

EFFECT OF HEMATITE NANOFILLER ON THE ATTENUATION OF PTFE/Fe₂O₃ NANOCOMPOSITES AT MICROWAVE FREQUENCY

Bello Murtala Alhaji^{1,2}, Raba'ah Syahidah Azis^{1,3}, Muhammad Kashfi Shabdin¹, Nurul Huda Osman¹, Abubakar Yakubu⁴

¹*Department of Physics, Faculty of Science
University Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia*

²*Department of Physics
Shehu Shagari College of Education, Sokoto, P.M.B. 2129, Sokoto, Nigeria*

³*Nanomaterials Synthesis and Characterization Laboratory (MSCL), Institute of
Nanoscience and Nanotechnology (IONS2), Universiti Putra Malaysia, 43400 UPM
Serdang, Selangor, Malaysia*

⁴*Department of Physics, Kebbi State University of Science and Technology
Aliero, Nigeria*

**Corresponding author, Email: rabaah@upm.edu.my*

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ABSTRACT

The use of nanocomposites in microwave applications has received considerable attention because of their exceptional electromagnetic properties. This study investigates the effect of incorporating hematite (Fe₂O₃) nanofillers on the attenuation properties of polytetrafluoroethylene (PTFE)/Fe₂O₃ nanocomposites at microwave frequencies. The nanocomposites were prepared by dispersing hematite nanoparticles of 5, 7.5, 10, 12.5, and 15 % within a PTFE matrix using the dry mixing method. The fabricated nanocomposites were characterized for their morphological and electromagnetic properties. The results showed a uniform dispersion of hematite nanoparticles within the PTFE matrix which was confirmed by field emission scanning electron microscopy (FESEM). The attenuation of the PTFE/Fe₂O₃ nanocomposite substrate with varying percentages of Fe₂O₃ nanofiller was determined using the rectangular waveguide (RWG) method. The measurement involved analyzing the magnitudes of the transmission coefficient (S₂₁) obtained from the waveguide. The results indicated that as the content of Fe₂O₃ nanofiller was increased, the attenuation also increased. The composite with 15% PTFE/Fe₂O₃ exhibited the highest attenuation with a magnitude of 14.09 dB, while the composite with 5% Fe₂O₃ nanofiller showed the lowest attenuation with a magnitude of 12.40 dB. These findings offer valuable insights for the development of PTFE/Fe₂O₃ nanocomposites in applications requiring effective microwave attenuation, such as electromagnetic shielding and absorption devices.

Keywords: Nanocomposites; microwave attenuation; hematite nanofillers; polytetrafluoroethylene (PTFE); electromagnetic properties

INTRODUCTION

Nanocomposites have garnered significant interest in recent years because of their unique characteristics and potential applications in various fields. These materials include a matrix phase integrated with nanoscale fillers, resulting in enhanced thermal, electrical, mechanical, and electromagnetic properties [1]. In particular, nanocomposites have shown great promise in microwave applications, where precise control of electromagnetic properties is crucial for efficient device performance [2]. Understanding the behavior of materials in an electromagnetic field is crucial, particularly in electronics, communication, military hardware, and industrial applications, including shielding [3]. Measuring the transmission coefficient of materials in the microwave frequency range is common in various fields. It is essential to have a comprehensive understanding of transmission coefficient measurement to accurately assess the attenuation properties of materials for effective microwave absorption.

Polytetrafluoroethylene (PTFE) is a widely used matrix material in various applications, including composites. PTFE is a type of fluoropolymer known for its exceptional chemical resistance, high thermal stability, low coefficient of friction, and excellent electrical insulation properties [4,5,6]. These unique characteristics make it highly suitable for a wide variety of industries, such as aerospace, automotive, electronics, and medical [7]. As a matrix material, PTFE provides several advantages. Its low dielectric constant and loss tangent make it an excellent choice for applications requiring minimal signal loss and distortion, particularly in high-frequency and microwave systems. PTFE also offers excellent mechanical strength and dimensional stability, which contribute to the durability and long-term performance of composites.

