SYNTHESIS AND CHARACTERIZATION OF BISMUTH FERRITE NANOPARTICLES WITH SILLENITE PHASE VIA SOL GEL AUTO COMBUSTION ROUTE

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Dedicated to my beloved parents,
Abd Mubin Bin Othman and Kalsom Begum Binti Fazal Ahmad ,
to my siblings, family and family in law,
for the continuos support and prayers.
To my dearest wife, Nur Fatin Binti Sulaiman,
to my precious daughter, Nur Aleesa Binti Mohamad Helmi,
my source of strength,
thanks for always being there for me.
To my friends,
thank you so much,
for their patience, support, love and encouragement.
May Allah bless all of you...

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ABSTRACT

Bismuth ferrite $BiFeO_3$ (BFO) is one of the multiferroic materials that has high ferroelectric behavior and weak magnetic orders. The intrinsic coupling between the electric polarization and magnetic moments makes BFO potential material for advanced technological applications in different fields. The crystal structure plays very crucial role towards electric and magnetic properties of the nanoparticles. Determination and synthesis of the nanoparticles with specific phase is very important for specific applications. Substitution or doping of ferrites with other metal ions allows variations in their electric and magnetic properties which can be optimized for particular applications. Therefore, this research focuses on the synthesis and characterization of BFO nanoparticles with sillenite phase by sol gel auto combustion route. BFO is doped with cobalt, nickel, and magnesium to explore the role of dopants, concentration of dopants and annealing temperature especially for structural (phase) and electro-magnetic characteristics. Three different doping elements i.e. cobalt, nickel, and magnesium are used to study the role of dopants inside the BFO $Bi_{1-x}Co_xFeO_3$, $Bi_{1-x}Ni_xFeO_3$, and $Bi_{1-x}Mg_xFeO_3$ for five different concentrations 5%, 10%, 15%, 20% and 25% in context of structural and electro-magnetic characteristics. Structural and electro-magnetic characteristics of synthesized doped BFO nanoparticles are investigated using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), vibrating sample magnetometer (VSM) and energy dispersive X-ray (EDX). Results show that, the annealing temperature plays a crucial role towards the phase formation and crystallinity of BFO. At lower annealing temperature 400°C, all BFO nanoparticles possessed R3c rhombohedral phase with poor crystal structure. However, as the annealing temperature is increased, significant increase in the growth of sillenite phase is observed. Dopants such as Co, Mg, and Ni significantly improve the crystal structure, particle size and electro-magnetic characteristics of BFO. Cobalt-doped BFO nanoparticles have shown good ferromagnetic behaviour with high remnant magnetization and coercivity. Ni and Mg-doped BFO possess, super paramagnetic structure with low remnant magnetization and coercivity. Dopants significantly reduce the particle size of the host BFO. The increase in dopant concentration in BFO nanoparticle considerably promotes the formation of sillenite phase. Highly crystalline sillenite phase for BFO nanoparticle is obtained for 25% dopant concentration.

ABSTRAK

Bismut ferit $BiFeO_3$ (BFO) adalah salah satu bahan multiferoik yang memiliki sifat feroelektrik yang tinggi dan susunan magnet yang lemah. Gandingan intrinsik antara kekutuban elektrik dengan momen magnet menjadikan BFO bahan berpotensi untuk aplikasi teknologi maju dalam bidang yang berbeza. Struktur hablur memainkan peranan penting terhadap sifat elektrik dan magnet nanozarah. Penentuan dan sintesis nanozarah dengan fasa tertentu penting untuk aplikasi tertentu. Penggantian atau pengedopan ferit dengan ion logam lain membolehkan variasi dalam sifat elektrik dan magnet yang boleh dioptimumkan untuk aplikasi tertentu. Oleh itu, kajian ini memberi tumpuan kepada sintesis dan pencirian nanozarah BFO dengan fasa sillenit oleh laluan sol gel pembakaran auto. BFO di dip dengan kobalt, nikel, dan magnesium untuk meneroka peranan dopan, kepekatan dopan dan suhu penyepuh lindapan terutamanya untuk ciri struktur (fasa) dan elektro-magnet. Tiga elemen pengedopan yang berbeza yakni kobalt, nikel, dan magnesium digunakan untuk mengkaji peranan dopan dalam BFO $Bi_{1-x}Co_xFeO_3$, $Bi_{1-x}Ni_xFeO_3$, dan $Bi_{1-x}Mg_xFeO_3$ untuk lima kepekatan yang berbeza 5%, 10%, 15%, 20% dan 25% dalam konteks ciri struktur dan elektro-magnet. Sifat struktur dan elektro-magnet bagi nanozarah BFO terdop yang disintesis disiasat menggunakan pembelauan sinar-X (XRD), mikroskopi imbasan elektron pancaran medan (FESEM), magnetometer sampel bergetar (VSM) dan sinar-X sebaran tenaga (EDX). Keputusan menunjukkan bahawa, suhu penyepuh lindapan memainkan peranan penting terhadap pembentukan fasa dan penghabluran BFO. Pada suhu penyepuh lindapan yang lebih rendah 400°C, semua nanozarah BFO mempunyai fasa rombohedral R3c dengan struktur hablur yang lemah. Walau bagaimanapun, apabila suhu penyepuh lindapan meningkat, peningkatan ketara pertumbuhan fasa sillenit diperhatikan. Dopan seperti Co, Mg, dan Ni secara ketara meningkatkan struktur hablur, saiz zarah dan ciri elektro-magnet bahan BFO. Nanozarah BFO terdop kobalt telah menunjukkan sifat feromagnet yang baik dengan kemagnetan baki dan koersiviti yang tinggi. BFO terdop Ni dan Mg mempunyai struktur super paramagnet dengan kemagnetan baki dan koersiviti yang rendah. Dopan dengan ketara mengurangkan saiz zarah BFO. Peningkatan kepekatan dopan dalam nanozarah BFO sangat menggalakkan pembentukan fasa sillenit. Fasa sillenit hablur yang tinggi untuk nanozarah BFO diperoleh pada kepekatan dopan 25%.

