig. 5(b). Electrical conductivity response as a function of frequency plays a vital role in the case of EMI shielding materials. For this purpose, dielectric characterization of the polymer composites is also carried out at room temperature using impedance analyser (Wayne Kerr 6500B) in the frequency range 100 Hz – 5 MHz. AC conductivity is measured for all the samples and the trend is shown in Fig. 6(a). AC conductivity increases both as a function of filler loading as well as frequency. Other important parameters, in case of polymer composites are the dielectric constant and dielectric loss. The dissipation factor 'D' and capacitance 'C' are obtained for hybrid composites (12 mm diameter) as well. The dielectric constant of the polymer composites is increased from 2 (PVC) to 7 (PVC and PVC-PANI15-GNP5) at 100 Hz. Both PVC/PANI-15 and PVC/PANI-15/GNP-1 also show an enhancement in dielectric constant as compared to polymer only as shown in Fig. 6(b). The dielectric constant along with dielectric loss (Fig. 6(c)) are indicators for dissipation of electromagnetic energy inside the polymer composite. Various dielectric characteristics are presented in Table 1, measured at 5 MHz frequency.

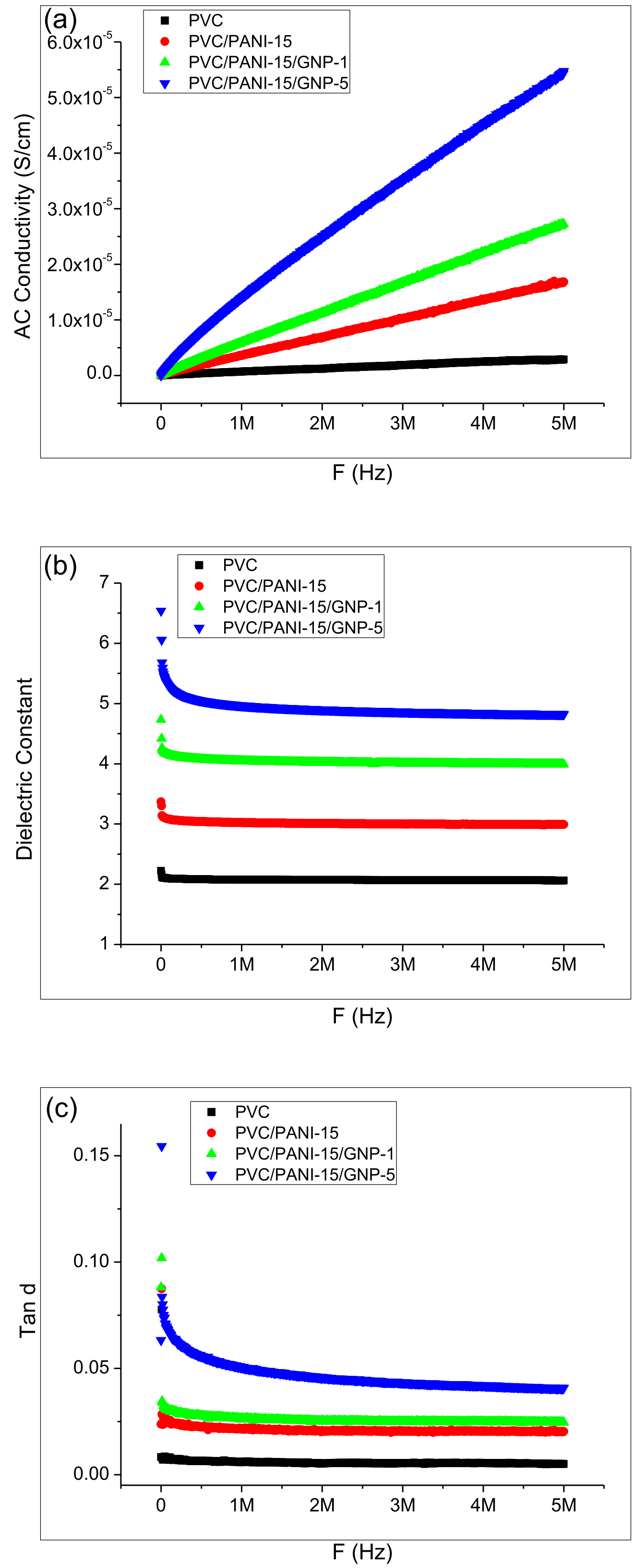

Fig. 6. (a) AC conductivity (b) dielectric constant and (c) Tan d of PVC, PVC-PANI blend and hybrid polymer composites up to 5 MHz frequency.

