



UNIVERSITI PUTRA MALAYSIA

**CHARACTERISATION OF LOSSY LOW DIELECTRIC CONSTANT SODA
LIME SILICA-HIGH DENSITY POLYETHYLENE COMPOSITES FOR
MICROWAVE APPLICATION**

ABUBAKAR DANTANI MELI

FS 2019 4



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MICROWAVE APPLICATION**

By

ABUBAKAR DANTANI MELI

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

March 2019

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DEDICATION

I dedicate this work to my late parents, May Allah have mercy on them, and to my wife and children who have sacrifice their comfort to eneble me pursuit my studies, and also to entire family members.



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

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March 2019

Chairman : Associate Professor Zulkifly Abbas, PhD
Faculty : Science

Composites material of low dielectric constant and high loss factor for microwave absorbing application are much needed. Composites materials of low dielectric constant and high loss factor, reflect electromagnetic waves less and absorb more. Composites of these characteristics can be use for security protection and to curb the menace of electromagnetic interference (EMI) pollution arising from the increasing number of the telecommunication users through mobile telephones, local area networks, wide area networks and radars systems. Generally microwave absorbing composites comprises of filler material in a host matrix. These fillers comprises of an element or more that carry out most of the absorption. Absorbing materials are applied in range of applications to remove the unrequired radiation that may affect an operation system.

This research presents characterisation of lossy low dielectric constant soda lime silica-high density polyethylene (SLS-HDPE) composites for microwave absorbing application. The total mass of each blended composite was 25 g and contained 63 μm size SLS glass. The Brabender Plastograph EC blending machine was used to blend the composites at a speed rotor of 50 rpm for 10 minutes. Brabender was set at 170 °C of heating temperature which was chosen to restrict the flow of matrix HDPE whose melting point is 160 °C. SLS glass was chosen for this study because it is a solid waste output from milling process which is broadly and readily available. SLS glass shows a better mechanical properties in comparison to other glass. SLS glass and HDPE are biodegradable and both have low density unlike many other manufacturing materials. Different percentage of filler were used and blended to produce SLS-HDPE composites to give different absorbing properties in the HDPE matrix. The structural

crystallinity of the composites was characterised via X-ray diffraction (XRD) machine.

Theoretical calculations of the reflection and transmission coefficients of placed samples inside a rectangular waveguide and the placed samples on top of an open microstrip were simulated by using the Finite Element Method (FEM) in connection to COMSOL software. The measurement of the reflection and transmission coefficients, and also the dielectric properties, was carried out using the PNA (N5227) Network Analyzer for both rectangular waveguide and microstrip under room temperature for frequencies ranges 8 GHz to 12 GHz. Investigative analysis between the calculated and measured scattering parameters was also carried out. The permittivity complex of the composites was found to be mixing ratio dependent between SLS-HDPE. At X-band frequency 10 GHz, the real permittivity of SLS-HDPE composites which is the dielectric constant was found to be between 2.44 to 2.91 respectively, while the imaginary permittivity which is the loss factor values were found to be from 0.05 to 0.17. The dielectric constant and loss factor of the composites can be predicted using the regression equations by putting the fractional values of the fillers composition. Both the dielectric constant and dielectric loss of the SLS-HDPE composites increased with increased in SLS fillers percentages. Thus, these, could result to obtain lower transmission coefficient $|S_{21}|$ and higher values of the magnitude of the reflection coefficient $|S_{11}|$ by the impedance matching theory. However, the obtained results from the scattering parameters was further used to determine the absorption loss of different SLS-HDPE percentages of samples composites. Lastly, investigation of the effect of different SLS filler percentages on the electric field was carried out by observing the pattern of electric field distribution of the samples of SLS-HDPE composites placed in a rectangular waveguide technique. Electric field distribution via FEM showed that the higher the SLS fillers, the lower the transmittance of the field distribution across the sample from the input port to the output port of the rectangular waveguide. Observation showed a reduction in field strength as SLS fillers increases confirming the results obtained for the complex permittivity of the composites are $\epsilon' = 2.44$ for 10%SLS, 2.55 for 20%SLS, 2.65 for 30%SLS, 2.83 for 40%SLS and 2.91 for 50%SLS respectively while the loss factor values are $\epsilon'' = 0.05$ for 10%SLS, 0.08 for 20%SLS, 0.10 for 30%SLS, 0.15 for 40%SLS and 0.17 for 50%SLS where the highest permittivity measured $\epsilon' = 2.91$ and $\epsilon'' = 0.17$ were for the fillers of 50% SLS composites further addition of SLS filler will result to the flexibility and increase the absorption which can generate more heat in the composites and eventually the composites will breakup. The 50%SLS composites shows better thermal and electrical properties, higher dielectric constant, higher loss factor and higher loss tangent. The 50%SLS composites has the highest absorbing properties, hence it is the best for microwave absorbing application.

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

**PENGLASIFIKASIAN KEHILANGAN DIELEKTRIK KONSTAN
RENDAH OLEH SODA KAPUR SILIKA-KOMPOSIT POLIETELINA
BERKEPADATAN TINGGI UNTUK APLIKASI GELOMBANG MIKRO**

Oleh

ABUBAKAR DANTANI MELI

Mac 2019

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Bahan komposit dengan penyerap gelombang mikro yang mempunyai ciri-ciri seperti pemalar dielektrik yang rendah dan kehilangan yang tinggi amatlah diperlukan untuk aplikasi penyerapan. Bahan komposit dengan penyerap gelombang mikro yang mempunyai pemalar dielektrik yang rendah dan faktor kehilangan yang tinggi menyerap lebih banyak gelombang elektromagnetik daripada memantulkannya. Penghasilan komposit yang mempunyai sifat sebegini boleh digunakan sebagai perlindungan keselamatan dan juga boleh diguna pakai untuk mengawal ancaman dari pencemaran interferens elektromagnetik (EI) yang muncul terhasil daripada penambahan bilangan penggunaan alat telekomunikasi melalui telefon bimbit, rangkaian kawasan tempatan, rangkaian kawasan luas dan sistem radar. Penyerap gelombang mikro secara amnya merangkumi sesuatu bahan pengisi di dalam matrik polimer. Pengisian tersebut mengandungi satu atau lebih elemen yang berfungsi untuk melakukan penyerapan tersebut. Penyerap digunakan di dalam pelbagai aplikasi untuk menghapuskan radiasi yang tidak diinginkan yang boleh mengganggu operasi sebuah sistem.

