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Article

Electromagnetic Shielding Effectiveness of Glass Fiber/Epoxy Laminated Composites with Multi-Scale Reinforcements

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Abstract: In this study, an experimental investigation has been performed to understand the electromagnetic interference-shielding effectiveness (EMI-SE) of glass fiber/epoxy laminated composites embedded with carbon nanotubes (CNTs) and Fe_3O_4 nanoparticles, reinforced with micro carbon fibers along the thickness direction. Micro carbon fibers were reinforced along the thickness direction between the laminates using an electro-flocking process and a vacuum infusion process used to fabricate the composites. The EMI-SE of the composites was measured in the X-band frequency range (8–12 GHz). The effect of carbon fibers of three different lengths (80 µm, 150 µm, and 350 µm) with two different fiber densities (1000 and 2000 fibers/mm^2) and two different amounts of Fe_3O_4 nanoparticles (0.5 and 1 wt.%) on total SE, absorption, and reflection was investigated. Due to the synergetic effect of Fe_3O_4 nanoparticles, CNTs, and carbon fibers, the final EMI shielding of the composites was mainly dominated by the absorption process. The absorption was more pronounced in the composites of longer carbon fibers with improved electrical conductivity. The presence of Fe_3O_4 nanoparticles also enhanced total SE values with improved magnetic permeability. The composite with micro carbon fibers of 350 µm length and 2000 fibers/mm^2 density with 1 wt.% of Fe_3O_4 nanoparticles showed the maximum value of total SE.

Keywords: electromagnetic shielding; glass/epoxy composites; carbon nanotubes; carbon fibers; iron oxide nanoparticles; electro flocking

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1. Introduction

Electromagnetic shielding (EMS) is often used to cut down emissions or enhance the protection of electronic equipment. In the past, enclosures of electronic equipment were produced using highly conducting metal to accomplish the required shielding. However, design constraints called for reducing the weight of electronic devices with conducting polymers and composites [1–4]. There have been several studies on the use of polymer composites to enhance EMS for the last decade. Researchers investigated the effects of various reinforcements on the EMS of the polymer composites. Jou et al. [5] investigated the electromagnetic shielding effectiveness (SE) of carbon nanotubes (CNTs) embedded polymers. The authors studied the use of CNTs of two different aspect ratios of 500 and 10,000 in liquid crystal polymers (LCPs) and melamine resins (MF). It was identified in their study that at a higher aspect ratio, a greater SE value was observed. Park et al. [6] reported the use of both single-walled and multi-walled CNTs functionalized with the epoxy linkage of the RET polymer on EMS. Through functionalization, they achieved good dispersion of CNTs in the polymer matrix and single-walled CNTs embedded composites showed superior EMS compared to those of multi-walled nanocomposites. Jalali et al. [7] improved the EM shielding especially absorption component of carbon fiber reinforced polymer composites filled with metallic nanoparticles of iron, cobalt, nickel, and iron oxide at higher frequency. The iron nanoparticles of 50 nm improved the total shielding effectiveness of a carbon fibre/polymer nanocomposite up to 15 dB in the 8.2–12.4 GHz range frequency.

Joshi et al. [8] studied graphene nanoribbon (GNR)/Polyaniline (PANI)/epoxy composite film for effective shielding material in the X-band frequency range of 8.2–12.4 GHz. The authors examined the effect of the GNR percentage and the thickness of the film on the shielding effectiveness. Their composites show −40 dB shielding which is sufficient to shield more than 95% of the EM waves in the X Band. Using chemical vapor infiltration, Jia et al. [9] fabricated carbon fiber-reinforced multilayered pyrocarbon-silicon carbide ((PyC-SiC)n) matrix (C/(PyC-SiC)n) composites by means of layer-by-layer deposition of PyC and SiC. The influence of the number of PyC-SiC sequences (n = 1, 2 and 4) on the electrical conductivity and EMI shielding performance of C/(PyC-SiC) n composites were determined. The total SE of the composites increased from 34 to 42 dB in the frequency range of 8.2–12.4 GHz with the increase of PyC-SiC sequences number due to the improvement of electrical conductivity and polarization of the multilayered matrix. Saini and Choudhary [10] fabricated non-covalently functionalized (polyaniline coated) MWCNTs/polystyrene composites with an extremely low percolation threshold (0.12 vol.% MWCNT) using a solution processing route. In their electromagnetic interference studies, due to high conductivity and porosity, these composites exhibited shielding effectiveness of −23.3 dB. This shielding is dominated by absorption (−18.7 dB) with a marginal contribution from reflection (−4.6 dB). Hu et al. [11] recently reinforced Polyvinylpyrrolidone (PVP) functionalized CNTs and KH-550 silane coupling agent treated Fe_3O_4 in lignin-based polyurethane for EMI shielding effectiveness measurements. They reported excellent EMI SE values and they were attributed to the synergistic effects between Fe_3O_4, CNT and lignin. Shuba et al. [12] investigated the electromagnetic response of a composite material consisting of single-walled carbon-nanotubes in a wide range from low-frequency (25–10^7 Hz) up to the infrared region. They reported that the real part of the permittivity of the p-CNT film in the terahertz range is competitive.

