Development and Assessment of Nano-Composites for Electromagnetic and Microwave Absorbing Properties – A Computational Approach



BY

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Dedications

Dedicated to.....

Muhammad Athar My Late Father

Anwar Sultana

My Late Mother

Assad Ullah My Husband

and

My Family



Statement of Originality

I hereby declare that my thesis work titles "Development and assessment of nano-composites for electromagnetic and microwave absorbing properties – A computational approach" is carried out by me under the supervision of Dr. Fouzia Malik at Research Center for Modeling and Simulation (RCMS) in National University of Sciences and Technology (NUST). I solemnly declare that to the best of my knowledge, this is my original work and it contains no material which has been accepted for the award of other degree in my name, in any other university. Also no material previously published or written by other person has been included in this except where due reference has been made to the previously published work.

Ayesha Tazeen

MS Computational Sciences & Engineering

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List of Abbreviations

EMR	Electromagnetic Radiations
MAMs	Microwave Absorptive Materials
MOF	Metal Organic Framework
SWCNTs	Single Walled Carbon Nanotubes
MWCNTs	Multi Walled Carbon Nanotubes
CST	Computer Simulation Technology
PU	Polyurethane
EP	Epoxy
SE	Shielding Effectiveness
RL	Reflection Loss
EMI	Electromagnetic Interference
EMT	Effective Medium Theory
PEC	Perfect Electric Conductor

Abstract

Microwave absorptive materials (MAMs), are gaining great attention due to their demand in aerospace, electronics, medical and military fields. These materials shield microwaves by absorption or reflection of the radiations. In the present study, the electromagnetic parameters of materials are determined based on the physical principle of interaction between electromagnetic field and the electromagnetic medium. Herein; polyurethane (PU) and epoxy nanocomposites *i.e.*; PU/SWCNT, PU/MnO₂, Epoxy/SWCNT and Epoxy/MnO₂ are designed and tested for their microwave absorption behavior. Maxwell Garnett effective medium theory is used to calculate the effective permittivity of the 0.4% samples of nanomaterials. Then these composite materials are modelled to find out the scattering parameters (S-Parameters) in X and Ku band region (8-18GHz) of microwave energy. The Reflection Loss, total Shielding and percentage absorption are calculated for all these samples. In X-band region, 5mm thick sample of 0.4% Epoxy/MnO₂ composite showed -20.6dB reflection loss (RL), which is the highest of all the four composites. It is important to note that $Epoxy/MnO_2$ composite has the highest values of real part of effective permittivity, as calculated by Maxwell Garnett effective medium theory. That may be the reason for enhanced microwave absorptive property of this composite. As far as SWCNTs composite concerns, 40% PU/SWCNTs composite provides maximum R_L value of -14.8dB in X-band region. The high concentration of SWCNT, and small length can be responsible for low reflection loss of SWCNTs composites.

Whereas, in Ku band region, again Epoxy/MnO₂ composite with 4mm thickness furnished highest value of reflection Loss, *i.e.* -21.3dB. It was found that both SWCNTs and MnO₂, the nanofillers, use reflection phenomenon for microwave shielding. The improved microwave absorbing properties suggested promising application of Epoxy/ MnO₂ nanocomposites in high-performance microwave absorber. Outcomes of this research can lead to futuristic nature of nanocomposites to be used as microwave absorbents for military applications.

Chapter 1

Introduction

Introduction

1.1 Background:

Electromagnetic radiations (EMR), for centuries, remained center of attention for researchers. James Clark Maxwell (1865), for the first time proposed the light as electromagnetic phenomenon and introduced the relationship between electric and magnetic fields to charges and currents, as a set of four mathematical equations. Heinrich Hertz (1887), later proved the existence of these radiations through his experiments. Electromagnetic radiation consisting of oscillating electric and magnetic field, is the form of energy that can travel through space without any supportive medium. These radiations constitute a spectrum, the electromagnetic spectrum – that has various frequencies associated with it. Different spectral regions from low to high frequency range include radio waves, microwaves, infrared, visible, ultra-violet, X-rays and Gamma rays.

Microwave radiations are important part of electromagnetic spectrum with relatively shorter wavelengths than radio waves. They are widely used in telecommunication industry, radar signaling, food processing etc. Continuous exposure to microwaves can pose serious health threats to mankind. Also, their use in radar signaling make the aircrafts vulnerable in enemy's territory.

So, along with finding new applications of microwave radiations, researchers are also working to find techniques or materials that can minimize the harmful effects of microwave radiations. Microwave absorptive materials (MAMs), are point of special interest to material scientists in this regard. [1-3].

1.2 Microwave Radiations:

Microwave radiations are the non-ionizing part of electromagnetic spectra having wavelength shorter than radio waves and larger than infrared radiation with frequency range between 300MHz to 300GHz and the wavelength between 30cm to 1mm. Microwave frequency region is assigned various ranges or bands by IEEE (Institute of Electrical and Electronics Engineers), as it is the main communication frequency region. The same frequency range is used by NATO and EU. [4]

1.3 Microwave Frequency Bands:

Microwave frequency bands as proposed by IEEE and some of their applications are presented in the table. The regions have important military and non-military applications.

Designation	Frequency Region	Applications
L Band	1-2 GHz	GPS, Mobile Phones
S Band	2-4 GHz	Microwave ovens, Mobile Phones
C Band	4-8 GHz	Telecommunications
X Band	8-12 GHz	Radar, Satellite communications
Ku Band	12-18 GHz	Molecular Rotational Spectroscopy
K Band	18-26.5 GHz	Radar, Satellite Communication
Ka Band	26.5-34 GHz	Satellite Communication, Spectroscopy
Q Band	33-50 GHz	Satellite Communication
U Band	40-60 GHz	Satellite Communication
V Band	50-75 G Hz	Scientific Research
W Band	75-110 GHz	Millimeter wave radar research
F Band	90-140 GHz	Satellite television broad casting
D Band	110-170 GHz	Millimeter wave scanner

Table 1.1: Microwave Frequency bands and Their Applications [5]

1.4 X – Band Region (8.20-12.40 GHz):

X-band has assigned the frequency range of 8 - 12 GHz by IEEE with wave length from 2.5cm to 3.75cm. Its significance is its use as radar frequency range. Due to shorter wavelengths it has higher power and can identify and detect the target easily.



Fig 1.1. Microwave Frequency Bands [6]

1.5 Ku – Band Region (**12.4 – 20 GHz**)

This region of microwave spectra is mainly used for setellite communication. Having high frequency this range is used to achieve greater bandwitdth than lower frequency regions.[7]

1.6 Microwave Technology

Microwave technology has revolutionized the whole wireless communication industry. Higher frequencies of microwave radiations, results in reduced antenna size make them good candidates for high capacity satellite and terrestrial communications. As microwave radiation, supports larger bandwidths hence it transmits higher data with low power consumption. Therefore, it is used in personal communication systems (PCS), wireless local area networks (WLANs), blue tooth, to name the few.

Various other useful impacts of microwave technology are in electronics, navigation, radar systems, food industry, spectroscopy, research applications, etc. [8,9,10]

Numerous applications of microwave technology have made these radiations a prominent member of electromagnetic spectra. However, we can't overlook the disadvantages of microwave radiations associated with it as well. Interference: When almost every electronic gadget around us is utilizing electromagnetic radiations there is always a chance of interference among the devices. For example, use of microwave oven in the vicinity of a Wi-Fi can interfere with its signals, and result in weakening the Wi-Fi signals in the phone or the laptop. In hospitals, where different life saving procedures and instrument uses electromagnetic radiations possibility of EMR interference is always there, which can lead to some life threatening situation for a patient. [11]

Health related problems: We ourselves are always exposed to some form of electromagnetic radiations, of which microwave radiation is a prominent part. This can lead to various health problems. These effects are of two types: Thermal – high frequency, high power associated with heat; Non-Thermal – Low frequency, low power. Various papers discuss the impact of continuous exposure of microwave radiations to central nervous system of human being – causing many long term and short-term brain problems. Many other medical conditions like reproductive, endocrine, cognitive and even cancer have been reported in various studies. [12,13,14,15].

Radar Signaling: Microwave are important in radar signaling technology, as these are used to detect the aircrafts, ships, missiles etc. However, this same technology become damaging when one wants to get edge in wars by attacking the enemy's territory - enemy detect the warheads beforehand. [16]

Electromagnetic Pollution: Electromagnetic pollution is now considered as the fourth major environmental pollution factor after air, water, soil and noise. But the major contributing part in this pollution is microwave spectral region, as microwave technology is used in almost all the communication devices like wireless, mobile phones, Wi-Fi signals, radar signals etc.

All these problems associated with extensive use of microwave technology make it clear that we have to develop the materials that can act as a shield from these harmful radiations from electromagnetic spectrum. [17,18,19]

1.7 Microwave Absorbing Materials:

Microwave absorbing materials (MAMs) are central to research in material's domain due to their importance in stealth technology and power of pollution control caused by electromagnetic radiations. They can dissipate microwave radiations by converting them in heat. As microwave technology is now used in almost every field of life as discussed earlier, the extensive variety of materials ranging from inorganic to organic origin and hybrid – are available commercially. To shield microwave radiations, MAMs work either as good reflectors or absorbers of microwave energy. Material scientists are working to get even more effective materials that can have strong shielding power to microwaves, cheap and having good mechanical strength.

