UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF OIL PALM EMPTY FRUIT FIBRE POLYCAPROLACTONE NICKEL ZINC–FERRITE AND NICKEL OXIDE COMPOSITES FOR MICROWAVE SHIELDING

AHMAD FAHAD AHMAD

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By

AHMAD FAHAD AHMAD

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

December 2015

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DEDICATIONS

To my late father and mother (Allah bless them),

--- and ---

My lovely wife and sons,

For their great patience and encouragement

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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

PREPARATION AND CHARACTERIZATION OF OIL PALM EMPTY FRUIT FIBRE POLYCAPROLACTONE NICKEL ZINC–FERRITE AND NICKEL OXIDE COMPOSITES FOR MICROWAVE SHIELDING

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AHMAD FAHAD AHMAD

December 2015

Chairman : Associate Professor Zulkifly Abbas, PhD
Faculty : Institute For Mathematical Research

Electromagnetic shielding is an issue that needs to be resolved in the design and development of many electronic systems and components. A number of light-weight polymers have been proposed with shielding effectiveness (SE) as high as 80dB. However not all electromagnetic interference or compatibility applications demand materials with very high shielding effectiveness value. For example, the Wireless Avionics Intra-Communications protocol imposed only a minimum shielding effectiveness of only 5dB for the aircraft structural design.

This thesis presents the development of natural oil palm fruit fibre reinforced polycaprolactone (PCL) composites filled with nickel zinc ferrite (NZF) for microwave shielding and absorbing applications. The oil palm empty fruit bunch fibre (OPEFB) exhibit good mechanical properties when compared with other natural fibres. The OPEFB fibres and PCL are biodegradable, cheap and less hazardous compared to many other industrial materials. OPEFB+PCL composites with NZF fillers provide cost effective solutions for applications requiring a minimum of 5dB shielding effectiveness. In this work, the properties OPEFB+PCL composites with nickel oxide (NiO) fillers were examined in the microwave frequencies. Different composition of NZF and NiO fillers will give different absorbing properties in the OPEFB+PCL matrix. Too low percentage of fillers in the OPEFB+PCL will not provide sufficient SE whilst too high percentage would be excessively expensive.

The fillers were prepared by the conventional solid state method. Different compositions of filler were doped and blended to produce NZF+OPEFB+PCL and NiO+OPEFB+PCL composites. The crystalline structure of the composites was analyzed using X-ray diffraction (XRD) machine. The elemental compositions were examined using Scanning Electron Microscopy (SEM) and Fourier transform infrared (FTIR) techniques. Thermal analyses were carried out using TGA and DTG, scanning electron microscope (SEM) and energy dispersive X-ray analysis (EDX). The theoretical calculation of the transmission and reflection coefficients of the sample placed in the waveguide was computed using Nicholson-Ross-Weir method and Finite Element Method. Then later was accomplished using COMSOL software.

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The transmission and reflection coefficients as well dielectric properties were measured using an Agilent N5230A Network Analyzer from 8GHz to 12 GHz at room temperature. The permittivity of the composites was found to be dependable on the mixing ratio values between OPEFB, PCL, NiO and NZF. The dielectric constants of the composites were found between 1.87 and 5.82 with corresponding loss factor from 0.09 to 1.21 in the X-band frequency. OPEFB+PCL composites showed magnetic properties when doped with just a small percentage of NZF doping. Increasing the NZF content in the composites from 2.5 % to 15% will increase the real part of permeability from 1.043 to 1.095 whilst the imaginary part increased from 0.042 to 0.054. Both the dielectric constant and loss factor of OPEFB+PCL+NiO and OPEFB+PCL+NZF composites were found to be linearly dependent on the percentage of the fillers. Both the dielectric constant and loss factor of the composites can be predicted from the regression equations by inserting values of the fractional composition of the fillers. Both the dielectric constant and loss factor of the OPEFB+PCL composite increased with increasing percentages of NZF fillers. These in turn will lead to higher values of the magnitude reflection coefficient $|S_{11}|$ and lower transmission coefficient $|S_{21}|$ in accordance with the impedance matching theory.

