UNIVERSITI PUTRA MALAYSIA

ONE-POT SYNTHESIS AND CHARACTERIZATIONS OF (SrFe12O19)X - (Ni0.5Zn0.5Fe2O4)1-X NANOCOMPOSITE FERRITES

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Nanocomposite ferrites are known as an exchange - spring magnet when they are well exchange coupled. This phenomena has been studied by synthesized $(SrFe_{12}O_{19})$ - $(Ni_{0.5}Zn_{0.5}Fe_2O_4)$ nanoparticles using a one-pot thermal treatment method. This technique is a single reaction mixture involving metal nitrates and the capping agent which is polyvinyl pyrrolidone (PVP). Nanocomposite ferrite nanoparticles were succeed to synthesize via single reaction technique. The starting materials used were PVP as the capping agent, deionized water and metal nitrates as the precursors. This study was divided into 3 sections. For the first part, we investigated the effect of PVP as a capping agent. Composite ferrites with 20% of hard and 80% of soft ferrites were fabricated by varying the concentrations of PVP (0 to 0.06 g/ml). For the second part, we studied the influence of phase distributions on the particles. Nanocomposite ferrites of $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ were synthesized by varying the contents of *x* (0.1 to 0.9). The last part of this research was on calcination temperatures. Samples of $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ where (*x* = 0.8 and 0.9) were calcined at 600 ºC to 1000 ºC for 3 hours.

Several characterizations were carried out such as X-ray Diffraction (XRD), Vibrating Sample Magnetometer (VSM), Fourier Transform Infra-red (FT-IR), Thermogravimetric Analysis (TGA), microstructure analysis using Field Emission Scanning Electron Microscope (FESEM) and Transmission Electron Microscope (TEM). From the XRD analysis, both soft and hard phases were coexist for every samples. The optimum concentration of PVP was 0.06 g/ml which gave a better magnetic properties compared to other concentrations of PVP used. In addition, $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ nanocomposite with x = 0.9 shows highest $\boldsymbol{H_c}$ value with 5692 G and the optimum

calcination temperature was found out to be at 800 ºC. From the analyses show that the hard/soft nanocomposite ferrites were able to synthesize by this one-pot thermal treatment method. Therefore, this technique is one of the promising method to fabricate nanocomposite permanent magnet.

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

SINTESIS SECARA SERENTAK DAN PENCIRIAN BAGI $(SrFe_{12}O_{19})_x$ - $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ NANOKOMPOSIT FERIT

Oleh

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Nanokomposit ferit dikenali sebagai pertukaran spring magnet apabila bertukar ganding dengan baik. Fenomena ini telah dikaji dengan mengsintesis $(SrFe_{12}O_{19})$ - $(Ni_{0.5}Zn_{0.5}Fe_2O_4)$ nanopartikel menggunakan kaedah rawatan termal satu bekas. Teknik ini adalah campuran reaksi tunggal melibatkan logam nitrat and ejen pelekat iaitu polyvinyl pyrrolidone (PVP). Nanokomposit ferit nanopartikel berjaya di sintesis dengan teknik reaksi tunggal ini Bahan permulaan yang digunakan adalah PVP sebagai ejen pelekat, air ternyahion dan logan nitrat sebagai pelopor. Kajian ini dibahagikan kepada 3 bahagian. Bagi bahagian pertama, kami mengkaji pada kesan PVP sebagai ejen pelekat. Komposit ferit dengan 20% ferit keras dan 80% ferit lembut dibentuk dengan mempelbagaikan konsentrasi PVP (0 to 0.06 g/ml). Untuk bahagian kedua, kami mengkaji pengaruh pengagihan fasa ke atas partikel. Nanokomposit ferit $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ telah di sintesis dengan mempelbagaikan kandungan x (0.1 to 0.9). Bahagian terakhir bagi kajian ini adalah ke atas suhu pembakaran. Sampel $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ dimana (x = 0.8 and 0.9) dibakar pada 600 ºC to 1000 ºC untuk 3 jam.

Beberapa pencirian telah dijalankan seperti pembelauan sinar-X (XRD), magnetometer sampel bergetar (VSM), inframerah transformasi Fourier (FT-IR), penganalisis termogravimetri (TGA), analisis mikrostruktur menggunakan mikroskop electron pengimbas pancaran medan (FESEM) dan mikroskop electron penghantaran (TEM). Daripada analisis XRD, kedua-dua fasa ferit lembut dan keras wujud bagi setiap sampel. Konsentrasi optimum bagi PVP adalah 0.06 g/ml memberikan ciri magnetik yang lebih baik berbanding lain-lain konsentrasi PVP yang digunakan. Tambahan pula, $(SrFe_{12}O_{19})_x$ – $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ nanokomposit dengan x = 0.9 menunjukkan nilai $\boldsymbol{H_c}$ yang tertinggi dengan 5692 G dan suhu pembakaran optimum telah didapati pada 800 ºC. Analisa

menunjukkan bahawa nanokomposit ferit keras/lembut mampu di sintesis dengan kaedah rawatan termal satu bekas ini. Oleh itu, teknik ini adalah salah satu kaedah yang menjanjikan bagi mengahasilkan magnet kekal nanokomposit.

