SYNTHESIS AND ELECTROMAGNETIC CHARACTERIZATION OF POLYCAPROLACTONE FILLED WITH HEMATITE AND OPEFB FIBER NANOCOMPOSITE

MENSAH, EBENEZER EKOW

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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 the Almighty God, my beloved wife and children

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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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July 2019

Chairman : Associate Professor Zulkifly Abbas, PhD
Faculty : Science

The most common materials used for microwave absorbing applications are ferrites. However, ferrites are expensive, heavy, non – biodegradable and have low dielectric loss properties especially at high frequencies. This study presents the development of novel composites using recycled ferrite in conjunction with biodegradable oil palm empty fruit bunch (OPEFB) fiber and polycaprolactone (PCL) as an alternative for reducing the limitations of ferrite – based microwave absorbing materials. Hematite ($\alpha - Fe_2O_3$) was recycled from mill scale waste (steel waste) material and the particle sizes reduced to nanosize after several hours of high energy ball milling (HEBM). The relationship between the reduced particle sizes and the dielectric properties was then determined. $\alpha - Fe_2O_3$/PCL and $\alpha - Fe_2O_3$/OPEFB fiber/PCL nanocomposites with different loadings (5 to 25%) of 16.2 nm $\alpha - Fe_2O_3$ nanofiller were fabricated and characterized for their dielectric, magnetic and microwave absorption properties. The material composition and structural properties were analyzed using X – ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X – ray spectroscopy (EDX), high resolution transmission electron microscopy (HRTEM), Fourier transform infrared spectroscopy (FTIR) and Brunauer – Emmett – Teller (BET) techniques. The relative complex permittivity and permeability of the samples were respectively measured using the open – ended coaxial probe and the rectangular waveguide techniques while the microwave absorption properties were measured with the microstrip at 1 GHz to 4 GHz. The results showed that the relative complex permittivity of the recycled $\alpha - Fe_2O_3$ increased with reduced particle size. The dielectric loss factor (ε'') increased from 0.17 to 0.46 when the particle size was reduced from 1.73 μm to 16.2 nm at 8 GHz. Within the X – band (8 GHz – 12 GHz) frequency range, the relative complex permittivity properties of the recycled $\alpha - Fe_2O_3$ particles were higher as compared to a commercial $\alpha - Fe_2O_3$ (Alfa Aesar). Additionally, the relative complex permittivity (ε^*) values of the nanocomposites increased with recycled $\alpha - Fe_2O_3$ nanofiller content and were higher in the α–

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Fe_2O_3/OPEFB/PCL nanocomposites than the α – Fe_2O_3/PCL nanocomposites. This is due to the high loss factor of the incorporated OPEFB fiber.

Attenuation and power loss due to absorption equally increased with recycled α – Fe_2O_3 nanofiller loadings. At 2.4 GHz, the range of attenuation for the α–Fe_2O_3/OPEFB/PCL nanocomposites was from 2 dB to 2.6 dB while the power loss values were from 15 dB to 17.3 dB. The attenuation values for the α – Fe_2O_3/PCL nanocomposites were however from 1.8 dB to 2 dB while the power loss values were in the range of 13.6 dB to 15.2 dB. The recycled α–Fe_2O_3/OPEFB/PCL nanocomposites can therefore serve as promising alternatives for microwave absorbing applications in the 1 – 4 GHz in view of their low cost, low density, biodegradability and attractive absorption behaviour. Recycled hematite at reduced particle size has the potential for use as a filler in other polymeric composites and its application can reduce the cost of ferrite – based microwave absorbing materials significantly without compromising the absorption efficiency of the materials.