The total shielding effectiveness for OPEFB+PCL, NiO+OPEFB+PCL and NZF+OPEFB+PCL composites were found between 1.11 to 1.46 dB, 2.91 to 3.69 dB and 5.06 to 6.19dB, respectively. The minimum 5dB shielding effectiveness required for commercial aircraft structural shielding can be satisfied by using OPEFB+PCL composites with only 2.5% NZF fillers.

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

PERSEDIAAN DAN PERCIRIAN SIFAT KOMPOSIT TANDAN KOSONG KELAPA SAWIT POLIKAPROLAKTON NIKEL ZINK-FERRITE DAN NIKEL OKSIDA UNTUK PEMERISAIAN MIKROGELOMBANG

Oleh

AHMAD FAHAD AHMAD

Disember 2015

Pengerusi : Profesor Madya Zulkifly Abbas, PhD
Fakulti : Institut Penyelidikan Matematik

Perlindungan elekromagnetik adalah satu isu yang perlu diselesaikan dalam rekabentuk dan pembangunan kebanyakan komponen dan sistem elektronik. Pelbagai polimer ringan telah dicadangkan dengan keberkesanan lindungan (SE) setinggi 80dB. Namun begitu bukan semua aplikasi ganguan atau keserasian elektromagnetik memerlukan bahan dengan nilai lindungan yang tinggi. Sebagai contoh, protokol Wireless Avionics Intracommunication hanya menetapkan lindungan minima 5dB untuk rekabentuk struktur kapalterbang.

Tesis ini mempersembahkan pembangunan fiber kelapa sawit sebagai bahan pengukuhan komposit policaprolactone (PCL) dengan pengisian nikel zink ferrite (NZF) untuk aplikasi lindungan dan serapan gelombang mikro. Fiber dahan kelapa sawit (OPEFB) mempaunyai sifat mekanikal yang lebih baik berbanding dengan fiber semulajadi yang lain. Fiber OPEB dan PCL adalah mudah teruari secara biologi,murah dan kurang toksid berbanding bahan industry yang lain. Komposit OPEFB+PCL dengan pengisi NZF menawarkan penyelesain yang berkesan lagi murah untuk aplikasi yang memrlukan keberkesanan lindungan 5dB. Dalam kajian ini, komposit OPEFB+PCL dengan pengisian nickel oxide (NiO) juga diuji pada frekuensi gelombang mikro. Lain kandungan peratusan pengisi NZF dan NiO akan member lain nilai serapan dalam matriks OPEFB+PCL. Peratusan terlalu rendah dalam OPEFB+PCL tidak akan. Member SE yang mencukupi sementara pertusan pengisian yang terlalu tinggi pula menjadi bahan terlalu mahal.

Pengisi disediakan mengguna kaedah konvensional keadaan pepejal. Pengisi dengan peratusan berbeza digabung untuk menghasil komposit NZF+OPEFB+PCL dan NiO+OPEFB+PCL. Struktur hablur komposit dianalisa mengguna kaedah belauan sinar-X (XRD). Kandungan elemn dinilai menggunakan kaedah pengimbas mikroskop electron (SEM) dan teknik infra merah transformasi Fourier (FTIR). Aanalisa terma dilakukan secara kaedah TGA, DTG, SEM dan EDX. Pengiraan teori pekali penghantaran dan pantulan untuk sapel yang diletakan dalam pandu gelombang dilakukan menggunakan kaedah Nicholson-Ross-Weir dan Elemen Berangka mengunakan perisian COMSOL.

