

RESEARCH PAPER

Study of the Mechanical and Absorption Properties of Fe₂O₃/Epoxy Nanocomposite in the Microwave Frequency Range

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ABSTRACT

This research aims to investigate the effect of adding iron oxide nanoparticles (Fe₂O₃) at different concentrations to epoxy resin on both its mechanical and absorption properties within the X-band (8–12 GHz). Nanoparticles containing 1%, 3%, 5%, and 7% Fe₂O₃ were synthesized, and their mechanical behavior was evaluated using tensile strength, Young's modulus, hardness, and impact resistance tests. The results showed that the medium concentrations (3% and 5%) achieved the best improvement in stiffness, rigidity, and energy absorption capacity, due to good particle dispersion and enhanced interfacial interaction within the polymer matrix. In contrast, the high concentration of 7% led to particle agglomeration and a decrease in some mechanical properties. Regarding microabsorbents, the enhanced samples exhibited a strong absorption response across the frequency range, with deeper and more regular absorption peaks appearing at 3% and 5% concentrations, indicating an effective balance between dielectric and magnetic loss mechanisms and impedance compatibility. At 1%, absorption performance was limited, while irregularities appeared in the 7% sample due to agglomeration. Taken together, these results suggest that the addition of Fe₂O₃ at 3–5% concentrations gives the epoxy a balanced combination of mechanical enhancement and absorption efficiency, making these composites promising candidates for wave-absorbing and radar applications.

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INTRODUCTION

Polymer composites reinforced with nanomaterials are among the most important advanced materials used in engineering applications, particularly in electromagnetic shielding and microwave absorption [1]. Epoxy resin is a suitable matrix for these applications due to its rigidity and chemical stability; however, its mechanical and absorption properties need enhancement to achieve better performance in

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radar bands [2,3]. Iron oxide (Fe₂O₃) is considered one of the effective nanomaterials for improving the behavior of composite materials, given its suitable magnetic and dielectric properties, good thermal stability, and ability to interact with the polymer matrix [4–6]. Incorporating Fe₂O₃ particles into the epoxy contributes to improving mechanical properties, such as Young's modulus due to increased structural rigidity, hardness due to resistance to local deformation, and



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impact strength by inhibiting crack growth and altering crack trajectories. Furthermore, Fe₂O₃ particles contribute to improved electromagnetic absorption through magnetic and dielectric loss mechanisms, making the composite suitable for use in the X-band (8–12 GHz) used in radar systems [7,8]. Although numerous studies exist in this area, a comprehensive evaluation of the effect of different Fe₂O₃ weight ratios on the mechanical and absorption properties still requires further investigation [9,10]. Therefore, this research aims to investigate the effect of adding Fe₂O₃ nanoparticles at different weight ratios to epoxy resin on Young's modulus, hardness, and impact strength, as well as to evaluate the absorption properties in the microwave range (8–12 GHz) and analyze the resulting enhancement mechanisms. Polymer composites reinforced with nanomaterials are a rapidly developing research area, particularly when it comes to developing materials that combine mechanical strength with microwave absorption properties. Iron(III) oxide (Fe₂O₃) is one of the most widely used nanomaterials in this context due to its suitable magnetic properties, thermal stability, low cost, and ability to interact with polymer matrices [11]. Several studies have shown that adding Fe₂O₃ nanoparticles to epoxy resin improves the mechanical properties of the composites. In a study by Al-Mamun et al. (2021), epoxy composites were prepared reinforced with α -Fe₂O₃ nanoparticles. The results showed a significant increase in stiffness, tensile strength, and Young's modulus compared to pure epoxy, due to the excellent surface interaction between the nanoparticles and the polymer matrix. The researchers also noted that the good dispersion of the nanoparticles plays a crucial role in enhancing the material's properties and reducing stress concentration [5]. In a recent study, researchers enhanced an epoxy matrix using Fe₂O₃ nanoparticles embedded in carbon fibers to develop multifunctional composites that combine high mechanical strength with efficient electromagnetic wave mitigation. The study demonstrated that incorporating Fe₂O₃ with carbon fibers within the epoxy not only improved electromagnetic shielding performance but also enhanced structural strength and tensile strength, confirming the effectiveness of this strategy in improving mechanical properties while maintaining multidimensional functionality [12]. Regarding microwave absorption properties,

