STRUCTURAL, MAGNETIC AND DIELECTRIC PROPERTIES OF NICKEL-MAGNESIUM SUBSTITUTED COBALT FERRITES NANOPARTICLES AND CORE-SHELL NANOCOMPOSITES

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Special dedications to my beloved wife, parents and my supportive supervisors…
Thanks for the love and memories

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ABSTRACT

Cobalt ferrite has gained great scientist interest because of its important applications in various fields of science and technology. However, the magnetic character of the particles used for many applications depends crucially on the size, shape and purity of these nanoparticles. Hence the need for developing fabrication processes that are relatively simple and yield controlled particle sizes is desired. This work involves the study of structural, magnetic, dielectric properties and morphology of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ ferrite nanoparticles (x = 0.0, 0.1, 0.2, 0.3, 0.4, 0.5), which are synthesized by chemical co-precipitation method. In addition, the core-shell nanocomposites of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$/Polyaniline were successfully synthesized via chemical polymerization method. The ferrite samples were then sintered at selected temperatures of 700 °C, 800 °C, 900 °C and 1000°C for 8 hours. X-ray powder diffraction indicated that the core material is having a single phase of spinel cubic structure. The crystallite size of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ nanoparticles was found in the range of 25-40 nm. The infrared spectra of the synthesized samples displayed two absorption bands characteristic of the spinel ferrites at 585–595 cm^{-1} and 390–400 cm^{-1}, which correspond to vibrations of tetrahedral and octahedral bonds, respectively. The Field Emission Scanning Electron Microscope and Transmission Electron Microscope images of ferrite nanoparticles show different aggregations at different sintering temperatures and concentrations. The combination of both Ni-, Mg- substituted cobalt ferrites showed that the substitution of Mg^{2+} ions for Fe made more pronounced effects on magnetic and dielectric properties at room temperature. The values of saturation magnetization (M_s) and coercivity (H_c) are enhanced by increasing of Mg concentration up to $x = 0.1$. By increasing Mg^{2+} substitution, the M_s and H_c increase from 57.35 emu/g ($x = 0.0$) to 61.49 emu/g ($x = 0.1$) and 603.26 Oe ($x = 0.0$) to 684.11 Oe ($x = 0.1$), respectively. In contrast, the M_s decreases from a maximum value 12.00 emu/g ($x = 0.1$) to a minimum value 5.39 emu/g ($x = 0.4$) when ferrites are encapsulated with Polyaniline. However, the H_c increases from a maximum value 766.94 Oe ($x = 0.1$) to a minimum value 646.17 Oe ($x = 0.0$). At 1 kHz, dielectric constant ε' shows a maximum value at 86.22 for $x = 0.1$ and minimum value at 56.67 for $x = 0.3$. In addition, the dielectric loss ε'' shows a maximum value of 10.98 for $x = 0.2$ and minimum value of 9.45 for $x = 0.0$. For nanocomposites, ε' reaches a maximum value of 68.32 ($x = 0.1$) and minimum value of 46.73 ($x = 0.3$) at 1 kHz. In addition, ε'' shows a maximum value of 49.42 ($x = 0.2$) and a minimum value of 36.33 ($x = 0.3$).

