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RESEARCH ARTICLE

Influence of Fe_3O_4 and Carbon Black on the Enhanced Electromagnetic Interference (EMI) Shielding Effectiveness in the Epoxy Resin Matrix

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Abstract

The present study aims to investigate the shielding properties of the electromagnetic interference of polymer nanocomposites with different weight percentages of magnetite nanoparticles and cost-effective carbon black nanoparticle (CBN) on different thicknesses. X-ray diffraction test, Raman spectroscopy, the scanning electron microscopy, and the transmission electron microscope analysis were used for investigating the crystallographic structure, morphology and microstructure of the material. The nanocomposites were successfully prepared using a simple mixing and casting. Their shielding efficiency was measured by a vector network analyzer (VNA) in the frequency range of 8.2 ~ 12.4 GHz. The maximum total shielding efficiency was 36.6 dB at 8.2 GHz for a weight percentage of 15% Fe_3O_4 composite and 50% CBN (0.7 mm thickness). The results showed that with an increase of nanocomposite thickness, there is a shift of absorption shielding efficiency peak toward a higher frequency. In addition, nanocomposites had the greatest shielding effectiveness in the low-frequency range. It was found that the proper combination of electrical and magnetic losses causes excellent wave absorption. These findings indicated that epoxy resin with a combination of optimal weight percentage of magnetite and carbon black nanoparticle can be used as a suitable shielding in low thickness.

Keywords Electromagnetic · Shielding properties · Magnetite nanoparticles · Carbon black nanoparticles · Reflective loss

Introduction

Nowadays, given the increasing use of equipment utilizing electromagnetic radiation in their operation, the effects of these waves on the human body or the efficiency of various devices have been investigated [1]. Exposure to electromagnetic fields has been listed as a potential carcinogen for humans by the International Agency for Research on Cancer (IARC) (group 2B) [2]. Various studies on the effect of exposure to electromagnetic fields include the risk of cancer [3], electromagnetic hypersensitivity [4], cognitive effects [5], neurological disease [6], Central Nerve System [7]and reproductive disorders [8]. The control measures to protect workers against electromagnetic fields (radio waves) include following the instructions for working with electromagnetic fields, compliance with permissible exposure limits, reducing exposure time, using technical and engineering methods as well as the appropriate personal protective equipment [9]. Among these, the application of technology and engineering methods, such as using shields is one of the methods to prevent or reduce exposure [10]. Some common types of shields against waves include metals, carbon, ceramics, cement, polymers, hybrids, and composites [11].

Polymer nanocomposites are another class of electromagnetic shields that, in addition to being used to control the exposure of employees, are also used in electrical and electronic equipment and prevent the electromagnetic interference (EMI) [12]. These materials have good conductivity, lower density, and higher corrosion resistance compared with metals [13]. Today, with the advent of nanotechnology, using nanocomposite shields to prevent EMI is a new approach that has recently been considered by researchers [14]. Carbon compounds are one of the best compounds to be used as absorbents due to their good electrical and thermal conductivity [15]. Carbon black nanoparticle (CBN) is an amorphous form of

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carbon commercially obtained by thermal decomposition or oxidation of hydrocarbons. Carbon black is widely used in the industry as a filler to modify the electrical and optical properties of dispersed materials [16]. Black carbon is used as a conductive filler and reinforcing material for polymer matrices because of its unique properties, which include a wide surface area, good chemical stability, excellent electrical conductivity, and low cost [17]. Ferromagnetic particles with a diameter less than the critical diameter (Dc) exhibit single-zone behavior. The Dc is the maximum diameter of the rod which is quenched to produce 50% martensitic. In single-zone particles, magnetic losses are generally caused by spin rotation, while for larger particles (multi-zone) these losses are mostly due to the movement of the zone walls [18]. Due to the proper attenuation of waves in single-zone ferromagnetic materials, these materials have been widely used in electromagnetic wave absorption applications [19]. Magnetic iron oxide or magnetite (Fe_3O_4) is one of the ferromagnetic materials with relatively high magnetic properties, which has an excellent ability to absorb electromagnetic waves. Fe_3O_4 has been investigated as an absorber due to its permittivity and permeability properties, appropriate saturation magnetization and high curie temperature [20].

One of the constant challenges in designing and constructing electromagnetic shields is to achieve a shield with high absorption efficiency in the wide frequency band. The purpose of this research was to investigate the efficiency of electromagnetic interference shielding using cost-effective nanocomposites containing carbon black and iron oxide nanoparticles (as fillers) in the epoxy resin matrix and the frequency range of 8.2 to 12.4 GHz.

Materials and methods

Materials

In this study, an epoxy resin was used as a matrix, and spherical nanoparticles CBN and Fe_3O_4 were used as a filler (98%, Electrical conductivity: 0.30 Ω.cm, spherical particles of size: 25–50 nm). Epoxy resin (EI-403) is produced by Mokarrar Engineering Material Co. Nanomaterials were supplied from Us Research Nanomaterial.

