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PREPARATION AND CHARACTERIZATION OF OIL PALM FIBER-REINFORCED PCL COMPOSITES FOR REDUCTION OF ELECTROMAGNETIC INTERFERENCE

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By

RADZI RAHMAN BIN IBRAHIM

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

December 2017

DEDICATION

To my father and mother (Allah bless them)

And

my friends

For their great patience and encouragement

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairman : Associate Professor Zulkifly Abbas, PhD
Faculty : Science

Microwave absorbers are used in a wide range of applications to eliminate stray or unwanted radiation that could interfere with a system's operation. For example, the Wireless Avionics Intra-Communications protocol imposed only a minimum shielding effectiveness of only 5dB for the aircraft structural design. It generally consist of a filler material inside a material matrix. The filler consists of one or more type of material that do most of the absorbing. The matrix material is chosen for its physical properties (temperature resistance, weather ability, etc.).

This thesis presents the development of a natural oil palm empty fruit bunch fiber (OPEFB)-reinforced polycaprocalctone (PCL) composites with Iron Oxide (Fe_2O_3) for microwave shielding and absorbing applications. Oil palm empty fruit bunch fiber exhibit excellent mechanical properties when compared with other natural fibers. The OPEFB and PCL are biodegradable, cheap and less dangerous compared to industrial materials.

The fillers which is OPEFB and Fe_2O_3 were prepared by the conventional solid-state method. Different compositions of filler were doped and blended to produce OPEFB-PCL and OPEFB-PCL- Fe_2O_3 composites. The crystalline structure of the composites was analyzed using X-ray diffraction (XRD) machine to ensure the sample only containing the fillers without any contamination while the theoretical calculation of the transmission coefficients of the sample placed in the waveguide was computed using Finite Element Method (FEM) and was accomplished using COMSOL software. The transmission and reflection coefficients as well as dielectric properties were measured using a PNA (N5227) Network Analyzer from 8 GHz to 12 GHz at room

temperature by using open ended coaxial (OEC) and rectangular waveguide (RWG) technique. The permittivity of the composites was found to be dependable on the mixing ratio values between OPEFB, PCL, and Fe_2O_3. Both the dielectric constant and loss factor of the OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composite increased with increasing percentages of OPEFB and Fe_2O_3 .fillers respectively. The dielectric constants of OPEFB-PCL composites were found to between 2.50 and 3.44 with similar loss factor from 0.17 to 0.40 in the X-band frequency. Furthermore the dielectric constant of OPEFB-PCL-Fe_2O_3 composites were found to be between 3.17 to 3.50 and similar loss factor from 0.25 to 0.40 respectively in the X-band frequency. These, in turn, will lead to higher values of the magnitude reflection coefficient |S11| and lower transmission coefficient |S21| by the impedance matching theory. OPEFB-PCL composites with Fe_2O_3 fillers provide cost-effective solutions for shielding effectiveness.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENYEDIAAN DAN PENCIRIAN FIBER KELAPA SAWIT ASLI (OPEFB) PENGUKUHAN POLYCAPROLACTONE (PCL) KOMPOSIT UNTUK PENGURANGAN GANGGUAN ELEKTROMAGNETIK

Oleh

RADZI RAHMAN BIN IBRAHIM

Disember 2017

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Penyerap gelombang mikro kebiasannya digunakan di dalam aplikasi skala besar untuk menghilangakan radiasi yang menyimpang atau yang tidak diperlukan dari mengganggu operasi sistem. Sebagai contoh, protokol *Wireless Avionics Intra-Communications* memutuskan hanya perlindungan minima efektif sebanyak 5dB sahaja dibenarkan untuk struktur reka bentuk pesawat. Penyerap gelombang mikro secara amnya terdapat pengisi bahan di dalam bahan matrik. Pengisi mempunyai satu atau lebih konstituen yang melakukan penyerapan. Bahan matrik dipilih kerana ciri fizikalnya (rintangan suhu, kelebihan tahan cuaca, etc).

