UNIVERSITI PUTRA MALAYSIA

MICROWAVE ABSORBER BASED ON BIOCOMPOSITES DERIVED FROM AGRICULTURAL WASTES WITH REINFORCEMENT OF POLY LACTIC ACID

ELFARIZANIS BINTI BAHARUDIN

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By

ELFARIZANIS BINTI BAHARUDIN

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

January 2021

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DEDICATION

This thesis is dedicated to

Those who kept faith in me:

With love, respect and a bunch of memories
There is neither might nor strength except with Allah
Indeed, we belong to Allah and indeed to Him we will return.

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

MICROWAVE ABSORBER BASED ON BIOCOMPOSITES DERIVED FROM AGRICULTURAL WASTES WITH REINFORCEMENT OF POLY LACTIC ACID

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ELFARIZANIS BINTI BAHARUDIN

January 2021

Chairman : Professor Alyani binti Ismail, PhD
Faculty : Engineering

The development of new microwave absorber (MA) materials has been motivated by an increasing ecological and environmental consciousness. Biocomposites is natural fibre-reinforced polymers has low density and biodegradable nature obtain from natural fibers which are cheap, continuous supply, easy and safe handling. The idea of this research is to develop a MA from biocomposite derived from oil palm fronds (OPF), empty fruit bunch (EFB), pineapple leaf fiber (PALF) and strengthened with poly-lactic acid (PLA). The fibers samples are prepared in 0.3 mm particle size, meanwhile the polymer and biocomposites are in ring shaped forms using mix and blend process and injection moulding. Their dielectric constant, (ε') and loss tangent, (tan δ) are extracted using open ended coaxial probe and S-parameter Reflection measurements. OPF, EFB and PALF show ε'with 1.92, 1.88 and 2.04 respectively and tan δ with 0.105, 0.096 and 0.102 respectively. PLA ε' and tan δ is about 1.99 ± 0.25 and 0.044 ± 0.02 respectively in the X band. Each type of biocomposite mixture of fiber and PLA composition (wt%) namely OPFPLA, EFBPLA, and PALFPLA prepared in 10 wt%, 20 wt%, 30 wt%, 40 wt% and 50 wt% fiber loadings. The optimum ε' and tan δ for each biocomposite is 2.718 and 0.165 at fo = 8.6 GHz for OPFPLA5050 with 50 wt% OPF and 50 wt% PLA, meanwhile 2.079 and 0.168 at fo = 8.8 GHz for EFBPLA5050 with 10 wt% EFB and 90 wt% PLA and 2.23 and 0.09 at fo = 8.4 GHz for PALFPLA with 50 wt% PALF and 50 wt% PLA. The MA performance is observe from CST simulation, where S_{11} and S_{21} parameters are the key factors of MA absorption rates (AR), fractional bandwidth (FBW) and its thickness, d is used to determine the new formulation of figure of merit (FOM) for the percentage of wave absorption per unit milimeter (mm). OPFPLA5050 MA with d = 9 mm resonates at 8.6 GHz show the optimum performance with AR of 40.9%, FBW of 31.2% and the FOM is 4.5% per unit mm using normal incident in TE and TM polarization. When incident wave transmitted in variation angles of θ, the AR and FBW increase as θ increases until 45^{o} in TE and

TM polarization. Then, OPFPLA5050 MA is fabricated using 30 x 30 x 1 cm^3 mould and hot press machine. It is measured using free space and bistatic reflection methods. The results shows OPFPLA5050 MA with *d* = 10 mm resonates at 8.17 GHz with AR is 35.9%, FBW is 7.42 % and FOM is 3.6% per unit mm. Due to fabrication error, the MA thickness is different form the simulation although their plots show same trends except for the shifted resonant frequency. A fine-tune simulation shows that the fabricated MA consider to have ε' = 2.46 and tan δ = 0.12 and frequency at 8.17 GHz where the corresponding AR is 32%, FBW is 26.8% and FOM is 3.2% per unit mm which is close to the fabricated results. Then, a similar trend observed with the simulations when θ varies from 0º in 45º in TE and TM polarization. Therefore, these findings on natural fiber reinforced PLA biocomposites have significant implications in the application of microwave absorber. The unique composition between oil palm frond fibers and PLA demonstrates promising wave absorption for further exploration as alternative material in future research.

