
**UNIVERSITI PUTRA MALAYSIA**

# *MILL SCALE DERIVED IRON OXIDE NANOPARTICLES AND SURFACE MODIFICATION FOR WASTEWATER TREATMENT*

**AZDIYA SUHADA BT ABDUL RAHIM @ ARIFIN**

**ITMA 2018 12**

# MILL SCALE DERIVED IRON OXIDE NANOPARTICLES AND SURFACE MODIFICATION FOR WASTEWATER TREATMENT

By

**AZDIYA SUHADA BT ABDUL RAHIM @ ARIFIN**

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

**January 2018**

**DEDICATIONS**

Alhamdulillah.

Every challenge need self-efforts as well as guidance of elders especially those who very close to our heart. My humble effort, I dedicate to my sweet and loving

## Mok, Hubby and Hud

Whose affection, love, encouragement and pray of days and nights make be able to get such success and honor.

Along with all supports and respected, my siblings

**Azmi, Azmarina, Azli, Azmajura, Azmalisya, Azriehisyam, Azdiya Suhana, Najahtul Akma, Syafeq and Azreen.**

Lastly,

**Ayoh,**

**I miss you. A lot.**

اَللّٰهُمَّ اغْفِرْلَهُوَرْحَمْهُ وَعَافِهِوَاعْفُ عَنْهُ

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

**MILL SCALE DERIVED IRON OXIDE NANOPARTICLES AND SURFACE MODIFICATION FOR WASTEWATER TREATMENT**

By

**AZDIYA SUHADA BT ABDUL RAHIM @ ARIFIN**

**January 2018**

**Chairman: Ismayadi Ismail, PhD**
**Institute: Institute of Advanced Technology**

A balance ecosystem is very important to sustain livelihoods, human well-being, population of species and environment. Pollutions such as land pollution and water pollution imbalanced and disturbed the ecosystem, affected the living things in different ways. Tremendous accumulation of mill scale waste consisting of iron oxides, metal alloys and impurities such as dust and sand contributing to the land pollution whereas discharging untreated wastewater into water bodies contributing to the water pollution endanger the environmental ecosystem. To make use of the mill scale waste, iron oxide was extracted by using magnetic separation technique (MST) and Curie temperature separation technique (CTST) and recycled in wastewater treatment. However, extremely small sizes of iron oxide nanoparticles tend to agglomerate and limited the performance of iron oxide nanoparticles in wastewater treatment. Hence the surface modification of iron oxide nanoparticles with surfactant, Cetyltrimethylammonium bromide (CTAB) was needed to overcome the agglomeration problem. The extracted iron oxide was milled for 4, 6, 9 and 12 hours using High Energy Ball Milling (HEBM) machine to obtain different average particle sizes of the iron oxide nanoparticles. The morphological, physical and magnetic properties of the extracted iron oxide nanoparticles from mill scale waste before and after surface modification were studied. The performance of extracted iron oxide nanoparticles in wastewater treatment was also studied by analyzing the colour, Total Solids (TS), Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) of wastewater before and after treatment. Highly crystalline iron oxide extracted from the mill scale waste consisted of hematite, magnetite and wuestite phase which undergo phase transformation and decreasing average particle size with prolong milling time. The iron oxide nanoparticles (IONp) were agglomerated and their surface area per volume of the iron oxide nanoparticle were low. The surface modification of iron oxide nanoparticles with CTAB (IONpCTAB) were successful as HRTEM image show average coating layer of 2.7 nm on the surface of iron oxide

nanoparticles. The surface modification overcome the agglomeration problem and increased the surface area per volume of the iron oxide nanoparticles but decreased the magnetic properties of iron oxide nanoparticles. The colour, total solid (TS), total dissolved solid (TDS) and total suspended solid (TSS) were greatly removed from the untreated wastewater after treated using both iron oxide nanoparticle without and with surface modification. The presence of CTAB helps in decolorized the colour of wastewater from dark brown to light yellow colour. CTAB also increased the BOD and COD values of wastewater after treated with iron oxide nanoparticles modified with CTAB. The surface area per mass of iron oxide plays important role in wastewater treatment as the performance of iron oxide nanoparticles increased with increasing surface area per mass of iron oxide nanoparticles resulted by reduction of average particle sizes of iron oxide nanoparticles.

