UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF ZINC OXIDE POLYCAPROLACTONE NANOCOMPOSITES USING RECTANGULAR WAVEGUIDE AND MICROSTRIP TECHNIQUES

ABUBAKAR YAKUBU

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By

ABUBAKAR YAKUBU

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

October 2015

DEDICATION

Allahu Akbar, Allahu Akbar, Walillahi hamd

In loving memory of my late Son, Father and Grandfather. I pray Allah Subahana-Wata-Allah (S.W.T) grant them mercy and a place in Aljannatu – Firdausi, Amen.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the Degree of Doctor of Philosophy

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October 2015

Chairperson : Zulkifly Abbas, PhD
Faculty : Science

A growing number of demanding applications in high frequency electronics and telecommunications depends on the absorbing properties of materials. The electromagnetic properties of microwave absorbers and radar-absorbing materials are critical issues that need to be resolved in many military applications dealing with reduction of radar signature of aircraft and ships. For industrial equipment and home appliance applications, the Electromagnetic Compatibility Compliance Directive (ECCD), demands electromagnetic interference side effects be eliminated or marginally minimised. The equipment must not disturb radio and telecommunication as well as other appliances. Additionally, the ECCD also governs the immunity of such equipment to interference and seeks to ensure that this equipment is not disturbed by radio emissions when used as intended. Many type of absorbing materials are commercially available. However, many are expensive and not environmentally friendly.

This thesis describes the synthesis and characterization of zinc oxide (ZnO) nanoparticles and ZnO-PCL nanocomposites using the microwave irradiation and melt blend techniques respectively. Two types of pellets with dimension of 6.0 cm X 3.6 cm and 0.11 cm X 0.22 cm were prepared for the measurement of complex permittivity of the different % of the composites using open ended coaxial probe (OEC), while the latter dimension were used in a rectangular waveguide (RWG) in measuring both the permittivity and permeability of the different % composites.

Comparison of permittivity between OEC and RWG results were carried out for all materials used in this study (PTFE, PCL and composites with different percentages of ZnO nanofillers). The effect of the different % ZnO nanofiller on the permittivity of the composites were also investigated. Attenuation, power loss and absorption due to sample thickness and ZnO nanofiller inclusion in the composites were investigated using finite element method (FEM) and RWG methods, whilst transmission and reflection coefficient were measured, simulated and calculated using RWG, FEM, and Nicholson Ross Weir (NRW) methods respectively.

Microstrip and FEM techniques were used to determine both the transmission and reflection coefficients and electric field distribution for the different % ZnO-PCL nanocomposites pellets when placed on top a microstrip. Comparison of the measured and calculated scattering parameters were also investigated. Furthermore, the results obtained from the scattering parameters were used to determine the attenuation of the different % of ZnO-PCL nanocomposites pellets. Finally, the effect of the different % ZnO nanofiller on electric field was investigated by visualizing the electric field distribution of the ZnO-PCL nanocomposites pellets placed on top a microstrip using finite element method. Findings from investigations showed that the complex permittivity values obtained using the OEC method were in good agreement with the RWG technique, whilst increase of ZnO nanofiller percentage into the polymer matrix increased the dielectric constant, loss factor, attenuation, absorption, real permeability, imaginary permeability and reflection coefficient of the composites. The attenuation obtained for the 70 % filler composition was -18 dB which is good for microwave absorption whilst the microwave irradiation technique was able to synthesize ZnO nanoparticles with an average particle size of 57.5 nm.

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

SINTESIS DAN PENENTUAN SIFAT-SIFAT ZINK OKSIDA POLYCAPROLACTONE NANOKOMPOSIT DENGAN MENGGUNAKAN TEKNIK PANDU GELOMBANG SEGI EMPAT TEPAT DAN MIKROSTRIP