The use of nanofillers in polymer matrices offers advantages in achieving desirable electromagnetic properties [8]. Nanoparticles modify the dielectric and magnetic behavior, enhancing electromagnetic wave absorption. Hematite (Fe_2O_3) nanoparticles are promising fillers for microwave absorption due to their high magnetic susceptibility and resonance frequency in the microwave range [9]. Incorporating hematite nanoparticles into a PTFE matrix is expected to improve microwave absorption properties. Previous studies have demonstrated improved microwave absorption by incorporating Fe_2O_3 nanoparticles into various polymer matrices. For instance, Shamsaddin Saeed et al. [10] investigated $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4/\text{PANI}/\text{MWCNT}$ nanocomposite and reported high microwave absorbing performance. Similarly, Manjappa et al. [11] studied $\alpha\text{-Fe}_2\text{O}_3$ and MWCNT/Graphene in LDPE-Based Composites and observed improved absorption in the microwave frequency range. Building upon these studies, our research aims to investigate the impact of hematite nanofiller on the attenuation properties of PTFE/ Fe_2O_3 nanocomposites at microwave frequencies. By varying the proportion of hematite nanoparticles, we will evaluate their effect on the microwave absorption characteristics of the nanocomposites.

EXPERIMENTAL DETAILS

Synthesis of hematite nanoparticles and PTFE/ Fe_2O_3 nanocomposites preparation

The hematite Fe_2O_3 was obtained from industrial chips known as “mill scale”. The mill scale was processed first by weighing and crushing for 24 h using conventional milling. Subsequently, the grinded powder underwent purification through the Magnetic Separation Technique (MST) and Curie Temperature Separation Technique (CTST), as reported previously [11,12,13]. The resulting FeO powder was dried for 24 h in an oven at 65 °C before being oxidized at 600 °C for 6 h to generate hematite Fe_3O_4 powder. To

achieve nano-sized particles, the hematite powder was subjected to high-energy ball milling (HEBM) using SPEX D8000 for durations of 9 h, respectively.

As illustrated in Figure 1, the PTFE/Fe₂O₃ nanocomposites were prepared by incorporating PTFE powder with different mass ratios of Fe₂O₃ nanoparticles. The PTFE and Fe₂O₃ composites were synthesized using a dry powder technique. The mixing process involved blending the Fe₂O₃ nanofiller with PTFE for 10 min using a blender/wing mixer. The content of Fe₂O₃ in each composite was incrementally increased by 2.5% from 5 to 15%. The resulting compositions were then pressed for 5 min under a pressure of 10 MPa using a hydraulic press. After pressing, the composite samples were heated in a furnace by 3 °C per minute until reaching a temperature of 380 °C. The samples were maintained at this temperature for at least 1 h to allow proper coalescence of the PTFE matrix particles and eliminate any voids [14]. Sintering was carried out using a furnace from Nabatherm GmbH in Lilienthal, Germany. The cooling process from 380°C to room temperature was conducted at a rate of 1.0 °C per min. The sample used in the rectangular waveguide has dimensions of 0.114 cm by 0.228 cm.