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

**SISA SERPIHAN BESI MENGHASILKAN BESI OKSIDA DAN MODIFIKASI PERMUKAANNYA UNTUK RAWATAN SISA AIR**

Oleh

**AZDIYA SUHADA BT ABDUL RAHIM @ ARIFIN**

**Januari 2018**

**Pengerusi: Ismayadi Ismail, PhD**
**Institut: Institut Teknologi Maju**

Ekosistem yang seimbang sangat penting untuk mengekalkan kehidupan, kesejahteraan manusia, populasi spesies dan alam sekitar. Pencemaran seperti pencemaran tanah dan pencemaran air membuatkan ekosistem yang tidak seimbang dan terganggu, di mana ia mempengaruhi hidupan dengan berbagai cara. Pengumpulan sisa serpihan besi yang mengandungi besi oksida, aloi logam dan bendasing seperti habuk dan pasir menyebabkan pencemaran tanah manakala pengeluaran sisa air tanpa rawat ke badan air menyebabkan pencemaran air membahayakan ekosistem alam sekitar. Oleh itu, dengan menggunakan sisa serpihan besi, besi oksida diperolehi dengan menggunakan teknik pengasingan magnet (MST) dan teknik pengasingan suhu Curie (CTST), lalu di kitar semula dalam rawatan sisa air. Walaubagaimanapun, terlalu kecil saiz besi oksida membuatkan ia bergumpal dan menyekat prestasi besi oksida bersaiz nano dalam rawatan sisa air. Oleh itu, modifikasi permukaan besi oksida bersaiz nano dengan Cetyltrimethylammonium bromide (CTAB) diperlukan untuk mengatasi masalah pergumpalan tersebut. Besi oksida yang diperolehi dari sisa serpihan besi telah dikisar selama 4, 6, 9 dan 12 jam menggunakan pengisar bebola berkuasa tinggi (HEBM) untuk memperolehi purata saiz zarah yang berbeza. Sifat – sifat morfologikal, fizikal dan magnet besi oksida bersaiz nano dihasilkan daripada sisa serpihan besi sebelum dan selepas modifikasi permukaannya dikaji. Prestasi besi oksida bersaiz nano dalam rawatan sisa air juga dikaji dengan mengkaji warna, jumlah pepejal (TS), jumlah pepejal terlarut (TDS), jumlah pepejal terampai (TSS), permintaan biokimia oksigen (BOD) dan permintaan kimia oksigen (COD) sisa air sebelum dan selepas rawatan. Besi oksida yang diperoleh dari sisa serpihan besi mengandungi fasa hematit, magnetit, wustit mengalami perubahan fasa dan purata saiz zarah berkurang selagi tempoh pengisaran bertambah. Besi oksida (IONp) bergumpal dan luas permukaan zarah besi oksida bersaiz nano rendah. Modifikasi permukaan besi oksida bersaiz nano dengan CTAB (IONpCTAB) telah menangani masalah penggumpalan dengan adanya lapisan bersalut pada permukaan besi oksida setebal 2.7 nm yang diperoleh dari imej HRTEM dan menambahkan luas permukaan zarah besi oksida bersaiz nano, tetapi mengurangkan sifat magnet besi oksida bersaiz nano. Kedua – dua IONp dan

IONpCTAB digunakan untuk merawat sisa air buangan. Banyak jumlah pepejal, jumlah pepejal terampai dan pepejal terlarut berjaya dibuang daripada sisa air buangan selepas dirawat dengan besi oksida bersaiz nano dan juga selepas dirawat dengan besi oksida bersaiz nano yang telah dimodifikasi permukaannya. Kehadiran CTAB membantu menurunkan warna sisa air daripada coklat gelap kepada kuning cerah. CTAB juga meningkatkan nilai BOD dan COD dalam rawatan sisa air buangan selepas dirawat menggunakan besi oksida bersaiz nano yang telah dimodifikasi dengan CTAB. Luas permukaan besi oksida bersaiz nano memainkan peranan penting dalam rawatan sisa air buangan kerana prestasi besi oksida bersaiz nano meningkat dengan peningkatan luas permukaan besi oksida bersaiz nano disebabkan oleh pengecilan purata saiz zarah.