Oleh

ABUBAKAR YAKUBU

Oktober 2015

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Semakin banyak aplikasi dalam elektronik frekuensi tinggi dan telekomunikasi yang dikehendaki__bergantung kepada sifat-penyerapan bahan. Sifat elektromagnet penyerap gelombang mikro dan bahan penyerap radar adalah isu kritikal yang perlu diselesaikan dalam banyak aplikasi ketenteraan berurusan dengan pengurangan isyarat radar pesawat dan kapal. Untuk aplikasi peralatan industri dan perkakas rumah, Arahan Pematuhan Keserasian Elektromagnet (ECCD), menuntut agar kesan sampingan gangguan elektromagnet dihapuskan atau dikurangkan sedikit. Alat ini tidak boleh mengganggu radio dan telekomunikasi serta peralatan lain. Tambahan pula ECCD juga mengawal ketahanan peralatan untuk turut serta dan berusaha untuk memastikan bahawa alat ini tidak diganggu oleh pancaran radio apabila digunakan seperti yang sepatutnya. Banyak jenis bahan penyerapan boleh didapati secara komersial. Walau bagaimanapun, kebanyakannya adalah mahal dan tidak mesra alam.

Tesis ini menerangkan sintesis dan pencirian zink oksida (ZnO) zarah nano menggunakan penyinaran gelombang mikro dan ZnO-PCL nanokomposit menggunakan teknik cair dan gabung. Dua jenis pelet dengan dimensi 6.0 cm X 3.6 cm dan 0.11 cm X 0.22 cm disediakan untuk mengukur ketelusan kompleks yang berbeza peratusan komposit dengan menggunakan prob sepaksi hujung terbuka (OEC), manakala dimensi yang kedua digunakan dalam pandu gelombang segi empat tepat (RWG) untuk mengukur ketelusan dan kebolehtelapan yang mempunyai peratusan komposit yang berbeza.

Perbandingan ketelusan antara keputusan OEC dan RWG telah dijalankan bagi semua bahan-bahan yang digunakan dalam kajian ini (PTFE, PCL dan komposit dengan peratusan yang berbeza ZnO nanopengisi). Kesan perbezaan peratusan ZnO nanopengisi ke atas ketelusan bagi komposit juga disiasat. Pengecilan, kehilangan kuasa dan penyerapan kerana ketebalan sampel dan ZnO nanopengisi dimasukkan dalam komposit telah dikaji menggunakan kaedah unsur terhingga (FEM) dan kaedah RWG, manakala pekali penghantaran dan pantulan diukur, disimulasi dan dikira menggunakan kaedah RWG, FEM, dan Nicholson Ross Weir (NRW).

Teknik mikrostrip dan FEM telah digunakan untuk menentukan pekali pantulan dan penghantaran dan taburan medan elektrik untuk peratusan ZnO-PCL nanokomposit pelet yang berbeza apabila diletakkan di atas mikrostrip. Perbandingan parameter penyerakan yang diukur dan dikira juga dikaji. Tambahan pula, keputusan yang diperolehi daripada parameter penyerakan telah digunakan untuk menentukan pengecilan peratusan ZnO-PCL nanokomposit pelet yang berbeza. Akhir sekali, kesan peratusan ZnO nanopengisi yang berbeza pada medan elektrik telah dikaji dengan menggambarkan corak taburan medan elektrik pada ZnO-PCL nanokomposit pelet yang diletakkan di atas mikrostrip dengan menggunakan kaedah unsur terhinnga. Penemuan daripada penyiasatan menunjukkan nilai-nilai ketelusan kompleks diperolehi dengan menggunakan kaedah OEC yang diperolehi bersetuju dengan teknik RWG, manakala kenaikan dalam peratusan ZnO nanaopengisi dalam matriks polimer meningkatkan pemalar dielektrik, faktor kehilangan, pengecilan, penyerapan, kebolehtelapan nyata, kebolehtelapan bayangan dan pekali pantulan komposit tersebut. Pengecilan yang diperoleh bagi 70% komposit nanopengisi iaitu -18 dB adalah bagus untuk penyerapan mikrogelombang manakala teknik penyinaran mikrogelombang mampu mensintesis zarah nano ZnO dengan saiz purata sebesar 57.5 nm.

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With absolute submission to the will of Allah (SWT), I want to thank the most gracious, and most merciful for the uncountable blessings and guidance throughout this period.

