IMPROVEMENT OF MICROWAVE ABSORPTION PROPERTIES VIA CERAMIC METHOD AND MECHANICAL ALLOYING PROCESSING OF Ti-Co-Mn-Ni SUBSTITUTED BARIUM FERRITE

By

CHEN HONGXU

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

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Chairman : Associate Professor Raba'ah Syahidah binti Azis, PhD
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Many attempts have been made to identify nanomaterials with high reflection loss to improve electromagnetic microwave absorption. In this study, barium hexagonal nanocomposites ($BaFe_{12}O_{19}$) and substituted barium hexagonal ferrite composites $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4) were synthesized using high energy ball milling to investigate their magnetic and electromagnetic microwave adsorption properties. The goal is to address the challenge of creating homogeneous, fine-grained materials for high-GHz band applications, where electromagnetic interference remains a problem.

Initially, the samples underwent processing optimization by several stages: mixing by mortar (M), calcination (C), sintering (S) and high energy ball milling (HEBM) (H) to determine the best method to obtain highly crystalline nanoparticles for Microwave Absorber (MA) materials. The calcination-sintering-HEBM (CSH) method proved to be the most effective and was used to prepare the MA composite. The raw powder underwent conventional milling for 5 hours, calcination at 1000 ºC for 5 hours, sintering at 1300 ºC for 5 hours, and then mechanical alloying for 5 hours. The samples were then investigated their structural, microstructural, elemental, magnetic, and microwave properties using an X-ray diffraction (XRD), a field emission scanning electron microscopy (FESEM), an energy dispersive X-ray analysis (EDX) a vibrating sample magnetometer (VSM), and a vector network analyzer (VNA). XRD data revealed that all the samples are crystalline ferrite with a hexagonal crystal structure. The XRD spectra showed that the peaks of the milled samples are broader than those of the un-milled samples, with the smallest particle size being 79.09 nm, as indicated by FESEM micrographs. VSM results exhibited that saturation magnetization (M_s) decreases with milling time and Mn content. The $BaTiCo_{0.5}Mn_{0.4}Ni_{0.1}Fe_{10}O_{19}$ sample with the substitution value of x =0.4 had the lowest reflection loss (RL) of −40.37 dB at 13.7 GHz with at a thickness of 2 mm through a CSH synthesis. Thus, $BaTiCo_{0.5}Mn_{0.4}Ni_{0.1}Fe_{10}O_{19}$ demonstrated excellent reflection loss and higher absorption of incident electromagnetic radiation, addressing the urgent problem of electromagnetic pollution.

Keyword: Calcination, Mechanical Alloying, M-Type, Nano-Ferrite Composite, Sintering.

SDG: GOAL 9: Industry, Innovation and Infrastructure

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

PENAMBAHBAIKAN SIFAT PENYERAPAN GELOMBANG MIKRO MELALUI KAEDAH SERAMIK DAN PEMPROSESAN PENGALOIAN MEKANIKAL BARIUM FERIT YANG DISUBSTITUSI DENGAN Ti-Co-Mn-Ni

Oleh

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Banyak usaha telah dilakukan untuk mengenal pasti nanomaterial dengan kehilangan pantulan yang tinggi untuk meningkatkan penyerapan gelombang mikro elektromagnetik. Dalam kajian ini, nanokomposit heksagonal barium ($BaFe_{12}O_{19}$) dan komposit ferit heksagonal barium yang disubstitusi $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4) telah disintesis menggunakan penggilingan bola berkecepatan tinggi untuk menyiasat sifat magnetik dan penyerapan gelombang mikro elektromagnetiknya. Tujuannya adalah untuk menangani cabaran mencipta bahan homogen berbutir halus untuk aplikasi jalur tinggi-GHz, di mana gangguan elektromagnetik masih menjadi masalah.

Pada awalnya, sampel menjalani pengoptimuman pemprosesan melalui beberapa peringkat: pencampuran dengan mortar (M), kalsinasi (C), pensinteran (S), dan penggilingan bola berkecepatan tinggi (HEBM) (H) untuk menentukan kaedah terbaik untuk memperoleh nanopartikel kristal tinggi untuk bahan Penyerap Gelombang