CBN properties

Scanning electron microscope (SEM) (model S-4160 -Hitachi, Japan), Raman spectroscopy (532 nm laser beam, Takram P50C0R10 model, Teksan Co., Iran), and X-ray diffraction (XRD) (Cu Kα λ = 0.154 nm, Ultima IV model, Rigaku Co., Japan) pattern results of CBN are shown in Fig. 1.

Fe_3O_4 properties

Figure 2 shows SEM image, transmission electron microscope (TEM) image (model Zeiss EM10), Raman spectroscopy, and XRD results of Fe_3O_4.

Nanocomposite synthesis

In this study, the desired nanocomposites were prepared using the mixing and casting melt mixing method with different filling percentages in epoxy resin matrix with a thickness of 0.7 mm. The Fe_3O_4 and Carbon black powders as filler were added to the epoxy resin at different weight percentages (from 15 to 50) and then, they were adequately blended. The nanoparticles weight percentage of the composites is presented in Table 1. Mixing was performed at 60 rpm for 15 min for all the samples. The constituents of nanocomposites in each case were first mixed manually based on the weight percentage and then mixed by the extruder (Brabender Co. produced the 25 DS twin-screw extruder, which has a screw diameter of 25 mm and a length-to-diameter ratio of 40). The speed of movement of the screws was adjusted to 60 rpm. In the end, each mixture was poured into glass casts and cured at 100 C for 2 h [22]. The hole with an outer diameter of 6.9 mm and an inner diameter of 3 mm [23] was used for measuring EM properties of the nanocomposites.

To remove the moisture in the nanoparticles, they were placed in a dryer at 90 °C for 2 h [24].

EMI Shielding Efficiency Analysis

The electromagnetic properties of the fabricated nanocomposites were measured by an E8362B vector network analyzer (VNA) (E8362B model, Agilent Co., USA) in the frequency range of 8 to 12.5 GHz (X) based on the transmission/reflection method. To this end, the sample was placed in a coaxial waveguide with the same internal and external diameters as the sample. The values of the microwave permeability and permittivity were extracted based on the Nicholson-Rose-Weir method using scattering indices. The amount of EM wave attenuation of the encountering wave is expressed in decibels by assessing the total shielding efficiency (SE_T). When the SE_T value reaches 20 dB, it can reduce 99% of the EM waves [25]. The SE_T of the fabricated nanocomposites was calculated according to Eqs. (1, 2 and 3) [26–29].

