



UNIVERSITI PUTRA MALAYSIA

***DEVELOPMENT OF POLYLACTIC ACID FILLED WITH OPEFB
FIBER, ACTIVATED CARBON AND MWCNTs COMPOSITES USED
AS EMI SHIELDING MATERIALS***

IBRAHIM ISMAILA LAKIN

FS 2021 29



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By

IBRAHIM ISMAILA LAKIN

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

February 2021

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DEDICATION

I hereby dedicate this thesis to the Almighty Allah who has been my help, sustainer, provider, guide, encouragement, and keeper throughout my studies and also to my parents (Late Alhaji Ibrahim Lakin and Maimuna (Kande) Pate), whose prayers and support has kept me going. Finally to my wife, brothers, and sisters who are always by my side.



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

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February 2021

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Microwave absorbers are used in a wide range of applications to eliminate or reduce electromagnetic radiation that could interfere with a system's operation. There are several materials such as metals, various carbonaceous materials (graphene, carbon nanotubes, graphite powder and carbon fibers), conductive polymers, and their composites which are the ideal candidates for microwave absorbing applications. Conventionally metals have been used for this reason for a long time and act as the most effective microwave absorber, but they have drawback for corrosion, heavy weight and difficult processability. Industries are now concentrating on easy-to-process electrically conducting materials based on conductive polymers, conductive fillers in polymer matrix that not only have semiconductor properties but can also withstand corrosive environments, inexpensive and are light in weight. This thesis presents the development of microwave absorbers using polylactic acid (PLA) and empty fruit bunch (OPEFB) as the host. Two types of absorbers were developed in this work as an alternative for reducing the limitations of metal-based microwave absorbing materials. The first absorber utilized activated carbon (AC) fillers synthesized from OPEFB. The second type used commercial multiwalled carbon nanotubes (MWCNTs) fillers obtained from US Research Nanomaterials, Inc. OPEFB fiber was crushed and sieved to various fiber sizes using laboratory sieve test. The relationship between the different OPEFB fiber sizes and the dielectric properties was then determined.

The material composition and structural properties were analyzed using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy dispersive X-ray analysis (EDX), and fourier transform infrared (FTIR) techniques. The relative complex permittivity of the composites was measured using open-ended coaxial probe technique while the microwave absorption properties were measured with the microstrip. The results showed that relative complex permittivity of the OPEFB fiber increased with reduced fiber size. Additionally, relative complex permittivity of the OPEFB-PLA,

OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs increased with filler content and were higher in the OPEFB-PLA-MWCNTs composites. At 10 GHz the dielectric constants of OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites were found to be between 2.98 to 3.40, 3.14 to 3.96 and 3.40 to 4.25 respectively while the loss factor values were from 0.34 to 0.48, 0.41 to 0.52 and 0.51 to 0.64. The measured $|S_{11}|$ and $|S_{21}|$ were used to determine the reflection loss (SE_R), absorption loss (SE_A), and total electromagnetic interference (EMI) shielding effectiveness (SE_T) of all the composites. The SE_T values were found to increase with filler loadings and were higher in the OPEFB-PLA-MWCNTs composites than the OPEFB-PLA and OPEFB-PLA-AC composites which is ascribed to the MWCNTs' high loss factor. At 12 GHz, the range of SE_T values for OPEFB-PLA-MWCNTs composites was from 20.31 to 25.01 dB while OPEFB-PLA-AC and OPEFB-PLA composites were from 17.45 to 22.25 dB and 10.67 to 12.50 dB respectively, which suggest their usefulness for microwave absorption. AC derived from OPEFB fiber has the potential for use as a filler in polymeric composites and its application can reduce the cost of MWCNTs-based microwave absorbing materials significantly.

