Comparative study of polymer-based nanocomposites microwave absorption performance in X-band

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Abstract
The present work focused on electromagnetic (EM) absorption performance comparison of different polymer nanocomposite structures. Synthesis and fabrication of polymer nanocomposite composed of graphene, polyurethane (PU), epoxy was done using in situ polymerization and resin transfer moulding respectively. The effect of polymer on EM properties of composite was carried out using waveguide measurement technique in X-band. Reflection Loss (RL) and shielding effectiveness (SE) were measured for various nanocomposites using EM wave theory. From the results it is observed that 2.5 wt% of PU/epoxy graphene nanocomposite has shown RL value $-35$ dB at 12.1 GHz and SE value of $-45$ dB.

1. Introduction
The rapid development of electronic gadgets, commercial, biological, military and defence systems creates electromagnetic (EM) noise known as EM pollution. EM pollution causes human health diseases like sleeping disorders, nervousness and headache [1]. This pollution worsens the functioning of electronic equipment and their durability. So there is a necessity to mitigate EM pollution by using EM shielding materials. There are two functional methods proposed in EM shielding, one of which is absorption/reflection of radiation by certain polymeric/metalllic shielding materials placed in the vicinity of radiating components. These shields also act as radiation barriers to other electronic components in the devices. In the second method, the shielding materials are made more effective by incorporating dielectric particles into them which allows for reduction of thickness of the shielding material [1–4]. In current study the second method has been chosen knowingly absorption dominated microwave shielding.

Carbon based electrically conducting materials have advantages like lightweight, corrosion resistant, good processibility over other conventional shielding materials in addition to adjustable conductivity [1–23]. Conducting carbon based materials like carbon black (CB) [1, 13, 23], carbon nanotube (CNT) [1, 5–8, 13, 16, 20, 23], carbon nano fiber (CNF) [1, 8, 23] and graphene [1–4, 9, 11–15, 17–23] were exhibited EM shielding due to their remarkable electromagnetic properties. Among all, graphene posses electrical conductivity value of 6000 Scm$^{-1}$, high aspect ratio and good microwave attenuation. Moreover graphene shows diminishing of electrical conductivity due to the existence of poor interfacial interactions within it and conversely increase of interfacial interference impedance. This problem can be overcome by adding epoxy resin to pure graphene which will be leading to create a strong wander waal interactions in between graphene sheets [1, 9, 14, 15, 23]. It is reported in the literature that CB based structures have [24, 25] exhibited maximum shielding effectiveness value of $-15$ dB with 8 wt% filler concentration [13, 23] and EMISE value of $-19$ dB for MWCNTs with 20 wt% [1, 13, 16, 20, 23]. It is perceived from previous studies that graphene has improved EM absorption performance than MWCNTs [4, 20, 23].

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In recent years porous structures were used as shielding materials and these structures can reduce back reflections of EM radiations, impedance mismatch and also improves EM penetration depth into the structure. Microwave absorption performance of different polymer based materials like PMMA [2], polystyrene [3, 16], polyurethane [4, 7, 12, 17–20], PPY [11], PANI [15, 26], PET [26], epoxy resin [4, 7, 8], PVA [6] has been investigated. Best of the author’s knowledge, few studies were observed to improving the EM absorption performance using conducting polyurethane-based structures with low filler concentrations. Moreover computational model approach is observed to be essential for microwave absorption analysis of graphene based structures. In this regard, few researchers have developed computation models to study the shielding performance of SWCNTs [27, 28].

Finally, thin EM absorption structure is a paramount for the reduction of EM pollution. A systematic model is essential for the development of cutting edge technologies to design effective thin EM absorbing structures. The broad objective of the present work is reduction of EM radiations internm of EMI shielding and RL using lightweight polymer nanocomposite materials. A systematic study is conducted which includes fabrication of epoxy and polyurethane (PU) as base materials and reinforced with various wt% of graphene separately to measure EMI shielding effectiveness (EMISE) and RL pertained to X-band. These results are compared with pure polymer structures such as epoxy and PU. Moreover present work results will be compared with available literature.

2. Experimental details and analysis

2.1. Materials
Raw graphene with surface area 120 m² g⁻¹; contains more than 95% of carbon and average thickness of graphene sheet is 6 to 8 nm and conducting polymer polyurethane (PU), DMF (Dimethylformamide) were purchased from Sigma Aldrich, India. Other materials E–glass fiber (650 GSM), room temperature hardener (HY 951) and Epoxy resin (LY 556) were purchased from Ram Composites, India.