Besides adding nanoparticles and short fibers in polymers, researchers also investigated the use of embedding nanoparticles in laminated composites for electromagnetic shielding applications. Chen et al. [13] used copper wire and polyamide filaments as core yarn and later wrapped with polypropylene filaments. They utilized identical yarn for both warp and weft to realize isotropic shielding behavior. Plane-wave shielding properties (SE) of isotropic fabrics and laminated composites were determined at 30–1500 MHz using the coaxial transmission-line method. In their findings, the shielding effectiveness of a single layer was not satisfactory for conventional applications and the multi-layer fabrics deliver sufficient plane-wave shielding effectiveness (20–55 dB) when the wave was normally incident and composite thickness was more than 1.6 mm. Huang et al. [14] used copper wires, stainless-steel (SS) wires, and polyester (PET) filaments to make copper/PET, SS/PET, and copper/SS/PET composite ply yarns. Later authors knitted these ply yarns into electromagnetic shielding fabrics with various knitting-needle densities. Their findings indicated that the copper/SS/PET fabrics demonstrated an SE value of 10 dB, greater than that of the copper/PET or SS/PET fabrics. Tugirumubano et al. [15] recently evaluated electromagnetic interference shielding of bimetal (stainless steel-copper, stainless steel-nickel, and copper-nickel)/carbon prepreg fibers textile composite materials. A combination of stainless steel-copper-carbon fiber with a shielding effectiveness of 131.6 dB was found to be the best for electromagnetic interference shielding. Very recently, Rojas et al. [16] studied electromagnetic shielding effectiveness of serrated and rectangular strips of highly porous carbon nanotube buckypaper (BP) incorporated glass fiber/epoxy resin composites. About 90% of attenuation (8.2–10.8 GHz) was achieved for laminates containing between 0.75 and 2.5 wt.% CNTs. Greater attenuation values of −41.04 dB (>99.99%) at 8.3 GHz were achieved in the reflectivity analysis for laminates with serrated strips at 0.75 wt.% of CNTs.

Although there are many studies reported on electromagnetic shielding effectiveness of laminated composites with several metal/textile weaves and reinforcements, there are no studies reported in the literature on the reinforcing of micro carbon fibers (CF) between the laminates along the thickness direction, in addition to CNTs and Fe_3O_4 nanoparticles in glass fiber/epoxy composites on EMS effectiveness. The through thickness reinforcement of micro carbon fibers generates a three-dimensional electrically conductive network inside the composites. These micro CF can penetrate through the laminates and help to make contact with CNTs in the matrix and micro CF of the neighboring plies [17]. Hence, in this study, novel laminated composites were made by reinforcing micro carbon fibers of three different lengths and two different fiber densities along the thickness direction while embedding CNTs and two different weight percentages of Fe_3O_4 nanoparticles. The effect of above parameters on total SE, absorption SE, and reflection SE in X-band frequency (8–12 GHz) is investigated.

2. Materials and Methods

2.1. Materials

Glass fabric of 10 oz/yd^2 supplied by Fibre Glast Development Corporation, Brookville, OH, USA is used as laminate. Thermoset epoxy of system 2000 resin and 2120 hardener supplied by the same company was used as the matrix. Multi-walled carbon nanotubes (MWCNTs) having an external diameter of 20–30 nm, internal diameter of 5–10 nm, length of 10–30 µm, and purity greater than 95% was supplied by Cheap Tubes Inc., Cambridgeport, VT, USA. In this study, three types of carbon fibers were used. Carbon fibers of length of 80 µm and 150 µm with a diameter of 7–9 µm were supplied by Asbury Graphite Mills, Bloomsbury, NJ, USA. Carbon fibers of length of 350 µm and 7 µm diameter were supplied by E&L Enterprises, Galliano, LA, USA. All fibers consist of 99% carbon content and a mass density of 1.8 g/cc. Fe_3O_4 nano particles of 20 nm (>99.5% purity) were supplied by US Research Nanomaterials, Inc. All lab supplies used for vacuum infusion were procured from Fibre Glast Development Corporation, Brookville, OH, USA.

2.2. Dispersion of CNTs and Fe_3O_4 Nano Particles

Due to the high surface area of MWCNTs and clustering Fe_3O_4 nano particles, they were dispersed properly in the epoxy matrix while making the composites [18]. Based on previous study, for all composite types, 0.3 wt.% of CNTs was used [17]. First, a measured amount of CNTs was hand mixed in the epoxy resin for 10 minutes. Then the mix was placed in an ice bath and a combination of shear mixing and an ultrasonication process was performed for 60 minutes. The shear mixture (IKA model number RW 16 basic overhead stirrer) was set at 600 rpm. Ultrasonicator (Cole-Parmer model CP 750) was set to pulse cycle of 4 seconds on and 9 seconds off. To prevent the generation of excessive heat during the sonication process, the mix was always placed in an ice bath and maintained at room temperature. After 60 minutes, the mix was placed into a vacuum chamber for 60 minutes to remove the entrapped air bubbles. After de-gassing, the measured amount of hardener of the epoxy was added into the mix and hand mixed for 10 minutes. Then, the whole mix was placed again in the vacuum chamber for additional de-gassing. For the composites with Fe_3O_4 nano particles (0.5 and 1 wt.%), the measured powder was added, and ultra-sonicated & shear mixed for another 60 minutes after mixing of CNTs.

2.3. Electro-Flocking of Micro Carbon Fibers

Micro carbon fibers of three different lengths (80 µm, 150 µm, and 350 µm) were reinforced on the laminates along the thickness direction using an electro-flocking process [19]. Figure 1 shows the electro-flocking set up, where carbon fibers were uniformly spread on the bottom plate (electrode) and glass fabric coated using a paint brush with epoxy embedded with CNTs (CNTs and Fe_3O_4 nano particles) was attached to the top plate (electrode). A high electrical potential of 30–80 kV was applied between the electrodes. The potential difference drove the carbon fibers from the bottom plate to top plate and embed into the glass fabric. This process was repeated for 9 layers to make a laminated composite with 10 layers, where the top layer was not flocked. The carbon fiber lengths and fiber densities of 1000 and 2000 fibers/mm^2 were chosen based on our previous study [17].

