UNIVERSITI PUTRA MALAYSIA

ENHANCEMENT OF FERUM MIXED CARBON NANOFILLER CHITOSAN POLYMER COMPOSITES FOR EMI SHIELDING

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by

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Thesis Submitted to the Department of Physics, Faculty of Science, Universiti Putra Malaysia, in partial Fulfillment of the Requirements for the degree of Bachelor of Science in Physics with Education (Hons)

FEBRUARY 2022

DEDICATION

This thesis is dedicated to:

My project supervisor, Dr. Nurul Huda Osman

My parents, Mohd Ridzuan and Jamilah

My lecturers,

especially my course coordinator, Dr Shuhazlly

My siblings,

My friends,

My partner,

and others that help me throughout completion of the thesis.

Thank you for the supports, encouragements and help.

ABSTRACT

ENHANCEMENT OF FERUM MIXED CARBON NANOFILLER CHITOSAN POLYMER COMPOSITE FOR EMI SHIELDING

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FEBRUARY 2022

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Electromagnetic Interference shielding is introduced to protect internal electromagnetic signal/waves from being disrupted by external electromagnetic signal/waves and a way to reduce the coupling of electrostatic and electromagnetic fields. Chitosan is used as conducting polymer of this shielding materials as for its biodegradable and environmentally friendly. As for the enhancement in the conductivity, carbon nanofiller is introduced. As a result of this study, the shielding effectiveness is compared between pure chitosan film, Fe_3O_4/Ch (0.5wv%) film, Gr/Fe_3O_4/Ch (0.5wv%) film and f-MWCNT/Fe_3O_4/Ch (0.5wv%) film. Some characterizations were done and the total shielding effectiveness were observed.

ABSTRAK

PENAMBAHBAIKAN LOGAM CAMPURAN PENGISI NANO KARBON POLIMER KOMPOSIT KITOSAN DALAM PERISAI GANGGUAN ELEKTROMAGNET

Oleh

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Perisai Gangguan Elektromagnet diperkenal bagi melindungi signal elektromagnet dalaman daripada gangguan signal elektromagnet luar dan cara untuk mengurangkan gandingan elektrostatik dan medan electromagnet. Kitosan digunakan sebagai polimer pengalir elektrik dalam bahan perisai ini kerana sifatnya yang biodegradasi dan mesra alam. Dalam penambahbaikan kekonduksian, pengisi nano karbon diperkenal. Sebagai hasil penyelidikan ini, keberkesanan perisai dibandingkan antara filem kitosan tulen, filem Fe_3O_4/Ch, filem Gr/Fe_3O_4/Ch dan filem f-MWCNT/Fe_3O_4/Ch. Beberapa ujian perwatakan telah dijalankan dan jumlah nilai keberkesanan perisai telah diperhatikan.

ACKNOWLEDGEMENT

In the Name of Allah, the Most Merciful. The Almighty Allah deserves praise and glory; thank you for providing me with the strength and inspiration to complete this project.

To begin, I would like to express my heartfelt appreciation to my parent, my supervisor, Dr Nurul Huda Osman, and course coordinator, Dr. Shuhazlly, for their unwavering support, advice, and guidance throughout the research and thesis writing process. Your sacrifices will never go unnoticed. You made my journey possible and gave a huge impact to myself.

Additionally, I want to express my gratitude to my supportive partner Nurin Nadiah for all of her emotional contributions during the thesis writing process. She had been motivated me a lot. Last but not least my best guidance, Hazeem Ikhwan and Nurul Najiha for guiding me from the start until the end without complaining and had treated me like their own younger brother. I will never forget. I truly appreciate you and your time you spent helping me in many occasions.

Finally, I would like to express my gratitude to everyone who contributed directly or indirectly to the project's success.

.

APPROVAL

This thesis entitled "ENHANCEMENT OF FERUM MIXED CARBON NANOFILLER CHITOSAN POLYMER COMPOSITE FOR EMI SHIELDING" by AMIRUL AIMAN BIN MOHD RIDZUAN (Matric No.: 197535), was submitted to the Department of Physics, Faculty of Science, Universiti Putra Malaysia and has been accepted as partial fulfilment of the requirement for the degree of Bachelor of Science in Physics with Education (Hons).

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LIST OF ABBREVIATIONS

EM	Electromagnet
EMI	Electromagnetic Interference
RFI	Radio-frequency Interference
Ch	Chitosan
Fe_3O_4	Ferum (III) Oxides
Gr	Graphite
f-MWCNTs	Functionalized Multiwall Carbon Nanotubes
SE_A	Absorption Shielding Effectiveness
SE_R	Reflection Shielding Effectiveness
SE_T	Total Shielding Effectiveness
Fe	Ferum
dB	Decibel
XRD	X-Rays Diffraction
VNA	Vector Network Analyzer

CHAPTER 1

INTRODUCTION

1.1 History of Electromagnetic Interference (EMI)

In 1933, Comité International Spécial des Perturbations Radio (CISPR) under an International Electrotechnical Commission (IEC) sub-committee in Paris had secretly obtained and recognized the field of Electromagnetic Interference (EMI) in an effort to understand better the long-term complications that could sprout due to radiofrequency technology.

In 1934, CISPR began developing and propagating standards for suggested permitted emissions and immunity limits for electronic devices, which have evolved into most of the world's EMC regulations.

From the 1960s to 1980s, researchers were increasingly concerned and worried about electromagnetic emissions interference. So, in 1967, they developed testing and verification standards and regulations, as well as emissions and susceptibility limitations for electronic equipment used in military applications. As the system and electronic devices have gotten compatible over the years, these restrictions continued to change.

Throughout the years, electromagnetic fields and interferences have become more well-documented topics of conversation, discussion and concern. The National Energy Policy Act of 1992 permitted the Department of Energy to sponsor a $65 million five-year study initiative with the utility industry to determine the effects of electromagnetic field power line exposure on human health and to give public information.

On top of this, researchers, especially in the aerospace field, had investigated the EMI and the related issues to understand how the interference is created and the ways that the new system can minimize the transmission of the interference to the surround while also being able to hold back the inevitable interference from other sources.

Since its founding, EMI shielding awareness has emerged and led to many business companies devoted to manufacturing with various shapes, types and materials. EMI shielding is available as solid enclosures, wire mesh, screens, gaskets, O-rings, cable shields, and coatings.

1.2 Background of Study

Electromagnetic (EM) waves radiation has risen in the past decades due to the growth of microprocessors usage in this current device such as mobile phones. With the rapidly evolving communication systems and electronic devices, we should take this EM interference more serious. This electromagnetic interference (EMI) can be an objective, and significant problem as EM waves radiation can harm our body, health, electronic devices and system. With electronic devices getting more compact, not surprisingly more complex, study and research about this must be enhanced to face those whole new challenges in the future.

EMI is known when the function of an electronic device is disrupted. This occurs when a gadget is in the radio frequency range so that it is surrounded by an electromagnetic field triggered by various electronic devices. This electromagnetic interference has the potential to cause problems. EM waves can interfere with the activity or ability of electrical equipment or components while being operated. The disturbance of electromagnetic interference would affect the performance of a device. In our daily lives, we are all exposed to EMI. Here seem to be a few examples:

- Noise on microphones from a cell phone.
- Disturbance in the audio or video signals on radio or television.
- We are supposed to turn off mobile phones during flights, especially while taking off or landing, because the EMI from an active mobile phone interferes with navigation signals.

These show EMI may interrupt, obstruct, or otherwise cause an appliance to perform poorly, otherwise damaging it. Study and research about electromagnetic interference (EMI) shielding must be developed and enhanced.

EMI shielding is described as the reflection and absorption of electromagnetic radiation by a component that functions as a barrier to the radiation penetrating the shielding materials. (Shukla, 2019). The purpose of EMI shielding is to prevent electromagnetic interference (EMI) or radio frequency interference (RFI) from disrupting electronic appliances and systems. EMI shielding can then be used to isolate the electromagnetic field source or as an enclosure of the electronic equipment that needs to be protected.

The causes of electromagnetic interference (EMI) are numerous, including both artificial and natural sources. The consequences might vary from minor disruption and data loss to system failure and even malfunctioning. Shielding is a must to protect electrical devices from reducing the circuit's performance, especially electronic devices with small voltages and currents, which can be disrupted. There are various types of EMI shielding materials. Effective EMI shielding material is usually made of metal, although it comes in a variety of shapes and sizes. Here it comes to valuing one material's effectiveness to shield electronic devices or systems to see how great it works.

The EMI shielding method blocks the field via conductive or magnetic barriers. By using this EM shielding technology, the energy of electromagnetic waves is lowered in order to prevent interference with the operation of electronic components. At the moment, electromagnetic shielding and microwave energy absorption materials have been devised and synthesised to lower the likelihood of electronic equipment malfunctioning due to electromagnetic noise and pollution.