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Pekali penghantaran dan pantulan serta sifat dielektrik diukur mengunakan Penganalisa Rangkaian Agilent N5230A dari frekuensi 8GHz hingga 12 GHz pada suhu bilik. Ketelusan elektrik komposit didapati bersandar kepada nilai nisbah campuran OPEFB, PCL, NiO and NZF. Pemalar dielektrik diperolehi adalah diantara 1.87 and 5.82 sementara faktor kehilangan nya antara 0.09 to 1.21 dalm frekuensi jalur-X. Komposit OPEFB+PCL mempamirkan sifat magnetic dengan hanya sedikit pendopan NZF. Penambahan kandungan NZF dari 2.5% kepada 15% sahaja akan menaikkan nilai hakiki ketelusan magnet dari 1.043 kepada 1.095 sementara nilai khayalannya bertambah dari 0.042 kepada 0.054. Kedua-dua pemalar dielektrik dan faktor kehilangan komposit OPEFB+PCL+NiO dan OPEFB+PCL+NZF adalah berkadar terus dengan peratusan pengisi. Nilai pemalar dielektrik dan faktor kehilangan komposit boleh diramal terus dari persamaan regresi sekiranya diketahui nilaipecahan pengisi. Kedua pemalar dielektrik dan faktor kehilangan komposit bertyambah dengan peratusan penambahan NZF. Seterusnya ini akan meningkatkan nilai magnitud pekali pantulan $|S_{11}|$ dan menurun nilai pekali penghantaran rendah $|S_{21}|$ bertepatan dengan teori ketidakserasian impedans.

Jumlah keberkesanan lindungan komposit OPEFB+PCL, NiO+OPEFB+PCL and NZF+OPEFB+PCL adalah diantara 1.11 hingga 1.46dB, 2.91 hingga 3.69dB dan 5.06 hingga 6.19dB, masing-masing. Keberkesanan lindungan minima 5dB yang ditetapkan untuk struktur kapalterbang komersil boleh dicapai menggunakan komposit OPEFB+PCL dengan hanya pengisian 2.5% NZF.

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I certify that a Thesis Examination Committee has met on 30 December 2015 to conduct the final examination of Ahmad Fahad Ahmad on. his thesis entitled "Preparation and Characterization of Oil Palm Empty Fruit Fibre Polycaprolatone Nickel Zinc-Ferrite and Nickel Oxide Composites for Microwave Shielding" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

α	Attenuation constant
s	Spacing between ring and the feed line
c	Velocity of light
ε^*	Complex permittivity
ε'	Dielectric constant
ε''	Loss factor
PCL	Polycaprolactone
PLA	Polylactic acids
EM	Electromagnetic
EMI	Electromagnetic interference shielding
σ	Electrical conductivity
ε	Permittivity
μ	Permeability
SE	Shielding effectiveness
σrμr	The reduction of absorption
σr/μr	The reduction of reflection
dB	Decibels
δ	Substrate depth
NiO	Nickel Oxide
OPEFB	Oil palm empty fruit bunch
NZF	Nickel Zinc Ferrite
NRW	Nicholson-Rose-Weir
FEM	Finite Element Method
XRD	X-ray diffraction
TGA	Thermogravimetric analysis
DTG	Differential thermal
FTIR	Fourier transforms infrared
SEM	Scanning electron microscopy
EDX	Energy dispersive X-ray
T/R	Reflection/Transmission
RFI	Radio frequency interference
J	The current density
D	The electric displacement
B	The magnetic flux density
r	Mean radius of the ring
γ	Propagation constant
ρ_q	The electric charge density
β	Phase constant
∇	Laplacian vector
d	Sample thickness
η	Impedance
η_0	Impedance in free space
ω	Angular frequency
f	Frequency
fr	Critical frequency
ε_r	Relative dielectric of the substrate

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λ_g	Guided wave length
ε_{eff}	Effective permittivity
H_z	Longitudinal magnetic field
E_z	Longitudinal electric field
P_1	Power measured with the material inserted
P_2	Power measured without material inserted
MC	Moisture content
S_1	Input reflection coefficient of port one
S_{12}	Transmission coefficient port one
S_{22}	Input reflection coefficient of port two
S_{21}	Transmission coefficient port two
TE	Transverse Electric
TM	Transverse Magnetic
D_w	Relative density of the water.
D_S	Relative density of solid rubber.
k_0	Free space wave number
m_{dry}	Mass after dried
m_{wet}	Mass before drying
h,s ,d	Thickness of dielectric layers
$\varepsilon^*(x,y)$	Complex permittivity profile
$\varepsilon_{r1,}\varepsilon_{r2,}\varepsilon_{r3,}\varepsilon_{r4}$	Permittivity of dielectric layers
TEM	Transverse electromagnetic modes
$\tan\delta$	Loss tangent
EMP	Evanescent Microwave Probe
FSK	Frequency shift keying
FDTD	Finite Difference Time Domain Method
MMM	Microwave moisture measurement
MoM	Method of Moment
MSA	Microwave antenna
NDT	Non-destructive testing
OPC	Ordinary Portland cement
MUT	Materials under test,
PNA	Professional network analyzer
DXF	Drawing exchange format
SOLT	Short-open-load-thru
ASTM	American Society for Testing and Material Standards
FDTD	Finite difference time domain
MATLAB	Matrix laboratory