several studies have indicated the effectiveness of Fe₂O₃ in enhancing microwave absorption within the X-band (8–12 GHz). In a study by Zhang et al. (2022), Fe₂O₃-based composites reinforced with Diatomite were synthesized. These materials achieved a reflectance loss of -54.2 dB at a frequency of approximately 11.5 GHz, along with an effective absorption bandwidth ranging from 9.76 to 18 GHz, demonstrating the ability of Fe₂O₃ to absorb microwaves with high efficiency when prepared with a suitable structure [13]. Azis et al. also studied... (2024) Properties of Fe₂O₃ nanoparticles and Fe₂O₃/CNTs. The results showed that the particle microstructure and crystal size directly influence absorption behavior, with magnetic loss mechanisms such as Néel and Brownian relaxation playing a pivotal role in wave energy absorption. The study demonstrated that optimizing the balance between dielectric and magnetic losses is key to achieving effective absorption [14]. A study by Liu et al. (2020) demonstrated that the development of nano-iron oxide (α -Fe₂O₃)-based composites leads to a significant improvement in X-band electromagnetic wave absorption. This improvement is attributed to the complementary interaction between the magnetic loss of iron ferrite and the dielectric loss of the carrier matrix. The results indicate that optimizing the microstructure and increasing interfacial polarization contribute to better impedance matching and reduced reflectance loss, making Fe₂O₃ a promising material for designing wave-absorbing and radar-based materials and coatings [9].

MATERIALS AND METHODS

Epoxy resin was used as the polymer matrix, while iron oxide nanoparticles (Fe₂O₃) were added at 1%, 3%, 5%, and 7% by weight. The epoxy and Fe₂O₃ were mechanically mixed, and the mixture was then ultrasonically dispersed to ensure homogeneous distribution. A hardener was added at a 2:1 ratio, and the mixture was poured into custom molds. The samples were left to harden at room temperature, and then heat treatment was performed to improve their properties. The mechanical properties were evaluated using tensile, hardness, and impact tests according to standard parameters, while the absorption properties were measured using an X-band lattice analyzer.

Sample Preparation

Epoxy resin was used as the primary matrix with a hardener at a suitable weight ratio of 2:1. Iron oxide (Fe₂O₃) nanoparticles were added at different weight ratios (1%, 3%, 5%, and 7%) to the epoxy before adding the hardener. To achieve better homogeneity, magnetic stirring followed by ultrasonic dispersion was used to ensure uniform particle distribution and prevent agglomeration. The mixtures were then poured into special silicone molds, vacuum degassing was performed, and the samples were left to cure at room temperature for 24 hours, followed by a mild heat treatment to ensure complete curing.

Characterizations

X-ray diffraction (XRD)

The measurement was performed using a Cu-Kα X-ray diffraction device ($\lambda = 1.5406 \text{ \AA}$) in a θ -2 θ system within an angular range of 10°–80° with a scan step of 0.04°, an operating current of 30 mA and a voltage of 40 kV. X-ray diffraction (XRD) was used to study the structural properties and degree of crystallinity of epoxy composites reinforced with ferrite nanoparticles. Analysis of the diffraction patterns allows for the determination of the semi-crystalline nature of the epoxy matrix, as well as the identification of the characteristic crystalline phases of the Fe₂O₃ nanoparticles. It also contributes to evaluating the effect of the nanoparticle additions on the crystalline structure of the composite.

The interplane distances (d-values) were calculated using Bragg's Law [15]:

$$2d_{hkl} \sin\theta = n\lambda \quad (1)$$

The average crystalline size of the ferrite nanoparticles was estimated using the Debye-Scherrer equation [15]:

$$D = (K\lambda)/(\beta \cos \theta) \quad (2)$$

Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is used to study the morphology and surface structure of materials, providing high-resolution images that reveal particle shape, size, and distribution. In this research, SEM was used to characterize pure ferrite (Fe₂O₃) particles prior to their use in synthesizing compounds.

Mechanical Testing

Tensile Test

The tensile test was performed using a Universal Testing Machine (UTM) according to ASTM D638-87. Number of samples: 5. The following were obtained:

Stress-strain curve, Young's modulus (from the slope of the linear portion of the curve), tensile strength, and elongation at fracture.