In order to gauge the effectiveness of shielding, a precise set of test and measurement criteria can be applied to a shielded device. One of the most essential markers to determine the

efficiency of EMI shielding is attenuation. The term "measurement" refers to comparing an electromagnetic signal intensity before shielding against the electromagnetic signal intensity after shielding. This measurement procedure is known to be measured in decibel (dB) that correspond to the difference in field strength with and without a protective medium. Determining the degree of attenuation for an EMI shield may be difficult, and the procedures for obtaining the findings might vary depending on the shielding application.

In general, metal can be the most compromise material for EMI shielding rather than other materials. This is because metal has a significant reflectivity and an excellent conductive material to isolate devices from EM waves and signals. This can be reflected in the Faraday cage principle as metal can absorb, reflect and relay electromagnetic interference and make the inside of the cage is entirely out of external electric fields while most plastics are insulators. As a result, electromagnetic waves may easily travel through them. Conductive barriers must be used as shields to stop the waves. Plastics are not suitable to be used for EMI shielding in their original state, which brings us to more complex procedures.

To conclude, a study has been explored to find one material that can be used is EMI shielding. The effectiveness of minimum reflection and absorption is yet to be determined. A lot of hard work has poured into this effort.

1.3 Mechanism of EMI Shielding

The principle behind how a material is able to shield EMI can be seen in Figure 1.1. The primary mechanism of EMI shielding is reflection. For reflection of the radiation, the shield must have mobile charge carriers (electrons or holes) that interact with the radiation's electromagnetic fields (Chung *et al*, 2001). They function mainly by reflection due to the mobile charge carriers or free moving electrons in them. These free moving electrons cause the

material to have good electrical conductivity. Metals are by far the most common materials for EMI shielding due to its excellent ability in electrical conducting.

A secondary mechanism of EMI shielding is absorption. For significant absorption of the radiation by the shield, the shield should have electric and magnetic dipoles which interact with the electromagnetic fields in the radiation. Metals may provide the electric dipoles with a high dielectric constant value. The magnetic dipoles may be provided by Fe_3O_4 or other materials with a high magnetic permeability value.

Besides reflection and absorption, a shielding mechanism is multiple reflections, which refer to the reflection at various surfaces or interfaces in the shield. This mechanism requires the presence of a large surface area or interface area in the shield. The loss due to multiple reflections can be neglected when the distance between the reflecting surface or interface is significant (such as porous or foam material/material containing filler) compared to the skin of depth.

The absorption loss is a function of the product σ and μ. In contrast, the reflection loss is a function of the ratio σ/μ, where σ, is the electrical conductivity relative to copper and μ is the relative magnetic permeability. Silver, copper, gold and aluminium are excellent for reflection due to their high conductivity. Superpermalloy and mumetal are excellent for absorption due to their magnetic permeability.

The losses, whether due to reflection, absorption or multiple reflections, are commonly expressed in dB. The sum of all the losses is the shielding effectiveness or total shielding effectiveness (SE_T). The absorption loss is proportional to the thickness of the shield. This loss due to absorption is known as absorption loss (SE_A). The partial loss due to reflection is known as the reflection loss (SE_R).

The ability of the EMI shielding material to attenuate or minimize the EM signal is defined by the term electromagnetic shielding effectiveness (SE). It is the ratio of the incident field strength to the transmitted field strength as expressed in Equation 1 to 3.

$$SE_P = 10\log(P_{in} / P_{out}) \quad (1)$$

$$SE_E = 20\log(E_{in} / E_{out}) \quad (2)$$

$$SE_H = 20\log(H_{in} / H_{out}) \quad (3)$$

where the P, E and H are the strength of plane wave, electric field and magnetic field of the EM waves. Subscriptions in and out represent the magnitude of the field strength that is incident on and transmitted through an EMI shielding. Both electromagnetic waves are electrical € and magnetic (H) fields orthogonal to each other. The EM wave propagates at a right angle to a plane comprising an electrical field and a magnetic field, and its properties depend on its frequency and related photon energies.

The skin depth of good electric conductor can be expressed in Equation 4.

$$\delta = \sqrt{\frac{1}{\pi\omega\mu\sigma}} \quad (4)$$

Where,

μ is the relative permeability of EMI shielding

ω is the frequency

σ is electrical conductivity of shielding material

εo is the permittivity of free space ($8.854 \times 10^{-12} F/m$)

Based on the Equation 4, the skin depth will inversely proportional to the magnetic permeability, electric conductivity and frequency.

The total SE of EMI shielding material, is the total of three type of mechanism from reflection (SE_R), absorption (SE_A), and multiple reflection (SE_M) as Equation 5.

$$\mathrm{SE_T} = SE_A + SE_R + SE_M \qquad (5)$$

Losses due to reflection, absorption and multi-internal reflecting structures are impedance functions, and thus their values are different for the electrical field, the magnetic field, and the plane wave. On the contrary, the absorption phenomenon does not depend on the impedance, and hence the absorption loss will have the same value for all these three fields. Is negligible when it is greater than 10 dB.

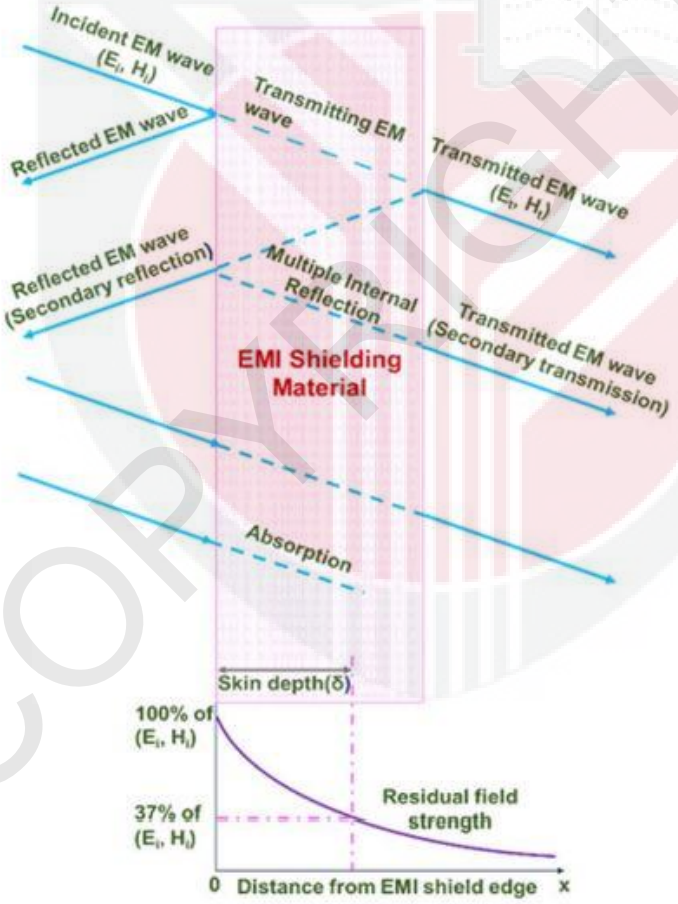


Figure 1.1: Picture shows pictorial depiction of the EMI shielding mechanism and the skin depth of an EMI shielding material.

1.4 Problem Statement

As we all know, EMI can interfere with the operation and functionality of electrical equipment. EMI can cause everything from short-term disruptions and data loss to system failure and death. The major source of worry is the interaction of electrons in metal conductors with the electric field in the radiation that interferes with electronics. Electronics can be harmed as a result of the interference.

As we all can see, many types of shielding material have been discovered until now, including metals, carbons, ceramic, cement, polymers, hybrids and composite. Note that, hybrids and composites are the most common ways to customize the structure to improve shielding efficiency. The major functional materials for offering shielding capabilities are metals and carbons. Ceramics, cement, and polymers are generally ineffective unless coupled with a functional component.

In selecting the various types of materials, this study concerned the enhancement of Fe_3O_4 mixed carbon nanofiller (Graphite and f-MWCNTs) with chitosan polymer composite for EMI shielding. Fe_3O_4 act as metal to increase the value of the dielectric constant and the magnetic permeability. In contrast, the carbon nanofiller increase the electrical conductivity. Many experimental procedures need to be done to prove the effectiveness of the EMI shielding is being enhanced by these materials.

However, chitosan polymer is introduced as a conducting polymer for biodegradable, making them more environmentally friendly. Chitosan is a renewable nature of biomass polymer with low conductivity. These carbon nanofillers are involved in enhancing the electrical properties of the shielding material.

1.5 Objectives

- To prepare the conductive filler with chitosan polymer composite
- To measure the electrical properties and EMI shielding efficiency of the polymer composite.

1.6 Thesis Outline

This study is conducted to observe the effectiveness of EMI shielding using (Fe_3O_4) compound mixed carbon nanofiller (Graphite and f-MWCNTs) with chitosan polymer composite.

