UNIVERSITI PUTRA MALAYSIA

HIGH SHIELDING EFFECT IN X-BAND MICROWAVE FREQUENCIES OF FERRITE-OPEFB-PTFE NANOCOMPOSITES

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By

AHMAD KHAMIS

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This thesis is dedicated to my parents, late uncle, siblings and beloved wife.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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Chairman : **Associate Professor Zulkifly Abbas, PhD**
Faculty : **Science**

Ferrites are the most common materials for microwave absorbing applications that widely used to eliminate undesired or stray radiated electromagnetic signals which could interfere with a system's operation. This project investigates using the recycled Ferrite (Fe_2O_3) jointly with biodegradable oil palm empty fruit bunch fiber (OPEFB) as fillers and Polytetrafluoroethylene (PTFE) as the host matrix for microwave shielding applications. Hematite (Fe_2O_3) fillers were recycled from the steel waste (mill scale waste) material and the particle sizes decreased to 11.3 nm after six hours of high energy ball milling. Four different batches of composites were fabricated as follows: Fe_2O_3-PTFE (batch 1), OPEFB-PTFE (batch 2), OPEFB-PTFE (batch 3), and Fe_2O_3-OPEFB-PTFE (batch 4) composites. The materials composition, complex permittivity, complex permeability, scattering parameters, density, structural, thermal, and tensile properties of prepared samples were investigated. The COMSOL software based on finite element method (FEM) was used to calculate the scattering parameters and visualize the electric field distribution in the nanocomposites. The coefficient of thermal expansion (CTE) of PTFE sample and 25%Fe_2O_3 (A) nanocomposite was respectively $65.28\times10^{-6}/\ ^{o}C$ and $39.84\times10^{-6}/\ ^{o}C$, therefore, the recycled Fe_2O_3 nanofiller enhanced the thermal properties of the nanocomposites. The density increased from 2.2 g/cm^3 to 2.54 g/cm^3 when the content of Fe_2O_3 increased from 5 wt.% to 25 wt.% while it decreased from 2.08 g/cm^3 to 1.5 g/cm^3 when OPEFB increased 5 wt.% to 25 wt.%. At 10 GHz, the complex permittivity of Fe_2O_3-PTFE nanocomposites increased from $(2.2 - j \times 0.10)$ to $(3.1 - j \times 0.22)$, while, the complex permeability increased from $(1.03 - j \times 0.017)$ to $(1.1 - j \times 0.038)$ when the percentage of Fe_2O_3 increased from 5% to 25%. The transmission coefficients $|S_{21}|$ of the nanocomposites decreased with increasing Fe_2O_3 content. The $|S_{21}|$ values of Fe_2O_3-OPEFB-PTFE nanocomposites ranged from 0.84 to 0.62, while, the values of Fe_2O_3-PTFE nanocomposites varied from 0.86 to 0.74 at 8.2 GHz. The $|S_{11}|$ values of Fe_2O_3-OPEFB-PTFE nanocomposites ranged from 0.52 to 0.63, while, the values of Fe_2O_3-PTFE nanocomposites varied from 0.50 to 0.62 at 8.2 GHz. The comparison between the measured and calculated scattering parameters showed a very good agreement. In addition, the total shielding effectiveness (SE) values increased with increasing the content of Fe_2O_3 nanofiller. At 10 GHz, the range of total SE values for Fe_2O_3-PTFE nanocomposites was from 12.8 dB to 18.3 dB, while, the range of Fe_2O_3-OPEFB-PTFE nanocomposites was from 15.6 dB to 19.6 dB. The prepared nanocomposites can therefore be used as promising alternatives for microwave shielding applications due to their shielding effectiveness, low density, low cost, and biodegradability. The recycled Fe_2O_3 nanoparticles can be used as fillers in polymeric composites for microwave shielding applications due to their low cost, good thermal stability and high shielding effectiveness.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KESAN KUAT PEMERISAIAN DALAM FREKUENSI MIKROGELOMBANG JALUR -X BAGI NANOKOMPOSIT FERIT-OPEFB-PTFE