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

**PEMBANGUNAN ASID POLIKLAKTIK DIISI DENGAN SERAT OPEFB,
KARBON AKTIF DAN KOMPOSIT MWCNTS DIGUNAKAN SEBAGAI
BAHAN PENGHANTARAN EMI**

Oleh

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Penyerap mikrogelombang digunakan dalam pelbagai kegunaan untuk menghilangkan atau mengurangkan sinaran elektromagnet yang boleh mengganggu operasi sistem. Terdapat beberapa bahan seperti logam, pelbagai bahan berkarbonat (graphene, nanotub karbon, serbuk grafit dan gentian karbon), polimer berkonduksian, dan kompositnya yang sangat sesuai untuk kegunaan penyerap mikrogelombang. Secara konvensional, logam telah lama digunakan untuk tujuan tersebut dan ia bertindak sebagai penyerap mikrogelombang yang paling berkesan, tetapi ia sukar untuk diproses, berat dan boleh menyebabkan hakisan. Kini, industri tertumpu kepada bahan pengalir elektrik yang mudah diproses berasaskan polimer berkonduksian, pengisi berkonduksian dalam matriks polimer bukan sahaja bersifat semikonduktor tetapi juga murah, ringan dan dapat menahan persekitaran yang mengkakis. Kajian ini membentangkan perkembangan penyerap mikrogelombang menggunakan asid polilaktik (PLA) dan tandan kosong (OPEFB) sebagai perumah. Dua jenis penyerap dibangunkan dalam kajian ini sebagai alternatif dalam mengurangkan kekurangan bahan penyerap mikrogelombang berasaskan logam. Penyerap pertama menggunakan pengisi karbon aktif (AC) yang disintesis daripada OPEFB. Jenis kedua menggunakan pengisi nanotub karbon berbilang dinding komersial (MWCNTs) yang diperolehi dari US Research Nanomaterials, Inc. Serat OPEFB banyak dijumpai sebagai sisa pepejal dari kilang kelapa sawit. Serat OPEFB dihancurkan dan diayak kepada pelbagai saiz serat menggunakan ujian saringan makmal. Hubungan antara pelbagai saiz serat OPEFB dan pemalar dielektrik ditentukan.

Komposisi bahan dan sifat struktur dianalisis dengan menggunakan belauan sinar-X (XRD), pemancaran medan mikroskopi imbasan elektron (FESEM), analisis tenaga serakan sinaran-X (EDX), dan teknik transformasi Fourier inframerah (FTIR). Kebertelusan komposit relatif rumit diukur menggunakan teknik kuar sepaksi terbuka sementara itu, sifat serapan mikrogelombang diukur menggunakan mikrojalur. Dapatan

menunjukkan kebertelusan komposit relatif rumit serat OPEFB meningkat dengan saiz serat yang dikurangkan. Selain itu, kebertelusan komposit relatif rumit bagi OPEFB-PLA, OPEFB-PLA-AC, dan OPEFB-PLA-MWCNT meningkat dengan kandungan pengisi dan lebih tinggi di dalam komposit OPEFB-PLA-MWCNT. Pada 10 GHz, pemalar dielektrik OPEFB-PLA, OPEFB-PLA-AC, dan komposit OPEFB-PLA-MWCNT masing-masing didapati berada di antara 2.98 hingga 3.40, 3.14 hingga 3.96 dan 3.40 hingga 4.25. Sementara itu, nilai faktor kerugian adalah dari 0.34 hingga 0.48, 0.41 hingga 0.52 dan 0.51 hingga 0.64. Ukuran $|S_{11}|$ and $|S_{21}|$ digunakan untuk menentukan kehilangan pantulan (SE_R), kehilangan serapan (SE_A), dan jumlah gangguan elektromagnet (EMI) keberkesanan pemerisaian (SE_T) bagi semua komposit. Nilai SE_T didapati meningkat dengan muatan pengisi dan lebih tinggi pada komposit OPEFB-PLA-MWCNTs berbanding komposit OPEFB-PLA dan OPEFB-PLA-AC yang dikaitkan dengan faktor kehilangan tinggi MWCNT. Pada 12 GHz, julat nilai SE_T bagi komposit OPEFB-PLA-MWCNT adalah dari 20.31 hingga 25.01 dB sementara komposit OPEFB-PLA-AC dan OPEFB-PLA masing-masing dari 17.45 hingga 22.25 dB dan 10.67 hingga 12.50 dB. Ini membuktikan kebergunaannya dalam serapan mikrogelombang. AC, terbitan dari gentian OPEFB berpotensi untuk digunakan sebagai pengisi dalam komposit polimer dan kegunaannya dapat mengurangkan kos bahan penyerap gelombang mikro berasaskan MWCNT dengan ketara.