Overall, this draft consists of five chapters gathered all together. The first chapter mainly focuses on the history of electromagnetic interference (EMI), the background of the study, the mechanism of electromagnetic interference (EMI) shielding, problem statement, and objective.

In chapter 2, the literature review has been written down based on the past researcher, study on EMI findings and its effectiveness. This chapter explains a lot more theory and the material that has been used in this study.

In chapter 3, all the methodologies for conducting this study are explained.

In chapter 4, the result of the effectiveness based on (Fe_3O_4) compound is presented. Analysis on x-rays diffraction (XRD), conductivity, permittivity and magnetic permeability are shown. The total shielding effectiveness is observed. This chapter also presented the comparison and table obtained from the analysis.

The final chapter, chapter 5, presents the summary of this study. The conclusion and recommendation also have been included for future study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

As the system and electronics have gotten more compatible over the years, a lot of research has been done to understand how the interference is created and the ways to minimize the transmission of the interference to the surround, to overcome the whole new challenges these days.

The origins of electromagnetic interference (EMI) are many and include both artificial and natural sources of interference. The consequences might range from minor interruption and data loss to system failure and even malfunctioning, among other things. Shielding is required to prevent electrical devices from lowering the circuit's performance, particularly electronic devices with modest voltages and currents, which may be disturbed if they are not shielded. EMI shielding materials are available in a variety of forms.

This chapter discusses the theory that supports this research. I will review the prior study that has been done by other researchers in terms of producing good EMI shielding by materials that have been discovered.

2.2 Theory of EMI Shielding

Many theories have been put up in the wake of the prior investigation. Researchers believe that their concept and explanation will yield the best results for the best EMI shielding. As the goal of their research is different, there are several strategies to improve EMI shielding.

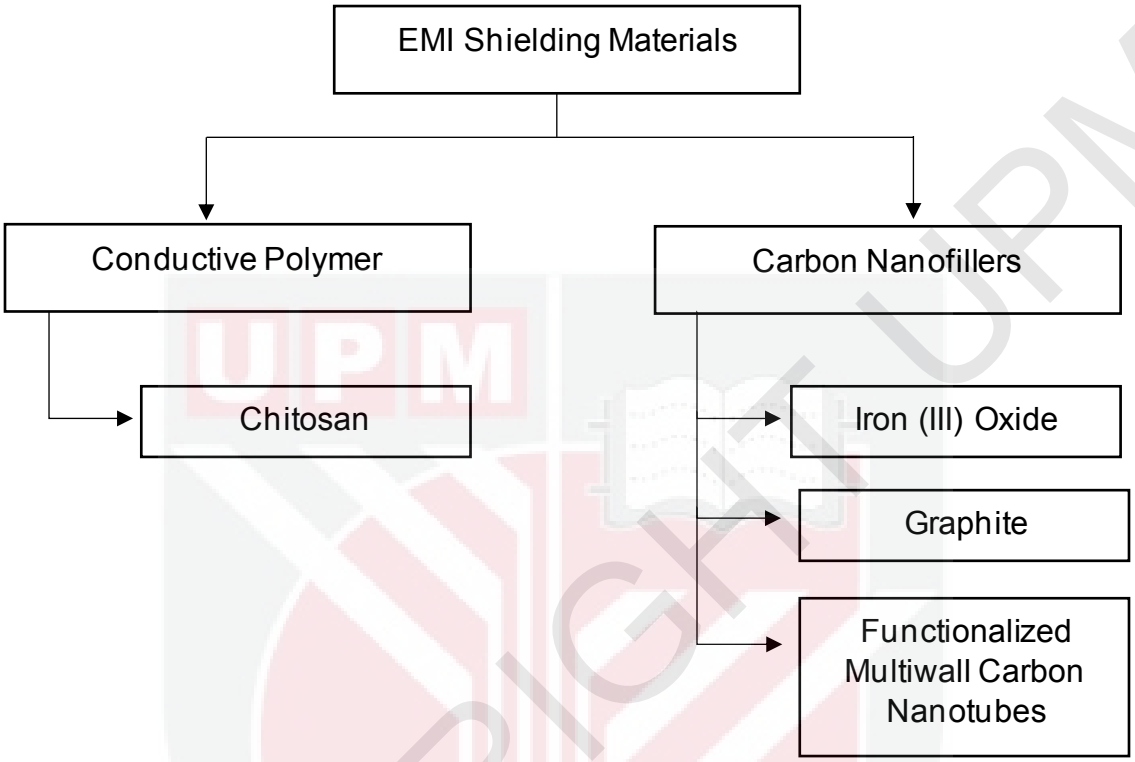
The concept, absorption and reflection of radiation are the primary mechanisms of shielding. Shielding should be effective in preventing the penetration of the radiation.

In EMI shielding, reflection is the most crucial protection mode against electromagnetic interference. According to Chung, for a shield to effectively reflect radiation, it must contain mobile charge carriers (electron or hole) that interact with the electromagnetic fields produced by the radiation. As a result, the shield tends to be electrically conductive. When it comes to EMI shielding, metals are the most popular choice because the presence of excess electrons makes them primarily work through reflection.

Absorption is a secondary mechanism of EMI shielding. In order for the shield to be effective in absorbing radiation, it must have both electric and magnetic dipoles that interact with the electromagnetic fields in the radiation being absorbed by the shield. Metals have a high dielectric constant value; therefore, they may supply electric dipoles with a lot of energy. Iron oxide (Fe_3O_4) or other materials with a great magnetic permeability value may be used to produce high magnetic dipoles in the system.

In addition to reflection and absorption, a shielding system may include multiple reflections, which refer to reflections occurring at various surfaces or interfaces inside the shield. Multiple reflections can cause substantial loss, which can be ignored in cases when the distance between the reflecting surface and the interface is significant (e.g., porous or foam material or material containing filler) as compared to the depth of the skin of the depth. It is necessary for this mechanism to operate in order for the shield to have a wide surface area or interface area.

2.3 EMI Shielding Materials



2.3.1 Chitosan (Ch) as Conducting Polymer

Chitosan is a natural polymer with abundant amino and hydroxyl groups that possesses many good properties such as low cost, chemical inertness, high mechanical strength, high hydrophilicity, good film-forming ability, biocompatibility, and no toxicity. Very recently, chemically modified electrodes and enzymatic biosensors with chitosan support the research of Tan *et al.* (2005).

Chitosan is from renewable resources, and it is obtained from the shell of shellfish and the waste of the seafood industry. It has novel properties such as biocompatibility, biodegradability, antibacterial, and wound-healing activity. Recently, functional biomaterial research has been directed towards the progress of improved scaffolds for reformative medicine as in our daily life. Tissue engineering is an important therapeutic strategy for the present and future medicine. Furthermore, the current revision recommended that chitosan and its derivatives are promising candidates as supporting material for tissue engineering application owing to their porous structure, gel-forming properties, ease of chemical amendment, high affinity in vivo macromolecules, and others at the site, according to Kim *et al.* (2008).

For this research, chitosan is introduced to be the conducting polymer for biodegradability, making them more environmentally friendly. Chitosan is a renewable nature of biomass polymer (Fang Liu *et al.*,2020). However, Ch is low in conductivity at its initial condition (Sathish *et al.*,2021). Therefore, carbon nanofiller is introduced to this research as conductive filler. Carbon nanofiller are involved to enhance the poor electrical, thermal and mechanical properties of the shielding material. Carbon nanofiller are incorporated with the polymer matrices to enhance the mechanical strength, conductivity and permeability, which improves reflection as well as absorption. Thus, a great material of shielding polymer is created.

2.3.2 Iron (III) Oxide, (Fe_3O_4) as Conductive Filler

Compared to all other iron oxides, Fe_3O_4 has been the most thoroughly explored magnetic nanostructure due to its simplicity of production, excellent biocompatibility, superparamagnetic composition, excellent stability, and low toxicity, among others characteristics. In the literature, there are many low-cost preparation techniques for Fe_3O_4 nanostructures that may be discovered. Consequently, magnetite has a wide range of applications in various fields such as magnetic storage systems, food analysis, magnetic resonance imaging (MRI) and EMI applications, particularly in the field of magnetism, where it is high magnetic moment is hugely helpful.

Fe_3O_4 nanostructures also have a cubic inverse spinel structure of two Fe^{3+} and one Fe^{2+} valence states, with oxygen forming a closed-pack structure that is similar to that of a cubic spinel structure. It is a type of half-metallic substance that is absolutely necessary, in which electron hopping occurs between the Fe^{2+} and Fe^{3+} ions. As a result, the remarkable magnetic and dielectric characteristics of the Fe_3O_4 nanostructure make it a promising alternative for magnetic and electric attenuation components in the EMI shielding mechanism. Fe_3O_4 might be regarded an excellent alternative for energy-related applications, such as electromagnetic interference (EMI).