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LIST OF ABBREVIATIONS

BFO	-	Bismuth Ferrite
BFCO	-	Co-doped Bismuth Ferrite
BFMO	-	Mg-doped Bismuth Ferrite
BFNO	-	Ni-doped Bismuth Ferrite
EDX	-	Energy dispersive X-ray
FESEM	-	Field emission scanning electron microscopy
FWHM	-	Full width half maximum
RhB	-	Rhodamine B
TGA-DSC	-	Thermogravimetry Analysis-Differential Scanning Calometry
VSM	-	Vibrating sample magnetometer
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

μ_B	-	Magnetic moment
Å	-	Angstrom
a	-	Lattice constant
°C	-	Degree Celsius
d	-	Interplanar spacing
D	-	Crystallite size
H	-	Magnetic field
H_c	-	Coercivity
M	-	Magnetization
M_r	-	Retentivity / remanence magnetization
M_s	-	Saturation Magnetization

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Ferrites, the ceramic like materials with unique electric and magnetic characteristics are iron oxide with one or more other metals such as manganese, cobalt, barium, nickel, magnesium, aluminium, and iron itself in chemical mixture (Evans et al., 2016). They are brittle, hard and polycrystalline consisting of a large number of minor crystals. In common practice, a ferrite is defined by $M(Fe_xO_y)$, where M symbolizes a metal which established divalent bonds, such as nickel ferrite and manganese ferrite $NiFe_2O_4$ and $MnFe_2O_4$. Ferrites possess a certain behavior of magnetism named as ferrimagnetism, which is different as compared to the form of ferromagnetism possess by metals such as cobalt, nickel, and iron (Boudouris, 2016). Therefore, the magnetic moments of constituent atoms for ferrite materials line up in two or different orientations. Thus, partial cancellation of the magnetic moment left a net magnetic field on ferrites which is less strong as compared to ferromagnetic material. This asymmetry in atomic orientations is due to existence of two or more different types of metal ions which leads to net magnetism in a peculiar crystalline structure (Chandamma et al., 2017). Ferrites can possess different type of crystalline structures as spinel, garnet, perovskite, and hexagonal. The most significant characteristics of ferrites are high magnetic permeability and electrical resistance which makes them suitable materials for the memory cores of digital devices, where the small ferrite ring is capable of storing binary bits of information (Kakade et al., 2016).

Multiferroics exhibit ferroelectricity and ferromagnetism simultaneously, which have attracted much interest owing to the ability to change the magnetic states by applying an E-field through magnetoelectric (ME) coupling (Dong et al., 2015). Ferroelectric material is generally categorized as pyroelectric. However, not all the pyroelectric materials exhibit ferroelectric behavior. Below the Curie temperature (the transition temperature of ferroelectric material), pyroelectric and ferroelectric materials tend to possess spontaneous polarization or electric dipole moment. This polarization can be manipulated or reoriented through the presence or absence of electric field. The reversal of this orientation of dipole moment is called 'switching'. For non-polar material, above the Curie temperature, is the paraelectric phase. The high magnitude of the spontaneous polarization is obtained below the Curie temperature (Ye et al., 2015). Ferromagnetic materials are used in a variety of applications such as capacitors, storage media devices, transducers, magnetic tape recording, transformers, sensors, photocatalytic activity, etc. All these applications are operated in ultra-low power consumption devices by manipulating the spin of electron using external electric field (Liu and Sun, 2014). Ferromagnetic materials are doped with other elements to enhance coercivity and retentivity while retaining the actual structure.

