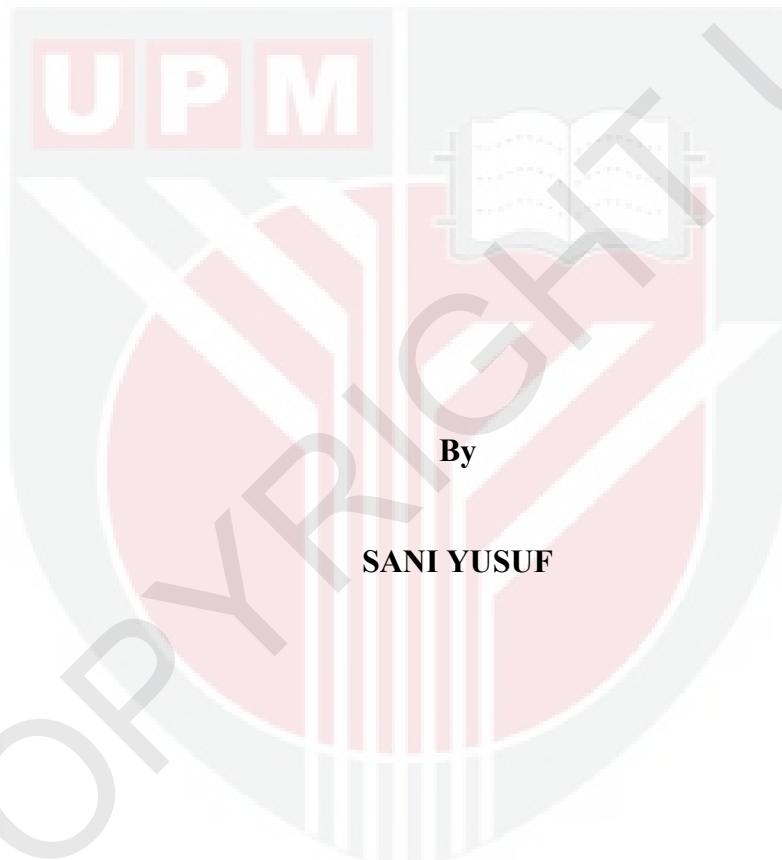




**SYNTHESIS AND ELECTROMAGNETIC CHARACTERIZATION OF
HIGH FREQUENCY RADAR ABSORBING MATERIALS USING
NANOSTRUCTURED SPINEL FERRITES NANOCOMPOSITE**



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

March 2024

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Doctor of Philosophy

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NANOSTRUCTURED SPINEL FERRITE NANOCOMPOSITES**

By

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March 2024

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Radar absorbent materials (RAMs) have been studied and developed to reduce or eliminate harmful electromagnetic radiation in many applications. Scientists have synthesized and analyzed numerous nanocomposites for effective radar-absorbing nanocomposites, but factors like impedance matching, absorber weight, and composite thickness issues require further research. An alternative approach is proposed for the development of a novel spinel ferrite of $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$, $\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$, and $\text{Mg}_{0.8}\text{Zn}_{0.1}\text{Co}_{0.1}\text{Fe}_2\text{O}_4$. Spinel ferrite's low dielectric loss and magnetic coercivity decrease microwave absorption properties, necessitating the incorporation of Calcium titanium oxide (CTO), Multiwall carbon nanotubes (MWCNT), and Molybdenum disulfide (MoS_2) into the prepared ferrite composition to improve the microwave absorption properties of the nanocomposites. The spinel ferrite and CTO were synthesized via a co-precipitation method and hydrothermally with the MWCNT by the acid functionalization process. This study aims to create microwave-absorbing material compositions ranging from 8 to 18 GHz and investigate the nanostructured