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CHAPTER 1

INTRODUCTION

1.1 Preliminary Concepts of Composites

Composites are made up of individual materials referred to as constituent materials. There are two main categories of constituent materials: matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties (Kozłowski et al. 2008).

A synergism produces material properties unavailable from the individual constituent materials, while the wide variety of matrix and strengthening materials allows the designer of the product or structure to choose an optimum combination (Chawla, 2012).

Historical or natural examples of composites are abundant: brick made of clay reinforced with straw, mud wall with bamboo shoots, concrete, concrete reinforced with steel bar, granite consisting of quartz, mica and feldspar, wood (cellulose fibres in lignin matrix), etc. The matrix material experiences a melting event, after which the part shape is essentially set. Depending upon the nature of the matrix material, this melting event can occur in various ways such as chemical polymerization or solidification from the melted state. A variety of molding methods can be used according to the design requirements. The principal factors impacting the methodology are the nature of the chosen matrix and reinforcement materials. Another important factor is the gross quantity of material to be produced.

Many commercially produced composites use polymer matrix material often called a resin solution. There are many different polymers available depending upon the starting raw ingredients. There are several broad categories, each with numerous variations. The most common are known as polyester, polycaprolactone (PCL), polylactic acids (PLA), ester, epoxy, phenol, polyimide, polyamide, polypropylene and others.

The development of natural fibre composite for structural application is still at infancy stage. Due to the attractive properties like high specific strength and high specific modulus, natural fibre composite rapidly gains popularity in the use of automobile applications and structural applications. Compare to synthetic fibre composite, natural fibres are low cost and abundant in agro base country. The use of natural fibres in composites can reduce the impact of environmental issues.

This study is a preliminary stage to made natural fibre composite as structural application where only dielectric properties is focused. In fact, the force of the impact of this new material in structural application is equally important. The use of natural fibre composite in structural application is possible but requires more study and development in future. On the other hand fibre percentage influences the

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dielectric behavior properties directly, where the dielectric properties are improved and also affected by fibre aspect ratio where high fibre aspect ratio composite usually improve the loss tangent properties of the composite.

1.2 Short History of Electromagnetic Interference (EMI) Shielding

Electromagnetic shielding is the practice of using specialized materials to reduce the electromagnetic fields or waves from entering an enclosure. The shielding performance depends very much on the type of material used, its thickness, the size of the shielded volume and the frequency of the fields of interest and the size, shape and orientation of apertures in a shield to an incident electromagnetic field. Typical materials used for electromagnetic shielding include metal sheet, metal screen, and metal foam. Any holes in the shield or mesh must be significantly smaller than the wavelength of the radiation that is being kept out, or the enclosure will not effectively approximate an unbroken conducting surface.

The designing of an Electromagnetic Interference (EMI) shielding material with a certain level of attenuation, meeting a set of physical criteria, maintaining economics and regulating shielding have been proposed. The main motivation behind the proper design of shield is to make a product that can comply to National International Electromagnetic Interference Regulatory Standards. Owing to their aesthetic appeal, plastics have replaced metal cabinets of electronic housings.

However, plastics are transparent to electromagnetic radiations and cannot be earthed to provide electrostatic control. Investigation of new effective materials applicable as microwave absorbers for EMI shielding of various electronic devices ranks among important present-day activities (Paligová et al. 2004). Extensive studies have been carried out to develop new microwave absorbing materials with high magnetic and electric loss (Qin, 2012).

