






Electromagnetic shielding effectiveness performance of carbon fiber reinforced polymer (CFRP) composites with hematite and goethite in far-field

Hematit ve götit takviyeli karbon fiber polimer kompozitlerin uzak alan elektromanyetik kalkanlama etkinliği performansı

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Abstract

Carbon fibers (CFs) are indispensable materials in our daily life. The excellent bearing capacity, remarkable dielectric property, ease of production, and corrosion resistance of CFRP composites distinguish them from all other options in addition to them, CFRPs may also shield from electromagnetic interference (EMI). In this study, two-layer CF reinforced epoxy composites reinforced with two different hematite (alpha-Fe₂O₃) and goethite (FeO(OH)) particle sizes of 50 nm and 45 µm (325 mesh) were produced using the manual lay-up method. Then, far field electromagnetic shielding effectiveness (SE) with 700 MHz - 6000 MHz range were examined. The maximum shielding effectiveness was determined to be at 5200 MHz with 39.28 dB for 5 wt.% FeO(OH), at 4700 MHz with 38.38 dB for 10 wt.% Fe₂O₃(325 mesh), at 3800 MHz with 37.15 dB for 15 wt.% Fe₂O₃(50 nm).

Keywords: Carbon fibers, Far field, Electromagnetic shielding, carbon fiber reinforced polymer, Hematite, Goethite.

Öz

Karbon fiber (carbon fiber: CF) katkılı malzemeler, günlük hayatımızda vazgeçilemez malzemelerdir. CF takviyeli kompozitlerinin (CFRP) üstün mukavemeti, yüksek dielektrik özelliği, üretim kolaylığı ve korozyon direnci, CFRP'leri diğer malzemelerden öne çıkaran özelliklerinden yalnızca birkaçıdır. Bu çalışmada partikül boyutları 50 nm ve 45 µm (325 mesh) olan iki farklı hematit (alfa-Fe₂O₃) ile götit (FeO(OH)) takviyeli, 2 katmanlı CF takviyeli epoksi kompozitler el ile yatırma yöntemi kullanılarak üretilmiştir. Üretilen kompozitler 700 MHz-6000 MHz aralığında uzak alan elektromanyetik kalkanlama özelliklerini tespit etmek için testlere tabi tutulmuşlardır. Maksimum kalkanlama etkisi, ağırlıkça %5 FeO(OH) takviyeli kompozit için 39.28 dB ile 5200 MHz'de, %10 Fe₂O₃(325 mesh) takviyeli için 38.38 dB ile 4700 MHz'de, %15 Fe₂O₃(50 nm) takviyeli için 37.15 dB ile 3800 MHz'de belirlenmiştir.

Anahtar kelimeler: Karbon fiber, Uzak alan, Elektromanyetik kalkanlama, Karbon fiber takviyeli polimer kompozit, Hematit, Götit.

1 Introduction

The fast growth of wireless communications, particularly with the advent of 5G and Artificial Intelligence (AI) affords us a delightful and intelligent lifestyle. Although the fast development of wireless technology has enhanced our quality of life, the widespread use of wireless gadgets has unavoidably led to electromagnetic (EM) pollution, which is currently considered the fourth largest cause of pollution after air, water, and noise [1]. To reduce EM radiation pollution, the development of high-performance microwave absorption (MA) and electromagnetic interference (EMI) shielding materials is a crucial research effort [2].

Current EMI shielding materials consist mostly of metals and their alloys, conductive polymers, and carbon-based materials. The greatest benefit of metals and alloys is their excellent electrical conductivity. But these materials also have disadvantages, such as high density, low corrosion resistance, difficult production process, high cost and metal coatings' poor

wear resistance [3]. Due to their improved electrical characteristics, outstanding flexibility, cheap production cost, environmental friendliness, ease of fabrication, and chemical inertness, carbon materials and their composites have gained remarkable interest in the field of EMI shielding [4]. The most practical structural EMI shielding material among all the possibilities is considered to be carbon fibers (CFs)/polymer composites because of its high bearing capacity, outstanding dielectric characteristic, ease of manufacture, and corrosion resistance [5]. However, high electrical conductivity frequently leads in significant reflection loss and EMI shielding efficacy is dominated by reflection. Thus, the microstructure of the nanomaterials, the structure of the shield, and the presence of other materials, such as those with dielectric or magnetic dipoles, all have a significant impact in the absorption of EM waves [4]. In addition, CFs' deeper skin, low magnetism and high conductivity cause EMI impedance mismatch to rise. Modifying CFs with Fe and its derivatives, such as magnetite (Fe₃O₄) and hematite (Fe₂O₃), may therefore be a helpful strategy to solve this issue [6]. One of the thermoset resins that

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is widely used in industry and is simple to make and utilize is epoxy resin. Additionally, thermosetting polymers such epoxy compounds are employed as binders with Fe-derived materials because they serve as the appropriate dispersion matrix and prevent the agglomeration of Fe-derived nanostructures [6].