Tesis ini membentangkan tentang perkembangan komposit fiber kelapa sawit (OPEFB) dikukuhkan dengan polycaprolactone (PCL) diisi bersama besi oksida (Fe_2O_3) untuk perlindungan mikro gelombang dan aplikasi penyerapan. OPEFB mempunyai ciri-ciri mekanikal yang baik apabila dibandingkan dengan fiber asli. OPEFB dan PCL adalah biodegradasi, murah dan kurang merbahaya sekiranya dibandingkan dengan bahan industri.

Pengisi iaitu OPEFB dan Fe_2O_3 telah disediakan dengan menggunakan cara konvensional bentuk pepejal. Pengisi dengan komposisi yang berbeza digabung dan digaul untuk membentuk komposit OPEFB-PCL dan OPEFB-PCL-Fe_2O_3. Struktur kristal komposit telah diperiksa menggunakan mesin pembiasan sinar-X (XRD) untuk memastikan tiada bahan luar selain daripada pengisi manakala pengiraan secara teori untuk transmisi dan refleksi koefisien untuk sampel di dalam pandu gelombang dilakukan dengan Cara Elemen Terhad (FEM) dan telah dilaksanakan menggunakan perisian COMSOL. Transmisi dan refleksi serta ciri penebat diukur menggunakan

PNA (N5227) Penganalisa Rangkaian dari 8 GHz hingga 12 GHz dalam suhu bilik mengunakan teknik pembuka berakhir sepaksi (OEC) dan teknik segi empat tepat pandu gelombang (RWG). Permitiviti komposit telah ditemui bergantung kepada nilai nisbah diantara OPEFB, PCL dan Fe_2O_3. Kedua-dua penebat pemalar dan faktor hilang untuk OPEFB-PCL komposit bertambah dengan pertambahan peratusan pengisi OPEFB. Penebat pemalar untuk komposit OPEFB-PCL adalah diantara 2.50 hingga ke 3.44 dan nilai faktor hilang 0.17 hingga 0.40 dalam frekuensi X. Tambahan lagi penebat pemalar untuk OPEFB-PCL- Fe_2O_3 komposit adalah masing-masing diantara 3.17 hingga 3.50 dan nilai faktor hilang 0.25 hingga 0.40 dalam frekuensi X. Ini secara tidak langsung akan meyebabkan nilai magnitud koefeksi refleksi |S11| yang tinggi dan nilai transmisi koefeksi |S21| yang rendah bersamaan dengan teori impedans salin. OPEFB-PCL komposit bersama pengisi Fe_2O_3 menyediakan solusi yang kos-efektif untuk perlindungan efektif

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First and foremost I would like to praise to the almighty ALLAH for giving me the strength, guidance and patience to complete this work. I could never have done this work without the faith I have in You. May blessing and peace be upon Prophet Mohammad SallaAllahuAlaihi Wasillam, who was sent for mercy to the world.

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I certify that a Thesis Examination Committee has met on 21 December 2017 to conduct the final examination of Radzi Rahman bin Ibrahim on his thesis entitled "Preparation and Characterization of Oil Palm Fiber-Reinforced PCL Composites for Reduction of Electromagnetic Interference" 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 Master of Science.

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

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A	attenuation constant
S	spacing between ring and the feed line
C	capacity
c	velocity of light
ε^*	complex permittivity
ε'	dielectric constant
ε''	loss factor
PCL	polycaprolactone
EM	electromagnetic
EMI	Electromagnetic interference shielding
σ	electrical conductivity
ε	Permittivity
μ	Permeability
dB	Decibels
δ	Substrate depth
Fe_2O_3	Ferrite
OPEFB	Oil Palm Empty Fruit Bunch
NRW	Nicholson-Rose-Weir
FEM	Finite Element Method
XRD	X-Ray diffraction
T/R	Reflection/Transmission
J	The current density
D	The electric displacement