ABSTRAK

Ferit kobalt telah menarik minat yang tinggi para saintis disebabkan kepentingan aplikasinya dalam pelbagai bidang sains dan teknologi. Namun begitu, sifat magnet partikel tersebut sangat bergantung terhadap saiz, bentuk dan kandungan ketulenan bahan partikel nano tersebut. Justeru, keperluan di dalam menghasilkan proses fabrikasi yang lebih baik dan mudah serta kebolehupayaan mengawal saiz partikel nano yang terhasil sangat diperlukan. Penyelidikan ini melibatkan kajian terhadap struktur, magnet, sifat dielektrik dan morfologi bagi $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ partikel nano ferit (x = 0.0, 0.1, 0.2, 0.3, 0.4, 0.5), di mana ia telah disintesis melalui kaedah pemendakan kimia. Tambahan lagi, komposit nano rangka-teras $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$/Polianalina telah berjaya disintesis melalui kaedah pempolimeran kimia. Sampel ferit yang terhasil telah disinter pada suhu 700 °C, 800 °C, 900 °C dan 1000°C selama 8 jam. Pembelauan sinar-X serbuk ferit menunjukkan bahawa bahan ferit tersebut adalah spinel berfasa tunggal dan berbentuk kubik. Saiz kristal bagi partikel nano $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ telah diperolehi dalam julat 25-40 nm. Spektrum infra merah bagi sampel disentesis menunjukkan dua jalur serapan pencirian ferit spinel pada 585–595 cm^{-1} dan 390–400 cm^{-1}, masing-masing merujuk kepada getaran ikatan tetrahedral dan oktahedral. Imej mikroskop elektron pengimbas pancaran medan dan mikroskop elektron transmisi bagi partikel nano menunjukkan perbezaan agregat pada suhu pensinteran dan konsentrasi yang berbeza. Gabungan antara Ni-, Mg- sebagai pengganti dalam ferit kobalt menunjukkan bahawa penggantian ion Mg^{2+} bagi Fe memberi kesan dan perubahan yang ketara terhadap sifat magnet dan dielektrik pada suhu bilik. Nilai pemagnetan tepuan (M_s) dan daya koersif (H_c) meningkat dengan penambahan konsentrasi Mg sehingga x = 0.1. Dengan peningkatan penggantian Mg^{2+}, M_s dan H_c masing-masing menunjukkan peningkatan daripada 57.35 emu/g (x = 0.0) kepada 61.49 emu/g (x = 0.1) dan daripada 603.26 Oe (x = 0.0) kepada 684.11 Oe (x = 0.1). Sebaliknya, M_s menyusut daripada nilai maksimum 12.00 emu/g (x = 0.1) kepada nilai minimum 5.39 emu/g (x = 0.4) apabila Polianalina ditambah ke atas ferit. Namun, H_c didapati meningkat daripada nilai maksimum 766.94 Oe (x = 0.1) kepada nilai minimum 646.17 Oe (x = 0.0). Pada 1 kHz, pemalar dielektrik ε' menunjukkan nilai maksimum 86.22 bagi x = 0.1 dan nilai minimum 56.67 bagi x = 0.3. Sebagai tambahan, kehilangan dielektrik ε'' menunjukkan nilai maksimum 10.98 bagi x = 0.2 dan nilai minimum 9.45 bagi x = 0.0. Bagi komposit nano, ε' mencapai nilai maksimum 68.32 (x = 0.1) dan nilai minimum 46.73 (x = 0.3) pada 1 kHz. Tambahan pula, ε'' menunjukkan nilai maksimum 49.42 (x = 0.2) dan nilai minimum 36.33 (x = 0.3).

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LIST OF ABBREVIATIONS

Ag	-	Argentum
Al	-	Aluminum
a-PANI	-	Amorphous Polyaniline
APS	-	Ammonium Persulfate
Au	-	Aurum
Bi	-	Bismuth
c-PANI	-	Crystalline Polyaniline
Co	-	Cobalt
Cr	-	Chromium
CTAB	-	Cetyl Trimethylammonium Bromide
Cu	-	Copper
DBSA	-	Dodecyl Benzene Sulfonic Asid
DCTATPR	-	Direct Current Transferred Arc Thermal Plasma Reactor
EB	-	Emeraldine Base
EM	-	Electromagnetic
ES	-	Emeraldine Salt
ESR	-	Electron Spin Resonance
EXAFS	-	X-Ray Absorption Fine Structure
FCC	-	Face-centered Cubic
Fe	-	Ferrum (iron)
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared
Hg	-	Mercury
HRTEM	-	High Resolution Transmission Electron Microscopy
IL	-	ionic liquid