Recent studies have produced carbon fiber reinforced polymer (CFRP) composites reinforced with Fe-derived materials using a variety of techniques. Liu et al. [7] reinforced Fe₃O₄ (1, 3 and 5 wt.%) in CFs/cement composites and researched EMI shielding characteristics in the X band (8. 2-12.4 GHz). They discovered that the cement matrix's 5 wt.% Fe₃O₄ and 0.4 wt.% CFs reinforcement boosted the EM shielding effect by 34.4%. Electrophoretic deposition was used to cover CFs with nano-Fe₃O₄ by Salimkhani et al. [8], they examined the CFs' magnetic and microwave characteristics. The largest reflection loss for the composites, according to their observations, was - 7.8 dB at 9.3 GHz for a layer with a thickness of 2 mm. In order to test the effectiveness of EMI shielding, Anaraki et al. [9] produced Fe-FeO reinforced epoxy nanocomposites (20, 40, and 60 wt.%). They discovered that increasing the amount of nanoparticles increased the shielding performance, and the maximum effectiveness was discovered with 55 dB at 12.3 GHz for 60 wt.% reinforced composites. High-density polyethylene (HDPE) reinforced composites with iron scale reinforcement and EM field shield research were made by Jakubas et al. [10], they compared the different types of iron scale and found that iron scale in the form of flakes provides the maximum shielding performance (45 dB at 10 GHz for 80 wt.% and approximately 70 dB at 8.1 GHz for 20 wt.%). Consequently, in the literature graphite, graphene oxide and magnetite are studied for EMI SE [11]-[13]. In the current work, Fe₂O₃ and goethite (FeO(OH)) particles were used to reinforce the epoxy and CFs/epoxy matrix, and sandwich composites were produced. The impacts of different FeO(OH) and Fe₂O₃ reinforcement wt.% on EMI shielding effectiveness, as well as the effect of Fe₂O₃ powder particle size, were investigated.

2 Materials and methods

The epoxy resin (Metkon Epocold-R) was mixed with the hardener (Metkon Epocold-H) at a 5:1 ratio, respectively. Fe₂O₃ powders (Alfa Aesar, 98%, Lot: N09B013) in two distinct particle sizes 325 mesh and 50 nm, as well as FeO(OH) powders (19 μm, particle size analysis was measured with the Malvern-Mastersizer3000e) have been used as reinforcement materials. Twill CF textiles with a 3k 245 g/m² areal mass were used as the primary reinforcing material. A flat glass mould (250 x 250 mm) was employed to create the composites. Wax was added to make it simpler to extract the composite from the glass mould. The wax and CF textiles were obtained from Dost Kimya company. The steps below were used to produce composites: First, a sonicator (Bandelin Sonoplus HD 3200) was used to disperse Fe₂O₃ or FeO(OH) in epoxy resin for two hours.

After sonication, a magnetic stirrer (Heidolph MR Hei-Standart) was used to mix the mixture for 30 minutes at 500 rpm. Hardener was added to the prepared mixture and continued to be mixed with a magnetic stirrer throughout the production. The dimension of the CFs was 200x200 mm, and they were stacked 90° apart in the same direction. Wax was applied to the glass mould before production. After being well mixed in the magnetic stirrer, the mixture was then applied to the CFs using a brush. As a consequence, the hand lay-up technique is used to fabricate the composites. The prepared

composites were dried under ambient conditions for 24 hours. The reinforcement types and amount of their additions for each sample is given in Table 1.

Table 1. Types and wt.% of reinforcements of prepared composites.