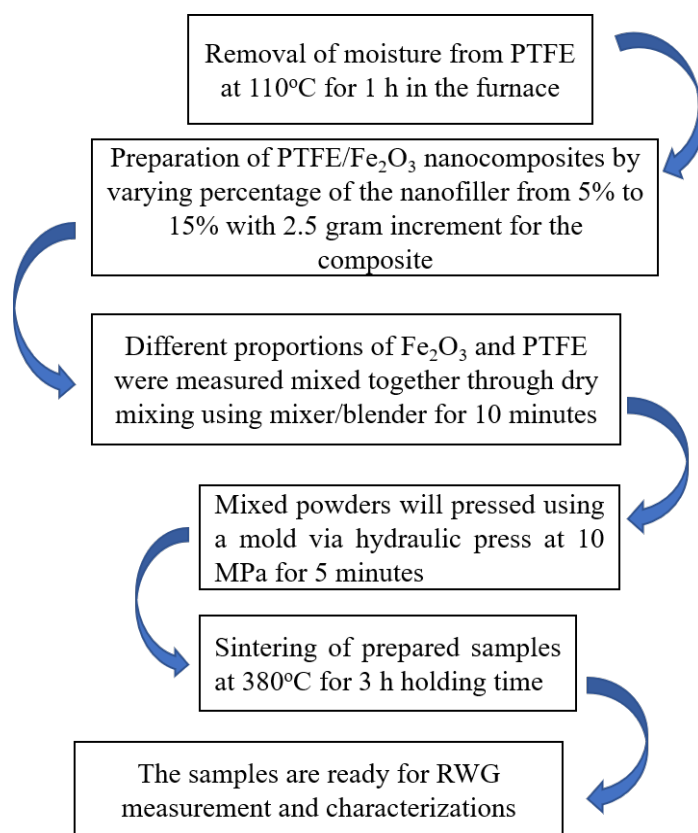


Figure 1. Preparation of PTFE/Fe₂O₃ nanocomposites

Characterization of PTFE/Fe₂O₃ nanocomposites

The surface topology of hematite Fe₂O₃/PTFE nanocomposites was analyzed using a field emission scanning electron microscope, and the dielectric properties, transmission (S_{21}), reflection (S_{11}), and attenuation of the composites were evaluated at 8-12 GHz employing a rectangular waveguide (RWG) measurement technique. A vector network analyzer (N5227A) from Agilent Technologies, USA, Santa Rosa, was attached to the RWG adapters for the measurement.

The FEM analysis procedures involved the following steps: (a) the solution region is divided into a finite number of elements or sub-regions; (b) formulating the governing equations for each element; (c) combining the elements to form the overall solution domain; and (d) solving the resulting system of equations to obtain the solution. The values of complex permittivity of the PTFE/Fe₂O₃ nanocomposites were obtained through measurements and utilized as computation inputs at the frequency range of 8 to 12 GHz (X-band). By analyzing the samples' transmission coefficients, the attenuation resulting from material absorption was determined. The deduced attenuation values were derived from the calculated magnitude of S₂₁ using the following formula:

$$\text{Attenuation (dB)} = -20\log(|S_{21}|) \quad (1)$$

RESULTS AND DISCUSSION

The dielectric constant increase is attributed to the enhanced interfacial polarization caused by the presence of interfacial lattice disorder in the PTFE and Fe₂O₃ nanofiller, resulting in a higher dielectric constant as the particle sizes decrease [15]. Another reason behind the increase in the dielectric constant is the compactness observed in smaller particle sizes, which reduces the presence of air gaps and promotes better interaction among the constituent particles, as shown in Figure 2, resulting in a rise in surface density. The interfacial polarization and increased surface density contribute to an overall increment in the dielectric constant. Conversely, the reduction in dielectric constant with an increase in frequency from 8 to 12 GHz is consistent with materials exhibiting interfacial polarization [16].

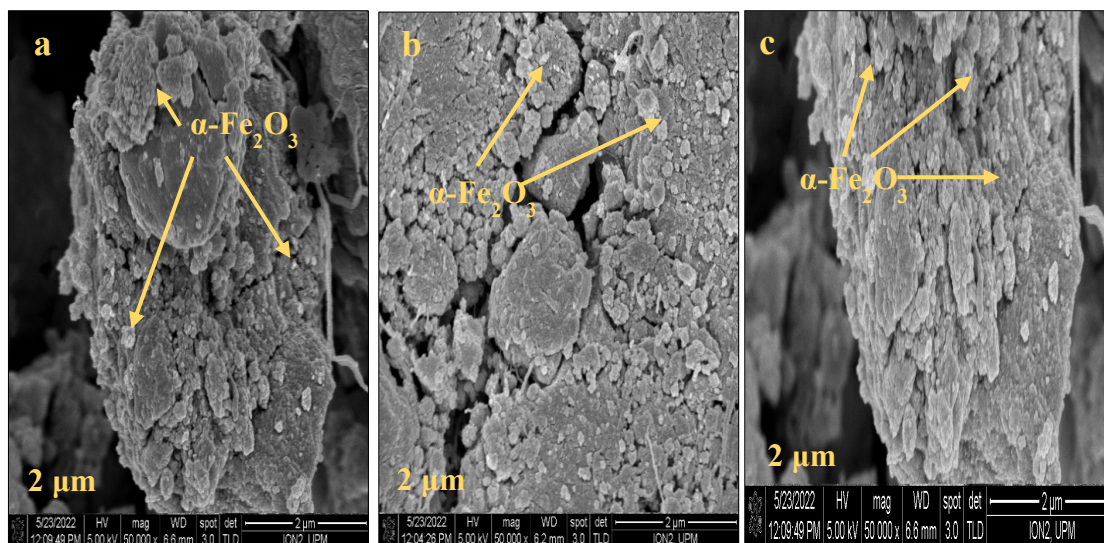


Figure 2. FESEM micrographs of (a) 5 %, (b) 10 %, and (c) 15% Fe₂O₃ for PTFE/Fe₂O₃ nanocomposites