and microwave properties of the synthesized $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$ @CTO @MWCNT (A3), $\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$ @CTO@MoS₂ (B3), and Mg_{0.8}Zn_{0.1}Co_{0.1}Fe₂O₄@CTO (C3) nanocomposites at various thicknesses. The focus is on radar absorption and EMI shielding, with the aim of developing suitable absorbers and enhancing these properties consistently with the radar frequency bands. The nanocomposite samples were mixed with epoxy resin and placed in a manufacturing sample holder. XRD analysis revealed a single-phase ferrite in the A series nanocomposite, with crystallite sizes ranging from 55.6 to 75.3 nm. XRD reveals the hexagonal phase of synthesized B3 nanocomposites, with three separate diffraction peaks for MoS₂ and a crystallite size range of 55.6 to 74.2 nm. The C-series hybrid nanocomposite samples show no impurity phases in the XRD spectra. Raman spectra show peaks that are consistent with the single crystalline phase of prepared ferrites, CTO, MWCNT, and MoS₂. The XRD patterns, Raman spectra, and FTIR indicate the successful incorporation of both spinel ferrites, CTO, MWCNT, and MoS₂ phases in the nanocomposite. Both A, B, and C series samples show a decreased pattern of saturation magnetization (M_s), indicating the required superparamagnetic "S" form. The saturation magnetization of A-series samples decreased from 21.838 to 11.080 emu/g, B-series samples from 36.715 to 20.103 emu/g, and C-series samples from 79.93 to 15.69 emu/g. The A3 nanocomposite showed the highest reflection loss (RL) value of -39.12 dB (99.98% absorption) at 13.3 GHz with a thickness of 2 mm. The B3 hybrid nanocomposite reached the highest RL of 30.8 dB at 13.3 GHz with a thickness of 3 mm and a bandwidth of 2.5 GHz. The as-synthesized C3, with a thickness of 2 mm, demonstrated a maximum RL value of -24.0 dB around 11.0 GHz and a bandwidth of 2.0 GHz, ranging from 12.1 GHz to 10.1 GHz. The A3-prepared nanocomposite is the best candidate for microwave and radar absorption. The

encouraging results suggest that the MWCNTs and the composite's dielectric properties improved microwave absorption at microwave frequencies. The synthesized nanocomposites were expected to be very useful in many applications, particularly in military applications such as radar cross-section reduction and the prevention of electromagnetic interference.

Keyword: Co-Precipitation Method, Microwave Absorption, Nanocomposite, Radar Absorbent Materials (RAMs) and Reflection Loss.

SDG: GOAL 9: Industry Innovation and Infrastructure

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai
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**SINTESIS DAN CIRI-CIRI ELEKTROMAGNETIK BAHAN MENYERAP
RADAR BERKEKERAPAN TINGGI MENGGUNAKAN
NANOSTRUCTURED SPINEL FERIT NANOKOMPOSITS**

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Bahan penyerap radar (RAM) telah dikaji dan dibangunkan untuk mengurangkan atau menghapuskan sinaran elektromagnet berbahaya dalam banyak aplikasi. Para saintis telah mensintesis dan menganalisis banyak nanokomposit untuk nanokomposit menyerap radar yang berkesan, tetapi faktor seperti padanan impedans, berat penyerap dan isu ketebalan komposit memerlukan penyelidikan lanjut. Pendekatan alternatif dicadangkan untuk pembangunan ferit spinel novel $Cu_{0.5}Ni_{0.5}Fe_{1.9}Mn_{0.1}O_4$, $Ni_{0.6}Co_{0.4}Fe_{1.8}Zn_{0.2}O_4$, dan $Mg_{0.8}Zn_{0.1}Co_{0.1}Fe_2O_4$. Kehilangan dielektrik spinel ferrite yang rendah dan koersiviti magnetik mengurangkan sifat penyerapan gelombang mikro, memerlukan penggabungan Kalsium titanium oksida (CTO), tiub karbon berbilang dinding (MWCNT), dan Molibdenum disulfida (MoS_2) ke dalam komposisi ferit yang disediakan untuk meningkatkan sifat penyerapan gelombang mikro nanokomposit. Ferit spinel dan CTO telah disintesis melalui kaedah kerpasan bersama dan secara hidrotermal dengan MWCNT melalui proses kefungsian asid. Kajian ini bertujuan untuk mencipta komposisi bahan yang menyerap gelombang mikro antara 8