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I wish to further express my warmest salutations and heart felt gratitude to our research group members who have toiled day and night, through thick and thin, to excel in our educational struggle thus far. You were all awesome! May I also use this opportunity to express my gratitude to staff and postgraduate students of Institute of Advanced Technology (ITMA), Universiti Putra Malaysia, for their candid support, I love you all.

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Finally, I thank my wife, daughters, (Anisa, Amrah, and Aisha) brothers, sisters and my sweet happy mother for their continuing prayer, love and support throughout this period.

Thank you very much.

Assalamu - alaikum.

I certify that a Thesis Examination Committee has met on the 12/10/2015 to conduct the final examination of Abubakar Yakubu on his thesis entitled "Synthesis and Characterisation of *Zinc-Oxide Polycaprolactone* Nanocomposites using Rectangular Waveguide and Microstrip Techniques" 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.

Members of the Thesis Examination Committee were as follows;

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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. Members of the Supervisory committee were as follows;

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Signature: ______________________________
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LIST OF ABBREVIATIONS

EMI	Electromagnetic interference
PCL	Poly-caprolactone
ZnO	Zinc-Oxide
EM	Electromagnetic
CNT	Carbon nanotube
OEC	Open ended coaxial probe
RWG	Rectangular waveguide
EF	Electric field
HE	Magnetic field
CVD	Chemical vapour deposition
XRD	X-ray diffraction
AFM	Atomic force microscope
SEM	Scanning electron microscope
EDX	Energy dispersive X-ray
TEM	Transmission electron microscope
FTIR	Fourier transform infrared spectroscopy
SEI	Secondary electron imaging
BEI	Back scattered electron imaging
FEM	Finite element method
NRW	Nicholson Ross Weir
FDM	Finite difference method
MOM	Method of moment
FDTD	Finite difference time domain
MWO	Microwave office

HFSS	High frequency structural simulation
CEM	Computational electromagnetic
PNA-L	Professional network analyser
VNA	Vector network analyser
TRL	Thru, Reflect, Line
RF	Radio frequency
FWHM	Full wave half maximum
MUT	Material under test
ECAL	Electronic calibration
SMA	Sub-miniature
TE_{10}	Transvers electric mode
TM	Transverse magnetic mode
TEM	Transverse electromagnetic mode
TR	Transmission/Reflection
S_{11}	Reflection coefficient
S_{21}	Transmission coefficient
ε_r	Complex permittivity
μ_r	Complex permeability
PTFE	Polytetrafluoroethylene (Teflon)
EDTA	Ethylenediaminetetra-acetic acid
DIW	De-ionized water
JCPDS	Joint committee on powder diffraction standard
ECCD	Electromagnetic Compatibility Compliance Directive
MTI	Maximum transmitted intensity

CHAPTER 1

INTRODUCTION

The need for microwave absorbers and radar-absorbing materials is on the rise in military applications dealing with reduction of radar signature of aircraft and ships, whilst in civilian applications dealing with reduction of electromagnetic interference among electronic and telecommunication components. Nanocomposite absorber that uses zinc oxide nanoparticles in conjunction with a polymer matrix produces flexibility for fabrication and properties control, as the composite can be manipulated through changes in both the nanoparticle filler and the host matrix. Depending on the application for which the absorber is intended, the percentage of filler and the host 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. The suppression of eddy current due to electromagnetic interference are enhanced by sizes of particles in the absorber material. In this regard, metal type nanocomposites were widely used for EM wave absorption. Magnetic particles encapsulated in carbon nanotube (CNT) composites and magnetic particles coated with carbon have been the focus for EM wave absorbers (Tang et al, 2014; Wen et al, 2011). However, the process involved in the fabrication of magnetic particles doped (CNT) is unfavourable for the application of absorbing nanocomposites. This condition has led to the push in looking for new absorbing nanocomposites materials. This search has led scientists to ZnO nanoparticles which can be used as high efficiency microwave absorbing materials due to its high complex permittivity and complex permeability (Tan et al, 2014; Cao, et al, 2007).