LEDs	-	Light emitting diodes
Li	-	Lithium
Mg	-	Magnesium
MHz	-	Megahetz
Mn	-	Manganese
MnO	-	Manganese Oxide
Ni	-	Nickel
PANI	-	Polyaniline
Sb	-	Antimony
SC	-	specific capacitance
SCS	-	solution combustion synthesis
TEM	-	Transmission Electron Microscopy
Ti	-	Titanium
VSM	-	Vibrating Sample Magnetometer
XRD	-	X-ray Diffractometer
Zn	-	Zinc
[BMIM]Br	-	1-Butyl-3-Methyl-Imidazolium Bromide

LIST OF SYMBOLS

σ_{ac}	-	AC conductivity
Å	-	Angstrom
ω	-	applied frequency
N_A	-	Avogadro's number
θ	-	Bragg angle
C_0	-	capacitance of the condenser with the region of space (without vacuum)
C	-	capacitance of the condenser when the space is filled with dielectric medium
q	-	charged of an electron
V_{cell}	-	cell volume
H_c	-	coercivity
α_1	-	cosines of the angles between M_s and the x axes
T_c	-	crystalline temperature
D	-	diameter of crystallite
ΔE	-	different of energy
dI	-	distance between centers of two charges
ρ_B	-	density of bulk
ρ_x	-	density of x-ray
μ	-	electronic dipole moment
K_1	-	first order of cubic anisotropy constants
R•	-	free radical
v	-	frequency of radiation
g	-	g-factor
D	-	grain size
δ	-	inversion parameter

a	-	lattice constant
β	-	Bohr magneton
β	-	line broadening at half the maximum intensity (FWHM)
ΔH_{PP}	-	peak-to-peak line width (in *G*)
$\Delta H_{1/2}$	-	line width (in *G*) at half-height of the absorption peak
M	-	magnetization
B	-	magnetic field strength
n_B	-	magnetic moment
μ_0	-	magnetic permeability of free space
K	-	magnetocrystalline anisotropy
M_w	-	molecular mass or molecular weight
M	-	monomer
Z	-	number of formula units in a unit cell
N_2	-	octahedral cluster
h	-	Planck constant
ε'	-	permittivity real
ε''	-	permittivity imaginary
μ''	-	permeability imaginary part
μ'	-	permeability real part
P	-	porosity
ε_r	-	relative permittivity
τ^2	-	relaxation time
M_r	-	remanence
M_s	-	saturation magnetization
m_S	-	saturation moment
K	-	shape factor
K_1	-	second order of cubic anisotropy constants
S	-	specific surface
v_1	-	tetrahedral cluster
φ	-	volume fraction
d_x	-	X-ray density
λ	-	X-ray wavelength

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

In recent years, the synthesis and characterization of ferrites and their modifications have attracted more attention due to their remarkable electrical, magnetic and magneto-electric properties, which are interesting for scientific and technological applications. Ceramic-like ferromagnetic materials have been considered as highly important electronic materials for more than half a century. According to Still, a Magnetite (Fe_2O_4) which is known as a natural genuine ferrite has been recognized more than two millennium years ago by ancient people due to its magnetism and was used as a mariner's compass in China [1]. Nanoscale ferrites are likely to become an integral part of the future nanotechnology primarily as their electrical, permittivity and magnetic elements [2]. The properties of ferrites are dependent on size, shape, distribution of particles and chemical composition, which are in turn influenced by the synthesis technique.

Among ferrites, cobalt ferrites, $CoFe_2O_4$ are the most widely used magnetic materials for having low cost and high performance in high frequency applications. $CoFe_2O_4$ with inverse spinel structure is well known for having a relatively large magnetic anisotropy, moderate saturation magnetization, remarkable chemical stability, and mechanical hardness [3]. These properties, along with their great physical and chemical stability, make $CoFe_2O_4$ nanoparticles suitable for potential applications in electromechanical transducers, biomedicine and magnetic data storage systems. However, the magnetic character of the particles used for many applications depends crucially on the size, shape and purity of these nanoparticles [4]. Hence the need for developing fabrication processes that are relatively simple and yield controlled particle sizes is desired. Several, popular methods including co-precipitation, thermal decomposition and/or reduction, micelle synthesis, hydrothermal synthesis, and laser pyrolysis techniques can all be directed at the synthesis of high-quality magnetic nanoparticles [5].