Sample No	Reinforcement types and wt.%
S1	-
S2	5 wt. % Fe ₂ O ₃ (325 mesh-45 μm)
S3	10 wt. % Fe ₂ O ₃ (325 mesh-45 μm)
S4	15 wt. % Fe ₂ O ₃ (325 mesh-45 μm)
S5	5 wt. % Fe ₂ O ₃ (50 nm)
S6	10 wt. % Fe ₂ O ₃ (50 nm)
S7	15 wt. % Fe ₂ O ₃ (50 nm)
S8	5 wt. % FeO(OH)
S9	10 wt. % FeO(OH)
S10	15 wt. % FeO(OH)

Different measurement methods are used for EMI-SE. The first method is made by using the Vector Network Analyzer shown in Figure 1 to determine S-parameters, absorption and reflection effects [14]-[17].

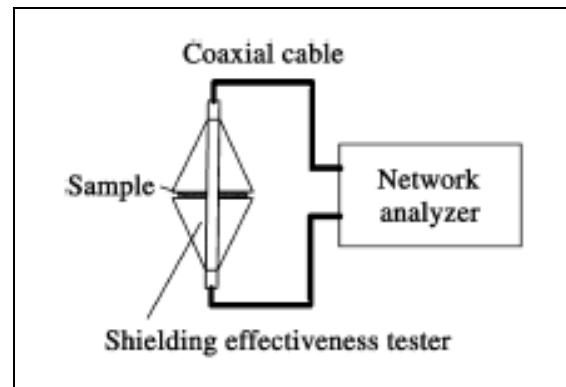
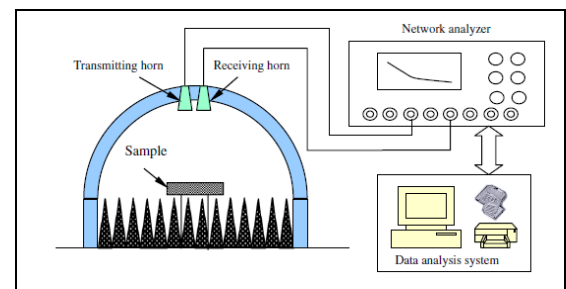
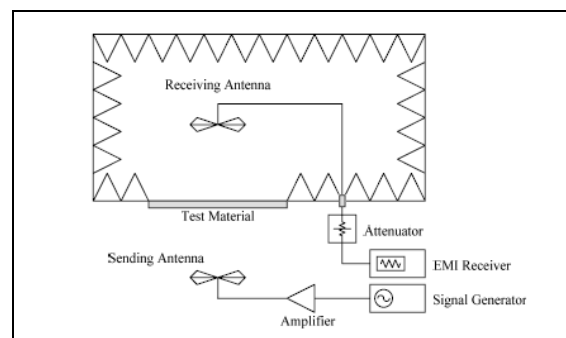


Figure 1. VNA measurement.

The second method is made by using the spectrum analyzer and electromagnetic field generator as shown in Figure 2.



(a)



(b)

Figure 2. Spectrum analyzer and field generator measurement.

The second method is applied by two ways. The first one is shown in Figure 2(a). The spectrum analyzer and the field generator are on the same side. Electric field is generated, and then reflected from the sample, finally reaches to the spectrum analyzer [18]. The second one is shown in Figure 2(b). The spectrum analyzer and the field generator are on the opposite side. Electric field is generated, and then transmitted from the sample, finally reaches to the spectrum analyzer [19]. There are many different types of materials used for EMI shielding. Like other carbon materials, carbon fibers (CFs) have a high mechanical strength, a low density and high electrical conductivity [20]. Due to being a conductor, electromagnetic field having very high frequency could penetrate the sample as skin depth [21]. CFs are conductor and the magnitude of the reflection coefficient is very close to 1 so that most of the coming electromagnetic waves are reflected from the sample surface. For that reason dominant EMI shielding mechanism is reflection. Schematic representation of experimental design is shown in Figure 3. The distance between the Receiver Spectrum Analyzer (RSA) and Transmitter Signal Generator (TSG) is set to 70 cm. Measurements are carried out under room conditions. The measurement setup includes a directional field generator (400 MHz-6 GHz) and spectrum analyzer (1 MHz-9.4 GHz). The diffractions from the edges are neglected. Measurements are performed at 100 MHz intervals between 700 MHz and 6 GHz and interpolation techniques are not used. Therefore, sharp changes are appeared in the measured values. As the intervals are reduced, changes will be smoother. The measurement scenario is designed in three positions are called M, L and R. In M position, the test material is placed at the midpoint of RSA and TSG as it is illustrated in Figure 3(a). In L position, the test material is placed at the vicinity of the TSG as it is depicted in Figure 3(b). In the last position, is called R, the test material is placed at the vicinity of the RSA as it is demonstrated in Figure 3(c).