In the aerospace industry, innovative solutions are required to shield effectively sensitive electronic equipment such antennas form EMI without adding a lot of weight to aircraft. EMI can jam cockpit radio and radar signals, preventing pilots from sending and receiving crucial information. The U.S. Air Force initiated an 18-month study of the use of carbon nanotube sheets to create a shielding layer on the surfaces of lightweight composites (Greenemeier, 2009). The carbon nanotube sheets are designed to act as a Faraday cage that can block out external static electrical fields from sensitive circuitry. This carbon nanotube shielding technique offers more than 80dB shielding effectiveness. Unfortunately, the technique is very expensive. For commercial aircrafts, more cost effective solution is desirable as only a minimum 5dB shielding effectiveness is required for the aircraft structural shielding for antenna based on the Wireless Avionics Intra-Communications specifications shielding effectiveness is required for the aircraft structural shielding for antenna based on the Wireless Avionics Intra-Communications specifications (ITU, 2013).

This EMI may intentionally or unintentionally react with either electronic device, causing a degradation of performance of other equipment, or systems that share the same environment leading to loss of time, energy, resources and also adversely affect human health. Furthermore, electronic equipment that produces electromagnetic

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radiation must be isolated or shielded to prevent it from degrading the performance of surrounding equipment.

Electromagnetic radiation or shielding has been a subject of intense research since the 1930‘s which continued with attempts to find solution of the overlapping waves. Therefore, in the recent past, a wide variety of materials (Singh et al. 2006; Abbas et al. 2007; Saini et al. 2013) have used EMI shielding with a broad range of electrical conductivity (σ), good electromagnetic attributes such as permittivity (ε) or permeability (μ) and engineered geometries. Previously the problem of EMI was solved through isolation of the electronic device through some metallic housing. Nowadays polymeric material has gained popularity due to its flexibility, light weight, corrosion resistance, and lower cost than metal.

1.3 Shielding Materials

Different engineering design and applications require different minimum value of shielding effectiveness (SE). For military designs, the typical SE is usually more than 80 dB for. Selection of shielding materials is certainly a factor in the design. SE of a material depends on both the conductivity and permeability, but for high frequency shielding, conductivity dominates. Materials with higher conductivity will provide higher values of SE. The composites are usually engineered to process chemical and physical properties of materials and generally, enhance their properties. Commonly, the composites consist of fillers and matrix. The fillers are surrounded by matrix material in order to provide the supports for that material by keeping their positions. The matrix material must be flexible, light weight, corrosion resistance, and lower cost than metal.

PCL and other conducting polymers are well known materials for shielding electromagnetic waves in both near and far fields (Lakshmi et al. 2009). PCL is a class of polymers is an excellent microwave absorbing material (Geetha et al. 2009). Adding biodegradable fibres such as the OPEFB will not only reduce the cost and enhanced structural properties of the composite but also the ability to control its electromagnetic properties especially when operating at high frequencies.

For SE applications, Nickel-zinc ferrites (NZF) are the best filler candidate for the composites due to its high resistivity values at microwave frequencies especially in the X-band (8 to 12 GHz). NZF is also widely used as microwave absorbing materials (Rozman, 2005). The electromagnetic properties of the composites are usually characterized in terms of its dielectric constant, loss factor, reflection, transmission coefficients and the shielding effectiveness.

1.4 Interactions of Microwaves with Materials

The microwave constitutes only a small portion of the electromagnetic spectrum (300 MHz to 300 GHz) corresponding to wavelengths between 10^{-1}m and 10^{-3}(Stutzman et al. 2012). But their uses have become increasingly important in investigations of material properties. Material characterization is essential for the proper selection and

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implementation of a substance when used in industrial, scientific and medical applications.

The dielectric parameters over a wide range of temperature are needed to assess their suitability for use in telecommunications, dielectric waveguides, lenses, dielectric resonators and microwave integrated circuits (MICs). The electromagnetic spectrum consists of various types of electromagnetic signals as listed in Table 1.1. The range of microwave frequency is between 300MHz and 300GHz (Pozar, 1998).