Concurrently, nanocomposite materials combining an electrically conducting polymer and magnetic nanoparticles also have been intensively investigated due to their fascinating application such as electrochemical display devices [6], molecular electronics [7], sensors [8], electrical-magnetic interference (EMI) shields [9][10], and microwave absorption materials[11]. On top of this, the synthesis of magnetic particle/polyaniline nanocomposites not only achieves a combination of their properties, but also overcomes the shortcomings in the preparation of inorganic nanomaterials, according to reports related to their preparation and properties [12]. Among the conducting polymers, polyaniline, (PANI) has received a great deal of attention due to its unique electro-physico-chemical behavior, environmental properties and relatively easy synthesis.

In this work, nickel-magnesium substituted cobalt ferrite Co-Ni-Mg Fe_2O_4 and their substituted ferrite/polyaniline nanocomposites (Co-Ni-Mg-Fe_2O_4/PANI) have been successfully synthesized. Co-Ni-Mg-Fe_2O_4 is the magnetic core, and PANI is the conducting shell to become core-shell structure. The Co-Ni-Mg-Fe_2O_4 nanoparticles were prepared by co-precipitation method and the Co-Ni-Mg-Fe_2O_4/PANI composites were synthesized via polymerization method. The structural, morphological, magnetic and dielectric properties were investigated in details through X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscope (FESEM), Transmission Electron

Microscope (TEM), Vibration Sample Magnetometer (VSM), Electron Spin Resonance (ESR) and Two Probe of Impedance Analyzer.

1.2 Problem Statement

A potential of Co ferrites has extensively been explored for highly important applications in various fields of science and technology. Their structural, morphology, magnetic and electrical properties would be the main indicators as functional magnetic materials as for specific applications. These properties of ferrite are very much sensitive to technique adopted for the synthesis, preparative parameters, initial ingredients and heat treatment. Magnetic properties of ferrites can be suitably tailored by varying the composition of cations. Due to the above parameters, there may be a change in cations distribution, which may result in the unexpected magnetic, electrical and dielectric properties. This means that by changing the type of the magnetic ions as well as by selective substitution of non-magnetic atoms on the tetrahedral (A) and octahedral (B) sites, will lead to interesting spin configurations.

Note that, there are several studies focusing on the effect of other co-substituted ferrites. The influence of magnetic ion substitution such as Mn^{2+} [13] and Gd^{3+} [14] on various structural, magnetic, electric and dielectric properties of $CoFe_2O_4$ have been reported in the literature. Nevertheless, several researchers have reported on non-magnetic ions such as Al^{3+} [15], Y^{3+} [16], Zn^{2+} [17], Cu^{2+} [18] or Cd^{2+} [19] substituting $CoFe_2O_4$. Magnesium ions with non-magnetic nature are known for achieving control over magnetic parameters in developing technologically important materials and they have strong B sites preference.

It was observed that when the non-magnetic divalent cations such as Zn, Mg, are substituted for magnetic cations such as Ni, Co, Mn, the saturation magnetization

(M_s) increase up to 50% substitution, beyond which these values decrease. In addition, Mg^{2+} ions causes appreciable changes in the structural and electrical properties of the ferrites [20] [21]. Thus, the substitution of magnetic Ni^{2+} and non-magnetic Mg^{2+} ions on Co ferrite will markedly modify the magnetic properties. The aim of this work is to study the structural properties of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$, $0.0 \leq x \leq 0.5$ in step of 0.1 as a function of Ni and Mg contents and to define their correlation with morphology, magnetic and dielectric properties. Since Ni-Mg substituted Co ferrite nanoparticles in series $0.0 \leq x \leq 0.5$ is a new contributor in family of mixed ferrites, it would be considered as pioneer to combine this material into conductive polyaniline matrix to develop a core-shell structure of nanocomposites. The structure of core-shell for nanocomposites is categorized as versatile by combining the electrical and magnetic properties, where this is also has a plenty rooms need to be explained and explored.