Kajian ini mengemukakan penghasilan dan pencirian komposit *lossy* SLS-HDPE berpemalar dielektrik rendah untuk aplikasi penyerapan gelombang. Jumlah jisim setiap campuran komposit ialah 25 g dan mengandungi kaca SLS bersaiz 63 μm . Mesin pengadunan *Plastograph EC Bradender* digunakan untuk mengadun komposit pada kelajuan rotor 50 rpm untuk 10 minit. Mesin tersebut ditetapkan pada suhu pemanasan 170 °C yang mana suhu tersebut dipilih untuk menyekat aliran HDPE matrik yang mempunyai takat lebur 160 °C. Kaca SLS dipilih dalam kajian ini kerana

kaca ini ialah hasil pembuangan berbentuk pepejal dari proses pengilangan yang mampu didapati secara mudah dan murah. Kaca SLS ini menunjukkan ciri mekanikal yang baik berbanding jenis kaca yang lain. Kaca SLS dan HDPE mudah didegradasi dan kedua-duanya mempunyai ketumpatan yang rendah berbanding bahan penghasilan yang lain. Peratusan bahan pengisian yang berbeza digunakan dan diadun untuk menghasilkan komposit SLS-HDPE untuk memberi ciri penyerapan di dalam HDPE matrik yang berbeza. Struktur kristal komposit dicirikan menggunakan mesin belauan sinar-X (XRD).

Pengiraan secara teori pekali pantulan dan trasmit sampel yang telah disusun di dalam segi empat tempat gelombang pandu dan juga sample yang telah diletakkan di atas sebuah mikrojalur yang terbuka disimulasi menggunakan Kaedah Unsur Terhingga (FEM) yang bersambung dengan perisian COMSOL. Pengukuran pekali pemantulan dan penghantaran, dan juga ciri-ciri dielektrik, kedua-dua diukur menggunakan PNA (N5227) Network Analyzer segi empat gelombang pandu dan mikrojalur di suhu bilik berdasarkan frekuensi 8 GHz ke 12 GHz. Penyiasatan di antara parameter penyerakan yang dikira dan yang diukur juga dikaji. Ketelusan kompleks komposit didapati terikat kepada nisbah pengadunan SLS-HDPE. Pada gelombang frekuensi X-band 10 GHz, ketelusan sebenar pemalar dielektrik komposit SLS-HDPE yang juga merupakan pemalar dielektrik dan faktor kehilangan masing-masing bernilai antara 2.44 ke 2.91, sementara nilai faktor kehilangan adalah 0.05 ke 0.17. Pemalar dielektrik dan faktor kehilangan komposit boleh dijangka dari persamaan regresi dengan meletakkan nilai-nilai pecahan komposit pengisian. Kedua-dua pemalar dielektrik dan faktor kehilangan komposit SLS-HDPE bertambah dengan penambahan peratus pengisian SLS. Oleh itu, perkara mungkin akan menghasilkan pekali penghantaran $|S_{12}|$ dan nilai magnitud pekali pemantulan yang lebih tinggi $|S_{11}|$ berdasarkan teori padanan impedans. Walaubagaimanapun, keputusan yang didapati daripada parameter penyerakan telah digunakan untuk menentukan kehilangan penyerapan berdasarkan peratusan sampel komposit yang berlainan. Akhir sekali, kajian perbezaan peratusan SLS kepada medan elektrik telah dijalankan dengan memerhati pola taburan medan elektrik oleh sampel yang diletak dalam segi empat gelombang pandu. Taburan medan elektrik daripada FEM menunjukkan bahawa semakin tinggi pengisian SLS, semakin rendah taburan medan yang ditransmit melintasi sampel daripada ruang masuk kepada ruang keluar segi empat gelombang pandu. Pemerhatian menunjukkan pengurangan kekuatan medan apabila pengisian SLS meningkat, membuktikan hasil permitiviti kompleks komposit adalah bernilai $\epsilon' = 2.44$ untuk 10% SLS, 2.55 untuk 20% SLS, 2.65 untuk 30% SLS, 2.83 untuk 40% SLS dan 2.91 untuk 50% SLS sementara nilai faktor kehilangan adalah $\epsilon'' = 0.05$ untuk 10% SLS, 0.08 untuk 20% SLS, 0.10 untuk 30% SLS, 0.15 untuk 40% SLS dan 0.17 untuk 50% SLS di mana ketelusan tertinggi bernilai $\epsilon' = 2.91$ and $\epsilon'' = 0.17$ adalah untuk pengisian komposit 50% SLS, penambahan pengisian SLS yang akan menyebabkan komposit mudah lentur dan meningkatkan penyerapan haba yang mana akan menghasilkan lebih haba di dalam komposit dan kemudian menyebabkan komposit itu berpecah. 50% SLS menunjukkan ciri-ciri termal dan elektrik yang lebih baik, pemalar dielektrik yang

lebih tinggi, faktor kehilangan dan kehilangan tenjen meningkat. Komposit 50% SLS mempunyai sifat menyerap yang paling tinggi, maka ini menunjukkan ianya terbaik untuk aplikasi penyerap gelombang mikro.