Hardness Test

The hardness of epoxy samples reinforced with ferrite nanoparticles was measured using a Shore D Durometer to assess the samples' resistance to surface penetration. This test measures the penetration depth of a pointed metal tip into the material's surface under a standard load, with the measured values, expressed in Shore D (HD), representing the material's hardness. Measurements were performed at room temperature after calibration of the instrument, with multiple readings taken at different locations for each sample and the arithmetic mean calculated. The addition of ferrite nanoparticles to the epoxy matrix increases the hardness values by restricting the movement of the polymer chains and improving interfacial bonding, thus enhancing the surface deformation resistance of the nanocomposite.

Impact Strength Test

Impact strength was tested using a Charpy Impact Tester according to ASTM D6110. The fracture energy was recorded and converted to impact strength (kJ/m²).

Electromagnetic absorptivity test

Electromagnetic absorption tests were performed using a Vector Network Analyzer (VNA) in the X-band (8–12 GHz). Samples were cut into standard shapes suitable for waveguides. The scattering coefficients (S_{11} and S_{21}) were measured, and from these, the reflection and transmittance coefficients were calculated using the Nicolson-Ross-Weir (NRW) algorithm. The reflection loss (RL) was calculated using the equation [11]:

$$\left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \log_{10} 20 = RL(\text{dB}) \quad (3)$$

Where Z_{in} is the input impedance of the samples and Z_0 is the vacuum impedance (377 Ω) [16].

RESULTS AND DISCUSSION

XRD for Pure Nano powdered Ferrite (Fe₂O₃)

The X-ray diffraction pattern of the pure nanopowdered ferrite (Fig. 1) shows sharp, distinct peaks within the 2θ angle range, with good agreement between the angle values and intercalated distances (d(hkl) exp) and standard values (d(hkl) std), as detailed in the Table 1 for pure ferrite. This agreement indicates the purity and stability of the crystalline phase and the absence of significant crystalline impurities. The recorded peaks correspond to the hematite phase (α-Fe₂O₃) or the cubic/spinnly ferrite structure, as per the ICDD PDF reference card(15). Calculations

of the nanocrystal size using the Scherer equation show that the values fall within the range (≈ 14–44 nm), confirming the nanoscale nature of the powder and its high degree of crystallinity. This pattern represents a key reference when studying the effect of mixing these nanoparticles with epoxy [11,17].

SEM Test

Scanning electron microscopy (SEM) images of pure ferrite reveal irregularly shaped particles with distinct agglomerations, reflecting the material's nanoscale nature and high surface energy. The particle dimensions visible in the image indicate

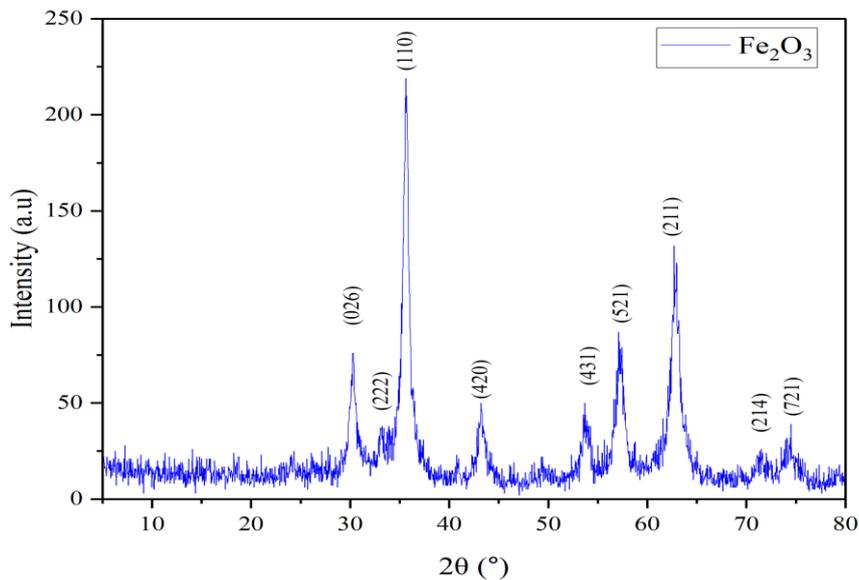


Fig. 1. X-ray diffraction pattern of nano Fe₂O₃.