ACKNOWLEDGEMENTS

In the name of Allah, the most gracious and the most merciful who has thought man by pen. All thanks to Allah who has allowed me to see this day, the day I have been dreaming of, may the peace and blessings of Allah be upon his noble prophet Muhammad (SAW). First of all, I would like to use this opportunity to express my heartfelt thankfulness to my supervisor Assoc. Prof. Zulkifly Abbas PhD, for his support, advice, guidance, and encouragement which helped me towards achieving this goal. My sincere appreciation also goes to members of my supervisory committee; Assoc. Prof. Dr. Nor Azowa Ibrahim, and Dr. Mohd Amiruddin Abd Rahman for kind guidance and support towards the completion of this project.

I would also like to extend my sincere gratitude to Kaduna State University (KASU) for the sponsorship, my appreciation also goes to Universiti Putra Malaysia for the opportunity given to pursue my goals.

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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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	iii
ACKNOWLEDGEMENTS	v
APPROVAL	vi
DECLARATION	viii
LIST OF TABLES	xiii
LIST OF FIGURES	xv
LIST OF ABBREVIATIONS AND SYMBOLS	xix
CHAPTER	
1 INTRODUCTION	1
1.1 Problem Statement	2
1.2 Research Objectives	3
1.3 The Scope of the study	4
1.4 Organization of the Thesis	4
2 LITERATURE REVIEW	6
2.1 Microwave Absorbers	6
2.2 OPEFB Fiber Properties	9
2.3 Polylactic acid (PLA) Properties	12
2.4 Activated Carbon (AC) Properties	13
2.5 Multiwalled carbon nanotubes (MWCNTs) Properties	16
2.6 Overview of Composites Preparations	16
2.7 XRD Characterization	18
2.8 Microwave Characterization Techniques	19
2.8.1 Open Ended Coaxial (OEC) Probe	19
2.8.2 Completely Filled Rectangular Waveguide (RWG)	19
2.8.3 Microstrip Line Technique	20
2.9 Electromagnetic Interference Shielding Effectiveness	21
2.10 Numerical Solutions	22
2.10.1 Finite Element Method (FEM)	22
2.10.2 Method of Moment (MoM)	23
2.10.3 Finite Difference Method (FDM)	24
2.11 Summary	24
3 METHODOLOGY	25
3.1 Sample preparation	26
3.1.1 OPEFB preparation	26
3.1.2 Synthesis of AC	26
3.1.3 Preparation of composites	27
3.2 Dielectrical Characterization	29

3.2.1	Permittivity Measurement (Open Ended Coaxial Technique)	29
3.3	Scattering Parameters Measurements	30
3.3.1	Microstrip Measurement Technique	30
3.3.2	Finite Element Method (FEM)	32
3.4	Structural and Morphological Characterization	38
3.4.1	X-Ray Diffraction (XRD)	38
3.4.2	Field Emission Scanning Electron Microscopy (FESEM)	39
3.4.3	Transmission Electron Microscopy (TEM)	39
3.4.4	Fourier Transform Infrared (FTIR) Spectroscopy	40
3.4.5	BET Analysis	40
3.4.6	Error Analysis	40
3.5	Summary	41
4	RESULTS AND DISCUSSION	42
4.1	X-Ray Diffraction (XRD)	42
4.1.1	XRD of OPEFB-PLA Samples	42
4.1.2	XRD of OPEFB-PLA-AC	43
4.1.3	XRD of OPEFB-PLA-MWCNTs	44
4.2	FESEM and EDX spectrum	46
4.2.1	OPEFB, PLA, and AC	46
4.2.2	OPEFB-PLA	49
4.2.3	OPEFB-PLA-AC composites	50
4.2.4	OPEFB-PLA-MWCNTs composites	51
4.3	FTIR analysis	52
4.4	Permittivity results Using Open-ended Coaxial Probe Technique	55
4.4.1	PLA-OPEFB samples different grain size	56
4.4.2	OPEFB-PLA different weight percentages	59
4.4.3	OPEFB-PLA-AC	64
4.4.4	OPEFB-PLA-MWCNTs	68
4.5	Material Characterization using Microstrip Technique	71
4.6	Scattering Parameters $ S_{11} $ and $ S_{21} $ of the Open and Covered Microstrip	76
4.6.1	Variation of measured $ S_{11} $ and $ S_{21} $ magnitudes	76
4.6.2	Comparison of Measured and Simulated Scattering Parameters (Microstrip)	80
4.6.3	Microstrip Covered with OPEFB-PLA composites	81
4.7	Material Reflectance and Absorbance	87
4.7.1	Total Shielding Effectiveness	91
4.8	Summary	94
5	SUMMARY AND FUTURE WORK	95
5.1	Summary of the Study	95
5.2	Main Contributions and Findings	95
5.3	Recommendations for future studies	96