Microwave behaves much like light wave in that it travels in straight lines, refract, reflect, diffract, scatter, and interfere according to the same physical length. However they are different in behavior because of the difference in wavelength. Microwave wavelengths are typically 10^5 larger than optical wavelengths. Thus microwave tend to interact with materials and structure on a macroscopic scale. For example, microwaves are capable of penetrating most non-metallic materials, reflecting and scattering from internal boundaries and interacting with molecules (Bahr, 1982).

Table 1.1: The electromagnetic spectrum (Pozar, 1998)

Wavelength (m)	Usual division of radiation	Frequency (Hz)
10^{-14}	Cosmic radiation	3×10^{22}
10^{-13}, 10^{-12}, 10^{-11} 10^{-10}, 10^{-9}	X and Gamma radiation	3×10^{21}, 3×10^{20}, 3×10^{19} 3×10^{18}, 3×10^{17}
10^{-8}, 10^{-7}	Ultraviolet	3×10^{16}, 3×10^{15}
10^{-6}	Visible light	3×10^{14}
10^{-5}, 10-^{4}	Infrared	3×10^{13}, 3×10^{12}
10^{-3}, 10^{-2}, 10^{-1}	**Microwaves**	**3×10^{11}, 3×10^{10}, 3×10^{9}**
1	Radio waves	3×10^{8}

Microwaves do not heat or change in any way the material due to the extremely low energy emitted. Testing with microwave is dominated by the basic properties of microwaves. Since their penetration in good conducting materials is minimal, they are mainly used to test non-conducting materials. On the other hand, microwaves are affected by a large number of material properties. In lossless or lossy dielectrics, material composition, uniformity of the material, moisture and contamination content and such diverse properties as porosity are some of the properties that can be measured.

Many ideas have been attempted to adapt these phenomena to microwave applications. Inherently conducting polymers are excellent microwave absorbers and make ideal materials for effecting welding of plastics (Domenech et al. 2004). The two important applications concerned with the use of microwave properties are EMI shielding and radar absorbing materials.

The application of microwave technology can be found in various fields such as communications, broadcasting, military, environmental remote sensing, weather monitoring and forecasting, ground positing system, astronomy and medical system.

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Much of the success of today's microwave technology is owed to decades of relentless efforts, hard works and painstaking researches by Andre-Marie Ampere, Carl Friedrich Gauss, Michael Faraday, Oliver Heaviside, Heinrich Hertz and James Clerk Maxwell, just to name but a few (Pozar, 2006). The interest in this work is the interaction of microwaves with materials. This includes parameters of absorption in materials, scattering, attenuation, reflection and transmission. These effects are exploited in various testing arrangements to allow for quantitative measurements in materials.

1.5 Electrical Properties

Microwave is a very important medium in modern life especially at the industry and academic field. There are huge number of existing and possible applications of microwave techniques and instrumentation in the enhancement of industries.

The interaction between microwaves and materials can be deduced from Maxwell's equations and material properties. The relations define a variety of properties such as mode of propagation, reflection, refraction, transmission and impedance ((Luo et al. 2015). Both the permittivity and permeability are complex numbers of which the imaginary part is associated with losses.

This rich and complex system of properties allows a very wide range of measurement techniques at microwave frequencies. Until this date, many different methods have been proposed for microwave measurements of electromagnetic properties of materials (Afsar et al. 1986). However, there are two main groups of measurement technique based on sample location: waveguide or coaxial line methods and free space measurement methods.

The first groups of methods have been elaborated in detail (Hippel, 1954) but, in many practical cases, they are not applicable. The main disadvantage of these methods is the extreme difficulty of machining a sample to fit into the waveguide or coaxial fixture with negligible air gap.

1.6 Problem Statement

Materials with good absorption are in demand to control EMI in commercial and industrial electronics. The most common shielding material is mu-metal, an alloy of 5% copper, 1.5% chromium, 14% iron and 79.5% nickel. However, the mu-metal are corrosive and expensive. Usage of different metals for shielding could easily lead to galvanic corrosion which in turn increased the nonlinearity behavior and decreased its shielding effectiveness. In recent years, conventional plastic materials filled width conductive materials are gaining interests as alternatives to mu-metal.