1.3 Objectives of Research

The main objectives of this research are:

1. To synthesize single-phase $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ ($0.0 \leq x \leq 0.5$) in step of 0.1 powdered materials by co-precipitation method.

2. To determine the influence of Ni^{2+} and Mg^{2+} concentration on the structural, particle size, magnetic and dielectric properties of the $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ ferrites material at 900 °C.

3. To determine the influence of sintering temperature on the structural, particle size and magnetic properties of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ ferrites material.

4. To determine the influence of polyaniline embedded on the magnetic and dielectric properties of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ ferrites material.

1.4 Scope of Research

In this work, ferrite nanoparticles phase of $Co_{0.5}Ni_{0.5-x}Mg_xFe_2O_4$ with x = 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5 were synthesized using co-precipitation method. The synthesis of the core-shell ferrite/PANI nanocomposites using polymerization method. The stoichiometric molar amounts of $Ni(NO_3)_2{\cdot}6H_2O$, $Mg(NO_3)_2{\cdot}6H_2O$, $Co(CH_3COO)_2{\cdot}4H_2O$ and $Fe(NO_3)_3{\cdot}9H_2O$ were introduced. The ferrites samples were sintered at selected sintering temperatures of either 700 °C, 800 °C, 900 °C and 1000°C for 8 hours. Determination of structural properties and morphology of ferrite nanoparticles and nanocomposites have been performed by using XRD, FTIR, FESEM and TEM. Determination of magnetic properties of ferrite nanoparticles and nanocomposites were performed by using ESR and VSM. Determination of dielectric properties of ferrite nanoparticles and nanocomposites were performed by two-probe method using impedance analyzer.

1.5 Significant of Research

The combination of Co, Ni and Mg to be ferrite nanoparticles with specific formula and in the form of ferrite/PANI nanocomposites are novel. Our aim is to merge the advantages of both Co and Ni ferrites (ferromagnetic behavior) and to utilize from the existence of Mg (paramagnetic behavior) in small constant ratio to ensure the large magnetization of the ferrites. It is unexpected that the addition of Mg improves the magnetization by high saturation magnetization, higher dielectric properties and low loss over a wide range of frequency. The properties of Co ferrites are remarkable such as high coercivity, moderate saturation magnetization, strong anisotropy along with good mechanical hardness and chemical stability. On the other hand, Ni ferrites possess high resistivity and permeability at high frequencies. The chosen methods of co-precipitation and polymerization are economical. The simple, repeatable, homogeneous and environmental friendly preparation may contribute towards the controlled growth of high quality ferrite nanopowders, potentially as candidates for memory storage media and microwave devices.

1.6 Organization of the Research

This thesis is divided into seven chapters as follow:

Chapter One provides a brief introduction of the research under taken. This includes the research background and overview, problem statement, objectives, scope of research, significant of research and organization of the research.

Chapter Two provides a comprehensive review of background related to this topic and current knowledge on spinel cobalt ferrite and their chemical composition. It covers fundamental of magnetism, growth mechanism of cobalt ferrites and their composites, including the formation of core-shell ferrite/polyaniline nanocomposites. This includes some theoretical aspects involves and uses in this project.

The experimental work employed in this study is described in details in Chapter Three. It includes the chemical used, formulation and preparation of Ni-Mg substituted cobalt ferrites and core-shell formation of ferrites/polyaniline nanocomposites samples. The structural, morphology, magnetic and dielectric properties determination using XRD, FTIR, FESEM, TEM, VSM, ESR and impedance analyzer are also described in detail in this chapter.

The experimental results and finding of the research are presented in Chapter Four. It includes the characterization of ferrite samples in term of different ratio of Ni and Mg substitution, different sintering temperature and the formation of core-shell structure on $Co_{0.5}Ni_{0.4}Mg_{0.1}Fe_2O_4$/ polyaniline nanocomposites. This chapter is

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