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LDPE-based Composites for Electromagnetic Interference Shielding in X-Band: The Synergetic Effect of Magnetic $MgFe_2O_4$ and Conductive MWCNT - Graphene

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Abstract

To achieve the material's synergistic effect for better EMI shielding, this study focuses on evaluating the critical concentration of Magnesium Ferrite ($MgFe_2O_4$) nanoparticles with the combination of Graphene and Multi-Walled Carbon Nanotubes (MWCNT) dispersed in a Low-density polyethene (LDPE) polymer medium. Using urea as fuel, a solution combustion synthesis technique was used to create the magnetic filler $MgFe_2O_4$. At 900 °C, the calcined nanoparticles exhibit distinct phases. MWCNT and graphene were utilized as conductive fillers. Using a chemical exfoliation procedure, graphene was created. The fillers were analyzed using XRD, FTIR, Raman spectroscopy, SEM, EDS, and VSM. The XRD reveals the $MgFe_2O_4$ synthesized from solution combustion synthesis (SCS) is in pure phase without any impurity. The SEM result reveals the morphology of the prepared nanoparticle was Nano sized, well crystalline particles are formed. The composite of LDPE was studied for EMI shielding. The composite sample with a critical concentration of 50 wt. % LDPE, 5wt. % $MgFe_2O_4$, 40 wt. % graphene and 5 wt. % MWCNT (50:5:40:5) shows a superior SE value of 33.59 dB at 10.3 GHz.

Keywords: $MgFe_2O_4$; solution combustion; EMI shielding; TGA; MWCNT; Graphene; permittivity; permeability; mechanical strength

Introduction

One of the helpful technologies that allow us to transmit data and information over very great distances without encountering any impediments is electromagnetic waves. Unfortunately, the circuits are disrupted by electromagnetic waves, which usually causes the gadgets to malfunction. However, the growing telecommunication field and demand for novel types of wireless devices and technologies made humankind use a wide range of EM wave spectra for various applications such as medical diagnostics, radios, Bluetooth, television, cellular phones, Wi-Fi, 5G, radars, satellite communications, etc. [1]. This has also become an essential part of new civilization as modern trends and advancements upgrade technologies in lifestyle, military upgradation, and high-speed data transfer for today's essentials for every country [2, 3]. However, the other side of overexposure to EM waves leads to a death threat to birds, honeybees, and marine creatures. Also, it affects DNA mutation by altering the base pairs' bonding change, which eventually leads to genetic change in our newborn generation [4, 5].

Moreover, exposure to EM radiation also increases stress levels, insomnia, allergy, cancer, vertigo, and mentally returned, which affects mental health [6, 7]. To overcome such things, many research groups searched for novel techniques. Shielding is one of the promising fields, so EM reflectors are extensively used to develop EM wave-absorbing materials. Metal foils did shielding, and sheets were a primary selection, but the weight, corrosion, flexibility, and cost restrict its usage for use in a wide application [8].

Some people also prepared EMI shielding material with magnetic, conducting nanofillers with polymers. Still, the polymers they are adopting here are PANi, Poly acetylene, and polypyrene, which effetely have less mechanical and thermal properties and chemical stability [9]. So, one can think of using thermoplastic as a medium for holding magnetic and conducting nanofillers [10]. On the other hand, ferrites are an excellent choice of magnetic materials to absorb the magnetic component of the EM waves. Also, they are ceramic with good magnetic saturation, are chemically inert, and have high curie temperatures, making them a suitable choice in EMI shielding matter. Moreover, these composites are light in weight, low cost, easy to process, recyclable, thermally stable, chemically inert, gain electrical conductivity, and are helpful in general environmental conditions without any problem.

Graphene and MWCNT have been utilized in the current work to improve the EMI shielding effect. The optimized combination of these two fillers improves thermal stability by acting as a charring agent. Conducting fillers like graphene [11] and MWCNT [12] have good aspect ratios to introduce in a polymer and will bring a good amount of electrical conductivity if loaded in small amounts as well [13]. In addition, it offers the base polymer's value [14]. Therefore, these carbon filters are the cheap and best source of conducting filler, which helps develop conducting polymer composites for engineering and technical applications.