hingga 18 GHz dan menyiasat sifat struktur nano dan gelombang mikro Cu_{0.5}Ni_{0.5}Fe_{1.9}Mn_{0.1}O₄@CTO@MWCNT (A3), Ni_{0.6}Co_{0.4}Fe_{1.8}Zn_{0.2}O₄@CTO@MoS₂ (B3), dan Mg_{0.8}Zn_{0.1}Co_{0.1}Fe₂O₄@CTO (C3) nanokomposit pada pelbagai ketebalan. Tumpuan adalah pada penyerapan radar dan perisai EMI, dengan tujuan untuk membangunkan penyerap yang sesuai dan meningkatkan sifat ini secara konsisten dengan jalur frekuensi radar. Sampel nanokomposit dicampur dengan resin epoksi dan diletakkan di dalam bekas sampel pembuatan. Analisis XRD mendedahkan ferit satu fasa dalam nanokomposit siri A, dengan saiz kristal antara 55.6 hingga 75.3 nm. XRD mendedahkan fasa heksagon nanokomposit B3 yang disintesis, dengan tiga puncak pembelauan berasingan untuk MoS₂ dan julat saiz kristal 55.6 hingga 74.2 nm. Sampel nanokomposit hibrid siri C tidak menunjukkan fasa kekotoran dalam spektrum XRD. Spektrum Raman menunjukkan puncak yang konsisten dengan fasa kristal tunggal ferit yang disediakan, CTO, MWCNT, dan MoS₂. Corak XRD, spektrum Raman, dan FTIR menunjukkan kejayaan penggabungan kedua-dua fasa spinel ferit, CTO, MWCNT, dan MoS₂ dalam nanokomposit. Kedua-dua sampel siri A, B, dan C menunjukkan corak penurunan kemagnetan tepu (M_s), menunjukkan bentuk "S" superparamagnet yang diperlukan. Pemagnetan tepu bagi sampel siri A menurun daripada 21.838 kepada 11.080 emu/g, sampel siri B daripada 36.715 kepada 20.103 emu/g, dan sampel siri C daripada 79.93 kepada 15.69 emu/g. Nanokomposit A3 menunjukkan nilai kehilangan pantulan (RL) tertinggi iaitu -39.12 dB (99.98% penyerapan) pada 13.3 GHz dengan ketebalan 2 mm. Nanokomposit hibrid B3 mencapai RL tertinggi 30.8 dB pada 13.3 GHz dengan ketebalan 3 mm dan lebar jalur 2.5 GHz. C3 as-sintesis, dengan ketebalan 2 mm, menunjukkan nilai RL maksimum -24.0 dB sekitar 11.0 GHz dan lebar jalur 2.0 GHz, antara 12.1 GHz hingga 10.1 GHz. Nanokomposit yang disediakan A3 adalah calon terbaik untuk penyerapan gelombang

mikro dan radar. Keputusan yang menggalakkan menunjukkan bahawa MWCNT dan sifat dielektrik komposit meningkatkan penyerapan gelombang mikro pada frekuensi gelombang mikro. Nanokomposit yang disintesis dijangka sangat berguna dalam banyak aplikasi, terutamanya dalam aplikasi ketenteraan seperti pengurangan keratan rentas radar dan pencegahan gangguan elektromagnet.

Kata kunci: Kaedah Kerpasan Bersama, Penyerapan Gelombang Mikro, Nanokomposit, Bahan Penyerap Radar (RAM) dan Kehilangan Pantulan.

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LIST OF SYMBOLS

H_c	Magnetic Coercivity
M_r	Remanence
M_s	Saturation Magnetizations
V_{cell}	Volume of a Unit Cell
Z_0	Impedance in Air
Z_{in}	Substance Impedance
ϵ'	Real Complex Permittivity
μ'	Real Complex Permeability
μ''	Imaginary Complex Permeability
μ_0	Vacuum Permeability
μ_B	Bohr magneton
ϵ_∞	Relative dielectric permittivity
K	magneto-crystalline anisotropy constant
$\tan\delta$	Loss-Tangen
α	Attenuation Constant
ϵ''	Imaginary complex permittivity
λ	Lamda
σ	Electric Conductivity
τ	Polarisation Relaxation Time
ω	Angular Frequency

LIST OF ABBREVIATIONS

a, b, c	Lattice parameters or Lattice Constant
DTA	Differential Thermal Analysis
EDX	Energy Dispersive X-Ray
EM	Electromagnetic
EMI	Electromagnetic Interference
EMW	Electromagnetic Wave
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transforms Infrared Spectroscopy
GHz	Gigahertz
hkl	Miller indices
HRTEM	High Resolution Transmission Electron Microscopy
MA	Microwave Absorbers
MAMs	Microwave Absorbing Materials
MOS ₂	Molybdenum Sulphide
MWCNTs	Multiwall Carbon Nanotubes
RAMs	Radar Absorbing Materials
RL	Reflection Loss
SAED	Selected Area Electron diffraction
SF	Spinel Ferrite
TEM	Transmission Electron Microscope
TGA	Thermogravimetric Analysis
VNA	Vector Network Analyzer
VSM	Vibrating Sample Magnetometer
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Introduction

