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SYNTHESIS AND CHARACTERIZATION OF BARIUM HEXAFERRITES DERIVED FROM STEEL WASTE USING SALT-MELT TECHNIQUE

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By

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

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December 2017

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This project focused on the synthesis of barium hexaferrites derived from steel waste (also known as mill scales). The steel flakes were ball milling for several hours to form fine powders. The fine powders are purified using magnetic separation technique (MST) and Curie temperature separation technique (CTST). The purified steel waste powder then oxidized at 500 ºC to form hematite, Fe_2O_3. Fe_2O_3 powder was characterized using X-ray Fluorescence (XRF), Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction (XRD) and energy-dispersive X-ray analysis (EDX). The XRD and XRF results indicate the purified powder obtained are iron oxide Fe_2O_3 (ICDD 98006-9763) and show that the obtained powders have high content of Fe_2O_3 for about 99.051%. The Fe_2O_3 were used to synthesize $BaFe_{12}O_{19}$ by using salt-melt method. The samples were synthesized using different powder to salt weight ratio of $BaCO_3$ + Fe_2O_3 and ammonium nitrate salt (NH_4NO_3). The NH_4NO_3 was melted at 170 ºC. The mixture of $BaCO_3$ and Fe_2O_3 were added into the NH_4NO_3 melt solution and kept stirring for several hours using magnetic stirrer under control temperature of 170 ºC. The heating temperature was then increased up to 260 ºC for 24 hours to produce ash powders. The XRD show the peak of $BaFe_{12}O_{19}$ for all the samples and the presence of small amount of impurity Fe_2O_3 for sample's ratio 1:5 and 1:6. Based on FTIR spectra, the bands appears at 542.71 cm^{-1} and 432.48 cm^{-1} corresponding to metal-oxygen bending and vibration of octahedral sites of $BaFe_{12}O_{19}$. The FESEM images shows that the grains of the samples appear to stick to each other and agglomerate at different masses throughout the image with the grain size 5.26, 5.88, 6.14, 6.22 and 6.18 µm for ratio 1:3, 1:4, 1:5, 1:6 and 1:7 respectively. From the VSM analysis, the magnetic properties of sample with a ratio 1:3 shows the highest value of coercivity H_c of 1317 Oe, saturation magnetization M_s of 91 emu/g and remnant M_r of 44 emu/g. The dielectric measurement shows that the $BaFe_{12}O_{19}$ samples have high dielectric loss and low ac conductivity. The complex permeability measured from VNA shows that the real part of permeability in the range of 1.0 to 1.2. The imaginary parts of permeability is in the range of 0 to 0.1. The minimum reflection loss is approximately around -3.9 dB to -4.33 dB for all samples.

The first matching frequency in average of 8.8 GHz are due domain wall resonance for all samples while natural resonance appears at second matching frequency in average of 11.0 GHz. This project is significance have potential for used in the low cost, recycle approached permanent magnet fabrication that potentially used in motor, loudspeakers and electromagnetic (EM) absorber applications.

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

SINTESIS DAN PENCIRIAN BARIUM HEKSAFERIT YANG DIPEROLEH DARIPADA BAHAN BUANGAN BESI MENGGUNAKAN TEKNIK CAIRAN GARAM

Oleh

NOR NADHIRAH BINTI CHE MUDA

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Fakulti: Institut Teknologi Maju