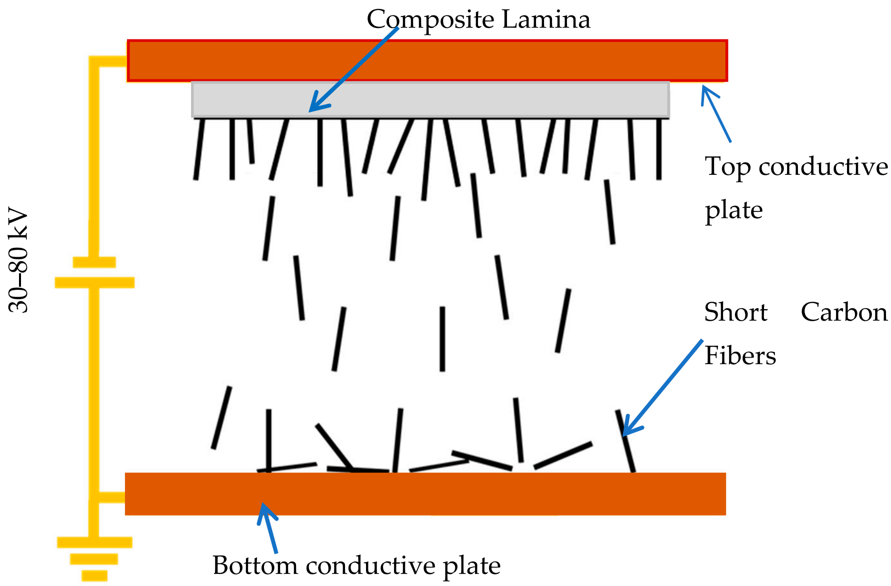

Figure 1. Schematic of wet electro-flocking method.

2.4. Composite Fabrication Using Vacuum Infusion

A standard vacuum infusion process [20,21] was employed to fabricate composites where carbon fiber flocked laminates were first stacked in the vacuum bag. Two vacuum tubes were connected on each end of the vacuum bag. One tube was attached with a vacuum pump for the resin outlet, and the other tube was connected to a resin bucket for the resin inlet. The whole vacuum system was thoroughly sealed using the double-side gum tape. The epoxy mix with CNTs (CNTs and Fe_3O_4 nano particles) prepared in the Section 2.2 was infused into the vacuum bag through the resin inlet with a vacuum pump. During the infusion process, the original orientation of flocked fibers changed, and this could not be avoided. The infused composite was left at room temperature for 48 hours and later specimens of the required dimensions (12.7 mm width × 24.5 mm height × 2.5 mm thickness) were cut.

2.5. Electromagnetic Shielding Effectiveness Measurements

The total shielding effectiveness (SE), and that of absorption and reflection were measured using the set up shown in Figure 2. The set up consisted of a vector network analyzer (VNA-8510C), two standard WR-90 coaxial launchers to guide EM wave, and a copper sample holder in between the launchers. The wave guide launchers used in this study were in the frequency range of 8–12 GHz. The composite samples were fitted in the opening of the sample holder. The sample holder is bolted between the launchers. The incident EM wave had a power of 10 dB. After calibration of the set-up, the wave transmittance loss in dB for conditions of no sample and with sample were collected from VNA and later subtracted to determine the total shielding effectiveness (SE) and those of absorption and reflection. VNA provides the S parameters {S_{11}, S_{22}, S_{12}, and S_{21}} in the frequency range 8–12 GHz. The measurements of S parameters were provided in an incremental frequency of 0.4 GHz within the above frequency range. S_{11} or S_{22} represents a reflection coefficient and S_{12} or S_{21} represents a transmission coefficient of the EM wave. The total SE, SE due to reflection, and SE due to absorption are expressed in terms of S parameters as below:

$$\text{Total SE} = -10\log_{10}(|S_{12}|^2)\ \text{dB} \tag{1}$$

$$\text{SE due to Reflection (SER)} = -10\log_{10}(1 - |S_{11}|^2)\ \text{dB} \tag{2}$$

$$\text{SE due to Absorption (SEA)} = -10\log_{10}(|S_{12}|^2/(1 - |S_{11}|^2))\ \text{dB} \tag{3}$$

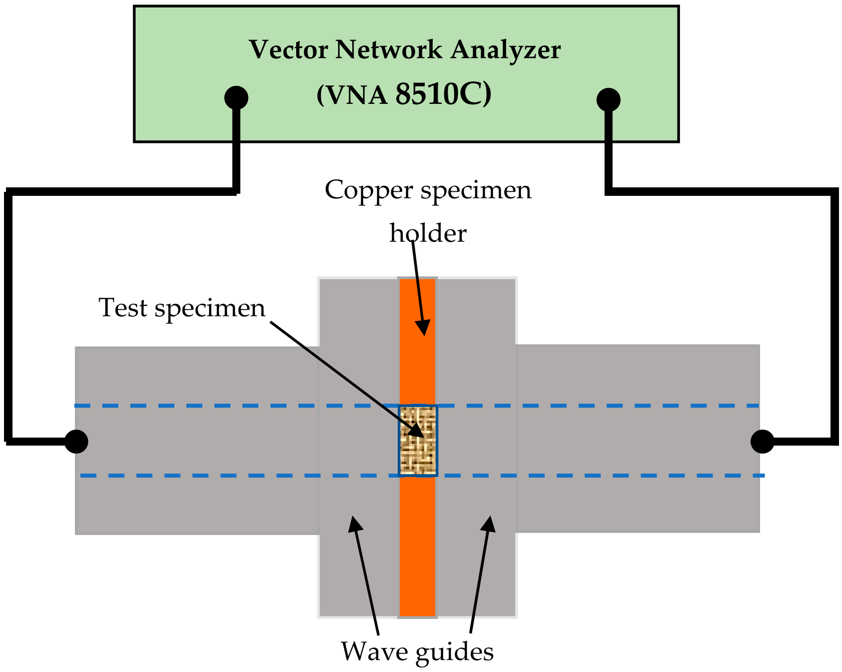

Figure 2. Schematic illustrating the method for measuring electromagnetic wave shielding effectiveness.