Shown in Figure 1.1 and 1.2 are areas in telecommunication where zinc-oxide -polycaprolactone (ZnO-PCL) nanocomposites can be applied in telecommunication as absorbing material (Wahab, et al, 2013; Liu, et al, 2008), with worldwide consumption of ZnO in a wide range of applications, ranging from tyres to ceramics, from pharmaceuticals to agriculture, and from chemicals to electronics (Kołodziejczak-Radzimska & Jesionowski, 2014).

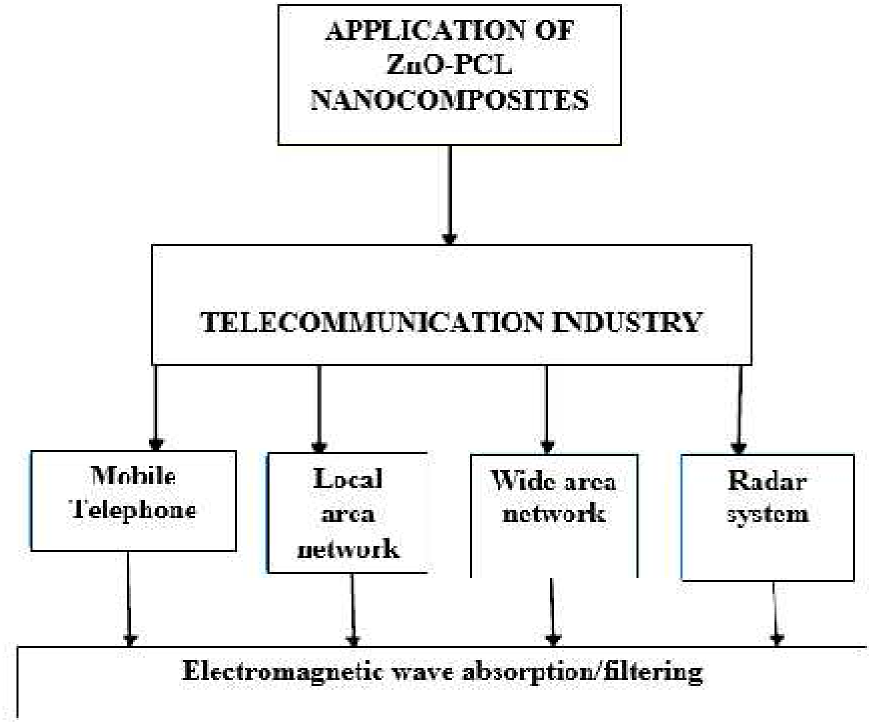

Figure 1.1: Schematic representation of application of ZnO-PCL nanocomposites

The advantages of using ZnO-PCL nanocomposites is the ease in realizing large scale synthesis of ZnO nanoparticles which is cost effective. Due to ZnO unique geometrical morphology, cage like ZnO/SiO_2 nanocomposites exhibited a strong attenuation of microwave at X band frequency (Cao et al, 2007).