Projek ini memberi tumpuan kepada sintesis barium heksaferit yang diperoleh dari bahan buangan besi (atau dikenali sebagai sisik besi). Sisik besi telah dikisar menggunakan bebola pengisar selama beberapa jam untuk menghasilkan serbuk halus. Serbuk halus tersebut kemudiannya ditulenkan menggunakan teknik pemisahan magnetik (MST) dan teknik pemisahan suhu Curie (CTST). Serbuk sisa besi yang telah ditulenkan kemudiannya dioksidakan pada 500 ºC bagi menghasilkan hematit, Fe_2O_3. Serbuk Fe_2O_3 dicirikan dengan menggunakan pendarfluor sinar-X (XRF), Spektroskopi jelmaan Fourier Inframerah (FTIR), belauan sinar-X (XRD) dan analisis serakan sinar-X (EDX). Hasil XRD dan XRF menunjukkan serbuk yang telah ditulenkan adalah oksida besi Fe_2O_3 (ICDD 98006-9763) dan mempunyai kandungan Fe_2O_3 yang tinggi iaitu 99.051%. Fe_2O_3 tersebut digunakan untuk mensintesis $BaFe_{12}O_{19}$ menggunakan kaedah cairan garam. Sampel di sintesis menggunakan nisbah berat sebuk ($BaCO_3$ + Fe_2O_3) dan garam amonium nitrat (NH_4NO_3) yang berbeza. NH_4NO_3 dicairkan pada suhu 170 ºC. Campuran $BaCO_3$ dan Fe_2O_3 dimasukkan ke dalam larutan NH_4NO_3 dan di kacau selama beberapa jam dengan menggunakan pengacau magnet pada suhu 170 ºC. Suhu pemanasan kemudiannya ditingkatkan kepada 260 ºC selama 24 jam untuk menghasilkan serbuk abu. XRD menunjukkan puncak $BaFe_{12}O_{19}$ untuk semua sampel dan kehadiran sedikit bendasing Fe_2O_3 untuk nisbah sampel 1:5 dan 1:6. Berdasarkan spektrum FTIR, jalur ini muncul pada 542.71 cm^{-1} dan 432.48 cm^{-1} menunjukkan ikatan oxygen-metal dan getaran di tapak oktahedron $BaFe_{12}O_{19}$. Imej FESEM menunjukkan bahawa biji-bijian sampel kelihatan saling melekat dan bergumpal dengan saiz butiran 5.26, 5.88, 6.14, 6.22 dan 6.18 µm bagi nisbah 1: 3, 1: 4, 1: 5 , 1: 6 dan 1: 7. Daripada analisis VSM, sifat magnet dari nisbah sampel 1: 3 menunjukkan nilai daya paksa, H_c yang tertinggi iaitu 1317 Oe, ketepuan permagnetan, M_s 91 emu/g dan baki magnetisme 44 emu/g. Pengukuran dielektrik menunjukkan bahawa sampel $BaFe_{12}O_{19}$ mempunyai kehilangan dielektrik yang tinggi dan kekonduksi ac yang rendah. Kebolehtelapan kompleks yang diukur menggunakan VNA menunjukkan nilai purata

1.0 hingga 1.2 untuk kebolehtelapan sebenar. Kebolehtelapan khayalan menunjukkan nilai julat 0 hingga 0.1. Kehilangan pantulan minima adalah sekitar -3.9 dB hingga -4.33 dB untuk semua sampel. Frekuensi padanan pertama iaitu 8.8 GHz adalah disebabkan resonans dinding domain bagi semua sampel manakala resonans semula jadi muncul pada kekerapan padanan kedua iaitu pada purata 11.0 GHz. Projek ini mempunyai potensi digunakan dalam bahan kitar semula berkos rendah untuk fabrikasi magnet kekal, penyerap motor, pembesar suara dan elektromagnetik (EM).

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I certify that a Thesis Examination Committee has met on (date of viva voce) to conduct the final examination of Nor Nadhirah binti Che Muda on her thesis entitled Synthesis And Characterization Of Barium Hexaferrites Derived From Steel Waste Using Salt Melt Technique 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 (insert the name of relevant degree).

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LIST OF ABBREVIATIONS

μ	Complex permebility
μ’	Real permeability
μ”	Imaginary permeability
ANSM	Ammonium nitrate salt melt
$BaFe_{12}O_{19}$	Barium hexaferrites
CTST	Curie temperature separation technique
EDAX	Energy dispersive X-ray analysis
Fe_3O_4	Magnetite
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infra-red
G	Gauss
H_c	Coercivity
IR	Infra-red
L_s	Inductance
M_s	Saturation magnetization
MST	Magnetic separation technique
Oe	Oersted
T	Temperature
T_c	Curie temperature
VNA	Vector network analyzer
VSM	Vibrating sample magnetometer
XRF	X-ray fluorescence
α-Fe_2O_3	hematite
ε_r	Relative permittivity
$\varepsilon_{r'}$	Real part of permittivity
$\varepsilon_{r''}$	Imaginary part of permittivity
ρ_{exp}	Experiment density
ρ_{xrd}	XRD density
σ_{AC}	AC conductivity