Table 1. X-ray diffraction peaks of Fe₂O₃.

2θ (Deg.)	FWHM (rad)	dhkl STd.(Å)	dhkl Exp.(Å)	Crystallite size (nm)	Average Crystallite size (nm)	hkl
30.2760	0.4723	2.9562	2.949	19.399	25.07888	026
33.1223	0.4723	2.6928	2.702	20.0117		222
35.6818	0.4723	2.5322	2.514	20.641		110
43.2912	0.6298	2.0967	2.088	17.297		420
53.9238	0.9446	1.9295	1.698	14.298		431
57.2737	0.4723	1.7006	1.607	31.192		521
62.8587	0.3936	1.6159	1.477	44.493		211
71.3405	0.9446	1.4794	1.320	26.651		214
74.3282	0.9446	1.2746	1.275	31.723		721

a nanoscale, providing a large surface area. These observations are consistent with X-ray diffraction (XRD) results, which showed an average crystalline grain size of approximately 25 nm. This fine crystalline size represents the internal structure of the particles, while the agglomerations visible in the SEM images represent secondary clusters of these nanocrystals.

Mechanical Properties

Stress-Strain Curves and Young's Modulus

The tensile test results indicate that adding Fe₂O₃ to epoxy resulted in significant changes in mechanical strength [18]. Pure epoxy exhibited a relatively high Young's modulus, while the value decreased with the addition of 1% Fe₂O₃ due to an insufficient particle quantity to form an effective reinforcing network, as well as the potential for imperfect dispersion leading to stress concentrations. At 3%, the Young's modulus improved thanks to an increased number of particles interacting with the matrix and a more homogeneous distribution, allowing for better stress transfer. However, at 5%, the value

decreased further, attributed to the potential for particle aggregation at this concentration, which could create weak zones within the material. At 7%, the Young's modulus reached its highest recorded value, indicating that this percentage allowed for the formation of a near-continuous network of Fe₂O₃ particles within the epoxy, capable of enhancing strength and limiting polymer chain movement. High R² values demonstrate the accuracy and reliability of measurements in analyzing the mechanical behavior of the material [19,20].

Hardness Tests

The hardness results show that Shore D values were significantly affected by the addition of Fe₂O₃ particles to the epoxy. Pure epoxy registered a value of 68, while the hardness decreased slightly at 1% to 66. This is typically attributed to the uneven dispersion at lower percentages, resulting in relatively weak areas within the matrix. At 3%, the hardness increased to 70 due to the increased number of particles interacting with the epoxy and the formation of better mechanical support at the