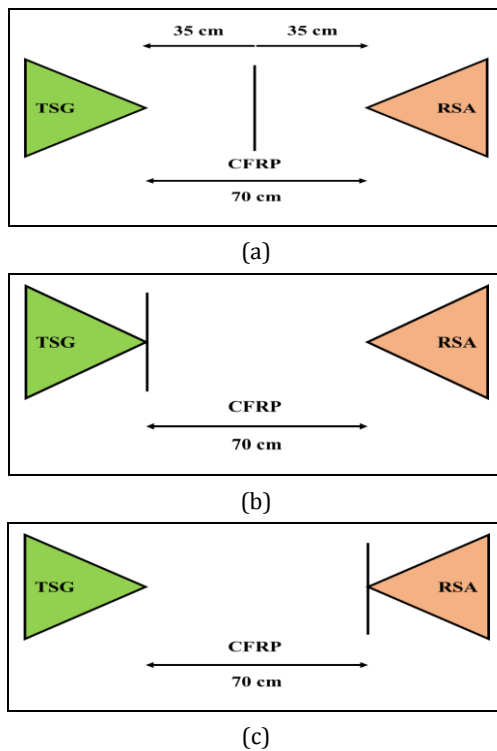


Figure 3. Schematic representation of experimental design.

EMI SE measurements are carried out between 700-6000 MHz and shielding performances are recorded. Aaronia HF60105 spectrum analyzer (RSA) and Aaronia DFG4060 Directional Field Generator (TSG) is used.

3 Results

Before the measurement with test material, first of all free space loss should be determined. Even if that there is no any obstacle between the transmitter and the receiver antenna, electromagnetic wave emanates from the TSG is attenuated. Free space measurements are shown in Figure 4.

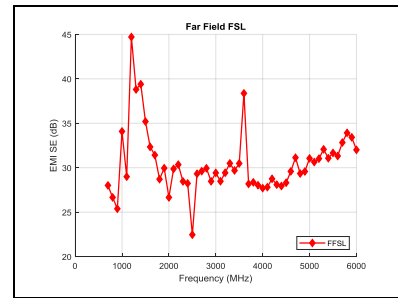


Figure 4. Free space loss.

The EMI SE performance for all specimens in M position is given in Figure 5.

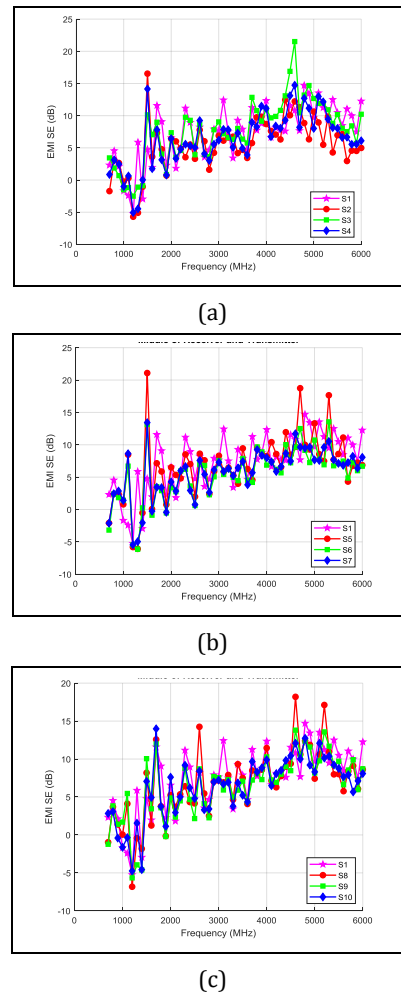


Figure 5. EMI SE values for CFRP composites. (a): Fe₂O₃ (325 mesh). (b): Fe₂O₃ (50 nm), and (c): FeO(OH) in the M position.

As it is seen in Figure 5, the most remarkable shielding performance is observed as 21.51 dB at 4600 MHz for S3, 21.08 dB at 1500 MHz for S5, and 18.19 dB at 4600 MHz for S8, respectively. While the highest shielding is obtained at the rate of 5 wt.% in FeO(OH) and Fe₂O₃ (50 nm) added composites, it is received at 10 wt.% in Fe₂O₃ (325 mesh) reinforced composites. It is understood that there is no need for reinforcement of 15 wt.% for the M position because the best shielding values are obtained in the order of 5 wt.% and 10 wt.%.