Microwave absorption materials are useful substances with minimal reflection and dispersion coefficients that can absorb microwaves and electromagnetic energy. The fundamental idea of microwave absorbers (MA) is to first transform microwave energy into another sort of energy using a specific physical mechanism, and then transform that other kind of energy back into heat energy using a dissipation motion. Unquestionably, microwaves are composed of rapidly oscillating, perpendicular magnetic and electric fields. They drew an increasing amount of attention in order to utilize the unwanted microwave radiation. The essential idea behind microwave absorption is the fusion of magnetic and dielectric loss processes to transform incoming microwave energy into other energy. Based on their fundamental operating principles, microwave absorption materials may be classified as interference or absorption types (Cao et al., 2020a). When an incident electromagnetic wave penetrates the material's interior, electromagnetic waves are typically reflected, absorbed, and transmitted (Shu et al., 2021a). Impedance matching and attenuation properties are often the two factors to be taken into account when evaluating electromagnetic wave absorption performance (Wu et al., 2021). A material should have an impedance that is equal to that of empty space in order to allow electromagnetic waves to penetrate it as much as is feasible. The three main types of losses are magnetic loss, conductance loss, and dielectric loss. Materials with

nanoscale dielectric, magnetic, or composite structures may be used to absorb microwaves.

Two materials, each of which has distinct chemical and physical characteristics, are combined to form a composite material (Ahmad et al., 2019). It is produced from a new material that is considerably different from the substance it was originally composed of when these two distinct components, termed the matrix and reinforcement, are combined (Barkoula et al., 2008). Low installation costs, lesser toxicity, and the capacity to construct complex components in one piece are some of the advantages of composites. Composites have improved aeronautical efficiency, and a nation's air force is looking into them to increase the effectiveness and efficiency of its squadrons and air crew. In fighter aircraft, speed and visibility are important for mission fulfilment. Fighter planes must penetrate enemy territory without leaving any trace of an invasion. The use of composites may make aircraft less apparent to an opponent's detection system. "Stealth technology" (ST) or low-observable technology is used to make ships, planes, missiles, submarines, and satellites undetectable by radar, sonar, infrared, and other detection systems (Palmer et al., 2012). Since the advent of contemporary warships, ST has grown to be one of the most important research topics in the field of military equipment. Various studies on the use of ST in armament systems such as missiles, planes, and battleships have been conducted (Kim et al., 2006). Composites are also employed in the manufacture of stealth aircraft as radar absorbing materials (RAMs).

1.2 Research Background

The development of an acceptable solution for thin and broadband RAMs is receiving increasing attention from researchers as a consequence of recent advances in technology. According to reports on the topic, utilizing a single layer to offer a -10 dB absorption bandwidth and a thinner layer is inadequate. However, sophisticated electromagnetic (EM) techniques such as excellent heterogeneous composite RAMs have been described as an efficient step towards the development of a thin and broadband Radar wave absorber for stealth technology. There is a lot of interest in radar absorbent materials (RAMs) since military organizations employ them to provide warheads and fighter aircraft stealth characteristics. RAMs are devices that alter electromagnetic waves by absorbing and dissipating heat. They may be used for a variety of exciting tasks, including environmental preservation, radar stealth, microwave darkrooms, electromagnetic compatibility, broadcasting, television visuals, and interference with multi-story structures. The main objectives of stealth and camouflage designs are to minimize the visibility of an aircraft or any of its components to hostile radar surveillance systems as well as to decrease the Radar Cross Sections (RCS) of prospective targets. There are two ways to achieve "invisibility": (i) Radar Cross Section, that is, reduction by Shaping, which assesses a target's capacity to reduce back scattered radar signals towards the radar receiver; and (ii) RAMs, which absorb incoming radar signals and cover the surface of the vehicles or hardware. The first approach has obvious drawbacks for applications that dynamically change the required form. Therefore, the development of adaptive and intelligent architectures with weight-limited RAMs is crucial (Liang et al., 2004). An absorbent material must be able to cancel out both the electric and magnetic