New materials with SE as high as 130 dB have been reported by (Luo et al. 2015). These materials are useful in protecting the EMI of military equipment where SE of at least 80 dB from 10 KHz to 18 GHz is not an option. However, not all EMI applications demand materials with extremely high SE value. For some applications, the objective of shielding is just reducing the interference signals.

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The minimum SE required for the aircraft structural design is only 5dB based on the Wireless Avionics Intra-Communications specifications (ITU, 2013). It would be unnecessarily expensive to require 60 dB of shielding instead of 25 dB for enclosure if the associated cables and their connecting hardware provide only 30 dB (Kaiser, 2006). The performance of materials with low SE can be improved by increasing the shielding thickness (Lakshmi et al. 2009).

This project presents an extensive investigation of the morphological, structural and microwave properties of OPEFB+PCL composites with NZF fillers as cost effective materials for applications requiring a minimum of 5dB shielding effectiveness. The results shall be compared with nickel oxide (NiO) fillers which are more commonly used at low frequencies.

Different fillers will give different absorbing properties. These fillers will host in OPEFB+PCL matrix. PCL is a biodegradable material is cheap, has good physical and mechanical properties, and flexible enough to be molded into various packing shapes for EMI applications. Different composition of NZF and NiO fillers will give different absorbing properties in the OPEFB+PCL matrix. Too low percentage of OPEFB+PCL will not provide sufficient SE whilst too high percentage would be excessively expensive. The minimum percentage of NZF fillers required to produce at least 5dB SE will be investigated.

1.7 Research Objectives

The main objectives of the study are:

i) To prepare OPEFB, NiO and NZF ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) based PCL polymer matrix with different OPEFB, NiO, NZF filler percentages, and then characterize their structural properties by using XRD, TGA, DTG, FTIR, SEM as well as EDX.

ii) To determine S-parameters of different OPEFB+PCL, NiO+OPEFB+PCL and NZF+OPEFB+PCL compositions that placed in a closed T/R rectangular waveguide. And then compare the obtained measurement results with the extracted values that achieved by the theoretical approach of NRW and FEM techniques.

iii) To determine the dielectric constant and loss factor properties of various OPEFB+PCL, NiO+OPEFB+PCL and NZF+OPEFB+PCL composite percentages by using open-ended coaxial technique at 8-12 GHz microwave frequency range.

iv) To determine the best composition of OPEFB, NiO and NZF in the PCL matrix to provide maximum shielding effectiveness and absorption of 1mm sample thickness that change the medium loss of PCL polymer into a preferable high loss material.

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1.8 Scope of the Study

An overview of natural fibre based composites; shielding materials and effectiveness are highlighted in this Chapter 1. The problem statement and main objectives of this study are also presented. Chapter 2 presents presents a review on the basic materials used as composites in this work: OPEFB, PCL, NZF and NiO.

The numerical electromagnetic methods and measurement techniques to determine the electromagnetic properties of materials are also discussed. Chapter 3 highlights the basic theory of electromagnetic wave propagation. The Nicholson-Ross-Weir technique (NRW) and finite element method (FEM) to calculate the transmission and reflection coefficients of a sample place in a closed waveguide are also discussed.

This is followed by a review on the theory of shielding effectiveness. Chapter 4 describes the details of the preparation and characterization techniques of bio-composite materials of OPEFB+PCL, NiO+OPEFB+PCL and $Ni_{0.5}Zn_{0.5}Fe_2O_4$-OPEFB+PCL. A detailed description on the implementation of FEM using COMSOL software to calculate the transmission and reflection coefficient is also presented. Chapter 5 presents the results and discussions on material characterization using XRD, TGA and DTG, SEM, EDX and FTIR techniques.

The results on the electromagnetic properties of the materials are discussed in detail emphasizing on the dielectric properties, reflection and transmission coefficients as well as its shielding effectiveness. Finally, Chapter 6 provides a summary of the main results reported in this thesis. Based on these results, some general conclusions are drawn and recommendations for future research are given.

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