

INTERCALATING POLYMER WITH NANO STRATUM GRAPHENE FOR EMI SHIELDING

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Abstract

Electromagnetic interference (EMI) has been identified as one of the ever-increasing unintended consequences of the widespread use of electronic devices and systems. There is a growing demand for effective shielding materials and fillers to control EMI and related concerns. Increased filler content usually results in higher costs, poor dispersion, and unwanted agglomeration, making polymer composites mechanically fragile and difficult to produce. As a result, developing a robust composite with conductive filler content while maintaining good EMI shielding performance is extremely desirable. Hence in this paper, a hybrid polymer composite based on polyetherimide is suggested by dispersion of conducting polymers like polyacetylene and MWCNT fillers with a Graphene substrate. Then the improvement in EMI shielding effectiveness is investigated. The blending techniques of the polymer matrix and the reinforcing filler materials are discussed and the design of the Graphene substrate was done with a nano filler based coating on it. The effectiveness of the shield is then analyzed with ANSYS-HFSS software. S-parameters of the shield like S11, S12, and S21, etc. are determined using the plots obtained and this showed superior performance. Hence by implementing the proposed design the high-performance EMI shielding materials can be fabricated by the combination of different fillers for the shield. This will improve the electrical, mechanical, and EMI shielding properties of the composites.

Keywords: Crosslinked Graphene substrate; Coupled Nano stratum coating technique; electromagnetic interference; graphene; Intercolated Polyacetophenimide; MWCNTs

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Introduction

Electromagnetic interference (EMI) has become a major concern owing to rapid advancements in the electronics industry and telecommunications networks. Conducting polymers are a convenient choice for EMI shielding applications (Ravindren *et al.*, 2019). Various important forms of conducting polymers have been produced using Nano electronics and nanotechnology (Panda and Acharya, 2019). Polymer nanocomposites, made up of polymer matrices and nanometrical reinforcement phases, are commonly used in composite materials due to their ease of processing and shaping.

Graphene is a two-dimensional substance with nanoscale diameter and thickness, used for improving thermal and electromagnetic interference shielding properties of nanocomposites (Ha *et al.*, 2019). It has been used in radar absorption and EMI shielding applications for hybrid compounds with Graphene Nano platelets as fillers. Graphene oxide (GO) is a continuous layer with strong polymer compatibility, wide surface region, and excellent chemical stability. It is also used to stabilize metal, ceramic, and polymer matrix materials. Graphene's unique properties and low mass density make it a promising material for future applications (Abutalib, 2019).

Graphite Nano platelet GNPs have high thermal and electrical conductivity with a lower mass density, in comparison to conventional 2D Nano fillers, such as Nano clays. In comparison the manufacturing cost for GNPs compared to carbon nanotubes is comparatively low (CNTs) (Gao *et al.*, 2018). But further expansion of polymer composites properties and applications is yet to be made feasible by adding Carbon Nanotubes (CNTs) and Graphene as Nano fillers.

The study examines the necessity for an upgraded EMI shield with enhanced properties, optimised filler content and sturdy manufacturing. It highlights the need for a novel technique to overcome these issues. A new polyacetophenemide matrix containing CNT fillers and a Graphene substrate is described in this paper. The work is divided into four chapters. The first three chapters consist of an introduction, a description of the finished task, details of simulation and results. The last chapter discusses the work's conclusion.

Intercalating Polymer with Nano Stratum Graphene for EMI Shielding

High-frequency electrical radiation with electromagnetic interference (EMI) has become

a major threat to the security of information. Electrical conductivity in shielding is necessary to maintain high conductivity which is generally fulfilled by filler particles. Nastier filler particles, on the other hand, result in severe electrical percolation and device impedance inadequacies, which significantly reduce composite conductivity. Also large quantity of fillers tends to improper dispersion in the matrix and also results in low thermal stability which makes the shield unstable at high temperatures. Hence here for the betterment of the shield with inherent conductivity and improved properties a novel Intercolated Polyacetophenimide matrix is proposed in which multi walled carbon nano tubes are added as fillers. Polyetherimide with Thiophene and Acetylene polymers mixed with di butyl sebacate are blended to form the mixture. A modified coupled magnetic stirrer technique is suggested for this which ensures proper dispersion. Furthermore, by employing a novel Crosslinked Graphene substrate technology, the shield's electrical conductivity and thermal stability can be increased. After weighing all the factors, the most important one is how to fabricate the polymer nano composite shield without sacrificing its qualities. This can be done by using a novel technique called Coupled Nanostratum coating, which uses a designed die to fuse polymer film to the Graphene substrate while creating an even coating on the substrate (Guan *et al.*, 2018). The unique approaches described above improve shielding performance by reducing filler quantity while maintaining proper filler dispersion, improving electrical conductivity, increasing thermal stability, and increased connectivity.