Mikro (MA). Kaedah kalsinasi-pensinteran-HEBM (CSH) terbukti paling berkesan dan digunakan untuk menyediakan komposit MA. Serbuk mentah menjalani penggilingan konvensional selama 5 jam, kalsinasi pada 1000 ºC selama 5 jam, pensinteran pada 1300 ºC selama 5 jam, dan kemudian pengaloian mekanikal selama 5 jam. Sampel kemudiannya disiasat untuk sifat struktur, mikrostruktur, unsur, magnetik, dan gelombang mikro menggunakan pembelauan sinar-X (XRD), mikroskopi elektron pancaran medan (FESEM), analisis sinar-X serakan tenaga (EDX), magnetometer sampel bergetar (VSM), dan penganalisis rangkaian vektor (VNA). Data XRD menunjukkan bahawa semua sampel adalah ferit kristal dengan struktur kristal heksagonal. Spektra XRD menunjukkan bahawa puncak sampel yang digiling adalah lebih lebar daripada sampel yang tidak digiling, dengan saiz zarah terkecil ialah 79.09 nm, seperti yang ditunjukkan oleh mikrograf FESEM. Keputusan VSM menunjukkan bahawa magnetisasi tepu (Ms) berkurang dengan masa penggilingan dan kandungan Mn. Sampel $BaTiCo_{0.5}Mn_{0.4}Ni_{0.1}Fe_{10}O_{19}$ dengan nilai substitusi x = 0.4 mempunyai kehilangan pantulan (RL) terendah iaitu -40.37 dB pada 13.7 GHz dengan ketebalan 2 mm melalui sintesis CSH. Oleh itu, $BaTiCo_{0.5}Mn_{0.4}Ni_{0.1}Fe_{10}O_{19}$ menunjukkan kehilangan pantulan yang sangat baik dan penyerapan yang lebih tinggi terhadap radiasi elektromagnetik yang terlibat, menangani masalah mendesak pencemaran elektromagnetik.

Kata kunci: Jenis-M, Kalsinasi, Komposit Nano-Ferit, Paduan Mekanikal**,** Penyinteran.

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2θ	2 theta degree
a, *c*	Lattice parameters
a.u	Arbitrary unit
B	Magnetic flux density
BHF	$BaFe_{12}O_{19}$
c	Velocity of light
CNT	Carbon nanotube
CSH	Calcination- Sintering-High energy ball milling
CSM	Calcination- Sintering -Motar
d	Interplanar spacing
D	Particle size
dB	Decibel
EDX	Energy-dispersive X-ray
EM	Electromagnetic
f	Microwave frequency
FESEM	Field emission scanning electron microscopy
G	Gauss
GHz	Gigahertz
GOF	goodness of refinement fitting
H_A	Anisotropy field for *c*-axis anisotropy
H_c	Coercivity
HEBM	High-energy ball milling (Mechanical alloying)
H_i	Internal field
hkl	Miller indices

K_1	Magnetocrystalline anisotropy
K_u-band	12 – 18 GHz
MA	Microwave absorber
MAM	Microwave absorbing material
MHz	Megahertz
M_r	Residual magnetization
M_s	Saturation magnetization
R_{exp}	Expected profile parameter, and the chi-square fitting
RL	Reflection loss
R_p	Un-weighted profile parameter
R_{wp}	Weighted profile parameter
SBHF	Ti-Co-Mn-Ni Substution $BaFe_{12}O_{19}$
SG	space group
SH	Sintering-High energy ball milling
SM	Sintering-Motar
t	Thickness
$tan\ \delta_\mu$	Magnetic loss angle
$tan\ \delta_\varepsilon$	Dielectric loss angle
t_m	Matching thickness
UPM	Universiti Putra Malaysia
V	Unit cell volume
VNA	Vector network analyzer
VSM	Vibrating sample magnetometer
wt%	Weight percent
X-band	8 – 12 GHz
XRD	X-ray diffraction
Z_{in}	Input impedance of the absorber

Z_o	Impedance of free space
α	Extinction coefficient
$\varepsilon = \varepsilon' - j\varepsilon''$	Complex permittivity, ε' represents the real part, ε'' is the imaginary part
λ	Wavelength of the X-ray
$\mu = \mu' - j\mu''$	Complex permeability, μ' represents the real part, μ'' is the imaginary part
ρ_{xrd}	X-ray density
χ^2	the goodness of fitting

CHAPTER 1

INTRODUCTION

1.1 Background of study

The ongoing advancements in science and technology, coupled with the modernization of digital devices like computers, smartphones, and airplanes, as well as the extensive development of 5G, result in electromagnetic radiation that poses a significant threat to human health and the environment (Wang et al., 2014; Wongkasem et al., 2021). Investigating superior electromagnetic performance and microwave absorption materials becomes crucial to resolving this issue. The electromagnetic absorption capabilities of typical electromagnetic wave-absorbing nanomaterials, such as carbon, Fe, Fe_3O_4, and CNTs, have advanced significantly in recent years (Wen et al., 2023).