2.2. Fabrication of PU/Graphene composite
Polyurethane consisting graphene with different wt% (0, 0.5, 1, 1.5, 2 and 2.5 wt%) were synthesized using solution blending method. PU granules were broken down in DMF using a magnetic stirrer for 6 h at 60 °C and similarly graphene particles were uniformly dispersed in DMF for 6 h at 60 °C using probe sonicator. Two separate solutions namely graphene/DMF and PU/DMF were added and then stirred at 80 °C temperature until the solution has become powder using magnetic stirrer. Thus obtained powder was kept in vacuum oven to remove excess moisture at a temperature of 120 °C for 48 h.

2.3. Fabrication of PU/Graphene/epoxy/E–glass fiber composite
Proper dispersion of graphene based samples in epoxy is a major challenge in preparation of graphene/PU/epoxy nano composites. Good distribution state of filler (Graphene and PU/Graphene) in the composite affects its electrical and physical properties and this depends on the processing technique. The distribution of filler material in the matrix has been achieved by means of solution mixing process. Graphene filler material has dispersed in the epoxy resin at various wt% (0.5 to 2.5 wt%). The required quantities of filler material, fiber weight and matrix material have been calculated using rule of mixtures concept for a given size and thickness of laminate. Graphene/epoxy solution was sonicated using ultra tip sonicator for 90 min to get proper distribution of graphene in the epoxy resin. Based on the chemical properties of epoxy and hardener, the ratio of epoxy/graphene and epoxy/graphene/PU were added separately to the hardener while maintaining the solution ratio as 100:11 for all weight percentages. E–glass fiber was cut to specific dimensions (50 × 50 mm²) and soaked with dispersed graphene composites and hardener mixture. These fabric layers were stacked and packed to form 2 mm thickness. This process was repeated for all graphene composite wt%.

2.4. Microwave absorption measurements
The nanocomposite sample was cut into specific dimensions 22.86 × 10.16 × 2 mm³ for measuring EM properties and S—parameters as shown in figure 1(a), using waveguide measurement setup.

The schematic diagram of EM radiation incident on PU/graphene/epoxy based single layered structure and wave propagation within the composite has been shown in figure 1(b). Absorption performance of fabricated sample was measured internm of RL and it is measured using equations (1) and (2) [29].

\[
RL_{dB} = 20 \log_{10} \left( \frac{Z_i - Z_0}{Z_i + Z_0} \right)
\]
Here $Z_i$ is input impedance at air and absorber interface $Z_0$ (free space impedance) value is 377 $\Omega$, $f$ indicates the frequency of incident EM radiation in GHz, $\varepsilon_r$ and $\mu_r$ indicates the relative permittivity and permeability of fabricated sample.

When electromagnetic wave incidents on the surface of the composite material, the total energy is divided into three parts which are the reflection power ($R$), absorption power ($A$) and transmission power ($T$) of the composite. Total shielding efficiency ($SE_T$) can be expressed in terms of the $S$—parameters which were calculated using equation (3) [1, 6, 8, 10, 13, 15, 29].

$$SE_T = 10 \log_{10} \frac{1}{T} = 10 \log_{10} \left( \frac{1}{|S_{21}|^2} \right) = 10 \log_{10} \left( \frac{1}{|S_{11}|^2} \right)$$ (3)

### 3. Results and discussion

Crystalline nature of graphene composites are observed using XRD (Rigaku) within the incident angle range ($2\theta$) from 10.0154° to 90.9524° under standard atmospheric conditions and results are presented in figure 2(a).

Graphene powder is exhibited a strong diffraction peaks at 26° (002) [24, 25, 30]. PU granules has shown strong peaks at 20.8° (110), which confirm the presence of short range amorphous PU matrix [24, 25, 30]. In XRD pattern of PU graphene composite, graphene diffraction peak is vanished which is attributed to exfoliation during the sonication process. From the results it is observed that positions of diffraction peaks are unchanged after adding graphene particles. Intensity peak is weakened with increase of weight fractions due to interfacial interactions between PU and graphene which leads to reduction of PU crystalline size [24, 25, 30].