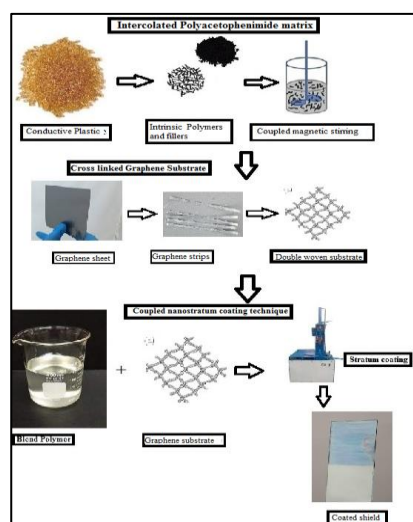


Figure1. Block diagram of the proposed work

The block diagram of the proposed methodology is given in Figure 1.

Intercalated Polyacetophenimide Matrix

Conducting polymer based EMI shields are used recently with conductive filler materials in order to improve the conductivity and thermal stability of the shielding. Here the polymer matrix is made of Polyacetophenimide, which is a blend of Polyetherimide as the polymer matrix with conducting polymers like Thiophene and Acetylene blended to it. Polyetherimide is a high performance engineering thermoplastic. Its characteristics include high strength and rigidity at elevated temperatures, dimensional stability, long term heat resistance and good electrical properties. Since EMI shielding material should be of conductive in nature and in order to reduce filler content, we have chosen the blend of polymers of conductive nature (Thiophene and Acetylene) with improved properties. Multi walled CNT fillers are used here to improve the reflective property of the shield. The properties of the Polyetherimide polymer (Ahmad *et al.*, 2017) are shown in Table 1 below.

Multiwalled Carbon nanotubes are used as fillers in the matrix in order to improve the thermal and electrical conductivity. The properties of the filler particles are depicted in Table 2. Conducting polymers are used for reducing the filler addition in the matrix and also to improve the conducting properties. Thiophene and Acetylene are used for this. The properties of Thiophene (Roncali, 1992) are given in Table 3.

Table 1. Properties of Polyetherimide

Density	1,270 (kg/m ³)
Melting point	219 (°C)
Thermal conductivity	0.22 (W/mK)
Tensile modulus	3,276 (MPa)
Tensile strength	126 (MPa)
Poisson's ratio	0.36
Coefficient of thermal expansion	5.58×10 ⁻⁶ (C ⁻¹)
Glass transition temperature	227 (°C)
Melt index	0.42 (g/min)
Linear thermal expansion	3.3 (°C)

Table 2. Properties of MWCNTs

Purity %	98%
-OH Content	1.76 Wt%
Outer diameter	10-20 nm
Inner diameter	5-10 nm
Length	0.5-2.0 µm
Surface area	>200 m ² /g
Density	0.22 g/cm ³
Electrical conductivity	>100 S/cm

The properties of Acetylene (West, 1980) is shown in Table 4.

The chosen plastic polymers are electrically conductive, therefore very little filler is needed to create the necessary weight percentage of each polymer powder to create the polymer blend. This reduces clumsy dispersion without sacrificing conductivity capabilities.

Polyetherimide with Thiophene and Acetylene polymers mixed with di butyl sebacate can be blended to form the mixture by a modified coupled magnetic stirrer technique, which ensures proper dispersion of MWCNTs which are added as filler material. The purpose of adding di butyl sebacate is to enhance the plastic property of the shield. The blending technique of magnetic stirrer is mainly used for proper dispersion of the filler material and even mix of the material.