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

PENYERAP GELOMBANG MIKRO BERASASKAN BIOKOMPOSIT DARIPADA SISA PERTANIAN YANG DIPERKUKUH DENGAN POLI LAKTIK ASID

Oleh

ELFARIZANIS BINTI BAHARUDIN

Januari 2021

Pengerusi : Profesor Alyani binti Ismail, PhD
Fakulti : Kejuruteraan

Peningkatan kesedaran terhadap ekologi dan persekitaran menjadi motivasi kepada penyelidikan pembangunan bahan-bahan bagi menyerap gelombang mikro (MA). Biokomposit adalah serat yang diperteguhkan dengan polimer yang berkepadatan rendah dan bersifat biodegradasi dari sumber semula jadi yang murah, mudah didapati serta senang diuruskan. Idea penyelidikan ini adalah membangunkan penyerap MA dari biokomposit yang berasal dari sisa pertanian seperti pelepah kelapa sawit (OPF), tandan buah kosong (EFB) dan serat daun nanas (PALF) yang diperkuat oleh poli-laktik asid (PLA). Serat sisa pertanian tersebut dihancurkan menjadi sampel bersaiz 0.3 mm, manakala sampel PLA dan biokomposit dihasilkan seperti bentuk cincin menggunakan proses gaul dan kisar serta kaedah acuan suntikan. Sifat pemalar elektrik (ε') dan tangen kehilangan, (tan δ) bagi bahan-bahan tersebut diekstrak menggunakan kaedah prob koaksial terbuka dan refleksi S-parameter. OPF, EFB dan PALF menunjukkan nilai ε' masing-masing dengan 1.92, 1.88 dan 2.04 manakala tan δ dengan nilai 0.105, 0.096 dan 0.102. Bagi PLA, ε' dan tan δ adalah 1.99 ± 0.25 dan 0.044 ± 0.02 masing-masing pada jalur X. Setiap biokomposit OPFPLA, EFBPLA dan PALFPLA, iaitu campuran serat dan PLA, disediakan dalam komposisi (wt%) sebanyak 10 wt%, 20 wt%, 30 wt%, 40 wt% dan 50 wt% muatan serat. Nilai-nilai optimum ε' dan tan δ setiap biokomposit adalah 2.718 dan 0.165 pada fo = 8.6 GHz (OPFPLA5050) yang mempunyai 50 wt% OPF dan 50 wt% PLA, manakala 2.079 dan 0.168 pada fo = 8.8 GHz (EFBPLA5050) dengan 10 wt% EFB dan 90 wt% PLA dan akhirnya 2.23 and 0.09 pada fo = 8.4 GHz (PALFPLA) dengan 50 wt% PALF dan 50 wt% PLA. Prestasi MA dari simulasi CST dilihat pada parameter pantulan (S_{11}) dan hantaran (S_{21}) serta ia adalah kunci utama kadar serapan (AR) MA, jalur lebar pecahan (FBW) manakala ketebalan (*d*) MA adalah untuk menentukan angka merit (FOM) iaitu nisbah kadar serapan gelombang per unit milimeter (mm). OPFPLA5050 MA dengan *d* = 9 mm menghasilkan resonan pada 8.6 GHz dan ia menunjukkan prestasi optimum dengan

AR sebanyak 40.9%, FBW selebar 31.2% dan FOM adalah 4.5% per unit mm dengan hala tuju normal dalam pengutuban TE dan TM. Ketika hala tuju dipancarkan dalam sudut θ, didapati nilai AR dan FBW meningkat sehingga θ mencapai sudut 45^o pada pengutuban TE dan TM. Kemudian, OPFPLA5050 MA difabrikasi menggunakan acuan 30 x 30 x 1 cm^3 dan juga mesin tekanan panas. Ia diukur menggunakan kaedah ruang bebas serta kaedah refleksi bistatik. Keputusan menunjukkan OPFPLA5050 MA dengan d = 10 mm mempunyai resonan pada 8.17 GHz dengan AR sebanyak 35.9%, FBW selebar 7.42 % dan FOM adalah 3.6% per unit mm. Disebabkan kelemahan pada proses fabrikasi, maka hasil ketebalan MA adalah berbeza dari nilai optimum simulasi walaupun bentuk graf yang terhasil mempunyai corak yang sama kecuali pada anjakan frekuensi resonan. Simulasi pembaikan bagi menentukan perbezaan keputusan ukuran dilakukan dan didapati bahawa MA yang telah fabrikasi mempunyai ε' = 2.46 dan tan δ = 0.12 pada frekuensi 8.17 GHz. Hasil dapatannya adalah AR, 32%, FBW, 26.8% dan FOM 3.2% per unit mm di mana ia hampir sama dengan keputusan ukuran MA yang telah difabrikasi. Hasil simulasi tersebut juga menunjukkan corak yang hampir sama dengan hasil simulasi asal apabila sudut hala tuju θ dipelbagaikan dari 0^o ke 45^o dalam pengutuban TE and TM. Oleh yang demikian, penyelidikan terhadap pembangunan penyerap MA dari biokomposit yang diperbuat daripada serat semula jadi diperkukuhkan dengan PLA ini menunjukkan hasil yang signifikan dan mempunyai implikasi penting terhadap aplikasi penyerap gelombang. Komposisi yang unik antara OPF dan PLA membuktikan penyerapan gelombang dalam medium adalah meyakinkan dan ia sebagai bahan alternatif yang berpotensi untuk kajian yang lebih jauh pada masa hadapan.