Magnetic nanostructures have captured the interest of many researchers in recent years, particularly in the generation of high microwave absorption materials. Their cheap cost and ease of synthesis, besides the great biodegradability and biocompatibility features of iron and other constituents, have considered them appealing materials in comparison to other transition metals in terms of possible uses in EMI shielding. Iron is one of the most potential alternatives

for a variety of applications, including catalysis, microwave absorption, water pollution control, conductive polymers, and a variety of other fields of study.

Iron is well-known for being the highest-temperature ferromagnetic material having the highest saturation magnetism at ambient temperature. The design of Fe-based nanostructures has increased significantly. This is due to the numerous benefits that nanostructured materials have over bulk materials, such as a high aspect ratio, decent porosity, and a strong magnetic moment (superparamagnetic behavior). Alternatively, pure Fe can be observed in the body-centered cubic (BCC) structure or the face-centered cubic (FCC) configuration.

Since the high conductivity of Fe elements and the strong skin effect at GHz high frequency, it has been observed that oxide shells not only protect Fe from oxidation in the presence of air but also prevent the forefront diffraction gratings as previously observed in pure Fe sheets that show negligible microwave absorption. This is the primary reason why Fe structures have only been included in a huge number of studies.

More significantly, iron may be found in various forms, sizes, and dimensions, including nanowires, nanoparticles, nanorods, nanotubes, hollow fibres, microspheres, and dendrite-like microstructures, all of which increase the reflection loss yet decrease the conductivity of the material. For the purpose of overcoming the mentioned difficulty, iron such as ferric oxide, magnetite, and ferrous oxides (FeO) have been recommended for use in the development of more efficient microwave absorption materials due to their ability to function as semiconductors (highly resistive).

EMI-preventing materials with higher permittivity are made possible by anchoring transition metal oxides such as ZnO, MnO_2, and SnO_2 with an iron component. This means that by utilizing the double attenuation process, the combination of these oxides with Fe components may reduce both dielectric and magnetic losses in materials, resulting in improved

microwave absorption performance. After all, the usage of Fe/metal oxide composites is constrained by challenges in processing, agglomeration during synthesis, and poor dispersion, all of which are significant disadvantages.

2.3.3 Graphite (Gr) as Conductive Filler

Recently, nanostructures such as graphene composite films and foams have received great interest for their unusual two-dimensional structures and outstanding electrical conductivity. The shielding efficacy of graphene/polymer composite materials is largely sensitive to filler loading, with a general requirement for the embedded graphene nanosheets to create good conductive links as a condition of use.

Firstly, graphene oxide is a non-conductive material as the absence of percolating pathways between carbon cluster, but it can be restored by varying their synthesis and processing (Rao *et al.*, 2018). There are different methods to synthesize GO for conductivity, such as thermal reduction and chemical reduction which have been generally accepted to restore electrical conductivity. It was believed that as the EMI shielding effectiveness rises with the high amount of electrical conductivity, the primary mechanism is reflection, whereas absorption is the secondary mechanism (Rao *et al.*, 2018)

In 2017, Zhou E et al. proposed a post doping method for large-area potassium doped graphene films (GF-K). Surprisingly, the electrical conductivity of GF-K began to approach benchmark metals. Study shows, that the shielding effectiveness (SE) increased from 70-85 dB for graphene films to over 130 dB for GF-K. It has previously been recorded as one of the best shielding effectiveness (SE) with the thickness of GF-K was only 31 μm.

Recent research has focused on fabricating conductive composite films made of graphene and poly (ethylene/vinyl acetate) that can be used to reduce the effective thickness of

EMI shielding. Electrical conductivity of 2.5 S cm^{-1} in the composite films, identical to that of the foams mentioned above, could be produced with films as thin as 0.36 mm in effective thickness, resulting in the same shielding performance (23-27 dB) as the foams. Thin films/papers appear to be a more beneficial configuration for graphene-based composites than thick films/papers because they have a smaller effective thickness, high mechanical flexibility, and are easier to produce, allowing for a broader range of application possibilities.

2.3.4 Functionalized Multiwall Carbon Nanotube (f-MWCNT) as Conductive Filler

According to Sun et al., carbon nanotubes (CNTs) are an effective electromagnetic interference shielding material due to their outstanding electrical conductivity, low weight, and corrosion resistance. Sun et al. prepared graphene foam/MWCNT into the composites in proportions of 2.7/2 wt%, resulting in an EMI shielding effectiveness value of 75 dB, which is 25 dB greater than the value of the composites made without MWCNT.

In addition, Jiantong et al. found at 2017 that the electrical conductivity of the EP/f-MWCNT nanocomposite increased significantly (2.32×10^{-10} S/cm to 1.04×10^{-5} S/m) when the f-MWCNT concentration increased from 0.56 vol% to 2.85 vol% of f-MWCNTs subsequently. According to these findings, the improved electrical conductivity of the nanocomposites was due to the orientation of the f-MWCNTs.

We can see, composites with a more extensive f-MWCNT loading exhibited enhanced electrical conductivity, implying that a more significant number of free electrons in the composites were available to interact with the penetrating radiation when exposed to the radiation. Anil Kumar et al. also demonstrated that f-MWCNT/PANI nanocomposites (0 to 21 vol% CNT) exhibit greater electrical conductivity than pure PANI due to the creation of a

conducting route between the CNT and the PANI matrix in their study. The enhanced electrical conductivity of nanocomposites significantly improved the predicted EMI shielding, which was previously believed to be poor.

These shows that, the amount of f-MWCNT in nanocomposites enhanced the electrical conductivity and predicted EMI shielding of the composites. It has been proposed to employ functionalized multiwalled carbon nanotubes (f-MWCNTs) in EM shielding materials because of their promising electromagnetic properties, high flexibility, and high electrical conductivity, among other characteristics. According to the researchers, the system, which includes f-MWCNT and results in increased conductivity in an exceptionally thin and flexible matrix, is the primary contributor to the high values of shielding efficacy.

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, the research's methodologies lists were explained. To do this, prior research and previous work on Electromagnetic Interference (EMI) were used as references. These methods were selected and be done in laboratory of University Putra Malaysia (UPM). All the steps enlisted can be carried out through proper procedures in the lab.

3.2 Produce Graphene Oxide

Pre-Oxidized Graphene

3 g of Graphite Powder, 2.5 g of Potassium Persulfate ($P_2S_2O_8$) and 2.5 g of Phosphorus Pentoxide (P_2O_5) are mixed into 12 mL of Sulfuric Acid (H_2SO_4)

↓

Keep into oil bath of 80° C in temperature for four and half hours

↓

Dilute the solution with 500 mL of DI water and left overnight

↓

The mixture is filtered and washed with DI water using 0.22 µm PTFE membrane to remove any remaining acids

↓

Left overnight for drying process then only can proceed with Hummer's Method

The Hummer's method (Graphene Oxide)

The pre-Oxidized Graphene was added into 120 mL of Concentrated Sulfuric Acid (temperature of 0^oC).

↓

Gradually add 15 g of Potassium Permanganate ($KMnO_4$) into the mixture.
Stir the mixture under 800 rpm and must keep it under 20^oC using ice bath.

↓

After that, stir the mixture for 2 hours under 35^oC , then add 250 mL of DI water and put it in ice bath for 2 hours.

↓

Add 700 mL of DI water into the mixture , only then can be added with 20 mL of 30% Concentrated Hydrogen Peroxide (H_2O_4).

↓

The mixture change to yellowish colour indicates that well oxidized Graphene Oxide was produced.

↓

Mixture is filtered and wash with 1:10 Hydrocholric Acid (HCL) to remove metal ions, follow by 1 L of deionised water to remove acid.

↓

Resulting solid is dried and diluted to make GO dispersion.

3.3 Reducing Graphene Oxide to r-GO

Thermal Reduction

Thermal Reduction is a process which use heat treatment totally. This process is known as Annealing Reduction.

Annealing (Thermal) Reduction

Reducing GO by heat treatment

Anneal Graphine Oxide (GO) at temperatures of 100^oC , 200^oC and 300^oC for 1 , 2 and 3 hours.

r-GO are obtained.

Chemical Reduction

Chemical Reduction

Reduce Graphene Oxide using $NaBH_4$ as reducing agent.

Start suspension process with 10mM of $NaBH_4$ and add deionized water into it.

GO solution is added into the suspension after 10 minutes of magnetic stirring at 300 rpm.

Then, resulting mixture is treated with reducing aget by magnetic stirring at 200 rpm for 2 hours.

Inhomogeneous brown suspension is obtained.

Then, the synthesized r-GO solution is filtered by vacuum filtration method.

The mixture is filtered and washed with 1:10 HCL solution (1 Liter) to remove metal ions, followed by 1 L of deionised water to remove acid.

The resulting solid is dried in air and diluted to make r-GO dispersion.

