UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF MAGNETITE FERROFLUID FOR GENERATING CURRENT INDUCED

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PREPARATION AND CHARACTERIZATION OF MAGNETITE FERROFLUID FOR GENERATING OF INDUCED CURRENT

By

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

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PREPARATION AND CHARACTERIZATION OF MAGNETITE FERROFLUID FOR GENERATING OF INDUCED CURRENT

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November 2014

Chairman : Associate Professor Mansor Hashim, PhD
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In this research, morphology, average particle size and magnetic properties of magnetite (Fe_3O_4) particles were studied and mixed with a carrier liquid to obtain a ferrofluid. Further, an attempt to use the ferrofluid to generate induced electric current was to be carried out. Magnetite (Fe_3O_4) nanoparticles were prepared by wet milling using mechanical alloying in a hardened steel vial using a SPEX8000D mill with different milling times of 10 hours, 20 hours, 30 hours and 40 hours to obtain magnetite nanoparticles in bigger quantities compared with other method. Firstly, micron-size magnetite was milled with water using different milling times of 10 hours, 20 hours, 30 hours and 40 hours. After that, the powder was dried for a day. The material was crushed with mortar and pestle and sieved to obtain a fine powder. Next, the magnetite milled with oleic acid with different times of 10 hours, 20 hours, 30 hours and 40 hours. After that, the powder was washed with hexane mixed with ethanol. Finally the powder must be dried for a day. For the next sample, the sample was milled with water and mixed with hydrochloric acid, HCl, diluted with 100 ml water in a beaker at 70^oC. Besides, 0.1ml oleic acid as surfactant was mixed with 10 ml acetone and a co-surfactant in another beaker. This solution had to be put slowly into a beaker contains magnetite and was slowly stirred. Then, 10 ml ammonia solution was put into this beaker to give a colloidal suspension. The top layer of this suspension was centrifuged by using methanol mixed with acetone. This wet powder mass was then extracted and dried for 3 hours. The magnetic nanoparticles were analyzed by XRD, TEM, FTIR and VSM analysis. The result showed that superparamagnetic magnetite nanoparticles were obtained, suggesting that the top–layer suspension was suitable to be used as ferrofluid particles. The phase of magnetite was confirmed by X-ray diffraction (XRD) using a Philips X-ray diffractometer. The average particle size of magnetite was studied using a Transmission Electron Microscope (TEM). The magnetic properties studies were carried out by using a Vibrating Sample Magnetometer (VSM). The XRD patterns showed an improvement of crystallinity with increasing milling time. The XRD patterns also showed the all samples as magnetite nanoparticle and no impurities coming from this sample. FTIR analysis showed peaks of pure magnetite and oleic acid. Hysteresis analysis from VSM shows that when milling time increased, the saturation magnetization increased but the coercivity decreased parallel with average particle size decrease. TEM micrographs show that with increase milling time, average particle size becomes decreased. The magnetite nanoparticles from the 40 hours milling

time were mixed with silicon oil to yield a ferrofluid. This ferrofluid was used to generate induced current by passing it in a plastic tube through a magnetic field. The experiment on induced current showed that the induced current generally increased when the weight of magnetite increased.