The coupled magnetic stirrer technique (Loos, 2015) involves the melting of the polymer materials assisted with continuous stirring with magnetic bars. Melting and processing is a versatile and widespread method for dealing with insoluble polymers, especially thermoplastic polymers. The fact that thermoplastic polymers soften when heated is used in this technique. Melt processing is the process of melting polymer pellets into a viscous liquid. Rheological shear mixing can be used to incorporate any additives, such as carbon nanotubes, into the melt. Using the magnetic stirring technique, a stir bar is moved around in liquid samples by means of a rotating magnetic field. With its quick movement and agitation, this stir bar fully blends the sample. Hence a melted polymer blend with good dispersion of fillers and improved conducting properties is obtained. Moreover, a Graphene substrate is added to the shield to improve its thermal stability and offer a continuous conducting property (Figure 2).

Table 3. Properties of Thiophene

Molar mass	84.14 g/mol
Density	1.051 g/mL
Melting point	-38°C
Boiling point	84°C
Magnetic susceptibility (χ)	-57.38·10 ⁻⁶ cm ³ /mol
Refractive index (n_D)	1.5287

Table 4. Properties of Acetylene

Molar mass	26.038 g·mol ⁻¹
Density	1.1772 kg/m ³
Melting point	-80.8°C
Conductivity	4.4×10 ⁻⁵ Siemens/cm
Magnetic susceptibility (χ)	-12.5×10 ⁻⁶ cm ³ /mol

Cross-linked Graphene Substrate

To further enhance the shield's performance, a substrate composed of Graphene sheets is included. Here, it is suggested to divide the graphene sheets into strips with a $0^\circ/90^\circ$ orientation for the strips in order to maximise material utilisation and enhance the effective conducting property (Figure 3).

Because of its outstanding electrical properties and super-high specific surface area, Graphene, has been touted as a very promising and effective EMI shielding material. To date, light-weight and high-performance EMI shielding has been achieved using carbon-based shields with various nano/microstructures. Because of its extraordinary charge transport, thermal, optical, and mechanical capabilities, Graphene, has emerged as an exotic substance of the twenty-first century. Graphene's strong conductivity, on top of all of this, makes it an ideal option for such applications. The properties of Graphene (Kausar, 2018) are shown in Table 5.

Table 5. Properties of Graphene

Melting point	3,800 K
Thermal conductivity	$3\text{-}5 \times 10^3$ W/mK
Current density	$>10^8$ A/cm ²
Electron mobility	$>10,000$ cm ² /V.s

Hence here the stripped Graphene sheets are suggested to make the substrate over which nano coating has to be done. The Graphene sheets should be cut and oriented in crosslinked pattern which ensures continuous conductivity and thermal stability characteristics. Graphene has an electrical conductivity of 2.62×10^2 S/m. The use of Graphene as the substrate in the shielding material enhances the properties of the EMI shield in terms of absorption and reflection losses. Then the coating of the polymer is done over the substrate to make the EMI shield (Figure 4).

Coupled Nano Stratum Coating Technique

The nano stratum technique should be done for the coating of the polymer over the Graphene substrate which provides the shield material with enhanced property due to the combining of polymer coating and the substrate. The coating of the polymer over the substrate can be done by a non-contact coating technique which uses a slotted die as per the thickness of the coating.

Slotted-die coating (Patidar *et al.*, 2020) uses a coating process in which the slotted-die is normally kept at a considerable standoff distance over the substrate. Hence this noncontact ensures good surface of coating. A fluid bridge connects

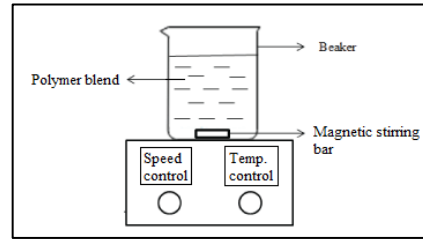


Figure 2. Temperature controlled Magnetic stirrer

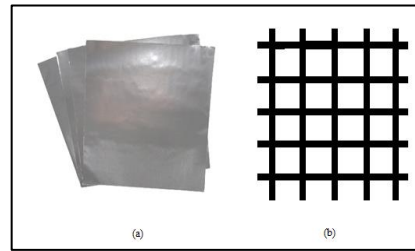


Figure 3. Graphene sheets (a) full sheet [IndiaMART], (b) $0^\circ/90^\circ$ oriented Graphene strips