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

SINTESIS DAN PENCIRIAN ELEKTROMAGNET POLIKAPROLAKTON BERISI HEMATIT DAN OPEFB SERAT KOMPOSIT NANO

Oleh

MENSAH, EBENEZER EKOW

Julai 2019

Pengerusi : Profesor Madya Zulkifly Abbas, PhD
Fakulti : Sains

Bahan yang kerap digunakan untuk aplikasi penyerapan mikro gelombang adalah ferit. Walau bagaimanapun, ferrite adalah mahal, berat, tidak terbiodegradasikan dan mempunyai sifat kehilangan dielektrik yang rendah terutama pada frekuensi tinggi. Kajian ini membentangkan mengenai pembangunan satu novel komposit yang menggunakan ferit yang dikitar semula sehubungan dengan serat tandan buah kelapa sawit yang biodegradasi (OPEFB) dan polikaprolakton (PCL) sebagai alternatif untuk mengurangkan keterbatasan bahan penyerapan mikro gelombang berasaskan ferit. Hematit (α - Fe_2O_3) telah didapati daripada bahan sisa skala kilang (sisa buangan keluli) boleh dikitar semula dan saiz zarah telah dikurangkan menjadi nano-saiz selepas beberapa jam melalui proses penggilingan bola tenaga yang tinggi (HEBM). Hubungan antara saiz zarah yang dikurangkan terhadap sifat dielektrik telah ditentukan. α - Fe_2O_3 / PCL dan α - Nanoplastik Fe_2O_3 / OPEFB / PCL nanokomposit dengan berisi α - Fe_2O_3 nanofiller yang berbeza muatan (5 hingga 25%), yang difabrikkan dan dicirikan sifat dielektrik, magnetik dan sifat penyerapan gelombang mikro. Komposisi bahan dan sifat struktur dianalisis dengan menggunakan pembelauan sinar-X (XRD), pengimbasan mikroskop elektron (SEM), spektroskopi serakan tenaga sinar-X (EDX), mikroskop penghantaran elektron resolusi tinggi (HRTEM), transformasi Fourier spektroskopi inframerah (FTIR) dan Brunauer - Emmett-Teller (BET). Ketulusan kompleks dan kebolehtelapan sampel diukur dengan menggunakan proba sepaksi hujung terbuka dan pandu gelombang segi empat tepat sementara sifat penyerapan gelombang mikro diukur dengan menggunakan mikrostrip pada 1 GHz hingga 4 GHz. Kadar ketelusan dielektrik oleh α - Fe_2O_3 yang dikitar semula meningkat apabila saiz zarah yang dikecilkan. Kadar faktor kehilangan dielektrik (ε") meningkat dari 0.17 ke 0.46 apabila saiz zarah dikecilkan daripada 1.73 μm hingga 16.2 nm pada 8 GHz. Dalam jalur-X julat frekuensi (8 GHz – 12 GHz), sifat ketelusan kompleks relatif bagi zarah α - Fe_2O_3 yang dikitar semula adalah tinggi berbanding dengan α - Fe_2O_3 komersial (Alfa Aesar). Tambahan pula, nilai ketelapan dielektrik (ε*) nano-komposit meningkat dengan kandungan kitar semula terisi nano

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(nanofiller) α – Fe_2O_3 dan lebih tinggi bagi sampel α – Fe_2O_3/OPEFB/PCL nano-komposit, berbanding α–Fe_2O_3/PCL nanokomposit. Ini disebabkan oleh faktor kehilangan yang tinggi yang disebabkan oleh penggabungan serat OPEFB.

Atenuasi dan kehilangan kuasa disebabkan oleh penyerapan meningkat dengan muatan terisi nano α – Fe_2O_3 kitar semula. Pada 2.4 GHz, julat attenuasi untuk α-Fe_2O_3 /OPEFB /PCL nanokomposit adalah daripada 2 dB hingga 2.6 dB manakala nilai kehilangan kuasa adalah daripada 15 dB hingga 17.3 dB. Nilai atenuasi untuk α-Fe_2O_3/PCL nanokomposit adalah daripada 1.8 dB hingga 2 dB manakala nilai kehilangan kuasa berada dalam lingkungan 13.6 dB hingga 15.2 dB. Kitar semula α–Fe_2O_3/OPEFB/PCL nanokomposit dengan ini boleh digunakan sebagai satu alternatif sebagai penyerap gelombang mikro beraplikasi dalam julat frekuensi 1-4 GHz, sebagai bahan kos murah, ketumpatan rendah, terbiodegradasikan and mempunyai sifat penyerapan yang menarik. Kitar semula hematit dengan saiz zarah yang mengecil mempunyai potensi digunakan sebagai 'filler' (pengisi) dalam komposit polimer dan aplikasi ini boleh mengurangkan kos bahan penyerap gelombang mikro berasaskan ferit, paling penting, tidak berkompromis dengan kecekapan penyerapan bahan ini.

