

SYSTHESIS AND CHARACTERIZATION OF ALUMINIUM SUBSTITUTED
NICKEL MAGNESIUM FERRITE NANOPARTICLES

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To my lovely parents and my helpful Professor

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ABSTRACT

With excellent magnetic and electrical properties, ferrite magnetic nanoparticles have wide range of applications in wide range of fields. With tuneable physical and chemical properties, ferrites can have more applications and become more suitable for advancing technology. Normally, two approaches are used in tuning the physical properties of ferrites, first approach is changing the synthesis methods and the second approach is altering the types of cations being introduced into the ferrites. In this study, Aluminium substituted nickel magnesium ferrite with general formula $\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$ were synthesised with co-precipitation method. Three annealing temperature were chosen at 600°C, 800°C and 1000°C. The samples were characterised using X-ray diffractometer (XRD) and Fourier Transform Infrared Red Spectroscopy (FTIR) for structural analysis. Field emission scanning electron microscope (FESEM) was used to analyse the morphological property of the samples. Energy dispersive X-ray spectrometer was used to study the composition of the samples. The dielectric properties of the samples were analysed by using impedance analyser. XRD and FTIR shows the single phase spinel structure of the samples. Crystallite size decreases with increasing ratio of aluminium and magnesium except for the samples annealed at 600°C, and increases when the annealing temperature increases. Lattice constant of samples annealed at 800°C and 1000 °C are almost constant but becomes smaller when annealed at 600°C, which may due to redistribution of the cations with different annealing temperature. The degree of crystallinity increases as the annealing temperature increase, when the ratio of aluminium and magnesium reach 0.8, the degree of crystallinity deteriorated. FESEM micrograph shows the homogeneous distribution of the nanoparticles. Increment of dielectric constant and low loss factor of this substituted ferrites make it a good candidate for high frequency device applications.

ABSTRAK

Dengan sifat magnetik dan elektrik yang baik, nanopartikel ferit yang bermagnet mempunyai aplikasi yang banyak dalam bidang yang luas. Dengan sifat fizikal dan kimia yang boleh diubahsuai dengan mudah menjadikan nanopartikel ferit mempunyai aplikasi yang lebih banyak dan lebih sesuai digunakan dalam bidang teknologi yang semakin maju. Dua cara yang biasa digunakan dalam mengubahsuai sifat-sifat fizikal ferit nanopartikel, iaitu menggunakan cara sintesis yang berlainan atau mengubah kation yang berlainan dalam ferit. Dalam kajian ini, nikel magnesium ferit yang digantikan oleh aluminium dengan formula $Ni_{1-x}Mg_xFe_{2-x}Al_xO_4$ telah disintesis dengan menggunakan cara kimia dekomposisi dan disepuhlintap pada suhu 600 °C, 800 °C dan 1000 °C. Sampel yang sudah siap telah dicirikan dengan XRD dan FTIR untuk mengkaji struktur sampel. FESEM telah digunakan dalam kajian morfologi sampel. EDX digunakan untuk mengkaji komposisi sampel. Sifat-sifat dielektrik telah dikaji oleh penganalisis impedan. Hasil XRD dan FTIR menunjukkan sampel mempunyai struktur spinel. Saiz kristal berkurang apabila nisbah magnesium dan aluminium meningkat dalam ferit ini kecuali sampel yang disintesis pada suhu 600°C, saiz kristal meningkat apabila suhu sepuhlintap meningkat. Pemalar kisi tidak berubah untuk sampel yang disintesis pada suhu 800°C dan 1000°C tetapi telah meningkat berbanding dengan sampel yang disintesis pada suhu 600°C, yang mungkin disebabkan oleh pengagihan semula kation pada suhu sepuhlintap yang berlainan. Darjah penghabluran meningkat apabila suhu anil untuk menyintesis sampel ini meningkat, apabila nisbah magnesium dan aluminium capai 0.8, tahap kristal telah jatuh. FESEM menunjukkan taburan saiz nanopartikel adalah hampir sama. Peningkatan pemalar dielektrik dan keturunan faktor kehilangan menjadikan ferit yang mempunyai gantian sebagai calon yang sesuai untuk aplikasi frekuensi tinggi.

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LIST OF ABBREVIATIONS

EDX	Energy Dispersive X-Ray
FESEM	Field Emission Scanning Electron Microscope
FWHM	Full Width Half Maximum
FTIR	Fourier Transform Infrared Red
XRD	X-Ray Diffraction
AR	Analytical Grade

CHAPTER 1

INTRODUCTION

1.1 Background of study

Ferrites, a type of dielectric material and magnetic material are object of great interest recently due to wide range of applications including high density magnetic storage, contrast agents for magnetic resonance imaging (MRI), magnetic field assisted cancer treatment, ferrofluids, drug delivery (S.Rana *et al.*, 2007), electronic devices such as inductors, transformers (Andris Sutka *et al.*, 2015), gas sensing (Ghosh.P *et al.*,2015), and photocatalysts replacing titanium dioxide (TiO₂). (Kong Zi.Y *et al.*, 2015).

