Study of mechanical, electrical and EMI shielding properties of polymer-based nanocomposites incorporating polyaniline coated graphene nanoparticles

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Abstract

The Electromagnetic Interference (EMI) shielding characteristics of PVC based nanocomposites were studied when reinforced with newly developed Thermally Reduced Graphene Oxide (TRGO) coated with Polyaniline (PANI) nanoparticles. Various techniques were utilized to characterize prepared nanocomposite films like Scanning Electron Microscopy (SEM), direct current (DC) conductivity measurement, impedance analyses, and EMI shielding. EMI shielding was measured in three different regions of the electromagnetic spectrum like the Microwave region (0.1 GHz to 20 GHz), Near InfraRed (NIR), and Ultraviolet region (UV). The coating of PANI over TRGO provides compatibility of nanoparticles with a polymer matrix that leads to enhanced dispersion. EMI shielding was found to be 104 dB for 40 wt% filler content, because of the formation of a mature and dense interconnected network structure of filler. UV and NIR transmittance was also studied and less than 0.5% transmission in the whole UV (200 nm 400 nm) and NIR (700 nm 2500 nm) region was observed.

1. Introduction

Electromagnetic pollution is considered major pollution that causes Electromagnetic (EM) waves to interact with electronic equipment and causes instability in their functioning [1–3]. To counteract this, EMI shielding is a need of the hour and is found to be a profound area in research [4]. EMI shielding is the shielding of the accessory from EM waves by deploying a hurdle between the EM environment and the accessory [5]. EMI shielding requires an important property of electrical conductivity and that makes metals by choice a priority but is not used nowadays because of some of its demerits such as high cost, difficult processing and high weight, etc. Polymers, in comparison to metals, are a good choice because of their low weight flexibility, ease of processing, and low cost [6]. Intrinsically conducting polymers (ICPs) such as Polyaniline, Polypyrrole, Polythiophene are a class of polymers that because of their conductivity are used in many electrical applications and are a direct choice for this application [7]. Polyaniline, an ICP that is known for its environmental stability, high conductivity, and low density [8, 9] is considered for many applications such as solar batteries, flexible solar cells, actuators, storage devices, conducting fibers, supercapacitors [10], and EMI shielding [11], etc. Graphene an allotrope of carbon having layers of sp2 hybridized carbon atoms is well known because of its high electrical conductivity, high strength, and chemical inertness [12–14] is being used in many applications such as biomedical [15], drug delivery [16], energy storage [13] and construction applications to increase the strength of cement [17] and polymeric solar cells [18], etc. Different approaches for EMI shielding by using different materials such as CNTs [6, 19], ferrites [11, 20, 21], graphene [22], etc. have been used till now.

Fayzan et al. prepared PANI/NiFe/PS and PVC/PANI/GNP composite and obtained shielding effectiveness of 35 dB and 51 dB at 0.1–20 GHz and 11–20 GHz with the thickness of 0.25 mm and 0.1 mm respectively.
PANI/TRGO/PVC composite was made and obtained shielding was 56 dB in the frequency range of 11–20 GHz with a thickness of 0.10–0.13 mm. PS/PANI blend was prepared and shielding effectiveness up to 45 dB was obtained at a thickness of 0.25 mm [7, 20–26]. Nina et al developed the fabric of polyaniline nanofibers with a thickness of 0.1 mm and their composite with graphite and got 11–15 dB shielding effectiveness in the frequency range of 8.2–18 GHz. Nanofibers of polyaniline were also prepared and showed shielding effectiveness up to 71–77 dB in the frequency range of 8.2–18 GHz [9, 27]. Seema et al prepared polyaniline-carbon fiber composite
and shielding of 31.9 dB was observed at 12.4 GHz with a thickness of 1.5 mm [28]. PVA/graphene composite was prepared by Sandeep et al and shielding effectiveness of 19.5 dB was observed in the frequency range of 8.2–12 GHz (X-band) with graphene content up to 0.5% [29]. Rajini et al synthesized films of PANI and graphene and their work showed 42 dB sand 32 dB shielding effectiveness in 4–8 GHz and 8–12 GHz respectively [5]. A composite of NBR and graphene was prepared by Vivek et al and it showed shielding up to 57 dB at 7.5–12 GHz with a thickness of 3 mm [30]. Jun et al prepared Fe3O4/CNTs/HPCF composite
and observed shielding effectiveness up to 50.9 dB in the frequency range of 2–18 GHz with a thickness of 1.5–3 mm [31].