CHAPTER 1

INTRODUCTION

1.1 Background study

High volume of industrial waste generated daily. This demands an effective and good management of waste disposal. With a limited number of landfill to handle increasing volume of wastes, there is a need to recycle wastes for other uses. Steel wastes such as mill scales were found having significant values and interest to produce ceramic ferrites (Septiani et al., 2016), cement clinker, negative electrode for alkaline storage batteries, and iron powder for powder metallurgy. Besides, mill scales are used as a raw material in granular refractory and as a material for the road construction (Maghool et al., 2017). The use of steel slags would reduce the production of natural high-quality materials and the impact of environmental from waste disposal can be avoided. Steel slags have mostly been used in surface layers of road pavements because of their high resistance to polishing. Thus, the necessary roughness characteristics of the road surface to be maintained for longer and prevent the skid resistance for road safety (Andreas et al., 2014). In Malaysia about 4.1 million tonnage of crude steel are produced (Global Steel Trade Monitor 2017). Steel play an important role as raw materials used in engineering industries, manufacturing sectors and transportation equipment. Thus, most of the steel waste generated from the industry such as metal engineering, electric and electronic engineering. Some of generated steel waste are recycled and some of them are end up at landfill.

Ceramic ferrites are basic materials of electronic industry. Ceramic ferrites is the material composed of iron oxides and metal oxides which have combined magnetic and electrical properties. Due to increasing interest, ceramic ferrites have been studied for several decades. There are different classes of magnetic materials such as soft ferrites (spinel cubic structure and garnet structures) and hard ferrites (hexagonal structure). Among these classes of magnetic materials, hexagonal ferrites have attracted much attention because of their potential applications in permanent magnet (Gieras 2002), magnetic recording media (Shimizu et al., 2016) and microwave devices (Pullar 2012b). The magnetic properties of ferrite materials are strongly depends on the distribution of metallic ions among crystallographic lattice sites which in turn is sensitive to the synthetic method used. Thus, there are variety of methods have been employed to synthesis them. The most popular method are chemical and mechanical alloying. Both method have their own advantages. Mechanical alloying is the process of mixed the oxides powder are milled together to obtain a homogeneous alloy. The advantage of mechanical alloying is the ability to produce high mass of product. Over the years, chemical method have overcome many of limitation methods of mechanical alloying and conventional ceramic method to produce ferrites such that it produces can control particle size, morphology and microscopic homogeneity since the properties of ferrites depends on the microstructure. Chemical routes is the method that the reaction initiates from a chemical solution. The powder processing of chemical method usually in a liquid/solid medium which may produce intermediate, finely divided mixed hydroxides or mixed organic salts to assist in the subsequent diffusion process. In this study, ammonium salt melt method was employed to synthesis $BaFe_{12}O_{19}$.

1.2 Ferrites

Ferrites commonly compose of magnetic oxide, which contain iron oxide as a primary component (Pullar 2012a). Ferrites generally contain pure iron and contains 0.06 percent carbon at room temperature. The first magnetic materials available were lodestone which was discovered in early 800 B.C. Lodestone is the natural state of magnetic materials and in the form of magnetite (Fe_3O_4). The materials were named Magnes because it was found in the district of Thessaly, Magnesia (Cullity and Graham 2011). The study of ferrites for its structural, electrical and magnetic properties has started in the year 1930 for practical use. Since then, there are tons research on ferrites.

Ferrites are ferromagnetic materials which has broad range of applications such as microwave absorber (Sugimoto et al., 1998), motor (Gieras 2002), electronic devices (Qinghui et al., 2012). Ferrites are hard, brittle and ceramic like materials with magnetic properties which make them very useful in electrical devices. Generally, there are two type of ferrite, which are soft ferrites and hard ferrites. Both soft and hard ferrite have different magnetic properties. Soft magnetic ferrite can easily magnetized and demagnetized. Soft ferrites material has low coercivity, high magnetic saturation, and narrow hysteresis loop. Compare to soft ferrite, hard ferrite are hardly demagnetized and magnetized. Hard ferrites have wide hysteresis loop and high coercivity (**Figure 1.1**). Hard ferrites are widely used in magnetic recording media (Shimizu et al., 2016), microwave devices (Mu et al., 2008) and permanent magnet such as motor, loudspeakers and refrigerators magnet (Campbell 1994).