The results confirm that composites with 5% mass content exhibit the lowest loss, which aligns well with the findings for the dielectric constant (Figure 3(a)). Figure 3(b) depicts the loss factor, which represents the propagated wave energy loss through the composites. Furthermore, the dielectric measurement results confirm the increase in mass percentage of Fe₂O₃ nanofiller within the polymer matrix, in which the composite material with the highest mass percentage shows the highest dielectric constant among the investigated composites.

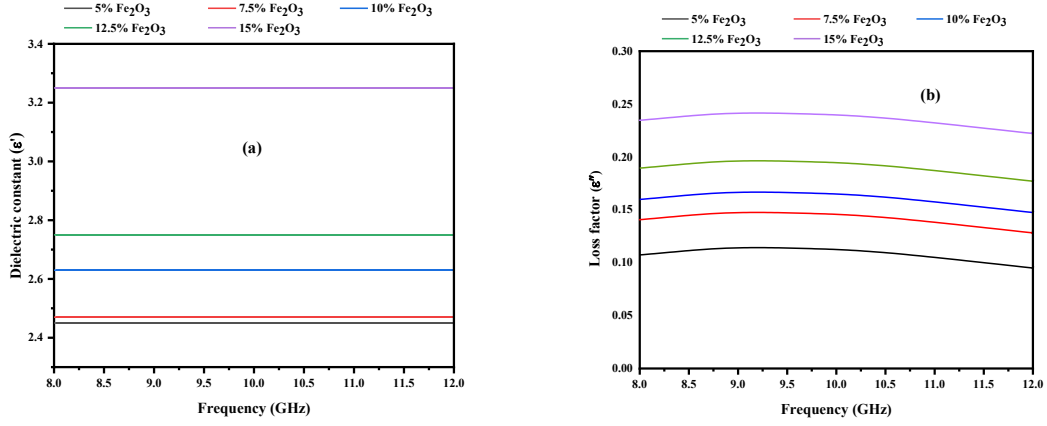


Figure 3. Variation in (a) dielectric constant, and (b) loss factor of PTFE/Fe₂O₃ nanocomposites

The measured results of S_{11} and S_{21} using the VNA for PTFE/Fe₂O₃ composites with mass percentages of 5, 7.5, 10, 12.5, and 15% are depicted in Figure 4. The findings from the measurements indicate that the transmission coefficient surpasses the reflection coefficient for all composites. For the 5, 7.5, 10, 12.5, and 15% composites, the mean value of S_{21} was calculated to be 0.8796, 0.8795, 0.8710, 0.8627, and 0.8591, while S_{11} was found to be 0.3345, 0.3355, 0.3290, 0.3460, and 0.3347, respectively. These findings suggest a dependency of the transmission coefficient on the sample's nanofiller percentage, indicating that as the nanofiller increases, the transmission coefficient decreases. Furthermore, according to the impedance mismatch theory, there is an inverse relationship between the magnitude of S_{21} and the dielectric properties, indicating that higher dielectric properties result in lower S_{21} values [17]. The relationship between S_{21} , attenuation, and nanofiller percentage is presented in Table 1 and graphically represented in Figure 5.