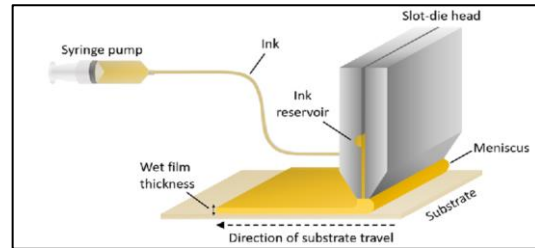


Figure 4. Slotted coating technique

the slot-die tip and the substrate surface, allowing the coating fluid to flow from the slot-die to the substrate. The coating bead is the usual name for this fluid bond. The ratio between the applied volumetric pump rate and the coating rate is used to manage the thickness of the resulting wet coated layer. Unlike self-metered coating technologies like blade and bar coating, the slotted-die does not use any damaging physical contact or scraping to alter the thickness of the coated layer. A temperature control can be coupled with the machine so as to maintain the wettability of the polymer blend. As a result, the coating of the substrate is made using the technique. The height of the slot-die, on the other hand, is important in defining the quality of the coated film because it limits the distance that the meniscus must span to keep the coating process steady. Hence depending on the thickness and width of coating the parameters can be varied.

$$l = \frac{F}{W \cdot S} \quad (1)$$

Where l -Thickness of the wet coated layer, W -Width of coating (W), F -Volumetric pump rate, S -Coating speed or relative speed between the slot-die and the substrate during coating.

A pre-metered liquid coating mechanism is suggested for slotted-die coating. The thickness of the layer can be increased by raising the pump rate, while the thickness is decreased by increasing the coating speed or coating width. For a specific slotted-die process, the coating width is usually a fixed value. As a result, the major factors used to calculate, regulate, and alter the wet film thickness (Carvalho and Kheshgi, 2000) in a highly predictable manner are pump rate and coating speed which are given by the equations.

$$T = l * \frac{C}{\rho} \quad (2)$$

Where T is the final thickness, C is the solid concentration, ρ is the volumetric density of the coated material. Thus the provision of the stratum provides electron percolation and high conductivity constantly between the neighboring filler particles. Thus it acts as an enhancement to the thermal and electrical properties of the shield material. The property improvement is determined using the HFSS software which depicts the properties like absorption, transmission and reflection.

Simulation and Results

This section provides a description of simulations done and the performance analysis of the proposed structure. Comparison section is also included to show the improvement of proposed shield material.

Simulation Details

For the suggested work the analysis of the EMI shield was then done using ANSYS HFSS. The dielectric permittivity, permeability, and mass density are given as the input for the model. The values for the inputs are found by applying the formula of rule of mixtures for the composite shield material. Dielectric permittivity of the composite (Ávila *et al.*, 2015) is determined from the given Equations (3), (4) and (5) and are given as input.

$$\epsilon = (1 - V_m) \epsilon_m + V \epsilon_f \quad (3)$$

Where ϵ - permittivity, V_m - Volume fraction of matrix, ϵ_m - permittivity of matrix, ϵ_f - permittivity of fiber.

The permeability of the composite (Thabet and Repetto, 2014) is determined using the Equation (4)

$$P = P_m + 2P_m \frac{P_f - P_m}{P_f + P_m - V_f(P_f - P_m)} \quad (4)$$

Where P_m - permeability of matrix composite, P_f - permeability of the fiber composite, V_f - Volume fraction of fibers.

The density of the polymer (Tam *et al.*, 2012) composite is found using the Equation (5)

$$\rho V = \rho_m V_m + \rho_f V_f \quad (5)$$

Where ρ_m , ρ_f - density of matrix and fibers, V_m , V_f - Volume matrix and fibers.

Results from Analysis

The results from the analysis of the software are described below. The model of the strip material in HFSS is shown which is surrounded by a boundary box.

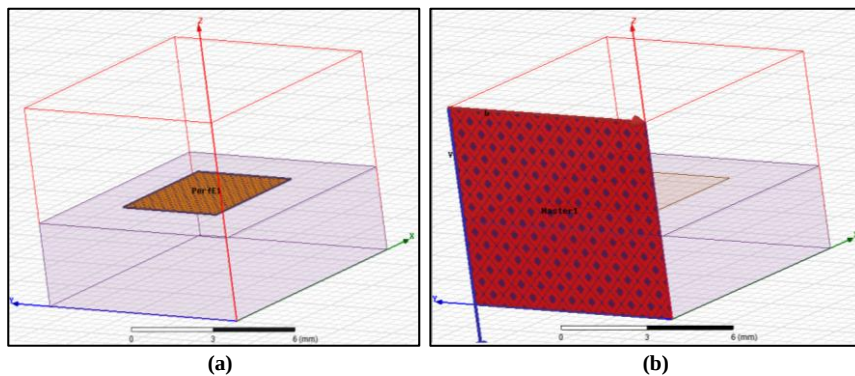


Figure 5. a) Model of the strip material, b) Model with boundary box

The model of the shield was done with a patch of 4×4 cm with 2 mm thickness with a boundary box dimension of 9×9×3 cm. It is shown in Figures 5(a) and 5(b).