3.4 Characterization: X-Rays Diffraction (XRD)

Technique of X-ray diffraction (XRD) is used to identify whether the material is crystalline or amorphous depending on the structure of the substance. It is standard practice in materials research to use X-ray diffractograms to analyze a crystallographic structure. When doing an XRD experiment, an item is exposed to X-rays and then utilized to determine the intensities and scattering angles of the resultant X-rays. Identification of materials may be accomplished by the examination of their diffraction patterns, which is an extremely popular use for XRD investigations. The XRD technique detects phases and demonstrates how the real structure differs from the ideal structure as a result of tensions and flaws within the material.

X-rays are electromagnetic waves, whereas crystals are atomic structures that are regular in their organization. The electrons in crystal atoms interact with X-rays and scatter them due to this interaction. In the technological world, these phenomena are referred to as elastic scattering, with the electron serving as the scatterer. Spherical waves are generated by a systematic arrangement of scatterers in a circular pattern. As Bragg's law demonstrates, for the vast majority of the time, these waves interfere and cancel each other out (destructive interference), but in a few specific directions, they may assist each other (constructive interference):

$$2d\sin\theta = n\lambda$$

Where,

d= spacing between diffracting planes

θ= incident angle

n= integer

λ= beam wavelength

X-ray diffraction is a non-destructive material analytical technique utilised in a wide variety of industries, including medicine and aerospace. Additionally, it provides information on crystal structures, phases, preferred crystallographic structure (texture), and other structural parameters such as crystallite size, crystalline nature, strain, and crystal defects, among other things, as well as information on crystal flaws. Constructive interference of an X-ray beam scattered at certain angles from each pair of lattice planes in a sample, resulting in an X-ray peak in the spectra, is responsible for the formation of XRD peaks. The molecular placements inside the lattice planes are accountable for defining peak intensities in crystallographic structures.

3.5 Characterization: Two Point Probes (LCR)

LCR meters are measuring devices that measure the impedance of a physical characteristic known as resistance, which is also known as impedance in electrical terms. Z is a quantifier that denotes resistance to the flow of alternating current, and it measures resistance to the flow of alternating current (AC). It may be calculated from the current I flowing through the measurement object and the voltage, V across the measurement target's terminals. The fact that impedance is represented as a vector on a complex plane means that LCR meter may be used to detect the ratio of current to voltage RMS values as well as the phase difference between current and voltage waveforms in real time.

BNC connectors are used on all terminals of LCR meters that use the automatic balancing bridge technique, which is the most common. The shielded coaxial design of the connections dictates how well the measurement and detection signals are protected from extraneous noise. In this setup, two terminals make contact with the measurement target. It has been shown that when the measuring target has a low impedance, measurable values such as wire resistance and contact resistance are significantly affected. During high-frequency and

high-impedance measurements, the stray capacitance between the two cables also contributes to inaccuracy, as the measurement signal goes to both the stray capacitance and the measurement target.

To better understand the behavior of material resistance, it was necessary first to define it in terms of resistivity and conductivity. At room temperature, a material's resistance, inductance, and conductance as a function of the material thickness can be measured at room temperature. With the use of the LCR meter, we were able to get these measurements.

3.6 Characterization: Vector Network Analyzer (VNA)

A network analyzer is an equipment that is used to examine electrical networks in order to discover their properties. Due to the ease with which electrical networks' reflection and transmission can be monitored at high frequencies, network analyzers are frequently used to monitor s–parameters on a network. The amplitude and phase characteristics of a vector network are evaluated using a vector network analyzer (VNA). VNAs are more than just a common type of network analyzer; they are also quite useful. A VNA may also be referred to as a gain-phase meter or an autonomous network analyzer, depending on the context. Network analyzers have a basic design that includes several components such as a signal generator, a test set, one or more receivers, and a display. The majority of VNAs include two test ports, which allows for the measurement of four S parameters in one instrument (S11, S21, S12 and S22). Instruments having more than two ports are available for purchase on the commercial market. A test signal is required by a network analyzer, which can be generated or obtained from a signal generator or signal source. Almost every network analyzer on the market today has an inbuilt signal generator. The high-performance network analyzer is equipped with two different sources.

During testing, the signal generator's output is received by the test set, which then feeds it to the device being tested, and the signal to be measured is received by the receivers. Frequently, it serves to create a reference channel for the wave that has been incident. It is necessary to provide the reference channel to the receivers in a VNA in order for it to serve as a phase reference.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

Chapter 4 discussed the expected results obtained from experiments and characterization that have been carried throughout the research. The discussion will mainly focus on the morphology analysis and structure of the synthesized Ch polymers with different carbon nanofiller. The characterization used in this study are XRD, Two Point Probe (LCR) and Vector Network Analyzer (VNA). There are many interesting and meaningful results are expected to be obtained and discussed comprehensively in this section.

4.2 X-Ray Diffraction

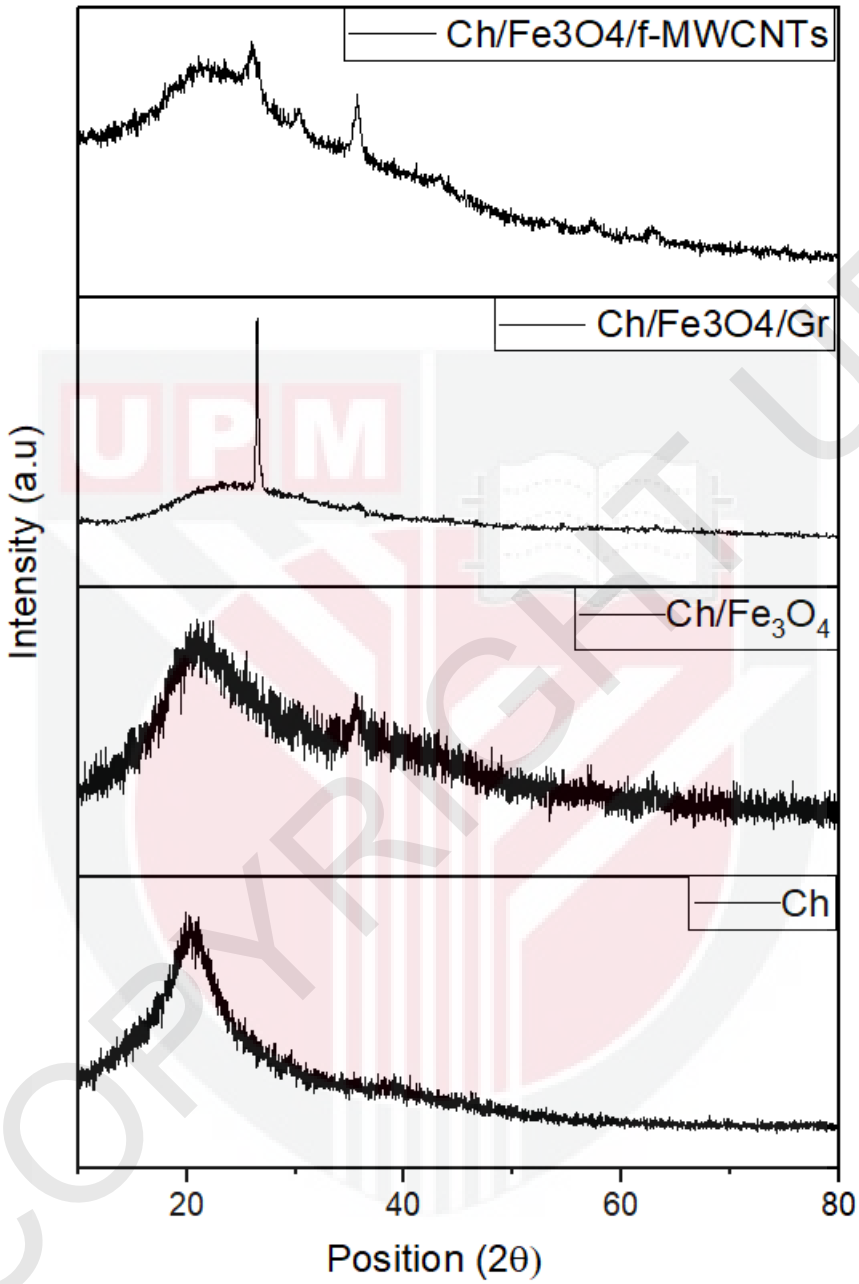


Figure 4.1: X-Ray diffraction pattern for Ch, Ch/Fe_3O_4, Ch/ Fe_3O_4/Gr, Ch/ Fe_3O_4/f-MWCNTs

Figure 4.1 represents the XRD patterns of the polymer composites film employed in this analysis, as shown. According to the data, a peak can be found around 20.9°, which may be attributed to the crystalline domain of the Ch polymer, which is associated with the crystal

within the chitosan membrane, as observed in the data. According to Aziz et al., this peak has also been seen in 2019, where the lattice parameters of the unit cell of crystal associated to θ= 20.9° are as follows: a = 4.4, b = 10.0, c = 10.30 (Å), and β = 90°. It can be seen that the broad peak is located at roughly 2θ = 41°. It has been demonstrated in earlier research that the large peaks within the XRD patterns of pure Ch polymers are caused by inter-chain segment scattering when the polymers are in the amorphous state and that this is due to the inter-chain segment scattering.