1.8 Types of Microwave Absorbing Materials:

In general, MAMs are divided into following categories on the basis of their chemical nature: [20]

- 1. Inorganic microwave absorbing materials
- 2. Organic microwave absorbing materials
- 3. Hybrid microwave absorbing materials

i. Inorganic MAMs:

There are many inorganic microwave absorbing materials that are commercially available. The most important ones are

- i. Iron based absorbing materials Ferrites, Carbonyl Iron, polycrystalline iron fibers are some of them.
- Carbon based absorbing materials Carbon black, Graphene, Carbon nanotubes, Carbon nano rods etc.
- iii. Ceramic based absorbing materials Zirconia, Silicon Carbide, Alumina etc.

ii. Organic MAMs:

Conducting Polymers are the promising new absorbing materials due to their electrical properties. The examples are Polyaniline, Polypyrrole, Polyazomethine esters are few examples of such polymers.

iii. Hybrid MAMs:

These multifunctional materials are the combination of organic and inorganic materials, to generate the desired properties. These are either, chemically combined forming a single structure as metal organic framework (MOF), or blend of the two materials (organic/inorganic) formed by physical methods e.g. nano composites. MOFs can also be used as polymer nano composite when blended with polymers as a filler. [21,22]

1.9 Nano – Composites:

Nano composites are multicomponent, multiphase non-gaseous hybrid materials, in which at least one of the phases has at least one dimension less than 100 nm. These materials can be formed by conducting organic polymers used as matrix and the inorganic nano particles such as graphite, gold, carbon nano tubes, graphene, tungsten sulphide etc. dispersed in the matrix as a filler. The small quantity of nanofiller enhance various properties of polymers tremendously, including microwave absorption properties. Nanocomposites can be made without polymer as matrix in them as well. [23,24]

Classification of Nano-composites:

Nano-composites can be classified on the basis of their nanoscale dimensions, dispersion medium or the chemical nature of the nanofiller in the matrix.

i. Classification on the basis of nanoscale dimensions:

Depending upon the number of nanoscale dimensions of the nano fillers, the nano composites are classified into following categories:

- a. Zero dimensional (0D) nanocomposites which has all the dimensions within 100nm
 e.g. Nanoparticles, Bucky balls
- b. One dimensional or 1D nanocomposites having two dimensions within nanoscale,
 e.g. fibers, nanotubes
- c. Two dimensional or 2D nanocomposites- with at least one dimension in nanoscale e.g. platelet, graphene, metal dichalcogenides

d. Three dimensional or 3D nanocomposites –with none of the dimension is confined in nanoscale e.g. nanoparticle dispersions, bundles of nanowires. [25, 26]

ii. Classification on the basis of dispersion medium:

On the basis of dispersed matrix and dispersed phase materials the nanocomposites can be classified into two basic categories:

1. Non-polymer based nanocomposites:

The nanocomposites which don't use polymer as a matrix are non-polymer nanocomposites. These may be further classified as:

- a. Metal-based nanocomposites
- b. Ceramic nanocomposites
- c. Ceramic-ceramic nanocomposites

2. Polymer based nanocomposites:

These have the filler material dispersed in polymer matrix. It is important branch of nanocomposites, as very small quantity of nanomaterial enhances the mechanical, thermal and absorptive properties of bulk matrix medium which is polymer. These can be further categorized as: [27]

- a. Ceramic / Polymer nanocomposites
- b. Inorganic / Organic polymer nanocomposites
- c. Inorganic / Organic Hybrid nanocomposites
- d. Polymer / Layered Silicate nanocomposites



Fig 1.2. Classification of nanocomposites [28]

iii. Classification on the basis of nanofiller:

Depending on the chemical nature of the filling material, nanocomposites can be classified into various types. Some important are:

a. Carbon based nano composites:

These composites have revolutionized the whole stock of already present microwave absorptive materials. Only small quantity of Carbon nano particles in polymers, improve different properties of a polymer - converting otherwise non-absorptive material to a good microwave radiation absorber. The materials may include: Carbon nanotubes (CNTs), Carbon fibers (CF), Graphene etc. [29 - 31]



Fig 1.3. Carbon Nanomaterials [32]

b. Carbide based nanocomposites:

Silicon Carbide (SiC), is the foundational nanoparticles in this class. Other materials like Al/SiC, Ni/SiC etc., derived by SiC has shown good absorbing properties. [33]

c. Oxides based nanocomposites:

These are important nanomaterial with respect to their response in GHz frequency range. Few examples are: Iron Oxide, Titanium Oxide, Manganese dioxide, Silicon Oxide, Graphene Oxide, Boron Oxide etc. [34 - 36]

d. Sulphide based nanocomposites:

Metal sulphide are considered as materials that produces relatively high reflection losses, hence they are important microwave absorptive materials. Important examples are: Molybdenum Sulphide, Tungsten Sulphide, Copper Sulphide, Manganese Sulphide. [37, 38]

i. Metal / Alloy based nanocomposites:

Metals and their alloys, are magnetic materials. These are used in polymer matrix as nanoparticles to achieve high absorption properties. Most important metals are Fe, Ni and Co etc., and their alloys like Fe/Ni alloy, FeSiAl alloy, Ni powder, Carbonyl iron etc. [39 - 41]

ii. Metal – Organic Frameworks:

These porous, high-surface area materials are relatively new. They are made up of inorganic metal ions linked with organic ligands to form cage like structure. These hybrid materials have shown promising results as microwave absorbers in recent studies. [43]



Fig. 1.4. Metal Organic Frameworks [43]

Chapter 2

Literature Review

Literature Review

Recent Progress of Nanomaterials for Microwave Absorption:

Microwave absorbing materials (MAMs), has gained immense attention in the past few decades due to their applications in stealth technology for military purposes and in telecommunications (Joseph & Sebastian, 2013). Material scientists have studied many different materials in an attempt to get better micro wave absorbers to minimize the undesirable effects of microwave radiations. The discovery of nanomaterials revolutionized the world of materials including MAMs. Many nanomaterials have so far been synthesized and tried as potential MAMS. The process is not limited to experimental work. Different modelling techniques have been used to produce new materials and their absorption capabilities tested in different microwave frequency ranges computationally. Computational optimization techniques have allowed researchers to develop low cast multifunctional microwave absorptive materials with greater mechanical strength. This combination of experimental and computational techniques opened new doors in the development of microwave absorptive materials. [44].

2.1 Effect of Concentration and sample thickness on absorption

Hussein, Jehangir et al. (2020) prepared the composite of functionalized multi-walled carbon nanotubes with different percentage concentration of Cobalt Oxide, Iron Oxide & Cobalt-Iron Oxide with polyurethane. The study involved the microwave absorption capability of nine samples between 5-50GHz. They reported the maximum reflection loss of -45dB for 20% CoFe functionalized CNT/PU composite between 15-20 GHz frequency. [45]

Singh, Kumar, and Singh (2020) reported the enhanced microwave absorption of SiC nanocomposites with small addition of SWCNTs in Ku band of electromagnetic spectrum. They reported a reflection loss of -37.11 dB at 14.23GHz with the sample thickness of 2.5mm. [46]

Zhu (2019) in his work assessed different effective medium theories to construct the polymer based nanocomposite comprised of Barium Strontium Titanate (BST) and Nickel Zinc Ferrite (NZF) in CST Microwave Studio and calculated their effective permittivity and permeability from 1 - 4 GHz region. He emphasized that the use of CST MWS can give better results in measuring the dielectric properties of dielectric substances.



Fig 2.1. Diagram of the Generation, Simulation and Calculation Process [47]

Guo, Jiang, Fu, and Research (2018) synthesized modified [(1-x)MnO₂-xMWCNTs] /waterborne Polyurethane composites that exhibited not only good microwave absorption properties but also better thermal stability and mechanical strength. Out of different samples the one that contained 20% MWCNTs showed the maximum reflection loss of -28.7dB at 6.4 GHz frequency range. The sample thickness was 2.5mm. The microwave absorption value decreased with increase in concentration of MWCNTs in the composite. [48]

2.2 Dielectric Properties of MAMs

Yakovenko et al. (2019) tested the composites made up of MWCNTs/Epoxy in 1 - 67GHz frequency range for their dielectric properties experimentally. They also calculated the effective permittivity of various volume fractions of composites using the Maxwell Garnett mixing model for nanocomposites taking aspect ratio of nanotubes into consideration. The experimental and calculated results only coincide at volume fraction as small as 0.006%. This work established the importance of interface between nano filler and matrix particles in determination of dielectric properties of the nanomaterial. [49]

He, Duan, Pang, and Zhang (2019) designed single component microwave absorber mesoporous MnO_2 with controlled pH in Wax matrix. The sample with pH 5 showed maximum reflection loss of -31.785dB at 14.60GHz. This experiment used a high surface area, light weight compound that showed the maximum RL value in Ku band region of microwave region.