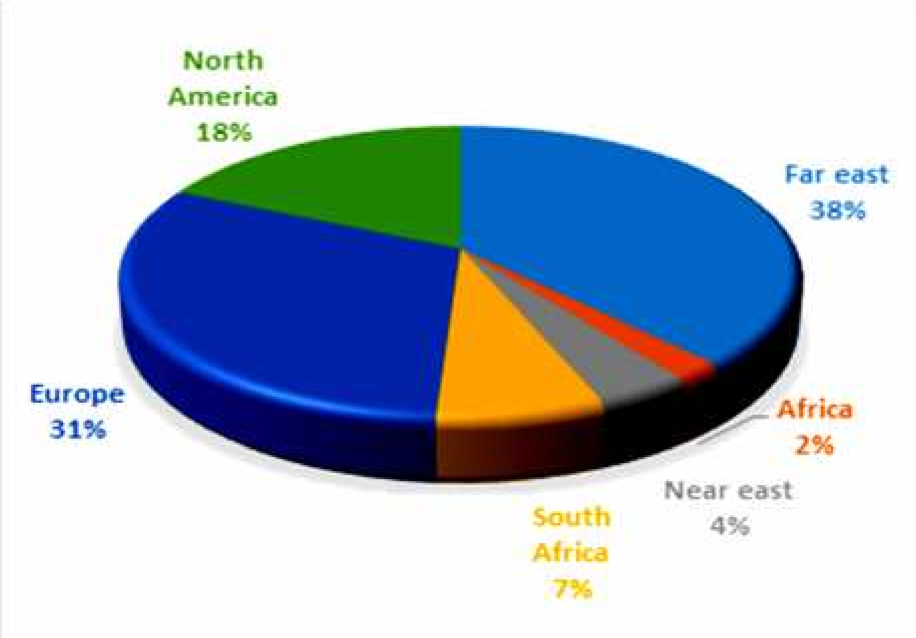

Figure 1.2: Worldwide consumption of ZnO

Su et al, (2014), reported that ZnO nanowire polyester composites are strong absorption materials for microwave at X band frequency which is due to its interfacial multi-polarization at the interface between the polyester and the ZnO nanowires with a high surface to volume ratio. The anisotropic energy of nano sized particles might be increased due to the surface anisotropic field affected by very small size effect. This phenomenon causes a shift in the resonance peak to higher frequency value which is important for EM wave absorption at higher frequency.

Detailed studies in measurement of permittivity of solid materials using open ended coaxial probe (OEC) has not been carried out. In the light of the above, measurement of complex permittivity using OEC technique will be investigated and the results will be compared with standard recommended technique like the rectangular waveguide method (RWG). Other investigations will involve the effect of different (%) ZnO nanofiller inclusion in the host matrix on materials complex permittivity, complex permeability, scattering parameters, absorption, attenuation, power-loss, and electric field distribution. Further understanding in the applications of dielectric materials can be found in (Pozar, 2009; Pozar, 2012; Laverghetta, 2005).

1.1 Nanocomposites

The term nanocomposites materials could be explained in different ways depending on the perspective with which it is viewed. Simply put, nanocomposites are compounds which encompasses two or more unlike components mixed together at a nano-meter scale. The composites materials could either be organic or inorganic in nature. In the inorganic state, the composite could be three dimensional, two dimensional, one dimensional and even zero dimensional. In nanocomposites there is a tendency of mixing different properties together that are so far impossible within a single material (Zhao, et al, 2008). Within the class of nanocomposites, the polymeric nanocomposites have a promising future because of its high performance properties. The four types of polymeric nanocomposites are the clay-polymer nanocomposites, the metal-polymer nanocomposites, the oxides-polymer nanocomposites, and the carbon nanotubes nanocomposites. The two main methods employed in obtaining oxide-polymer nanocomposites are the ex-situ synthesis and in-situ synthesis method. The changes in properties of nanocomposites are mainly caused by phenomena such as size confinement, predominance of interfacial phenomena and quantum mechanisms (Liang, 2007). The dependence of bulk properties of nanocomposites is mainly due to (Kochetov, 2012);

- ✓ properties of the filler
- ✓ filler size,
- ✓ filler type,
- ✓ host matrix:
- ✓ crystallinity,
- ✓ nature (thermoplastic or thermosetting),
- ✓ degree of dispersion and of agglomeration,
- ✓ Synthesis methods.

1.2 Properties of Polymer Nanocomposite

The dielectric properties of polymer composite are mainly controlled by the conductive fillers. Consequently, the nature or type of fillers determines the dielectric characterisation of polymer composites. Examples of conductive fillers are semiconductors, metals, carbonic materials and intrinsic conductive polymers (Saini and Arora, 2012; Xu et al, 1999). Polymer that are conductive have attracted a lot of interest in the recent due to their excellent flexibility and easy preparation procedures as against conventional inorganic semiconductors. They are applied in areas of electronics as flexibility conductors and shielding devices especially with regards to electromagnetic radiation (Saini and Arora, 2012; 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 that large filler contents lead to a poor composite (He and Tjong, 2014; Liang, 2007). The use of nano sized reinforced polymers has led to the production of nanocomposites with unique dielectric and mechanical properties. Nanofiller material comes in different forms, these forms could be in metals, semiconducting oxides, dielectric ceramics and carbon materials (Liang, 2007).