Table 1. Transmission coefficient (S_{21}) and attenuation of PTFE/Fe₂O₃ at different mass percentage

Fe ₂ O ₃ nanofiller (%)	S_{21}	Attenuation (dB)
5	0.9279	12.40
7.5	0.9207	13.06
10	0.9196	13.40
12.5	0.9156	13.64
15	0.9087	14.09

The graph demonstrates that the 5% Fe₂O₃/PTFE composite exhibits an attenuation of 12.40, while the 7.5, 10, 12.5, and 15% PTFE/Fe₂O₃ composites show an attenuation of 13.06, 13.40, 13.64, and 14.09 dB. Notably, there was a noticeable and significant rise in attenuation with the increment in filler content. This increase in attenuation can be attributed to the reduction in particle size, which causes the specific surface area of Fe₂O₃ to increase. Consequently, there is enhanced interaction between the particles of the crosslinking agent and polymer chains, resulting in increasing attenuation as the percentage of Fe₂O₃ nanofiller increases [18,19].

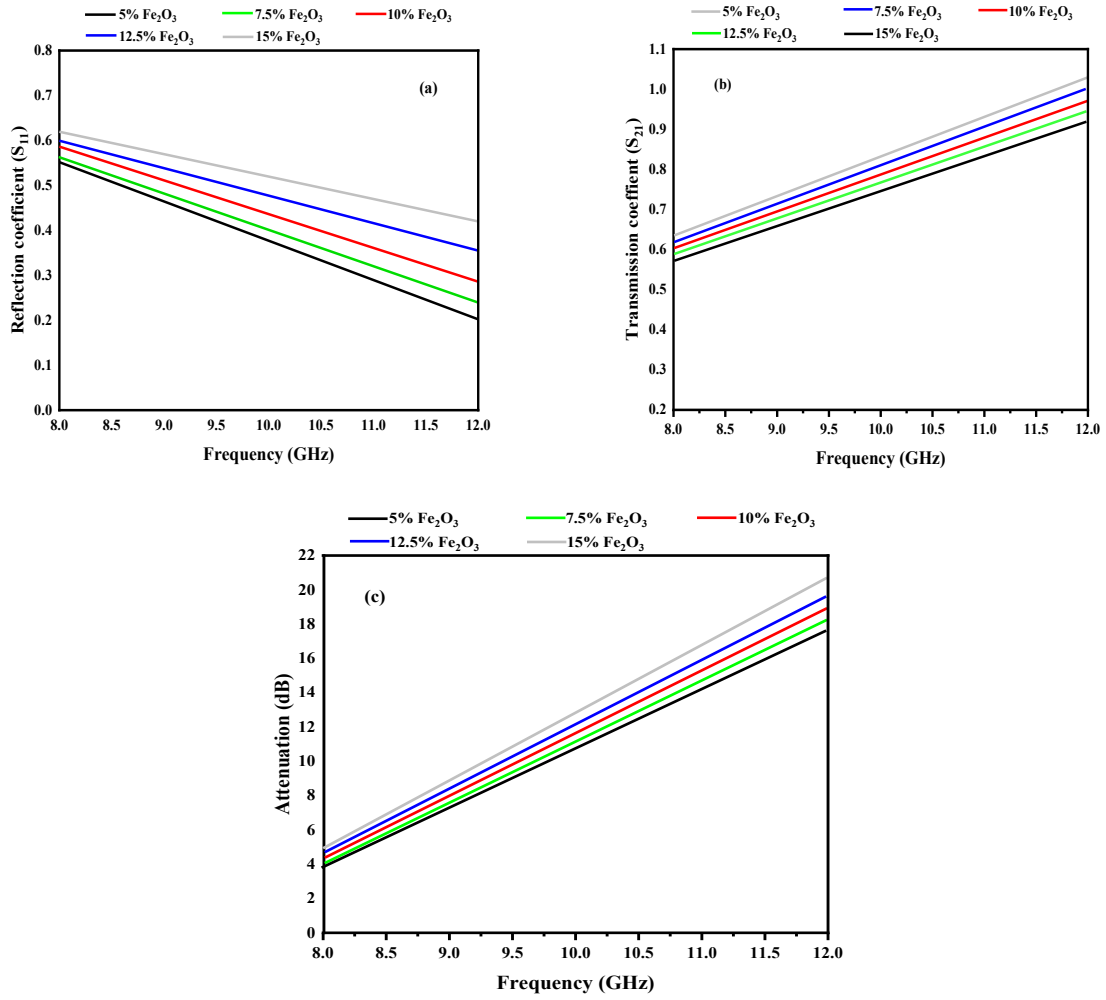


Figure 4. (a) Reflection coefficient measured for all the samples, (b) transmission coefficient measured for all the samples, and (c) calculated attenuation of various PTFE/Fe₂O₃ nanocomposites compositions