Fig 2.2. Scheme of Primary Electromagnetic Microwave Attenuation Process Involved in MnO₂ Composites Absorber [50]

2.3 Morphological and Structural Effects:

Savi, Giorcelli, and Quaranta (2019) simulated single layer nanocomposite consisted of 4% MWCNTs with epoxy resin as a matrix on metallic plate. He used high aspect ratio MWCNTs (approx. 1400) and with high surface area (approx. 300g/m2) in his model and proved that the morphology of nanofillers like CNTs are more important in determining their microwave absorption properties than their concentration. [51]

Kuchi et al. (2018) used Co-Arc discharge process to synthesize $Fe_3O_4/SWCNT$ composite. Samples made up of $Fe_3O_4/SWCNTs$ in paraffin wax were tested for microwave absorption in Vector Network Analyzer. A sample of 2.5mm thickness with 30% Iron Oxide – CNT load showed the remarkable absorption of -36.9 dB at 10.5GHz (X-band region). The good microwave absorption attributed to combined electric and magnetic losses due to suitable mass ratio of CNT to Fe_3O_4 . [52] Bora, Vinoy, Ramamurthy, and Madras (2017) prepared Polyaniline-MnO₂ Nano rod composite at low temperature, and checked its microwave absorption capacity in X – band and Ku – band region. In X-band region the maximum shielding was found 35dB where SE_A was approximately 28dB and SE_R was 11dB.In Ku – band Shielding effectiveness was 39dB, where SE_A was 29dB and SER was 10dB.The authors also compared the shielding effectiveness of already reported materials at various thickness which are mostly in the range of 2 – 2.5mm. The important aspect of their work is the good shielding effectiveness value obtained at as small thickness as 169 micrometers for X-band and 81 micrometers for Ku band. [53]

Wang et al. (2016) synthesized 3D hollow MnO₂ microspheres with SiO₂ sphere templates and compared their microwave absorption properties with 1D MnO₂ nanoribbons prepared in the absence of SiO₂ templates. They reported the improve absorption properties of hollow MnO₂ in Ku band with -40dB minimum RL at 14.2GHz frequency. They concluded that the light weight and low cost of hollow MnO₂ spheres make them promising materials to be used as MAMs. [54]

Che et al. (2015) used three different commercially available MWCNTs (Baytubes C150P, Nanocyl NC7000 & VAST), in epoxy resin to produce nanocomposite. They used two simple techniques to make the dispersions, the solution dispersion technique and the ball milling method. They emphasized that Nanocyl NC7000 gave better performance as microwave absorber in X-band region irrespective of the method used, however for bulk composite formation they found ball milling method better than solution dispersion, as it saves the mechanical agitation through ultra-sonication and evaporation of solvent used in solution dispersion method. [55]

Hong, Xiao, Luo, and Li (2015) used a formula based on Reynold's Hugh theory to calculate dielectric properties (effective permittivity), of Carbon Fiber (CF), nanocomposites in epoxy resin with different lengths and volume fractions at X-band region. They found that complex permittivity of composite decrease with fiber length and increased with concentration i.e. volume fraction. They attributed this behavior to the change in depolarization factor with change in volume fraction and length of the carbon fibers. [56]

Lv, Ji, Liang, Zhang, and Du (2015) synthesized a ternary composite made up of 30nm wide rod-like strip of MnO₂ with Fe deposited on it. Afterwards graphene nanoparticles were loaded

on it as well. In this experiment the permittivity value of MnO_2 , $MnO_2@Fe$ and $MnO_2@Fe$ -GNP were calculated and compared. $MnO_2@Fe$ -GNP gave highest reflection loss of -17.5 dB. [57]

Wang and Zhao (2013) prepared various samples of carbonyl Iron / MnO₂ nanocomposite dispersed in paraffin wax and checked their microwave absorption properties in 2 – 18 GHz frequency range. The minimum reflection loss of -39.1 dB was observed at 4.4 GHz and the RL values under -20dB were observed at 2-8 GHz. Higher frequencies i.e. X-band and Ku – band didn't show the considerable reflection loss. Therefore, they concluded that Carbonyl Iron/MnO₂ nanocomposite can be good microwave absorbing materials in S-band and C-band only. [58]

Liang, Yang, and Choi (2012) reported the MWCNT-epoxy composite with 1-10% CNT loading showed the increase in microwave absorption in 2-20GHz frequency range. 20 – 26% absorption was noted at 18-20 GHz frequency range, with increase in the volume percentage of MWCNTs loading. [59]

Zhang et al. (2014) worked on irregular shape MnO₂ nanoparticles (NP) and MnO₂ Nano rods composites in epoxy resin for their microwave absorption properties in 2-18 GHz frequency range. They reported that 60% NP containing composite showed only -11.6dB reflection loss at 9.12GHz frequency where as 12% NR containing composite showed -20dB reflection loss at 9.6GHz frequency, the thickness of both the samples was taken as 3mm. This result shows the importance of regular shapes of nano fillers in achieving the microwave absorption. The MnO₂ nano rods with high aspect ratio proved to be better MAM than its corresponding irregular shape MnO₂ nanoparticles. [60]

Decrossas, Sabbagh, Hanna, and El-Ghazaly (2011) found the effective complex permittivity of bundled network of Single Walled Carbon nanotubes with different densities over a wide range of frequencies, ranging from 10MHz – 50GHz. They used only fraction of material for testing and used vector network analyzer to test the material. The importance of the method was the use of only nanotubes networks in analyzer rather than its composite with some polymer matrix. The experimental results were in accordance with the simulation results. [61]



Fig 2.3. Molecular Dynamics Simulations of Single-Walled Carbon Nanotube Bundles Under Mechanical Loading [62]

Li et al. (2012) used Maxwell Garnett effective medium technique to measure the effective complex permittivity of solid materials. In their experiments, researchers used solid particles dispersed in silicone oil to make composites at different volume fractions. From the measured permittivity of oil and the composite they derived the permittivity of solid inclusions using MG mixing formula. The process doesn't involve to take solid shapes into consideration, and measured values for alumina, glucose and pearl experimentally validated as well. [63]

Sun, Gao, Li, Wu, and Yellampalli (2011) investigated MWCNTs doped in rare earths and aligned nanotubes for their microwave absorption properties within 2-18 GHz range. They found the improved microwave absorption in X-band range when carbon nanotubes were doped with 1% rare earths. At 10.88 GHz these showed -29 dB reflection loss. Whereas the aligned nanotubes gave better absorption properties at higher frequency region of 2-18GHz frequency. [64]

Folgueras, Alves, Rezende, and Management (2010) characterized two different paint formulations for their microwave absorption properties in X-band region. One formulation of the polyurethane paint contained carbonyl iron and /or polyaniline showed the attenuation of 60 - 85% of incident electromagnetic radiation. Silicone sheets with conducting polymer polyaniline as filler also produced and these showed up to 90% attenuation from microwave

radiations. Simulations for these sheets also performed to find out the relationship between electromagnetic absorption parameters with thickness of the sheets. [65]

Koledintseva, Drewniak, DuBroff, Rozanov, and Archambeault (2009) modelled the carbon based shielding composite materials using Maxwell Garnett theory for inclusion concentration below the percolation threshold, and McLachlan formulation for concentration of inclusions above percolation threshold. They used the genetic algorithm to optimize the parameters to get maximum shielding effectiveness for the desired frequency range. They used CST studio for modelling of these composite. [66]

Kučerová et al. (2009) prepared various Polyurethane matrix samples containing different types of carbon nanofillers in different concentrations. The carbon filler used were, flat carbon nanoparticles, carbon microfibers and multi walled carbon nanotubes. The use of fillers improved the mechanical properties like hardness, resistance to deformity of the matrix. They found that the enhancement of mechanical properties mainly depends on the dispersion of nanofiller in the polymer matrix. Well dispersed fillers improved the mechanical strength of poly urethane matrix than not dispersed fillers. [67]



Fig 2.4. Chemical Structure of Polyurethane

2.4 Thermal Studies:

Since last decade, researchers are working to investigate effect of temperature on stability of composites being used as MAM's. Liew, K. M., Wong, C. H., He, X. Q., & Tan, M. J. (2005) studied the thermal stability of SWCNTs and MWCNTs through modelling and simulation. They concluded that SWCNTs were thermally more stable than MWCNTs. Closed capped carbon nanotubes were found thermally stable up to 2000K while open ended were stable up

to 1000K temperature. They also concluded that other factors like length, diameter and structure also determine the stability of carbon nano tubes. [68]

Tinsley, D. M., & Sharp, J. H. (1971) performed the thermal analysis of MnO₂ in controlled atmosphere of Nitrogen, air and oxygen. They find that in air the MnO₂ show stability to heat up to 823K. [69]

2.5 Gap Identification:

Extensive research work has been done on micro wave absorptive materials. With advent of nanotechnology, new materials have been synthesized and characterized for shielding effectiveness. However, only limited number of computational studies are available discussing the microwave absorptive properties of materials. While comparative work using different nanocomposites with different thickness is nearly non-existent.

Many experimental studies for composites containing carbon nano tubes have been performed, but mostly used very small concentrations of carbon nano tubes in their composites and samples with limited thickness has been characterized. As for as MnO₂ is concerned, few studies are available where MnO₂ is used with other materials like graphene to find out their microwave absorptive properties but no study is available that shows the comparative shielding effectiveness of its nano composites with epoxy and poly urethane.

The complete theoretical study, that uses effective medium theory to find out the permittivity of composites and then evaluate the shielding effectiveness of those composites using microwave studios and other material programs is not available.

2.6 Problem Statement:

A variety of metal based nanomaterials have been used as microwave absorbing materials (MAMs). However, these materials have high density, apt to agglomerate, intolerant to high temperature and have strong affinity to oxide, which greatly limit their applications as MAMs. Therefore, MAMs with light weight, small thickness, strong chemical stability, wide absorption frequency range and excellent absorption performance are greatly desired.

