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PREPARATION AND CHARACTERIZATION OF ZIRCONIUM BASED MAGNETIC PARTICLES

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PREPARATION AND CHARACTERIZATION OF ZIRCONIUM BASED MAGNETIC PARTICLES

YOGETASRI A/P JEYAPALAN

This project is submitted in partial fulfillment of
the requirements for the degree of Bachelor of Engineering with Honours
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To my beloved parents and dearest friends

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ABSTRAK

Dalam kajian ini, partikel-partikel magnetik berasaskan zirkonium telah dihasilkan melalui kaedah sepemendakan. Hubungan antara parameter boleh kawal (tempoh sebelum mengacau, suhu mengacau, dan tempoh selepas mengacau) dan permukaan gerak balas (cas titik sifar) yang diperolehi untuk penghasilan partikel-partikel magnetik dimodel melalui kaedah permukaan gerak balas menggunakan rekabentuk Box-Behnken. Model yang dicadangkan adalah:

$$y = 7.35295 - 0.00130X_1 - 0.02992X_2 - 0.01023X_3 - 0.00006X_1X_2 + 0.00020X_1X_3 + 0.00022X_2X_3 + 0.00002X_1^2 + 0.00025X_2^2 - 0.00009X_3^2$$

di mana y ialah pH_{ZPC}, X_1 ialah tempoh sebelum mengacau, X_2 ialah suhu mengacau and X_3 ialah tempoh selepas mengacau. Partikel-partikel magnetik dianalisa melalui kaedah pentitratan, spektroskopi Fourier ubah inframerah (FTIR), dan diperhatikan di bawah mikroskop. Kaedah pentitratan menentukan cas titik sifar (pH_{ZPC}) pada partikel-partikel magnetik. FTIR mendedahkan ikatan kimia dalam partikel dan pemerhatian di bawah mikroskop menunjukkan morfologi permukaan partikel-partikel magnetik.

ABSTRACT

In this study, zirconium based magnetic particles have been fabricated by co-precipitation method. Relationship between controllable input parameters (pre-stirring duration, stirring temperature, and post-stirring duration) and obtained response surfaces (zero point charge, pH_{ZPC}) for the fabrication of the magnetic particles is modelled through response surface methodology by using Box-Behnken design. The proposed model is:

$$y = 7.35295 - 0.00130X_1 - 0.02992X_2 - 0.01023X_3 - 0.00006X_1X_2 + 0.00020X_1X_3 + 0.00022X_2X_3 + 0.00002X_1^2 + 0.00025X_2^2 - 0.00009X_3^2$$

where y is the pH_{ZPC}, X_1 is the pre-stirring duration, X_2 is the stirring temperature and X_3 is the post-stirring duration. The magnetic particles are analysed by potentiometric titration, Fourier Transform Infrared spectroscopy (FTIR) and observed under microscope. Potentiometric titration determines the point of zero charge (pH_{ZPC}) of the magnetic particles. FTIR reveals the chemical bonding of the particles while microscope observation shows the surface morphology of the magnetic particles.

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NOMENCLATURE

$FeCl_2.4H_2O$	-	Ferum (II) chloride tetrahydrate
$FeCl_3$	-	Ferric chloride
FTIR	-	Fourier Transform Infrared
HCl	-	Hydrochloric acid
KBr	-	Kalium bromide
NaCl	-	Sodium chloride
NaOH	-	Sodium hydroxide
pH_{ZPC}	-	pH at zero point charge
σ_0	-	Surface charge density
$Zr(SO_4)_2$	-	Zirconium sulphate

CHAPTER 1

INTRODUCTION

1.1 Background

Magnetic particles have been developed comprehensively in diagnostics and other research applications for the purification of cells and bio-molecules, such as antibodies, nucleic acids, and polypeptides. They provide various benefits, including ease of separation and suitability for automation. Coated with molecules, these magnetic microspheres are perfect for the practical capture and separation of target. Unwanted sample constituents may be washed away with a simple magnetic separation step. Magnetic microparticles allow us to deal with countless applications in the life sciences, such as, cell separations, immunoassays, and suspension arrays.

1.2 Problem statements

The zirconium-based magnetic particles obtainable in the present are not optimized in their physical and chemical properties yet. These existing properties of the particles have caused problems to control quantitatively parameters of the elements detected to obtain accurate standard magnetization field strength and its direction according to specified temperature and pressure. These problems are essential technical problem which may result in harm and serious consequences of these problems have to be analysed and further improved.

1.3 Scope and objectives

The aim of this research is to study on zirconium based magnetic particles and analyse them accordingly. The specific aims are:

i. To fabricate the zirconium based magnetic particles using a simple method;

ii. To model the relationship between the controllable input parameters (pre-stirring duration, stirring temperature, and post-stirring duration) and the obtained response surfaces (pH_{ZPC}) for the fabrication of the zirconium based magnetic particles;

iii. To characterize the zirconium based magnetic particles.

Appropriate procedures are implemented in the experiments to obtain good results which can be used for further analysis.

1.4 Significance of study

Zirconium based magnetic particles represent a new group of materials with promising mechanical, electronic and chemical properties. Since the discovery of zirconium in 1789, considerable efforts have been made to study the application of this new material. The unique mechanical properties, chemical stability and the properties of zirconium compounds have been intensively researched for various applications. Many applications based on zirconium particles have been developed in the medical and biotechnology field.

While zirconium have proven to be quite successful in their designed usage, a desire for longer lifetimes of components and increased duty cycle puts more demand on materials performance. This demand has led to more in-depth studies of characterization of zirconium based magnetic particles to optimization of the composition and to new data of mechanical properties. Phenomena occurring on a microscopic scale are given emphasis for a better understanding of the particles' behaviour.