Materials and Methods

Materials

$Fe(NO_3)_3$, $MgNO_3$, urea, graphite powder, APS, H_2SO_4, procured from SD-fine chem, MWCNT was kindly provided by Ad-nanotech, India. LDPE of melt flow index (1g/10min) was supplied from Sabic polymers, India.

Methods

Synthesis of $MgFe_2O_4$ from Solution Combustion Synthesis (SCS) Method

In the synthesis procedure, a Petri dish with a 100 ml capacity is used to dissolve the stoichiometric ratio of ferric nitrate, magnesium nitrate, and urea. The redox mixture is kept in a muffle furnace, preheated at 500 ±10°C. After a brief period, the water evaporated, leaving behind a dense, concentrated gel that caught fire and self-burned with the creation of numerous gaseous molecules. The resulting substance was fluffy and porous. It was used to create phase-pure $MgFe_2O_4$ nanoparticles by grinding them and then heating them at 900°C for 3 hours [11].

Preparation of GNP

Graphene Nanoplatelets were prepared from a chemical exfoliation process by APS as an oxidizer and H_2SO_4 mixture with graphite powder. After being thoroughly stirred for three days, the reaction mixture was diluted with water and an ice bath. The finished product was microwave irradiated for 30 minutes at 80°C to produce yellow, worm-like graphene. This graphene was then filtered and washed before being heated to 300°C in an N_2 gas flow to produce expanded graphene [12].

Preparation of LDPE composite

The LDPE composites with different stochiometric ratios of fillers (LDPE, MWCNT, GNP and $MgFe_2O_4$) are prepared as shown in Table (1). All ingredients are mixed in Brabender plastic order, meld mixing instrument at 20 rpm, 20 min for each sample to ensure proper mixing of the fillers into the polyethene matrix. The well-blended matrix was then removed and compressed at 110°C for 1 hour in a 2x3 cm rectangle mould. The sample obtained was used for EMI shielding measurements in the VNA apparatus for getting S-parameters.

Sl. No.	***Polymer (in wt.%)***	***Conductive filler (in wt.%)***		***Magnetic filler (in wt.%)***
	LDPE	***MWCNT***	***GNP***	***$MgFe_2O_4$***
1	50	5	40	5
2	50	5	35	10
3	50	5	25	20
4	50	5	15	30
5	50	5	5	40
6	50	0	0	50

Table 1: Preparation of LDPE composites with a stochiometric ratio.

Characterization studies

A powder X-ray diffractometer, model BRUKER D8, was used to record the XRD patterns of magnetic and conducting fillers using Cu K radiation (λ= 1.5405Å). The synthesis of graphene and MWCNTs was studied using Raman spectroscopy (Renishaw India). The surface morphology of the samples was examined using a ZEISS Sigma 300 scanning electron microscope (SEM) outfitted with AMETEK EDAX. Using VSM at ambient temperature and a maximum applied field of 1.5 T, the magnetic properties of ZnFe2O4 were examined. Using a vector network analyzer (R&S®ZNB vector network analyzer), electromagnetic shielding effectiveness was tested in various bands of the microwave region (8.2-12.4 GHz).

Results and Discussion

Powder X-Ray diffraction (PXRD) analysis

Figure 1(a) represents the XRD graph of synthesized $MgFe_2O_4$ nanoparticles. The well-resolved patterns and intense peaks reveal pure and crystalline particle formation. It is also evident from the XRD pattern that $MgFe_2O_4$ is monophasic and polycrystalline and spinel structure. The diffraction peaks that are associated with the planes (2 2 0), (3 11), (4 0 0), (4 2 2), (3 3 3), (4 4 0) and (6 2 0). A well-defined phase of $MgFe_2O_4$ was formed in our material, and the Ferrite is free of the residual -Fe2O3 phase that is often expected during synthesis at comparatively lower temperatures. This phase corresponds well with the JCPDS (73-2410) file. Using Scherrer's formula, 35 nm is calculated as the $MgFe_2O_4$ crystallite size [13].