2.7 Preface and Significance of Current Research:

Nano composites are the main focus of the current researches by the material scientists. However, no work is available that use to calculate both the effective permittivity of nanocomposites theoretically and then to find out shielding effectiveness computationally. The present research aims to find effective permittivity of four different nano composites through famous Maxwell Garnett effective medium theory and use of these parameters to find out the scattering parameters and shielding effective ness of these materials in two different frequency ranges i.e. X-band range and Ku band range.

The research also focuses on the comparative behavior of the four composites in the above mentioned frequency ranges. The comparison is based on the thickness of the sample taken. The method used in this study can be useful to find out the effective properties of nanocomposites, *i.e.* permittivity and permeability through effective medium theories. The model designed for this project can be used to find scattering parameters and thus shielding effectiveness in any study where permittivity and permeability value of the material is given for different frequency ranges.

2.8 Objectives:

- To determine the effective permittivity of nanocomposites (epoxy and polyurethane with SWCNTs and MnO₂), by using effective medium theory.
- To evaluate effect of thickness on microwave absorption through scattering parameters, reflection loss and total shielding effectiveness.
- To compare the total shielding values of nanocomposites with pristine SWCNT and MnO₂.
- To find out the effective nanocomposites to be used in coatings at various frequency ranges from 8-18 GHz.

Chapter 3 Material and Methods

Material and Methods

The current work is focused to find out the microwave absorption tendencies of nanocomposites composed of: (i) Single Walled Carbon Nanotube bundles (SWCNTs) with (a) Polyurethane (b) Epoxy resin and ii) Rod-like Manganese dioxide nano strips with (a) Polyurethane (b) epoxy resin. For this purpose, we have used Computer Simulation Technology (CST-Microwave Studio Suite 2019) to determine scattering parameters S11 and S21. CST studio is based on Maxwell Electromagnetic Equations.

3.1 Electromagnetic Theory

James Clerk Maxwell (1865), proposed that the electric currents and charges creates electric and magnetic fields and the electric field creates the magnetic field and vice versa. In his theory he successfully unified the concept of electricity, magnetism and light Electromagnetic theory is the set of 4 equations that describe the electromagnetic interactions. The equations proposed by J.C. Maxwell, are based on the earlier experimental and mathematical work of Ampere, Michael Faraday and Gauss. The present form of these equations come from the work of Oliver Heaviside who simplified and limited the equations to electric and magnetic fields and their sources. These equations are used both in their differential as well as integral form. The equations describe the creation of the electric and magnetic fields by distribution of charges and the change in their strengths with time. [70-72]

The differential form of equations is:



In these equations:



It is easy to understand the physical meaning of Maxwell equations using their alternative integral form. Equation no 1 and 2 can be converted into integral form by using Stoke's Theorem, whereas equations 3 and 4 can be converted into their integral form by using Divergence Theorem.



Fig 3.1. Stoke's Law
Hence the integral form of these equations are:

$$\int_{l} \overline{H} \cdot d\overline{l} = \oint_{a} \left(\frac{\partial \overline{D}}{\partial t} + \overline{J} \right) \cdot d\overline{a}$$
(5)
$$\int_{l} \overline{E} \cdot d\overline{l} = \oint_{a} \frac{\partial \overline{B}}{\partial t} \cdot d\overline{a}$$
(6)

$$\int_{S} \overline{D} \cdot d\overline{a} = \int_{V} \rho \ dV \tag{7}$$

$$\int_{S} \bar{B} \cdot d\bar{a} = 0 \tag{8}$$

3.2 Dielectric Properties

The dialectics are the non-conducting materials having the tendency to store electric charges, hence show polarization in the presence of external electric field

i. Polarization

The perfect dielectrics don't allow their charges to be carried away by the external electrical field. The charges under the influence of electric field slightly move from their mean position, thus try to create polarization. However, at the same time an opposite force, a "restoring force" try to stop this disturbance of charges from their actual position. The dominating strength of either of the two forces decide the amount of displacement take place between positive charges, that move towards electric field and the negative charges move away from it. The separation between these charges is equal to the dipole moment produced. This polarization produced in electric field make it possible for dielectrics to store the electrical energy in them. Similarly, magnetic materials show magnetic polarization under the influence of external electrical or magnetic field.

Different materials can have different types of charge distribution in them, so we have different type of polarizations. Such as, atomic, ionic, orientation and interfacial polarization. Each type corresponds to different frequency ranges and can also material specific, such as ionic polarization can be shown by Ionic compounds only. Similarly, orientation polarization is low

frequency phenomenon while electronic polarization occurs at higher frequencies like infrared frequency region. In microwave frequency range material usually undergo to dipolar or orientation polarization phenomenon. [73]

ii. Complex Permittivity & Permeability

Complex permittivity or dielectric constant ε_r is the ability of a substance to store electrical energy in an electric field where as Maxwell equation no. 1 & 2 are:

$$\nabla \times \overline{H} = \frac{\partial D}{\partial t} + \overline{J}$$
(9)
$$\nabla \times \overline{E} = -\frac{\partial \overline{B}}{\partial t}$$
(10)

In vacuum the electric flux density vector D and magnetic flux vector B can be written as

$$D = \varepsilon_0 E$$
 (11)
 $B = \mu_0 H$ (12)

Where ε_0 is the free space permittivity with value 8.85×10^{-12} F/m and μ_0 is the free space permeability having value 1.2566×10^{-6} H/m. In a material these relationships do not remain this simple and become:

$\mathbf{D} = \boldsymbol{\epsilon}_0 \mathbf{E} + \mathbf{P}$	(13)
(where P is the polarization)	
$B = \mu_0 H + M$	(14)
(M is the magnetic polarization)	

Polarization P is in direct relationship with electric field E and magnetic polarization is in direct relationship with magnetic field H:



 $(\chi_m \text{ is the magnetic susceptibility})$

Together with Ohm's Law (J = σ E) and Maxwell equations in time harmonic field we get the complex permittivity value as

$$\varepsilon = \dot{\varepsilon} - j \ddot{\varepsilon} = \varepsilon_0 \left(\dot{\varepsilon} \mathbf{r} - j \ddot{\varepsilon}_r \right)$$
(17)

Where σ is the conductivity of material, $\dot{\epsilon}$ is the real part of permittivity and ϵ (double prime) is the imaginary part. The real part is concerned with the energy stored by the medium and imaginary part is about the energy loss within the medium.

An analogous treatment of eq 2 results in the relationship:

$$\mu = \mu' - j \mu'' = \mu_0(\mu'_r - j\mu''_r)$$
(18)

Another important factor uses to characterize the dielectrics is dissipation factor also known as tan\delta. It is the ratio of imaginary part of permittivity to its real part, i.e.

$$\tan \delta_{\rm e} = \varepsilon'' / \varepsilon' \tag{19}$$

For magnetic loss tangent the equation is:

Both these properties, permittivity and permeability are frequency dependent phenomenon. [74-75]

iii. Dispersion and Relaxation in Dielectrics

The frequency dependency of complex permittivity and permeability is called "Dispersion". Kramers-Kronig relation is used to study the behavior of real and imaginary permittivity values in different frequencies.

The adjustment of polarization of dielectric in presence of electric field is known as "relaxation". For example, in low frequencies like that of microwave frequency the dipoles in a material has enough time to rotate and to keep in step with change in electric field but in higher frequencies real permittivity $\dot{\epsilon}$ decreases as individual dipoles can't keep in step with changing external field. [76]

3.3 Shielding Effectiveness

Shielding effectiveness (SE), is the tendency of a material against electromagnetic radiations. It is measured in terms of reduction in intensity of incident electromagnetic radiations while crossing the shield. When electromagnetic wave strikes the shield part of it is reflected at the interphase due to impedance mismatch between the incident wave and the shield. This is called Reflection Loss (RL). A part of it is absorbed by the shielding material, which is called the Absorption Loss. The main mechanism followed in absorption loss is the heat generation by the material due to the presence of dipoles in it (electric, magnetic or both depending on type of material). Part of the incident wave is attenuated due to multiple reflections within the material. Mathematically, we can calculate shielding effectiveness as [77-79]

$$SE_T = SE_R + SE_A + SE_M$$
(21)

Where

 SE_R = Shielding effectiveness due to reflection

 $SE_A =$ Shielding effectiveness due to absorption

 SE_M = Shielding effectiveness due to multiple internal reflections



Fig 3.2. Schematic of EMI Shielding Mechanism [80]

Multiple internal reflection factor is usually excluded at higher frequencies \sim 1GHz or greater. So total shielding can be written as:

$$SE_{T} = SE_{R} + SE_{A} \qquad (22)$$

SE_R & SE_A, the two values can be calculated in terms of Reflectance and Transmittance

$$SE_R = 10 \log (1/1-R)$$
 (23)

And

$$SE_A = 10 \log (1-R/T)$$
 (24)

Putting eqs 23 & 24 in equation 22 will give total shielding as

$$SE_T = 10 \log (1/T)$$
 (25)

The values of Reflectance (R) and Transmittance can be calculated through Scattering Parameters as:

$$\mathbf{R} = |\mathbf{S}11|^2 = |\mathbf{S}22|^2 \quad (26)$$

And

$$T = |S12|^2 = |S21|^2$$
 (27)

3.4 Effective Medium Theories

It is now a well-known fact that composite materials containing dielectric matrix with conductive inclusions can be used as shielding materials. To evaluate the effective properties of the composites different effective medium theories are used.