The EMI SE performance for all specimens in L position is demonstrated in Figure 6.

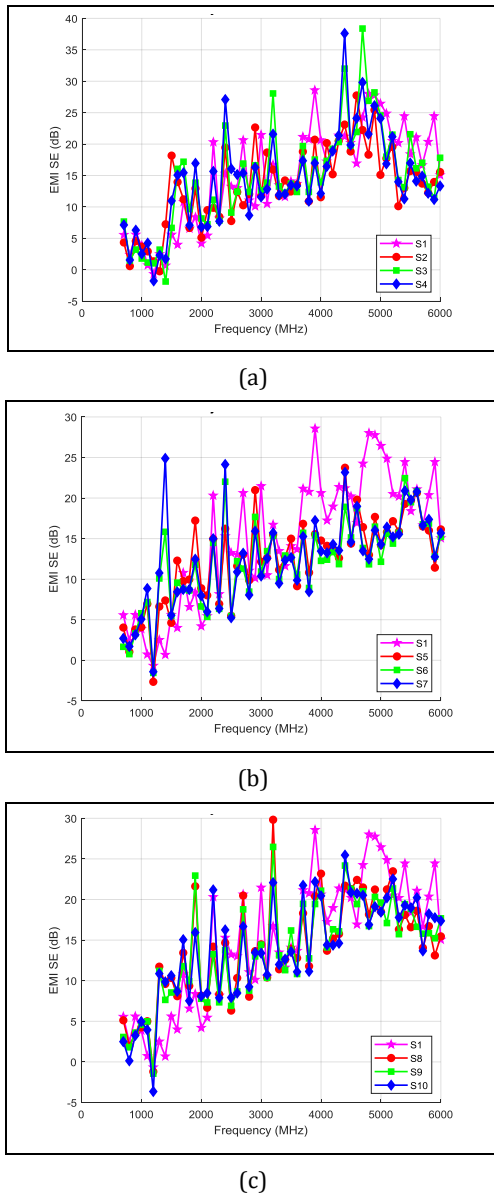


Figure 6. EMI SE values for CFRP composites. (a): Fe₂O₃ (325 mesh). (b): Fe₂O₃ (50 nm), and (c): FeO(OH) in the L position.

As it is demonstrated in Figure 6, the best EMI SE performance for produced composites is measured at 38.38 dB at 4700 MHz for S3, 24.89 dB at 1400 MHz for S7, and 26.48 at 3200 MHz for S9. In this position, it is observed that the shielding performance of Fe₂O₃ (325 mesh) added composites, especially those with 10% and 15% reinforced, are good. When

comparing the other reinforced composites with the S1 composites, surprisingly, S1 specimens also gave good EMI SE results. The reason for this may be that the electromagnetic waves produced by the transmitter pass the S1 sample and cannot be collected sufficiently with the receiver due to the gap after specimen. However, Fe₂O₃ and FeO(OH) reinforced materials showed better EMI SE properties than the S1 specimen at some frequencies such as 2400 and 4500 MHz for Fe₂O₃ (325 mesh), 1400 and 2300 MHz for Fe₂O₃ (50 nm), 4400 and 5800 MHz for FeO(OH).

The EMI SE performance for all specimens in R position is depicted in Figure 7.