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

PENYEDIAAN DAN PENCIRIAN DARIPADA MAGNETITE FERROFLUID UNTUK MENGHASILKAN ARUS TERARUH

Oleh

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Pengerusi : Profesor Madya Mansor Hashim, PhD
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Dalam penyelidikan ini, morfologi, purata saiz zarah dan sifat magnet pada magnetit (Fe_3O_4) dipelajari dan dicampur dengan cecair pembawa untuk mendapatkan ferrofluid. Seterusnya, satu usaha ferrofluid untuk menjana arus elektrik akan dilaksanakan. Magnetit (Fe_3O_4) nanopartikel digunakan dengan pengisaran basah dengan menggunakan pengaloian mekanikal dalam keluli vial keras menggunakan sebuah pengisar SPEX8000D dengan masa pengisaran yang berbeza iaitu 10 jam, 20 jam, 30 jam dan 40 jam untuk mendapatkan nanopartikel magnetit dalam kuantiti yang lebih besar berbanding dengan kaedah lain. Pertama, magnetit bersaiz mikron telah dikisar dengan air menggunakan masa-masa pengisaran yang berbeza iaitu 10 jam, 20 jam, 30 jam dan 40 jam. Selepas itu, serbuk basah itu dikeringkan dalam sehari. Bahan ini telah dihancurkan dengan mortar dan alu dan diayak untuk mendapatkan serbuk halus daripada bendasing. Seterusnya, serbuk magnetit dikisar bersama dengan asid oleik dengan menggunakan masa yang berlainan iaitu 10 jam, 20 jam, 30 jam dan 40 jam. Selepas itu, serbuk itu dibasuh dengan campuran heksana dan etanol. Akhirnya serbuk itu dikeringkan untuk sehari. Untuk sampel seterusnya, sampel telah dikisar dengan air dan dicampur dengan asid hidroklorik, HCl, dicairkan dengan 100 ml air dalam bikar pada suhu 70ºC. 0.1 ml asid oleik sebagai surfaktan dicampur dengan 10 ml aseton dan bersama surfaktan dalam bikar yang lain. Larutan ini diletakkan secara perlahan-lahan ke dalam bikar yang mengandungi magnetit dan dikacau perlahan-lahan. Kemudian, 10 ml larutan ammonia telah dimasukkan ke dalam bikar ini untuk memberi penggantungan koloid. Lapisan atas penggantungan ini telah diasingkan dengan menggunakan campuran metanol dan aseton. Serbuk basah ini kemudian diekstrak dan dikeringkan selama 3 jam. Nanopartikel magnetik dianalisis oleh XRD, TEM, FTIR dan analisis VSM. Hasilnya menunjukkan bahawa magnetit berzarah nano yang superparamagnetik diperolehi, menunjukkan bahawa lapisan atas bersesuaian untuk digunakan sebagai ferrofluid. Fasa magnetit telah disahkan oleh pembelauan sinar-X (XRD) dengan menggunakan Philips X-ray diffractometer. Saiz purata zarah magnetit telah dikaji menggunakan Mikroskop Elektron Transmisi (TEM). Kajian sifat magnet telah dijalankan dengan menggunakan magnetometer sampel bergetar (VSM). Corak XRD menunjukkan peningkatan penghabluran dengan meningkatkan masa pengisaran. Corak XRD juga menunjukkan semua sampel sebagai magnetit nanopartikel dan tiada bendasing yang datang dari sampel ini. Analisis FTIR menunjukkan puncak magnetit tulen dan asid oleik. Analisis Histeresis dari VSM menunjukkan bahawa apabila

pengisaran masa meningkat, pemagnetan tepu meningkat tetapi coercivity menurun selari dengan purata penurunan saiz zarah. Mikrograf TEM menunjukkan bahawa dengan peningkatan masa pengisaran, saiz purata zarah menjadi berkurangan. Nanopartikel magnetit dari masa 40 jam pengisaran telah bercampur dengan minyak silikon untuk menghasilkan ferrofluid. Ferrofluid ini telah digunakan untuk menjana arus aruhan dengan melepaskan ia dalam tiub plastik melalui medan magnet. Ujikaji ke atas arus teraruh menunjukkan arus aruhan biasanya meningkat apabila berat magnetit meningkat.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF SYMBOLS AND ABBREVIATIONS

XRD	X-ray diffraction
VSM	Vibrating Sample Magnetometer
FTIR	Fourier Transform Infrared Spectroscopy
EDX	Energy-dispersive X-ray
TEM	Transmission electron microscopy
MA	Mechanical Alloying
B	Magnetic induction/Flux magnet
HEBM	High-energy ball milling
BPR	Ball to powder weight ratio
H	Magnetic field strength
Eqn.	Equation
a.u	Arbitrary unit
2θ	2 theta degree
M	Molarity
V	Volume
Fe_3O_4	Magnetite
H_c	Coercivity
B_s	Saturation induction/saturation flux magnet
ρ_{xrd}	X-ray diffraction density
M_s	Saturation magnetization
M	Molecular weight
N	Avogadro's constant
M_r	Remanence magnetization
Λ	Wavelength
H	Hours

HCl	Hydrochloric acid
NH_4OH	Ammonia solution
Hkl	Miller indices
Nm	Nanometer
G	Gram
Φ	Magnetic flux
E	Electromotive force (emf)
Oe	Orsted
A	Lattice constant
D	d spacing
S	Electron spin moment
L	Electron orbital moment
T_C	Curie temperature
K	Magnetocrystalline anisotropy
V	Voltage
M_o	Spontaneous magnetization
D_p	Critical diameter
D_c	Critical size
SP	Superparamagnetic
SD	Single domain
MD	Multi domain
$FMNP_S$	Ferromagnetic Nanoparticles
NP_S	Nanoparticles
HRTEM	High resolution transmission electron microscopy
J	Journal

CHAPTER 1

INTRODUCTION

1.1 Background of the study

A ferrofluid is a colloidal dispersion of finely separated magnetic particles in a carrier fluid generally referred to as magnetic fluid or magnetic liquid. It is a functional liquid material which exhibits normal liquid behavior coupled with magnetic properties. In the presence of magnetic field or other fields such as centrifugal or gravitational field, the particles remain uniformly dispersed throughout the carrier liquid due to the unique properties (Chen et al., 2011). Thereby a synthesis of a "black material attracting itself" and "oxide ferroso-ferrique" from FeO and Fe_2O_3 with "HO" was described by Mandel (2011).