Oleh

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Ferit adalah bahan yang sering digunakan sebagai penyerap mikrogelombang yang diguna secara meluas untuk menghapuskan isyarat sinaran elektromagnet sesat yang boleh mengganggu operasi sistem. Kajian ini menggunakan bahan kitar semula Ferit (Fe_2O_3) digabung dengan serat tandan buah kelapa sawit kosong (OPEFB) sebagai pengisi dan Politetrafluoroetilena (PFTE) sebagai matriks perumah bagi aplikasi pemerisaian mikrogelombang. Pengisi Hematit (Fe_2O_3) yang dikitar semula daripada bahan sisa keluli (sisa sisik besi) dan saiz zarah mengecil sehingga 11.3nm selepas enam jam pengisaran bola bertenaga tinggi. Empat kelompok komposit yang berlainan difabrik seperti berikut: komposit Fe_2O_3-PTFE (kelompok 1), OPEFB-PTFE (kelompok 2), OPEFB-PTFE (kelompok 3), dan Fe_2O_3-OPEFB-PTFE (kelompok 4). Kandungan bahan, ketelusan kompleks, kebolehtelapan kompleks dan parameter penyebaran, ketumpatan, struktur, haba dan sifat tegangan sampel telah dikaji. Perisian COMSOL berdasarkan kepada kaedah unsur terhingga (FEM) digunakan untuk mengira parameter serakan dan menggambarkan taburan medan elektrik dalam nanokomposit. Pekali pengembangan terma (CTE) bagi sampel PTFE dan nanokomposit 25%Fe2O3 (A) masing-masing adalah 65.28×10-6/ ^{o}C dan 39.84×10-6/ ^{o}C. Oleh itu, pengisi nano kitar semula Fe_2O_3 meningkatkan sifat terma nanokomposit. Ketumpatan meningkat daripada 2.2 g/cm^3 kepada 2.54 g/cm^3 ketika kandungan Fe_2O_3 meningkat daripada 5 wt.% kepada 25 wt.% sementara ia menurun daripada 2.08 g/cm^3 kepada 1.5 g/cm^3 ketika OPEFB meningkat 5 wt.% kepada 25 wt.%. Pada 10 GHz, ketelusan kompleks nanokomposit Fe_2O_3-PTFE meningkat daripada (2.2 – j × 0.10) kepada (3.1 – j × 0.22), manakala, kebolehtelapan kompleks meningkat daripada (1.03 – j × 0.017) kepada (1.1 – j × 0.038) apabila peratus Fe_2O_3 meningkat daripada 5% kepada 25%. Pekali pemancaran nanokompsoit $|S_{21}|$ menurun dengan peningkatan kandungan Fe_2O_3. Nilai $|S_{21}|$ nanokomposit Fe_2O_3-OPEFB-PTFE berjulat antara 0.84 hingga 0.62, manakala, nilai nanokomposit Fe_2O_3-PTFE berbeza daripada 0.86 hingga 0.74 pada 8.2 GHz. Nilai $|S_{11}|$ bagi nanokomposit Fe_2O_3-OPEFB-PTFE berjulat antara 0.52 hingga 0.63, manakala, nilai nanokomposit Fe_2O_3-PTFE berbeza daripada 0.50 hingga 0.62 pada 8.2 GHz. Perbandingan antara parameter serakan yang diukur dan dikira menunjukkan keseragaman. Tambahan lagi, jumlah nilai keberkesanan pemerisaian (SE) meningkat dengan peningkatan kandungan pengisi nano Fe_2O_3. Pada 10 GHz, jumlah julat nilai SE bagi nanokomposit Fe_2O_3-PTFE adalah dari 12.8 dB hingga 18.3 dB, manakala, julat nanokomposit Fe_2O_3-OPEFB-PTFE adalah dari 15.6 dB hingga 19.6 dB. Oleh itu, nanokomposit Fe_2O_3-OPEFB-PTFE boleh digunakan sebagai alternatif terbaik bagi aplikasi pemerisaian mikrogelombang disebabkan oleh keberkesanan perisainya, ketumpatannya yang rendah, kos rendah, dan keterbiodegradasikan. Nanozarah Fe_2O_3 yang dikitar semula boleh digunakan sebagai pengisi dalam komposit berpolimer bagi aplikasi pemerisaian mikrogelombang kerana ia mempunyai kestabilan haba yang baik, berkos rendah dan mempunyai keberkesanan perisai yang tinggi.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS AND SYMBOLS