$$SE_T(dB) = SE_A(dB) + SE_R(dB) + SE_M(dB) \quad (1)$$

$$SE_A = 20log\left|e^{\frac{d}{\delta}}\right| \quad (2)$$

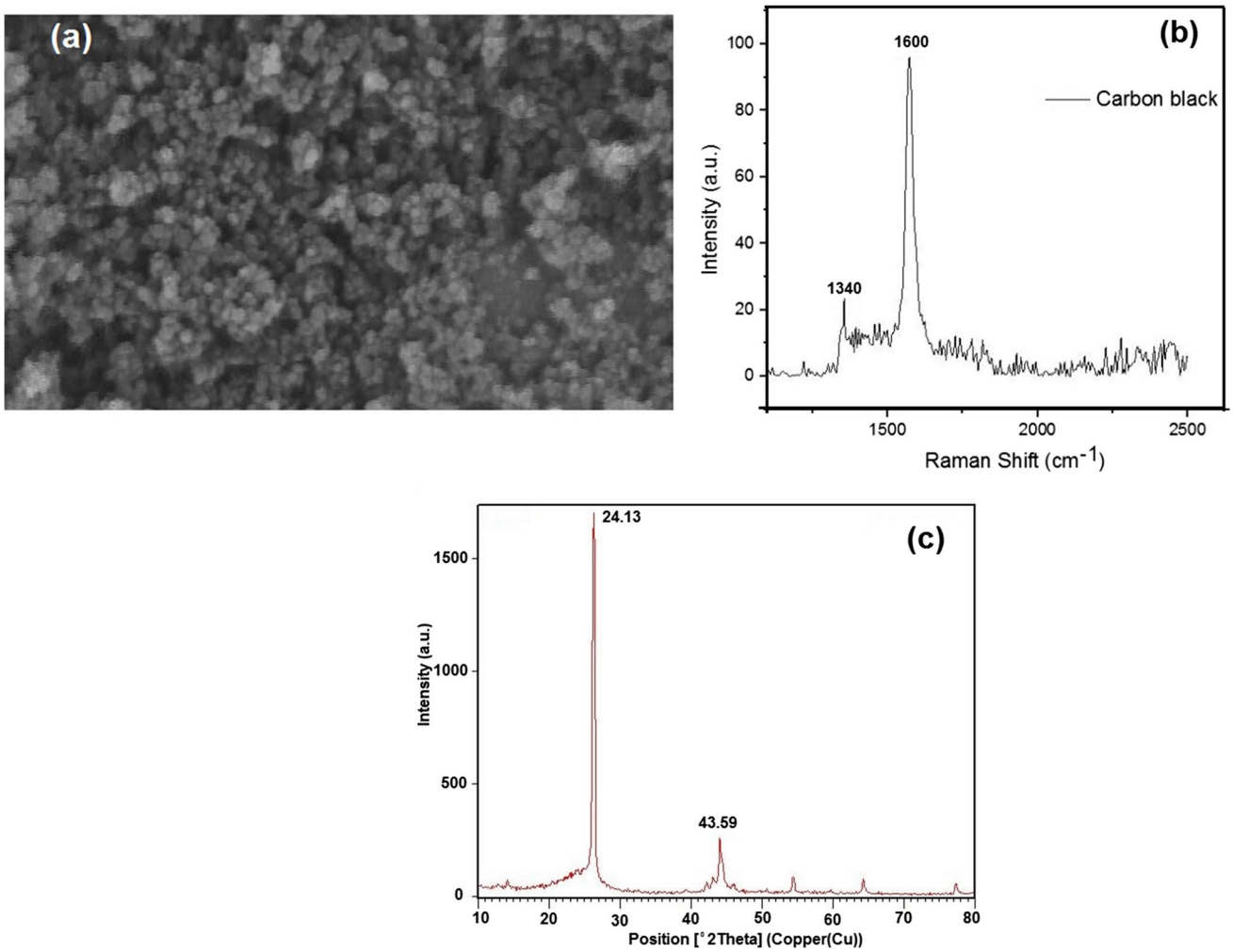

Fig. 1 (a) SEM image, (b) Raman spectroscopy, and (c) XRD results of CBN

$$SE_R = \mathrm{R_L} = 20log\left|\frac{Z_{in}-1}{Z_{in}+1}\right| \tag{3}$$

where SE_R represents shielding efficiency (SE) via reflection, absorption shielding efficiency (SE_A) represents SE duo to absorption and $\mathrm{SE_M}$ represents SE via multiple reflections. Z_{in} is the normalized input impedance at free space and material injector. d is the thickness of composite, and δ is the skin depth, which is shown in Eq. (4) [27, 30]. $\mathrm{SE_M}$ can be neglected if $\mathrm{SE_A}$ is more than 10dB.

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}} \tag{4}$$

Reflective loss (RL) is a part of the electromagnetic wave energy reflected towards the source when it comes in contact with the absorber. RL depends on the ratio of the electromagnetic wave impedance to the absorbent impedance and the conductivity and permeability of the shielding material. An RL value equal to 10 dB means that 90% of the emitted wave is absorbed by the absorbent [31]. The transmission line concept, which can be summarized by Eqs. 5 and 6, can be used to determine RL values [32].

$$\mathrm{R_L} = 20log\left|\frac{Z_{in}-1}{Z_{in}+1}\right| \tag{5}$$

$$\mathrm{Z_{in}} = \sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left[j\left(\frac{2\pi fd}{c}\right)\right]\sqrt{\varepsilon_r\mu_r} \tag{6}$$

where, ε_r and μ_r are the complex permittivity and magnetic permeability of the nanocomposite, respectively, f is the frequency, and c is the light speed in open space.