REFERENCES	97
APPENDICES	110
BIODATA OF STUDENT	113
LIST OF PUBLICATIONS	114



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LIST OF TABLES

Table		Page
2.1	EMI shielding performance of various composites	9
2.2	Mean complex permittivity of different OPEFB powder fiber size at 1-4 GHz. (Abdalahadi et al., 2017)	12
2.3	Mean complex permittivity of different OPEFB powder fiber size at 1-4 GHz. (Abdalahadi et al., 2017)	12
2.4	Summary of AC preparation techniques	15
2.5	Summary of polymer composites preparation techniques	17
2.6	Comparison between FEM, MoM, and FDM techniques	23
3.1	Raw materials used for preparation of OPEFB-PLA composites	28
3.2	Raw materials used for preparation of OPEFB-PLA-AC composites	28
3.3	Raw materials used for preparation of OPEFB-PLA- MWCNTs composites	28
3.4	Properties of rectangles for geom2	32
3.5	Properties of circles for Geom3	33
3.6	Properties of rectangle for Geom3	33
3.7	Boundary condition and boundary's number	35
4.1	EDX analysis of OPEFB	48
4.2	Summary of various peaks and their corresponding vibrations	54
4.3	Empirical equation, the regression coefficient for a dielectric constant with respect to OPEFB content	61
4.4	Empirical equation, the regression coefficient for loss factor with respect to OPEFB content	63
4.5	Real and imaginary parts of permittivity for all samples at 8-12 GHz	64
4.6	Empirical equation, regression coefficient for the dielectric constant with respect to AC content	66

4.7	Empirical equation, the regression coefficient for the loss factor with respect to AC content	67
4.8	Empirical equation, regression coefficient, for dielectric constant with respect to MWCNTs content	69
4.9	Empirical equation, regression coefficient for the loss factor with respect to MWCNTs content	70
4.10	The mean relative error of $ S_{11} $ and $ S_{21} $ for open microstrip	81
4.11	The mean relative error with respect to OPEFB content for $ S_{21} $	86
4.12	The mean relative error with respect to AC content for $ S_{21} $	86
4.13	The mean relative error with respect to MWCNTs content for $ S_{21} $	87
4.14	Comparison of SET for various composites	92

LIST OF FIGURES

Figure		Page
2.1	EMI SE values of segregated PLLA/PCL/MWCNTs/Ni composites with respect to the alignment of Ni particles (Shi et al., 2018)	8
2.2	Absorption loss for OPEFB/PLA/F ₂ O ₃ composites for different percentages of F ₂ O ₃ filler (Abdalahadi et al., 2018)	8
2.3	Classification of Natural Fiber	9
2.4	Structural organization of the three major constituents in the fibre cell wall (Abdalahadi, 2018)	10
2.5	Chemical structure of (a) cellulose (b) hemicelluloses (c) lignin (Abdalahadi, 2018)	11
2.6	X-ray Diffractograms of (a) jute stick powder (JS), (b) Hydrochar (JSC) and (c) Hydrochar activated carbon (JSCAC) (Choudhury et al., 2016)	18
2.7	A sketch of filled dielectric waveguide (a) reflection only and (b) reflection and transmission	20
3.1	Flow chart of methodology	25
3.2	Preparation process of OPEFB substrate	26
3.3	OPEFB fiber activation process	27
3.4	Preparation of substrate	29
3.5	Schematic diagram of complex permittivity measurement using OEC	30
3.6	Fabricated microstrip	31
3.7	Schematic diagram of S-parameters measurement using the microstrip sensor	31
3.8	Application scalar variables dialog box	34
3.9	Subdomain settings dialog box	35
3.10	Boundaries involved in perfect electric conductor (PEC) boundary condition	36
3.11	Schematic Diagram of Meshed Microstrip for FEM Simulation	37