In the current work, graphene particles coated with polyaniline nanoparticles were prepared. Graphene provides excellent EMI shielding properties, but it doesn’t disperse well in polymer matrix creating stress concentrating points that are undesirable. In comparison to it, PANI disperses much better in polymer matrix also has poor EMI shielding properties as compared to graphene. To avoid the dispersion problem of graphene, it is coated by PANI during the in situ polymerization of PANI. This would cause graphene to disperse more...
evenly in the polymer matrix. PVC is chosen as a host matrix in this work because it is cheap, chemically stable, both rigid and flexible [32].

2. Materials and method

Graphite Powder (45 Micron), Potassium permanganate (K\text{MnO}_4) (99%), Sulfuric acid (H\text{SO}_4) (96%), Formic acid (H\text{PO}_4)(98%), Aniline(99.9%), Tetrahydrofuran (THF)(99%) and Ammonium Persulfate (APS)(99%) were purchased from Sigma-Aldrich. Polyvinyl chloride (PVC) (99%) was donated by engro group of industries Pakistan., Distilled Water (DI), Hydrogen Peroxide(H\text{O}_2)(30%) were purchased by the local vendor.

2.1. Fabrication of thermally reduced graphene oxide (TRGO) nanoparticles

A mixture of graphite powder (3 g), K\text{MnO}_4 (18 g), H\text{SO}_4(360 ml), and H\text{PO}_4(40 ml) is made and was left for stirring for 24 h at 50 °C. 30% Solution of H\text{O}_2(20 ml) and ice of DI water (400 ml) were added to the obtained
Graphite oxide (GO) solution to stop the reaction and reduce unreacted potassium permanganate. GO particles were then separated by centrifuge and washed with DI water 3–4 times to remove acid content. GO particles were then reduced thermally for 72 h in a muffle furnace at 200 °C. Nanolevel thermally reduced graphene oxide (TRGO) particles were obtained. A schematic flowchart of the process is shown in figure 1.

2.2. Fabrication of TRGO nanoparticles coated with Polyaniline (PANI) (TRGO-PANI)
Homogeneous solutions of TRGO/aniline/formic acid and APS/formic acid were prepared separately in an ice bath keeping their temperature below 5 °C. APS/Formic acid solution was then poured drop by drop in TRGO/aniline/formic acid to start the polymerization reaction of aniline. The solution was left for stirring for 3 h keeping the temperature of 0 °C until the solution’s color turned black to dark green. TRGO-PANI nanoparticles separated by centrifuge and dried at 90 °C for 24 h. A schematic flow chart for the preparation of TRGO-PANI nanoparticles is shown in figure 2.
2.3. Fabrication of PVC/TRGO-PANI nanocomposite

PVC and TRGO-PANI nanoparticles are mixed in THF in different concentrations (shown in table 1) and stir for 1 h. Sonicated in a bath sonicator for 2 h to remove any agglomeration of particles. Then the solution was left for stirring for 5 h. Poured in a petri dish and dried at room temperature for 24 h. A schematic of the preparation of PVC/TRGO-PANI nanocomposite films is shown in figure 3. A uniform thickness of 0.25–0.27 mm was obtained and measured by SEM (figure 4).

3. Scanning electron microscopy (SEM)

Scanning Electron Microscopy (TESCAN MIRA3) is used for the morphological characterization of TRGO, TRGO-PANI nanoparticles, and PVC/TRGO-PANI nanocomposites films. SEM images of TRGO at different magnifications are shown in figure 5. It is evident that TRGO is successfully prepared with a sheet thickness of 11–14 nm. Similar results of SEM were also found in the literature [33]. After coating PANI on the surface of TRGO, the thickness of sheets increased to 24 nm measured by SEM as shown in figure 6. PANI is uniformly coated on the TRGO surface.

SEM images of pure PVC, PVC/TRGO-PANI 15 wt%, and 40 wt% nanocomposite films are shown in figure 7. All three films were frozen in liquid nitrogen and then broken to expose a fresh cross-section of the film. Liquid nitrogen is used to avoid chain elongation and disturbing the structure of filler during the breaking of the film. The absence of any prominent feature in the PVC image indicates the amorphous nature. As 15 wt% nanoparticles added, a well dispersed and homogenous interconnected network structure is formed inside the amorphous PVC matrix. This structure becomes denser and more mature by increasing the amount of filler to 40 wt%. similar results of SEM were also found in the literature [7, 22, 23].