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TABLE OF CONTENTS

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LIST OF TABLES

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LIST OF FIGURES

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LIST OF ABBREVIATIONS

EMI	Electromagnetic interference
EMC	Electromagnetic compatibility
OPEFB	Oil Palm Empty Fruit Bunch
$\alpha - Fe_2O_3$	Hematite
PCL	Polycaprolactone
OECP	Open ended coaxial probe
RWG	Rectangular waveguide
XRD	X-ray diffraction
EDX	Energy dispersive x – ray
FESEM	Field emission scanning electron microscopy
HRTEM	High resolution transmission electron microscopy
BET	Brunauer – Emmett – Teller
FEM	Finite element method
NRW	Nicholson Ross Weir
FDM	Finite difference method
MoM	Method of moment
FDTD	Finite difference time domain
PNA-L	Professional network analyzer
VNA	Vector network analyzer
TRL	Thru, Reflect, Line
RF	Radio frequency
FWHM	Full wave half maximum
ECAL	Electronic calibration
SMA	Sub-miniature
TE	Transverse electric

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TM	Transverse magnetic
TEM	Transverse electromagnetic mode
T/R	Transmission/Reflection
S_{11}	Reflection coefficient
S_{21}	Transmission coefficient
ε_r	Complex permittivity
μ_r	Complex permeability
ε'	Dielectric constant
ε''	Loss factor
PTFE	Polytetrafluoroethylene (Teflon)
ICSD	Inorganic crystal structure database
FTIR	Fourier transform infrared spectroscopy
dB	the decibel
TRM	True, Reflect, Match
LRL	Line, Reflect, Line

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CHAPTER 1

INTRODUCTION

The recent rapid development of the electronics industry in the microwave frequency domain has intensified the electromagnetic interference (EMI) issue among devices having both civilian and military applications. EMI refers to stray or unwanted electromagnetic signals found within 300 MHz and 300 GHz, which are radiated and/or conducted from sources such as wireless networks and devices, mobile phones, car alarms, USB devices, FM/AM radio antennas and digital TV emitters. EMI has the potential to cause errors, system failures, malfunctions or other faults in neighboring electronic equipment and installations. As a result, EMI absorbing materials are needed to deliver electromagnetic compatibility (EMC) in various electronic devices operating in the microwave region to improve on their quality and reliability. In recent years, much attention has been focused on ferrite materials (spinel, garnet and hexagonal ferrites) for EMI absorption applications due to their superior saturation magnetization, reduced electrical losses, large electrical resistivity as well as chemical stability. Ferrites such as zinc ferrite (Raju, 2017), cobalt ferrite (Li et al., 2017), magnetite (Zhang et al., 2018) and La – Ni substituted barium ferrite (Shen et al., 2017), and many others have been used in composites which have shown improved microwave absorption over a wide frequency range. However, ferrite absorbers lack the right balance of low density, low cost, high dielectric loss performance, thinness, strong mechanical properties, and biodegradability. Subsequently, the development of new techniques to reduce the limitations of ferrites has received a lot of attention in the last few years. Nevertheless, the utilization of carbon bio – based materials, recyclable materials and biodegradable materials has received very little focus in spite of their economic and environmental benefits. This research therefore presents a novel technique to reduce the limitations of ferrite – based microwave absorbing materials using low – cost recycled $\alpha - Fe_2O_3$ together with biodegradable materials. The focus is on the effectiveness of particle size reduction by the ball milling technique in enhancing the complex permittivity characteristics of the recycled $\alpha - Fe_2O_3$ as well as the incorporation of OPEFB fiber and polycaprolactone matrix for the fabrication of novel nanocomposites for microwave absorbing applications within the L – S (1 – 4 GHz) frequency range.

1.1 Problem Statement

Ferrites are the most common materials used for solving EMI problems in microwave and electronic devices due to their excellent electromagnetic properties at microwave frequencies. However, ferrites are often synthesized through chemical techniques which could be multi – staged, complicated and expensive. Moreover, ferrites also have low dielectric loss properties especially at high frequencies and are non – biodegradable. This research therefore presents a new method to reduce the cost of ferrite – based microwave absorbing materials by using recycled ferrite in conjunction with biodegradable materials. The method involves the retrieval of hematite (α – Fe_2O_3) from recyclable mill scale waste and the subsequent improvement of the

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dielectric properties by particle – size reduction to nanosize using high energy ball milling. The relationship between the particle – size reduction and the dielectric properties of the recycled α – Fe_2O_3 particles was then established. The recycled α – Fe_2O_3 with improved dielectric properties could be useful in reducing the limitations of the frequently utilized ferrites in microwave absorbing applications. α – Fe_2O_3 is a ferrite (corundum – kind iron oxide), stable in ambient conditions with unique magnetic and electric properties.