In the XRD pattern of Ch/ Fe_3O_4 given above, the six features are as follows: six peaks at 2θ= 10°; 30°; 35°; 44°; and 59°; and 62°; corresponding to their indices (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), (6 1 1) respectively. Shagholani et al. (2015) found comparable findings, which they assign to crystalline magnetite with a spinal structure, which is identical to the one presented here. Calculated using the Debye-Scherrer technique, the crystallite sizes associated with the value of θ=15, 2.53(Å) are 12nm. For the Ch/ Fe_3O_4 sample, an amorphous peak occurred between 20 and 28 at 2θ, is associated with chitosan. This demonstrates that the structure of Ch/Fe_3O_4 has not altered over the period in which the procedure was conducted. The wide nature of the diffraction peaks in the XRD pattern indicated that the nanoparticles had tiny particle sizes, which was supported by the pattern of XRD diffraction.

According to Rajendra's research published in 2015, the diffraction peak appeared at 2 = 26.5°, indicating that the graphite peak has a d-spacing of approximately 3.35Å, a graphite peak feature. The XRD pattern of the graphite doped chitosan suggested the production of a single-phase composite, and the peaks were formed at 26.5°, which indicated the formation of a single-phase composite. Following the doping with graphite, the broad peak at roughly 2θ = 20°, which was caused by the chitosan, diminished in intensity, confirming that graphite has been doped on the surface of the chitosan particle(s). The presence of a prominent graphite peak in the GDCC spectrum, combined with a small chitosan peak, demonstrated that the

integration of graphite into the matrix was successful and successfully provided support for the chitosan. As shown above, the graphite structure in the Ch/ Fe_3O_4 /Gr sample exhibits polycrystalline features.

The X-ray diffraction (XRD) spectra of MWCNTs reveal the crystal structure of the material, with seven characteristic peaks in 2 = 30.54º, 35.87º, 43.65º, 54.03º, 57.61º, 63.12º, 74.74º that may be ascribed to the d(220), d(311), d(400), d(422), d(511), d(533). When looking at the XRD pattern of Fe_3O_4, the peak broadening of Fe_3O_4 reveals that the produced magnetite crystallites in the composite are significantly tiny (13.02 nm), which is consistent with the Scherrer equation's prediction. The XRD curve of Fe_3O_4 /MWCNT nanocomposites clearly shows diffraction peaks ascribed to both acid-treated MWCNTs and nanocrystalline Fe_3O_4, demonstrating the presence of MWCNT structure and Fe_3O_4 nanoparticles, respectively (Safari & Gandomi, 2014).

4.3 Electrical Properties

4.3.1 Bulk Resistance and Conductivity

Sample	Filler Concentration (w/v %)	Thickness (mm)	$R_B(\Omega)$	Conductivity (S/m)
Chitosan	-	0.000126	4.45×10^3	3.0×10^{-7}
Fe_3O_4/Ch	0.25	0.000209	2.95×10^3	9.0×10^{-7}
Gr/Fe_3O_4/Ch	0.5	0.000164	2.01×10^3	1.04×10^{-6}
f-MWCNTs/ Fe_3O_4/Ch	0.5	0.000137	7.58×10^2	2.3×10^{-6}

Figure 4.2: Table of Conductivity

As shown in figure 4.2, all the samples have been analyzed for conductivity. First of all, to obtain this result I had set up an experiment using LCR meter in the laboratory. After the machine had been calibrated properly, the sample had been put between the two probes of LCR

meter. All the sample's results were collected to further Nyquist Plot graph to obtain R_B value from the intersection between semicircle and residual spike.

R_B value for Ch found to be the highest value and f-MWCTNs/Fe_3O_4/Ch have the lowest value. By using the conductivity formula, all the samples had been calculated for its conductivity as shown by figure 4.2. A high value of conductivity materials must be correlated to high amount of free moving electron. As per result, we can see f-MWCTN/Fe_3O_4/Ch has the highest value of conductivity due to its properties of having the most amount of free moving electron in its structure.

The contribution of f-MWCNTs is way more effective in increasing conductivity rather than Fe_3O_4 filler. We can see the contribution of Gr is lower compared to f-MWCNT for the Chitosan. The difference by the contribution is 7.4×10^{-7} S/m while f-MWCNT is 2.0×10^{-6} S/m. Its obvious the contribution of f-MWCNT offered for conductivity is very high compared to the Gr. All films have in range of 0.120mm to 0.210mm thickness.

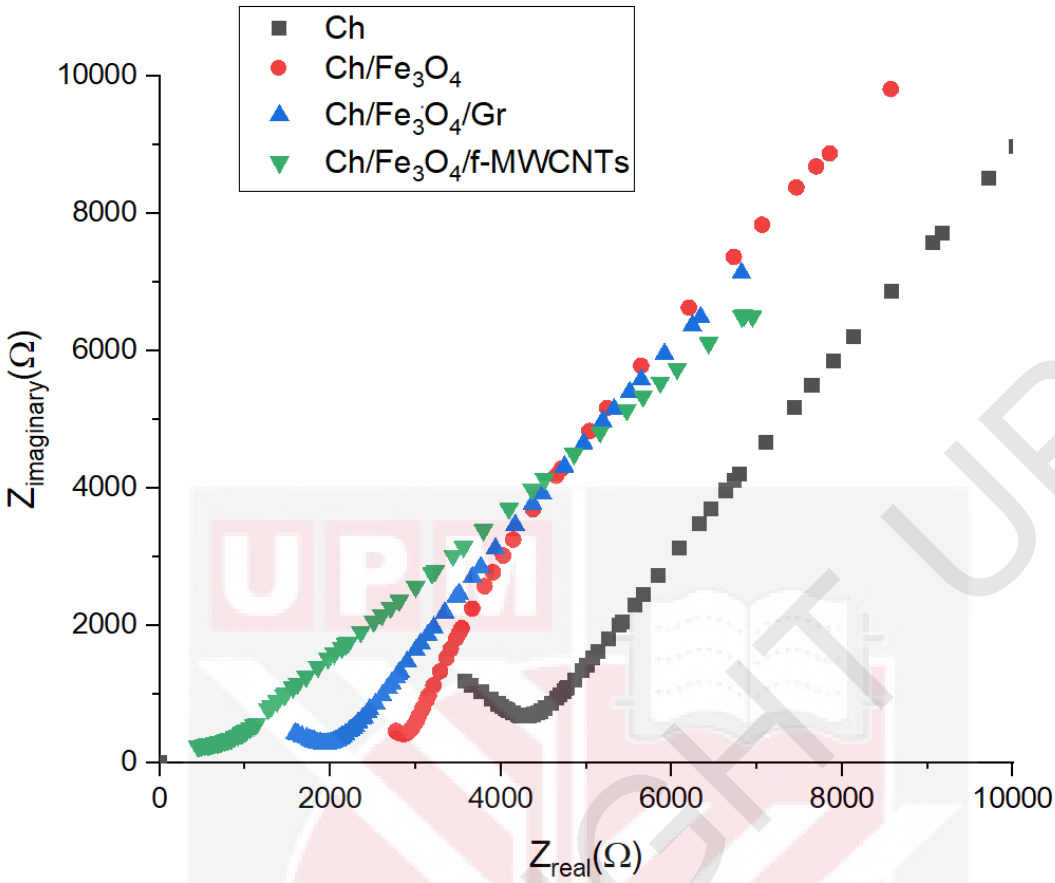


Figure 4.3: Impedance plot

All samples tested with 0.25 wt% to 0.5 wt% nanofiller were measured between 10 Hz and 20 GHz at room temperature, as shown in Figure 4.3. There was a semicircle with a residual spike. It was determined that the spike was caused by electrical contact between the electrode and the sample. As a general rule, the filler begins to agglomerate at more significant concentrations, increasing the interfacial impedance. It was found that when the f-MWCNT filler was added to a polymer matrix, the bulk resistance of the material decreased. According to the percolation theory, polymer conductivity can be improved by increasing filler concentration. Furthermore, f-MWCNT filler was used to increase the charge carrier's conduction channel in the network connection.