The PXRD of Graphene Nanoplatelets is depicted in Figure 1(b). A standard Crystallography Open Database (COD) number matches the pattern. The most pronounced peaks may be seen in the patterns at 26.53, 42.48, 44.54, and 54.64, which correspond to planes

(002), (100), (101), and (004), respectively [14]. The PXRD of MWCNT Nano platelets is depicted in Figure 1(c). The hexagonal graphite peak for the carbon nanotubes is indexed to the XRD of MWCNT diffraction peaks at 26 and 44, respectively, which correspond to (002) and (100) reflection planes (JCPDS No. 41-1487). Additionally, the pattern shows that the synthesized CNTs have a fair degree of crystallinity and a low percentage of amorphous carbon and impurities [15].

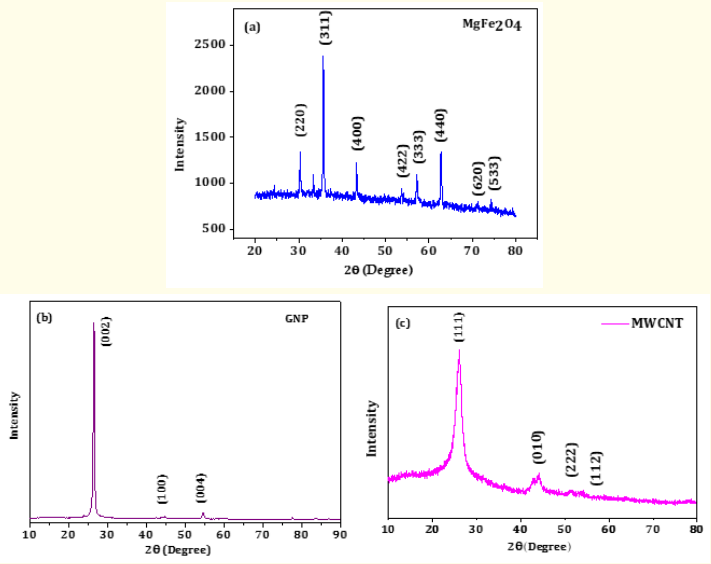


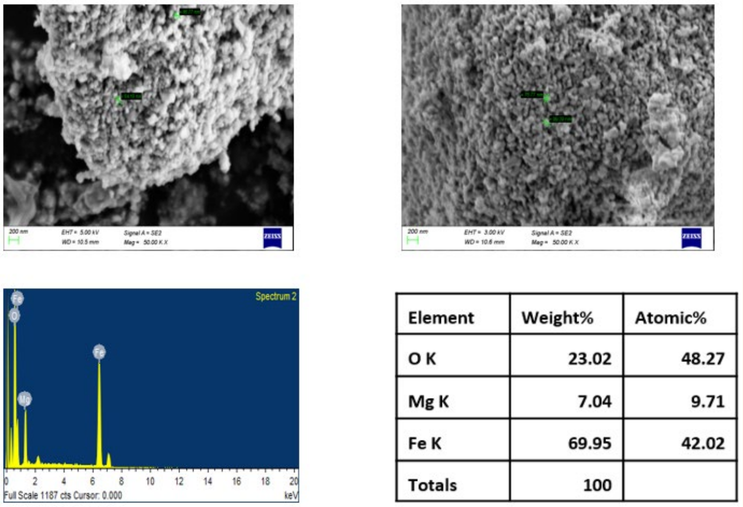
Figure 1: PXRD of: (a) magnetic $MgFe_2O_4$, (b) graphene and (c) MWCNT.