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REMOVE

I certify that a Thesis Examination Committee has met on 30th November 2017 to conduct the final examination of Hartini Binti Abdullah Ahmad on her thesis entitled "ONE-POT SYNTHESIS AND CHARACTERIZATION OF $(SrFe_{12}O_{19})_x$ - $(Ni_{0.5}Zn_{0.5}Fe_2O_4)_{1-x}$ NANOCOMPOSITE FERRITES" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

H_c	Coercivity
L_{ex}	Exchange length
FT-IR	Fourier Transform Infra-red
FESEM	Field Emission Scanning Electron Microscope
PVP	Poly(vinylpyyrolidone)
M_r	Remanence
M_s	Saturation magnetization
TGA	Thermogravimetric Analysis
TEM	Transmission Electron Microscope
VSM	Vibrating Sample Magnetometer
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background Studies

This study is focused on synthesis of nanocomposite magnet using ferrite nanoparticles. Ferrite was chosen because of the advantages that it offers such as low cost, good in heat resistance and high in corrosion resistance (Sugimoto, 1999). Other materials such as rare earth also have their own respective properties. However, ferrite seems to fulfill the requirements of producing an environmentally friendly magnet. In addition, ferrite does not need vacuum atmosphere to produce composite ferrite as it can be handle in ambient condition due to its properties that is not so corrosive. For this research, the hard ferrite was $SrFe_{12}O_{19}$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ as a soft ferrite were chosen to fabricate nanocomposite permanent magnet. Due to high in magnetization, anisotropic constant and also moderately high in Curie temperature (Sugimoto, 1999), thus make $SrFe_{12}O_{19}$ as better candidate compared to other M-type ferrite. Nickel zinc ferrite mostly chosen due to high in saturation magnetization, magnetic permeability and low dielectric losses (Mathew and Juang, 2007).

The composite ferrite was synthesized by using a thermal treatment method where it involves only a single reaction mixture while several other techniques required double reaction mixture. It is also simple, cost and time effective, no by product effluents, low calcination temperature and environmentally friendly technique (Naseri et al., 2010). Compared to other techniques, this method is newly established for the fabrication of composite magnetic materials. There are few other techniques like self-propagating combustion method (Moon et al., 2007), used to produce composite nanoparticles, yet some of the methods involve in complicated set up and dealing with high calcination temperature.

1.2 Nanocomposite Permanent Magnet

There are several types of permanent magnets and one of them is ferrite which is mostly chosen by researchers nowadays due to its magnetic properties, good in corrosion resistance and low cost. Ferrite can be divided into soft ferrite and hard ferrite. Both of these ferrites exhibit magnetic properties which completed each other when they are combined. For example, soft ferrite has low coercivity while high in magnetization and hard ferrite gives high value in coercivity while low in magnetization. As both soft and hard ferrites are well and homogeneously mixed, nanocomposite ferrite or exchange spring magnet can be obtained if both the ferrites phases are grown together in the same reaction mixture (Hazra et al., 2012; Hazra et al., 2014; Hazra et al., 2015). Therefore, the combination of these two ferrites can lead to a better magnetic properties with high in magnetization from soft and high in coercivity from hard ferrite (Roy et al., 2009; Rai et al., 2014).

However, there is a criterion for the exchange spring magnet to occur which a largely reversible demagnetization curve in conjunction with an isotropic saturation remanence ratio $\boldsymbol{M_r/M_s} \geq 0.5$ may be considered (Kneller and Hawig, 1991). Thus, loop obtained will acts as a magnetically one phase behavior even though crystallographically shows two phases magnetic materials (Hazra et al., 2012; Hazra et al., 2014; Hazra et al., 2015). In brief, a smooth loop of exchange spring magnet with no kink at the second quadrant is obtained. The example of an ideal hysteresis loop of exchange spring magnet is shown in Figure 1.1.

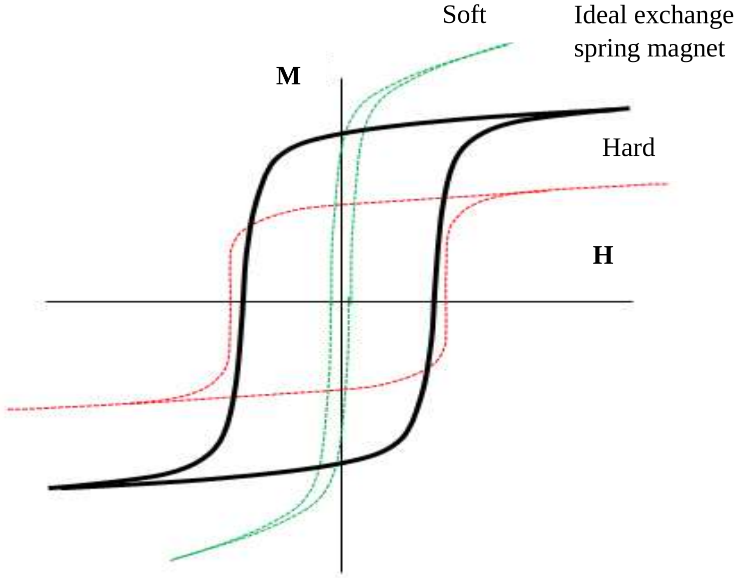

Figure 1.1: Illustration of magnetizing loops for exchange coupled hard-soft magnetic phases (Jimenez-Villacorta and Lewis, 2014)