Maxwell Garnett Theory

The most important and excessively used is Maxwell Garnett effective medium theory for homogeneous mixtures (MG EMT). Composite containing rod shaped inclusions have frequency dependent effective permittivity. To make use of the effective medium theory we need to know the electromagnetic parameters of the matrix and the inclusions. [81]

Maxwell Garnett multiphase formula to find effective permittivity of composite with various concentrations of inclusions is:

$$\varepsilon_{eff} = \varepsilon_m + \frac{\frac{1}{3} \sum_{i=1}^n v_f \left(\varepsilon_f - \varepsilon_m\right) \sum_{k=1}^3 \frac{\varepsilon_m}{\varepsilon_b + N_{ik} (\varepsilon_f - \varepsilon_m)}}{1 - \frac{1}{3} \sum_{i=1}^n v_f \left(\varepsilon_f - \varepsilon_m\right) \sum_{k=1}^3 \frac{N_{ik}}{\varepsilon_b + N_{ik} (\varepsilon_f - \varepsilon_m)}}$$

Where

 ε_m = Relative Permittivity of a Matrix Dielectric

 \mathcal{E}_f = Relative Permittivity of nanofiller

 $v_f =$ Volume fraction of the filler

 N_{ik} = Depolarization factor of *i*th type of inclusions

K = 1,2,3 corresponds to x, y, z Cartesian coordinates

To find out depolarization factor of an ellipsoid the formulae used are: [82 - 84]

$$N_{x} = \frac{(1-e^{2})}{2e^{3}} \left(\ln \frac{(1+e)}{(1-e)} - 2e \right)$$
$$N_{y} = N_{z} = \frac{1-N_{x}}{2}$$
$$e = \sqrt{1 - (\frac{2r}{l})^{2}}$$

3.5 Materials and their Properties

In the present work, following matrix and nanofiller have been used to design nanocomposites under required conditions.

Matrix (Polymer)	Nano fillers
Polyurethane (PU)	Single Walled Carbon Nanotubes Bundles (SWCNTs)
Epoxy (EP)	Manganese Dioxide (MnO ₂)

Properties of Polymer Matrix:

Polymer	Tensile Strength	Shear Strength
Polyurethane	70 kPa	40 kPa
Ероху	5.17 – 97.0 MPa	6.89 – 137 MPa

Properties of Nano fillers:

Nano fillers	Length (µm)	Diameter (µm)
SWCNTs	20	0.01
MnO ₂	0.4	0.03

3.6 Methodology

In order to determine effective permittivity of composite material with 0.4% volume fraction of nanofiller, we used Maxwell Garnett equation. Four composite materials were designed, comprising of two nanofiller i.e. SWCNTs and MnO₂, each one combining the polymer matrix (Polyurethane and Epoxy).

i. CST Microwave Studio

CST Microwave studio is a 3D Electromagnetic solver software to design, analyze and optimize systems for their performance in various electromagnetic frequency ranges. CST microwave studio uses different methods like Frequency Difference Time Domain (FDTD), Finite Element Method (FEM), Finite Integration Technique (FIT) or Transmission Line Matrix (TLM) to perform high frequency simulation tasks. The software is based on Maxwell electromagnetic equations, and the solvers to solve these equations use either of the methods mentioned above.

We designed nanocomposites in Computer Simulation Technology software (CST MW Studio Suite 2019), to find out their microwave absorption properties in X-band region (8.2 - 12.4)GHz) and Ku – band region (12.4 – 18 GHz) of microwave spectrum.

Create Project Template

Choose an application area and then select one of the workflows:



Fig 3.3. CST Microwave Studio Suite 2019

ii. Wave Guide Models

To find out the permittivity value of microwave absorptive materials, number of different methods are applied. One of them is to use rectangular waveguides. The sample under test (SUT), is carefully placed in the waveguide and the scattering parameters are calculated through passing the electromagnetic energy through waveguide for desired frequency range. The dimensions of these waveguides are given in literature. [85]

For microwave absorption Simulations such waveguides are modelled in 3D program and scattering parameters are obtained. For this purpose, we modelled two different waveguides in frequency ranges 8.2 - 12.4 GHz and 12.4 - 18 GHz.

iii. Setting Up the Simulations

8.2 – 12.4 GHz Range

The waveguide inner opening dimensions in this frequency range were set as 22.86mm and 10.16mm respectively. The background material taken was Perfect Electric Conductor (PEC). The time domain solver was selected for scattering parameters calculations using hexahedral mesh properties.



Fig 3.4. Waveguide Containing 1mm Epoxy with MnO₂ Composite - a CST Model

12.4 – 18 GHz Range

Two waveguide were created with 15.79mm length and 7.89mm width, the inner dimensions. The background material was taken as perfect electric conductor. The sample materials, the composite SWCNT bundles with polyurethane and epoxy resin and then MnO_2 –PU and MnO_2 - epoxy were taken in the center of the two waveguides. The two waveguide ports were created at the opposite faces of the waveguides. The simulations were run with electrical boundary conditions and scattering parameters were obtained.



Fig 3.5. Meshing in CST Studio with 41,580 Mesh Cells

Chapter 4

Results and Discussion

Results and Discussion

The result section of this chapter is divided in four parts.

- a. Reflection Loss and Shielding effectiveness studies in 8 12.2 GHz range (X Band Studies)
- B. Reflection Loss and Shielding effectiveness studies in 12.4 18 GHz range (Ku Band Studies)
- c. Comparative result analysis of SWCNT and MnO₂ Composites
- d. Final Conclusion

4.1 EMI Shielding in X – Band Studies:

Effective Permittivity (ϵ), Reflection Loss (RL) and Shielding effectiveness (SE) of the following 0.4% compositions (1-5mm) are discussed in the order.

- 1. PU / SWCNTs
- 2. Epoxy / SWCNTs
- $3. \ PU \ / \ MnO_2$
- 4. Epoxy / MnO₂

4.1.1 Effective Permittivity:

Composite formation changes the real and imaginary component of permittivity of both the polymer matrix and the nanofiller. Permittivity is the one of the key property that describes the microwave attenuation ability of the material. The real part of permittivity ε' determines how much microwaves penetration is allowed by the material and the imaginary part ε'' , determines the storage tendency of the penetrated radiation in the material. Whereas, dissipation factor tan δ determines the material's ability to convert the penetrated energy into heat. For good

shielding properties the material should have a balanced combination of all the three factors, ϵ' , ϵ'' and tan δ . [86]

The addition of filler in polymer matrix, changes the both the real and imaginary part of permittivity for both the filler and the matrix. So the composite that is formed, shows different values of permittivity's then the pure components. Hence, the modified values improve the EMI shielding property of the composite material. [87]

The following figures, show this change in permittivity values of the four composites, namely: PU/SWCNTs, PU/MnO₂, Epoxy/SWCNTs and Epoxy/MnO₂ composites. These values are obtained by using Maxwell-Garnett mixing formula, and are given in data tables given below.

$$\varepsilon_{eff} = \varepsilon_b + \frac{\frac{1}{3}\sum_{i=1}^n v_i \left(\varepsilon_i - \varepsilon_b\right) \sum_{k=1}^3 \frac{\varepsilon_b}{\varepsilon_b + N_{ik}(\varepsilon_i - \varepsilon_b)}}{1 - \frac{1}{3}\sum_{i=1}^n v_i \left(\varepsilon_i - \varepsilon_b\right) \sum_{k=1}^3 \frac{N_{ik}}{\varepsilon_b + N_{ik}(\varepsilon_i - \varepsilon_b)}}$$

Here

ε_b

= Permittivity of polymer matrix

 ϵ_i = Permittivity of nanofiller inclusions

 ϵ_{eff} = Effective permittivity of nanocomposite



 N_{ik} = Depolarization factor of the nanofiller inclusions

Fig 4.1. Real Permittivity (É) of Polyurethane, SWCNTs and Polyurethane Composite with SWCNTs

The figure 4.1 illustrates, the change in real permittivity $\dot{\epsilon}$ values of PU composite with SWCNTs. The permittivity values of pure SWCNT is below 1.9, and that of pure polyurethane is lies above 2.7, but after composite formation the value is in the range of 2.4.