Scanning Electron Microscopy (SEM) and Energy dispersive spectroscopy (EDS)

SEM and EDS were utilized to examine the microscopic appearance and composites of the produced nanoparticles. Fig. 2 (a and b) shows the solution combustion-derived $MgFe_2O_4$ nanoparticles after calcination for 3 hours. The $MgFe_2O_4$ has agglomerated structure even though a well-developed crystalline structure is observed from a close examination of the obtained SEM images. In addition, the nanoparticles got some irregular polygonal geometry, and the surface has a smooth appearance. The nanomaterial composites of the sample are confirmed by EDS Fig. 2(c and d) [16].

Vibrating- Sample Magnetometer (VSM) Studies

The saturation magnetization Ms and coercivity Hc of the $MgFe_2O_4$ sample, which was synthesized at 900°C, are shown in Fig. (3) as a function of applied fields, and another image was shown with the magnification of the VSM graph. It is assumed that the magnetization at 1.5 T corresponds to the saturation magnetization value. As the temperature increases, the coercivity Hc and saturation magnetization Ms rapidly drop. At room temperature, the coercivity Hc is only 38 Oe, the saturation magnetization Ms is 24.30 emu/g, and the remanence MR is 3.5 emu/g. The coercivity is insufficient with such comparatively large particles. This may be related to the fact that there are probably not many domains on the particles [11].



Element	Weight%	Atomic%
O K	23.02	48.27
Mg K	7.04	9.71
Fe K	69.95	42.02
Totals	100	

Figure 2: (a, b) Scanning electron microscopic (SEM) images of $MgFe_2O_4$ at different magnifications (c,d) the EDS and elemental composition.

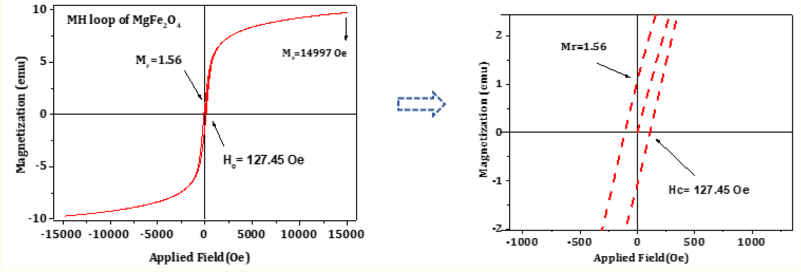


Figure 3: MH loop for $MgFe_2O_4$ nanoparticles at room temperature and magnified image.

EMI shielding characterization study

Shielding effectiveness total (SE_T)

Figure 4(a) shows SE_T as a function of $MgFe_2O_4$ concentration in the nanocomposites at specimens of 3.5 mm thickness. We can observe that the nanocomposite contained 50 wt. % LDPE, 5 wt. % $MgFe_2O_4$, 40 wt. % graphene and 5 wt. % MWCNT (50:5:40:5) contributed the highest shielding efficiency of 33.55 at 10.38 GHz frequency 12.71 dB since synergism was achieved in the combination. The later sample combination had low values. The sample with 5 wt. % of $MgFe_2O_4$ had 32.67 dB, the sample combination with 20 wt. % of $MgFe_2O_4$ had 29.91 dB, the 30 wt. % of $MgFe_2O_4$ had 25.28 dB, the sample with 40 wt. % of $MgFe_2O_4$ had 23.14 dB, while the sample with 50 wt. % of $MgFe_2O_4$ has a total shielding effectiveness of 7.07 dB. Besides 5% ferrite, the shielding was decreased, and the composite failed to give enhanced shielding.

Shielding effectiveness due to Absorption (SE_A)

Figure 4(b) shows the shielding effectiveness due to Absorption. The sample with 50 wt. % LDPE, 5 wt. % $MgFe_2O_4$, 35 wt. % graphene and 10 wt. % MWCNT (50:5:35:10) was having highest shielding efficiency value of 30.82 dB at 10.40 GHz frequency from absorption phenomena. The other samples are the following subsequent shielding values. The sample with 10 wt. % of $MgFe_2O_4$ has 28.49 dB, the sample with 20 wt. % of $MgFe_2O_4$ has 26.81 dB, the sample with 30 wt. % of $MgFe_2O_4$ has 22.15 dB, and the sample with 40 wt. % of $MgFe_2O_4$ has 20.25 dB, the sample with 50 wt. % of $MgFe_2O_4$ has a minor shielding of 7.80 dB, observed from the graph.