Bismuth ferrite $BiFeO_3$ (BFO) is one of the promising multiferroic materials due to its high ferroelectric behavior (below Tc ~ 830 ºC) and weak magnetic orders (below T_N ~ 370 ºC) (Li et al., 2007). It possesses a distorted structure of R3c rhombohedral perovskite and the unit cell has a hexagonal symmetry. Moreover, at room temperature, the BFO possesses behavior of antiferromagnetic with G-type spin formation along the pseudocubic [111]c or rhombohedral [001]h direction and having an overlaid unequal cycloid spin structure with a periodicity of 620 Å along the [110]h axis. Thus, parting a remaining moments from canted spin structures leads to weak magnetic properties (Lama et al., 2009). BFO has potential to play a promising role in next-generation memory devices, photocatalytic activities, semiconductor sensors, etc, due to the intrinsic coupling between the electric polarization and magnetic moments and photo sensitive characteristics (Jalil, 2017).

1.2 Problems Statement

The advancement in field of nanotechnology from nano-particle preparation to device fabrication evoke on account of the development of nano-science (Wu et al., 2015b). Nanoparticulate ferrites carry huge potential for advanced applications in different fields. The use of these nano-materials require fine control over the size distribution (Zhang and Zhao, 2017). Various synthetic routes such as hydrothermal, co-precipitation, electrochemical, arc discharge plasma, and sol gel are used to synthesize nanoparticles (Hu et al., 2015). However, sol-gel is an attractive, cost effective and efficient route to synthesize bismuth ferrite due to the simple preparation without involving any sophisticated machines (Luo et al., 2015). This technique allows good control of the concentration of the precursor to effectively yield optimum results.

Majority of investigations have been focused on the synthesis of BFO nanoparticle with pure or primary phase. Secondary phase or other phases have received less attention. Bismuth ferrite nanoparticles have hexagonal phase which belong to R3c space group with formulation of $BiFeO_3$. But, it has rarely been reported the synthesis and analysis of other phases such as sillenite phase with I23 Cubic structure with formulation of $Bi_{25}FeO_{40}$. Determination of the nanoparticle phase is very important for specific application. Substitution of ferrites with other metal ions allows variations in their electric and magnetic properties which can be optimized for specific applications. By controlling substituent ratio, the magnetic moment and coercivity of ferrite nanoparticles can be optimized. The substituents play key role on the structure, particle size, morphology, and magnetic properties.

Therefore, this research focuses on the synthesis and characterization of bismuth ferrite nanoparticles with sillenite phase by sol gel auto combustion route. Bismuth ferrite are doped with cobalt, nickel, and magnesium to explore the role of dopants especially for its structural (phase) and electro-magnetic characteristics.

1.3! Objectives of Research

The main objective of this research is to synthesize and characterize bismuth ferrite nanoparticles with sillenite phase by introducing dopant as nickel, cobalt, and magnesium via sol gel auto combustion route. The specific objectives are:

a. To synthesize doped bismuth ferrite (BFO) nanoparticles with sillenite phase via sol-gel auto combustion route.
b. To investigate the effect of annealing temperature on phase formation.
c. To investigate role of dopants on structural, phase and electro-magnetic characteristic of BFO nano-particles.

1.4! Scope of Research

This research focuses on synthesizing the doped bismuth ferrite nanoparticles by using wet chemical deposition route such as sol gel auto combustion method. High purity nitrate salts such as bismuth nitrate and iron nitrate are used for the growth of the nanoparticles. Citric acid is used as a fuel for the combustion and as chelating agent.

Three different doping elements cobalt, nickel, and magnesium are used to study the role of dopants inside the bismuth ferrite based on the structural and electro-magnetic characteristics. Five different doping concentrations of 5%, 10%, 15%, 20%, and 25% are used for each doping element. The phase formation of the nanoparticle is studied for three different annealing temperatures of 400°C, 500°C, and 600°C.

The structural and morphology analysis of the nanoparticle is conducted using x-ray diffraction spectroscopy (XRD) and Field emission scanning electron microscopy (FESEM) respectively. The electro-magnetic characteristics are studied using Vibrating sample magnetometer (VSM). The elemental composition confirmation is determined using Energy Dispersive X-ray (EDX) spectroscopy.

1.5 Significance of Study

The present study significantly contributes to provide better understanding about the role of dopants and annealing temperature towards the phase formation of BFO structure. Understanding of dopant concentration ratio would help to fabricate the nano-particles with optimized characteristics for specific applications. This study will provide a baseline for choosing the suitable dopant element for specific applications and other purposes.

1.6 Thesis Outline

The thesis is organized as follows. Chapter 1 gives an overview of the thesis. Chapter 2 gives a critical analysis on the research involving bismuth ferrite from the experimental perspectives. Chapter 3 describes the detail methodology of preparing, synthesis and characterization of the doped BFO nanoparticles under different experimental conditions. Chapter 4 discusses on the results and analysis based on the experiments. Chapter 5 conclude the research with summary and recommendation for future work.

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