3. Results and Discussion

3.1. Composites with No Fe_3O_4 Nano Particles

Figure 3 shows the EMS effectiveness results of (a) total SE, (b) absorption SE (SE_A), and (c) reflection SE (SE_R) for composites having carbon fibers of fiber density of 1000 fibers/mm^2 and 2000 fibers/mm^2 of all three carbon fiber lengths without Fe_3O_4 nano particles. Total SE, SE_A, and SE_R demonstrated higher values as the carbon fiber length increases from 80 µm to 350 µm. A maximum value of 22 dB of total SE is observed at 8.4 GHz for composites of 350 µm carbon fiber with 1000 fibers/mm^2. However, at the same frequency, a maximum value of 14 dB is noted for composites of 80 µm carbon fiber with 1000 fibers/mm^2. For SE_A, composites of 350 µm carbon fiber with 1000 fibers/mm^2 had a maximum value of 15.6 dB at the frequency of 8.4 GHz. However, composites of 80 µm carbon fiber with 1000 fibers/mm^2 has a maximum value of 9.81 dB at the frequency of 8.4 GHz.

It was well reported in the literature that the dielectric property of CNTs and carbon fibers provide improved attenuation of electromagnetic wave when they are reinforced in polymers [22,23]. Moreover, as the carbon fiber length increases, the electrical conductivity of the composite increases significantly as reported in our previous study [17]. The longer carbon fibers penetrate through the laminates during the stacking and vacuum infusion process and thus connect with CNTs in the epoxy of the laminates as well as with carbon fibers in neighboring laminates. Both leakage and tunneling currents increase due to the increased number of physical contacts and the smaller mean distance between the carbon fibers and CNTs. Leakage current regularly has more influence on conductivity than the tunneling current [24]. As it is evident from the Figure 3b, the total SE values have a major contribution from SE_A. Since SE_A is proportional to electrical conductivity of the composite, the former increases with carbon fiber length.

SE, due to reflection, is not a major contributor for total SE, for example, composites of 350 µm carbon fiber length with 1000 fibers/mm^2 at 8.4 GHz, is only 29% of the total SE where the rest is from absorption. The values of SE_R are lower for composites of shorter carbon fiber length (80 µm) for most of the frequencies because of their lower electrical conductivity. SE_R is proportional to conductivity and hence it is expected to have the trend shown in Figure 3c.