The generalized response to AC conductivity can be determined by the mathematical expression given below [24]:

$$\sigma_{AC} = \sigma_{DC} + A\omega^{t} \tag{1}$$

In the above equation σ_{AC} and σ_{DC} denote AC and DC conductivity, respectively while A is a temperature-dependent constant and ω is the angular frequency (2πf). The exponential factor 't' ranges from 0.7 to 0.9 at lower temperatures and vanishes at high temperatures. It is evident that AC conductivity directly depends on the frequency-induced dipole polarization. At low frequency region, σ_{AC} is relatively low and enhances at higher frequencies. The induced dipoles orient well along field direction at low frequencies while less relaxation time at high frequency region hinders the dipoles to reorient. The hoping mechanism due to the charges excitement with frequency is responsible for the enhancement in the effective conductivity. With the addition of conductive fillers and increase in concentration, further enhancement in the electrical conductivity is experienced as shown in Fig. 6(a) as a function of frequency (100 Hz–5 MHz). At first place, PANI inclusion (15%) paves a way to form an electrically conducting network ($\sigma_{AC} \sim 1.7 \times 10^{-5}$ S/cm at 5 MHz). A further enhancement ($\sim \times 2$) is observed at GNP 1% loading to the PVC-PANI blend ($\sigma_{AC} \sim 2.7 \times 10^{-5}$ S/cm at 5 MHz). A conducting network formation is evident with the increase in graphene content. The AC conductivity reaches up to 6×10^{-5} S/cm at 5 MHz for PVC/PANI-15/GNP-5 composite. An electrically conducting network formation effect on the EMI shielding effectiveness (SE) can be predicted by using the following equation [25–26]:

$$SE = 50 + 10\log\left(\frac{\sigma}{f}\right) + 1.7t\sqrt{\sigma f} \tag{2}$$

Here in this relationship, 't' is the thickness in cm, 'σ' is conductivity in S/cm and 'f' is the frequency in MHz. Eq. (2) clearly predicts the dependence of SE on the material's thickness and conductivity. The electrical conductivity enhancement trends up to 5 MHz are promising; as for all the samples it is increasing directly with frequency. It can be predicted that at higher frequencies (beyond 5 MHz), specifically in GHz frequency range these polymer composites may be useful for EMI shielding purpose as per Eq. (2). Another important aspect is that for the frequency range under discussion (100 Hz–5 MHz) magnetic dissipation is the prime part to achieve the EMI shielding effectiveness. Current work is related to the graphene and polyaniline fillers, both useful in electrical dissipation of the EM wave. Thus, the electrical conductivity trend (up to 5 MHz) is useful to predict that the composites may be applied as EMI shielding material. Based on the above discussion, the polymer blends and hybrid composites are tested for EMI shielding effectiveness via network analyser in the frequency range 10 MHz–20 GHz. The outer diameter of the tested samples is 7 mm while the inner hollow round is 3 mm in diameter. The EMI shielding theory is based on three main aspects when incident on a certain material; reflection (R), absorption (A) and transmission (T). It is represented mathematically in the following expression:

$$R + A + T = 1 \tag{3}$$

The shielding effectiveness, measured in decibels (dB), is expressed as the logarithmic ratio of incoming to transmitted energy as expressed below:

$$Shielding\ \ Effectiveness,\ SE\,(dB) = 10\log\left(\frac{E_I}{E_T}\right) \tag{4}$$

A network analyser is used to test the hybrid composites for EMI shielding effectiveness by calculating the complex scattering parameters known as S-parameters. The S-parameters of a network analyser are represented as S_{11} (or S_{22}) and S_{12} (or S_{21}) used for reflected and transmitted electromagnetic waves respectively. These parameters are used for calculations of reflection and absorption coefficients as below [27]:

$$R = |S_{11}|^2 = |S_{22}|^2 \tag{5}$$

$$T = |S_{12}|^2 = |S_{21}|^2 \tag{6}$$

Table 1
Various dielectric characteristics of PVC, PVC/PANI blends and PVC/PANI/GNP composites at 5 MHz.