The values of the characteristic performance of the shield were determined using S parameters. S₁₁ and S₁₂ represent the reflection coefficient and the parameters S₂₁ and S₁₂ represent the transmission coefficient which is determined by the S parameter plot.

Figure 6 represents the plot of reflection coefficient against frequency for the shield and it is found that the maximum reflection is 0.373 dB at 18.60 GHz. This good reflective property is due to the presence of the evenly dispersed multi walled CNT filler particles in the shield with even polymer coating on the substrate.

The transmission coefficient curve (Figure 7) depicts the amplitude of transmitted wave to the amplitude of the incident wave. Since S₁₂ for which the input and output ports are different, it measures the net transmitted power which is represented by the transmission coefficient and was found as 0.373 at 18.6 GHz. From the values of reflectivity and transmission the value of absorption (Geetha *et al.*,

2009) is calculated from Equation (6) and it is found to be 0.254.

$$S_A + S_R + S_T = 1 \quad (6)$$

Where S_A - shield absorption, S_R - shield reflection, S_T - shield transmission. Figure 8 denotes the 3D rectangular plot of the analysis. It gives the information about the reflection and transmission of the shield material vs. the frequency. From the plot it is clear that the maximum value for the parameters is 0.373 dB.

The Figure 9 denotes the 3D polar plot of the shield which denotes the combined S parameters of the shield with frequency. The value of the S parameters denoting the range are found to be 0.373 dB(minimum) and 0.40 dB(maximum) respectively with frequency.

A smith chart is to visualize the impedance of the system as a measure of frequency. It is shown in Figure 10. From the chart, the resistance of the shield lies on 0.075 circles and the source impedance lies on 0.8. Hence the value of resistance and impedance are 7.5 ohm and 0.8 ohm