4.3.2 Dielectric Properties

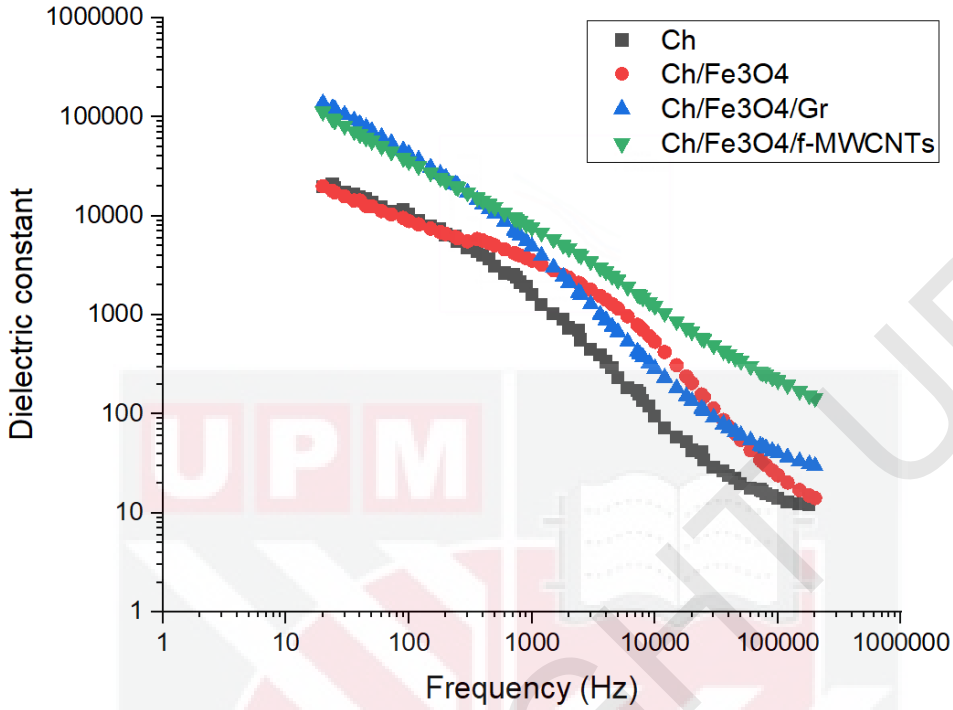


Figure 4.4: Dielectric constant

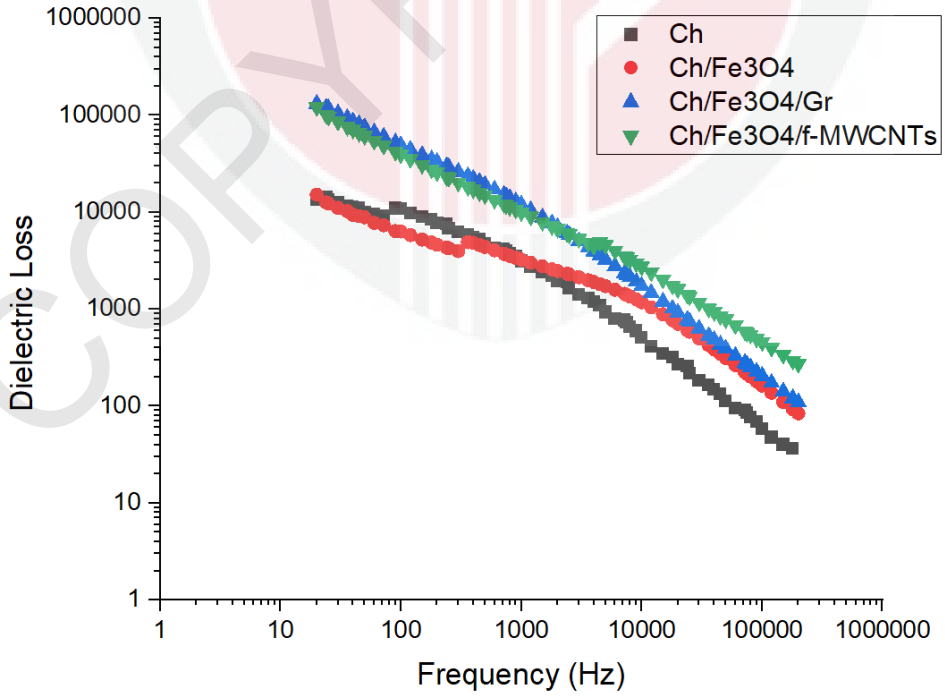


Figure 4.5: Dielectric loss

In Figures 4.4 and 4.5, the dielectric constant and dielectric loss as a function of frequency is depicted for all of the samples used in this investigation. The analyzed samples exhibited a frequency dependence, with the value dropping as the frequency increased. This correlated to the electrode polarization effects, showing that space charge effects occurred at a low frequency, consistent with the results. As the frequency of the samples rose, the dielectric loss in the samples reduced. The dielectric constant of f-MWCTNs/ Fe_3O_4 /Ch was found to be greater. The more the dielectric loss was caused by dissipation through the passage of charge in an alternating field, the easier it was to measure.

4.1 Shielding Effectiveness

4.4.1 Absorption Shielding Effectiveness

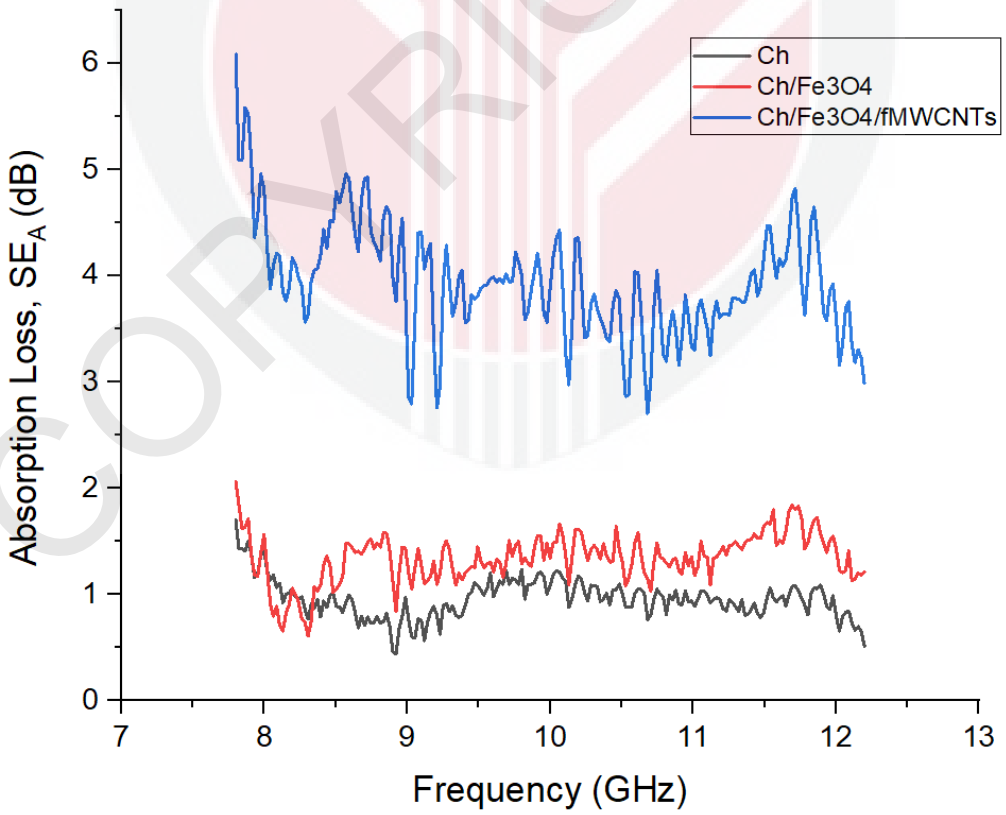


Figure 4.6: Absorption Loss, SE_A (dB)

Figure 4.6 shows the result due to absorption (SE_A) of all samples. Ch has the lowest SE_A compared to all the samples, while Ch/Fe_3O_4/f-MWCNT has the highest SE_A. The SE_A value was observed to increase with conductive nanofiller.