B	The magnetic flux density
r	Mean radius of the ring
γ	Propagation constant
σ	Conductivity
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
λ_g	Guided wave length
ε_{eff}	Effective permittivity
P_1	Power measured with the material inserted
P_2	Power measured without material inserted
S_{11}	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.
TEM	Transverse Electromagnetic Modes
FDTD	Finite Difference Time Domain Method
MoM	Method of Moment
NDT	non-destructive testing
VNA	Vector network analyzer
FDTD	Finite Difference Time Domain
MATLAB	Matrix Laboratory
PTFE	Polytetrafluoroethylene
DIW	De-ionized water
IC	integrated circuits
OEC	open ended coaxial probe
RWG	rectangular wave guide
MMCs	metal matrix composites
CMCs	ceramic matrix composites
PMCs	polymer matrix composites
VLSI	very large scale integrated
SiO_2	silica
$HfSiO_4$	hafinium silicate
$ZrSiO_4$	zirconium silicate
$BaTiO_3$	barium titanate
MWCNT	multiwall carbon nanotube
CNT	carbon nanotube
δ	loss tangent

CHAPTER 1

INTRODUCTION

The demand of low cost, biodegradable absorber is increasing rapidly day by day. Absorbers in the RF/microwave realm are materials that attenuate the energy in an electromagnetic wave. Absorbers are used in a wide range of applications to eliminate stray or unwanted radiation that could interfere with a system's operation. Absorber can be used externally to reduce the reflection from or transmission to particular objects and can also be used internally to reduce oscillations caused by cavity resonance. They can also be used to recreate a free space environment by eliminating reflections in an anechoic chamber. Often after a circuit is designed and tested it must be properly shielded and physically protected before it can be put into use. Due to the increase amount of circuit-related technology, the demand for microwave absorber has increases driven by the need optimal electrical and functional performance.

Polymers such as Polycaprolactone has been found to be used in various fields of research and industries. This polymer is often used as an additive for resins to improve their processing characteristics and their end use properties such as higher impact resistance and biodegradable. Polymer-fiber composites are inexpensive because of the abundant of natural fiber. It also able to improve mechanical and electrical properties of the composites (Ibrahim et al, 2011). OPEFB is a solid waste and was chosen for this research to utilise excessive waste produced.

The composite product from this research can be used as a substrate in many applications such as electromagnetic shielding, integrated circuits (IC), absorber and transmission lines components which can be found in mobile communication, aerospace and defence industry.

1.1 Composites

Composites consist of two constituents: matrix (binder) and fillers (reinforcement). The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions.

Composites are combinations of materials differing in composition, where the individual constituents retain their separate identities. These separate constituents act together to give the necessary mechanical strength or stiffness to the composite part. They are composed of two or more distinct phases (matrix phase and dispersed phase) and having bulk properties significantly different from those of any of the constituents.

The predominant useful materials used in our day-to-day life are wood, concrete, ceramics, and so on. Surprisingly, the most important polymeric composites are found in nature and these are known as natural composites. The connective tissues in mammals belong to the most advanced polymer composites known to mankind where the fibrous protein, collagen is the reinforcement. It functions both as soft and hard connective tissue.

Composites in structural applications have the following characteristics:

- They generally consist of two or more physically distinct and mechanically separable materials.
- They are made by mixing the separate materials in such a way as to achieve controlled and uniform dispersion of the constituents.
- They have superior mechanical properties and in some cases uniquely different from the properties of their constituents (Mayer et al, 1998).

Typically, two goals are achieved in composite making. The first goal is to improve strength, stiffness, or toughness, or dimensional stability by embedding particles or fibers in a matrix or binding phase. A second goal is to use less expensive, readily available fillers than a more expensive or scarce resin; this goal is increasingly important as petroleum supplies become costlier and less reliable. Still other applications include the use of some fillers such as glass spheres to improve processability, the incorporation of dry-lubricant particles such as molybdenum sulfide to make a self-lubricating bearing, and the use of fillers to reduce permeability. Advanced composites have high-performance fiber reinforcements in a polymer matrix material such as epoxy. Examples are graphite/epoxy, Kevlar/epoxy, and boron/epoxy composites. Advanced composites are traditionally used in the aerospace industries, but these materials have now found applications in commercial industries as well.