Fig 4.2. Imaginary Permittivity (ϵ'') of Polyurethane and SWCNTs and Polyurethane Composite with SWCNTs

The imaginary permittivity value ε'' , is modified and lies in the middle for Polyurethane/SWCNTs composite than for pure Polyurethane and pure SWCNTs

Permittivity Tables X - band

S.No	Frequency (GHz)	Real Permittivity (Poly Urethane) έ	Real Permittivity (SWCNT) έ	ε _{eff} (Effective Real Permittivity of Composite)	Imaginary Permittivity (Poly Urethane) ε''	Imaginary Permittivity (SWCNT) ε''	ϵ_{eff} (Effective Imaginary Permittivity of Composite)
1	8.0	2.74	1.92	2.39	0.17	1.45	4.77E-01
2	8.2	2.76	1.9	2.39	0.18	1.44	4.86E-01
3	8.5	2.77	1.86	2.38	0.2	1.43	5.06E-01
4	8.8	2.77	1.83	2.37	0.19	1.42	4.93E-01
5	9.2	2.80	1.81	2.37	0.22	1.40	5.21E-01
6	9.5	2.80	1.78	2.36	0.21	1.37	5.05E-01
7	9.8	2.84	1.75	2.37	0.23	1.36	5.23E-01
8	10.1	2.84	1.73	2.36	0.24	1.31	5.23E-01
9	10.4	2.85	1.7	2.35	0.24	1.31	5.23E-01
10	10.7	2.85	1.68	2.34	0.24	1.35	5.32E-01
11	11	2.85	1.66	2.32	0.26	1.29	5.39E-01
12	11.3	2.85	1.63	2.31	0.26	1.28	5.37E-01
13	11.6	2.85	1.61	2.30	0.24	1.28	5.17E-01
14	11.9	2.85	1.59	2.29	0.21	1.26	4.82E-01
15	12.2	2.85	1.56	2.28	0.21	1.24	4.78E-01

Table 4.1: Real and Imaginary Effective Permittivity of 0.4% PU Composite with SWCNTs in X – Band Region

The addition of SWCNTs in Polyurethane modify the effective permittivity values, as is apparent in table 4.1. At 8GHz frequency the real permittivity $\dot{\epsilon}$ for Polyurethane matrix is 2.74 and of SWCNTs is 1.92, while after composite formation the value is 2.39, which lies in middle of the two corresponding values. Similarly, the effective imaginary permittivity ϵ ", is changed after composite formation. At 8GHz the value for the PU/SWCNT composite the value is 4.77E-01, which is greater than the effective permittivity value of Polyurethane and less than the permittivity value of SWCNTs at the same frequency range

S.No	Frequency	Real	Real	ϵ_{eff} (Effective Real	Imaginary	Imaginary	ϵ_{eff} (Effective Imaginary
	(GHz)	Permittivity	Permittivity	Permittivity of	Permittivity (Epoxy)	Permittivity	Permittivity of
		(Epoxy)	(SWCNT)	Composite)	ε"	(SWCNT)	Composite)
		έ	έ			٤''	
1	8.0	4	1.92	3.06	0.03	1.45	3.05E-01
1	8.2	4	1.9	3.05	0.03	1.44	3.03E-01
2	8.5	4	1.86	3.03	0.03	1.43	3.01E-01
3	8.8	4	1.83	3.02	0.03	1.42	3.00E-01
4	9.2	4	1.81	3.00	0.03	1.40	2.96E-01
5	9.5	4	1.78	2.99	0.03	1.37	2.90E-01
6	9.8	4	1.75	2.97	0.03	1.36	2.89E-01
7	10.1	4	1.73	2.96	0.03	1.31	2.79E-01
8	10.4	4	1.70	2.95	0.03	1.31	2.79E-01
9	10.7	4	1.68	2.94	0.03	1.35	2.87E-01
10	11	4	1.66	2.92	0.03	1.29	2.76E-01
11	11.3	4	1.63	2.91	0.03	1.28	2.74E-01
12	11.6	4	1.61	2.90	0.03	1.28	2.74E-01
13	11.9	4	1.59	2.89	0.03	1.26	2.70E-01
14	12.2	4	1.56	2.87	0.03	1.24	2.67E-01

Table 4.2: Real and Imaginary Effective Permittivity of 0.4% Epoxy Composite with SWCNTs in X – Band Region

In case of Epoxy-SWCNT composite the values of real and imaginary permittivity are modified. At 8 GHz the real permittivity value for Epoxy is 4 and 1.92 for SWCNT which changes to 3.06. It is important to note that the real permittivity is greatly improved from the value for pure SWCNT. The imaginary part is modified as well, which is very low for pure Epoxy. AT 8 GHz this value is 0.03 for epoxy and for pure SWCNTs is 1.45, but for the composite it lies in the middle of the two components i.e. 3.05E-01.

S.No	Frequency	Real	Real	ϵ_{eff} (Effective Real	Imaginary	Imaginary	ε _{eff} (Effective Imaginary
	(GHz)	Permittivity	Permittivity	Permittivity of	Permittivity (Poly	Permittivity	Permittivity of
		(Poly Urethane)	(MnO ₂)	Composite)	Urethane)	(MnO ₂)	Composite)
		έ	έ		٤"	٤"	
1	8.0	2.74	24	7.22	0.17	20	1.11
2	8.2	2.76	23.9	7.23	0.18	20.5	1.17
3	8.5	2.77	23.6	7.20	0.2	21.5	1.29
4	8.8	2.77	22.8	7.07	0.19	21.1	1.23
5	9.2	2.80	20.7	6.81	0.22	22.4	1.40
6	9.5	2.80	19.9	6.69	0.21	21.4	1.34
7	9.8	2.84	19	6.59	0.23	20.3	1.42
8	10.1	2.84	18.9	6.58	0.24	20	1.47
9	10.4	2.85	18.1	6.46	0.24	21.1	1.48
10	10.7	2.85	16.2	6.13	0.24	21.6	1.49
11	11	2.85	14	5.72	0.26	22.4	1.60
12	11.3	2.85	13.8	5.69	0.26	21.8	1.59
13	11.6	2.85	13.7	5.67	0.24	21.1	1.48
14	11.9	2.85	13.7	5.67	0.21	21.1	1.33
15	12.2	2.85	13.5	5.64	0.21	20.8	1.33

Table 4.3: Real and Imaginary Effective Permittivity of 0.4% PU Composite with MnO₂ in X – Band Region

The real permittivity $\dot{\epsilon}$ value for polyurethane is in the range of 2 for all frequencies between 8-12.2GHz, which is low than the real permittivity $\dot{\epsilon}$ value for MnO₂, which has the highest value of 24 at 8GHz. The $\dot{\epsilon}$ for Polyurethane/SWCNTs composite is 7.22 for 8GHz frequency range, which is greater than that of pure Polyurethane and less than pure MnO₂. Due to less value of imaginary permittivity of pure polyurethane at all X band frequencies the ϵ'' value for Polyurethane composite with MnO₂ is also low.

S.No	Frequency	Real	Real	ϵ_{eff} (Effective Real	Imaginary	Imaginary	ε _{eff} (Effective Imaginary
	(GHz)	Permittivity	Permittivity	Permittivity of	Permittivity (Epoxy)	Permittivity	Permittivity of
		(Epoxy)	(MnO ₂)	Composite)	۳3	(MnO ₂)	Composite)
		έ	έ			ε''	
1	8.0	4	24	8.83	0.03	20	2.30E-01
1	8.2	4	23.9	8.81	0.03	20.5	2.30E-01
2	8.5	4	23.6	8.76	0.03	21.5	2.31E-01
3	8.8	4	22.8	8.60	0.03	21.1	2.30E-01
4	9.2	4	20.7	8.24	0.03	22.4	2.31E-01
5	9.5	4	19.9	8.09	0.03	21.4	2.31E-01
6	9.8	4	19	7.92	0.03	20.3	2.30E-01
7	10.1	4	18.9	7.90	0.03	20	2.30E-01
8	10.4	4	18.1	7.74	0.03	21.1	2.30E-01
9	10.7	4	16.2	7.36	0.03	21.6	2.31E-01
10	11	4	14	6.88	0.03	22.4	2.31E-01
11	11.3	4	13.8	6.84	0.03	21.8	2.31E-01
12	11.6	4	13.7	6.82	0.03	21.1	2.30E-01
13	11.9	4	13.7	6.82	0.03	21.1	2.30E-01
14	12.2	4	13.5	6.77	0.03	20.8	2.30E-01

Table 4.4: Real and Imaginary Effective Permittivity of 0.4% Epoxy Composite with MnO2 in X – Band Region

The real permittivity $\dot{\epsilon}$ for epoxy is 4 for all frequency values as taken from CST material library, the $\dot{\epsilon}$ for epoxy composite for MnO₂ is 8.83 at 8GHz frequency which is greater than its value for pure epoxy and less than that for pure MnO₂. However, the imaginary value of the epoxy composite with MnO₂, comes to be very low than that of pure epoxy i.e. 2.30E-01 at 8GHz. The possible reason for this is the low values of imaginary permittivity for pure epoxy which is only 0.03 for all frequency values.

4.1.2 Shielding Effectiveness:

EM shielding can take place though three mechanisms: Reflection, Absorption and Multiple Reflections, which are represented as three losses. These are the reflection loss, absorption loss and the multiple reflection loss. In case of all the four composites under study we find that the main contributor in shielding effect depends on Reflection mechanism in X band region, hence Reflection Loss value give us the effectiveness of the composite to attenuate the microwaves.



Fig 4.3. RL Values for 0.4% Polyurethane/SWCNTs Composites

The reflection loss (RL) value for Polyurethane composite with SWCNTs indicates that the RL values for 1mm thickness is the largest. At 10.7GHz, -14.8dB RL is shown by 0.4% polyurethane composite for 1mm thick sample. Other four samples with 2-4mm thickness show less than -10dB RL values for all X-band frequencies.



Fig 4.4. RL Values for 0.4% Epoxy/SWCNTs Composites

1mm thick 40% Epoxy composite with SWCNTs show -11.9dB RL value at 9.7GHz frequency, which is the highest value for all the composite samples of 1-5mm thickness. 2-4mm thick samples show the RL value in the range of 4 - 7dB in all X-band frequency ranges.



Fig 4.5. RL Values for 0.4% Polyurethane/MnO₂ Composites

The maximum reflection loss RL value for Polyurethane MnO2 composite is -12.4dB at 12.2GHz frequency which is the maximum limit of X-band frequency range. This value corresponds to 5mm thick sample of the composite. All the other samples i.e. 1-4mm thick samples show low values of RL, which lies in 2-6dB range.