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This thesis was submitted to the Senate of the 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:

Alyani binti Ismail, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Dayang Laila binti Abang Haji Abdul Majid, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Edi Syams bin Zainudin, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Adam Reda Hasan Alhawari
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Fauziahanim binti Che Seman, PhD
Associate Professor
Faculty of Electrical and Electronic Engineering
Universiti Tun Hussein Onn Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 12 August 2021

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ε	Permittivity
ε_r	Dielectric constant,
μ	Permeability
σ	Conductivity
ρ	Density
m	Mass
v	Volume
$\tan\delta$	Loss tangent
S_{11}	Reflection
S_{21}	Transmission
MHz	Mega hertz
CST	Computer Simulation Technology
EFB	Empty fruit bunches
OPF	Oil palm frond
PALF	Pineapple Leaf Fibre
PLA	Polylactic acid
MUT	Material under test
VNA	Vector network analyser
SUT.	Sample under test
CST	Computer Simulation Technology
MA	Microwave Absorber
w	Width
l	Length
d	Thickness

FOM	Figure of merit
AR	Absorption Rates
dB	Decibel

CHAPTER 1

INTRODUCTION

1.1 Research Background

Urbanization and economic growth over the past few years have increased the consumption of resources such as electronic, and energy resources. With the increment in demand due to technological change in the society, have also created an impact towards producing more new products and processes. With so many research and development to meet this needs, improvements and also limitations are revealed through the products and systems.

As the industries and manufacturer taking advantage of evolution of engineering technology, there are three principle of development which need to be considered. One is no waste where it is a discipline applied at all stages of design, appraisal, manufacturer, use, re-use, recycling and disposal. Second is the environment is to considered at every stage and finally 'valuable' where it preserves or enhances the environment and life processes (Baillie, 2004).

Biocomposite are those natural fibers reinforced with natural polymers (Mngomezulu *et al.*, 2014). Natural fiber are greatly used for applications in composite manufacturing (Faruk *et al.*, 2012). Natural fibers such as plant fibers are used to replace synthetic fibers in composites because of its sustainability. Natural polymer are biodegradable eco-friendly material with low environmental loads, which can be decomposed by microbes (Ohki *et al.*, 2007). There are may be natural, synthetic or semi-synthetic in origin polymer types. They are synthesized by the process called polymerization and if the combining monomers which may be two or more are different then the resultant is called copolymer (Chauhan, 2012). Biodegradable biocomposite are low cost, low relative density, and high specific strength, the interfacial adhesion between fiber and matrix is still an issue (Faruk *et al.*, 2012) which benefits the future.

Microwave absorbers (MA) are absorber materials that have been used for electromagnetic interference isolation and radio frequency absorption. There are many applications of (MA) from as low as 300 MHz up to 300 GHz (Raveendran and Sebastian, 2019), from a simple circuitry up to military application for stealth technology (Edries *et al.*, 2019). In the MA material, two important parameter that determines the absorption performance are the dielectric and magnetic losses. The higher the loss the more electromagnetic signal will be absorbed (Vinoy and Jha, 1996a). In addition to that, the MA design for example its physical thickness or resonator layer also plays an important role in achieving good absorber performance such as unity absorption rates, wide bandwidth, covers all polarizations and all incident angles (Tirkey and Gupta, 2019) which then reflect to the potential application of the MA themselves at the operating frequencies. According to