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I certify that a Thesis Examination Committee has met on 30 January 2018 to conduct the final examination of Azdiya Suhada bt Abdul Rahim @ Arifin on her thesis entitled "Mill Scale Derived Iron Oxide Nanoparticles and Surface Modification for Wastewater Treatment" 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.

Members of the Thesis Examination Committee were as follows:

**Halimah binti Mohamed Kamari, PhD**
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

**Rabaah Syahidah binti Azis, PhD**
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

**Norinsan Kamil Othman, PhD**
Associate Professor
Universiti Kebangsaan Malaysia
Malaysia
(External Examiner)

**NOR AINI AB. SHUKOR, PhD**
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 26 April 2018

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:

**Ismayadi Ismail, PhD**
Research Officer
Institute of Advanced Technology
Universiti Putra Malaysia
(Chairman)

**Abdul Halim Abdullah, PhD**
Associate Professor
Institute of Advanced Technology
Universiti Putra Malaysia
(Member)

**ROBIAH BINTI YUNUS, PhD**
Professor and Dean
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**Declaration by graduate student**

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| | |
|---|---|
| ADMI | American Dye Manufacturer's Institute |
| APTES | 3-amino propyltriethoxysilane |
| BET | Brunauer–Emmett–Teller |
| BOD | Biochemical oxygen demand |
| BPR | Ball-to-powder weight ratio |
| COD | Chemical oxygen demand |
| CTAB | Cetyltrimethylammonium bromide |
| CTST | Curie Temperature Separation Technique |
| EDAX | Energy Dispersive X-ray |
| FESEM | Field Emission Scanning Electron Microscope |
| FTIR | Fourier Transform Infra-red |
| Hc | Coercivity |
| HEBM | High Energy Ball milling |
| HRTEM | High Resolution Transmission Electron Microscope |
| IONp | Iron oxide nanoparticle |
| IONpCTAB | Iron oxide nanoparticles modified with CTAB |
| $M_S$ | Saturation magnetization |
| MST | Magnetic separation technique |
| nm | Nanometer |
| PC | Phosphatidylcholine |
| POME | Palm Mill Oil Effluent |
| TDS | Total Dissolved solids |
| TS | Total solids |
| TSS | Total suspended solids |
| VSM | Vibrating Sample Magnetometer |
| XRD | X-ray Diffraction |
| μm | Micrometer |

## GLOSSARY

| | |
|---|---|
| Turbidity | Cloudiness or haziness of a fluid caused by large invisible particles in water that cannot be seen by naked eyes. |
| Hydrophobic | Molecules that do not mix with water. |
| Hydrophilic | Molecules that mix well in water. |
| Effluents | Liquid waste or sewage discharged into water bodies. |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Steel production in Malaysia has achieved about 6.77 million metric tonnes and estimated to be increased due to increase demand in steel consumption which is about 10.34 million tonnes in 2016 (Malaysian Iron and Steel Industry Federation, 2017). In Sweden, over two million tonnes of industrial waste produced from iron and steel production. 38 % of iron and steel industrial waste were sold as various products, 42 % were recirculated internally and 20 % becomes landfill waste (Eva, 2017). The intensive of the steel productions grabbed attention of many parties related to the by-product resulted from the making process. One of the by-product is mill scale waste. Tremendous collected amount of mill scale waste become very serious pollution problems. Therefore, many trials were taken to turn these iron and steel industrial waste into valuable product. As in Malaysia, there were only few attempts in recycling mill scale waste in other application, especially from Universiti Putra Malaysia. Azis (2013) has tried to purify wustite from mill scale waste and then converted into hematite to produce barium / strontium hexagonal ferrite. Wustite obtained from mill scale waste converted into pure hematite could be used in other application. Also, yttrium iron garnet (YIG) synthesized by extracting hematite from mill scale waste has beed studied by Mohd. Sharani (2016). YIG possessed wide magnetization range and low dielectric loss that very applicable in microwave application. As from other country, Young and David (2004) tried to use mill scale waste as cement plants. However, the mill scale waste segregates from other feedstock materials during blending process due to the high density of mill scale waste. Meanwhile, hydrocarbon contains in mill scale waste were volatized and released to air as air pollution during sintering process. Hence, mill scale waste were claimed as not preferable as cement plants. Meynerts et. al (2013) reported that mill scale waste with addition of cement, hydrated lime and molasses or silicates produced briquette. However, briquette manufactured were not satisfied the strength of briquette required by the company. The thickness of these mill scale waste depends on the oxidation conditions, steel composition and surface finish (de Oliveira Lima et. al., 2010).