1.2 Classification of Composites

On the basis of matrix phase, composites are classified into metal matrix composites (MMCs), ceramic matrix composites (CMCs), and polymer matrix composites (PMCs) (Avila et al, 2003). The classifications according to types of reinforcement are particulate composites (composed of particles), fibrous composites (composed of fibers), and laminate composites (composed of laminates).

1.3 Properties of Polymer composite

The dielectric properties of polymer composite are mainly depended on the conductive fillers. Consequently, the type or nature of fillers determines the dielectric characterisation of polymer composites. Typical conductive fillers are semiconductors, metals, carbonic materials and intrinsic conductive polymers (Xu et al, 1999). Conductive Polymers, organic polymers that conduct electricity have attracted a lot of interest in recent time due to their excellent flexibility and easy preparation procedures as against the use of conventional inorganic semiconductors. Their industrial applications include flexible conductors and electromagnetic shielding devices (Ma, et al, 2005). In conventional conductive composites, carbon black particles of micro-meter sizes are used to achieve desired electrical characteristics. Researches have shown however that using high amount of filler contents can leads to a poor composite (Liang, 2007).

1.3.1 Fiber-polymer composite

A natural fiber composite with an exceptionally good combination of properties is not a dream today. Fiber-reinforced composites are known to be strong and light. This means that with proper processing techniques, fiber treatments, and compatibilizers/coupling agents, composites with optimum properties can be used to make automobiles lighter, and thus much more fuel efficient.

Few decades ago, polymers have replaced many of the conventional metals/materials in various applications. This was made possible because of the advantages polymers offer over conventional materials. The most important of this advantages are the ease of processing, productivity and cost reduction. In most of these applications, the properties of polymers are modified using fillers and fibers to suit the high strength and high modulus requirements. Fiber-reinforced polymers offer advantages over other conventional materials when specific properties are compared (Schneider et al, 1995). These composites are finding widespread applications in diverse fields from appliances to space crafts. Over the years, the attention of scientist and technologist have been drawn to the use of natural fibers instead of conventional reinforcement materials. However, some of the weaknesses of the fiber such as, incompatibility with the hydrophobic polymer matrix, the tendency to form aggregates during processing, and poor resistance to moisture serve as drawbacks to the potentials of natural fibers as reinforcement in polymers (Schloesser and Knothe, 1997).

1.4 High dielectric constant material

The continuing improvement in device density and performance has significantly impacted the feature size and complexity of the wiring structure. As very large scale integrated (VLSI) microelectronics technology has developed in this millennium, the need for specialized materials with low-K dielectric constants, as well as high-K dielectric constants, within such circuits has become critical. High-performance dielectric materials, known as high-K dielectric constant, materials, are expected to play increasingly important roles in the next generation of electronics and very large scale integrated (VLSI) microelectronics technology. Silica-based ceramic materials, such as silica (SiO_2), hafnium silicate ($HfSiO_4$), and zirconium silicate ($ZrSiO_4$), are common interlayer dielectric materials used in high density microelectronic packaging. Barium titanate ($BaTiO_3$) is a well-known dielectric materials used in many semiconductor devices due to its high values of dielectric constant but low loss factor. Many researchers have attempted to disperse high dielectric, barium titanates and other ceramic oxides, into polymers but no research on OPEFB has been done.

1.5 Characterization techniques

Both microwaves and light waves are electromagnetic waves but with different values of wavelength. The characteristics of travelling in a straight line enable them to reflect, refract, diffract, scatter, and interfere at boundary points with interacting media. Hence, their mode of interaction at the interface of these media varies due to their different wavelength. Microwave wavelengths range from 1 m to 1 mm corresponding to frequency range of 0.3 GHz to 30 GHz. This singular characteristic allows microwaves to interact with materials and structures 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) as cited by (Soleiman, 2009).

1.5.1 Permittivity

Measurement of complex dielectric constant is required not only for scientific but also for industrial applications. Areas in which knowledge of the dielectric properties of materials at microwave frequencies found application are microwave heating, biological effects of microwaves, and non-destructive testing (Weir, 1974).

Dielectric properties measurement is an important factor in defining the physical and chemical properties related to storage and energy loss in various kind of materials (Wee, et al, 2009).