OPEFB	Oil palm empty fruit bunch
OECP	Open ended coaxial probe
RWG	Rectangular waveguide
c	Velocity of light
ε^*	Complex permittivity
ε'	Permittivity (real part)
ε''	Permittivity (Imaginary part)
EMI	Electromagnetic interference
σ	Electrical conductivity
ε	Permittivity
μ	Permeability
μ'	Permeability (Real part)
μ''	Permeability (Imaginary part)
SE	Shielding effectiveness
dB	Decibels
FEM	Finite element method
XRD	X-ray diffraction
FESEM	Field emission scanning electron microscope
EDX	Energy-dispersive X-ray spectroscopy
HRTEM	High resolution transmission electron microscopy
T/R	Reflection/transmission
S_{11}	Input reflection coefficient of (port 1)

S_{12}	Transmission coefficient (port 1)
S_{22}	Input reflection coefficient of (port 2)
S_{21}	Transmission coefficient (port 2)
CTE	Coefficients of thermal expansion
TE	Transverse electric
TM	Transverse magnetic
VNA	Vector network analyzer
SE	Shielding effectiveness
EM	Electromagnetic
MST	Magnetic separation technique
CTST	Curie temperature separation technique
MAM	Microwave absorbing materials
MWCNT	Multiwall carbon nanotube

CHAPTER 1

INTRODUCTION

Chapter one contains six sections. The electromagnetic interference and absorbing materials are discussed in sections one and two, respectively. The third section focuses on the mechanism of materials interaction with microwaves. The fourth section presents the problem statements. Finally, the last two sections focus on this study objective and the study scope.

1.1 Introduction

The electromagnetic interference (EMI) which is commonly known as an electromagnetic (EM) pollution has increased to greater levels due to the quick development in the technology and electronic products. The demand to use the EM energy for different miniaturized and complex devices is still increasing in many important fields such as industrial, commercial, aerospace, and military. In addition, this increment in EM pollution is able to cause severe interference consequences and it also can negatively affect the near living beings and electric devices. Therefore, the electronic systems operations must be protected using an optimum shielding material. The use of lossy materials (dielectric or magnetic) is one of the solutions to solve the EMI problems because they can protect the electronic devices from the undesired and stray EM radiation by reflection and absorption (Maruthi et al., 2021).

In general, metals are considered the most conventional materials used to solve the EMI problems because they have an outstanding EMI shielding effectiveness (SE). Nevertheless, metals are generally expensive, rigid, heavy, and corrosive. The production of metal is high-priced and hard to process. In recent times, the conductive polymer composites are widely used as EMI shielding materials because of their lightweight, low cost, simple processability, strong resistance to corrosion, broad absorption and bandwidth properties comparing with conventional metal materials. In general, the polymer composites are simply fabricated by adding the conductive fillers into the conducting or insulating polymer matrix using a preferred fabrication method (Sankaran et al., 2018).

The microwave absorbing materials (MAMs) basically can attenuate the energy of electromagnetic (EM) waves or dissipate it via interference-effect or converting it into thermal energy. In addition, the electromagnetism parameters such as complex permeability and permittivity basically play a very significant role in the reflection and transmission of EM waves. The real parts of permeability and permittivity represent the storage capacity of the microwave energy in absorbing material, while, the imaginary parts of the aforementioned parameters symbolize the energy loss ability. Therefore, higher values of the imaginary parts of permittivity and permeability are usually expected to achieve better microwave absorbing performance. The ferrites have recently grabbed a great attention for their outstanding magnetic property, low cost remarkable chemical, and thermal stability. Many previous studies have revealed that the combination of magnetic and dielectric components can successfully improve the microwave absorbing performance of hybrids; therefore, the ferrites are usually composited with polymer to enhance the microwave absorption performance (Yin et al., 2020).