4. DC conductivity

Nanocomposites films were cut in rectangular strips of 10 mm * 30 mm dimensions. 2 prob method was used for the measurement of DC conductivity. Probes were placed at a 20 mm distance and a voltage of 10 V is applied across the probes. The current is measured by a highly sensitive ammeter. Using the following Equations (1)–(3), DC conductivity was calculated, and the results are shown in figure 8.

\[ R = \frac{V}{I} \quad (1) \]

\[ \rho = \frac{RA}{L} \quad (2) \]

\[ \sigma = \frac{1}{\rho} \quad (3) \]

PVC is insulative in nature and also proved by its DC conductivity value of 3.5 * 10^{-14} S cm^{-1}. A similar result for the conductivity of PVC was also reported [34]. By adding even 5 wt% of a filler, conductivity is increased by 5 orders of exponential because of the high electrical conductivity of TGO-PANI nanoparticles and the formation of interconnected network structures that provide a clear and easy path for electrons to move.
from one prob to another. DC conductivity keeps on increasing with the concentration of nanoparticles. A maximum of 0.027 S cm$^{-1}$ or 2.7 S m$^{-1}$ was measured with the filler amount of 40 wt% due to the dense and mature structure also described by scanning electron microscopy.

5. Dielectric properties

An impedance analyzer (Wayne Kerr 6500 B) was used to measure the capacitance and dissipation factor of all the prepared nanocomposite film between 20 Hz to 5 MHz to have an estimation of EMI shielding in the

![Figure 11](image-url)
Following equations (4) and (5) were used to calculate the dielectric constant ($\varepsilon'$) and dielectric loss ($\varepsilon''$) and shown in figures 9 and 10.

\[
\varepsilon' = \frac{Cd}{A\varepsilon_0}
\] (4)

\[
\varepsilon'' = \varepsilon' \times \tan \delta
\] (5)

$\varepsilon'$ and $\varepsilon''$ of pure PVC is about 1.5 and 0.001 respectively in the whole region due to its insulative nature and absence of electric dipoles. With the addition of extremely electrically conductive nanoparticles and the formation of multiple insulative/conductive interfaces, the dielectric constant increased rapidly with concentration. As the concentration of nanoparticles increases so as the number of interfaces resulting from the presence of electric dipoles. Dipoles orient themselves along with the external oscillating electric field consuming its energy in orienting in one direction in the first half cycle and then reorienting in the opposite direction in the second half cycle. Thus, the dielectric loss of nanocomposites is also very high in the low-frequency region. As the frequency increases, electric dipoles don’t get enough time to orient and then reorient in opposite direction with fast-changing electric and magnetic fields of electromagnetic waves, so values drop to a value independent of frequency and remain constant with respect to frequency but increase with the concentration of nanoparticles.
6. EMI shielding

EMI shielding can be done by the following three mechanisms: absorption (SEA), multiple reflections (SEMR), and reflection (SER). SEMR is generally ignored if total shielding effectiveness is more than 10 dB and so it doesn’t need to calculate. Total shielding effectiveness is calculated by the remaining two mechanisms and is given by [35]:

\[ SE_T = SE_A + SE_R = 10 \log \frac{E_{IN}}{E_{OUT}} \]  

Vector Network Analyzer (VNA) was used to measure Scattering parameters (S-Parameters) and by using the following equation SEA, SER and SET were calculated. A donut shape like sample was cut according to the required dimensions of equipment and then tested by VNA.

\[ SE_A = 10 \log \left(1 - \frac{S_{11}^2}{S_{22}^2}\right) \]  

\[ SE_R (dB) = 10 \log \left(1/(1 - S_{11})\right) \]  

\[ SE_T = SE_A + SE_R \]

Figure 11 tells the trend of SEA in relation to frequency for different concentrations of TRGO-PANI in the host matrix i.e. PVC. It is evident that the SEA is far less than SER as both electrical conductivity and magnetic permittivity is required for absorption. Given that TRGO-PANI nanoparticles are only electrically conductive so it shows SEA values that are very less than SER.

The shielding due to reflection is illustrated in figure 11(b) and total shielding effectiveness SET in figure 11(c). It clearly depicts an increase in shielding when the concentration of TRGO-PANI in PVC is increased by up to 40%. SER increases as the amount of filler increased ranging from 1.60 dB for pure PVC to 72 for 40% concentration of TRGO/PANI. 104 dB shielding effectiveness at 40% concentration and trend of the continuous increase in SET with the concentration of TRGO-PANI nanoparticles leads to the conclusion of the formation of the mature and dense interconnected network structure at higher concentration [12, 36] also indicated by SEM, DC conductivity and dielectric properties. The electric part of the Electromagnetic wave is dissipated by the electric dipoles present due to orientation and reorientation according to the oscillating electric...
field. As Dielectric loss is very high, so huge amounts of energy of EM waves were utilized to oscillate electric dipole. As the concentration of electric dipoles increased by adding more amount of filler, eventually more amount of energy dissipated inside the shielding nanocomposite film.