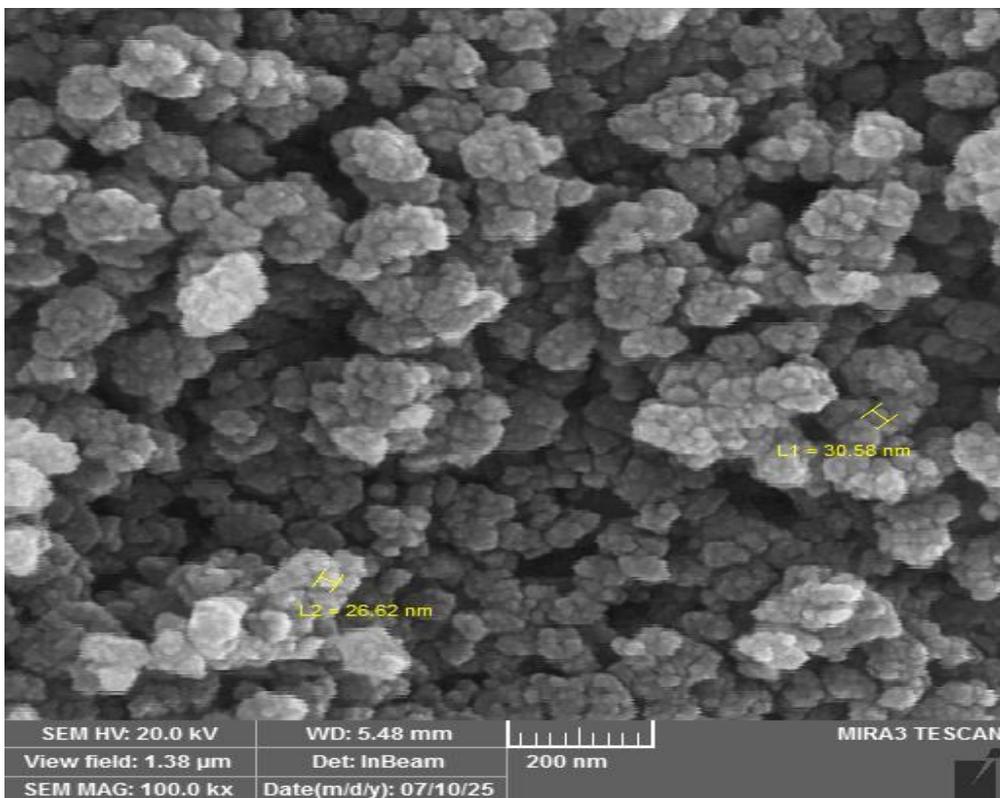


Fig. 2. SEM of nano Fe₂O₃.

particle-matrix interface, thus enhancing scratch and implant resistance. At 5%, the hardness decreased again to 68. This decrease is often explained by partial agglomeration of the particles at this percentage, leading to an uneven distribution

and resulting in less hard areas. Conversely, the hardness reached its highest value at 7%, registering 74.5, indicating that this concentration forms a stronger network of particles within the epoxy, capable of significantly enhancing surface

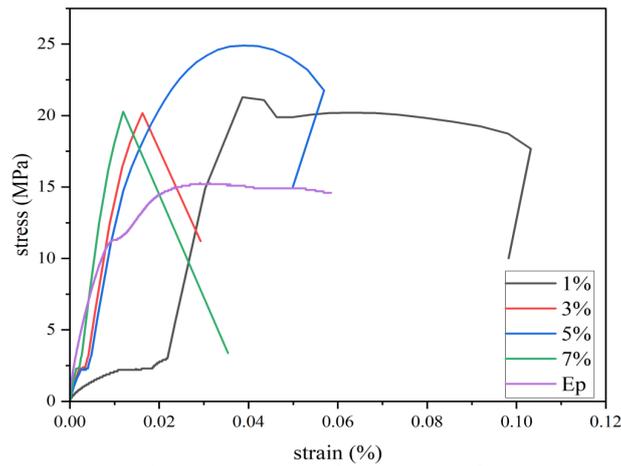


Fig. 3. Tensile stress-strain curve for tensile test of pure epoxy

Table 2. Values of tensile strength for pure epoxy and their nanocomposites

Sample	E(MPa)	R ²	UTM
epoxy	1673	0.9819	15.2
Ep+Fe ₂ O ₃ 1%	1126.46	0.987	21.28
Ep+Fe ₂ O ₃ 3%	1496.72	0.989	20.18
Ep+Fe ₂ O ₃ 5%	1227.86	0.98	24.9
Ep+Fe ₂ O ₃ 7%	1930	0.99	20.28

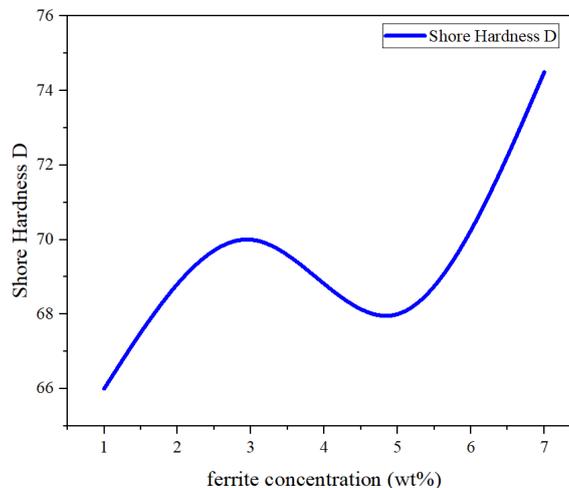


Fig. 4. Shore Hardness V(D) of composites.

resistance and increasing resistance to localized deformation. These results demonstrate that the overall hardness behavior depends primarily on the quality of particle dispersion and their degree of interaction with the matrix, and that a 7% Fe₂O₃ concentration was the most effective in improving the composite's surface hardness [18].