Fig 4.6. RL Values for 0.4% Epoxy/MnO2 Composites

In figure 4.6, it is evident that 5mm thick epoxy composite with MnO₂ sample shows the maximum reflection loss RL value of -20.6dB at 11.1GHz frequency. Other samples with 1-4mm thickness show as low as 1-5dBrange of RL values in X-band region.

Conclusion:

The data in the graphs shows that the Polyurethane/SWCNTs composites gives the maximum Reflection Loss values of -14.82dB and -11.98dB at 10.7GHz and 9.7GHz respectively. These values are achieved at 1mm thickness. The composites with thickness greater than 1mm all show decreased Reflection Loss values hence less attenuation power. For epoxy/MnO₂ composites, 5mm thick samples show -12.2db RL for polyurethane and -20.6db. This value of -20.6dB is highest for all the composites used in different simulations. The greater reflection loss means the higher attenuation power of a material. It can be safely concluded that epoxy/MnO₂ composite is an effective shielding material in X-band region.

4.2 EMI Shielding in Ku – Band Studies:

4.2.1 Effective Permittivity:

In Ku band region, the effective permittivity values are modified for all the four composites as in X band region



Fig 4.7. Real Permittivity of Epoxy, MnO2 and epoxy/MnO2 Composites

In Epoxy composite with MnO_2 the real permittivity is modified. The value lies between 4 (the real permittivity value for Epoxy), and 13 (the maximum real permittivity value for MnO_2).



Fig 4.8. Imaginary Permittivity of Epoxy, MnO₂ and epoxy/MnO₂ Composites

The imaginary permittivity part is largely reduced for nanocomposite, when we compared it with imaginary permittivity value of MnO_2 . The main reason can be the very low value of imaginary permittivity ε'' of epoxy, which is 0.03 for all frequencies in Ku band range. As in a nanocomposite the main constituent is the polymer matrix, while the nano filler is present in small amount.

Permittivity Tables Ku band:

S.No	Frequency (GHz)	Real Permittivity (Poly Urethane) έ	Real Permittivity (SWCNT) é	$\begin{array}{c} \epsilon_{eff} \ (Effective \ Real \\ Permittivity \ of \\ Composite) \end{array}$	Imaginary Permittivity (Poly Urethane) ε''	Imaginary Permittivity (SWCNT) ε''	$\begin{array}{llllllllllllllllllllllllllllllllllll$
1	12.5	2.8	1.29	2.11	0.19	1.05	4.17E-01
2	12.9	2.8	1.28	2.11	0.19	1.05	4.17E-01
3	13.3	2.8	1.20	2.07	0.15	1.04	3.73E-01
4	13.6	2.8	1.20	2.07	0.13	1.04	3.51E-01
5	14.0	2.8	1.20	2.07	0.13	1.02	3.47E-01
6	14.4	2.8	1.19	2.06	0.13	1.02	3.47E-01
7	14.8	2.8	1.17	2.05	0.14	1.01	3.56E-01
8	15.2	2.8	1.16	2.05	0.14	1.01	3.56E-01
9	15.7	2.8	1.14	2.04	0.17	1.00	3.86E-01
10	16.1	2.7	1.11	1.97	0.16	0.99	3.73E-01
11	16.6	2.7	1.09	1.96	0.15	0.80	3.23E-01
12	17.0	2.7	1.08	1.95	0.14	0.96	3.46E-01
13	17.3	2.7	1.08	1.95	0.13	0.94	3.31E-01
14	17.9	2.6	1.05	1.89	0.12	0.93	3.18E-01
15	18	2.6	1.02	1.87	0.12	0.92	3.16E-01

Table 4.5: Real and Imaginary Effective Permittivity of 0.4% Polyurethane Composite with SWCNTs in Ku - Band Region

The permittivity values (both real $\dot{\epsilon}$ and imaginary ϵ'' parts), are changed during the formation of nanocomposites, then their pure components, i.e. Polyurethane and SWCNTs. For example, at 12.5GHz, the $\dot{\epsilon}$ value for polyurethane is 2.8 and for SWCNTs is 1.29. After composite formation the effective permittivity ϵ_{eff} is adjusted on 2.11. The imaginary part of permittivity modifies too. At 12.5GHz it is 4.17E-01 for nano composite, while 0.19 for Polyurethane and 1.05 for pure SWCNTs bundle.

S.No	Frequency (GHz)	Real Permittivity (Epoxy) é	Real Permittivity (SWCNT) é	$\begin{array}{c} \epsilon_{eff} \ (Effective \ Real \\ Permittivity \ of \\ Composite) \end{array}$	Imaginary Permittivity (Epoxy) ε''	Imaginary Permittivity (SWCNT) ε''	$\begin{array}{c} \epsilon_{eff} \ (Effective \ Imaginary \\ Permittivity \\ Composite) \end{array}$
1	12.5	4	1.29	2.72	0.03	1.05	2.32E-01
2	12.9	4	1.28	2.71	0.03	1.05	2.32E-01
3	13.3	4	1.20	2.67	0.03	1.04	2.30E-01
4	13.6	4	1.20	2.67	0.03	1.04	2.30E-01
5	14.0	4	1.20	2.67	0.03	1.02	2.26E-01
6	14.4	4	1.19	2.66	0.03	1.02	2.26E-01
7	14.8	4	1.17	2.65	0.03	1.01	2.25E-01
8	15.2	4	1.16	2.65	0.03	1.01	2.25E-01
9	15.7	4	1.14	2.63	0.03	1.00	2.23E-01
10	16.1	4	1.11	2.62	0.03	0.99	2.21E-01
11	16.6	4	1.09	2.61	0.03	0.80	1.86E-01
12	17.0	4	1.08	2.60	0.03	0.96	2.15E-01
13	17.3	4	1.08	2.60	0.03	0.94	2.12E-01
14	17.9	4	1.05	2.58	0.03	0.93	2.10E-01
15	18	4	1.02	2.56	0.03	0.92	2.08E-01

Table 4.6: Real and Imaginary Effective Permittivity of 0.4% Epoxy Composites with SWCNTs in Ku – Band Region

The real permittivity $\dot{\epsilon}$ value for Epoxy composite for SWCNTs is adjusted in the middle of the two values, i.e. for pure epoxy and SWCNTs. At 12.5GHz this value is 2.72 for nanocomposite, while for epoxy it is 4, and for SWCNTs it is 1.29. Similarly, at this frequency i.e. 12.5GHz, the imaginary permittivity of nanocomposite is 2.32E-01, while it is 0.03 for pure epoxy and 1.05 for SWCNTs.

S.No	Frequency (GHz)	Real Permittivity (Poly Urethane) έ	Real Permittivity (MnO ₂) é	ε _{eff} (Effective Real Permittivity of Composite)	Imaginary Permittivity (Poly Urethane) ε"	Imaginary Permittivity (MnO ₂) E''	$\begin{array}{ll} \epsilon_{eff} \ (Effective \ Imaginary \\ Permittivity & of \\ Composite) \end{array}$
1	12.5	2.8	13	5.49	0.19	19.1	1.21E00
2	12.9	2.8	12.9	5.47	0.19	18.9	1.20E00
3	13.3	2.8	11.8	5.25	0.15	18.3	9.86E-01
4	13.6	2.8	11.4	5.16	0.13	17.7	8.70E-01
5	14.0	2.8	11.6	5.20	0.13	17.6	8.69E-01
6	14.4	2.8	12.1	5.31	0.13	17.6	8.69E-01
7	14.8	2.8	11.4	5.16	0.14	17.7	9.26E-01
8	15.2	2.8	10.3	4.93	0.14	16.7	9.17E-01
9	15.7	2.8	9.7	4.80	0.17	16	1.07E-01
10	16.1	2.7	9.6	4.68	0.16	16.3	1.02E-01
11	16.6	2.7	9.2	4.59	0.15	16.8	9.72E-01
12	17.0	2.7	9.2	4.59	0.14	15.8	9.08E-01
13	17.3	2.7	9.4	4.64	0.13	15.4	8.51E-01
14	17.9	2.6	9.1	4.47	0.12	14.6	7.89E-01
15	18	2.6	9	4.45	0.12	14.2	7.85E-01

Table 4.7: Real and Imaginary Effective Permittivity of 0.4% Polyurethane Composite with MnO₂ in Ku – Band Region

Pure MnO₂ have high real and imaginary permittivity values, when compared to Polyurethane. Such as, at 12.5GHz real permittivity value for polyurethane is 2.8, while it is 13 for MnO₂. The imaginary permittivity value for Polyurethane is 0.19 at 12.5GHz, and 19.1 for MnO₂. The permittivity values for their nanocomposite is 5.49 (real part) and 1.21 (img part). These values are close to the values for polymer matrix than for the nanofiller. Which shows the constituent in large quantity has greater effect on the permittivity values of the composite.