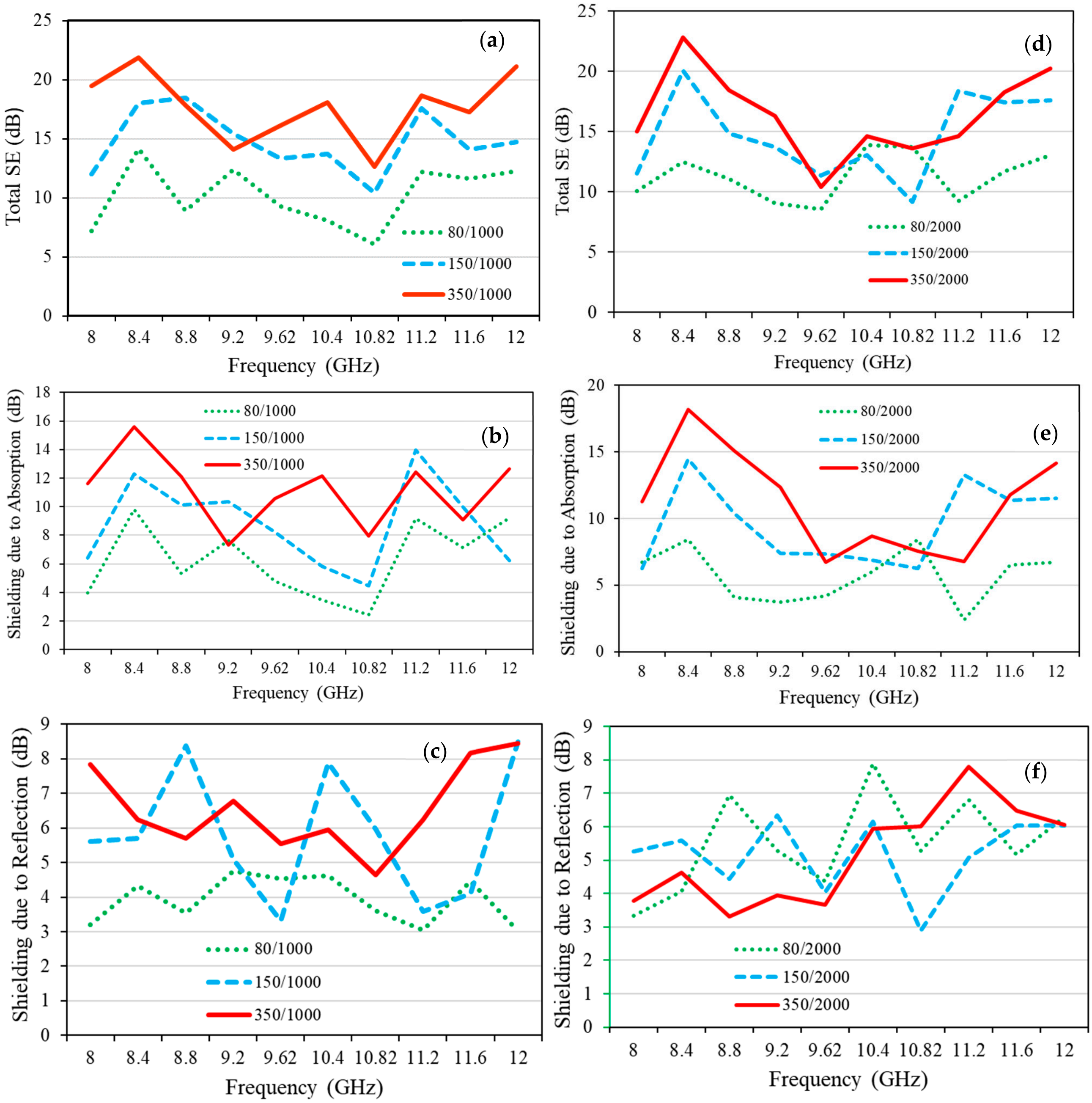

Figure 3. Shielding effectiveness of composites with no Fe_3O_4 nano particles and carbon fibers with fiber density of 1000 fibers/mm^2 (**a**) total SE, (**b**) absorption SE, and (**c**) reflection SE; carbon fibers with fiber density of 2000 fibers/mm^2 (**d**) total SE, (**e**) absorption SE, and (**f**) reflection SE.

When comparison is made between two fiber densities of 1000 fibers/mm^2 and 2000 fibers/mm^2, the total SE, SE_A, and SE_R are slightly higher at certain frequencies for composites of 2000 fibers/mm^2 compared to 1000 fibers/mm^2 as shown in Figure 3d–f. A maximum total SE value of 23 dB is observed at 8.4 GHz for composites of 350 μm carbon fiber length with 2000 fibers/mm^2, which is only a 5% increase compared to composites of same length with 1000 fibers/mm^2. In our previous study, as the carbon fiber density increased from 1000 fibers/mm^2 to 2000 fibers/mm^2, composites conductivity increased by 10 times for all carbon fiber lengths [16]. It is expected to see significant increases in both total SE and SE_A, however the fiber density surprisingly did not make the impact on the measurements. However, for SE_R, composites of 80 μm carbon fiber length at fiber density of 2000 fibers/mm^2 showed significant improvement of 70% when compared to that of

1000 fibers/mm^2. The increase in fiber density from 1000 fibers/mm^2 to 2000 fibers/mm^2 could have compensated the shortcomings of fiber length and improved the electrical conductivity of the composite.

3.2. Composites with Fe_3O_4 Nano Particles of 0.5 wt.%

Shielding effectiveness of composites having Fe_3O_4 nano particles with all carbon fiber lengths and both fiber densities is shown in Figure 4. The addition of Fe_3O_4 nano particles improved both the total SE and SE_A almost twofold when compared with composites having no Fe_3O_4 nano particles, as shown in Figure 3. The combined effect of dielectric loss due to presence of CNTs and carbon fiber; and magnetic losses due to presence of Fe_3O_4 nano particles is clearly evident for the improvement of the total SE and $SE_{A,}$ as shown in Figure 4. The addition of Fe_3O_4 nano particles enhances interfacial polarization between the particles and the surface polarization of the particles, which improves shielding effectiveness [25,26]. Furthermore, the particle size of Fe_3O_4 takes a very important role for the enhancement of total SE. The Fe_3O_4 nanoparticles used in this study have the size of 20 nm (>99.5% pure). For this size range, the orientation of the spins in each domain gets modified themselves and this contributes to the improvement of magnetization [27]. The surface to volume ratio is also high for this size range of nanoparticles which also contributes to the increase of magnetization. The composites of 350 µm carbon fiber length with 1000 fibers/mm^2 demonstrates a maximum total SE value of 41 dB at 8.8 GHz as shown in Figure 4a. This value is about 86% higher than that of same composites with no Fe_3O_4 nano particles. On the other hand, composites of 80 µm carbon fiber length with 1000 fibers/mm^2, showed maximum values of 14.4 dB at frequency of 12 GHz. This value is about 15% higher than that of no Fe_3O_4 nano particles shown in Figure 3a. The combined effect of higher electrical conductivity due to longer carbon fibers; and improved magnetic permeability due to addition of Fe_3O_4 nano particles increases the total SE value significantly for composites of carbon fiber density of 1000 fibers/mm^2. It is reported in the literature that the catalyst growth of crystalline graphite during carbonization process can be enhanced by adding Fe_3O_4 nano particles which might have further improved the composite's conductivity [28].

Like composites with no Fe_3O_4 nano particles, the contribution of SE_A to the total SE value is significant for composites with Fe_3O_4 nano particles as shown in Figure 4b. A maximum value of 37dB is noticed for composites of 350 µm carbon fiber length with 1000 fibers/mm^2 at a frequency of 8.8 GHz. This is about 84% of the total SE value for this composite type at the same frequency of 8.8 GHz. For the same composite type, SE_A with Fe_3O_4 nano particles is 3 times higher than that of no Fe_3O_4 nano particles at frequency of 8.8 GHz. Since SE_A is proportional to both conductivity and permeability of composite, the reported values follow the expected trend with the addition of Fe_3O_4 nano particles. However, the addition of Fe_3O_4 nano particles did not improve the SE_A for composites of 80 µm carbon fiber length with 1000 fibers/mm^2 when compared to other two carbon fiber length cases. For these composite types with Fe_3O_4 nano particles, about 20% improvement of SE_A is observed when compared to that of no Fe_3O_4 nano particles at the frequency of 12 GHz shown in Figure 3b.