Shielding effectiveness due to Reflection (SE_R)

All samples contribute significantly lower values to the total shielding, showing that the reflection mechanism contributed less to the overall shielding than the absorption mechanism. For example, in Fig. 4(c), the sample with 5 wt. % has an SE_R value of 3.21 dB, the sample with 10 wt. % of $MgFe_2O_4$ has a value of 3.09 dB, the sample with 20 wt. % of $MgFe_2O_4$ has a value of 3.12 dB, the sample with 30 wt. % of $MgFe_2O_4$ has a value of 3.01 dB, the sample with 40 wt. % of $MgFe_2O_4$ has a value of 3.0 dB and the sample with 50 wt. % of $MgFe_2O_4$ has a value of 2.68 dB, indicating that 50 wt. % samples will not reflect in EM wave since Graphene and MWCNT were absent and Ferrite was only present.

The shielding effectiveness in the dB scale

All the graphs correspond well with the SE_T graph, indicating that total shielding was contributed from SE_A, and all sample values have similar values to SE_T values. For example, from the bar column graph, it was evident that the sample with 5 % $MgFe_2O_4$ has a maximum SE_T of 33.59 dB, of which 3.11 dB of SE_R and 30.40 dB of SE_A are among the total contribution because of synergism. Likewise, the 10 wt.% of $MgFe_2O_4$ composite has 32.6 dB, among which 3.12 dB from SE_R and SE_A of 29.53 dB. Likewise, all other samples have rest positions, as shown in figure 4(d).

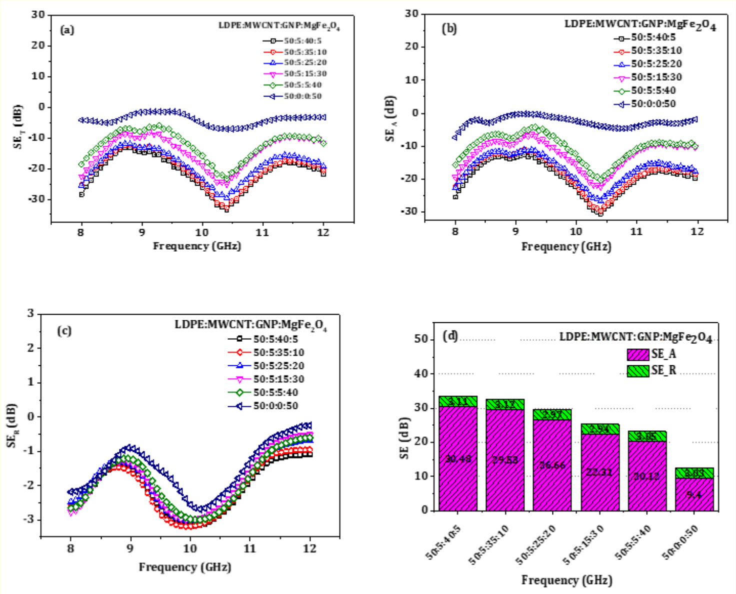


Figure 4: (a) Shielding effectiveness total, (b) Shielding effectiveness due to Absorption, (c) Shielding effectiveness due to Reflection, and (d) Shielding effectiveness in dB.