3.12	Philips X-pert system X-ray diffractometer	38
3.13	Image of Nova NanoSEM 230 FESEM	39
4.1	XRD patterns of OPEFB, PLA, and OPEFB-PLA% composites	43
4.2	XRD patterns of and OPEFB-PLA-AC composites	44
4.3a	XRD patterns of and OPEFB-PLA-MWCNTs composites	45
4.3b	RD pattern of MWCNTs (Bibi et al., 2017)	45
4.4	(a) FE-SEM of OPEFB and (b) EDX spectrum	47
4.5	FE-SEM of PLA and EDX spectrum	48
4.6	FE-SEM of AC and EDX spectrum	48
4.7	FE-SEM Micrographs of OPEFB-PLA composites (a) 20%, (b) 30%, (c) 40%, (d) 50% and (d) 60% OPEFB	49
4.8	FE-SEM of OPEFB-PLA-AC% composites	50
4.9	TEM images of (a), (b) MWCNTs and FE-SEM images of (c) OPEFB/PLA/6% MWCNTs (d) OPEFB/PLA/22% MWCNTs	51
4.10	FTIR spectrum of OPEFB-PLA% compositions	53
4.11	FTIR spectrum of OPEFB-PLA-AC% compositions	54
4.12	FTIR spectrum of OPEFB-PLA-MWCNTs% compositions	55
4.13	Variation in dielectric constant with frequency	57
4.14	Variation in loss factor	58
4.15	Variation in dielectric constant with respect to grain size	58
4.16	Variation in loss factor with respect to fiber size	59
4.17	Variation in the dielectric constant of OPEFB-PLA with respect to frequency	60
4.18	Variation in loss factor with respect to frequency	61
4.19	Variation in dielectric constant with respect to OPEFB at selected frequencies	62
4.20	Variation in loss factor with respect to OPEFB at selected frequencies	63

4.21	Variation in dielectric constant of OPEFB-PLA-AC with respect to frequency	65
4.22	Variation in loss factor of OPEFB-PLA-AC with respect to frequency	65
4.23	Variation in loss factor with respect to AC at selected frequencies	66
4.24	Variation in loss factor with respect to AC at selected frequencies	67
4.25	Variation in the dielectric constant of OPEFB-PLA-MWCNTs with respect to frequency	68
4.26	Variation in loss factor of OPEFB-PLA-MWCNTs with respect to frequency	69
4.27	Variation in dielectric constant with respect to MWCNTs at selected frequencies	70
4.28	Variation in loss factor with respect to MWCNTs content at selected frequencies	71
4.29	Electric field distribution and intensity plot for the different OPEFB-PLA composites (a) unloaded microstrip (b) 20% (c) 30% (d) 40% (e) 50% (f) 60% OPEFB	73
4.30	Electric field distribution and intensity plot for the different OPEFB-PLA-AC composites (a) 6% (b) 10% (c) 14% (d)18% (e) 22% AC	74
4.31	Electric field distribution and intensity plot for the different OPEFB-PLA-MWCNTs composites (a) 6% (b) 10% (c) 14% (d)18% (e) 22% MWCNTs	76
4.32	$ S_{11} $ for (a) OPEFB-PLA (b) OPEFB-PLA-AC and (c) OPEFB-PLA-MWCNTs composites using micro strip	77
4.33	(a) $ S_{21} $ for OPEFB-PLA% (b) $ S_{21} $ for OPEFB-PLA-AC and (c) $ S_{21} $ OPEFB-PLA-MWCNTs composite using microstrip	79
4.34	(a) S11 and (b) S21 for an open (without sample) micro strip	80
4.35	Variation in $ S_{11} $ for (a) OPEFB-PLA (b) OPEFB-PLA-MWCNTs and (c) OPEFB-PLA- MWCNTs composites for measurement method and FEM	84
4.36	Variation in $ S_{21} $ for (a) OPEFB-PLA (b) OPEFB-PLA-AC and (c) OPEFB-PLA- MWCNTs composites for measurement method and FEM	86
4.37	Variation in SER for (a) OPEFB-PLA (b) OPEFB-PLA-AC and (c) OPEFB-PLA- MWCNTs composites with frequency	89

