UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF MAGNETITE FERROFLUID FOR GENERATING OF INDUCED CURRENT

CHE SULAIMAN BIN AHMAD

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PREPARATION AND CHARACTERIZATION OF MAGNETITE FERROFLUID FOR GENERATING OF INDUCED CURRENT

By

CHE SULAIMAN BIN AHMAD

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

November 2014
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In this research, morphology, average particle size and magnetic properties of magnetite (Fe$_3$O$_4$) 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$_3$O$_4$) 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°C. 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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November 2014

Pengerusi : Profesor Madya Mansor Hashim, PhD
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I certify that a Thesis Examination Committee has met on 7 November 2014 to conduct the final examination of Che Sulaiman bin Ahmad on his thesis entitled "Preparation and Characterization of Magnetite Ferrofluid for Generating Induced Current" 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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<td>X-ray diffraction</td>
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<td>VSM</td>
<td>Vibrating Sample Magnetometer</td>
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<td>FTIR</td>
<td>Fourier Transform Infrared Spectroscopy</td>
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<td>EDX</td>
<td>Energy-dispersive X-ray</td>
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<td>TEM</td>
<td>Transmission electron microscopy</td>
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<td>MA</td>
<td>Mechanical Alloying</td>
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<td>B</td>
<td>Magnetic induction/Flux magnet</td>
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<td>HEBM</td>
<td>High-energy ball milling</td>
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<td>BPR</td>
<td>Ball to powder weight ratio</td>
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<td>H</td>
<td>Magnetic field strength</td>
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<td>Equation</td>
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<tr>
<td>a.u.</td>
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<td>2θ</td>
<td>2 theta degree</td>
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<td>M</td>
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<tr>
<td>V</td>
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HCl  Hydrochloric acid
NH₄OH  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
Tc  Curie temperature
K  Magnetocrystalline anisotropy
V  Voltage
Mo  Spontaneous magnetization
Dp  Critical diameter
c  Critical size
SP  Superparamagnetic
SD  Single domain
MD  Multi domain
FMNPₘ  Ferromagnetic Nanoparticles
NP  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 ferrosiferrique” from FeO and Fe$_2$O$_3$ 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 (Fe₃O₄) 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 a 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 includes brief 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.
REFERENCES