For SE_R, the addition of the Fe_3O_4 nano particles reduced the values of composites considerably, as shown in Figure 4c, when compared to those with no Fe_3O_4 nano particles, shown in Figure 3c. For example, the value of SE_R decreased by 25% with the addition of Fe_3O_4 nano particles for composites of 350 µm carbon fiber length with 1000 fibers/mm^2 at the frequency of 12 GHz. This is expected as the SE_R values are inversely proportional to magnetic permeability [29]. The addition of Fe_3O_4 nano particles increases the permeability values as reported in the literature [25,26]. The decrease of SE value due to reflection is much lower for composites of 80 µm carbon fiber length with 1000 fibers/mm^2, where the value reduced by 97% with addition of Fe_3O_4 nano particles for the frequency value of 9.2 GHz.

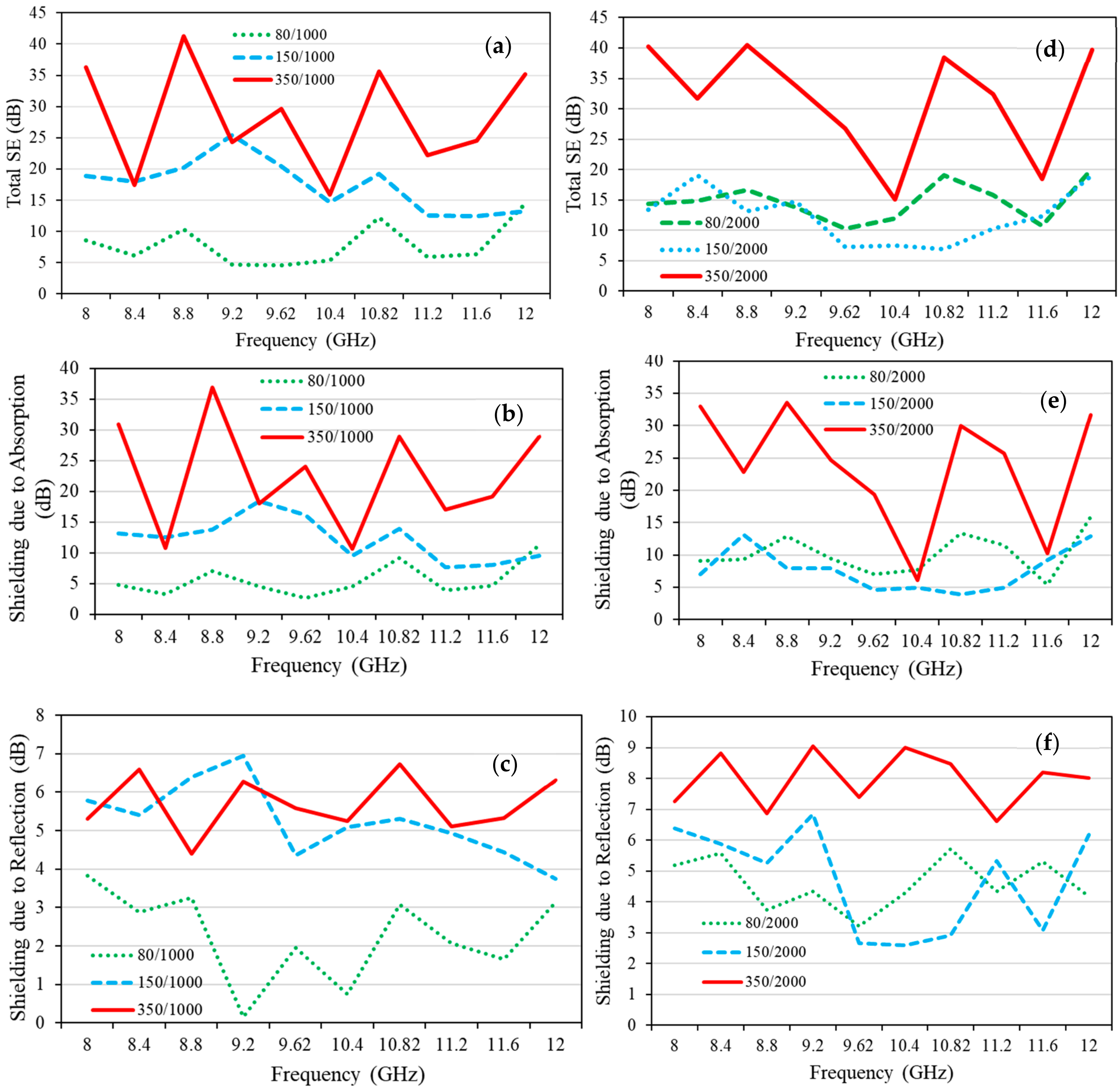

Figure 4. Shielding effectiveness of composites with Fe_3O_4 nano particles of 0.5 wt.% and carbon fibers with fiber density of 1000 fibers/mm^2 (**a**) total SE, (**b**) absorption SE, and (**c**) reflection SE; carbon fibers with fiber density of 2000 fibers/mm^2 (**d**) total SE, (**e**) absorption SE, and (**f**) reflection SE.