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

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LIST OF ABBREVIATIONS AND SYMBOLS

HDPE	High density polyethylene
SLS	Soda lime silica
OECP	Open Ended Coaxial Probe
RWG	Rectangular waveguide
c	Velocity of light
ϵ^*	Complex permittivity
ϵ'	Dielectric constant
ϵ''	Loss factor
EM	Electromagnetic
EMI	Electromagnetic interference shielding
σ	Electrical conductivity
ϵ	Permittivity
μ	Permeability
SE	Shielding effectiveness
dB	Decibels
FEM	Finite Element Method
XRD	X-ray diffraction
T/R	Reflection/Transmission
RFI	Radio frequency interference
J	The current density
D	The electric displacement
B	The magnetic flux density

γ	Propagation constant
ρ_q	The electric charge density
β	Phase constant
∇	Laplacian vector
d	Sample thickness
η	Impedance
η_0	Impedance in free space
ω	Angular frequency
f	Frequency
f_r	Critical frequency
ϵ_r	Relative dielectric of the substrate
H_z	Longitudinal magnetic field
E_z	Longitudinal electric field
P_1	Power measured with the material inserted
P_2	Power measured without material inserted
Z_0	Impedance of free space
Z_{in}	Impedance of the absorbing material
S_{11}	Input reflection coefficient of port one
S_{12}	Transmission coefficient port one
S_{22}	Input reflection coefficient of port two
S_{21}	Transmission coefficient port two
TE	Transverse Electric
TM	Transverse Magnetic
k_0	Free-space wave number

TEM	Transverse electromagnetic modes
MoM	Method of Moment
MUT	Materials under test,
PNA	Professional network analyzer
FDTD	Finite difference time domoi



CHAPTER 1

INTRODUCTION

As the telecommunication industries are increasing day by day, the usage of mobile telephones is also increasing, this led the industry into searching on how to curtails the effects of electromagnetic interference (EMI) pollution arising from the increasing number of the telecommunication users through mobile telephones, local area networks, wide area networks and radars systems. The need to have a microwave absorbing material at high frequency is necessary. Soda lime silica and high density polyethylene (SLS-HDPE) composites can be applied in many areas of telecommunication as absorbing material (Abbas et al., 2001)) as shown in the schematics, Figure 1.1.

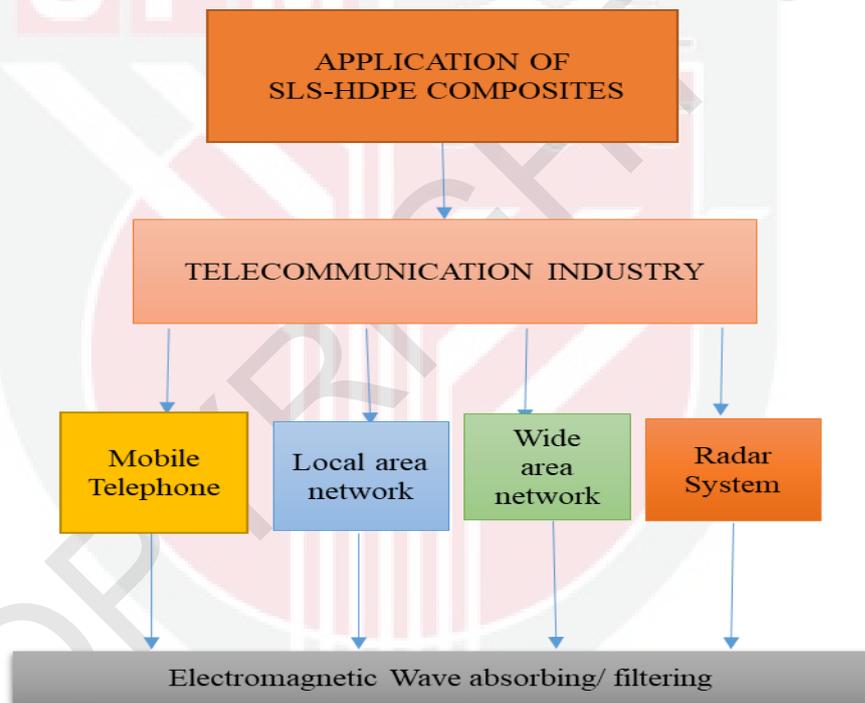


Figure 1.1 : Representation of schematic application of SLS-HDPE composites

The microwaves is a class of electromagnetic radiation, like radio waves and gamma-rays. Microwaves has various ranges of usage and applications, which include, wide area network, and telecommunications, perhaps the most popular usage known by the people, cooking. The electromagnetic radiation is transferred in particles and waves form in distinct wavelengths and frequencies. The electromagnetic spectrum or the (EM spectrum) is a broad range of wavelengths. The electromagnetic spectrum is further subdivided into seven different regions based on the order of their increasing in energy and frequency and decreasing in wavelength. Their common divisions are:

radio waves, microwaves, infrared (IR), visible light, ultraviolet (UV), X-rays and gamma-rays. Microwaves falls in between the electromagnetic range of radio and infrared wave light. The frequencies range of a microwaves is from about 1 gigahertz (GHz), or 1 billion cycles per second, up to about 300 gigahertz or 300 billion cycles per second, and wavelengths ranges from about 30 centimeters (12 inches), to 1 millimeter (0.04 inches). Each of these regions is further divided into number of bands, with descriptions such as, (L, S, C, X and K,) according to Ginger Butcher's book "Tour of the Electromagnetic Spectrum."

In the past decades, attention of researchers', material scientists and industries have been attracted to the use of natural fibers due to its peculiar advantages when compared to synthetic or conventional fibers. Natural fibers are environmentally friendly, and the issue of environment is at the top of the agenda both nationally and internationally. However, natural fiber due to its biodegradable nature unlike synthetic fibers has become center of attraction. This is in addition to low cost and low density (Mohanty et al., 2002) and (Singha, & Thakur, 2008).