Ferrites can be classified in three main types of crystalline structures, they are hexagonal ferrites with chemical formula MFe₁₂O₁₉, spinel ferrites with chemical formula MFe₂O₄ and garnet ferrites with chemical formula M₃Fe₅O₁₂ where M represents divalent ions. Different crystalline structure will have different properties hence different applications.

Spinel ferrites have been a subject of study due to their wide range of applications as shown above, with the high DC resistivity, low loss tangent and low eddy current loss, spinel ferrites are promising material for high frequency applications (Satish Verma *et al.*, 2012a). They are also used to absorb the microwave. Radar absorbing paint used in painting stealth aircraft are made of ferrites (Gui.I.H and Maqsood.A, 2008a).

Spinel ferrites with general chemical formula MFe_2O_3 , where M represents the divalent ions have crystal structure similar as naturally found Magnesium Aluminate ($MgAl_2O_4$). This crystal structure have cubic closed pack structure. This structure have two sites for cations to reside, which are tetrahedral site and octahedral site, normally referred as A sites and B sites. There are three types of spinel ferrites according to the distribution of the cations. When M^{2+} cations totally fill the tetrahedral sites and Fe^{3+} cations totally fill the octahedral sites, this crystal structure is called normal spinel structure. If the Fe^{3+} cations totally fill the tetrahedral sites and M^{2+} cations and Fe^{3+} cations both fill the octahedral sites, this crystal structure is called inverse spinel structure. Most ferrites spinel have both M^{2+} cations and Fe^{3+} cations fill both the tetrahedral sites and octahedral sites, this crystal structure is called mixed spinel structure. This structure have a parameter calls degree of inversion, normally symbolized by δ . For instance, zinc ferrite ($ZnFe_2O_4$) formed normal spinel structure. Nickel ferrite ($NiFe_2O_4$) and cobalt ferrite ($CoFe_2O_4$) formed inverse spinel structure, manganese ferrite ($MnFe_2O_4$) formed mixed spinel structure.

The net magnetization of the spinel ferrite are difference between magnetization of the A site and B site. Indeed, the magnetic moment of the ions in the A site and B site are arranged antiparallel with each other but still there is net magnetization due to unbalanced magnetic moment on the two sites, hence, spinel ferrites show ferrimagnetic properties. Substitution of other type of ions into the spinel ferrite may change the magnetic properties or electrical properties of the spinel ferrite because the original cations are forced to have new distribution among the A site and B site due to the new substitution (Raju.K *et al.*, 2013), for instance, introducing zinc or copper into nickel ferrite will obtain higher saturation magnetization (Rama Krishna.K *et al.*, 2012). By introducing aluminium into cobalt ferrites will increase its' DC resistivity (Gui.I.H and Maqsood.A, 2008b)..

The magnetic and electrical properties depends on their shape and shape which in turn depends on their synthesis methods. Conventional ceramic method requires high temperature for the synthesis of the samples, and normally will obtain nanoparticles with poor stoichiometry and inhomogeneity. Wet chemical methods such as sol gel auto combustion method, co-precipitation method and hydrothermal

method can produce better quality nanoparticles. Among the wet chemical methods, co-precipitation method have the advantages of low cost, high production rate, low temperature synthesis and produce nanoparticles with very small size (Binu P Jacob *et al.*, 2011a).

Nickel ferrite process inverse spinel structure in which Fe^{3+} ions fill both the tetrahedral sites and octahedral sites, and Ni^{2+} ions only fill the octahedral sites. Owing to its' high magnetocrystalline anisotropy and high saturation magnetization, nickel ferrite has many technological applications such as repulsive suspension for levitated railway system, microwave absorber and so on (Nermin Kasapoglu *et al.*, 2007). Magnesium ferrite have partially inverse spinel structure in which both the Fe^{3+} and Mg^{2+} ions fill both the tetrahedral and octahedral sites. Magnesium ferrite is a soft n-type semiconducting material. Magnesium ferrite has high magnetic permeability and high DC electrical resistivity and hence has many technological applications such as catalysts, colour imaging, sensors and so on (Bamzai.K.K *et al.*, 2014a). Indeed, both the magnesium ferrite and nickel ferrite are applied in many electronic devices because of their high permeability at high frequency and also very high resistivity (Abdul Gaffoor *et al.*, 2014a)

Higher loss tangent materials are suitable for high frequency heating devices. Loss tangent represents the efficiency with which the electromagnetic wave energy is converted to heat. Meanwhile, with higher loss tangent, the microwave absorption will improve. H. Moradmard *et al* showed that introducing magnesium into nickel ferrite will increase their dielectric loss, decrease the dielectric constant, at the same time, the crystalline size of the magnesium increase first and then decrease then increase again (2015a). The crystalline size's result is consistent with Abdul Gaffoor *et al.* (2014b). The dielectric constant's result is consistent with John Berchmans.L *et al.* (2004a)

Addition of Al^{3+} ions into the ferrites change the ferrites' structural properties, dielectric properties, electrical properties in quite interesting manner. Satish Verma *et al.* showed there is a significant decrease in the particles' size after the Al^{3+} ions are introduced to the ferrite samples (2012b). Indeed, many research have shown that introduction of Al^{3+} ions into nickel ferrite have increase its resistivity and dielectric

constant so this ferrite is very suitable to high applications which normally have low dielectric loss and eddy current loss (Maghsoudi.I *et al.*, 2013).