Raman spectroscopy is one of the important technique to characterize carbon based structures and composites which can also be used to present the interaction between polymer and carbon structures. Figure 2(b) presents the Raman shift results of pure graphene, PU and graphene composites. From the results it is evident that graphene have attained peaks at two different places which are 1358 cm$^{-1}$ (crystalline nature of carbon) and 1585 cm$^{-1}$ (SP$^2$ bonded carbon structure) and these peaks have pertained to D and G bands.
Thus it confirms the carbon bonding in graphene as well as its structure. Pure PU shows different peaks at 2930, 1742 and 1630 cm$^{-1}$ corresponds to stretching vibration of $–\text{CH}_2$, free carbonyl group and stretch vibration of C=C respectively. With increasing the weight fractions of graphene, peak intensity become weakened gradually and which will be vanished further with increase in graphene weight fractions. Thus it affirms existence of $\pi-\pi$ interactions in between PU and graphene particles. Peak intensity decrease can also be caused due to conversion of some of the SP² carbons into SP³ [24, 25, 30].

The structures of graphene and PU/graphene were recorded at 200 nm scale using TEM and are presented in figures 2(c) and (d) respectively. From the figure 2(c), the wrinkled graphene sheet is observed. Which is a renowned characteristic of graphene flake structure and this lattice damage is attributed to chemical and thermal
oxidation process. One more observation is recorded which is high transparency of structure is indicating the small number of atomic layers. TEM analysis of PU graphene 2.5 wt% composite in figure 2(d) shows the uniform dispersion of graphene in PU which leads to improve the microwave absorption performance of graphene composites [24, 25, 30].

The surface morphology imaging of graphene and PU/graphene composite structures were carried out using FESEM (JSM7100F) and results are presented in figures 2(e) and (f) respectively. Flake structure of graphene is obtained and is observed at 10 μm scale as shown in figure 2(e). Improved dispersion of 2.5 wt% of graphene in PU is confirmed from the SEM as shown in figure 2(f). Homogeneous dispersion of graphene in PU improves the shielding performance of composite [24, 25, 30].

EM properties permittivity and permeability of fabricated samples were measured using waveguide measurement setup (figure 3) in X-band. These properties are used as input for measurement of RL and EMISE for various PU/graphene/epoxy nanocomposites.

The variation of real (ε′) and imaginary (ε′′) part of relative permittivity in X-band can be seen in figures 3(a) and (b). This is due to the occurrence of electronic and atomic polarization in the composite material. Real permittivity of graphene/epoxy composite is varying from 3.9 to 5 on the other hand for PU/graphene/epoxy composite this value in between 4 to 7.5 in X-band region. Imaginary parts of graphene composites are in the range of 0.6 to 1.8 in X-band. From the figure 3(b) it is observed that permittivity values of graphene composites are increasing with increase in weight percentage of filler material. Real and imaginary permittivity values of PU graphene composite little bit high when compared pure graphene because of increase in electrical conductivity and electronic polarization.

Real (μ′) and imaginary (μ′′) parts of relative permeability values of fabricated samples are presented in figures 3(c) and (d). Generally, permeability value of dielectric material is 1. It is evident from figure 3(c) that graphene nanocomposite structures have shown more than 1 and it is corroborated to antiferromagnetic nature at room temperature [22, 23].

Electromagnetic wave absorption performance of low loading graphene/PU/epoxy composites is measured in terms of RL. Figure 4(a) presents the RL of all graphene composites in X-band frequency range. It is evident from figure that composite materials with 2 mm thickness of graphene 2.0 wt%, 2.5 wt% and PU/graphene 2.5 wt% have absorbed more than 85% of incident radiation. Maximum RL value −35 dB is obtained at 12.18 GHz for PU graphene 2.5 wt%. With increasing the weight fractions, total RL of fabricated samples are increasing in X-band. RL performance of PU/graphene/epoxy composites is majorly due to dielectric relaxation, polarization relaxation and defects raised with in the fabricated samples. It is noticed that electric
dipole polarization in the graphene based composites and multiple reflections of EM radiations in between polymer foam and graphene flakes are also caused for improvement of absorption performance in composite structures.

