UNIVERSITI PUTRA MALAYSIA

MICROWAVE CHARACTERIZATION OF ZINC/ALUMINIUM AND MAGNESIUM/ALUMINIUM LAYERED DOUBLE HYDROXIDES FILLED WITH POLYVINYL CHLORIDE COMPOSITES

ETHAR YAHYA SALIH

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By

ETHAR YAHYA SALIH

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

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DEDICATION

"„, and of knowledge ye have been vouchsafed but little"

Holy Qur'an

"the significant problems we face can't be solved by the same level of thinking that created them"

Albert Einstein

To my beloved parents for their endless help, encourage & support

Thank you for everything

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

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Chairman: Associate Professor Zulkifly Abbas, PhD

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In recent years, many conventional plastic materials filled with materials have been proposed as alternatives to the most expensive mu-metal for electromagnetic interference applications. Some of these new materials claimed to have shielding effectiveness (SE) values as high as 130 dB. However it would be unnecessarily expensive in many applications if the objective is only to block the interference signals when even a 1dB SE is sufficiently enough. Shielding can also be improved by increasing the material thickness.

This thesis presents an extensive investigation of the properties of Zinc/Aluminium (ZLDH) and Magnesium/Aluminium (MLDH) Layered double hydroxide (LDHs) filled with Polyvinyl Chloride (PVC) composites as new shielding materials for microwave applications. The ZLDH and MLDH were prepared using the co-precipitation method, while the PVC-ZLDH/MLDH composites were prepared in Tetrahydrofuran (THF) solvent. The d-spacing obtained from the XRD analysis were 8.95 and 8.13 Å for ZLDH and MLDH, respectively. The EDX analysis results indicate that all elements are traceable. The samples were placed in closed T/R rectangular waveguide. The dielectric properties of the PVC-ZLDH/MLDH composites were measured in the frequency range from 1 MHz to 1.2 GHz using the capacitance technique with an impedance analyser. The dielectric properties were also determined using closed T/R rectangular waveguide technique with a vector network analyser in the X-band frequency range (8-12 GH_Z) from the measured transmission and reflection coefficients ($|S_{11}|$, $|S_{21}|$). The higher percentage of the fillers the higher were the magnitude of the $|S_{11}|$ and the lower were the magnitude of $|S_{21}|$. The shielding effectiveness of PVC can be increased from 1.06 dB to 2.5 dB for 1 mm sample thickness by adding 7% and 5% of ZLDH and MLDH, respectively. The results of the dielectric properties demonstrated a noteworthy increase in both dielectric constant and loss factor of the PVC after introducing the fillers. It was found that adding 5% of the fillers changed the property of PVC from medium loss to high loss material. Theoretically, the calculation of magnitudes $|S_{11}|$ and $|S_{21}|$ were carried out using Finite Element Method (FEM), Finite

Integral Technique (FIT) and Nicholson-Rose-Weir technique (NRW); the results were compared with the measured $|S_{11}|$ and $|S_{21}|$. The results of the relative error of $|S_{11}|$ and $|S_{21}|$ suggest the FEM is the most accurate method. The mean relative errors of FEM were 0.158 and 0.045 for $|S_{11}|$ and $|S_{21}|$, respectively. In contrast, the respective mean relative errors of FIT were 0.20 and 0.069 whilst the NRW were 0.183 and 0.087.

Abstrak tesis dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan bagi mendapat Ijazah Sarjana Sains

PERINCIAN MIKROGELOMBANG PADA KOMPOSIT PLYVINIL KLORIDA YANG DIISI DENGAN ZINK/ALUMINIUM DAN MAGNESIUM/ALUMINIUM DUA LAPISAN HIDROKSIDA

Oleh

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Pada masa kini, bahan plastik yang diisi dengan bahan konduktif telah dikenal pasti sebagai alternatif kepada logam-mu yang lebih mahal untuk aplikasi perlindungan electromagnet. Ada diantara bahan-bahan tersebut yang didakwa mempunyai nilai efektif setinggi 130 dB. Bagaimanapun, nilai setinggi ini tidak perlu untuk pelbagai aplikasi memandangkan objektif utama adalah untuk mengurangkan gangguan signal sehinggakan nilai sebanyak 1 dB SE sudah memadai. Selain itu, perlindungan boleh diperbaiki dengan meningkatkan ketebalan bahan tersebut.