1.2.1 Polymer-Semiconductor Nanocomposites

Nanofillers with unique chemical, physical and mechanical properties that are intermixed with polymers comprises of composite materials with great advantage for technological breakthrough. Conventional metal oxides such as barium titanate ($BaTiO_3$), titania (TiO_2), alumina (Al_2O_3) and silica (SiO_2) are widely known as effective reinforcement materials to enhance the dielectric and mechanical properties of polymers. In the last decade, semiconducting oxides (ZnO, NiO, and MgO) have attracted much interest due to their potential for diverse electronic and photonic device applications (Murugadoss, 2012; Heo et al, 2004).

Recent research has demonstrated that the polymer-oxides nanocomposites exhibit excellent luminescent, optical, dielectric and bio-sensitivity properties. Studies have been conducted on the electrical properties of polymer-oxides nanocomposites prepared by in-situ polymerization and melt blending (Milani et al, 2013; Tripathi et al, 2013).

Hong, et al (2003), investigated the electrical properties of low density polyethylene-ZnO composites prepared by melt compounding. They reported that the nanocomposites exhibited a lower percolation limit and a slower decrease in resistivity with filler content when compared to conventional micro-composites. The dielectric breakdown strength was also found to be higher for the nanocomposites for all filler concentration.

Kango et al, (2013) reported that addition of inorganic nanoparticles into a polymer host material will definitely change the properties of the host matrix. They added that the resulting composite might show enhanced mechanical, thermal, electrical, and optical properties. They concluded that properties of polymer composites largely depend on type of nano filler, sizes and shape, concentration and their interactions with the polymer matrix.

1.2.2 Polymer-Metal Nanocomposites

Metals exhibit the most excellent electrical conductance among materials known. Metal nanoparticles, e.g. Ag, Cu, Al, Fe and Ni have been introduced into polymers to enhance the electrical, mechanical and dielectric properties of composite materials. Electrical conductivity of polymer nanocomposite can be greatly enhanced by metal nanoparticles at very low loading levels as a result of large surface area. Gonon and Boudefel (2006), investigated the electrical behavior of nanocomposites made of epoxy resin and Ag nanoparticles with particle size of 70 nm. Their result showed a very low percolation threshold as a result of filler segregation in the epoxy matrix.

Nanocomposites filled with metal nanoparticles can be synthesized via in-situ, polymerization and ex-situ processing routes. In-situ, vapor deposition polymerization of monomers with organometallic compounds and metal-monomer co-condensates has been reported (Kharissova et al, 2013; Sakai and Alexandridis, 2006). The method involves a gradual layer-by-layer deposition of metal and monomer vapors on substrate plates at a low temperature of about 77 K. The layer to layer deposition produces composites with very low concentration of about 0.01-1.0 wt % of metal particles in sub-micrometer dimension (Nicolais and Carotenuto, 2005). Current advances in the preparation of metal nanoparticles via chemical vapor deposition (CVD), laser induced gas phase and spray conversion procedure techniques have changed the way researchers understand polymer reinforcement (Bahlawane et al, 2012; Schubert and Husing, 2012). Ex situ polymerization of nanocomposite is the direct combination of nanoparticles into polymers via melt-compounding method.

1.3 Characterization Techniques

When discussing propagation of waves, the characteristics of microwaves and light waves are typically the same since both waves travel in a straight line. The characteristics of travelling in a straight line enable them reflect, refract, diffract, scatter, and interfere at boundary points with interacting media. 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).

1.3.1 Permittivity and Permeability

Measurements of complex permittivity and complex permeability are required not only for scientific but also for industrial applications. Example of areas in which knowledge of the properties of materials at microwave frequencies are microwave heating, biological effects of microwaves, and nondestructive 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 and permeability are complex numbers of which the imaginary part is associated with losses.

Scattering parameter, permittivity, permeability 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, free space measurement technique, open ended coaxial probe technique, and resonant method (Agilent Tech, 2011). Details of these techniques would be discussed in the ensuing chapters.

1.3.2 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 striking a sample as a function of incident and scattered angle, polarization, and wavelength or energy.