1.2 Absorbing Composites

The wide consumption of communication devices such as radar systems, local area network systems and telecommunications presently cause an enormous amount of EM energy everywhere including the living space of humans. The emission of EM waves can cause severe problems of EMI that not only can destroy the sensitive electronic apparatuses, but it also can affect physical health in an extraordinary negative manner. Many efforts are made on the EMI shielding applications to solve EMI problems which means to blockage the unwanted EM waves so that the waves cannot pass through the EMI shield. Nevertheless, the shielding materials cannot to totally dissipate the EM emission because of the reflection principle of the incident EM waves and these waves can reproduce repeated EMI pollution. The pragmatic and effective functions can be achieved using the MAMs with high absorption and low reflection, these type of materials have recently grabbed the attentions. It is because MAMs intrinsically dissipate EM waves through destructive interference or convert the EM energy into thermal energy. To fulfil the applications requirements, an eligible absorber should be designed with several characteristic features, including being thin and lightweight, with wide frequency bandwidth and powerful absorption. The microwave absorption and EMI shielding are two known strategies to resist the interference of incident EM signals, but they are evaluated with different measurement models because of their distinct concerns (Wang et al., 2017).

Since complex permittivity and permeability of the microwave absorbers usually play very important roles in controlling the properties of microwave absorption, two kinds of microwave absorbing materials are extensively investigated: the first one is materials with magnetic loss such as ferrite, the second one is dielectric loss materials like carbon-based materials. The improvements of microwave absorption materials with thin matching thickness and light weight are highly required (Li et al., 2015).

1.3 Interaction of Materials with Microwaves

The microwaves represent a section of the electromagnetic spectrum which move at the speed of light with a wavelength range from 1 mm to 1 m, which corresponds to the range of frequency from 300 GHz to 300 MHz (Sun et al., 2016). Moreover, Microwaves are electromagnetic waves which consist of a magnetic and electric fields orthogonal to each other (Mishra and Sharma, 2016).

The dielectric material can store the energy if an electric field (external) is applied. The polarizations can be caused by the orientation of the ions-atoms of the material and/or the tiny displacements of the ions-atoms. Therefore, the dielectric constant is an expressions of how a certain material is usually polarized (Webb, 2011)

The microwaves immediately interacted with the nuclei and atoms when they appeared in the merge of the universe for the first time. The microwaves can interact with molecules, nuclei, electrons, protons, atoms, and the clusters of molecules. Moreover, the microwaves can interact with all types of materials (in the macroscopic scale) such as dielectrics, clouds, gases, rocks, liquids, plasmas, metals, magnetic matter, and ionosphere.

The microwaves get absorbed, transmitted, and reflected. Moreover, the microwaves can cause a rotational excitation in the atoms and they also can make the dipoles of electric charges frenetically jiggle. The microwaves are able to heat the electric dipoles when they are part of a certain dielectric material. Moreover, microwaves can cause a rotation in the magnetic dipoles and they also can make a jump in the states of magnetic energy. However, free electrons can absorb microwaves in metallic objects.

In addition, microwaves can microscopically interact with materials through the atomic magnetic dipoles, atoms, and the conduction electrons. The microwaves impact on materials are macroscopically described by Maxwell equations and the electrodynamic characteristics of materials: σ (electrical conductivity), μ (magnetic permeability), and ε (electric permittivity) (Ulloa et al., 2019).