Khanna, 2020, by having MA design with a the closed ring structure indicates the absorption rates of 10% whilst a ring with dual split, the absorption increases to 90% for K and Ku-band application. As reported by Pandit *et al.*, 2018, more than 80 % of both of TE and TM polarized incident waves is absorbed for a wide incident angle for a single-layer of thickness 3.2 mm microwave absorber for stealth applications

This thesis presents, the microwave absorber based on the newly developed agriculture wastes derived biocomposite with the reinforcement of poly-lactic acid (PLA). The MA material properties that are significant at X band frequency range made from cheap, low density and biodegradable biocomposite that has dielectric properties with high loss tangents. The combination of prospective fibres and PLA show potential wave absorption will give impact to the research of microwave absorber application and environmental issues.

1.2 Problem Statement and Motivation

Due to the materials utilised at microwave frequencies, microwave materials have been widely used in a number of applications ranging from communication devices to military satellite services. The wavelength and velocity of electromagnetic waves vary within a material medium according to their dielectric properties. In recent studies of microwave absorber, there have been many reports in the use of magnetic, carbon based, biomaterials and metamaterial (Raveendran and Sebastian, 2019). Many studies have been drawn to biomaterial or biocomposites because of their ease of disposal and ability to compost after the expiration date, which is not often attainable with typical synthetic materials (Yıldızhan *et al.*, 2018). The use of natural fiber as filler for the biocomposites has also gained a lot of attention for their absorption properties. This includes the use of rice straw, sugar cane bagasse, coconut shell, rubber wood saw dust, rice husk, dried banana leaves, and bamboo-based ash (Raveendran and Sebastian, 2019). Natural fibers such as oil palm fronds (OPF) and empty fruit bunch (EFB) are agricultural wastes in Malaysia as the second largest oil palm plantation area in the world (Nordin, Sulaiman and Hashim, 2017) meanwhile pineapple leaf fiber (PALF) are a waste product of pineapple cultivation (Saravanakumar *et al.*, 2020) where Malaysia being part of 448,193 metric tonnes of the pineapple production (Adam, Yusof and Yahya, 2016). This means higher production in oil palm and pineapple will increase the amount of agricultural wastes and if not properly managed, they may intensify environmental issues. These fibers, on the other hand, have been widely examined in many fields such as biomass for biofuel (Abdul Khalil *et al.*, 2016), in the application of automotive and construction industries (Sudamrao and Patel, 2020), as fabrics for textile materials and in the manufacture of yarns and handicrafts in many countries (Leão *et al.*, 2015) due to their chemical and mechanical advantages which makes them less noticeable in microwave absorber applications.

Several reports show that there are many types of eco-friendly or renewable material binder used as matrix in the biocomposite for absorber application including polyester resin, and methyl ethyl ketone peroxide (Zahid *et al.*, 2013), polyol and isocynate to produce activated carbon loaded polyurethane foam (Se *et al.*, 2011) and multi wall carbon nano tube (MWCNT) (Lee *et al.*, 2016). PLA also one of the renewable material which is commercial bio-based, biodegradable, biocompatible, compostable and non-toxic polymer that has competitive material and processing costs, as well as having desirable mechanical properties (Nofar *et al.*, 2019). PLA have been utilized in many application including commodity and biomedical applications (Tsuji, 2016) and 3D printing application (Pal *et al.*, 2020). PLA has the drawback for low melt strength, slow crystallization rate, poor processability, high brittleness, low toughness, and low service temperature, which limit its applications (Nofar *et al.*, 2019). To overcome this PLA is blended with various natural fibers to improve the chemical and mechanical properties (Rajeshkumar *et al.*, 2021) as well as its dielectric properties. However, very less report found on the dielectric properties for fiber reinforced PLA biocomposites particularly between OPF, EFB and PALF with PLA respectively.