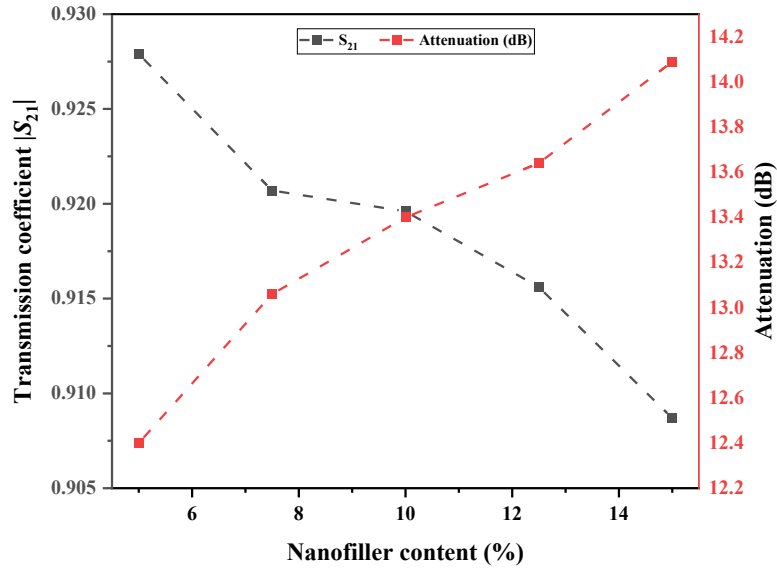


Figure 5. Transmission coefficient and attenuation vs. Fe₂O₃ mass percentage in PTFE/Fe₂O₃ nanocomposites

The electric field intensity in the PTFE/Fe₂O₃ composites was determined using the Finite Element Method (FEM) simulation. The simulation results for different sample mass percentages, namely 5, 7.5, 10, 12.5, and 15%, are presented in Figure 6. The rectangular waveguide used in the simulation is divided into three regions: Region I and Region III are surrounded by air, while Region II corresponds to the samples. The x -axis in the simulation geometry corresponds to the length of the waveguide spanning all three regions. Figure 3 depicts the plot of intensity against the amplitude of intensity for each region. To calculate attenuation based on the field intensity, two crucial variables were considered: Region I has the maximum incident intensity, while Region III has the maximum emergent intensity. In Table 2, representing the 5, 7.5, 10, 12.5, and 15% for the PTFE/Fe₂O₃ nanocomposites, the maximum incident electric intensity at Region I and Region III, having the maximum transmitted intensity, are recorded and summarized. Using the appropriate formula (1), the calculated attenuation for the PTFE/Fe₂O₃ composites with different mass percentages of Fe₂O₃ nanofiller at 12 GHz is shown in Table 2.