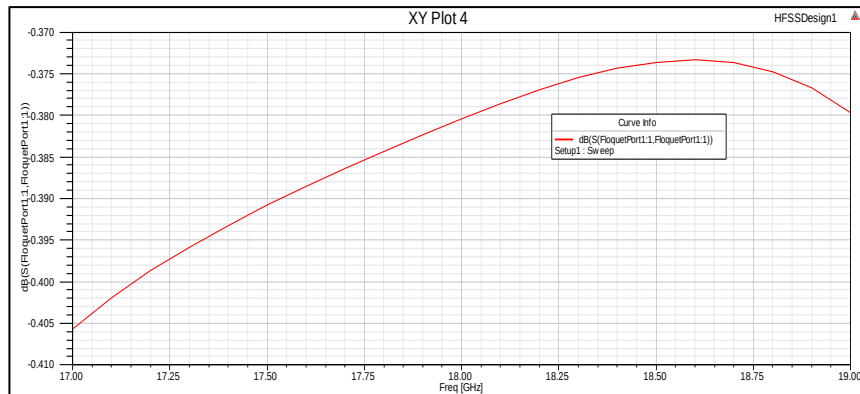


Figure 6. Reflection coefficient curve

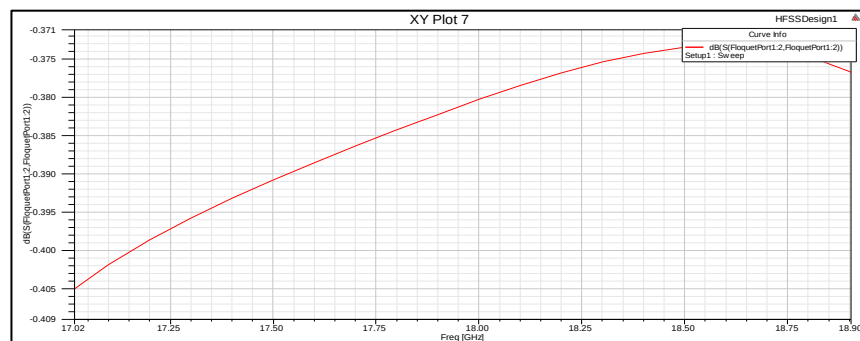


Figure 7. Transmission coefficient curve

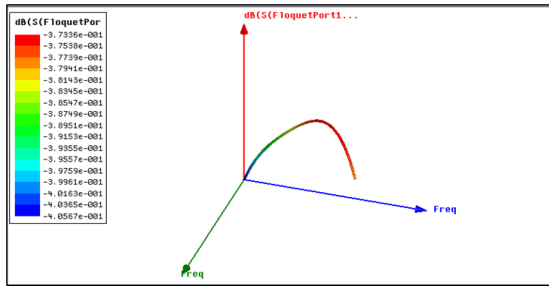


Figure 8. 3-D Rectangular plot

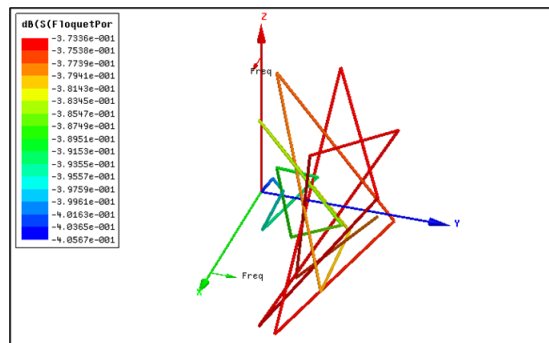


Figure 9. 3D Polar plot

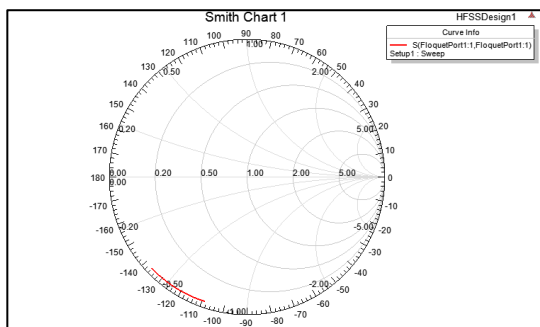


Figure 10. Smith chart

respectively. Thus the presence of fillers and Graphene substrate provides good conductivity with reduced resistance values. As a result, the novel techniques described above improves the performance of the shielding by reducing filler quantity, maintaining proper filler dispersion, improved electrical conductivity, increased thermal stability and also connectivity.

Comparison

The section compares the proposed material for various methods and material concentration.

From the Figure 11 the comparison of the absorption value with respect to filler concentration (Banerjee *et al.*, 2020) can be observed. Various

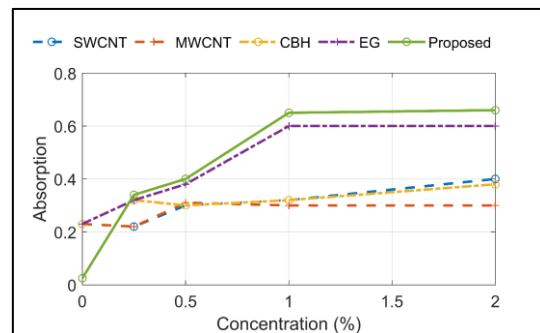


Figure 11. Comparison plot of Absorption vs filler concentration

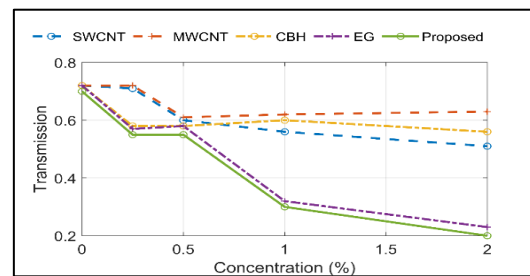


Figure 12. Transmission vs Concentration

filler concentrations of Single walled carbon nanotubes (SWCNT), multiwalled carbon nanotubes (MWCNT), Carbon black (CBH) and exfoliated graphite (EG) with 0.5 to 2% concentration are compared with the proposed material. Plot shows that the proposed material shows superior absorption capability of 0.25 with beyond 1% filler concentration.

In Figure 12 the comparison of the transmission value with respect to filler concentration (Banerjee *et al.*, 2020) is plotted. Various filler concentrations of Single walled carbon nanotubes (SWCNT), multiwalled carbon nanotubes (MWCNT), Carbon black (CBH) and exfoliated graphite (EG) with 0.5 to 2% concentration are compared with the proposed material. Plot shows that the proposed material shows the least transmission characteristics capability of 0.2 with 2% concentration of filler addition.