Perkin Elmer Lambda 950 equipment was utilized for the measurement of UV and NIR transmittance through nanocomposite films. Figure 12 shows the IR transmission of the composite and it can be seen clearly that as the concentration of TRGO-PANI is increased, it leads to very low transmittance that ranges from 83.53% for pure PVC to 0.4% for TRGO/PANI that is almost negligible as compared to pure PVC. This graph depicts the extremely low transmission of NIR waves by the filler particle because of the strong interaction of TRGO-PANI filler with the host.

Figure 13 demonstrates the UV transmittance through nanocomposite in direct relation with filler content. As the filler content increases, UV transmittance through the composite reduces with a drastic result than Pure PVC. This achievement again in debt to the interconnecting structure of the filler/polymer interface and filler itself.
7. Mechanical properties

Mechanical properties were analyzed by Zwick/Roell UTM Z100 using ASTM D3039 standard. Rectangular strips were cut from thin film with dimensions of 20 * 10 * 0.25 mm where 10 mm is width, 20 mm is gauge length and 0.25 mm is film thickness. 2 mm min^{-1} (10%/min) strain rates were set for the measurement. 3 tests were carried out for each sample and the best results are shown here. It was observed that PVC has a tensile strength of almost 6.2 MPa. By adding the both very low and high concentrations of nanoparticles (5 wt% and 40 wt%), both strength and elongation decreased to almost the same values because of various reasons like poor dispersibility, agglomeration, stress concentration points, very high chain restriction, etc. It appears that 15 wt% samples exhibit maximum enhancement in strength and elongation reaching at 9.8 MPa and 190% respectively. This indicated the maximum dispersion, chain restriction, and very few agglomerations and stress concentration points. SS curves, Strength, modulus, and elongation of all samples are shown in figure 14. 3 fractured specimens 1, 3, and of the tensile test was analyzed by SEM to observed the fracture behavior. SEM images are shown in figure 15. It is obvious from figure 15 a that pure PVC has no feature and fracture in a brittle manner as no prominent features appear in SEM because of its amorphous behavior. Moreover, it was also observed that PVC/TRGO-PANI-15 wt% specimen fractured in a ductile manner as no sharp facet like features appear in SEM micrograph while at 40 wt% does have lots of sharp facet like features. Confirming that nanoparticles are well dispersed and there is a strong interaction with the matrix that fracture happens in matrix phase and not on the interface. While at 40 wt%, it was observed that fracture happens at the interface because of
agglomeration that creates microcracks which eventually acted as stress concentration points and a cleavage plane for the crack to propagate. It can be deduced from SEM and the fracture analyses that at low concentrations, nanoparticles are well dispersed and have strong interaction with matrix and thus all the applied load transmitted throughout the specimen and have uniform elongation and low modulus. While at high concentration, due to agglomeration, all the load acts on the stress concentration points and propagating the cracks and breaking the specimen, and in turn modulus increases.

8. Particle size analysis

The particle size of PANi particles was determined by ‘ZetaSizer AT’ using a 0.5 wt% solution of TRGO and TRGO-PANI particles in water. The obtained particle size distributions are shown in figure 16. The average particle size of TRGO is found out to be 15 nm and that of TRGO-PANI nanoparticles to be 26 nm.

Zetasizer AT is an instrument of ‘Malvern Pananalytics’ that uses light scattering to measure the particle size from 1 nm to 1000 nm range with the accuracy of $+/−2\%$.

9. Conclusion

The EMI shielding of PVC composite is studied with the incorporation of PANi coated TRGO nanoparticles in the host PVC matrix. EMI characteristics of PVC were found to be excellent when fabricated with 40% TRGO-PANI filler. Shielding because of absorption was much low and the total shielding occurs due to the reflection mechanism or in other words to $\text{SE}_R$. 104 dB $\text{SE}_T$ was observed in the microwave region (0.1 GHz to 20 GHz). NIR and UV transmittance were also studied and found out to be less than 0.5% at a filler content of 40%. Results were found to be very inspiring and this research can lead to the new route to flexible EMI shielding materials. 15 wt% samples exhibit maximum enhancement in both tensile strength and elongation.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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