It may be seen because MWCNT-Graphene-$MgFe_2O_4$ continuity in polyethene nanocomposites is interrupted at increasing weight concentrations, leading to slight ohmic losses. However, the electrical conductivity was successfully obtained at a critical $MgFe_2O_4$ concentration, and the Nano $MgFe_2O_4$ significantly improved EM wave shielding. The high magnetic dipoles, which absorb the magnetic field associated with EM radiation, may cause improved attenuation. Magnetic dipoles in the Ferrite absorb the magnetic field component from the incident EM wave. This has occurred because $MgFe_2O_4$ nanoparticles possess a significant polarisation effect and multiple internal reflections caused by distributed $MgFe_2O_4$ in polymer composite. This will reveal the necessity of the critical concentration of $MgFe_2O_4$ nanoparticles to bring EM wave attenuation synergies. So, our studies give an idea for designing a suitable shielding material with magnetic and conducting filler to achieve shielding material [17].

Dielectric property studies

Any material's electromagnetic wave shielding capability is mainly governed by its dielectric values, such as permittivity and permeability. So, the extract ε', ε'', μ' and μ''. From S-parameters of the NRW algorithm were implicated here [18]. In microwave attenuation by Absorption, electrical conductivity is crucial. To produce the desired electrical conductivity of the nanocomposites, the insulative polymer, LDPE, was mixed with a conductive phase of MWCNT and Graphene. The $MgFe_2O_4$ was also optimally dispersed into this. Several samples such as 50:5:40:5, 50:5:35:10, 50:5:25:20, 50:5:15:30, 50:5:5:40, 50:0:0:50 were prepared and studied to identify the critical concentration which yields superior shielding from the 8 to 12 GHz range. The complex permittivity and permeability plots are shown in figure (5). Permittivity is a complex quantity with real ε' and imaginary ε'' Parts [19].

From figure 5 (a) and (b), the ε' and ε'' parameters of $MgFe_2O_4$ composites are shown. where the ε' reflects the degree of polarisation present in the substance, indicating the substance's capacity to store electrical energy, whereas ε››symbolizes the dissipation of electric energy. These two parameters cause a part of incoming EM waves to reflect by the reflection mechanism. The 50:5:40:5 $MgFe_2O_4$ showed maximum ε' and ε'', indicating the sample combination had the synergetic effect from those critical concentration ratios of LDPE:MWCNT: GNP: $MgFe_2O_4$ ratios. The CNT and graphenes are conductive fillers in the optimized ratio. It forms several conducting network paths; hence the power of EM waves was utilized to polarise the free electrical charges on the surface of conducting fillers such as Graphene and MWCNTs. The ε' represents the polarons present on the Graphene -MWCNT network. The part of energy also gets stored at the interfaces of the MWCNT and Graphene with the other fillers like $MgFe_2O_4$ nanoparticles and polyethene polymer chains. This is also known as the interfacial polarisation of the composite samples. ε'' was more in the 5 % sample, indicating that more electrical charges get trapped and stored, creating a tiny capacitor in the composite. ε'' indicates the attenuation by the absorption mechanism ε'' is high for the 5% $MgFe_2O_4$ combination. Maximum electrical energy loss occurred from the incident EM wave. The dielectric loss is also caused by the dielectric relaxation process and ac conduction loss due to the presence of Graphene and MWCNT, which are superior conductive fillers since $\varepsilon'' \approx \frac{\sigma}{\omega \varepsilon_o}$. The conductive fillers enhance the shielding through reflection phenomena through impedance mismatch and Absorption via huge dielectric loss. After 5% $MgFe_2O_4$, the electrical conductivity decreases since the low conductivity was obtained as $MgFe_2O_4$ was increased and conduction of electrical charges takes place in transverse passion between two consecutive conducting paths. This conducting path is mediated by insulative ferrites creating a capacitive effect at the given frequency. Thus, indicating more ε'' leads to more energy loss in the prepared composite [20].

To evaluate the magnetic loss that occurred from the composite, the μ' and μ'' are essential parameters. Figures 5 (b) and 5(c) represent the real and imaginary permeability associated with the composite. As the concentration of the magnetic filler was increased, the μ' also tended to increase, indicating that the magnetic loss is contributed mainly by the ferrite nanoparticles in the composite, and the rest fillers, MWCNT, Graphene, and LDPE, were non-magnetic. The μ'' of the 5% of $MgFe_2O_4$ was the maximum, displaying the composite maximum's magnetic loss at 10.3 GHz. The magnetic loss of the composite was caused by several eddy current losses, domain wall resonance, hysteresis loss, and natural magnetic resonance events. Similar works were shown in table (2), which had similar results to the present work [21].