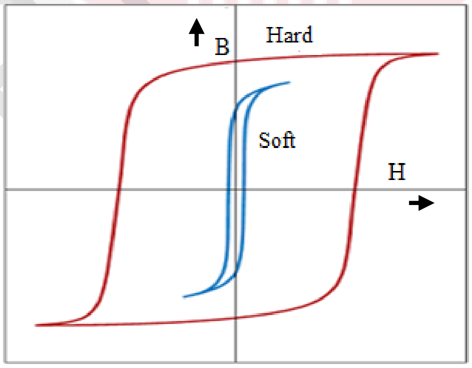

Figure 1.1: Comparison between soft and hard ferrites
(Carter and Norton 2013)

1.3 Hexagonal ferrites

Among the different classes of magnetic materials, hexagonal hard ferrites have attracted much attention because of their potential application in permanent magnets, microwave devices, motor and magnetic recording media (Pullar 2012a). All hexagonal ferrites are ferrimagnetic materials and their properties intrinsically depends on crystalline structures. Generally, the chemical formula for hexagonal ferrites can be written as $MO.Fe_2O_3$, where M is divalent ions such as Ba^{2+} and Sr^{2+}. There are different types of hexagonal ferrites such as M-type, Z-type, W-type and X-type. However in this study, M-type ferrite only considered. Mostly W, X and Z type are not interesting economically because of the difficulty of processing. Hexaferrites have a magnetoplumbite structure. The large hexagonal unit-cell contains 64 atoms which are 38 oxygen ions, 24 Fe ions and 2 divalent ions. Basically, the structure of m-type ferrite is the mixture of cubic closed packed and hexagonal packed layer formed by divalent ion and oxygen ions. The 24 Fe ions are distributed into octahedral, tetrahedral and trigonal bipyramedal sites.

1.4 Steel production and waste overview

Since 1960, the production of steel has increased almost five fold (World Steel Commitee 2016). In 2017, the steel production are expected to grow with marginal increase of 0.4%, to reach 1,494 million tonnes (Korb and Nyeberg 2016). In ASEAN, Malaysia is the fourth largest steel consuming country with a domestic steel consumption of 10 million tonnes. In Malaysia, the steel industry is divided by two categories namely Long Products and Flat Products (Lee 2015). Long products mostly used in the construction sector while the flat products are for industrial applications includes tubes, pipes, hot and cold rolled plates and sheets, coils and vessels. The production process of steel waste product from industry produce about two times more raw materials than the final product. There are huge amount of steel waste materials in form of scales, slags and dust (Martín et al., 2012). Globally, about 13.5 million tons of mill scales will be generated every year (Gaballah et al., 2013). About 500 kg/ton of solid wastes of different nature are generated in most iron and steel making processes. About 2% of mill scale produced in these solid wastes (Bagatini et al., 2011). Over the decades, the dust, slag and sludge produced by integrated steel plants was called waste. Due to intensive re-utilization of these wastes this term has been replaced with by-product (Çamci et al., 2002). The details about mill scales will be discussed in Chapter 2.

1.5 Problem statement

Hard ceramic ferrites ($BaFe_{12}O_{19}$) have gained great attention due to their potential applications as motor and electronic device applications. Mill scales are known for its iron richness have gaining interest in past years as a raw materials in preparing ferrites (Shahrani et al., 2016; Daud et al., 2016; Azis et al., 2015). The magnetic properties of ceramic derived from steel waste (mill scales) are usually low compared to commercial based $BaFe_{12}O_{19}$ (Low et al., 2015; Afghahi et al., 2017). Hameed et al., (2015) utilized quenching-melting technique to prepare hard magnetic glass ceramic from rolling mill scales wastes with magnetization of 16.665 emu/g and coercivity of 1001 G. Other literature reported the magnetic properties of the magnetization and coercivity of 43 emu/g and 860 G respectively using mechanical alloying (Azis et al., 2014) . Thus, in this study we intend to study the magnetic properties of $BaFe_{12}O_{19}$ by using chemical routes; ammonium nitrate salt melt method (Topal et al., 2006; Topal 2009). The study about microstructure, structural, magnetic, and dielectric properties are important to produce good performance of $BaFe_{12}O_{19}$ steel waste based. A research on low cost of $BaFe_{12}O_{19}$ are very significance since it can synthesis from steel waste material for electronic application. Yet, according to the previous reported on barium ferrite synthesis, none of these synthesis were reported on preparing $BaFe_{12}O_{19}$ from steel waste produced. This new approach of $BaFe_{12}O_{19}$ using chemical routes, ammonium nitrate salt melt method with an adjustment powder to salt weight ratio.

1.6 Objective

The interest of this research is to study the Barium Hexaferrites properties derived from steel waste using ammonium salt melt technique. Thus, this research embarks the following objectives:

I. To extract magnetic particle Fe_2O_3 from recycled steel waste.
II. To produce $BaFe_{12}O_{19}$ using magnetic particle from recycle steel waste by using ammonium salt melt technique.
III. To study the structural, microstructure, magnetic, and dielectric properties of $BaFe_{12}O_{19}$.

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