Thesis ini mengandungi kajian terperinci mengenai sifat komposit polivinil klorida (PVC) yang diisi dengan Zink/Aluminium (ZLDH) dan Magnesium/Aluminium (MLDH) dua lapisan hidroksida (LDHs) sebagai bahan pelindung baru bagi aplikasi mikrogelombang. Kedua-dua ZLDH dan MLDH disediakan dengan kaedah sejatan bersama manakala komposit PVC-ZLDH/MLDH disediakan dengan pelarut Tetrahidrofuran (THF). Nilai d-spacing yang didapati dari analisa XRD adalah 8.95 untuk ZLDH dan 8.13 untuk MLDH. Analisa EDX pula mendapati semua unsur dapat dikesan. Semua sampel diletakkan di dalam T/R pandu gelombang segi empat. Sifat dielektrik komposit PVC-ZLDH/MLDH diukur pada julat frekuensi dari 1 MHz ke 1.2 GHz dengan kaedah kapasitans dengan penganalisa rintangan. Sifat dielektrik komposit juga ditentukan dengan kaedah T/R pandu gelombang segi empat dengan penganalisis rangkaian vector dalam frekuensi jalur-X (8-12 GHz) dari pengukuran pekali pantul dan pekali hantar ($|S_{11}|$, $|S_{21}|$). Semakin tinggi peratusan pengisi, semakin tinggi nilai $|S_{11}|$ and semakin rendah nilai $|S_{21}|$. Keberkesanan pelindung PVC boleh ditingkatkan dari 1.06 dB kepada 2.50 dB untuk sampel yang mempunyai ketebalan 1 mm dengan menambah 7 % bahan ZLDH dan 5 % bahan MLDH. Hasil daripada pengukuran sifar dielektrik mendapati peningkatan ketara pada kedua-dua pemalar dielektrik dan kehilangan dielekrik pada PVC selepas ditambah dengan pengisi tersebut. Juga didapati bahawa penambahan sebanyak 5% daripada pengisi tersebut menukat sifat PVC daripada bahan kehilangan sederhana kepada bahan kehilangan tinggi. Secara teori, pengiraan nilai $|S_{11}|$ dan $|S_{21}|$ dibuat dengan mengunakan kaedah elemen hingga (FEM),

kaedah integrasi hingga (FIT) dan kaedah Nicholson-Rose-Weir (NRW); semua hasil pengiraan tersebut dibandingkan dengan nilai ukuran. Hasil pengiraan ralat relative kedua dua $|S_{11}|$ dan $|S_{21}|$ menunjukkan bahawa kaedah FEM adalah paling tepat. Nilai purata ralat realtif tersebut adalah sebanyak 0.158 untuk $|S_{11}|$ dan 0.045 untuk $|S_{21}|$**.** Sebaliknya, nilai purata ralat relatif bagi FIT adalah sebanyak 0.20 untuk $|S_{11}|$ dan 0.069 untuk $|S_{21}|$ manakala nilai purata ralat relatif bagi NRW adalah sebanyak 0.183 untuk $|S_{11}|$ dan 0.087 untuk $|S_{21}|$.

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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 Master of Science. The members of the Supervisory Committee were as follows:

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DECLARATION

Declaration by Graduate Student

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This is to confirm that:

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LIST OF ABBREVIATIONS

SE	-	Shielding Effectiveness
ZLDH	-	Zinc/Aluminium Layered double hydroxide
MLDH	-	Magnesium/Aluminium Layered double hydroxide
LDH	-	Layered double hydroxide
PVC	-	Polyvinyl Chloride
THF	-	Tetrahydrofuran
XRD	-	X-ray Diffraction
EDX	-	Energy Dispersive X-ray
T/R	-	Transmission/Reflection
MHz	-	Mega Hertz
GHz	-	Gaga Hertz
$\|S_{11}\|$, $\|S_{22}\|$	-	Magnitude of Reflection Coefficient
$\|S_{21}\|$, $\|S_{12}\|$	-	Magnitude of Transmission Coefficient
dB	-	Decibel
FEM	-	Finite Element Method
NRW	-	Nicholson-Rose-Weir
FIT	-	Finite Integral Technique
nm	-	Nanometre
RFID	-	Radio-Frequency Identification
AM	-	Amplitude Modulation
FM	-	Frequency Modulation