The complex permittivity and permeability, the microstructure of the absorber, sample thickness, and class of the material are used to determined microwaves absorption properties. In view of these, the nanocomposites type of metal were widely used. Carbon nanotube (CNT) composites coated with magnetic particles and magnetic particles encapsulated with carbon have attracted for electromagnetic wave absorbers (Abbas et al., 2001). The process involved in the fabrication of magnetic particles doped (CNT) is unfavourable for the application absorbing nanocomposites. This condition led to searching for new absorbing composites materials. This search has led scientists to

SLS microparticles that can be used as high efficiency microwaves absorber as a result of its low dielectric constant, high loss factor and low complex permeability $< \sim 1$ (Tan et al, 2014; Cao, et al, 2007). SLS plays a vital role in various ranges of applications, ranging from windowpanes, to glass containers, to granular form in closed breathing environments, to ceramics from pharmaceuticals to agriculture, and from chemicals to electronics.

The following pie chart shows world consumption of soda lime silica SLS (de Jong, 1989)

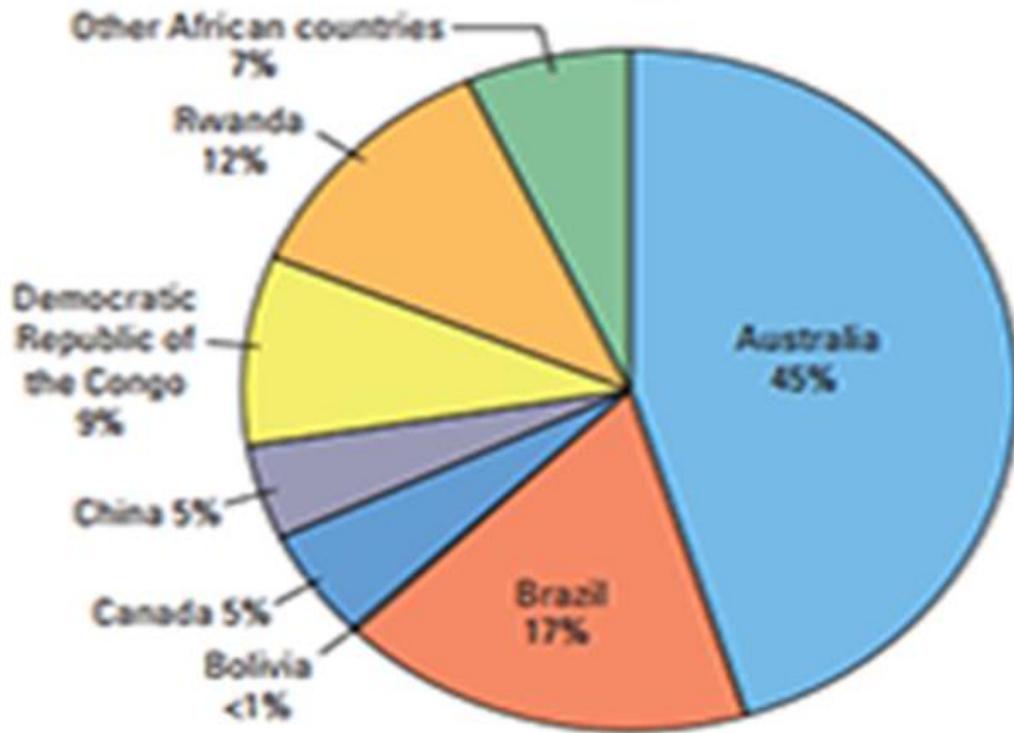


Figure 1.2 : Worldwide consumption of Soda Lime Silica (De Jong, 1989)

The low dielectric constant and high loss factor characteristics of SLS as well as low dielectric constant and low loss factor of HDPE explain the advantages SLS-HDPE composites as absorbing material when compounding to have a composite material of low dielectric constant high loss factor, and this give it the ability of reflecting electromagnetic wave less and absorb more which is good characteristics for microwave application, it is easy to synthesized large measure synthesis of SLS microparticles which is cost effective. Due to SLS unique geometrical morphology, cage like SLS/SiO₂ microcomposites exhibited a strong attenuation of microwave at X band frequency (Latif et al., 2015).

1.1 Soda Lime Silica

SLS glass and Soda lime silica glass waste composites have been used in a variety of applications because is a nontoxic waste that mainly obtained from packaging applications and in Europe the quantity production is increasing. In Italy, glass waste production has increased more than 9 % in the past five years and its use has risen above 14 % (Escalera et al., 2011). The recycling of these materials has also increased, due to the advent of better industrial organization. Over one million tons of SLS glass is now being recycled annually in Italy, 51.5 % of total production (Escalera et al.,

2011). The total percentage of glass recycling in Greece is quite low, ranging in the order of 27 %. (Pontikes et al., 2005). Recycling is mainly restricted in the largest cities and, similarly to Italy, differentiated collection is in limited use. Both countries should seek ways to increase recycling rates in order to meet the target of 60 % established by the European Union for 2008. In Malaysia the recycling is rather very low less than 5%. The total amount of glass in the Malaysian waste stream from 1994 was around 315 tons per day or 114 975 tons per year. The average market selling price at source for bottles for that year was US\$0.03 per kg or US\$32 per ton. Thus, the expected revenue from separating and selling glass at source is about US\$3.7 million per year. Assuming that only 5% of glass or bottles from the waste stream could be recycled, the estimated revenue is about US\$0.2 million per year (Zainab et al., 2008). Soda lime silica glass is relatively cheap, reasonably rigid, chemically stable, and extremely workable. As a result of these it can be remelted and resoftened in different occasion, it is suitable for glass recycling (Congcal.com.congcal., 2013). Soda lime silica glass is synthesized by melting the raw materials such as, lime (Ca(OH)_2), silicon dioxide (silica, SiO_2), sodium carbonate (Na_2CO_3), aluminium oxide (alumina, Al_2O_3), dolomite ($\text{CaMg}(\text{CO}_3)_2$), and small quantities of fining agents (e.g., sodium chloride (NaCl), sodium sulfate (Na_2SO_4), etc.) in a glass furnace at temperatures up to 1675 °C. (De Jong, 1989). In general, the integration of wastes glass into a fabrication process always brings about at least two environmental advantages: reduction in the disposal problems and conservation of natural raw materials. The present research was focused towards determining the likelihood of introducing SLS waste glass from urban scraps (glass packaging) into a microwaves absorber as absorbing material.