In present study, aluminium substituted magnesium nickel ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) will be synthesized using co-precipitation method with $x = 0.0, 0.2, 0.4, 0.6, 0.8$ and 1.0 , annealed at 600°C , 800°C and 1000°C respectively. This study will allow us to understand the properties of this ferrite when both aluminium and magnesium is introduced to the nickel ferrite.

1.2 Problem Statement

Nickel ferrite (NiFe_2O_4) is a type of soft magnetic material with high saturation magnetization and high resistivity, making it a useful material for applications in transformers and high frequency telecommunications. Magnesium ferrite (MgFe_2O_4) is also a soft magnetic material showing n-type semiconducting behaviour. Magnesium-nickel ferrite show special dielectric, electrical, magnetic and structural properties. Addition of Al^{3+} ions into the ferrite can alter the structural, magnetic and also electrical properties of the ferrites.

Until recently, there are detailed studies on electrical, dielectric, structural and magnetic properties on Ni-Mg ferrite ($\text{Mg}_{1-x}\text{Ni}_x\text{Fe}_{2-x}\text{O}_4$), but not any investigations about the structural, morphological and dielectric properties of the effects of annealing temperature on Ni-Mg ferrite. As far as we know there are no investigations about the effects of Al^{3+} ions' addition in to Ni-Mg ferrite. In this study, aluminium substituted nickel magnesium ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) with $x = 0.0, 0.2, 0.4, 0.6$ and 0.8 was synthesized and annealed at 600°C , 800°C and 1000°C .

Co-precipitation method was chosen due to its high production rate, low cost and better quality nanoparticles can be produced if compare to conventional ceramic method, hence suitable for large scale production. This study can be useful to study the change of properties of the samples due to different annealing temperature and also

the change of properties due to aluminium substitution for appropriate applications.

1.3 Objectives of the Research

The objectives of this study are:

- i. To synthesize ferrites aluminium substituted nickel magnesium ferrite with general formula $\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$ using co-precipitation method with different annealing temperature.
- ii. To determine the structural properties of aluminium substituted nickel magnesium ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) annealed at different temperature using X-ray diffractometer (XRD) and Fourier Transform Infra-Red spectroscopy (FTIR).
- iii. To determine the morphological properties of aluminium substituted nickel magnesium ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) annealed at different temperature using Field Emission Scan Electron Microscopy (FESEM).
- iv. To determine the dielectric properties (dielectric constant, loss factor ($\tan \delta$), dielectric loss) of aluminium substituted nickel magnesium ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) annealed at different temperature using impedance analyser.

1.4 Scope of Study

The scope of study is to synthesize the aluminium substituted nickel magnesium ferrite ($\text{Ni}_{1-x}\text{Mg}_x\text{Fe}_{2-x}\text{Al}_x\text{O}_4$) with different annealing temperature (600°C , 800°C , 1000°C) using co-precipitation method and with Sodium Hydroxide (NaOH) as precipitating agent. After synthesis, the samples will be characterized by X-Ray Diffraction (XRD) and Fourier Transform Infra-Red Spectroscopy (FTIR) to study the structural properties of the samples. The structural and morphological properties of the samples will be characterized by using Field Emission Scanning Electron Microscope (FESEM). Finally, the dielectric properties of the samples such as dielectric constant, dielectric loss and loss factor ($\tan \delta$) will be characterized using impedance analyser.

1.5 Significant of Research

Ni-Mg ferrites show good properties and find many applications in technological field. The structural properties, morphological, electrical, dielectric, and magnetic properties of Ni-Mg ferrite was studied by some researchers, whereas continuing to check out and improve the quality of the ferrite is important for different applications.

Addition of Al^{3+} ions into the ferrites change the ferrites' structural properties, dielectric properties, electrical properties in quite interesting manner too. With different annealing temperature, the structural and morphological of the ferrite can be altered which in turn alter the magnetic and electrical properties of the prepared ferrite. So, in this study, by additional of Al^{3+} ions into Ni-Mg ferrites and also by altering the annealing temperature to prepare these samples can help us to find out the better quality ferrite to be utilized in industrial and technological field.

1.6 Thesis Outline

This thesis consists of five chapters, with the first chapter depict the problem statement, significant of study, objectives of research scope of study and background of study. The second chapter encompass the literature review of the necessary knowledge of this study including history of ferrite, normal structure of ferrite, applications of ferrite, basic concept of dielectric loss, dielectric constant and dielectric properties of ferrite, basic concept of magnetism and the magnetic properties of ferrite and also some background study of this research. In chapter three, all the preparation process and characterization process will be discussed. Chapter four will have all the results of the characterization and the discussions of the result. Finally, conclusions and some suggestions for future research can be found in chapter five.

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