TV	-	Television
EMI	-	Electromagnetic Interference
Ω	-	Ohm
cm	-	Centimetre
UV	-	Ultraviolet
FTIR	-	Fourier Transform Infrared Spectroscopy
TGA	-	Thermo Gravimetric Analysis
DSC	-	Differential Scanning Calorimeter
NDT	-	Non-destructive Technique
EM	-	Electromagnetic
BEM	-	Bounded Element Method
MM	-	Moment Method
E	-	Electric Field Intensity
H	-	Magnetic Field Intensity
∅	-	Potential
ABC	-	Absorbing Boundary Conditions
B	-	Magnetic Flux Density
D	-	Electric Displacement
J	-	Current Density
ρ	-	Charge Density
w	-	Angular Frequency
σ	-	Conductivity
ε	-	Complex Permittivity

μ	-	Complex Permeability
k_0	-	Wave Number in Free Space
γ	-	Propagation Constant
∇_t	-	Laplacian Operator
D	-	Thickness
Z	-	Complex Impedance
R	-	Reflection Coefficient
T	-	Transmission Coefficient
VNA	-	Vector Network Analyser
PEC	-	Perfect Electric Conductor

CHAPTER 1

INTRODUCTION

Electromagnetic radiation or shielding has been a subject of intense research since the 1930's. The earliest was a simple resonant absorber (Barrick et al., 1970) fixed to the rear of 2 GHz antenna to improve its front-to-back ratio of the radiation pattern. The absorption or shielding effectiveness (SE) of a material is predetermined by its composition at given frequency. In recent years, variety of materials is commercially available appropriate for different application requirements. Some important requirements include high physical strength, low weight, high resilience, good flexibility, thickness, wide operational band width, high power capability, low outgassing and low cost.

Shielding materials for electromagnetic compatibility are required to reduce undesirable reflections from objects and devices and to eliminate electromagnetic interference between the reflection from the object and the inside of the enclosure and containers through use as gasket materials. For example, electromagnetic shielding is required to avoid signal from the core conductor to escape whilst simultaneously prevents signals from the 'outside' to interact with the core conductor. The coaxial cable shielding is in the form of a wire mesh surrounding an inner core conductor. Some cables even have set separate coaxial screens, one connected at both ends, the other at one end only to shield both electromagnetic and electrostatic fields. Shielding in microwave oven is accomplished by the oven's metal housing forming a Faraday cage with screen door allowing only visible light, with wavelength ranging between 400 nm and 700 nm to pass through the screen holes for visual inspection of the food from the outside. Radio frequency security for RFID chips embedded in various devices, such as biometric passports rely on electromagnetic shielding to prevent access to the stored data. Electromagnetic shielding materials are also used to prevent passive monitoring of keyboard emission that would allow passwords to be captured. In hospitals, shielding protection of medical and laboratory equipment against interfering signals such as AM, FM, TV, emergency services and other types of telecommunication signals.

1.1 An Overview of Electromagnetic Shielding Materials

Electromagnetic shielding is usually defined as the process of blocking external electromagnetic field (Barrick et al., 1970) in a space with barriers. Enclosures are shielded to isolate electronic and electrical devices from the 'outside world'. Flexible enclosures such as cables are shielded to isolate wires from its environment. Theoretically, shielding reduced the coupling of electromagnetic waves, electromagnetic fields and electrostatic fields. The shielding effectiveness depends very much on the type of the material used, its thickness, the size of the shielded volume and operating frequency.

The most common materials used for electromagnetic shielding include sheet metal, metal screen and metal foam. Holes or meshes in shielding materials must be smaller than the intended wavelength of the external radiation to ensure the enclosure will not assume them as an unbroken conducting surface. Another form of shielding approach is to coat the enclosure inner walls with a metallic ink or similar material especially for electronic components with plastic enclosure, the ink consists of a matrix filled with very small particulates metal, typically copper or nickel. The enclosure is sprayed with ink and left to dry to form a continuous conducive layer of metal, ready to be connected to the chassis ground of the equipment and thus enhancing shielding effectiveness of the enclosure.

A range of frequency for specific condition can be filtered by selecting suitable shielding material. Ferrites is one of the most commonly used material for radio frequency (RF) shielding as it satisfies most RF shielding needs, from computer and electrical switching rooms to hospital CAT-scan and MIR facilities.