The term dielectric constant is some time misleading, the dependence on frequency of dielectric materials causes it to have two parts, that is the real and imaginary permittivity. The ratio of the imaginary part to the real part of permittivity is called loss tangent (Kittel, 1996). Permittivity are complex numbers of which the imaginary part is associated with losses.

Scattering parameter and permittivity of materials measured using microwaves components are controlled by the basic properties of microwaves. In good conducting materials, microwave has low penetrating depth. For this reason, they are usually used to test non-conducting materials which include low-loss and lossy dielectric materials. To investigate the interaction between microwaves and materials, Maxwell's equation is often employed. Properties like propagation mode, reflection, refraction, transmission and impedance are defined from the equation. The broad nature of material properties allows the use of different techniques for measurement at microwave frequency range. A number of methods have been used in the measurements of electromagnetic properties at microwave frequencies. Amongst these methods are the transmission and reflection line technique, open ended coaxial probe technique, and resonant method (Agilent Tech, 2011). Details of these techniques would be discussed in the ensuing chapters.

1.5.2 Morphological Properties

X ray diffraction (XRD) is a non-destructive technique for the characterization of semi crystalline and crystalline materials. XRD investigates crystalline materials structure, phases, atomic orientations, and other structural parameters, such as average crystallite size, crystallinity, strain, and imperfections. X ray diffraction peaks are produced by constructive interferences of monochromatic beam of x rays scattered at specific angles from each set of lattice planes in a sample. The XRD technique is based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy.

1.6 Problem Statement

The research on high dielectric materials have been conducted immensely by ceramic and polymer researcher. In the past researches, substrates is made from commercial polymeric materials, however none of these materials are biodegradable. Most researches on OPEFB are targeted on polymer and polymer Fe_2O_3 composite but the need to study all materials together has been neglected. For this reason, this research is looking into all combination.

OPEFB natural fibers have caught the interest of many researchers due to its low density and low price, lighter, harmless, biodegradable, renewable, and their mechanical properties that can be comparable to those of inorganic fibers. Due to the abundant of OPEFB in Malaysia as an industrial waste product, the need to reuse this material has been significantly increases.

This work explore the application of a composite consisting of OPEFB, a biodegradable PCL and Fe_2O_3. This is because the mechanical properties, electrical properties of the natural fibers are dependent on the complex permittivity of the material.

The dielectric properties, transmission and reflection coefficients of the OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composite of various filler content were analysed theoretically and experimentally.

1.7 Objectives

The main objective of this work is to develop composites made of oil palm empty fruit bunch fiber (OPEFB) composites and polycaprolactone (PCL) for reduction of electromagnetic interference. Additionally OPEFB-PCL-Fe_2O_3 composites were also fabricated and characterized for comparison purpose. The specific objectives of this study are enumerated below;

1. To fabricate OPEFB-PCL and OPEFB-PCL-Fe_2O_3 substrates for XRD and microwave characterization techniques
2. To determine the effect of % OPEFB filler and % Fe_2O_3 filler on the values of the transmission and reflection coefficient of the OPEFB-PCL and OPEFB-PCL- Fe_2O_3 composites.
3. To determine the microwave characteristics of the OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composites in the frequency range from 8 to 12 GHz.
4. To compare the results obtained from transmission with the results from COMSOL simulation.

1.8 Scope of Study

In this study, an easy and lesser time consuming technique for preparing OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composites using the melt blending technique via Brabender melt blending machine would be carried out. The composites must be put in a room lower than 60ºC as the melting point of PCL is 60 ºC.

The effect of the different % OPEFB filler and % Fe_2O_3 on the dielectric properties would be measured using the open ended coaxial probe, and rectangular waveguide techniques. The effect of the OPEFB filler on the transmission and reflection coefficient also the power loss of the OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composite are also studied. It also proposes to use FEM COMSOL software in calculating scattering parameters and for simulating electromagnetic wave excited through OPEFB-PCL and OPEFB-PCL-Fe_2O_3 composites samples when placed inside a rectangular wave guide. The result obtained for scattering parameter through measurement and simulation also compared. The morphological characterization would be carried out using the XRD.

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