1.2 Polymers

A polymer, for example, High density polyethylene has been found to be applicable in various fields of research in the world today. It is frequently compounded with natural minerals so as to improve their properties. Glass fibre has been employed in the improvement of the stiffness and strength of thermoplastic material (Sanadi et al., 1995). Polymer-glass composites are mostly easy to produce because the soda lime silica glass waste materials are readily available, also it possess improved mechanical and electrical properties (Ibrahim et al., 2011). This work is aim at finding out the complex permittivity properties of the composites prepared using different ratios of environmentally friendly SLS and HDPE at microwave frequencies band. SLS is a solid waste and was chosen for this research in order to fabricate material having low reflecting and high absorbing properties, and also to reduce the excess SLS waste from the environment. At present time, the demand for wireless technologies is increasing promptly day by day where light weight, small size, easy installation devices are needed, and small profile microstrip antenna is needed. The antenna has become an essential in wireless communication system. A microstrip antenna has various ranges of usage ranging from spacecraft, higher performance aircraft, and satellite and missile application. The microstrip antenna play a very vital role in communication system, as a result of its attractive features such as light weight, low power handling low size, cost effective, easy to fabricate and easy in production (Singh et al., 2012). A

microstrip antenna have the ability of providing better performance of radiation and has a good acceptance when compare to other antenna because of its ability to focus energy into a tight beam (narrow beam width) to a particular direction in order to provide better performance of radiation (Rakesh et al., 2011). As the growth of microelectronics industries keep on increasing throughout the 21st century, further sophisticated processes and advanced materials have been in a high demand. After the advent of microprocessor, the quantity of active devices at chip has drastically increased, almost multiplying each year, as legendarily predicted by Gordon Moore in 1965. Almost all these are necessitated by the need for optimal electrical and functional performance. The SLS-HDPE composite can be used as a substrate in many applications such as electromagnetic absorption, integrated circuits (IC), transmission lines and microstrip antenna mechanisms which can be found in mobile communication, defence industry and aerospace.

Detailed study of complex permittivity measurement of solid material via open ended coaxial probe technique (OEC) is yet to be carried out. In the light of the above, measurement of using OEC technique would be investigated and results would be compared with standard recommended technique like the rectangular wave guide technique (RWG). Other investigations would include the effect of different percentages of SLS inclusion in the host matrix on materials complex (Yaacoba et al., 2015) permittivity, scattering parameters and absorptions. Further understanding in the applications of dielectric materials can be found in (Pozar, 2009; (Laverghetta, 2005).

1.3 Composites

The term composites materials can be explain in different forms depending on the angle in which one viewed it. Composites can simply be define as a domain that contained two and more unlike constituents mixed together. The composites materials could either be organic or inorganic in nature. In an inorganic state, the composite could be three dimensional, two dimensional, one dimensional and even zero dimensional. In composites there is possibility of mixing different properties together that are so far impossible within a single material (Kochetov, 2013). Among the classes of composites, the polymeric composites have a promising future because of its high performance materials. The four types of polymeric composites are the clay-polymer composites, the metal-polymer composites, the oxides-polymer composites, and the carbon nanotubes composites. The two main methods used in obtaining glass-polymer composites are the Ex-situ synthesis and In-situ synthesis method. The changes in properties of composites are mainly as a result of phenomena such as dimension confinement, majority of interfacial phenomena and quantum mechanisms (Liang, 2007). The dependence of bulk properties of composites are mainly due to (Kochetov, 2013);

Fillers properties such as filler dimension, filler kind, host matrix: nature; thermoplastic or thermosetting, degree of dispersion and of agglomeration, and Synthesis methods.

1.4 Properties of Polymer composite

The polymer composite complex permittivity properties are mainly controlled by the conductive fillers. Consequently, the nature or type of fillers determines the permittivity characterization of polymer composites. Typical conductive fillers are metals, semiconductors, carbonic materials and intrinsic conductive polymers (Xu et al., 1999). Conductive Polymers have attracted a lot of attention in the recent time as a result of their excellent flexibility property and also ease of preparation procedures contrary to conventional inorganic semiconductors. They can be applied in the field of electronics as flexibility conductors and as absorbing devices especially with regards to electromagnetic waves absorber (Ma et al., 2005). In conventional conductive composites, carbon black particles at micro-meter sizes are used to achieve desired electrical characteristics. Researches have shown that large filler contents leads to a poor composites (Liang, 2007; Balberg, 2002); the use of glass powder reinforced polymers, led to the production of composites that has unique dielectric, mechanical and chemical properties. Filler material comes in different forms; these forms could be in metals, semiconducting oxides, dielectric ceramics and carbon materials (Liang, 2007).

1.5 Glass-polymer Composite

A glass composite having remarkably good mixture of properties is not a dream today. Glass-reinforced composites are known to be light and strong. This means with accurate processing techniques, glass usages, and coupling agents/ compatibilizers, composites with optimum properties can be used to make automobiles lighter, and thus much more fuel efficient.