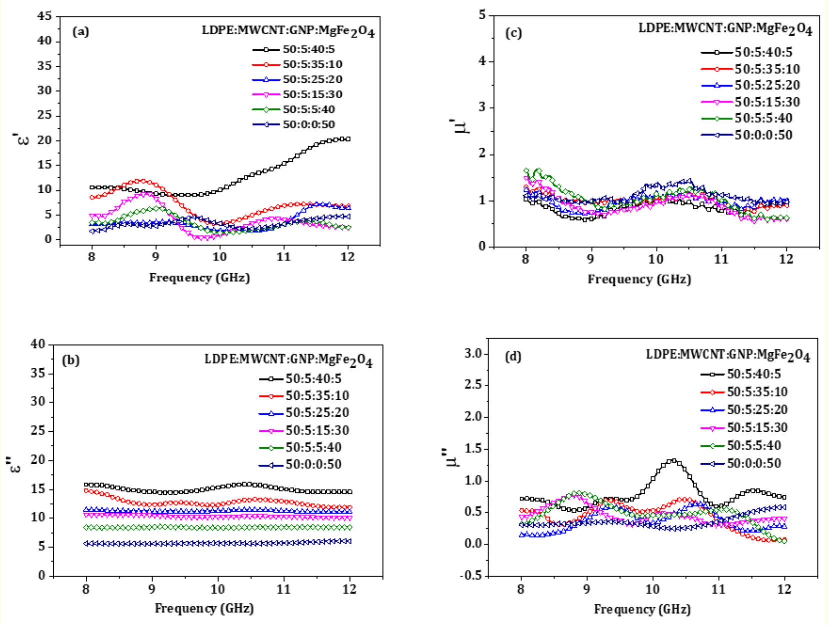


Figure 5: Dielectric properties (a) Real permittivity (ε'), (b). Imaginary permittivity (ε''), (c). Real part of permeability (μ') and (d). Imaginary part of permeability (μ'').

Sl.No	***Material***	***SE***	***Frequency***	***Reference***
1	$Ba_{2-x}La_xCo_{2-x}Mg_xFe_{12}O_{22}$	10.29 dB	8.2–12.4 GHz	[22]
2	$NiMgFe_2O_4$	17 dB	8.2–12.4 GHz	[23]
3	$MgFe_2O_4$/ PTh	40.17 dB	8.2–12.4 GHz	[24]
4	Mg-$ZnFe_2O_4$	35.10 dB	0-20 GHz	[25]
5	Polyaniline/ $Mg_{0.6}Cu_{0.4}Fe_2O_4$	32.8 dB	8.2–12.4 GHz	[26]
6	LDPE: MWCNT: GNP: $MgFe_2O_4$ (50:5:40:5)	33.59 dB	8–12 GHz	**This work**

Table 2: Comparison of EMI performance of MgFe$_2$O$_4$-based composites.

Conclusions

In this study, we tested a new MgFe$_2$O$_4$ nanoparticle, MWCNT, and graphene combination for its synergistic effect on EMI shielding utilizing an LDPE polymer matrix. MgFe$_2$O$_4$ is a soft ferrite that was synthesized by the SCS process. All the constituent materials in the composite were well characterized for its nanoscale. The composite with various concentrations was studied systematically. The matrix (LDPE: MWCNT: GNP: MgFe$_2$O$_4$: 50:5:40:5) was found to get superior ohmic, dielectric, and magnetic losses to obtain combined synergism amongst the two for effective shielding in X-band. An attenuation of 33.59 dB (99.99%) was obtained for 5 % MgFe$_2$O$_4$.

Conflict of interest

The authors declare no conflict of interest.

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