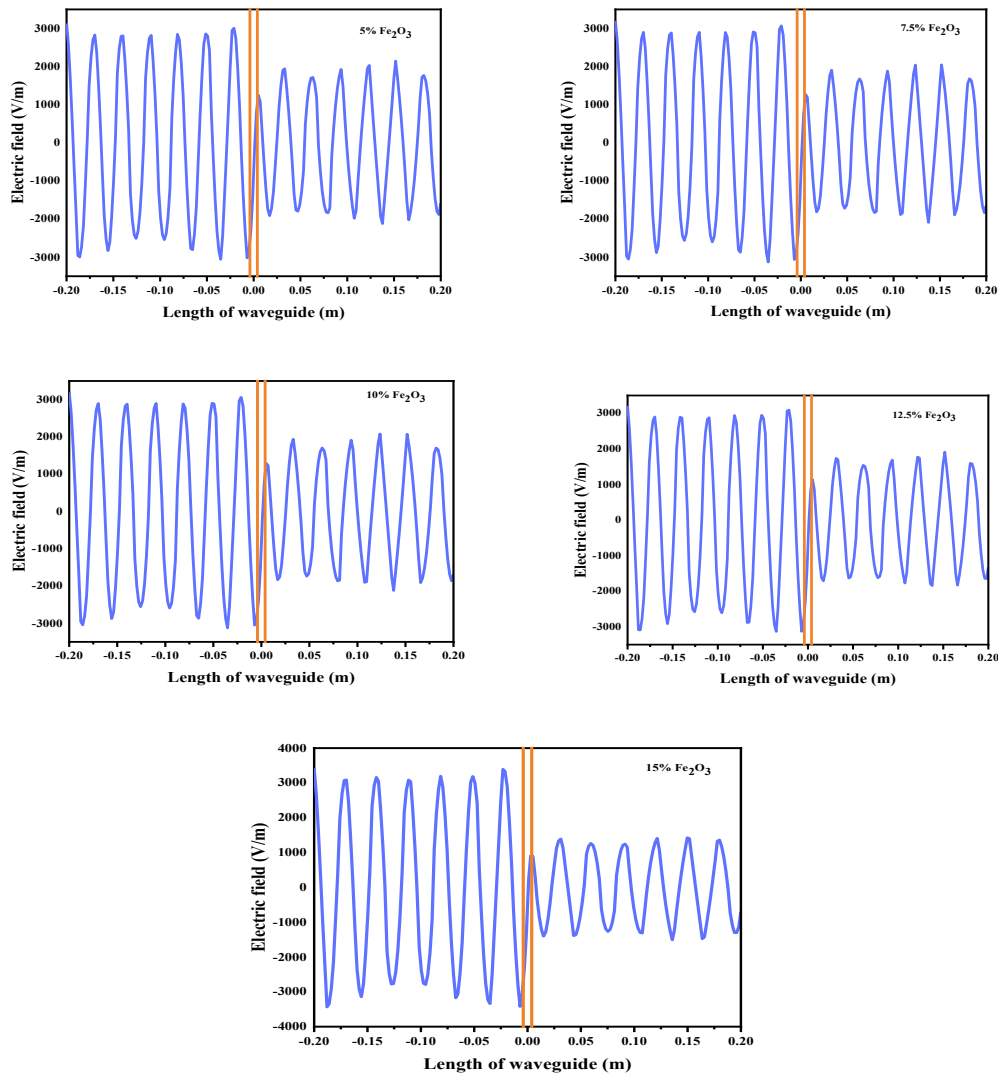


Figure 6. shows the electric field intensity at 12 GHz for PTFE/Fe₂O₃ nanocomposites with varying Fe₂O₃ mass percentages of 5, 7.5, 10, 12.5, and 15%

Table 2. The intensity of the incident peak in Region I and the intensity of the emerging peak in Region III for PTFE/Fe₂O₃ nanocomposites with varying Fe₂O₃ mass percentages of 5 to 15%

Fe ₂ O ₃ nanofiller (%)	Intensity (v/m)	Intensity (v/m)
	Region I	Region III
5	3130.09	1721.60
7.5	3200.68	1627.87
10	3195.23	1658.64
12.5	3209.62	1583.74
15	3424.96	1362.20

CONCLUSIONS

This study highlights the significance of nanocomposites in microwave applications and specifically focuses on the effect of incorporating hematite (Fe₂O₃) nanofillers on the attenuation properties of PTFE/Fe₂O₃ nanocomposites. Through the preparation and characterization of various nanocomposite samples with different percentages of Fe₂O₃ nanofiller, the study provides valuable insights into the relationship between nanofiller content and attenuation. The morphological analysis using field emission scanning electron microscopy (FESEM) confirms the uniform dispersion of hematite nanoparticles within the PTFE matrix, indicating a successful fabrication process. The measurement of attenuation using the rectangular waveguide (RWG) method reveals that increasing the content of Fe₂O₃ nanofiller leads to higher attenuation values. This observation suggests that the incorporation of hematite nanofillers enhances the microwave attenuation properties of the PTFE/Fe₂O₃ nanocomposites. The highest attenuation value was observed in the composite with 15% PTFE/Fe₂O₃ nanofiller, while the lowest attenuation was observed in the composite with 5% Fe₂O₃ nanofiller. These findings emphasize that such nanocomposites have potential applications in electromagnetic shielding and absorption devices, where the control and manipulation of microwave signals are crucial.

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