1.2 Layered Double Hydroxide (LDH)

Generally, the layered inorganic material is called layered double hydroxide (LDH), also have significance as fillers for the characterisation of polymer composites component. These types of materials have variety of chemical compositions depending on different interlayer anions and metal species and they are recognized due to their catalytic layered double hydroxide based polymer composites activities in organic synthesis (Gérardin et al., 2005). Other possible applications of the LDHs and their adapted forms contain the applications of biomedical e.g., controlled release of many kind of drugs and biomolecules (Du et al., 2007), heat stability improvement and polymer composites flame retardancy (Touloupakis at el., 2011), thermal, dielectric and optical properties (Ahmed et al., 2012), pesticides adsorption and controlled release, novelty of materials preparation for exact applications e.g., magnetic nanoparticle synthesis, visible luminescence (Musumeci at el., 2010), and UV/photo stabilization or in wastewater treatment (Tian et al., 2007). Comparing between the layered silicates and layered double hydroxide, the second has the structural homogeneity improvement, and this structural homogeneity is easy to be tuned throughout their production (Besse and Leroux, 2002). LDHs are used as filler for the production of the polymer composites is based on two major characteristics: (i) their layered crystalline geometry with various intercalating anionic species, and (ii) their ability to interchange these interlayer anions with much larger organic anionic species (Costa et al., 2008). The second reason is significant, as the perfect LDHs are not fit for the diffusion of giant polymer chains or chain segments into their gallery space unless through the organic modification of the interlayer distance is considerably increased. There are many types of LDHs each has a specific crystal structure; such as Zinc/Aluminium-nitrate (ZLDH) and Magnesium/Aluminium-nitrate (MLDH) and many other types such as Nickel/Aluminium-nitrate, Ferrite/Aluminium-nitrate, etc.

1.3 Composites

The composites are usually engineered to process chemical and physical properties of materials and generally enhance their properties. Commonly, the composites consist of reinforcement and matrix. The reinforcement materials are surrounded by matrix material in order to provide the supports for that material by keeping their positions. The reinforcement provides special physical (mechanical and electrical) and chemical will be imparted by the reinforcement to enhance the original matrix (polymer) properties. Mainly, the matrix and the desired reinforcement are provided to the composite by polymer and filler, respectively. Normally, the filler is chosen according to the properties which mean the filler is selected based on what is needed. Although, composites not based on polymers can be found for instance metal alloy. These kinds of composites are beyond the scope of this study. This study focuses on the layered double hydroxide, specifically ZLDH and MLDH, into PVC through dispersion of the fillers powder in the polymer matrix solution. The composites are prepared to investigate their electromagnetic properties, such as, the dielectric properties (dielectric constant, loss factor) and S-parameters ($|S_{11}|$, $|S_{21}|$).

1.4 Problem Statement

Absorption and shielding are two important issues that need to be resolved for many military and EMI applications. Materials with good absorption are in demand for many electronic applications to eliminate or reduce interaction between the electronic equipment or devices and the outside world. For EMI applications, the most common shielding material is mu-metal, a high permeability alloy of 5% copper, 1.5% chromium, 14% iron and 79.5% nickel. Unfortunately, the metal shield is not only susceptible to corrosion but also very expensive. Materials with high percentages of mu-metals caused galvanic corrosion leading to nonlinearity and decrease in shielding effectiveness of metallic shields (Niranjappa et al., 1996). Recently, conventional plastic materials filled with conductive materials are gaining interests as alternatives to mu-metal. Some of the new materials claimed to have a shielding effectiveness (SE) as high as 130 dB (Luo, X., & Chung, D. D. L. 1996). Such materials are especially important for example in certain military equipment demanding shielding effectiveness of at least 80 dB from 10 KHz to 18 GHz. However, it should be noted that not all EMI applications demand a material with very high shielding effectiveness value. The main objective of shielding is to block the interference signals. It would be unnecessarily expensive to require 60 dB of shielding instead of 25 dB for enclosure if the associated cables and their connecting hardware provide only 30 dB (Kaiser, 2006). Even a 1dB shielding is probably sufficient to remove the interference effect from the external sources. Nevertheless, the performance of materials with low shielding effectiveness can always be improved by increasing the shielding thickness (Lakshmi et al., 2009; Abbas et all., 2005).