S.No	Frequency (GHz)	Real Permittivity (Epoxy) é	Real Permittivity (MnO ₂) é	ε _{eff} (Effective Real Permittivity of Composite)	Imaginary Permittivity (Epoxy) ε"	Imaginary Permittivity (MnO ₂) E''	ε _{eff} (Effective Imaginary Permittivity of Composite)
1	12.5	4	13	6.66	0.03	19.1	2.30E-01
2	12.9	4	12.9	6.63	0.03	18.9	2.29E-01
3	13.3	4	11.8	6.37	0.03	18.3	2.29E-01
4	13.6	4	11.4	6.27	0.03	17.7	2.29E-01
5	14.0	4	11.6	6.32	0.03	17.6	2.29E-01
6	14.4	4	12.1	6.44	0.03	17.6	2.29e-01
7	14.8	4	11.4	6.27	0.03	17.7	2.29E-01
8	15.2	4	10.3	5.99	0.03	16.7	2.28E-01
9	15.7	4	9.7	5.83	0.03	16	2.28E-01
10	16.1	4	9.6	5.81	0.03	16.3	2.28E-01
11	16.6	4	9.2	5.70	0.03	16.8	2.28E-01
12	17.0	4	9.2	5.70	0.03	15.8	2.28E-01
13	17.3	4	9.4	5.75	0.03	15.4	2.27E-01
14	17.9	4	9.1	5.67	0.03	14.6	2.27E-01
15	18	4	9	5.64	0.03	14.2	2.26E-01

Table 4.8: Real and Imaginary Effective Permittivity of 0.4% Epoxy Composites with MnO₂ in Ku-Band Region

The effective permittivity (real part) ε_{eff} value for nanocomposite is 6.66 at 12.5GHz, while the imaginary part is 2.30E-01 for the same frequency. Comparing with the permittivity values of epoxy and MnO₂ at 12.5GHz both the real and imaginary part, we find the modification in these values.

4.2.2 Shielding Effectiveness:

In case of Ku band, all the four composites follow the reflection mechanism for electromagnetic shielding. The Reflection loss shown by these composites in 1-5mm thickness are shown in the following figures.



Fig 4.9. RL Values for 0.4% Polyurethane/SWCNTs Composites

The maximum RL value is recorded for 1mm thick composite sample is -14.1dB at 15.4GHz frequency. Three samples with 2-4mm thickness show less than 10dB reflection loss for all the frequencies in Ku band range. While 5mm thick sample shows increase in reflection loss with increasing frequency. The maximum RL is -12.5dB at 18GHz.



Fig 4.10. RL Values for 0.4% Epoxy/SWCNTs Composites

For epoxy composite with SWCNT, the maximum reflection loss RL is shown by 5mm thick sample, which is -16.3dB at 18 GHz. In these composites, 5mm thick sample shows the similar trend as shown by 5mm Polyurethane/SWCNTs composite, i.e. the RL increased with increase in frequency, hence maximum RL is at 18GHz, which is the maximum frequency in this study. 1mm thick sample gave maximum RL value of -10.5dB at 14.2GHz. While all the other thicknesses showed less than 10dB RL values for each frequency of Ku band.



Fig 4.11. RL Values for 0.4% PU/MnO2 Composites

In Polyurethane composite with MnO₂, the maximum reflection loss RL is -12.6dB, that is given by 4mm thick sample at 17.5GHz frequency. 5mm thick sample, also showed RL value of -11dB at 13.9GHz frequency. Other three samples, with thickness 1-3mm showed less than -10dB reflection loss.



Fig 4.12. RL Values for 0.4% Epoxy/MnO₂ Composites

The 4mm thick sample showed, maximum reflection loss RL value of -21.1dB at 15.6GHz frequency. This is the highest RL value of all the samples showed in Ku band region. The 5mm thick sample also gave good RL value of -19.4dB at 12.7GHz. Other samples with thickness 1-3mm showed 2-4dB reflection loss in Ku band region.

4.3 Comparison between SWCNT and MnO₂ Composites:

For comparison purpose, we have considered only those samples that gave maximum reflection loss values in X band in Ku band region of microwave frequency.

a. X-Band Region:



i. Polyurethane Composites with SWCNTs and MnO₂:

Fig 4.13. 1mm Thick Polyurethane Composite with SWCNTs in CST Studio Waveguide Model The simulations have run for 5 samples each with thickness from 1-5mm. However, we are comparing 1mm and 5mm thick samples of each composite, as these thicknesses showed comparatively better reflection loss values. It is noticed that for SWCNT bundles 1mm sample gave highest reflection loss value of -14.8dB at 10.7GHz region of X band, whereas the highest reflection loss value of -12.4dB is given by MnO₂ composite at 5mm thickness.



Fig 4.14: RL Values for PU/SWCNTs & MnO₂ Composites for 1mm and 5mm Thickness **ii. Epoxy with SWCNTs and MnO₂ Composites:**



Fig 4.15. RL Values for Epoxy/SWCNTs & MnO₂ Composites for 1mm and 5mm Thickness

In case of Epoxy composites with SWCNT bundles and MnO_2 the trend remains the same as is of Polyurethane composites. The CNTs give highest reflection loss of -11.9dB with minimum thickness (1mm), used in these simulations, though its value is smaller than its polyurethane counterpart. For MnO₂, the maximum Reflection loss is given by 5mm thick sample as also is the case in polyurethane, however this value is much greater than PU composite. The RL shown by this sample is -20dB at 11.1GHz, means 99% absorbance at this frequency. This value for Epoxy composite with MnO₂ is the largest value for all the samples simulated in X band frequency range.



Fig 4.16. 5mm thick Polyurethane Composite with SWCNTs in CST Studio Waveguide Model

b. Ku band Region:

In Ku band region we are taking 5 samples in consideration, namely 1mm and 5mm thick samples for polyurethane composite with SWCNTs and MnO_2 for each and one for 4mm Polyurethane composite for MnO_2 , as it gives high reflection loss value in Ku band.

i. PU with SWCNTs and MnO₂ Composites:



Fig 4.17. RL Values for PU/SWCNTs & MnO₂ Composites for 1mm, 4mm and 5mm Thickness In Ku band region, PU/SWCNTs show the same trend as in X band. The 1mm thick sample has greatest reflection loss value of -14.1dB at 14.1GHz frequency, but in MnO₂ composites, the trend changes a little. Instead of 5mm sample thickness, 4mm thick sample give higher reflection loss. This value is -12.6dB at 17.5GHz.

ii. Epoxy with SWCNTs and MnO₂ Composites



Fig 4.18. RL Values for Epoxy/SWCNTs & MnO₂ Composites for 1mm, 4mm and 5mm Thickness In epoxy – SWCNTs composites the 5mm thick sample gives higher reflection loss than 1mm sample, which is -16.3dB at 18GHz frequency. In Epoxy composite with MnO₂ composite 4mm sample give high reflection loss value of -21.1dB, corresponds to 99% energy attenuation, while the 5mm sample also show -19.4dB, reflection loss which is higher than any of the other composite studied in Ku band frequency region.

4.4 Conclusion:

SWCNT composites with both polyurethane and epoxy as polymer matrix give high reflection loss values with minimum thickness of 1mm of all the samples used in the X band. The maximum absorption is -14.8dB corresponding to 96% shielding. This is an important finding as the materials with small thickness but good absorption tendency of microwaves can be used as coatings, and polyurethane as polymer matrix can be excellent choice for composite formation, as it is the most competent candidate in modern coatings for buildings, vehicles and aircrafts.

MnO₂ composites show greater reflection loss values with increasing thickness of the samples. Therefore, 5mm samples give higher reflection loss values for both Polyurethane and Epoxy. However, 5mm sample of epoxy with MnO₂ give as high as -20.6 dB of reflection loss corresponds to 99% of attenuation, but with Polyurethane this value is only -12.4dB. Epoxy matrix is an important polymer as it has strong mechanical strength and better thermal resistance. With high reflection loss values in X band region the MnO₂ and Epoxy composites can be effective candidates as MAMs.

In Ku band region, which is mostly related to satellite communication, the MnO₂ epoxy composite give high value of reflection loss for both 4mm and 5mm thickness. 5mm sample of epoxy MnO₂ composite with -21db, at 15.4GHz frequency make it an important candidate to be used as MAM for the applications in this frequency region. With polyurethane the results for MnO₂ composites are not very promising.

The SWCNTs polyurethane 1mm composites maintain highest reflection loss value of -14.1dB in Ku band region as they are in X band region. But with epoxy composites 5mm sample of SWCNT bundles show increasing Reflection loss value with increasing frequency, with maximum RL of -16.3dB at 18GHz. So we can say that for epoxy SWCNTs the increasing thickness of the sample is the contributor in high value of reflection loss.

So we can conclude that SWCNTs are good nano fillers for polyurethane for 8-18GHz frequency range. The enhanced microwave absorption properties with low thickness of sample material is an advantage to use SWCNTs with polyurethane as coating material. However, SWCNTs are expensive and not easily available materials. Also, some other nanomaterials should be used with SWCNTs to enhance their microwave absorbing tendency, as they showed maximum of -14.8db reflection loss value. While MnO_2 is a better filler for epoxy in 8-18GHz
range. It can be safely concluded that MnO_2 which is economically cheap and easily available material with epoxy can be used as effective MAM in coatings.

4.5 Future Perspective:

MAMs have important military applications especially as radar absorptive materials. The current research is an important step forward in this regard. It is possible to design nanocomposite, if we know the permittivity values of the polymer matrix and the nanofiller through effective medium theories. The waveguide computational model created in this research can be used to find out the microwave absorption behavior of almost any material in 8-18GHz frequency range. The results can be used to synthesize and develop better future MAMs once their microwave absorption tendency is predicted through computational model.

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