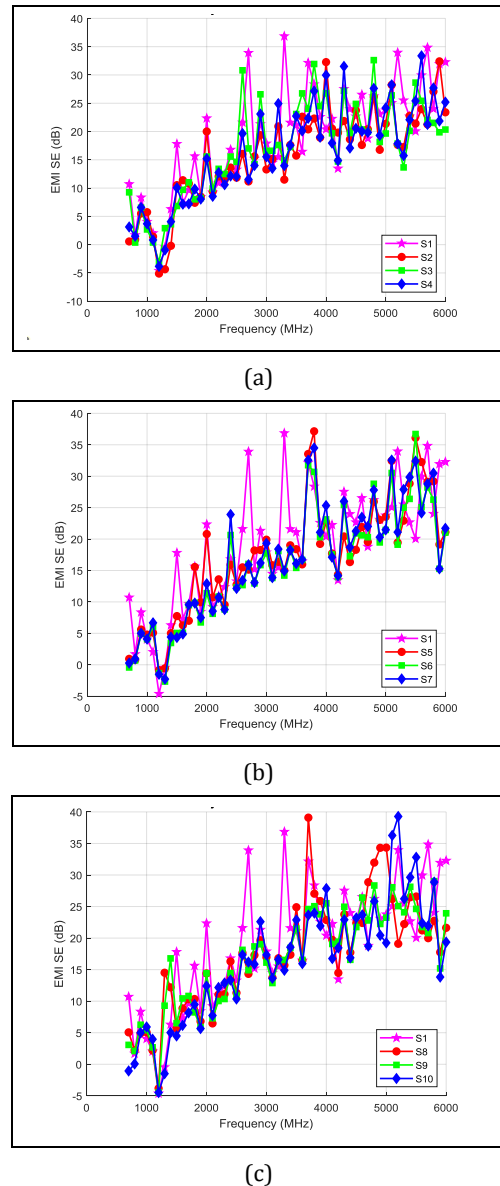


Figure 7. EMI SE values for composites (a) Fe₂O₃(325), (b) Fe₂O₃ (50 nm), and (c) FeO(OH) in the R position.

As it is depicted in Figure 7, the best shielding performance for R position, is 39.28 dB at 5200 MHz for S8, 21.08 dB at 1500 MHz for S5, and 32.62 dB at 4800 MHz for S3. It is seen that the EMI SE performance of unreinforced CFRP composites is higher in the R position than the L position. Among the reinforced

CFRP composites, it was observed that the EMI SE properties of the FeO(OH) reinforced composites are the highest.

The average of EMI SE values of the all specimens between 700-6000 MHz are found in Table 2. It is observed that the addition of reinforcement decreased the average values EMI SE in all measurement methods for all ratios (except S3 in the M position). According to the results, the highest average EMI SE value is 18.89 dB for the S1 specimen in the R position. The worst average EMI SE values are obtained in M position and it is found that the lowest EMI SE value is in the S6 sample with 5.38 dB. The best EMI SE performance is obtained in FeO(OH) reinforced composite materials among all composite materials under all measurement positions and the best performance in the average EMI SE values for all samples was observed in the R position.

Table 2. Average EMI SE values of specimens.

Sample No	M	L	R
S1	7.26	14.79	18.89
S2	5.55	13.31	15.68
S3	7.63	14.60	16.84
S4	6.32	14.17	16.21
S5	6.72	12.51	17.08
S6	5.38	12.04	15.93
S7	5.60	12.50	16.61
S8	6.67	14	17.31
S9	6.33	13.73	16.74
S10	6.39	13.87	16.33

As it is seen in Table II, EMI SE values of materials are different for each position resulted from the diffraction/scattering of the wave as mentioned in [22].

4 Conclusion

This study aims to investigate far-field EMI SE of Fe₂O₃ (325 mesh and 50 nm) and FeO(OH) reinforced CFRP composites. Fe₂O₃ and FeO(OH) reinforced CFRP are successfully produced. The results are summarized as follows:

- EMI SE values increased with the increase of frequency values for all samples,
- The highest EMI SE values for FeO(OH), Fe₂O₃(325 mesh) and Fe₂O₃(50 nm) reinforced materials is 39.28 dB, 38,38 dB and 37.15 dB, respectively,
- Considering the average EMI SE results, it is determined that the most efficient position is the R position, the worst position is the M position and the most effective reinforced material is FeO(OH). However, the highest average EMI SE value is found for S1 specimen with 18.89 dB at R position,
- Considering the average EMI SE results for Fe₂O₃, it is seen that 325-mesh grain size performed better for M and L positions, while 50 nm grain size performed better for R position. As a future work, magnetite, nickel, carbon black, FeNiCoCu alloy and Fe powder will used for comparisons.

5 Acknowledgment

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6 Author contribution statements

In this study, Uğur ERBAŞ worked on performing far field electromagnetic tests, evaluating the results and writing the article. Cantekin KAYKILARLI, Taha Yasin EKEN and Burak KÜÇÜKELYAS worked on the manufacturing of composite materials, introduction and writing of the article. Mehmet Barış Tabakcıoğlu worked on the main idea of the article, the evaluation of electromagnetic studies, the general layout and interpretation of the article.

7 Ethics committee approval and conflict of interest statement

"There is no need to obtain permission from the ethics committee for the article prepared".

"There is no conflict of interest with any person / institution in the article prepared".

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