Recently, many materials potentially suitable for many microwave applications have been proposed (Cruickshank, 2011). Among these materials, layered double hydroxide (LDHs) materials consisting of absorptive elements such as Aluminium, Zinc and Magnesium are probably the most attractive requiring minimal effort on sample preparation at a fractional cost of the more expensive ferrites. However, to-date, publications related to polymer with LDHs fillers as absorbing materials at microwave frequencies are not available in the literature.

This thesis presents an extensive investigation of the morphological, structural, dielectric and microwave properties of Polyvinyl Chloride with Zinc/Aluminium and Magnesium/Aluminium Layered Double Hydroxide as new and cost effective shielding materials for EMI problem. Different LDHs will give different absorbing properties. In this work only two types of LDHs will be explored namely, the zinc/aluminium-LDH (ZLDH) and magnesium/aluminium-LDH (MLDH). These LDHs will be used as fillers hosted in the polyvinyl chloride (PVC) matrix. The main advantages of PVC as the host material include low cost, good physical and mechanical properties, and flexible enough to be molded into various packing shapes for EMI applications. Different composition of MLDH and ZLDH will give different absorbing properties in the PVC matrix. Too low percentage of MLDH and ZLDH will not provide sufficient power whilst too high percentage would be excessively expensive.

1.5 Research Objectives

i) To prepare ZLDH and MLDH via co-precipitation method and different compositions of PVC-ZLDH, PVC-MLDH composites in THF solvent and characterise them using XRD, FTIR SEM as well as EDX.

ii) To determine the variation in both reflection and transmission coefficients of various compositions of PVC-ZLDH and PVC-MLDH composites placed in closed T/R rectangular waveguide and compare them with the extracted values obtained using Nicholson-Rose-Weir (NRW), Finite Element Method (FEM), and Finite Integral Technique (FIT).

iii) To determine the dielectric constant and loss factor of PVC-ZLDH and PVC-MLDH using waveguide technique in conjunction with Nicholson-Rose-Weir (NRW) method and RF Impedance/Material analyser in the lower and upper frequency ranges from 8 to 12 GHz and from 1 MHz to 1.2 GHz, respectively.

iv) To determine the optimum composition of ZLDH and MLDH in the PVC matrix to provide a minimum 2.5 dB shielding effectiveness for 1 mm sample thickness and to change property of PVC from a medium loss to high loss material.

1.6 Scope of Thesis

An overview of shielding effectiveness, LDHs and composites are highlighted and the main objectives of this study are clarified earlier in this chapter.

Chapter 2 presents a description of several materials to control the EMI shielding effectiveness from different aspects of views. This is followed by a historical background on the materials used in this thesis, LDHs and PVC. A brief introduction to various characterisation techniques to measure shielding effectiveness are covered in Chapter 2. A description about the dielectric properties is covered. Finally, the numerical methods, FEM and FIT, are briefly described.

In Chapter 3, the materials, LDHs and PVC, theory are discussed. This chapter also highlights the basic electromagnetic theory, wave equation, and plan wave in homogeneous media. The Nicholson-Ross-Weir technique (NRW) to determine the dielectric properties at X-band is covered in Chapter 3. A brief description of theory of shielding effectiveness is introduced in this chapter. Finally, this chapter discusses the FEM and FIT implementation in COMSOL and CST software packages, respectively, in section 3.7.

Chapter 4 explains the preparation of the fillers, ZLDH and MLDH, and their composites in the PVC matrix. The measurement set up of the S-parameters, reflection and transmission coefficients, and dielectric properties using T/R rectangular waveguide and RF Impedance/Material analyser are highlighted in Chapter 4. Nevertheless, in Chapter 4, the step by step procedure of the simulation methods, FEM and FIT, are covered.

In chapter 5, a discussion about the microstructure and electromagnetic properties of the composite samples are analysed in details. Chapter 5 provides the results of comprehensive S-parameters for different ratio of the fillers, ZLDH and MLDH, to the polymer matrix, PVC. This is followed by the comparison of the measured S-parameters with their identical, simulated using FEM and FIT and calculated using NRW, is presented.

Lastly, Chapter 6 summarises all the results and findings obtained in this study. Furthermore, this chapter presents several recommendations to future works in this field.

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