Sample representation	Dielectric constant	Tan δ	AC conductivity (S/cm)
PVC	2.06	0.009	2.8×10^{-6}
PVC/PANI-15	2.98	0.014	1.6×10^{-5}
PVC/PANI-15/GNP-1	3.98	0.025	2.85×10^{-5}
PVC/PANI-15/GNP-5	4.82	0.040	5.6×10^{-5}

The total shielding effectiveness is the combined effect of reflection, absorption and multiple internal reflections inside shielding materials [28]. The internal reflections may also be merged into absorption due to its characteristics. The schematic diagram for the shielding material involving various mechanisms is shown in Fig. 7. We can write an expression for total shielding effectiveness as

$$SE_T = SE_R + SE_A \tag{7}$$

Both reflection and absorption shielding effectiveness can be expressed as:

$$SE_R = 10\log\left(\frac{1}{1-|S_{11}|^2}\right) \tag{8}$$

$$SE_A = 10\log\left(\frac{1-|S_{11}|^2}{|S_{21}|^2}\right) \tag{9}$$

The above-mentioned equations (8) and (9) have been used to calculate total shielding effectiveness (Eq. (7)) of hybrid polymer composites as shown in Fig. 8(a–c). S_{11} and S_{21} parameters are measured using network analyser and put in above mentioned equation for SE_R and SE_A calculations. As the polymer matrix PVC is insulating, both reflection as well as absorption SE is around 0 dB which is the case for most of the polymer matrices. With the inclusion of PANI (15 wt%) particles, the attenuation due to reflection phenomena is minimal and the blend shows clearly the absorption pattern (Fig. 8(a–b)). The maximum SE_R for PVC/PANI-15 blend is around 4 dB (around 6–8 GHz and 15 GHz) while the maximum SE_A reaches to slightly above 20 dB. The absorption part is constantly increased up to 12 dB in the frequency range 1–12 GHz and remains constant 13–17 GHz. Beyond this point, absorption is increased again and reaches up to 22 dB. Thus, the total shielding effectiveness for polymer blend is enhanced over-all as compared to polymer only and reaches to 11–15 dB in the frequency range 11–17 GHz. The maximum total SE for polymer blend reaches up to 27 dB at 20 GHz. Incorporating GNP (1%) makes a slight enhancement in the already described trend of PVC/PANI-15 blend. The reflection part of shielding effectiveness is improved as compared to polymer blend may be due to the better electrically conducting network formation. The maximum SE_R observed for PVC/PANI-15/GNP-1 is around 6 dB at 15 GHz. The absorption part is enhanced as well and reaches up to 25 dB as shown in Fig. 8(b). The total shielding effectiveness for PVC/PAN-15/GNP-1, based on reflection and absorption phenomena, is to some extent enhanced in comparison with polymer blend and the maximum attenuation reaches up to ~30 dB at 20 GHz. With further increase in GNP loading up to 5%, the maximum total SE achieved is 51 dB (14–16 GHz) for PVC/PANI-15/GNP-5. The important thing to note is that the attenuation beyond 2 GHz frequency range is above 20 dB throughout and increases constantly. Normally for percolative polymer composite systems, 20 dB is a benchmark. The reported percolative systems with ≥20 dB usually have the shield material thickness in the 1–2-mm range [10]. In our case the hybrid composite thickness is ~0.11–0.13 mm, which if increased may enhance shielding effectiveness further. Likewise, the absorption phenomenon is dominant as compared to reflection and skin depth plays an important role in achieving higher absorbing attenuation. This aspect of thickness and skin depth has been explained well by N.C. Das et al. [29–35]. Here in our case, the SE_A for PVC/PANI-15/GNP-5 reaches up to 38 dB (max.)