The fiber density once again did not demonstrate significant influence on total SE and SE due to absorption even with the addition of Fe_3O_4 nano particles similar to composites with no Fe_3O_4 nano particles. However, it again played some improvement in SE_R at higher fiber density of 2000 fibers/mm^2. For composites of 350 μm carbon fiber length, an increase of 47% of SE_R at 9.2 GHz is noticed when the fiber density increased from 1000 fibers/mm^2 to 2000 fibers/mm^2. The improvement is 27 times for composite of 80 μm carbon fiber length at the same frequency of 9.2 GHz when the fiber density increased from 1000 fibers/mm^2 to 2000 fibers/mm^2. This once again reinforces the fact that higher density of 2000 fibers/mm^2 compensates for the shortcoming of being shorter a carbon fiber length of 80 μm.

3.3. Composites with Fe_3O_4 Nano Particles of 1.0 wt.%

Figure 5 shows the total SE, SE_A, and SE_R for composites with Fe_3O_4 nano particles of 1.0 wt.% and at two different fiber densities. The addition of 1.0 wt.% of Fe_3O_4 nano particles almost increased the total SE and SE_A values of all composites of fiber density of 1000 fibers/mm^2 by two times when compared to those of 0.5 wt.% of Fe_3O_4 nano particles. For example, composites of carbon fiber length of 350 μm with fiber density of 1000 fibers/mm^2 showed 125% improvement in total SE value at 1.0 wt.% of Fe_3O_4 nano particles compared to that of composites with 0.5 wt.% of Fe_3O_4 nano particles at a frequency of 8 GHz. This improvement is primarily due to the improvement in magnetic permeability of the composite. Moreover, the extra amount of Fe_3O_4 nano particles enhanced both the interfacial polarization between the particles and the surface polarization of the particles. The increase in total SE value is more pronounced for composites of carbon fiber length of 80 μm with fiber density of 1000 fibers/mm^2, where an improvement of 155% is noticed at 1.0 wt.% of Fe_3O_4 nano particles compared to composites with 0.5 wt.% of Fe_3O_4 nano particles at the frequency of 8.4 GHz.

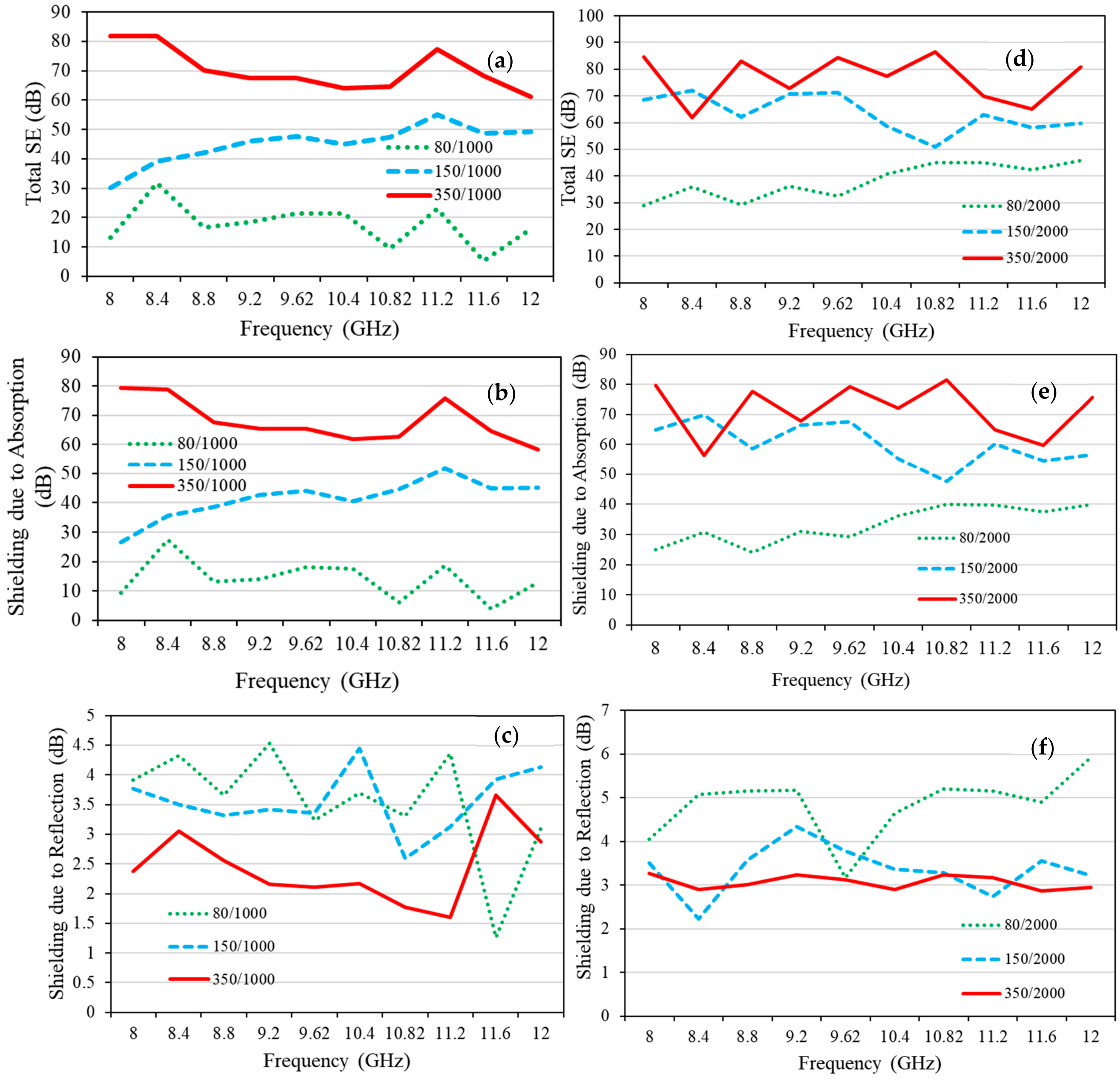

Figure 5. Shielding effectiveness of composites with Fe_3O_4 nano particles of 1.0 wt.% and carbon fibers with fiber density of 1000 fibers/mm^2 (**a**) total SE, (**b**) absorption SE, and (**c**) reflection SE; carbon fibers with fiber density of 2000 fibers/mm^2 (**d**) total SE, (**e**) absorption SE, and (**f**) reflection SE.