4.4.2 Reflection Shielding Effectiveness

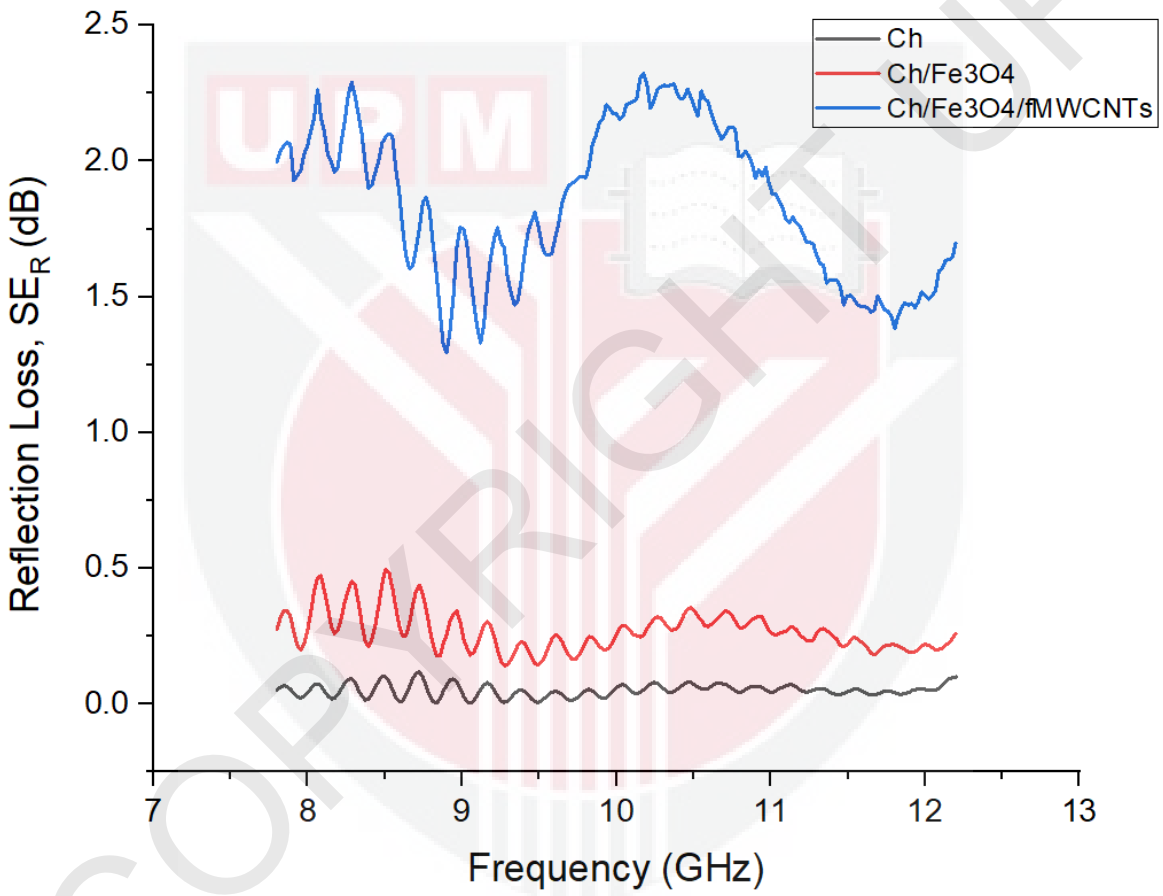


Figure 4.7: Reflection Loss, SE_R (dB)

Figure 4.7 shows the SE results due to reflection (SE_R) of samples. It was observed that Ch/Fe_3O_4/f-MWCNT produced the highest SE_R, while Ch is the lowest.

4.4.3 Total Shielding Effectiveness

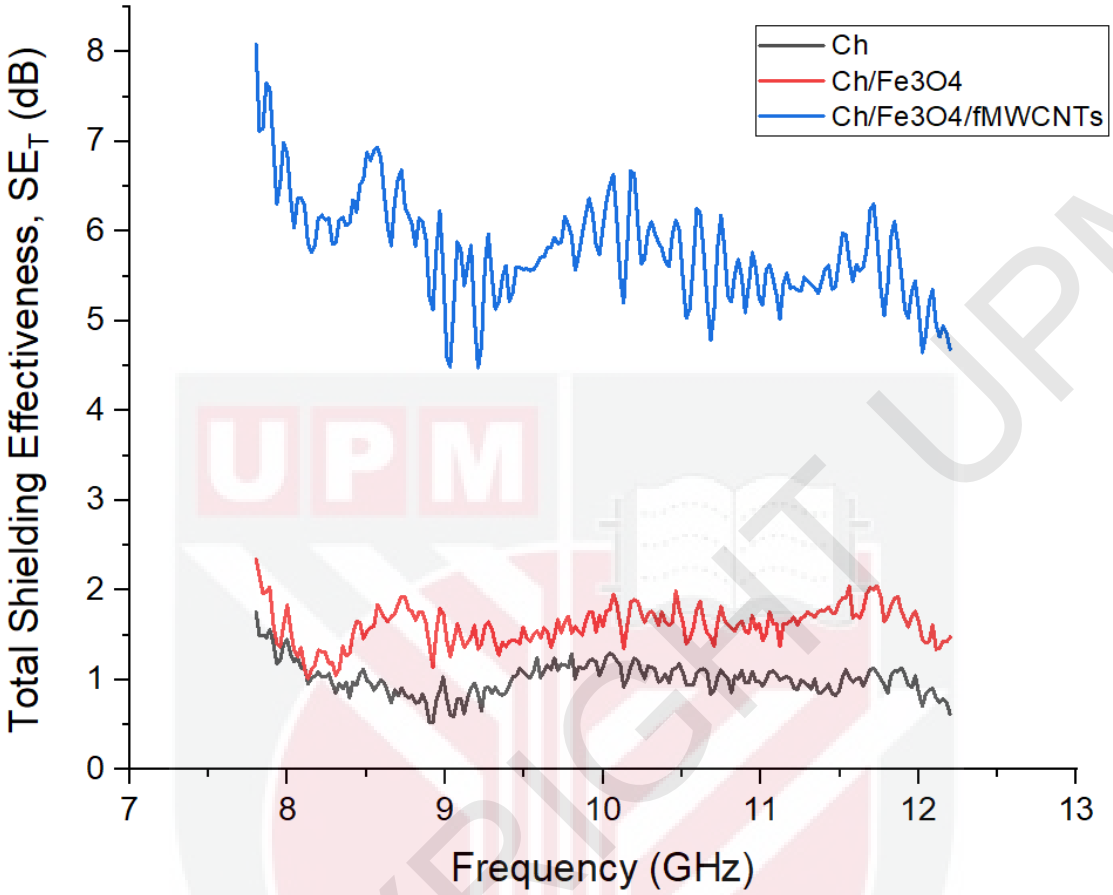


Figure 4.8: Total Shielding Effectiveness, SE_T (dB)

The total shielding effectiveness for samples is shown in Figure 4.8. Ch as expected, showed the lowest SE_T, while conductive nanofiller were proved to enhanced the shielding effectiveness. Ch/Fe_3O_4 produced a slightly better total shielding rather than pure Ch, but with f-MWCNT nanofiller produced the highest enhancement to the Ch. It is proved that conductive nanofiller will enhanced the shielding effectiveness while f-MWCNT gives the biggest attribution in this whole study.

CHAPTER 5

CONCLUSION

5.1 Introduction

This chapter discusses the overall finding of this study. In addition, the recommendation for improving shielding effectiveness through these carbon nanofillers type are also in this chapter.

5.2 Conclusion

Reflection, absorption, and multiple reflections are the three primary methods of attenuating EMI. In order for the shield to be effective at reflecting radiation from its surface, the shield must contain mobile charge carriers that can interact with the incoming electromagnetic field. As a result, the shielding material is more likely to be electrically conductive than other materials.

Absorption is correlated to the presence of electric and/or magnetic dipoles in the shielding material, which engage with the electromagnetic field when the shielding material is exposed to an electromagnetic field. A multiple reflection is a set of reflections that occur at various surfaces or interfaces inside a shield and are often observed in systems that have a lot of fillers with a lot of surface area and thus have a lot of interfacial area in the overall structure.

To summarize this study, all the characterization had been done for all 4 samples which is pure CH, Fe_3O_4/CH, Gr/ Fe_3O_4/CH, f-MWCNT/Fe_3O_4/CH. In theory, f-MWCNT has the highest shielding effectiveness while Ch has the lowest. With all the

characterization that have been done, it is proven that carbon nanofiller enhance the shielding effectiveness while reinforce the polymer composite. Observations have shown that the presence f-MWCNT improves electrical conduction through the carbon nanofiber network. Studies using f-MWCNT reinforced carbon nanofibers utilised in hybrid or multiscale architectures have revealed similar findings in the past, which are consistent with our observation. If we look at the matter from the standpoint of higher EMI SE performance, carbon-based fillers are chosen over metal fillers due to their large aspect ratio and low density as compared to metal fillers

5.2 Recommendation

There are numerous factors will influence the shielding effectiveness in the future. A various of parameters can be used to optimize the total shielding effectiveness of one's material. To increase the effectiveness, numerous factors must be considered, including the type of samples, the thickness of samples, the techniques/procedure, the theory taken from the prior researches, the material used for sample and etc. Additionally, there are a lot of biodegradable polymers such as Polylactide (PLA), Poly-butylene adipate terephthalate (PBAT), Epoxy etc. that can be used in to obtain a high shielding application. Despite the fact that biopolymer does not exhibit electrical conductivity, some of them are brittle and cannot be used in high-impact applications, they might be made more electrically conductive by modifying their insulating characteristics using a variety of nanofillers for use in specific sectors. These should be our main point of interest in the future study to investigate the applicability of various biodegradable polymer for EMI shielding application.

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