From the hysteresis loops illustrated in Figure 1.1 clearly shown the difference between hard and soft ferrites. As mentioned in previous section that soft ferrite provides high value in magnetization while low in coercivity and they are shown in the above hysteresis loop. Similarly, hard ferrite is low in magnetization and high in coercivity. Thus, by combining both ferrites would lead to an exchange spring magnet with improvement in the magnetization and coercivity as in the above figure.

1.3 Problem Statement

Magnetic materials play an important roles and being used in many applications such as, loudspeaker, AC motor and others. Ferrite magnetic materials have their individual properties such as high magnetization for soft ferrite and high coercivity of hard. Up to now, NdFeB permanent magnet has recorded among the highest magnetic properties compared to other magnetic materials. Even though it has stated a good magnetic properties, few issues arises as dealing with rare earth elements. The most critical issues included the high cost and also the risk on the environment. Ferrite is known as a low cost material and environmentally friendly. The idea on the fabrication of hard/soft nanocomposite ferrite is to improve and enhance the magnetic properties.

There are many methods that can be used to fabricate nanocomposite. However, most of the techniques require few steps during the process. Therefore, the simplest and easiest method is examined and studied. Among other techniques that available, this one-pot thermal treatment method seems promising to synthesize nanocomposite nanoparticle as it promotes simplicity, low cost, and environmentally friendly (Naseri et al., 2010). This technique is cost effectiveness as the starting materials are cheap metal nitrates and the solvent used is just a water instead of other chemical solution (Hazra et al., 2014).

At first, the synthesization of this ($SrFe_{12}O_{19}$)/($Ni_{0.5}Zn_{0.5}Fe_2O_4$) nanocomposite was done due to lack of research studied on this compound by using this one-pot thermal treatment method. Previously, this technique was used only to fabricate single ferrite (Naseri et al., 2010). Currently, there were several researchers (Hazra et al., 2012; Hazra et al., 2014; Hazra et al., 2015) had used similar method, yet they were synthesized different compound of magnetic ferrites.

1.4 Significance of the Study

Previously, research on nanocomposite permanent magnet was not intensively studied by most of the researchers. This is because, rare earth permanent magnets were quite famous among the group of magnetic materials due to their better magnetic properties. However, nanocomposite permanent magnet become an attention to most of the researchers as they found that the magnetic properties of nanocomposite were comparable with the current magnets available. Most of the magnets available nowadays are metal. As research on magnet keep on growing, researchers try to synthesis ceramic magnetic materials and ferrite is chosen. Ferrite is divided into soft and hard ferrites and these two types of ferrites were used in the synthesis of exchange spring nanocomposite permanent magnet. This nanocomposite ferrite supposedly produce better magnetic properties obtained from both soft and hard ferrites respectively.

Several steps could be made in the enhancement of magnetic properties of the nanocomposite ferrites. One of the method is to tailor the microstructure of the composite. The particle size affect the magnetic properties of the composite ferrite. Variation in the particle size is usually caused by the variation of calcination temperature. Therefore, study on effect of calcination temperature can be look into and deeply studied.

For this research, nanocomposite was synthesized by only a single reaction mixture (one-pot) rather than most technique that involves double reaction mixture. In this study, nanocomposite ferrite were prepared from an aqueous solution containing metal nitrates, PVP, and deionized water using a low temperature thermal treatment method, followed by grinding and calcination process. There is no other chemicals added into the solution and therefore, this method is an environmentally friendly. In addition, it also promotes the advantages of low cost, low calcination temperature and simplicity.

1.5 Research Objectives

The objectives of current research are:

1) To study the magnetic properties of $(SrFe_{12}O_{19})/(Ni_{0.5}Zn_{0.5}Fe_2O_4)$ nanocomposite ferrite prepared by one-pot thermal treatment technique.
2) To determine the effect of PVP as a capping agent in synthesis of nanocomposite hard/soft ferrite nanoparticles.
3) To optimize the calcination temperature of nanocomposite ferrites.
4) To examine the effect of phase distribution of soft and hard ferrites on their magnetic properties and phase formation.

1.6 Outline of the Thesis

This thesis consists of six chapters. For the first chapter, Chapter 1 is a brief discussion on the background studies, nanocomposite permanent magnet, problem statement, the significant of the study and the objectives of the research. Chapter 2 is the chapter that reported on the previous researches, related to the current work. Some theories on magnetic materials are covered in Chapter 3. In Chapter 4, material fabrication, materials preparation, and characterizations are explained in details. The results obtained from XRD, FT-IR, FESEM, TEM, VSM and TGA are discussed in Chapter 5. Finally, the last chapter, Chapter 6 is conclusion where major findings and some suggestions for future research are summarized.

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