Polycaprolactone is a biodegradable polymer which is easily blended, non – toxic lightweight, has good dielectric properties and binds well to α – Fe_2O_3. This desirable combination of characteristics makes it a suitable polymer matrix for hosting the recycled α – Fe_2O_3 nanofiller since the most commonly used host materials for ferrite – based absorbers are heavy, difficult to blend and non – biodegradable. Moreover, recycled α – Fe_2O_3 nanofiller compositions could have an effect on the permittivity, permeability and microwave absorbing properties of α – Fe_2O_3/PCL nanocomposites which has to be investigated. Furthermore, absorbers need to have high loss factor for higher absorbing properties. In this research, small grain – sized OPEFB fiber was incorporated into α – Fe_2O_3/PCL nanocomposites in order to provide the required high loss factor, while retaining the magnetic properties. The small grain – sized OPEFB fiber has a high loss factor and is biodegradable, cheap, low density with good thermal and mechanical properties and can easily be blended with PCL. Additionally, the low density can also make the nanocomposites lighter. The incorporation of OPEFB fiber into α – Fe_2O_3/PCL nanocomposites could enhance the permittivity, permeability and microwave absorbing properties of α – Fe_2O_3/OPEFB/PCL nanocomposites.

The conventional technique used to determine the complex permittivity and S – parameters of the α – Fe_2O_3/PCL and α – Fe_2O_3/OPEFB/PCL nanocomposites materials is to place the samples in a closed waveguide. The technique is difficult as the samples have to be inserted tightly into the waveguide without any air gaps. In this research, open ended coaxial probe (OEC) technique was used to measure the complex permittivity while and the microstrip measurement technique was used to acquire the S – parameters. The microstrip measurement method alone could not be used to describe the electromagnetic field distribution in the samples. Therefore, the visualization of the electromagnetic field in the samples was carried out where the samples were discretized into smaller meshes using the Finite Element Method (FEM).

1.2 Research Objectives

The main objectives of this research are as follows:

1. To synthesize α – Fe_2O_3 using mill scale (steel waste) and reduce the particle sizes to nanosize using high energy ball milling and characterize their morphological, structural and dielectric properties. The effect of particle – size reduction on the dielectric properties of the particles will then be examined.

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2. To fabricate $\alpha - Fe_2O_3$/PCL nanocomposites with different mass percentages of recycled $\alpha - Fe_2O_3$ nanofiller and investigate the effect of the nanofiller on the complex permittivity and permeability of the nanocomposites.
3. To fabricate $\alpha - Fe_2O_3$/OPEFB/PCL nanocomposites with different mass percentages of recycled $\alpha - Fe_2O_3$ nanofiller and OPEFB fiber and examine the effect of the fillers on the complex permittivity and permeability of the nanocomposites.
4. To investigate the effects of recycled $\alpha - Fe_2O_3$ nanofiller and OPEFB fiber on the scattering parameters, attenuation and power loss due to absorption for the $\alpha - Fe_2O_3$/PCL and $\alpha - Fe_2O_3$/OPEFB/PCL nanocomposites using the microstrip measurement. The measured scattering parameter results will be compared with those obtained from the theoretical calculations using the Finite Element Method (FEM).
5. To visualize the electric field distribution of the nanocomposites of different mass percentages of recycled $\alpha - Fe_2O_3$ nanofiller.