The utilization of electromagnetic wave absorption nanomaterials for mass production has been substantially impeded by some drawbacks, including a complex synthesis procedure for nanostructures and difficulties in mass production (Zeng et al., 2020). Therefore, to create nanomaterials that will increase the effectiveness of electromagnetic microwave absorption, the solid phase synthesis method—which offers a straightforward and affordable manufacturing process—was selected for this work (Ivanov et al., 2011). The solid phase synthesis process has limitations, though, including stoichiometric losses, inhomogeneous particle size and shape, poor junction behavior, a lack of repeatability, and multiphase characteristics (Ivanov et al., 2012). Therefore, optimizing the process variables, such as mechanical milling sintering,

sintering temperature, sintering time, type of substituent, and amount of substituent, is necessary to improve the performance of barium oxide as a microwave absorber and to optimize the solid phase synthesis method (Bodaghi et al., 2008).

Similarly, the effect of dielectric and magnetic nanomaterials in microwave absorption has yet to be explicitly explored as a function of particle size in numerous studies; it is undeniable that particle size appears to be a critical factor in microwave absorption (Elmahaishi et al., 2024). Thus, the size of nanomaterials is related to their tendency to enhance electromagnetic interactions, which provides a better match for microwave absorption (Elmahaishi et al., 2024). It is essential to investigate the materials' microwave absorption and electromagnetic properties and study process optimization to develop materials with superior microwave absorption and electromagnetic properties (Elmahaishi et al., 2022).

Wang et al. (2022) stated that single-phase M-type ferrite could surpass its performance limitations through ion doping or elemental substitution that enhances the physicochemical properties of M-type ferrite. The physical and chemical properties of M-type ferrites can also be improved by ion doping or other oxides. Doping or additional oxide doping can increase the coercivity and saturation magnetization strength and reduce the anisotropy field of m-type ferrites (Zhao et al., 2007).

Based on the above discussion, this thesis focuses on alloying methods and M-type element substitution to investigate the effect on microwave absorption properties.

1. Ceramic and alloying method study: Four different solid-state synthesis processes were designed to synthesize nanomaterials, using grinding time and heat treatment as essential considerations. We explore these methods to discover the most optimized solid-state synthesis technique.

2. Element substitution experiment: This experiment mainly investigated the barium hexagonal nanocomposite ($BaFe_{12}O_{19}$) and substituted barium hexagonal ferrite nanocomposite with a chemical formula of $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4) with the variation of Mn and Ni concentration on the microwave absorption property of the microwave absorption performance.

This study will thoroughly investigate the electromagnetic absorption characteristics of M-type ferrite nanomaterials, the preparation technique, the characterization procedure, and their particular applications to accomplish these two goals. In addition, $BaFe_{12}O_{19}$ and $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4) materials are prepared for the first time by solid-phase synthesis in the present experiments, aiming to enhance their performance in terms of broadband and high absorption levels and provide more in-depth theoretical and theoretical insights for the cost-effective production of microwave-absorbing and electromagnetism-enhancing nanomaterials with excellent microwave absorption and electromagnetic properties by providing a more in-depth theoretical and practical foundation.

With the advancement of science and technology, the usage of 5G in a significant number of buildings will enhance the danger of electromagnetic radiation to humans (Liu et al., 2017). Exploring materials with outstanding microwave absorption and electromagnetic properties has become the key to solving this problem, and people are also interested in M-type hexagonal ferrites with microwave absorption and electromagnetic properties (Chen et al., 2010). The microwave absorption and electromagnetic properties of $BaFe_{12}O_{19}$ and $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x=0.25, 0.3, 0.35, 0.4) are examined in this study. This thesis will explain the knowledge of alternative magnetic polymer nanocomposites with high-frequency absorption for microwave absorption, electromagnetism, calcination, and sintering applications. This experiment explored reflection and absorption to find effective absorbers with high absorption over a wide frequency range.

1.2 Brief introduction to electromagnetic properties of composite as M-type ferrite

M-type ferrite composites, also known as hexaferrites, exhibit unique electromagnetic properties that make them highly suitable for various applications in electronics and telecommunications (Mathews et al., 2021). These qualities arise from their inherent magnetic and dielectric properties at the nanoscale, which are significantly modified by their chemical composition, crystal structure, and the methods involved in their manufacture (Gleiter et al., 2019).