4.38	Variation in SEA for (a) OPEFB-PLA (b) OPEFB-PLA-AC and (c) OPEFB-PLA- MWCNTs composites with frequency	91
4.39	Variation in SET for (a) OPEFB-PLA (b) OPEFB-PLA-AC and (c) OPEFB-PLA-MWCNTs composites with frequency	93



LIST OF ABBREVIATIONS AND SYMBOLS

ω	Angular frequency
η_0	Impedance in free space
∇	Laplacian vector
γ	Propagation constant
ρ_q	The electric charge density
ϵ_r	The relative dielectric of the substrate
ABC	Absorbing boundary condition
AC	Activated carbon
B	The magnetic flux density
BET	Brunauer Emmett, and Teller
CEM	Computational electromagnetic
d	Sample thickness
D	The electric displacement
dB	Decibels
EDX	Energy-dispersive X-ray
EM	Electromagnetic
EMI	Electromagnetic interference shielding
E_z	Longitudinal electric field
f	Frequency
FDTD	Finite difference time domain
FEM	Finite Element Method
FESEM	Field electron scanning electron microscopy

FTIR	Fourier transforms infrared
H_z	Longitudinal magnetic field
J	The current density
k_0	Free-space wave number
MoM	Method of Moment
MUT	Materials under test,
MWCNTs	Multiwalled carbon nanotubes
OECP	Open Ended Coaxial Probe
OPEFB	Oil palm empty fruit bunch
P_1	Power measured with the material inserted
P_2	Power measured without material inserted
PANI	Polyaniline
PCL	Polycaprolactone
PEK	Poly (Ether Ketone)
PLA	Polylactic acids
PML	Perfectly match layer
PNA	Professional network analyzer
PVP	Polyvinylpyrrolidone
RAM	Radar absorbing material
RBC	Reflectionless boundary condition
RF	Radiofrequency
RWG	Rectangular waveguide
S_{11}	Input reflection coefficient of port one
S_{21}	Transmission coefficient port two
SE	Shielding effectiveness

SE_A	Absorption shielding effectiveness
SE_R	Reflection shielding effectiveness
SE_T	Shielding effectiveness
T/R	Reflection/Transmission
TE	Transverse Electric
TEM	Transverse electromagnetic modes
TM	Transverse Magnetic
XRD	X-ray diffraction
Z_{in}	Impedance of the absorbing material
Z_0	Impedance of free space
β	Phase constant
ϵ'	Dielectric constant
ϵ''	Loss factor
ϵ^*	Complex permittivity
μ	Permeability
σ	Electrical conductivity
ϵ	Permittivity