A ferrofluid is a stable colloidal homogenous suspension of magnetic nanoparticles which have a diameter around of 10 nm in an appropriate carrier (Maity et al., 2006). According to a report by Rheinlander (2000), nanoparticles in a magnetic fluid showed distribution of magnetic and non-magnetic parameters like particle size.

1.2 History of ferrofluid

A ferrofluid was found in early 1960. Ferrofluid technology is still new at this moment. The NASA Research Center first discovered a ferrofluid in 1960's. The initial work from Papell (1965) of the NASA Lewis Research Center on dilute magnetic dispersion in hydrocarbon was published in 1963 (Papell, 1965).

1.3 Historical overview of magnetite

The earliest magnetic material discovered by humans is magnetite. Magnetite is a naturally occurring as a magnetic ceramic (ferrite). Pieces of the mineral when brought near each other, would show attractive or repulsive force effects (Goldman, 1999). The mineral is believed to have been discovered in ancient Greece around 800 BC. The first application of magnets was found and used by the Vikings in compasses, in the ninth century, or perhaps even earlier.

The first scientific study of magnetism in magnetite known as De Magnet by William Gilbert was published in the year of 1600. After 200 years later, the new science of electromagnetism was developed through the work of physicists such as H. C. Oersted, A. M. Ampere, W. E Weber, M. Faraday, P. Curie, J. C. Maxwell and many others. During this time, researchers were starting to study material of a system which is the basic related to the basic of electromagnetic theory in general and crystal structures of the materials (Buchanan, 2004).

1.4 Magnetic nanoparticle

Soft magnetic nanoparticles are important material and used widely for a variety of technological application. Magnetite (Fe_3O_4) in the format soft magnetic nanoparticles that have been of they are major interests to many researchers because of being effectively used in ferrofluids, having magnetoresistance, exhibiting strong magnet property and generating low toxicity in biological and medical applications (Can et al, 2010).

1.5 Significant of study

Magnetite is important for producing ferrofluids which have particle sizes between 10-65 nm. There are many applications in of ferrofluids such as liquid seals in computer hardisks, friction reduction, magnetic domain observation and numerous optical and medical applications and for heat transfer in loudspeakers. In this research, ferrofluid is used in experimental attempts to produce electricity. The importance of using a ferrofluid is that it has the ability to be magnetized and demagnetized rapidly when entering and leaving a magnetic field region, thus yielding a significant magnetic flux change for electric induction.

1.6 Problem statement

This work attempts to produce a ferrofluid containing non-agglomerated magnetite nanoparticles. The nanoparticles are to be prepared in quantities more readily obtained and much greater than those produced by chemical, biological and biochemical methods. Thus high energy ball milling is the chosen method. The significant amount of nanoparticles in the ferrofluids is needed to show a proof of concept that such a ferrofluid can be used to generate electric current by induction.

1.7 Objective

In this thesis, the main objective is to prepare a magnetite-based ferrofluid containing magnetite nanoparticles by using mechanical alloying followed by a simple carrier liquid and nanoparticles mixing. Further, it is to be demonstrated that such as ferrofluid can generate electric currents. The work-step objectives of this research work are:

a) To prepare mono-dispersed, uniform and size controllable magnetite using mechanical alloying method.
b) To study in detail the effect of surfactant on magnetite particle size.
c) To investigate magnetic properties of magnetite nanoparticle.
d) To demonstrate how superparamagnetic particles can produce induced current.

1.8 Outline of the thesis

The thesis attempts to provide a good understanding of the structural, morphology and magnetic properties of nanostructured materials for the applications described above. Chapter 1 will focus on the general introduction about the research background, scope, problem statement and objectives of the study. Chapter 2 concerns the background and synthesis of magnetic nanoparticles and ferrofluids. Preparation of nanomaterials with other methods and related literature in view of preparation techniques together with characterization of magnetic nanoparticles were discussed as well. Chapter 3 focuses on the theoretical background which includesbrief introduction to magnetism and the underlying chemistry of metals and alloys. Chapter 4 highlights the methodology of the study including materials, sample preparation and characterization methods applied. Chapter 5 deals with the results and discussion of measurement data of as-prepared samples and those after oleic acid coating. Chapter 6 summarizes the results and gives some suggestions for future work.

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