M-type ferrites, with their high magnetic permeability and low conductivity, are excellent materials for high-frequency applications (Zeng et al., 2020). They exhibit high saturation magnetization due to the alignment of their magnetic moments, resulting in superior electromagnetic characteristics. Additionally, their inherent ferrimagnetism contributes to their magnetic solid anisotropy, leading to high coercivity values (Won et al., 2022). This means that M-type ferrite nanocomposites can maintain their magnetization even without an external magnetic field, a property highly valued in permanent magnet applications (Elmahaishi et al., 2020).

The electromagnetic properties of M-type ferrite nanocomposites also extend to their dielectric behavior (Pullar et al., 2012). These materials exhibit low dielectric loss, a measure of energy dissipated as heat when subjected to an alternating electric field (Abbas et al., 2007). M-type ferrite nanocomposites' magnetic and dielectric properties can be tailored to meet specific application requirements by varying factors such as particle size morphology or adding other materials to form composites (Althomali et al., 2024).

For instance, introducing other metal ions into the ferrite structure can enhance magnetic permeability, increase saturation magnetization, or reduce dielectric loss, depending on the ions used (Sardar al., 2024). In summary, the electromagnetic properties of M-type ferrite nanocomposites, ranging from their high magnetic permeability and low conductivity to their low dielectric loss, make them promising materials for various applications (Kaur et al., 2006). These properties can be fine-tuned by carefully manipulating their composition and structure, offering vast potential for innovation in electronics, telecommunications, and related fields (Nielsen et al., 1994).

1.3 Recent research and motivation for this work

Recent research on M-type ferrite nanocomposites has highlighted their potential as effective microwave absorbers, mainly due to their inherent magnetic and dielectric properties, which enable them to absorb electromagnetic waves and convert them into heat [Wang et al., 2022]. This microwave-absorbing capability is critical in many applications, including electromagnetic interference (EMI) shielding, radar stealth technology, and microwave devices [Kim et al., 2024].

Several recent studies have shown that preparing substituted hexagonal ferrites by conventional solid-phase reactions improved microwave absorption properties. For example, Won et al. (2022) showed that new opportunities for EMI material development were opened by solid-phase synthesis of MXene/$BaCo_{0.3}Ti_{0.3}Fe_{11.4}O_{19}$ hexagonal ferrite composites for millimeter-wave applications. In another study, Kaur et al. (2020) published that synthesized $Sr_{1-y}La_yFe_{12}O_{19}$ as microwave absorbing material in the frequency range of 18-40 GHz exhibited minimal reflection loss (RL) absorption more significant than 90%; this material has a great potential to be used as an efficient material microwave absorber.

In addition, substitution in M-type ferrite composites is also an effective way to enhance their microwave absorption capability. For example, the microwave absorption capabilities of Ce-substituted M-type ferrites are greatly improved, and $BaCe_{0.05}Fe_{11.95}O_{19}$ can be employed as an excellent microwave absorbing material in the 8.0-13.0 GHz range. (Chang et al., 2012). (Gujral et al., 2021) M-type $Ba_{0.5}Sr_{0.5}Co_xLa_xFe_{12-2x}O_{19}$ hexagonal ferrite, showing that doping improves impedance matching and increases microwave absorption and bandwidth of the absorbed prepared compositions.

Despite these achievements, there are still certain obstacles to overcome. One of the most critical difficulties is the trade-off between absorber thickness and absorption capacity. Thin absorbers are desired for practical applications, but their absorption capacity is generally modest (Gama et al., 2007). Another problem is achieving a broad absorption bandwidth, which is necessary for many applications with a wide range of operating frequencies (Zhang et al., 2021).

Motivated by the promising results of recent studies and the existing challenges, this thesis aims further to explore the microwave absorption properties of M-type ferrite nanocomposites. Specifically, this work will focus on different steps of solid-phase synthesis methods and dopant-substituted composite formulations to enhance M-type ferrites' microwave absorptivity and bandwidth. By addressing these issues, this research aims to facilitate the development of high-performance microwave absorbers and to advance current microwave technology.

1.4 Problem statement

The high emission of EM waves into the atmosphere is a severe health danger (Liu et al., 2017). To improve EM absorption properties, new systems have been evolved comprising magnetic fillers, including soft spinel ferrite and hard M-type ferrite (Chen et al., 2010). Microwave materials with good absorption are in high demand to solve electromagnetic interference (EMI) problems in commercial and industrial electronics. Nowadays, increasingly fierce military competition has promoted the development of various high-tech military equipment, especially stealth fighters and drones, which face challenges in effectively absorbing electromagnetic (EM) waves (Zeng et al., 2020). The electromagnetic radiation (EM radiation) produced by constantly evolving digital equipment, such as mobile phones, computers, and 5G stations, has had a negative impact on physical health (Qiang et al., 2015). Therefore, developing numerous improved materials for EM wave absorption is crucial. High reflection loss (RL), thin thickness, wide bandwidth, and low density are the primary markers for effective EM wave absorbers (Meng et al., 2015). Enhance impedance matching in electromagnetic absorbers to improve microwave absorption efficacy.