1.4 Problem Statement

Microwave materials with good absorption are in very high demand to solve the electromagnetic interference (EMI) problems in commercial and industrial electronics. Hematite is one of the most common materials usually used to solve EMI problems in electronic and microwave devices because of their excellent electromagnetic characteristic' at microwave frequency range. Nevertheless, hematite is often obtained using chemical methods that can be expensive, complicated, and multi-staged. Therefore, this study presents an effective technique to decrease the cost of hematite microwave shielding applications by using the recycled hematite with natural and biodegradable fiber. This technique includes the retrieval of hematite (Fe_2O_3) from the mill scale waste and the consequent enhancement of the loss factor and dielectric constant by decreasing the particles size into nano-size using high energy ball milling (HEBM). Polytetrafluoroethylene (Teflon or PTFE) is classified as a thermoplastic polymer, it is also considered as the greatest solvent and chemical resistant among the thermoplastics. The characteristics of Teflon such as chemical inertness, low moisture absorption and high operating temperature are very important for various microwave applications (Wu et al., 2013). Nevertheless, Teflon has particular drawbacks such as high coefficient of thermal expansion (CTE) (Murali et al., 2009) (Chen et al., 2003), low relative complex permittivity (ε^*) (Xie et al., 2017), and its total shielding effectiveness tends to zero (Al-Ghamdi et al., 2021). The recycled Fe_2O_3 powder have not been utilized as fillers in Teflon matrix for the microwave absorption applications. The recycled Fe_2O_3 can improve the thermal stability (Takeda et al., 2009), complex permeability and permittivity of PTFE matrix (Esa et al., 2015). Moreover, microwave absorbers should have high loss factor for better absorbing characteristics. In this study, OPEFB with 40 µm fiber size was embedded into Fe_2O_3-PTFE samples in order to enhance the absorption loss. OPEFB fibers are as lignocellulosic fibers where the cellulose and hemicellulose are reinforced in a lignin matrix similar to other natural fibers materials (Hassan et al., 2010). However, OPEFB is classified a waste of lignocellulosic agriculture which usually has a negative impact on the environment (Rosazley et al., 2016). OPEFB is conventionally composted to organic fertilizer, burned, or disposed of in land fields. Burning OPEFB is not a good solution and it is not recommended because it causes air pollution. Therefore, it is very crucial to optimally use OPEFB fiber in order to find solutions for the aforementioned problems and use the resource for valuable products at the same time (Ishola et al., 2012). OPEFB fiber is considered as a good potential to be highlighted in polymer composites because of its several advantages such as biodegradability, easy processing, relatively high in hardness, real strength, and low in density (Faizi et al., 2017). This research presents the fabrication and characterization of Fe_2O_3-PTFE, OPEFB-PTFE, and Fe_2O_3-OPEFB-PTFE composites for microwave shielding applications.

1.5 Research Objectives

The main objectives of this research are as follows:

1. To synthesize the Fe_2O_3 powder from the mill scale waste and decrease the particles size to nano-size using the high energy ball milling (HEBM) and characterize their structural properties.

2. To investigate the effect of nano-size Fe_2O_3 content on dielectric, magnetic, mechanical, structural, and thermal properties of Fe_2O_3-PTFE samples.
3. To investigate the impact of OPEFB percentage and fiber size on mechanical, structural, dielectric properties of OPEFB-PTFE composites
4. To determine the total shielding effectiveness and visualize the electric field distribution of Fe_2O_3-PTFE and Fe_2O_3-OPEFB-PTFE nanocomposites using rectangular waveguide technique.

1.6 The Scope of Study

In this research, Fe_2O_3 powder will be synthesized from mill scale waste and the particles size reduced into nanosize using HEBM technique for six hours. Four different batches of composites will be fabricated using recycled Fe_2O_3 nanofiller and OPEFB fiber as fillers and PTFE as a matrix. Fe_2O_3-PTFE nanocomposites (batch 1) will be fabricated with varying recycled Fe_2O_3 nanoparticles content (5-25 % wt.) in nanocomposites while OPEFB-PTFE composites (batch 2) will be fabricated with varying OPEFB content (5-25 % wt.) in composites. OPEFB-PTFE composites (batch 3) will be fabricated using different particle sizes of OPEFB (40 μm, 106 μm, 150 μm, 180 μm, and 250 μm). Moreover, Fe_2O_3-OPEFB-PTFE (batch 4) will be fabricated depending on different recycled Fe_2O_3 content (5-25 % wt.) and constant percentage of OPEFB (5% wt.). The complex permittivity of all batches will be determined using the open ended coaxial probe (OECP) and the rectangular waveguide (RWG) respectively. The complex permeability, reflection $|S_{11}|$ and transmission $|S_{21}|$ coefficients of batch 1 and 4 will be determined using the rectangular waveguide. The frequency ranges of OECP and RWG measurements will be 1-12 GHz and 8.2-12.4 GHz, respectively. The microstructural and morphological measurements of the samples will be carried out using techniques such as HRTEM, XRD, FESEM, and EDX.

The COMSOL software based on finite element method (FEM) will also be used to calculate the reflection and transmission coefficients and also to simulate the electromagnetic waves propagated through the samples. The results of $|S_{21}|$ and $|S_{11}|$ obtained from measurements and simulation will be compared. Error analysis of the comparison between simulation and measurements will be determined. Finally, the visualization of electric field in the samples will be conducted using COMSOL software in order to provide a clear understanding about the material's interaction with electromagnetic waves.

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