The shield effectiveness for absorption as a measure of frequency is shown in Figure 13. It compares the proposed material (Banerjee *et al.*, 2020) with various other material concentration of Graphene oxide over carbon (GCF) with the presence of epoxy (EP) and hybridisation of Fe_3O_4 Nanoparticles (MG). From the plot it is clear that the absorption for the proposed material is better

than the existing materials. The value is found to be 0.3475 dB at a frequency of 12 GHz.

The comparison of shield effectiveness for reflection as a measure of frequency is shown in Figure 14. It compares shield effectiveness of the proposed material with various other materials like polyvinylidene matrix (Ahmed *et al.*, 2020) with Graphene fillers. The matrix is hybridized with nickel ferrites as fillers and is compared for various concentrations. It is evident from the plot that the proposed material has better reflection than the materials that are currently in use. The value is found to be 2 dB at a frequency of 12 GHz and shows enhanced shield effectiveness.

The Figure 15 depicts the variation of resistance with temperature which is compared (Yussuf *et al.*, 2018) with other shield materials like Ammonium persulphate and ferric chloride. The plot makes it clear that the resistive power of the shield material is lower than the compared materials with increase in temperature. The value stays at 18 ohms for a temperature of 180°C.

Conclusions

In this work, polymer matrix materials were used to develop the EMI shield material. ANSYS-HFSS software was then used to analyse the material and identify its various properties. Graphene and conductive polymers were added to the materials chosen for the EMI shield to boost its performance, which was subsequently simulated using HFSS software. The S-parameter characteristics for absorption, reflection and transmission were determined with the plots and charts generated from the analysis. Hence it is concluded that the shield material has good SE_A value of 0.254 dB, SE_R value of 0.373 dB and the SE_T value of 0.373 dB at high frequency of 20 Hz. Finally, the proposed model is compared with different material reinforcement and filler concentration which showed a 30.75% increase in absorption characteristics, a 72% improvement in transmission characteristics and a 60 percent improvement in the reflection characteristics than the existing materials.

Nomenclature

L	- Thickness of the wet coated layer
W	- Width of coating (W),
F	- Volumetric pump rate,
S	- Coating speed, or relative speed between the slot-die and the substrate during coating
T	- the final thickness
C	- the solid concentration

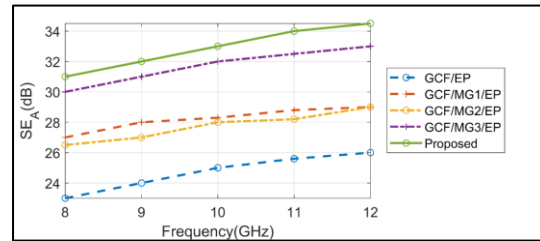


Figure 13. SE_A vs frequency

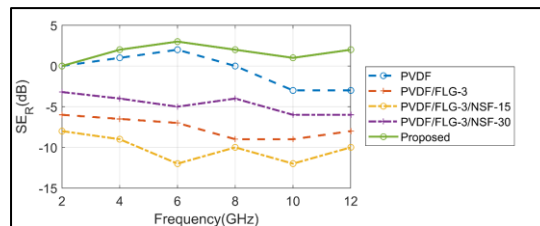


Figure 14. SE_R vs Frequency

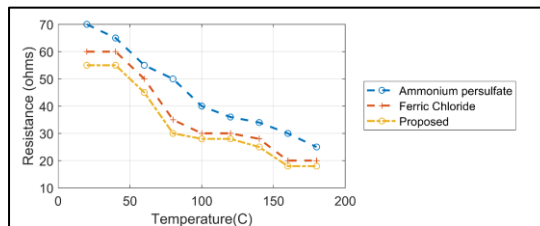


Figure 15. Resistance vs temperature

ρ	- the volumetric density of the coated material
ϵ	- permittivity
V_m	- Volume fraction of matrix
ϵ_m	- permittivity of matrix
ϵ_f	- permittivity of fiber
P_m	- permeability of matrix composite
P_f	- permeability of the fiber composite
V_f	- Volume fraction of fibers
ρ_m, ρ_f	- density of matrix and fibers
V_m, V_f	- Volume matrix and fibers
S_A	- shield absorption
S_R	- shield reflection
S_T	- shield transmission

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