Few years ago, polymers has substituted number of the conventional materials/metals in several ranges of applications. This was achieved due to the advantages polymers have as compare conventional materials. The most significant advantages of these polymers are, the simplicity in proccession, production and cost accessible. In significant number of these applications, fillers and glasses are used to shape properties of polymers to suit the high strength and high modulus requirements. The usage of glass to reinforced polymers has advantages for specific properties when compared to other conventional materials (Schneider et al., 1995). These composites have wide range of applications and usages in different areas, from space crafts to appliances. Over the years, the attentions of scientist and technologist have been drawn to the use of glass powder instead of conventional reinforcement materials. These glass powders are cost-effective, lower density and higher specific properties. They are nonabrasive and biodegradable, contrast to other reinforcing materials. More so, they are readily obtainable and their specific properties can be comparable to most of

other glass used in reinforcements (Colberg and Sauerbier, 1997). However, disadvantages such as mismatch with other polymer matrix like hydrophobic, lowly resistance to moisture and the tendency of developing aggregates during processing serve as setbacks to the potentials of glass as reinforcement in polymers (Schlosser and Knothe, 1997). Fillers 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 that improve the dielectric and mechanical properties of polymer-silicate composite. In the last decade, semiconducting oxides (SiO , CaO , and Na_2O) have attracted so much interest due to their potential for different electronic and photonic device applications (Colberg and Sauerbier, 1997). Recent researches have shown that the polymer-silicate composites exhibit excellent luminescent, optical, dielectric and bio-sensitivity properties. Researches have been conducted in the area of electrical properties of polymer-oxides compositions synthesized by in-situ polymerization and melt blending (Schneider et al., 1995). (Yaacoba et al., 2015) conducted a research on the electrical characteristics of the low density polyethylene-ZnO melt compounding technique was used to prepared the composites. They reported that the nanocomposites displayed a lower clarification limit and a slow decrease in resistivity with filler content as it was related to conventional micro-composites. The real permittivity breakdown strength was also found to be high for the nanocomposites for all filler contents. (Yaacoba et al., 2015), conducted a research to investigate the electrical and thermal assets of low density polyethylene-ZnO syntheses reinforced with ZnO whisker as well as nanoparticle. They stated that the dielectric constant and loss factor increased with the increase of ZnO content. Compared to ZnO whisker filled LDPE composites, the composites filled with ZnO nanoparticle exhibited increase in dielectric constant due to nanoscale effect.

1.6 Low Dielectric Constant Material

Persistent enhancement in a device performance and density has influence significantly the size features and convolution of the wiring configuration for chip interconnects. As the smallest device dimensions decrease lower than $0.18 \mu\text{m}$, the increase in transmission delay, energy dispersion, and crosstalk noise of the connected structure become regulating factors for ultra large scale integration (ULSI) of integrated circuits. Hence, to tackle these problems, new material for usage as interlayer dielectrics (ILD), and metal lines as a substitute designed are being fabricated to supplant the $\text{SiO}_2/\text{Al}(\text{Cu})$ interconnect technology. Thus this requires the incorporation of low dielectric materials and lower resistance conductors like copper as interlayer dielectric. The manufacturing industries are on its way to embracing the change to metallization of copper (Edelstein, 1997) and (Venkatesan, 1997), the industry is also working extremely to applied ILD using low dielectrics constant.

The insufficient and clear choice of low permittivity can be basically ascribed to the numerous challenges related to the successful incorporation of these materials into

future on chip connects. Apart from the low dielectric constant, material inter and intra level dielectrics must fulfil different number of requirements in order to be effectively incorporated, (Lee & Ho, 1997). These requirements include; resistance to processing chemicals, noble adhesion to the other interconnected-materials, high thermal and mechanical stability, little moisture absorption and cost effective.

Recently researcher's put in giant struggled to develop low dielectric constant, high loss factor composites devices that can concurrently gratify all of these requirements. The main task for now is to develop a composite material that is of low dielectric constant, high loss factor, and excellent mechanical and thermal strength. However, the categories of chemical structures that can influence structural steadiness are the ones having robust individual bonds and a high density of such bonds. Generally, the toughest bonds often have the highest polarizability; an increase in a bond density contributes to corresponding increase in polarization. For instance, the thermal steadiness of SiO₂ and inflexibility is partly due to the dense (2.20–2.40 g/cc) chemical network. The material density of SiO₂ and strongness the bond leads to a huge atomic polarization, hence resulted in an increased in dielectric constant. Organic polymer composites usually have a low dielectric constant as result of the low material density of these polymers (~1.00 g/cc), they also have low distinct polarizabilities bond. Regrettably, majority of these organic polymers are thermally decompose at distinctive back end of the line (BEOL) reducing (Al) treating temperatures (≥ 400 °C) due to the CC bonds of these polymers are not as strong as that of SiO bonds, usually polymers have greatly lower crosslinking density than the SiO₂. Processing of Cu damascene or inlaying and interconnecting (Lam et al., 2018) suggest the probability of decreasing the maximum BEOL processing temperature to (~ 350 °C), therefore increasing the processing temperature lead to materials low dielectric constant. The chemical and mechanical properties of the organic polymers are of inferior features as compared to that of oxide for similar reasons. The carbon-based materials that are frequently resistant to thermal disintegration and mechanical distortion are those possessing double and triple bonds, which have the tendency of increasing the dielectric constant of these materials. Low dielectric constant carbon-based polymers displaying sensible steadiness above 400 °C generally have dielectric constants between the ranges of 2.5 to 3.5. Since it is difficult to decrease the dielectric constant beneath 2.5 with entirely dense materials, it may be essential to introduce micro or mesoporosity to attain a low dielectric constant values (≤ 2.0). Mesoporous usually described materials having dimensions 2-50 nm in diameter, while micro porous is used to refer to materials having dimensions ≤ 2 nm in diameter. Perhaps it is proper to describe to micro porous solids as lower density materials than porous materials. The inclusion of voids brings about reduction in dielectric constant by decreasing the material density. In the same vain, the percentage of these porosity can be varied also the permittivity constant as well as material density. The network material that can be inorganic, organic, or a hybrid material can be incorporated by pores. Since inclusion of voids will compromise certain material properties which include mechanical strength, the chemical configuration of the porous network must be carefully designed to accomplish sufficient thermo mechanical steadiness. To achieve mechanical strength, the chemical configuration, SLS can be used to provide

an alternative to hybrid material because of its porous nature if compounded with polymer. The SLS has a very low density and the grain size would provide the required voids within the composites.