Atomic force microscope (AFM) studies can be divided into topographical applications and force curves in which forces are measured as a function of distance. Topographical applications involve getting an image of the sample surface to observe its structural or dynamic features. The method has been applied to a different types of surfaces including semiconductors, biological systems, nanostructures and polymers with imaging reaching the nanometer range and the atomic scale in some cases. For the force curves approach, the study allows the understanding of inter and intramolecular forces, and manipulate samples following dissection, dragging and cut. The method has also been used to study polymers systems and interfacial phenomena in various systems (Leite et al, 2007).

The Scanning electron microscope (SEM) is the most widely used analytical tools due to the detailed images it provides within a short time. It provides high resolution and thick depth of images of samples surface and near surfaces with wide magnification range. Application of SEM includes failure analysis, dimensional analysis, process characterization etc. SEM comprises of other sub function like the secondary electron imaging (SEI) signal, the backscattered electron imaging (BEI) signal. These signals provide "near surface" interpretation of sample morphology and information regarding sample composition, density and surface geometry respectively. To determine the elements and compounds of the sample, Energy Dispersive X-ray Analysis (EDX) is applied.

In Transmission Electron Microscopy (TEM) focused beam of electrons is used instead of light to see through the sample. TEM is a type of electron microscopy developed and programmed on light transmission microscopy. TEM is used to ascertain the followings (Ismayadi et al, 2009);

- ✓ The size, shape and arrangement of the particles which make up the sample as well as their relationship to each other on the scale of atomic diameters.

- ✓ The arrangement of atoms in the sample and their degree of order, detection of atomic-scale defects in areas with a few nanometers in diameter.

- ✓ The elements and compounds of the sample are composed of their relative ratios, in areas that a few nanometers in diameter exist.

Fourier Transform Infrared Spectroscopy (FTIR) analyses, and uses spectrum of molecular vibration in sample in order to identify or characterize organic materials such as polymers, lubricants, adhesives and cleaning agents. For semiconductor, FTIR is used to make quantitative measurement of hydrogen bonds and to measure the interstitial oxygen content in bulk.

1.4 Problem Statement and Hypothesis

The addition of ZnO nanoparticles into the polycaprolactone matrix is expected to enhance the dielectric properties, attenuation, absorption, and power-loss as well as decrease the prepared composites transmission coefficient making it a better microwave absorbing material. ZnO-PCL nanocomposites have been used extensively in many microwave applications. However, its potential has not been exploited fully due to lack of detailed information on the relationship between the filler composition and electromagnetic properties. The dielectric properties, transmission and reflection coefficients of ZnO-PCL nanocomposites of various filler content and types, host matrix and material properties were not analyzed in detailed both theoretically and experimentally. The conventional method to determine the complex permittivity of the ZnO-PCL nanocomposites materials is to place the sample in a closed waveguide. The technique is difficult as the sample must be inserted tightly into the waveguide without any air gaps. In this work, both the open ended coaxial probe (OEC) and waveguide techniques were investigated.

Dielectric measurement in a waveguide is usually calculated using the Nicholson Ross analysis. However, the technique does not offer an insight on the electromagnetic field distribution in the sample. Additionally, Nicholson Ross Weir method was originally designed for thick samples where the effect of multiple reflection is assumed to be negligible. In this work, the Finite Element Method (FEM) was used to discretize the sample into small meshes allowing accurate calculation of the scattering parameters and eventual visualization of the electromagnetic fields.

The microwave attenuation due to sample does not only depend on the complex permittivity but also the sample thickness. Thick sample measurements are always problematic when using waveguide technique due to air gap problems. In this work, the attenuation of the samples was also analyzed using the microstrip technique by placing a 6cm long ZnO-PCL nanocomposites on top of the open microstrip. Visualization of the effect of nanocomposites on the microstrip overlays is also carried out using FEM.