Generally, microwave absorber performance are based on its strong absorption, wide bandwidth, light weight (Li *et al.*, 2018) easy fabrication and low cost (Yah *et al.*, 2019). A good absorption is due to the material used where its depends on their complex permittivity and permeability for the concern frequency range (Yah *et al.*, 2019). For biocomposite material, the dielectric constant, ε' and lost tangent, tan δ are the key features for good absorption where the material with higher tan δ demonstrates higher absorption (Y. S. Lee *et al.*, 2013). In addition to that, high absorption also depends on the minimum reflection and transmission factors (Tian *et al.*, 2020) within the material. Many reports show free space reflectivity measurement (Kharber *et al.*, 2018), and monostatic and bistatic measurements (Masaki *et al.*, 2017) are used for determining the absorption of the MA. It is a non-destructive method where applicable to large samples and they are simple to prepare. This measurement yields reflection results, but it is also necessary to obtain transmission results. As a result, a free space tunnel method (Vinoy and Jha, 1996a) is used to obtain both parameters, reflection and transmission, from the same sample in order to determine absorption. Furthermore, large absorption bandwidth is one of the key features of MA (Tirkey and Gupta, 2019). Obtaining wideband properties is one of the primary difficulties while constructing a microwave absorber (Kumar and Singh, 2016) and to able to achieve that, the MA design tends to be thick and bulkier or multi-layer absorber. The concern with this is that, thick and bulky absorber limits its application and usually impractical (Yah *et al.*, 2019) and furthermore, the it gains in cost in the material and difficult in fabrication.

To reduce the effect on environment, to obtain good microwave absorption and to able practically fabricate a microwave absorber, the agricultural wates such as OPF, EFB and PALF are mixed with PLA to become a biocomposite where the material properties such as permittivity and loss tangent are characterized for microwave absorber application in the X-band region. The material with high loss tangent is determined from the characterized biocomposite using both reflection and transmission parameters factor from the free space measurements and the MA is designed with thin thickness at the same time produces wide absorption bandwidth. These biocomposites have the benefits of being a low-cost, low-density, lightweight, high-loss tangent dielectric material that can be fabricated into a flat, thin, single-layer microwave absorber with high absorption and wide bandwidth. The eco-friendly non-toxic material MA benefits to both microwave absorber application and the environment.

1.3 Research Aim and Objectives

The aim of this this research is to develop the prospective microwave absorber made from biocomposites and there are four objectives in order to accomplish this research work.

1. To characterize dielectric permittivity of natural fiber from oil palm frond, empty fruit bunch, pineapple leaf fiber, natural polymers from polylactic acid and also their biocomposites within X-band frequency range.
2. To analyse the absorption performance of microwave absorbers based on absorption rates, bandwidth, and thickness in relation to permittivity and loss tangent of various biocomposites loadings.
3. To evaluate the performance of the microwave absorber using the fabricated absorber.

1.4 Research Scopes

The scope of this research is the extent of work involved to which it is significant. Figure 1.1 shows the scope of this research. Mainly, it covers the selection of vegetable fiber of natural fiber such as the oil palm frond (OPF), empty fruit bunch (EFB) and pineapple leaf fiber (PALF). Each fiber properties in terms of dielectric constant, ε and loss tangent tan δ are identified using open ended coaxial probe technique. Next, the poli-lactic acid (PLA) which is from the natural polymer where its dielectric constant, ε and loss tangent tan δ are determine using transmission line method. The biocomposite which are the mixture of natural fiber and polymer are prepared into samples so that their properties can be obtained using transmission line method. The biocomposite properties are selected in the X band range to design the single layer microwave absorber (MA) using electromagnetic (EM) simulation. The MA performance are observed in terms of absorption rates, bandwidth, transverse electric (TE) and transverse magnetic (TM) polarization and also the incidents angles. Finally the validation is made upon the performance of the fabricated MA.