CHAPTER 1

INTRODUCTION

The need for microwave absorbers and radar-absorbing materials are on the rise in military application dealing with reduction of radar signature air crafts and ships whilst in civilian applications dealing with electromagnetic interference among electronic and telecommunication systems. Composite absorber that uses carbon based materials in conjunction with a polymer matrix produces flexibility which can be manipulated by changes (in formulations) in both the filler and the matrix. Depending on the application for which the absorber is intended, the percentage of filler and the matrix are two important factors to be understood. In addition, microwave absorption properties are determined by the complex permittivity and permeability, sample thickness, microstructure of the absorber, and class of material. In the industry of aerospace, for example, innovative solutions are needed to shield effectively sensitive electronic equipment such as antennas from EMI without adding much of weight to aircraft. In the past, the problem of EMI was tackled by isolating the electronic device through some metallic housing. Metals are excellent conductors of electricity that are capable to absorb, reflect, and transmit electromagnetic interference. Metals like Silver, Iron, Aluminum, Nickel Copper, and Barium were used in most of the EMI shielding applications (Xiang et al., 2007). The disadvantage was that the metals were easily susceptible to oxidation or corrosion and so could not be utilized for outside applications. The heavyweight and price of the metal shields also limit the use of metals as shielding materials (Jagatheesan et al., 2015). Nowadays, the most common method of shielding by reflection is the use of metallic plates or absorption by carbon-based materials. Polymeric materials have also acquired popularity due to their flexibility, lightweight, corrosion resistance, and lower cost to metals. Researches had also been carried out on the applications of polymer composites incorporated with conductive fillers, fibers, nanotubes, and dispersing particles (Chen et al., 2015). The metal plate problems centered primarily on the discomfort of poor mechanical flexibility due to high rigidity, high weight density and high cost. Low density, high conductivity, efficient broadband absorption, and excellent thermal stability are the main parameters for a typical EM absorbing material. The microwave absorption mechanism classifies the materials into two main sections; dielectric loss materials and materials that are magnetically lossy. (Arief et al., 2017). The design of an EMI shielding material with a certain degree of attenuation while meeting a set of environmental requirements, retaining economics and regulating shielding have been suggested. The main purpose behind the proper design of the shield is to create a product that can comply with International electromagnetic Interference Regulatory Standards. The research of new active materials used as microwave absorbers for the EMI shielding of various electronic devices is one of present-day activities (Paligova et al., 2004).

Measurement of complex permittivity is required not only for scientific but also for industrial applications. 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 misleading,

the dependency on dielectric material frequency 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 referred to as loss tangent. Permittivity and permeability are complex numbers of which the imaginary part is correlated with losses.

Scattering parameter, permittivity and permeability of materials measured using microwaves components are controlled by the basic properties of microwaves. In good conducting materials, microwave has low penetrating depth. To investigate the interaction between microwaves 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, free space measurement technique, open-ended coaxial probe technique, and resonant method (Teppati et al., 2013).

1.1 Problem Statement

The incorporation of OPEFB fiber into PLA matrix is expected to improve the complex permittivity, absorption, and attenuation as well as reduce the fabricated composites transmission coefficient making a better microwave absorbing material. OPEFB-PLA composites have been used extensively in many microwave application. However, due to lack of detailed knowledge on the relationship between the filler composition and the electromagnetic properties, its potential has not been fully exploited. The dielectric properties and scattering parameters of OPEFB-PLA composites of different filler and types, matrix and material properties have not been studied in depth both theoretically and experimentally (Abdalahdi et al., 2018). In developing absorbing composites, ferrites are commonly used. However, like any other metals ferrites are heavy, corrosive, expensive, and non-biodegradable. Therefore, the use of metals such as ferrites for EMI shielding could lead to galvanic corrosion which in turn increased the nonlinearity behavior and decreased its shielding effectiveness.

Oil palm empty fruit bunch fiber (OPEFB) offers various advantages such as low cost, low density, better thermal and insulating properties, good flexural strength, and biodegradability. Although microwave properties of OPEFB-PLA/PCL reinforced with Fe_2O_3 have recently been studied (Abdalahdi et al., 2018; Mensah et al., 2019) but the dielectric loss factor for the nanocomposites was found to be low and microwave absorbers need to have high loss factor for higher absorbing properties. In this research AC was synthesized from OPEFB and the dielectric properties subsequently improved after activating the OPEFB fiber via physical activation. The AC with improved dielectric properties could be useful in reducing the limitations of the frequently used fibers for microwave absorption applications. Recently, the addition of multiwalled carbon nanotubes (MWCNTs) into metal, ceramic, or polymer matrix has been carefully studied and a wide variety of different characteristics have been found (Kim et al., 2018 and Mondal et al., 2018). The incorporation of AC and MWCNTs fillers into OPEFB-

PLA composites could enhanced the permittivity and microwave absorbing properties of OPEFB-PLA-AC and OPEFB-PLA-MWCNTs composites.