1.5 Specific Objectives

The objectives of this study are enumerated below;

- To synthesize ZnO nano particle and ZnO-PCL nano-composites using microwave irradiation and melt blend method so as to study the effect of ZnO nanofillers on the complex permittivity and permeability of ZnO-PCL nanocomposites.
- To measure the dielectric constant and loss factor of ZnO-PCL nanocomposites using open ended coaxial probe and rectangular waveguide techniques. The latter technique is also used to measure the permeability of the nanocomposites.
- To study the effect ZnO-PCL nanocomposites thickness on scattering parameters, absorption, power loss using rectangular waveguide technique. The scattering parameters results are compared theoretically using Finite element method (FEM) and Nicholson Ross Weir (NRW) methods.
- To determine the attenuation of ZnO-PCL nanocomposites pellets using rectangular waveguide and microstrip methods and compare with calculated FEM results.
- To study the effect of ZnO nanofillers on both transmission and reflection coefficients of ZnO-PCL nanocomposites using microstrip technique and FEM and to visualize their electric field distribution using FEM.

1.6 Scope and Relevance of Study

In this study, an easy and lesser time consuming technique for preparing ZnO nanoparticle and ZnO-PCL nanocomposites using the microwave irradiation and melt blending technique via Thermo Haake melt blending machine were carried out.

The effect of the different % ZnO nanofiller on the dielectric properties were measured using open ended coaxial probe and rectangular waveguide techniques. The effect of ZnO nanofiller on the transmission and reflection coefficient of the ZnO-PCL nanocomposite pellets were also studied. It also proposes to use FEM COMSOL software in calculating scattering parameters and for simulating electromagnetic wave excited through ZnO-PCL nanocomposites samples when placed inside a rectangular wave guide and on top a microstrip. The result obtained for scattering parameter through measurement, simulation and calculation were also compared. Error analysis for the comparison is determined for both FEM and NRW techniques. The visualization of electric field of ZnO-PCL nanocomposites when placed on top a microstrip is pioneered in this study using finite element method. The micro-structural characteristics of materials with respect to sample size, bonding, surface roughness and filler dispersion were also studied for the pure PCL, prepared ZnO nanoparticles and ZnO-PCL nanocomposites.

1.7 Thesis layout

There are five chapters in this thesis with appendices attached at the end of the chapters. Chapter 1 briefly outlines generally on polymer nanocomposites, morphological and dielectric characterization, problem statements, and objectives of study, the scope of the study and finally, the thesis layout.

Chapter 2 presents reviews on ZnO-polymer nanocomposites, electromagnetic radiation (EM) measurement technique and limitations of some measurement techniques. Numerical methods associated with rectangular waveguide were also discussed.

In chapter 3, theories used in the research work are briefly outlined. Bragg's law, Maxwell equations, wave equation and FEM theory were all discussed. FEM formulation techniques on transmission and reflection coefficients calculation was also discussed.

Chapter 4 encompasses the entire method used in this study. The preparation of ZnO nanoparticle and ZnO-PCL nanocomposites were explicitly discussed. The use of FEM, PNA-L, NRW, OEC, RWG and microstrip methods are fully discussed in relation to microwave characterization. The morphological characterization using components like the XRD, TEM, SEM, EDX, AFM and FTIR were all discussed in details.

Chapter 5 is divided into five subsections. Section 5.1 deals with the morphology and characterization of all the samples used in this work. Section 5.2 deals with the dielectric characterization of the all the samples used in this research work using the open ended coaxial probe and rectangular wave guide methods. The effect of the ZnO nano inclusion on the permittivity of the composites was also investigated. Comparison between dielectric constant obtained using the two methods are shown. Section 5.3 details on the effect of sample thickness on the scattering parameters using rectangular waveguide and FEM. Absorption of the electromagnetic waves based on the scattering parameters was also discussed. Finally, the scattering parameters obtained from theory, calculation and measurement were compared.

Section 5.4 deals with the attenuation of the ZnO-PCL nanocomposites pellets using rectangular waveguide and microstrip methods. FEM was used to calculate the field intensity of samples when placed inside a rectangular waveguide and simulation of their field distribution. Finally, the attenuation from both methods were compared with their respective FEM calculated attenuations.

Section 5.5 deals with the measurement of scattering-parameters of ZnO-PCL nanocomposites pellets placed on top a microstrip. Comparisons between measured and calculated S_{11} and S_{21} magnitudes using microstrip and FEM were presented.

Visualization of electric field of the different percentages of the ZnO-PCL nanocomposites were simulated using Finite Element Method.

Finally, chapter 6 will draw conclusions based on findings and give suggestions for future studies.

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