EMI shielding efficiency of PU/graphene/epoxy composites are presented in figure 4(b). EMISE values of pure epoxy and PU/epoxy samples are very low i.e. approximately $-13$ dB and $-19$ dB respectively because of its transparent nature. By adding graphene to the pure epoxy and PU/epoxy structures, SE values are increasing with increase in weight percentages of graphene because of improvement in electrical conductivity. It can be seen from figure 4(b) that EMISE values of different wt% of graphene/epoxy composites are varying from $-13$ to $-27$ dB in X-band. Among all graphene/epoxy composites, 2.5 wt% graphene sample has exhibited maximum shielding performance i.e. $-30$ dB at 9 GHz. Moreover, graphene reinforced PU/epoxy nanocomposites have exhibited SE values in the range of $-19$ dB to $-45$ dB in X-band. Also maximum EMISE was attained by 2.5 wt% graphene/PU nanocomposite i.e. $-47$ dB at 8.3 GHz. Further, EMISE results of both epoxy and PU reinforced with graphene nanocomposites were compared. It is found that PU/graphene/epoxy nanocomposites have shown better shielding performance than graphene/epoxy nanocomposites. Electrical conductivity and conductive networks are playing a vital role throughout the composite structure in the estimation of EMI shielding efficiency. When EM radiation incident on the structure, it penetrates through the conductive networks and further weakened due to conductive decadence. Polarization relaxation, impedance matching and electronic dipole relaxation will also improve EM wave absorption performance of composite structures [12]. Also these mechanisms will be influenced by the presence of residual defects within the graphene. Moreover the large surface area of graphene is beneficial for allowing the incident EM radiations falling on the structure. Thus the EMI shielding performance will be enhanced for PU/graphene nanocomposites.

The optimum results (RL and SE) of PU/epoxy reinforced with 2.5 wt% graphene nanocomposite are compared with literature. These results are pertained to X-band as presented in table 1.
4. Conclusions

PU and epoxy polymer structures are reinforced with low weight concentrations of graphene (0 to 2.5 wt% with an increment of 0.5) to study the EM absorption performance in X-band. PU/graphene nanocomposite structures are synthesized using solution method latter it is reinforced into glass fiber to form a sample thickness of 2 mm using high pressure resin transfer molding technique. Here two different nanocomposites namely epoxy/graphene and PU/epoxy/graphene were fabricated for the analysis of EM wave absorption performance. The performance is measured in terms of RL and total SE which are calculated using EM properties pertaining to X-band. Moreover these results are compared with pure epoxy and PU structures.

From results it is observed that EM properties and EM absorption properties are increased with increase in wt% of filler material. It is predicted that PU/epoxy reinforced with 2.5 wt% graphene has shown maximum absorption capability approximately 85% in X-band. Also maximum RL value of $-35$ dB is obtained at 12.18 GHz for the same composite structure and it is corroborated to dielectric and polarization relaxation. Moreover best shielding effectiveness (SE) was observed for the same composite structure which is $-44.3$ dB in X-band. Thus it can be concluded that graphene 2.5 wt% reinforced PU/epoxy nanocomposite can be used in the field of electronic equipment, military and naval applications.

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Table 1. Microwave absorption comparison of different materials in X-band.

| S. No | Filler | Filler wt% | Polymer | Thickness | EMISE | Ref |
|-------|--------|------------|---------|-----------|-------|-----|
| 1     | Graphene | 5%         | PMMA    | 4 mm      | $-17.5$ dB | [1]   |
| 2     | Graphene | 30%        | Polystyrene | 2.5 mm   | $-30$ dB | [2]   |
| 3     | MWCNT   | 20%        | Epoxy resin | 1.75 mm  | $-19$ dB | [4]   |
| 4     | CNF     | 20%        | PPy     | 0.5 mm    | $-25$ dB | [10]  |
| 5     | Graphene | 5.5 (vol %) | PU      | 3 mm      | $-21$ dB | [11]  |
| 6.7   | Graphene | 20%        | Polycarbonate | 0.22 mm  | $-45$ dB | Present work |

Table 2. RL comparison of different materials in X-band.

| S. No | Filler | Filler wt% | Polymer | Thickness | RL | Ref |
|-------|--------|------------|---------|-----------|----|-----|
| 1     | Graphene | 10%        | TPU     | 2 mm      | $-7$ dB | [3]   |
| 2     | MWCNT   | 10%        | PVA     | 1 mm      | $-4$ dB | [5]   |
| 3     | TiO$_2$ coated MWCNT + Fe$_3$O$_4$ | 15% + 15% | TPU     | 2 mm      | $-8$ dB | [6]   |
| 4     | RGO     | 31.3%      | PU      | 2.5 mm    | $-6$ dB | [17]  |
| 5     | Graphene/Barium Titanate | 20% | PVP  | 5 mm      | $-10$ dB | [20]  |
| 6     | Graphene | 2.5%       | PU      | 2 mm      | $-6.12$ dB | Present work |

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