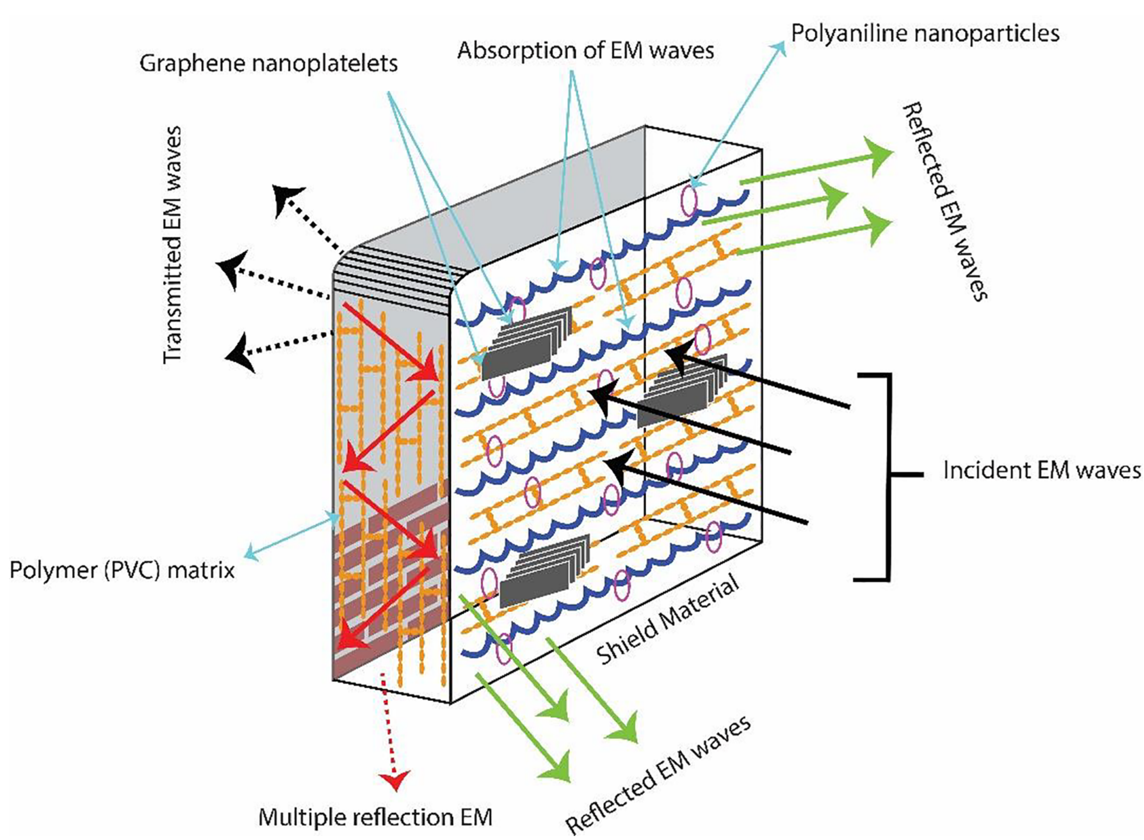

Fig. 7. Schemtic diagram for EM waves attenuation mechanism through a shielding material.