components in order to absorb them (Ting et al., 2012a; Zhong et al., 2016). Radar-absorbing substances absorb radar signals, reducing or eliminating reflection. They lessen the energy that the absorbing radar reflects back. Radar-absorbing materials (RAMs), often referred to as microwave-absorbing materials, come in two varieties: magnetic and dielectric-absorbing materials. An excellent RAM needs to be lightweight, thin, and able to handle a wide frequency range. This indicates that absorption is mostly used for its dielectric and magnetic properties. Thus, microwave absorption may be achieved using nanostructured dielectrics, magnetic materials, and composite materials. Nonmetals have been used in the manufacture of aeroplanes, primarily for the wings, tails, and control surfaces. Therefore, wings, tails, and control surfaces are the most prevalent nonmetallic materials. Examples include Horizontal boron/epoxy tail skins used in the manufacturing of F14 aircraft, horizontal and vertical boron/epoxy tail skins for F15 skins, graphite-epoxy horizontal and vertical skins and control surfaces (F16 aircraft), graphite-epoxy wing, forward fuselage, and control surfaces (Av-8B), and graphite-poxy control surfaces (Boeing 757). These materials may be integrated or covered with suitable RAMs to boost microwave absorption or minimize reflection (Ting et al., 2012a).

Despite their outstanding absorption capabilities, magnetic absorbers typically have excessively high densities. Magnetic materials that are sensitive to magnetic loss mechanisms include transition metals such as Fe, Ni, Co, Mn, Cu, Cr, and Zn. The most frequent magnetic loss processes are hysteresis loop (from irreversible magnetization that may be ignored with a modest external or applied magnetic field), domain wall resonance (which often occurs in the frequency range of 1-100 MHz), natural resonance, and eddy current losses (Liu et al., 2016). Dielectric absorbers lack

the absorptivity of magnetic absorbers but are much lighter in terms of weight. Absorption is influenced by dielectric loss mechanisms such as electronic or atomic polarisation, orientation (dipolar) polarisation, ionic conductivity, and inter-facial or space charge polarisation. When electric and/or magnetic dipoles align with an EM field, the material loses heat. This is called absorption loss, and it depends on the material's conductivity, dielectric permittivity, and magnetic permeability (Munir et al., 2015). When employed separately, both magnetic and dielectric materials have low absorption(Giannakopoulou et al., 2002; Wang et al., 2013). Dielectric losses (μ'') and magnetic losses (ϵ'') account for the majority of the absorption. As a result, RAM that contains both magnetic and dielectric fillers may provide more absorbent material.

A variety of constituents are mixed to provide a variety of structural and microwave absorption properties in nanostructured composite materials. For the purpose of creating magnetic nanocomposites, fillers such as ceramics (e.g., spinel ferrite and hexagonal ferrite), metal alloys, metal nanoparticles (e.g., Fe, Ni, and Co), metal oxides, and metal compounds are utilized (Atiq et al., 2016; Jin et al., 2018; Mohammadian et al., 2018). On the other hand, synthesizing nanostructured absorber materials is challenging. The choice of appropriate filler materials is required in addition to the host matrix, which is a continuous solid phase in which guest particles (atoms, molecules, ions, and so on) are implanted. The choice of suitable production methods and measurement strategies are additional factors. Spinel ferrites have the highest microwave absorption capabilities of any material known due to their high electromagnetic permeability and low dielectric loss (Xu et al., 2019a; Zavislyak et al., 2015). They have generated significant attention for high-frequency devices Zhou et al., 2013). Ferrites, however, have disadvantages such as increased frame density,

decreased dielectric losses, and a small or narrow absorption band (Yanhua et al., 2012). In order to achieve a broad absorption bandwidth, ferrite-based composites have a weight and thickness drawback, which limits their usage in fields like stealth and space technology. This thesis focuses on nanostructured composites that comprise dielectric, conductive, and magnetic absorption properties. Nanocomposites of various ferrites with MWCNT, two-dimensional conducting materials like molybdenum sulphide (MoS_2), and polymers have been thoroughly researched to address these difficulties (Abbas et al., 2016; Yu et al., 2013a).

1.3 Research Gap

It is still difficult to design a multi-component, innovative, lightweight microwave absorber. According to our knowledge, prior research on the three elements of a single-layer absorber as nanocomposite materials comprised of spinel ferrites, calcium titanium oxide (CTO), multiwall carbon nanotubes (MWCNTs), or spinel ferrites, calcium copper titanate CTO, and molybdenum sulphide (MOS_2) has never existed. Using a ternary composite of spinel ferrite-doped CTO with MWCNTs and spinel ferrite-doped CTO with MOS_2 powders, we aim to develop a lightweight multicomponent single-layer absorber with exceptional microwave absorption characteristics in the X and Ku band frequencies. The magnetic and microwave-absorbing properties of composites made from various kinds of materials are carefully investigated.