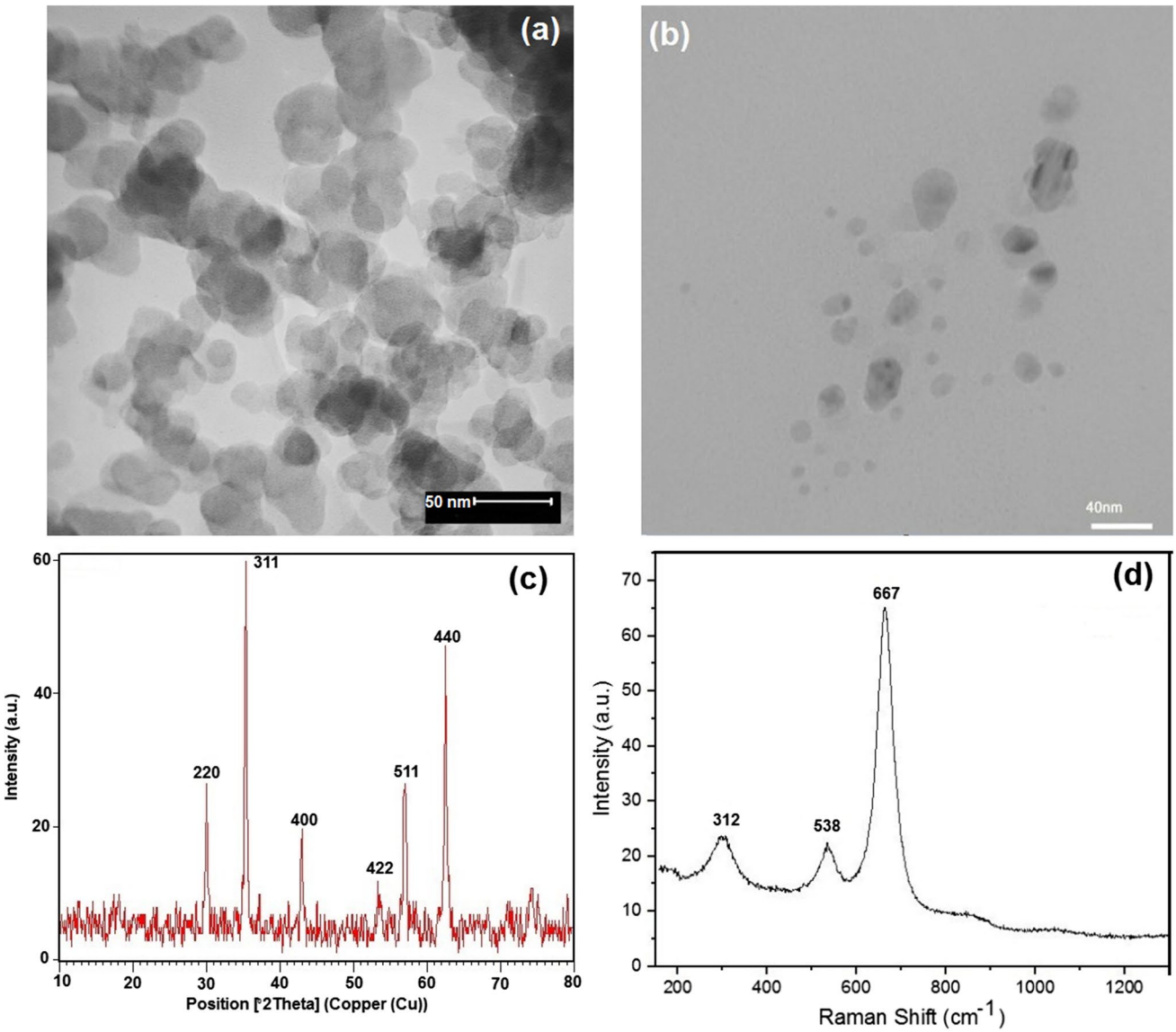

Fig. 2 Microstructure investigation of Fe_3O_4 (a) SEM image, (b) TEM image, (c) XRD pattern, and (d) Raman spectroscopy

Table 1 Carbon black and Fe_3O_4 weight percentages in making nanocomposite samples

Nanocomposites	FC1	FC2	FC3	FC4
Carbon black Nanoparticles (CBN)	15%	15%	15%	50%
Fe_3O_4 Nanoparticles	15%	30%	50%	15%

Results and discussion

Microscopy analysis

While the high electrical conductivity of Fe_3O_4 and CBN is one of the main reasons for the high EMI shielding efficiency of the composites fabricated in the study, the distribution of CBN is also significant. The uniform distribution of Fe_3O_4 and CBN particles in the matrix established a conducting network that provides the optimum EMI shielding possible. In Fig. 3, the obtained SEM micrographs of FC1 to FC4 composites are shown. The images show that in prepared composites, especially for FC1 and FC2 specimens, the mixed nanoparticles form an electron transfer network inside the samples.

The phase composition, chemical structure, and morphology of the composites were characterized by XRD, as shown in Fig. 4. The XRD result indicated both high crystallinity and purity of the Fe_3O_4/CBN composites.