1.7 Characterization Techniques

In dealing with a concept propagation of waves, the characteristics of light waves and microwaves are typically the same both of their components travel in a straight line. The ability for waves to traveled in a straight line enable them to reflect, refract, diffract, interfere, and also scatter at boundary points with interacting media. However, their mode of interaction at the interface of media varies due to their different wavelength. The wavelength of a microwaves ranges from 1m to 1mm corresponding to frequency ranges from 0.30 GHz to 30.0 GHz. The simple characteristic allows microwaves to have an interaction with materials and structures at macroscopic level. For instance, microwave has the ability of pass through most of non-conducting materials, reflected at the surface and scattering at internal boundaries and also interacting with the internal molecules of the sample (Bahr, 1982)

1.7.1 Permittivity

The complex permittivity measurement is required not only for scientific applications but also for industrial applications. Areas in which dielectric properties of samples at microwaves frequency have found applications are microwaves heating, non-destructive testing, and biological effects of microwave (Weir, 1974)

Complex permittivity measurement is a significant factor in describing the chemical and physical properties associated to energy storing and energy loss in different type of materials (Wee et al., 2009).

The term dielectric constant is some time misleading, the dependency of dielectric materials on frequency causes it to have two parts, that is the real and imaginary permittivity complex. The fraction of imaginary permittivity to the real permittivity part of permittivity is termed loss tangent (Kittel, 1996). Permittivity is complex figures of which the imaginary part is related to energy lost.

The permittivity as well as scattering parameters of materials measured using microwave components are controlled by the basic properties of microwave. In a good conducting material, microwave has low penetrating depth. In this regard, they are usually used to measured non-conducting materials which include low-loss and lossy dielectric materials. To study the interaction between microwaves and materials, Maxwell's equation is often required. Properties like propagation mode, reflection, refraction, transmission and impedance are defined from the equation. The broad nature and properties of a material allows different techniques to be use for

measurement at microwave frequency range. Various number of techniques have been employed in electromagnetic properties measurements of at microwave ranges of frequencies. Among these techniques are the reflection and transmission line technique, resonant technique and open ended coaxial probe technique (*Agilent Technology*, 2011). Details of all these methods would be elaborated in the following chapters.

1.8 Problem Statement

In recent years, there is a demand for composites material with lower dielectric constant higher loss factor for microwave application. Composite material having low dielectric constant high loss factor has the tendency to reflect electromagnetic waves less and absorb more. These characteristics are much needed to minimize the effects of electromagnetic interference (EMI) interference arising from the increasing number of the telecommunication users through mobile telephones, local area networks, wide area networks and radars systems. Unfortunately composites with low dielectric constant high loss factor are not found naturally, there is need for fabrication of such composites. The common absorbing materials are the mu-metals, that is; an alloy contains copper, iron, nickel, and chromium. Unpolluted iron or specifically referred to as Ferrites are frequently used for the fabrication of microwave absorbing composite. However, similar to various metals, ferrites are destructive and corrosive metals, expensive, heavy, and they are non-biodegradable. Ferrites has a higher complex permittivity, this means ferrites reflects more, the reflecting characteristic of ferrites is not good for security, because this reflection can be detected by radar or interfere with electronics gadgets. Used of various metals like ferrites for microwave absorbing device can easily be resulted to galvanic corrosion which can caused an increased the non-linearity behaviour and also caused a decreased its absorbing effects. However, Soda lime silica glass (SLS) was choosen to replaced ferrites due to its numerous chacteristics, SLS has a low dielectric constant, high loss factor, which means when compounding with polymers (HDPE), SLS-HDPE composites would have the ability to reflect less and absorb more. SLS is biodegradable, noncorrosive, cost effective and environmetally friendly.

This research investigates the application of soda lime silica glass powder (SLS) as a fillers for microwave absorbing device substitute to ferrite, with host matrix (HDPE). SLS scrap is considered as a glass industrial discard which can be obtained from the glass waste materials. SLS offer numerous advantages such as acceptable specific properties, better thermal properties, cheap, insulating characteristics and lower density, lower energy consumption when processing and biodegradable nature. SLS has the ability of providing rigidity, tensile strength, good flexural strength, and elongation at breakdown. It is assumed that glass of small particle size will show a higher density which will then resulted in the increase both the loss factor as well as dielectric constant of the SLS (Panigrahi, 2010). Though, till date, the influence of particle size of SLS on the value of complex permittivity is yet to be explored.

An aliphatic polyester and Polycaprolactone (PCL) polymers, has been suggested as the host matrix for microwave absorbing device (Ahmad et al., 2017). Polymer like PCL has been identified as biodegradable. Though, the PCL has limited electronics application as a result of it lower melting temperature ranges (57–60 °C) as well as lower mechanical properties. Polycaprolactone waste has a long disintegration period more than 2 years. Whereas HDPE decomposing period is between 6 months to 1 year (Pal et al., 2013) and (Meng et al., 2010).

In contrast, biopolymers such as high density polyethylene (HDPE) are found to have many superior qualities, melting temperature of 160°C and higher mechanical strength. HDPE also has additional major advantages which include easy processing capability, biocompatibility, non-toxic, higher mechanical stiffness, it has a low dielectric constant and thermal flexibility. More significantly its rare material, ethylene, can be derived from renewable materials, primarily starch. HDPE is a polymer type produced from renewable materials, it has been proposed that the polymers can be use to help reduce greenhouse gas pollutions and also diminish fossil energy consumption as it relate to petrochemical based conventional polymers. The observance of electric field spreading in the transmission line for microwave circuit is consider to be significant. Even though the electric field spreading of a microwave substrate in a closed waveguide has been described in the texts (Hotta et al., 2011). Eventhough none of these literatures has been published in respect to open transmission line technique like microstrip. Electrical properties of heterogeneous candidates are closely related to its constituent's composition. Previous researchers have investigated the influenced of ferrites on the attenuation of biobased absorbing composites. It is imperative to distinguish between the role of attenuation of the composite material and ferrites for cost-accessable production of bio-based ferrite absorbing composite and the bio-based host matrix.