1.3 The Scope of Study

In this study, $\alpha - Fe_2O_3$ will be synthesized using mill scale and the particles reduced to three different nano sizes using high energy ball milling technique for several hours. The dielectric properties of the recycled will be determined at X – band (8 – 12 GHz) frequency range in order to compare with recent research works. The nanocomposites will be fabricated using recycled $\alpha - Fe_2O_3$ with the smallest particle size and OPEFB fiber, through melt – blending by employing a Brabender Plastograph twin screw extruder. The mass percentage compositions of the recycled $\alpha - Fe_2O_3$ will be taken from 5% - 25% with 5% increment. A fixed ratio of 7:3 will be used for OPEFB fiber and PCL respectively. The relative complex permittivity and permeability of the nanocomposites with different mass percentages of recycled $\alpha - Fe_2O_3$ nanofiller and OPEFB filler will be determined using the open ended coaxial probe and the rectangular waveguide respectively. The dielectric and microwave characterizations of the nanocomposites will be conducted in the L – S (1 – 4 GHz) frequency range while the permeability properties will be performed in the X – band for comparisons. The morphological and microstructural characterizations of the samples will be carried out using specific techniques such as XRD, FTIR, SEM, EDX, HRTEM and BET.

The effect of the fillers on the transmission and reflection coefficients of the $\alpha - Fe_2O_3$/PCL and $\alpha - Fe_2O_3$/OPEFB/PCL nanocomposites will also be studied using the microstrip technique. The study also proposes to use FEM through the COMSOL software to calculate the scattering parameters (S_{11} and S_{21}) and also simulate the electromagnetic waves propagated through the $\alpha - Fe_2O_3$/PCL and $\alpha - Fe_2O_3$/OPEFB/PCL nanocomposites placed on top of the microstrip. The scattering parameter results obtained through measurement and simulation will then be compared. Error analysis of the comparison between measurements and FEM technique will be determined. The COMSOL software enables the visualization of the distribution of the electric fields around the system which provides a clear understanding about the material’s interaction with electromagnetic waves. FEM

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utilizes the inputs of the material's dielectric constant and loss factor values to accomplish the simulations.

1.4 Organization of the Thesis

This thesis consists of six chapters and an appendix. Chapter 1 gives a general introduction on EMI and the materials to be applied for its reduction in this research, followed by the statement of the problem, objectives of the study, the scope of the study and the thesis layout.

Chapter 2 presents reviews on the properties of ferrites, recycled α – Fe_2O_3, polycaprolactone and OPEFB fiber. Composite material synthesis methods and microwave characterization techniques are also reviewed in this chapter. Finally, the Finite Element Method (FEM) as a numerical technique for the simulation of electric field distribution and determination of the S – parameters of samples placed on the microstrip is reviewed.

Chapter 3 is the theory chapter and it discusses the theoretical concepts of the mechanical alloying technique, dielectric properties, polarization, the rule of mixtures, Bragg's law of diffraction and basic electromagnetic wave equations. It concludes with the transmission and reflection coefficients calculation procedures with FEM formulation techniques. Sample preparation, microstructural, morphological and electromagnetic characterizations will be discussed in Chapter 4. The use of the OEC, RWG, microstrip and FEM methods are fully discussed in relation to the electromagnetic characterization while morphological characterization using XRD, HRTEM, SEM, EDX, BET and FTIR are all discussed in details.

Chapter 5 is presented in six sections and it discusses the results of the material characterization and simulations involving all the samples used in this research. The first section presents and discusses the results of the morphological and structural characterization of the recycled α – Fe_2O_3 nanoparticles, α – Fe_2O_3/PCL and α – Fe_2O_3/ OPEFB/PCL nanocomposites using measurement techniques such as XRD, FTIR, SEM, HRTEM and EDX. This is followed by the second section which deals with the results of the complex permittivity measurements of the recycled α – Fe_2O_3 nanoparticles and the fabricated nanocomposites using the OEC and Rectangular waveguide techniques. The third and fourth sections respectively discuss the results of the permeability measurements and scattering parameter measurements of the α – Fe_2O_3/PCL and α – Fe_2O_3/OPEFB/PCL nanocomposites using the microstrip and numerical simulation (Finite Element Method). The fifth section describes and discusses the results of material absorption where the scattering parameters S_{11} and S_{21} of the nanocomposites obtained from the microstrip technique were used to calculate attenuation, absorption and power loss of the samples. The final section of the chapter reports on the results of FEM simulations and visualization of the intensity and electric field distribution for the α – Fe_2O_3/PCL and α – Fe_2O_3/OPEFB/PCL nanocomposites based on the microstrip.

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Finally, chapter 6 summarizes and draws conclusions based on the findings of this research and offers suggestions for future research in this area of study.

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