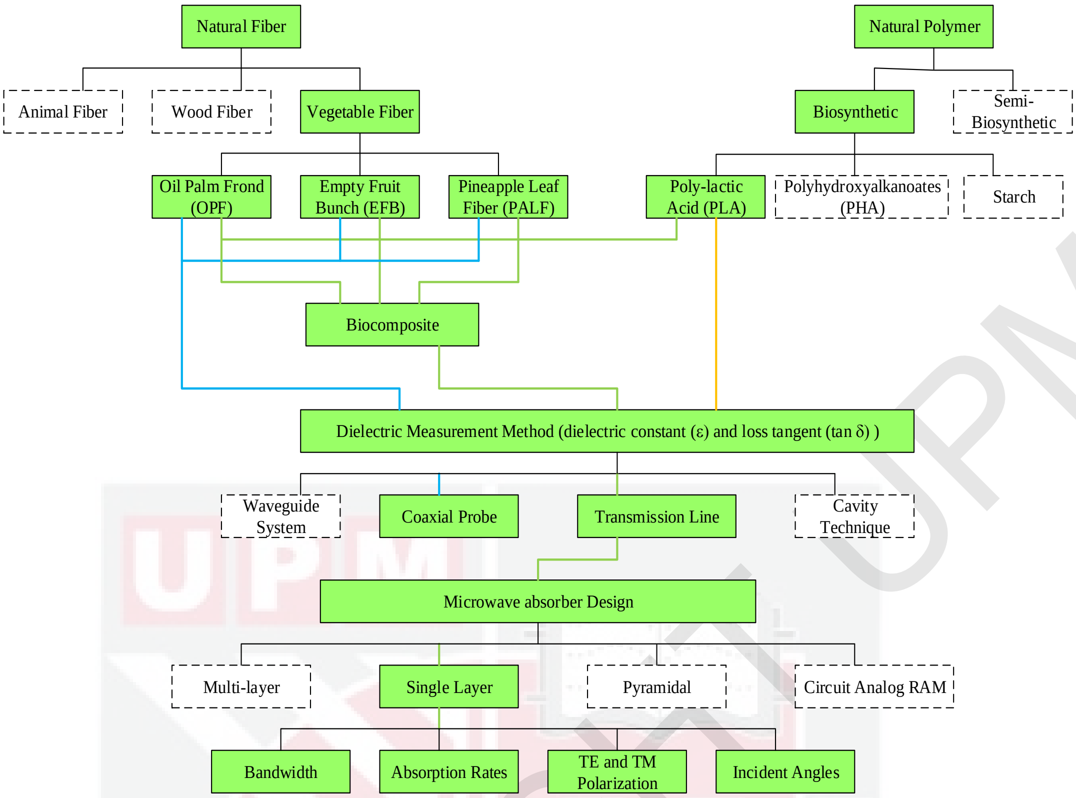

Figure 1.1 : Research Scopes

1.5 Thesis Outline

This thesis is organized into seven chapters as shown in Figure 1.2.

In chapter 1, the introduction on the research is presented. In the beginning of the chapter, it describes of the overview of the research background and the problem statement that motivates this research work. Then, followed by the aim and objectives that are achieved within the scopes and limitations of the research work. The generalized methodology is also presented to give a brief understanding of the research process and finally the chapter ends with the thesis outlines.

Chapter 2 reviews in detail the traditional and future trend of MA including design structure, material properties, reflectivity and transmission factor, absorption and bandwidth. In addition, several existing microwave absorber researches were discussed and together with microwave absorber performance characteristics. Then, the review on the natural fibers and polymer used in this research work which includes the chemical, physical dan mechanical properties. It also provides the foundation of dielectric material measurement techniques and also the MA measurement techniques at the end of the chapter.

Chapter 3 consist of a comprehensive explanation on the research methodology. This includes the process of preparing samples from the natural fiber and polymer and also the biocomposite. The explanations of the dielectric properties measurement to determine the material properties for fiber, PLA and biocomposite. The discussion also elaborates on the process of mixing fiber and matrix and also the fabrication of the optimum MA. The simulation of MA covers from the MA design until the results for the optimum MA. Meanwhile in the measurement setup for the MA validation, explains on the setup process and data collection for the fabricated MA.

Chapter 4 is mainly about the results of dielectric properties for natural fibers and polymer using open ended coaxial probe by highlighting the best material properties. Then, the dielectric properties results for the biocomposites using S-parameter reflection method according to different percentage loading and again the optimum properties is highlighted. Analysis and discussion on the results are also included as well.

Chapter 5 consist of the development of microwave absorber. Starting form the process of designing and optimizing the microwave absorber using CST Microwave Studio up determining the optimum MA for with the best material properties with respect to absorber performance I term of absorption rates, bandwidth and thickness. Then, explanation continues for the further simulation for the best MA in terms of TE and TM polarization and incident angles.

In Chapter 6, the analysis of microwave absorber performance is presented. It consist of the process of microwave absorber measurements, comparisons, results and discussions. In addition, the results of developed microwave absorber are supported by fine-tuned simulation result for error analysis.

This thesis ends with Chapter 7 that consist of the final conclusion of the research work. It also discussed the achievement made according to the research objectives. A few suggestions and recommendations are proposed to enhance the studied work mainly and enlarged the area of research generally.

Figure 1.2 : Thesis Outline

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