1.4 Problem Statement

The main goals of the ongoing research effort are to design an effective composite RAM with a large bandwidth and a thin architecture for microwave absorption and stealth applications, as well as investigate efficient methods for developing innovative composite RAMs. Some of the major difficulties and problems involved in creating thin and broad-band radar wave absorbers are as follows:

- i. Even though various studies have been conducted to develop nanocomposite RAMs, little attention has been paid to the effects of particle size and percentage variation on radar absorption.
- ii. Various research teams have developed different types of RAMs, but developing a suitable thin and wide-bandwidth radar wave absorber that balances composition and thickness-bandwidth tradeoffs remains a challenge.
- iii. Researchers have looked at a number of techniques, including the hydrothermal method, the sol-gel method, and others, to circumvent such thickness-bandwidth tradeoffs. But for researchers, choosing an appropriate composite RAM, number of layers, layer preferences, and thickness optimization for layer absorbers is a challenge.

The real challenge to achieving these goals is a lack of comprehensive scientific understanding of how the molecular constituents of materials and their mixtures reflect, absorb, and transmit incident microwave radiation. Therefore, extensive investigations, like the ones performed in this study effort, on the microwave absorption characteristics of different materials need to be synthesized and examined in order to locate the microwave absorbing materials with high absorptive capacity and suited for frequencies of 8 to 18 GHz. This thesis suggests a method for creating composite RAMs that combines spinel ferrite, calcium titanium oxide (CTO), multiwall carbon nanotube (MWCNT), and molybdenum sulphide (MoS_2) in order to produce an effective reflection loss (RL), a broad bandwidth, and a low coating thickness.

1.5 Selection of the material

The decision to use three different types of spinel ferrites ($\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$, $\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$, and $\text{Mg}_{0.8}\text{Zn}_{0.1}\text{Co}_{0.1}\text{Fe}_{1.9}\text{O}_4$) and incorporate them with different composites (CTO, MWCNT, and MoS₂) for microwave absorption studies involves several considerations:

1. Exploration of Composite Diversity:

Each combination of spinel ferrite and composite additive creates a unique composite material with potentially different microwave absorption properties.

The research work is interested in exploring a wide range of composite materials to understand how the interactions between different spinel ferrites and additives influence microwave-absorption.

This approach allows for a broad exploration of the composite landscape to identify promising materials for microwave absorption applications.

2. Tailoring Microwave Absorption Properties:

Different spinel ferrites and composites have varying microwave absorption characteristics. Incorporating them into different combinations may lead to tailoring the microwave absorption properties for specific frequency ranges or applications.

For example, Cu_{0.5}Ni_{0.5}Fe_{1.9}Mn_{0.1}O₄ have been chosen for its potential multiferroic properties, Ni_{0.6}Co_{0.4}Fe_{1.8}Zn_{0.2}O₄ for its magnetic properties, and Mg_{0.8}Zn_{0.1}Co_{0.1}Fe_{1.9}O₄ for its structural stability. Adding CTO, MWCNT, or MoS₂ can further modify these properties for optimized microwave absorption.

3. Comparative Studies:

Studying multiple combinations allows for comparative analyses to determine the most effective composite for microwave absorption. These lead to compares of the microwave absorption properties of each composite to understand which combination exhibits the best performance. This comparative approach helps to understand whether the observed effects stem primarily from the type of spinel ferrite, the additive, or their combined interaction in the microwave absorption process.

This approach allows for a broad exploration of composite diversity, tailoring properties for multiple applications, conducting comparative studies, exploring potential synergistic effects, and optimizing composite performance. The goal is to gain a comprehensive understanding of how both spinel ferrites and additives contribute to the properties of composite materials, guiding the development of advanced materials with tailored and enhanced properties. This approach allows for a broad exploration of the composite landscape and the development of advanced materials with tailored properties. Table 1.1 lists the justifications for selecting certain materials:

Table 1.1: List of substances and the justifications for selecting them as absorbent substances