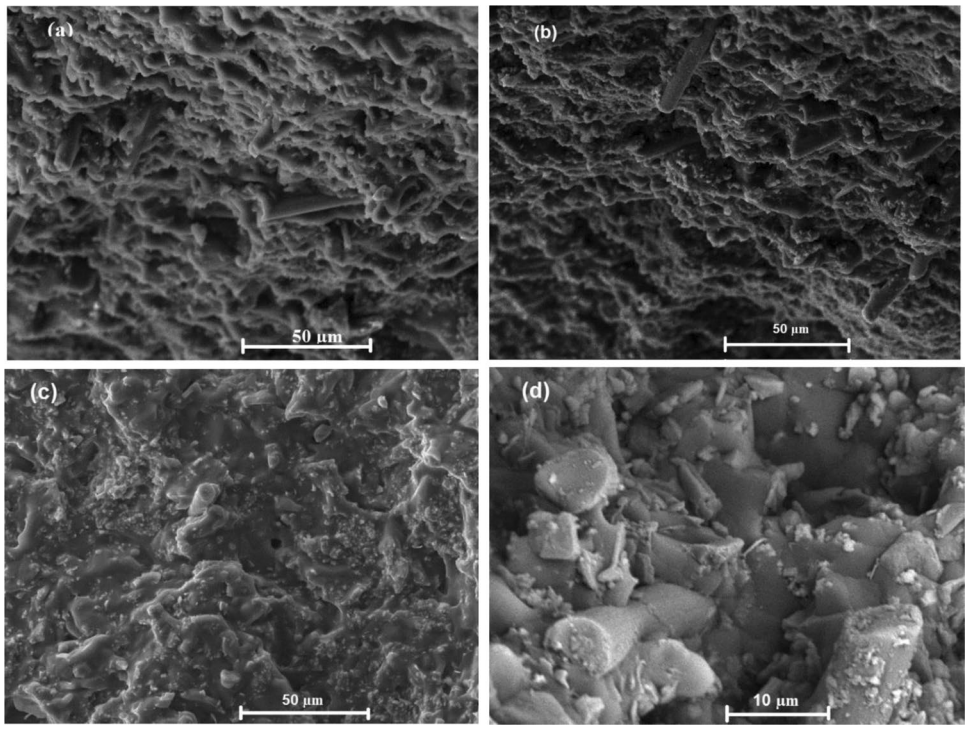

Fig. 3 SEMM images of (a) FC1, (b) FC2, (c) FC3, and (d) FC4 composites

EMI analysis

In the study, the EMI shielding effectiveness of different weight percentages of magnetite and CBNs was investigated. In the X-band, the electromagnetic shielding effectiveness of these materials was investigated (8.2–12.4 GHz). All the samples had a thickness of 0.7 mm. The SE_T, SE_R, and SE_A were determined using the scattering parameters obtained from the VNA. The efficiency of the fabricated samples' SE_A, SE_R, and SE_T as a function of frequency in a thickness of 0.7 mm is shown in Fig. 5.

According to Fig. 5, in all the samples, SE_T had a higher efficiency at lower frequencies compared with higher frequencies. Moreover, the value of SE_R at high frequencies was higher than that at low frequencies.

Figure 5, reveals that the mean SE_A value for samples FC1, FC2, FC3, and FC4 was 11.21 dB, 11.82 dB, 13.28 dB, and 11.85 dB, respectively. The mean SE_R value for samples FC1, FC2, FC3, and FC4 was -16.09 dB, -16.63 dB, -6.81 dB, and -17.61 dB, respectively. Moreover, the lowest and highest SE_T values in the prepared nanocomposites were 20.10 dB in FC3, and 36.6 dB in FC4, respectively. Therefore, as was shown in Fig. 5d, 50% weight percentage of carbon black in FC4 nanocomposite case more shielding effectiveness because of rising dielectric loss caused by electrical conduction in the specimen. On this basis, this composite has mean higher reflection loss compared with other composites, for instance, the composite has reflection loss -24.9 dB at 8.2 GHz, which was able to reduce over 99% of EM waves [33].

The sample FC3 contained a weight percentage of 50% magnetite and 15% carbon black had a higher mean absorption loss with a maximum of 13.47 dB at 11.89 GHz than the other samples. Therefore, adding magnetite nanoparticles rise in magnetic loss and it case more absorption loss. While, the sample FC3 had less reflection in mean-6.81 dB, because of the low weight percentage of carbon black (15%), which leads to lower electrical conductivity and less dielectric loss in the composite.

Because of the importance of the absorption efficiency of the prepared shields, the SEA of the nanocomposites was predicted using Eq. (2) in various thickness. The results of the prediction was shown in Fig. 6.

According to Fig. 6, in all four samples, the thickness of 3.2 mm had the highest SE_A with an average of 14.84 dB. The results showed that the maximum value of SE_A was 15.89 dB in the thickness of 3.2 mm, which appeared at a frequency of 11.89 GHz (sample FC3) and the minimum value of SE_A was 10.60 dB in the thickness of 0.7 mm at a frequency of 10.04 GHz (sample FC1). With an increase of nanocomposite thickness, there is a shift of SE_A peak toward higher frequency [Fig. 6 (d), 6(f) and 6(h)]. Knowing the SE values will help choose the right sample thickness for shielding.

Table 2 compares the Fe_3O_4/CBN (FC4) composite material to other commonly used absorbents. Compared with the reduced graphene oxide (rGO)–Fe_3O_4 (rGO:IO)