The current research aims to find the optimal solid-phase synthesis method for the large-scale production of high-quality nanoparticles. Of particular interest are Mn-Ti-

Co-Ni-substituted barium oxides, which exhibit excellent reflection loss performance in thin-layer structures and broadband conditions, which are essential for microwave radiation pollution reduction and military stealth technology. However, we face the following challenges and problems in fabricating thin-layer and broadband radar wave absorbers:

1. Although many researchers have used solid-phase synthesis to produce magnetic nanomaterials for microwave absorption, achieving materials with uniform shapes, fine particles, and consistent reproducibility remains a significant challenge.
2. Barium oxides have been extensively studied as microwave-absorbing materials; however, few studies have focused on developing thin and broadband radar wave absorbers using Ti-Co-Mn-Ni substituted barium oxides.
3. While various microwave absorber materials have been developed, issues related to electromagnetic interference and compatibility in the high GHz band remain unresolved.
4. Due to its tunable anisotropic field, M-type hexaferrite is considered an ideal microwave-absorbing material in the high GHz band. However, enhancing its dielectric loss remains challenging due to its reduced permeability in the high GHz band, resulting in weak resonant absorption and a narrow frequency range.

Therefore, our investigation into various solid-phase synthesis methods and different doping ratios has led to the identification of microwave-absorbing materials that are highly effective within the 8 to 18 GHz frequency range. These materials exhibit significant reflection loss, wide bandwidths, and reduced coating thicknesses, representing a key achievement in enhancing production quality and efficiency in this field.

1.5 Objective

Ferrites are magnetic oxide materials with superior electric-magneto properties suitable for elevated frequency applications. These properties include outstanding permittivity, excellent permeability, and minimal loss, which are influenced by microstructural characteristics (Dey et al., 2024). This research was performed to study the morphological, microstructural, magnetic, and dielectric properties of barium ferrite and Ti-Co-Mn-Ni substitution barium ferrite composite ($BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4)). The specific objectives of the study are as follows:

1. To investigate the effect of M-type particle size on the structure, morphological, magnetic, and microwave properties of M-type composites through various alloyed methods (SM, SH, CSM, CSH).
2. To study the barium hexagonal composite ($BaFe_{12}O_{19}$) and substituted barium hexagonal ferrite composite with a chemical formula of $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4)) with a variation of Mn and Ni concentration, in the structural, magnetic, morphological/ microstructural and microwave properties of hexagonal ferrites.

1.6 Thesis Outline

The current experiments concentrated on barium ferrite's structural, morphological, electromagnetic, magnetic, and adsorption examinations. The solid-phase synthesis of the barium hexagonal nanocomposite ($BaFe_{12}O_{19}$) and substituted barium hexagonal ferrite nanocomposite with a chemical formula of $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$ (x = 0.25, 0.3, 0.35, 0.4) were reviewed, as were the effects of varied heat treatments, high-energy milling duration, and Mn-Ni doped on $BaTiCo_{0.5}Mn_xNi_{0.5-x}Fe_{10}O_{19}$. This thesis is divided into six chapters. **Chapter 1** provides a general introduction to the research work, barium ferrite nanocomposite and electromagnetic properties of nanocomposite as an M-type ferrite, a statement of the problem, objectives of the study, and the significance of the study. **Chapter 2** reviews relevant literature on methods of preparing barium ferrite nanocomposite (BFN), sintering, calcining, doped M-type ferrite, and high energy milling time. The structural, magnetic and electromagnetic/magnetodielectric properties of BFN and its derived compounds were also reviewed. **Chapter 2** covers theoretical aspects of the subject, including the theory of the crystal structure, magnetic characteristics, and microwave absorption of barium ferrite. It explains magnetism and its type, intrinsic and extrinsic magnetic properties, and barium structure. **Chapter 3** deals with materials and sample preparation methods. The different characterization measurements involved in the research are presented. **Chapter 4** outlines the sample characterization results and discussion. Finally, Conclusions drawn from the research findings are presented and discussed in **Chapter 5**.

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