Materials	Reason
Copper Nickel Manganese Ferrite($\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$)	$\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$ is a soft magnetic material with high electrical resistance, superparamagnetic behavior, high saturation magnetization, low coercivity, exceptional chemical stability, and outstanding mechanical hardness. They are the most adaptable technical materials, particularly suitable for high-frequency applications due to their high resistivity. Suitable for use in inductors, magnetic heads, and high-frequency transformers. They also benefit from applications with low to microwave frequencies and low to high permeabilities. It is quite helpful to have a high resonant frequency, particularly in the microwave frequency range.
Nickel Cobalt Zinc ferrite ($\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$)	$\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$ is a high-performance permanent magnet that has low coercivity, a significant saturation magnetization, a high Curie temperature, and a large magneto crystalline anisotropy. Furthermore, it has excellent chemical and thermal stability, as well as corrosion resistance. They could work well as a surface material for RF/THz frequency selection (Arya et al., 2021). They also serve as low-magnetic and low-dielectric-loss materials for absorbing electromagnetic waves. Thus, they can serve as microwave absorbers in the GHz frequency range.
Magnesium, Zinc Cobalt Ferrite ($\text{Mg}_{0.8}\text{Zn}_{0.1}\text{Co}_{0.1}\text{Fe}_2\text{O}_4$)	$\text{Mg}_{0.8}\text{Zn}_{0.2}\text{Co}_{0.1}\text{Fe}_2\text{O}_4$ is a superparamagnetic soft magnetic material that exhibits high saturation magnetization behavior and a low coercivity value. A high anisotropy field makes it possible to effectively absorb microwave energy through the precession of the magnetic moment. Additionally, it produces high complex permeability values across a broad frequency range due to its huge saturation magnetization and high Snoek's limit. As a result, it makes $\text{Mg}_{0.8}\text{Zn}_{0.1}\text{Co}_{0.1}\text{Fe}_2\text{O}_4$ a very effective thin absorber that operates in the high-frequency region.
Calcium Titanium Oxide (CTO)	CTO is a ferroelectric material with excellent dielectric permittivity, high chemical stability, perfect band edge placement, significant catalytic activity, minimal toxicity, and simplicity of manufacture. As an electro-ceramic material, it has drawn a lot of interest. It is frequently referred to as a microwave ceramic due to its dielectric response in the microwave band. It may function as one of the basic elements in ferroelectric ceramics. The low dielectric permittivity, however, may be reduced by integrating it into an insulating matrix to guarantee little reflection from the sample's front surface.
Molybdenum disulfide (MoS_2)	Because of its outstanding interfacial polarisation effect, high electrical conductivity, and flexible structural design, molybdenum disulfide (MoS_2) has long been recognized as a competitive microwave absorption material. MoS_2 is regarded as a particularly promising microwave absorption material due to its vast specific surface area and the high number of active sites that may interact with EM radiation.
Multiwalled Carbon Nanotubes (MWCNT)	Multiwalled carbon nanotubes have the following characteristics: minimal percolation, high electrical properties, and a high specific surface area. Additionally, since it is integrated into polymers, it provides considerable design and control freedom for microwave absorption characteristics. Changes can be made to MWCNT-polymer composites by changing the loading fractions, matrix materials, complex permittivity, and loss tangent. CNT inclusion may also improve the composite's thermal stability and provide it with high real and imaginary permittivity. Thus, the values of complex permittivity may be greatly raised by utilizing modest quantities of MWCNTs. Also, for good microwave absorption, the conductivity needs to be at its best, since poor conductivity could lead to only partial microwave absorption.

1.6 Objectives

The primary aim of the research endeavor is to create RAMs (Radar Absorbing Materials) that are adaptable, light in weight, thin, and flexible. In pursuit of this objective, this research work conducted the synthesis and examination of nanostructured magnetic materials, dielectric materials, and conducting materials using co-precipitation and hydrothermal techniques. Below are the particular goals that need to be tackled:

- To develop a flexible, lightweight, thin, and reconfigurable RAM. Towards this goal, Nano-structured magnetic materials (Spinal ferrite), dielectric and conducting materials are investigated.
- To synthesize composites of nano-structured spinel ferrite incorporated into CTO ceramic, MWCNT, and MoS₂ (SF/CTO, SF/CTO/MWCNT, and SF/CTO/MoS₂) using coprecipitation and hydrothermal processing methods.
- To investigate the properties and high-frequency microwave absorption characteristics of nano-structured SF/CTO, SF/CTO/MWCNT, and SF/CTO/MoS₂ nanocomposites.
- To investigate the role of CTO, MWCNT, and MoS₂ in the microwave absorption performance of a reflection loss (*RL*) of ≤ 30 dB of the prepared nanocomposites materials.