Like previous composite types, the SE_A is a major contributor of the total SE value. For composites with a fiber density of 1000 fibers/mm^2 at 1.0 wt.% of Fe_3O_4 nano particles, SE_A contributes almost 97% of the total SE value. This composite scenario is the best case for several defense related applications where SE due to absorption is a major requirement of electromagnetic shielding. When comparison is made to check the impact of a higher amount of Fe_3O_4 nano particles on SE value due to absorption, composites of carbon length of 350 μm with fiber density of 1000 fibers/mm^2 showed 156% improvement at 1.0 wt.% of Fe_3O_4 nano particles compared to that of composites with 0.5 wt.% of Fe_3O_4 nano particles at the frequency of 8 GHz. An improved situation of SE_A is observed for composites of shorter carbon length of 80 μm with fiber density of 1000 fibers/mm^2, where an improvement of 195% is noticed at 1.0 wt.% of Fe_3O_4 nano particles compared to that of composites with 0.5 wt.% of Fe_3O_4 nano particles at the frequency of 8 GHz. When compared to case of composites of 0.5 wt.% of Fe_3O_4 nano particles (Figure 4b) against the composites with no Fe_3O_4 nano particles (Figure 3b), the additional amount of 1.0 wt.% of Fe_3O_4 nano particles (Figure 5b) against the composites of 0.5 wt.% of Fe_3O_4 nano particles (Figure 4b) showed much higher improvement in total SE values and SE_A values.

With the increase of Fe_3O_4 nano particles to 1.0 wt.%, the SE_R for composites of fiber density of 1000 fibers/mm^2 reduces significantly when compared to those of 0.5 wt.% of Fe_3O_4 nano particles for both carbon fiber lengths of 150 μm and 350 μm. The presence of more Fe_3O_4 nano particles further increases the magnetic permeability, which in turn reduces the SE_R as expected. For example, composites of carbon fiber length of 350 μm with fiber density of 1000 fibers/mm^2 showed a reduction of 280% in SE_R compared to those of 0.5 wt.% of Fe_3O_4 nano particles at the frequency of 10.82 GHz. However, for composites of carbon fiber length of 80 μm with fiber density of 1000 fibers/mm^2 showed 213% increase in SE_R at 1.0 wt.% of Fe_3O_4 nano particles compared to those of 0.5 wt.% of Fe_3O_4 nano particles at the frequency of 9.2 GHz. The reasons for such increase at this frequency are not clear and needs further investigation.

At the higher fiber density of 2000 fibers/mm^2, composites of carbon fiber lengths of both 80 μm and 150 μm showed improvements in total SE values and SE_A when compared to those of 1000 fibers/mm^2. The higher fiber density of carbon fibers demonstrates its impact in the presence of 1.0 wt.% of Fe_3O_4 nano particles to improve the shielding effectiveness by enhancing the interfacial polarization between the particles and the surface polarization of the particles. For example, composites of carbon fiber length of 150 μm at fiber density of 2000 fibers/mm^2, showed a 50% improvement in total SE value compared to that of fiber density of 1000 fibers/mm^2 at the frequency of 9.62 GHz. In the case of composites of carbon fiber length of 80 μm, a higher percentage of improvement of 185% was noticed for total SE value at the fiber density of 2000 fibers/mm^2 compared to those of fiber density of 1000 fibers/mm^2 at the frequency of 12 GHz. Composites of carbon fiber length of 350 μm and fiber density of 2000 fibers/mm^2 along with 1.0 wt.% of Fe_3O_4 nano particles showed the highest total SE and SE_A values compared to all composite types. The longer carbon fibers penetrate the neighboring glass laminates thus providing higher electrical conductivity as found in our previous study [17]. The higher amount of 1.0 wt.% of Fe_3O_4 nano particles provides greater magnetic permeability as reported in the literature [24,25]. Hence the synergetic effect of carbon fibers and Fe_3O_4 nano particles resulted in the highest total SE and SE_A values for this composite type. The increase of carbon fiber density from 1000 to 2000 fibers/mm^2 has no significant impact on the SE_R.

4. Conclusions

An experimental characterization was performed to investigate the effect of carbon nanotubes, micro carbon fibers, and Fe_3O_4 nano particles on electromagnetic shielding effectiveness in the X-band frequency range (8–12 GHz). Overall, the Fe_3O_4 nano particles have a major influence on the total shielding effectiveness as well as on the absorption. The major outcomes of this study are:

- SE value of absorption is a major contributor for the total SE value for all composite types.

- Carbon fibers (350 µm long fibers, the fiber density of 2000 fibers/mm^2) and Fe_3O_4 nano particles (1.0 wt.%) provided the highest total SE and SE_A.
- The increase of fiber density of carbon fibers from 1000 to 2000 fibers/mm^2 did not have considerable impact on total SE and SE value due to absorption in composites with no Fe_3O_4 nano particles and with 0.5 wt.% of Fe_3O_4 nano particles.
- At 1.0 wt.% of Fe_3O_4 nano particles, and at a higher fiber density of 2000 fibers/mm^2, composites of carbon fiber lengths of both 80 µm and 150 µm showed significant improvements of total SE values and SE values due to absorption when compared to those of 1000 fibers/mm^2.

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