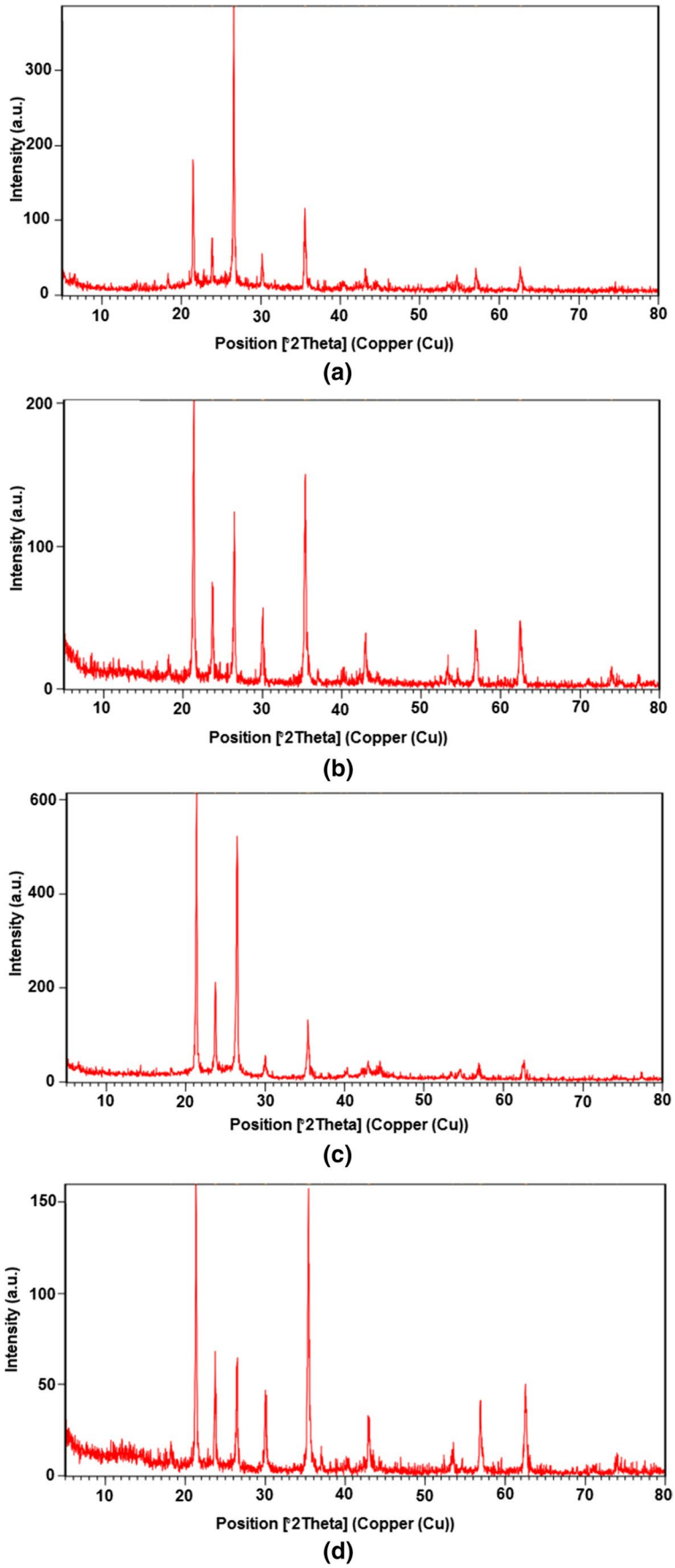

Fig. 4 XRD patterns of (a) FC1, (b) FC2, (c) FC3, and (d) FC4 composites

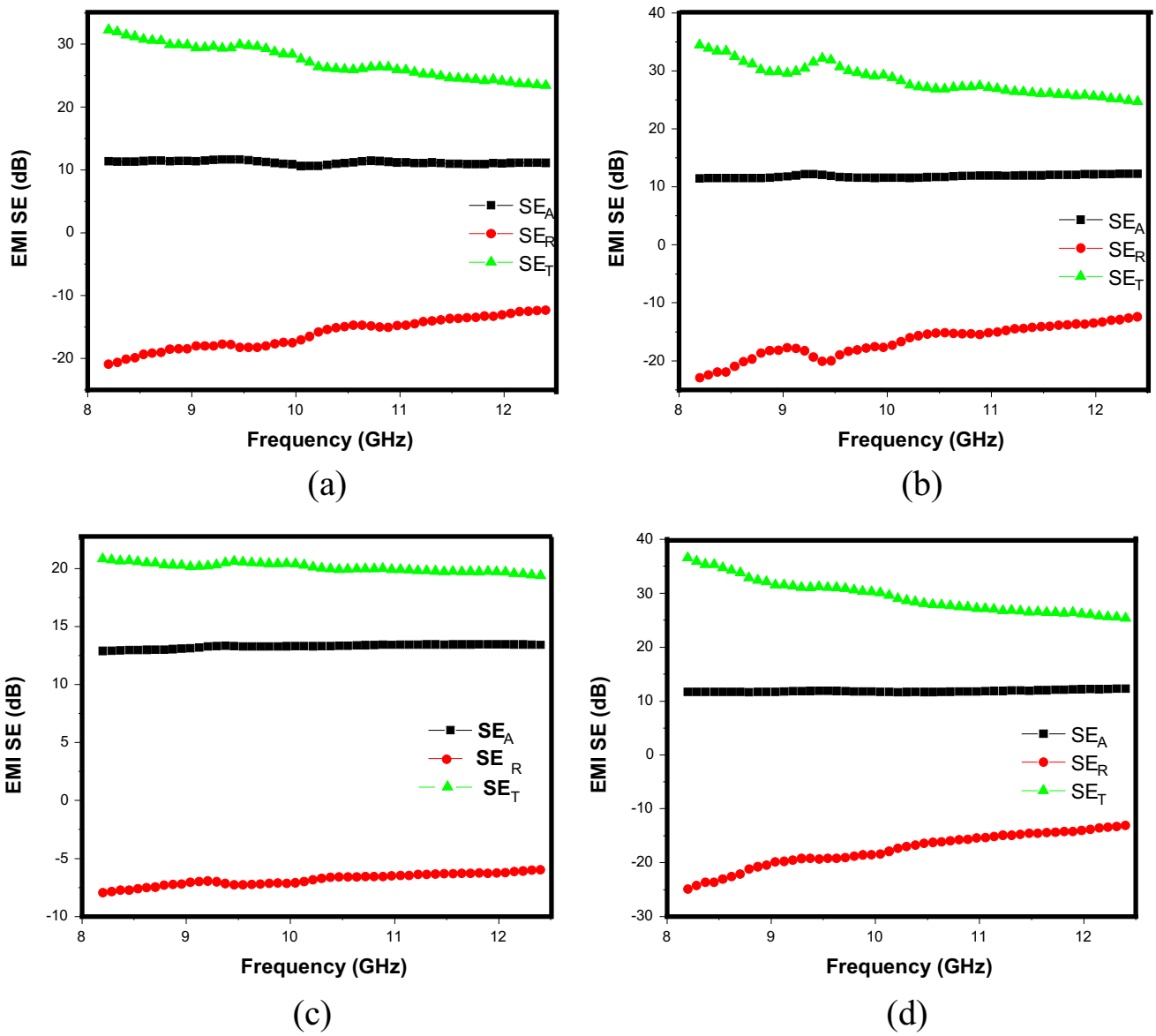

Fig. 5 Comparison of SE_T, SER, and SEA in samples (a) FC1, (b) FC2, (c) FC3, and (d) FC4 as a function of frequency (8.2–12.4 GHz) (the thickness of all the samples is 0.7 mm)

[34], acrylonitrile-co-butadiene-co styrene(ABS)/ carbon black(CB): carbon nanotubes(CNT) [35], chlorinated polyethylene(CPE)/ carbon black (CB) [36], poly (styrene-b-ethylene-ran-butylene-b-styrene)(SEBS) and carbon additives (SEBS/CB) [37], Ketjen carbon black (K-CB)-ethylene methyl acrylate copolymer (EMA) [38], graphene Nano platelets /Fe_3O_4/epoxy (G/Fe_3O_4/epoxy) [39], Fe_3O_4/graphene foam/poly dimethyl siloxane (Fe_3O_4/G/PDMS) [40], magnetic reduced graphene oxide (RGO)@Fe_3O_4 (RGO/Fe_3O_4) [41], silicone rubber/MWCNTs/Fe_3O_4 [42], PMMA/Fe_3O_4@ MWCNTs [43], it can be seen that the sample thickness is thinner for the Fe_3O_4/CBN composite material.

The Fe_3O_4/CBN composite tends to be a promising candidate for high-absorption materials as a result. CBN and Fe_3O_4 not only cause the dielectric and magnetic loss but also improve the EMI shielding efficiency of the composites by multiple internal reflections. Suitable impedance matching of the composite in the study allow waves to enter the material, and due to several internal reflections in them, the reflected internal radiation energy is subsequently absorbed by the carbon black [44].

In a review of the literature, it was found that similar studies have not been conducted to show the SE of the shielding material with the nanocomposites used in the present study, which was the limitation of this study.