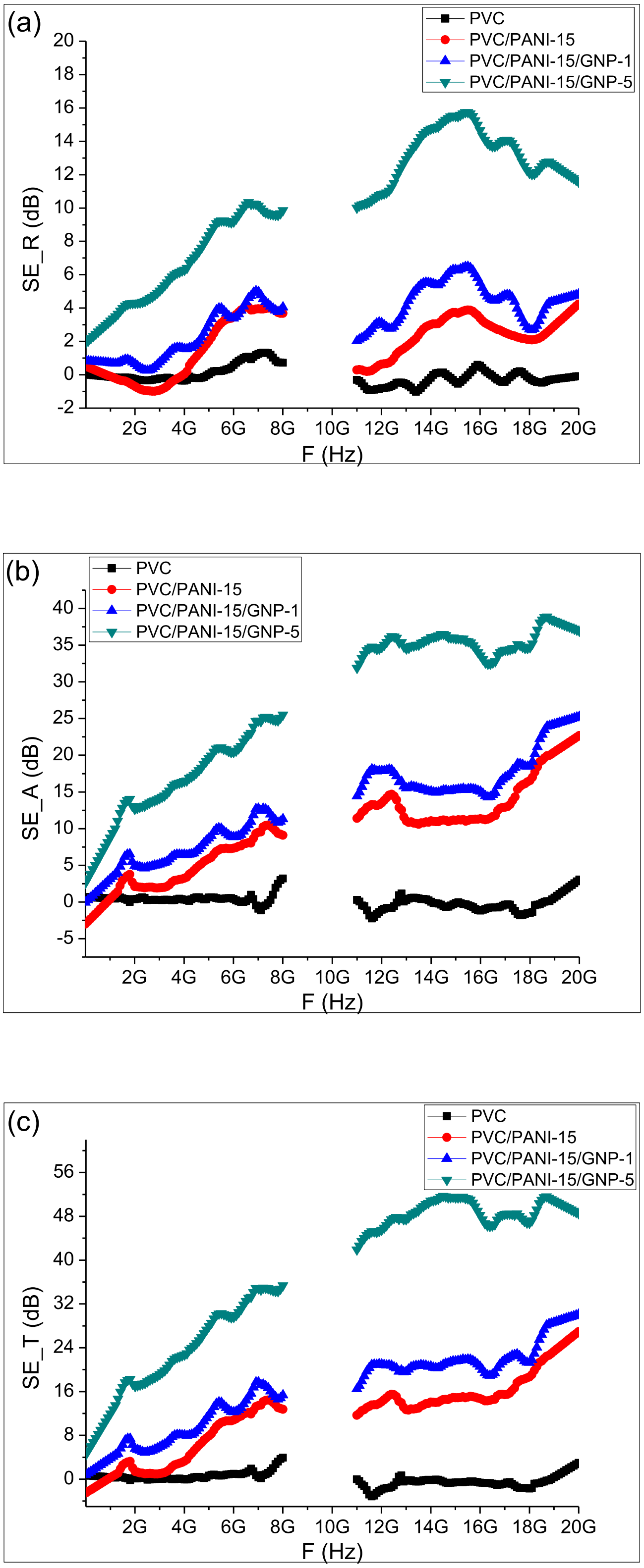

Fig. 8. EMI Shielding Effectiveness due to (a) reflection (b) Absorption and (c) total effect of hybrid polymer composites in a frequency 10 MHz–20 GHz.

at 18 GHz due to the charge carriers hoping mechanism. The effective absorption, which can be calculated as per Eq. (10), is indicative of the dominant absorption phenomena.

$$A_{eff} = \frac{1 - R - T}{1 - R} \tag{10}$$

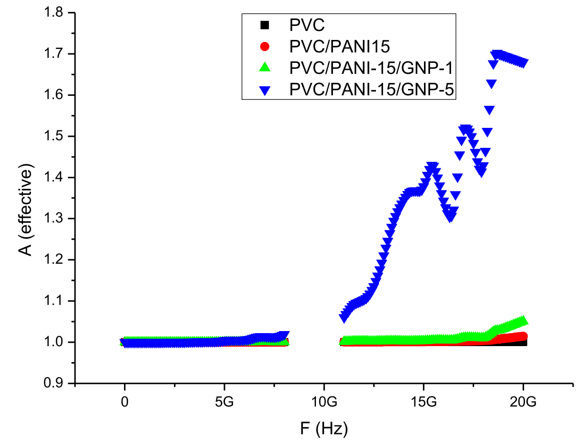

Fig. 9. Effective absorption for PVC, PVC/PANI blend and hybrid composites in the frequency range 10 MHz–20 GHz.

Based on Eq. (10), the effective absorption is calculated and represented in Fig. 9. Also, the increased graphene content provides free charge carriers, enhancing the reflection shielding effectiveness as SE_R is around 15 dB at 15 GHz shown in Fig. 8(a). Another interesting aspect is that generally attenuation is achieved at a certain frequency; however here in this case, the attenuation is expanded over a whole frequency range making these composites suitable for broadband applicability, especially in GHz frequencies. The functionalization of both PANI and GNP can make these hybrid polymer composites more useful in terms of EMI shielding applications.

Conclusion

Hybrid polymer composites based on the inclusion of conducting fillers, graphene nano platelets and polyaniline nano particles, are evaluated for EMI shielding. The dielectric characteristics, measured at 100 Hz–5 MHz frequency, provided a benchmark for the EMI shielding measurements. The dielectric constant increased up to 7 for PVC/PANI-15/GNP-5 as compared to 2 for polymer only. AC conductivity is also increased up to 6×10^{-5} S/cm for the maximum filler loadings in 100 Hz–5 MHz frequency range. The same composite set is experimentally evaluated for EMI shielding effectiveness (10 MHz to 20 GHz frequency regime), rendering maximum attenuation of ~51 dB in 18–20 GHz frequency range. These hybrid polymer composites may be a future prospect for applications in the broadband frequency spectrum.

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