1.5 Expected outcome

In the end of this study, it is expected that the optimized physical parameters of synthesizing zirconium-based magnetic particles are found and it can be implemented in other applications mainly in medical, biotechnology, and water and waste water industries. Besides that, the properties of the particles determined can be applied in new researches which could be carried out without any physical harm.

1.6 Summary of Contents

This section briefly reveals each of the chapters in a summarized form which helps readers to have a clearer picture and better understanding of the overall project. Below are the brief descriptions of each chapter:

i. Chapter 1: Introduction

   This chapter provides readers the overall review of the entire project. It highlights the introduction parts such as background of the study, problem statements, scope and objectives, significance of study, and expected outcome.

ii. Chapter 2: Literature Review

   This chapter reviews the previous researches on the topics of interest. It includes the background of the topic, preparation and characterization of the magnetic particles. Similar analytical methods used in previous studies were also highlighted in this chapter. The implementations of magnetic particles were also discussed here which includes medical, biotechnology, inspection, military, and data storage applications.

iii. Chapter 3: Methodology

Experiment methods and how zirconium-based magnetic particles synthesized are discussed in this chapter. It presents the designs of experiments in conducting runs which is a crucial aspect to study the optimized method of preparing the particles. The analytical methods and steps involved in conducting this research are also included in this chapter.

iv. Chapter 4: Results and discussions

This chapter reviews topics on the outcome of the study and shown with contour plot and graph for a clearer view of the readers. The graphical method allows readers to observe the effects of each independent parameter in obtaining zirconium-based magnetic particles.

v. Chapter 5: Conclusion

This chapter summarizes the study with the result obtained through the experiments and analysis. Further improvements and recommendations are also explained here.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter presents about similar studies conducted prior to this study. It includes the background of the topic, element of the magnetic particle, the natures of magnetism, types of magnetic particles exist, and preparation and characterization of the magnetic particles. The implementations of magnetic particles were discussed including medical, biotechnology, inspection, military, and data storage applications. Determination of surface charge density through potentiometric titration and point of zero charge attained in previous studies were also highlighted in this chapter.

2.2 Zirconium

Zirconium is the fortieth element listed in the periodic table and has the chemical symbol Zr. According to Watt (2008), all zirconium atoms have 40 protons in the nucleus. However, the number of neutrons in each atom varies. Some zirconium atoms have 56 neutrons in the nucleus while other might have only 50. Zirconium has five different isotopes and the most common is Zr-90. The least common zirconium isotope is Zr-96 which is radioactive. The density of zirconium is much like the density of iron. Craig and Anderson (1995) have stated that zirconium is very resistant to corrosive attack in most mineral and organic acids, strong alkalis and saline solutions. Zirconium is never found in nature as a pure metal because it bonds with other elements to form a compound.

2.3 Magnetism

Magnetism is a force between electric currents whereby two parallel currents in the same direction attract and two parallel currents in opposite directions repel. According to Maxwell (2005), magnetism represents aspects of the force of electromagnetism, which is one part of the fundamental electroweak force. Magnetic field is the region in space that is penetrated by the imaginary lines of magnetic force. The strength of the magnetic field is determined by the number of lines of force per unit area of space. Magnetic fields are created on a large scale either by the passage of an electric current through magnetic metals or by magnetized materials known as magnets. The elemental metals or alloys with related metallic elements are materials that respond strongly to magnetic fields. The magnetic force field within a magnetized body is polarized.

2.4 Types of magnetism

There are five basic types of magnetism that have been observed and classified on the basis of the magnetic behaviour of materials in response to magnetic fields at different temperatures by de Lacheisserie et al. (2005). The types of magnetism are ferromagnetism, ferrimagnetism, antiferromagnetism, paramagnetism, and diamagnetism.

According to Aharoni (1996), ferromagnetism occurs when the particles have a permanent mean magnetic moment. The larger effective magnetic anisotropy holds back the thermally activated motion of the core-moments. In a ferromagnet, magnetic moments of equal magnitude arrange themselves in parallel to each other. Cullity and Graham (2009) have mentioned about the advantages of ferromagnetic particles which have very strong magnetic properties and therefore the fast separation with an external magnetic field even in viscous media.

Ferrimagnetism is a specific type of ordering in a system of magnetic moments or the magnetic behaviour resulting from such order. Kaneyoshi (1991) have stated that a material is said to exhibit ferrimagnetic order when all moments on a given sublattice point in a single direction and the resultant moments of the sublattices lie parallel or antiparallel to one another. For a normal ferromagnet, there is a net moment or an algebraic sum of the sublattice moments. Ferrimagnets may also exhibit an angular momentum compensation point at which the angular momentum of the magnetic sublattices is compensated. Couture and Zitoun (2000) have mentioned that ferrimagnetic materials have high resistivity and have anisotropic properties. Ferrimagnets can be measured statically or at low frequencies to resemble ferromagnets with unusual temperature characteristics.

The magnetic moments of atoms or molecules in materials that exhibit antiferromagnetism are usually related to a regular pattern with neighbouring spins pointing in opposite directions on different sublattices. Generally, antiferromagnetic order may exist at sufficiently low temperatures. Antiferromagnetic materials occur less frequently in nature than ferromagnetic materials. According to Radu and Zabel (2007), antiferromagnets can couple to ferromagnets through a mechanism known as exchange bias, in which the ferromagnetic film is either grown upon the antiferromagnet or annealed in an aligning magnetic field, causing the surface atoms of the ferromagnet to align with the surface atoms of the antiferromagnet. Hopster and Oepen (2005) have found out that antiferromagnetism plays a vital responsibility in giant magnetoresistance.