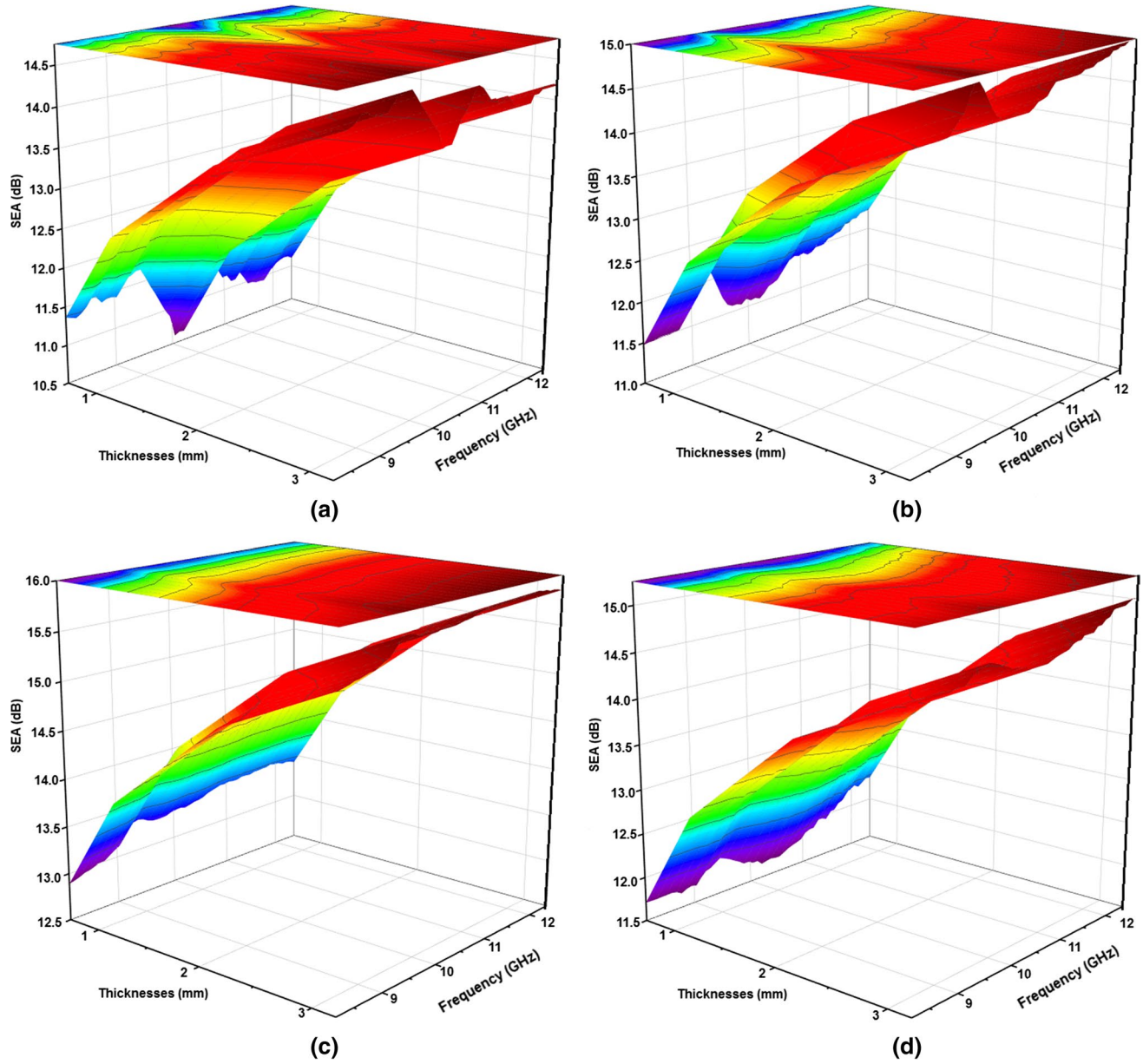

Fig. 6 SE_A as a function of frequency for samples (a) FC1; (b) FC2; (c) FC3; (d) FC4 with different thicknesses

Future research could be on optimizing morphology and structure to provide optimal nanocomposites.

Conclusion

In the study, four nanocomposites were prepared by mixing magnetite and carbon black nanoparticles was mixing in epoxy resin, successfully and their shielding EMI efficiency was investigated in the frequency range of 8.2–12.4 GHz. The mixing and casting method has provided a good dispersion of magnetite and CBN in the epoxy resin. The maximum value of SE was 36.62 dB at 8.2 GHz for epoxy resin nanocomposites with a content of a weight percentage of 50% carbon black nanoparticles and 15% magnetite and 0.7 mm in thickness. These magnetite/carbon black nanocomposites based on epoxy resin provide promising opportunities for EMI shielding materials. The Fe_3O_4/CBN composite not only causes the dielectric and magnetic loss but also improves the EMI SE of the composites by multiple internal reflections. Suitable impedance matching of the composite in the study allows waves to enter the material, and due to several internal

Table2 Summary of the last 5 years' EMI SE in sample thickness of different compounds based on Fe_3O_4 and carbon black

Composites	Measurement region (GHz)	Thickness (mm)	EMI SE (dB)	Reference
reduced graphene oxide–Fe_3O_4 (rGO:IO)	8 to 12	1	14.4–12.6	[34]
acrylonitrile-co-butadiene-co-styrene(ABS)/ carbon black(CB):carbon nanotubes(CNT)	8 to 12	2	29.37	[35]
chlorinated polyethylene(CPE)/carbon black (CB)	8.2–12.4	1	29	[36]
poly (styrene-b-ethylene-ran-butylene-b-styrene)(SEBS) and carbon additives (SEBS/CB)	8.2–12.4	5	17.5	[37]
Ketjen carbon black (K-CB)-ethylene methyl acrylate copolymer (EMA)	8.2–12.4	1	18.3	[38]
graphene nanoplatelets /Fe_3O_4/epoxy(G/Fe_3O_4/epoxy)	8–12	2	17	[39]
Fe_3O_4/graphene foam/poly dimethylsiloxane (Fe_3O_4/G/PDMS)	8.2–12.4	1	32.4	[40]
magnetic reduced graphene oxide (RGO)@Fe_3O_4 (RGO/Fe_3O_4)	8.2–12.4	1	13.45	[41]
silicone rubber/MWCNTs/Fe_3O_4	8.2–12.4	2	27.5	[42]
PMMA/Fe_3O_4@ MWCNTs	8.2–12.4	2.5	13.1	[43]
Fe_3O_4/CBN (FC4)	8.2 to 12.4	0.7	25.4–36.6	Present study

reflections in them, the reflected internal radiation energy is subsequently absorbed by the carbon black.

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Declarations

Competing Interest The authors declare that they have no conflicts of interest and no known competing for financial regarding this study.

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