1.9 Hypotheses

1. Addition of soda lime silica (SLS) filler into high density polyethylene (HDPE) host matrix is expected to increase the magnitude of the dielectric constant loss factor and loss tangent of the composite, also dielectric constant is expected to decrease with increase in frequency while loss factor and loss tangent to increase with increase in frequency.
2. The X-Ray diffraction (XRD) data of 100% SLS is expected to show a broad hump without sharp peak indicating the amorphous nature of SLS, while the XRD data of 100% HDPE is expected to show two sharp peaks indicating the semi crystal nature of HDPE. Addition of soda lime silica (SLS) filler into high density polyethylene (HDPE) host matrix is expected to decrease the sharpness of the peaks of the HDPE host matrix.
3. Addition of SLS filler into HDPE host matrix is expected to enhance the reflection coefficient S_{11} as well as the transmission coefficient S_{21} properties of the composites by increasing the magnitude of S_{11} and decreasing S_{21} with increase in the percentage of SLS, and also increase the magnitude of S_{21} with

increase in frequency and decrease the magnitude of S_{11} with increase in frequency.

4. Addition of SLS filler into HDPE host matrix is expected to improve the absorption properties of the composite by increasing the thermal properties and rigidity as a result of increase in SLS percentage.
5. Addition of SLS filler into HDPE host matrix is expected to enhance the attenuation by decreasing the attenuation with increase in %SLS filler content in the sample.

1.10 Objectives

The key objective about this research is to prepare SLS-HDPE composites from soda lime silica glass based on high density polyethylene using the method of hot pressure and characterize their crystallinity phase formation. The explicit objectives of this study are listed beneath:

1. To fabricate SLS-HDPE composites of low dielectric constant, high loss factor from soda lime silica glass based on high density polyethylene for microwave application using the method of hot pressure technique.
2. To determine the effect of different percentage of SLS fillers on dielectric constant, loss factor and loss tangent of the SLS-HDPE composites via the open-ended coaxial probe and rectangular waveguide methods in the frequency range from 8-12 GHz.
3. To investigate the influence of fillers on the scattering parameters in relation to absorption limits of SLS-HDPE composites via microstrip and rectangular waveguide methods. The scattering parameters values will be related theoretically to Finite element method (FEM) values.
4. To visualize the electromagnetic field distribution in a waveguide loaded with SLS-HDPE composites using finite element method (FEM).

1.11 Scope of Study

In this research, low dielectric constant, high loss factor biodegradable composites was fabricated using SLS as the filler and HDPE as host matrix. The technique of melt blending through Brabender machine was employed. The effect of different %SLS fillers on the permittivity complex was investigated via the rectangular waveguide method (RWG) as well as the method of open ended coaxial probe (OEC). The impact caused by increase in the percentage of SLS fillers on reflection coefficient S_{11} as well as transmission coefficient S_{21} on the SLS-HDPE composites was also be investigated. The use FEM COMSOL software in simulating electromagnetic wave excitation and for calculating scattering parameters through SLS-HDPE composites when placed on a microstrip and in a rectangular waveguide would be carried out. The results achieved for scattering parameter through simulation, measurement and calculation will be compared. Inaccuracy examination of the contrast would be identify by both FEM and

measurements techniques. Specific equipment like the XRD, would be used to carried out morphological characterization, as this research focuses on the surface part of the composites and not inner portion it XRD characterization would be used.

1.12 Thesis Layout

This thesis comprises five chapters with appendices attached at the end of the chapters. Chapter 1 briefly discusses in generally on SLS and polymer composites, morphological and dielectric characterization, problem statements, and objectives of study, the scope of the study and lastly, the thesis layout.

Chapter 2 described reviews on SLS-HDPE composites, electromagnetic radiation (EM) measurement technique and limitations of some measurement techniques. Amongst others, the free space methods were also discussed. Numerical methods associated with rectangular wave guide were also discussed.

Chapter 3 contained the theory, the chapter started by emphasizing on Bragg's law and continued to discuss on Maxwell's equation and wave equation. It finally extended discussion on the transmission and reflection coefficients calculation procedures with FEM formulation techniques.

Chapter 4 comprises the entire methods used in this study. The preparations of SLS-HDPE composites were explicitly explained. The use of OEC, RWG, FEM methods and the microstrip line measurement are fully discussed in relation to microwave application, and also the morphological characterization using the XRD is discussed in details.

Chapter 5 is divided into six subsections. Section 5.1 deals with the morphology characterization of all the samples used in this work. The characterization technique used was XRD. Section 5.2 deals dielectric characterization of different percentage of SLS to determine the effect percentage on the permittivity. Section 5.3 deals with the dielectric characterization of the all the composites used in this research work using the open ended coaxial probe and rectangular wave guide methods. The effect of % SLS on the permittivity of the composites was also investigated. Comparisons between dielectric constant obtained using the two methods are also shown. Section 5.4 deals with the effect of SLS-HDPE composite ratio on the scattering parameters using rectangular wave guide, microstrip line and FEM. Variation in scattering parameters of sample with SLS and absorption of the electromagnetic waves based on the scattering parameters were also discussed.

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

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LIST OF PUBLICATIONS

Abubakar Dantani Meli, Zulkifly Abbas, Nor Azowa Ibrahim, Mohammad Hafiz Mohammad, (2017). Determining the complex permittivity of soda lime silica glass-High density polyethylene (SLS-HDPE) composites material by Open ended coaxial probe technique for microwave applications “Journal of Advanced Research in Fluid Mechanics and Thermal Sciences” (submitted).

Abubakar Dantani Meli¹, Zulkifly Abbas^{1,2,*}, Mohd Hafiz Mohd Zaid a,b, Nor Azowa Ibrahim “Advances in Polymer Technology” (submitted). Effect of SLS Percentages on XRD Characterization and Complex permittivity of SLS-HDPE Composites using Waveguide technique.





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