Impact Tests

Impact test results showed a clear difference in the samples' ability to absorb impact energy with varying percentages of added Fe₂O₃. Pure epoxy recorded the lowest absorption value of 0.40 J, reflecting its brittleness and rapid crack propagation due to the absence of any reinforcing agents within the polymer matrix. With the addition of 1% Fe₂O₃, the absorption energy increased significantly to 5.71 J, indicating that the presence of even a small amount of nanoparticles can enhance fracture resistance by inhibiting crack propagation and improving stress transmission. The improvement continued at 3% Fe₂O₃, with absorption energy ranging from 7.08 to 7.21 J,

demonstrating that increasing the particle content led to a more efficient distribution within the epoxy and the formation of reinforcing pathways that enhance the material's impact resistance. Samples containing 5% Fe₂O₃ also achieved similar values, ranging from 7.53 to 7.79 J, indicating that this concentration represents a region of mechanical stability that allows the composite to absorb higher energy before failure occurs. In contrast, the absorption energy decreased significantly at 7% Fe₂O₃, reaching only 1.64 J. This decrease is attributed to the potential for nanoparticle agglomeration at high concentrations, leading to the formation of weak zones within the material and increased brittleness, thus reducing impact resistance. Overall, the results show that the best impact resistance performance is achieved at 3% and 5% Fe₂O₃ concentrations, while both excessively low and excessively high filler content degrade performance [21,22].

Discussion for all mechanical tests

The combined mechanical results indicate that

Table 3. Values of hardness for pure epoxy and their nanocomposites

Sample	Hardness
Fe ₂ O ₃ 1% Ep+	66
Fe ₂ O ₃ 3% Ep+	70
Fe ₂ O ₃ 5% Ep+	68
Fe ₂ O ₃ 7% Ep+	74.5
Epoxy	68

Table 4. Values of impact for pure epoxy and their nanocomposites

sample	hammer weight (J)	Air resistance (J)	Sample and Air Strength (J)	discription
Ep+Fe ₂ O ₃ 1%	22	0.08	5.71	Without failure
Ep+Fe ₂ O ₃ 3%	22	0.08	7.08	Without failure
	22	0.08	7.21	Without failure
	22	0.08	7.53	Without failure
Ep+Fe ₂ O ₃ 5%	25	0.09	7.79	Without failure
	25	0.09	1.64	
Epoxy	25	0.09	0.4	



reinforcing epoxy with Fe₂O₃ particles significantly improved the composite's performance, exhibiting percentage-dependent behavior. Young's modulus decreased at 1%, improved at 3%, and peaked at 7%, reflecting a clear effect of the particles on matrix stiffness. Hardness also improved at 3% and reached its highest value at 7%, demonstrating increased surface resistance to deformation with increasing filler content. In impact testing, energy absorption capacity increased significantly at 1%–5%, while it decreased at 7% due to agglomeration, which increases the material's brittleness. Overall, the comparison shows that intermediate percentages (3%–5%) achieve the best balance between stiffness, hardness, and impact resistance, while a large increase in Fe₂O₃ can lead to a decline in some properties due to distribution inconsistencies.

Interpretation of Absorbance for All Ferrite Ratios (1%–7%)

The absorbance curves for all samples within the X-band (8–12 GHz) show a strong response

with a clear difference in the depth and number of absorption peaks depending on the added Fe₂O₃ percentage [11]. In the 1% sample, absorption is good but characterized by shallower peaks, reflecting the limited magnetic and dielectric loss mechanisms due to the low nanoparticle density within the epoxy. At 3% Fe₂O₃, absorption becomes more pronounced, and the peaks become deeper and more regular, indicating improved dispersion and higher loss efficiency resulting from greater particle-matrix interaction. The 5% Fe₂O₃ sample exhibits the best overall performance, possessing the deepest peaks and widest coverage across the frequency range. This suggests an ideal balance between dielectric and magnetic losses and good impedance compatibility, making the material more capable of absorbing waves over a wide range. At 7% Fe₂O₃, absorbance remains high, but the peaks become sharper and more irregular, indicating the onset of particle agglomeration at this high concentration [15]. This causes variations in the material's microstructure, even though loss levels remain high. Generally, 3% and 5%