1.7 Scope and Limitations of the Research

The present research investigation aims to develop high-absorbing material compositions that can absorb microwave radiation at frequencies ranging from 8 to 18 GHz. Several microwave-absorbing composites were developed using the coprecipitation strategy as well as the hydrothermal technique. Different materials with various compositions, weight percentages, and thicknesses were being synthesized. However, this thesis will only provide an in-depth review of exceptional

and selected results. The materials being discussed are divided into three different series (A, B, and C): as-synthesized Copper-Nickel-Manganese ferrites ($\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_{1.9}\text{Mn}_{0.1}\text{O}_4$) mixed with as-synthesis calcium titanium oxide (CTO), and acidified Multiwall carbon nanotubes (MWCNT) (SF/CTO/MWCNT-A-series). As-synthesized nickel-cobalt-zinc ferrite ($\text{Ni}_{0.6}\text{Co}_{0.4}\text{Fe}_{1.8}\text{Zn}_{0.2}\text{O}_4$) mixed with as-synthesis CTO, and Molybdenum disulfide (MoS_2) (SF/CTO/MoS₂-B-series). and as-synthesized magnesium-Zinc-Cobalt ferrite ($\text{Mg}_{0.8}\text{Zn}_{0.1}\text{Co}_{0.1}\text{Fe}_2\text{O}_4$) mixed with as-synthesized CTO (SF/CTO -C-series). MWCNT and MoS₂ were purchased from commercial sources. The initial raw metal oxide powder materials used to make ferrite materials were weighted and stirred with magnetic stirrers, utilizing coprecipitation processes to reach nanoscale particle size. X-Ray Diffraction (XRD) was used to investigate the structural behavior of the prepared nanocomposite samples. TGA and DTA were used for the thermal analysis, while FESEM/EDX, HRTEM, SAED, RAMAN, FTIR, VSM, and VNA were used to analyze the morphology, microstructural, magnetic, and microwave absorption characteristics of the nanocomposite's samples. Coaxial transmission waveguide methods were used to investigate how MWCNT and MOS₂ filler concentrations affect the microwave absorption characteristics of the prepared nanocomposites.

1.8 **Outcome of the research**

Significant progress will be made in addressing the need for high-frequency microwave absorber material. Modern technological applications such as stealth technology and microwave electronics devices will profit from the created chemical. The topic of electromagnetic interference was also covered in this research.

1.9 An overview of nanomaterials used in microwave absorption

Electromagnetic (EM) microwave absorption materials are dependent on a combination of magnetic loss and dielectric loss (particularly polarisation, such as electron polarisation and interface polarisation) as well as other processes to generate microwave absorption. Magnetic materials (e.g., magnetic metals, alloys, and oxides), carbon-based materials (e.g., CNTs, MWCNT, graphene, carbon fiber), composite materials (e.g., magnetic carbon composite), and other nanomaterials (e.g., MoS₂, MXene, sulphide, nitride, and carbide) with unusual structures are currently the primary focus of microwave absorption material synthesis and design. In accordance with the class of materials, this thesis solely discusses the fundamental elements and particular examples of the numerous absorbing materials.

1.10 Outlines of the chapters

This thesis consists of six (6) chapters and an Appendix. Chapter one gives a general introduction to microwave absorbing materials (MAMs) and their applications, radar absorbers, and composite materials used for this research. Followed by the Research Gap, the statement of the problem, the objectives of the study, the scope and limitations of the study, and overview of nanomaterials used in microwave absorption. The research and reviews from earlier studies on microwave absorbers and RAM are presented in Chapter 2. The performance and examples of magnetic, dielectric, and composite materials are further addressed in this chapter. Additionally, discussions are had on the synthesis, microwave absorption capabilities, and relevant literature of both Molybdenum Disulfide (MoS₂) and Carbon Materials. In addition to the basic theory

of microwave absorption, the origin of the material, magnetization principles, dielectric substances, and material properties are further detailed in Chapter 3. Chapter 4 addressed the methods for figuring out the microwave and reflection properties using microwave characterization equipment, and the sample preparation was discussed in depth. Chapter 5 discusses the findings analyzed based on the design of the different kinds of RAM that were taken into consideration and all measurements. In Chapter 6, the study results are summarized and concluded. The thesis also has a comprehensive bibliography and offers some recommendations for further study in addition to the final chapter

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