The conventional method to determine the complex permittivity of the composites materials is to place the sample in a closed waveguide. The approach is difficult as the sample must be firmly placed into the waveguide avoiding any air gaps. In this work, the open-ended coaxial probe (OEC) technique was used to investigate the complex permittivity of OPEFB-PLA, OPEFB-PLA-MWCNTs, and OPEFB-PLA-AC composites. The microwave attenuation due to sample does not only depend on the complex permittivity but also the sample thickness. Thick samples measurements are always problematic when using waveguide technique due air gap problems. In this research, the attenuation of the samples was also analyzed using the open line transmission (microstrip) technique. Visualization of the electromagnetic field in the samples was carried out where the samples were discretize into small meshes using the Finite Element Method (FEM)

1.2 Research Objectives

The main objectives of this research are as follows;

1. To characterize the structure, microstructure and elemental properties of OPEFB-PLA, OPEFB-PLA-MWCNTs composites using XRD, FTIR, FESEM, and EDX techniques.
2. To determine the effect of OPEFB fiber size on its dielectric constant and loss factor values
3. To determine the complex permittivity and scattering parameters of OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites using the open-ended coaxial probe and microstrip line technique respectively. Also to calculate the Scattering parameter values of the composites using FEM and compare the results with microstrip line technique then visualize the electromagnetic field distribution of the composites using FEM for various filler percentage.
4. To calculate the reflection loss, absorption loss, and total EMI shielding effectiveness of OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites.

1.3 The Scope of the study

In this study, the melt blending technique using a Brabender machine to fabricate OPEFB-PLA biocomposites with MWCNTs and AC incorporated into the composites to enhance their electrical properties. The effect of the different percentages of MWCNTs and AC filler concentrations on the dielectric properties would be investigated using the open-ended coaxial probe technique. The use of a microstrip transmission line would be made to investigate the effect of MWCNTs and AC filler on the scattering parameters of OPEFB-PLA-AC and, OPEFB-PLA-MWCNTs composites. FEM COMSOL software would also be used to calculate the scattering parameters and for simulation of electromagnetic waves excited through OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites samples when placed on the microstrip transmission line. The results obtained through measurement and simulation would also be compared. The microstructure, elemental and bonding types would be characterized using equipment such as XRD, FESEM, EDX, and FTIR.

1.4 Organization of the Thesis

This thesis consist of six chapters and appendix. Chapter one gives the general introduction on EMI and the materials used for this research. Followed by the statement of the problem, objectives of the study, the scope of the study and the thesis layout.

Chapter two presents the reviews on the properties of OPEFB fiber, PLA, AC, and MWCNTs. Composite material synthesis methods and microwave characterization techniques are also reviewed in this chapter. Finally, the FEM as a numerical technique for the simulation of electrical field distribution and the determination of the S-parameters of samples placed on the microstrip is reviewed.

Chapter 3 presents the theory chapter and it discusses the theoretical concepts of the dielectric properties, polarization and basic electromagnetic wave equations. It concludes with the transmission and reflection coefficients calculation procedures with FEM formulation techniques. Furthermore, sample preparation, morphological, microstructural and electromagnetic characterizations are discussed in chapter 4. The use of the OEC, microstrip and FEM method are fully discussed in relation to the electromagnetic characterization using XRD, FESEM, EDX, 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 morphological and microstructural characterizations of the OPEFB fiber, PLA, AC powder, OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites using measurement technique such as XRD, FESEM, EDX, and FTIR. This is followed by the second section which presents and discusses the results of the complex permittivity of the OPEFB fiber, PLA, AC

powder, and the fabricated composites using the OEC technique. The third section of the chapter presents and discusses the results of FEM simulation and visualization of electric field intensity distribution for the OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites based on the microstrip. The fourth and fifth sections discuss the results of the S-parameters measurement of the OPEFB-PLA, OPEFB-PLA-AC, and OPEFB-PLA-MWCNTs composites using the microstrip technique and FEM respectively. The sixth section discusses the results of material absorption where the S-parameters S_{11} and S_{21} of the composites obtained from the microstrip technique were used to calculate the reflection shielding effectiveness (SE_R), absorption shielding effectiveness (SE_A) and the total shielding effectiveness (SE_T) of the composites.

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



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