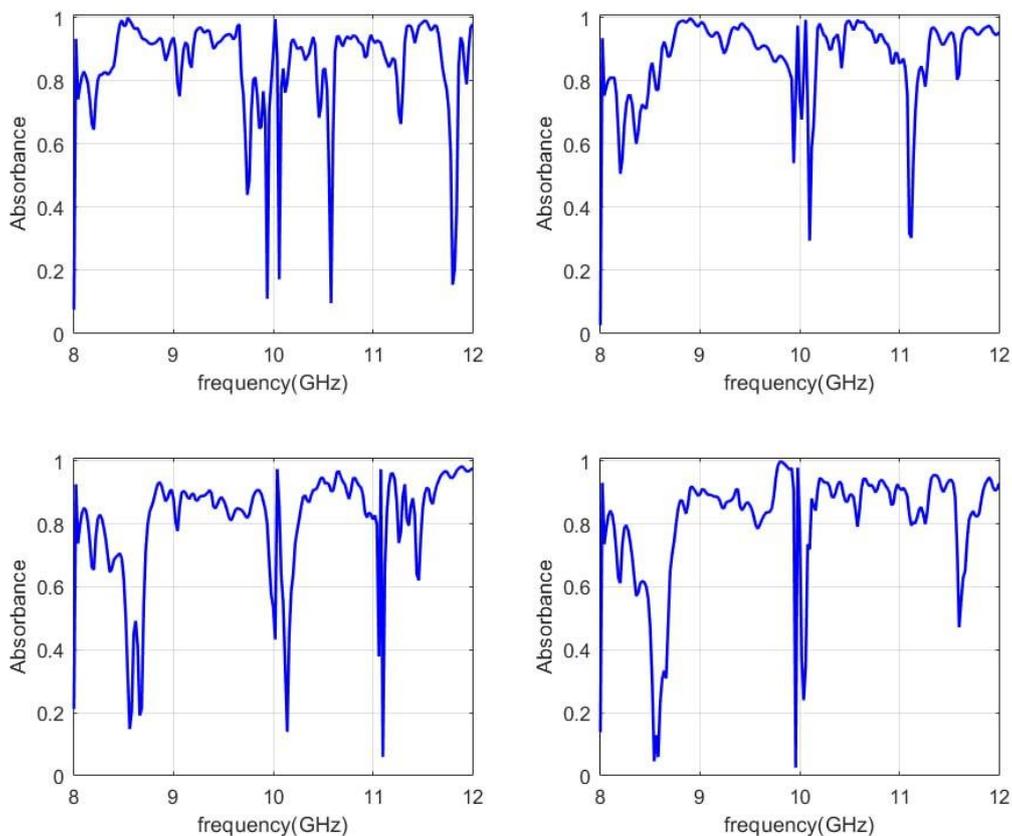


Fig. 5. The absorbance of the nanocomposite.



concentrations appear to achieve the most stable and efficient absorption, while 7% represents the threshold at which agglomeration problems begin. A 1% concentration is less effective due to the reduced amount of active material involved in the absorption mechanisms [7,23].

CONCLUSION

This research aimed to study the effect of adding iron oxide (Fe₂O₃) particles in different proportions to epoxy resin on both its mechanical and absorption properties in the X-band. The results of the mechanical tests showed that the average addition percentages, particularly 3% and 5% Fe₂O₃, achieved the best balance between the material's stiffness and strength. Young's modulus and stiffness improved due to the good dispersion of the particles within the matrix, and the composite's ability to absorb impact energy increased by inhibiting crack propagation and enhancing stress transfer. However, a 7% addition percentage showed a deterioration in some properties due to the formation of agglomerates, which reduced the material's homogeneity and increased its brittleness.

Regarding electromagnetic absorption, all samples exhibited a strong response within the 8–12 GHz range, with the 3% and 5% ratios demonstrating superiority. These ratios exhibited deeper and more regular absorption peaks, reflecting an effective balance between dielectric loss, magnetic loss, and impedance compatibility. Absorption stability decreased at the 7% ratio due to agglomeration, and at the 1% ratio due to a lack of active particles.

Overall, the results indicate that the addition of Fe₂O₃ at carefully considered ratios can simultaneously enhance the mechanical and absorption properties of epoxy. The 3%–5% ratio represents the optimal range for applications requiring good mechanical performance and high absorption efficiency, making these composites promising candidates for wave-absorbing and radar applications.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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