Usually, mill scale waste that formed on carbon steel at high temperature contain three main phase of iron oxide which are wustite (FeO), magnetite ($Fe_3O_4$) and hematite ($Fe_2O_3$). Iron oxide has been extensively used in many applications. For example, iron oxide were used in recording devices due to high coercivity properties of iron oxide. Besides, because of iron oxide hardness property is very good, iron oxide such as hematite and magnetite were also used as abrasive and polishing agent. Apart from that, iron oxide were used as ferrofluids. Ferrofluids contain of aqueous media or organic liquid and magnetic iron oxide nanoparticles with a very high degree of colloidal stability in magnetic field. Most ferrofluids were applied in many instruments such as Nuclear Magnetic Resonance spectroscopy (NMR).

Despite the unique properties of iron oxide nanoparticle, iron oxide nanoparticle suffers a major problem of agglomeration. The agglomeration of iron oxide nanoparticle limited and restricted the application and method preparation of the iron oxide nanoparticle. The generalizability of much published research on this issue is problematic. Hence, surface modification of iron oxide with suitable amine and carboxylic group has been introduced to overcome this problem. Xu et. al. (2012) stated that iron oxide nanoparticle was able to react with different functional group such as stabilizer, surfactant and steric polymer which has great potential in surface modification. Questions have been raised about the properties of the IONp after the surface modification. Thus, the main aim of this research is to investigate the properties of iron oxide nanoparticle before and after the surface modification. Furthermore, in past few years, several attempts have been made to use IONp in wastewater treatment and it was suggested as efficient, low cost, and environmental friendly adsorbent in wastewater treatment (Friedrich et al., 1998; Dimitrov, 2006; Dastjerdi and Montazer, 2010) due to the superior small nano sized particles, high surface area per volume ratio and surface modification exhibits high adsorption capability (McHenry and Laughlin, 2000; Afkhami et al., 2010; Pan et al., 2010). Extremely rapid population growth, industrialization and extensive agricultural has made water pollution problem become worst. These development activities somehow increases the amount of contaminant in the wastewater and then discharged into water bodies such as rivers, lakes and oceans. Example of the contaminants are inorganic compound, organic pollutants, heavy metals and other complex compounds (O'Connor, 1996; Fatta et al., 2011; Li et al., 2011). Hence, great varieties of wastewater treatment technologies were employed to remove contaminant in wastewater including adsorption processing. Therefore, this research set out to assess the potential of IONp and IONp modified with surfactant in the wastewater treatment as a preliminary study.

### 1.1.1 Mill Scale Waste

Mill scale waste (Figure 1.1) is one of the iron and steel industrial waste that formed as an oxidized layer during descaling hot metal process in a furnace at very high temperature. During hot metal process, the metals were converted into desired shapes such as bars and sheets continuously in hot rolling mills. To remove the mill scale waste from the metals, water spray technique was employed. Usually, mill scale waste formed on carbon steel at high temperature consisted of magnetic particles, nonmagnetic particles and other impurities. Most magnetic particle contained in mill scale waste was iron oxide particle. Three type of iron oxide such as wuestite, magnetite and hematite were mostly found in the mill scale waste.

**Figure 1.1 : Accumulation of mill scale waste**
(http://www.europeanrecycle.com/it/millscale/)

### 1.1.2 Iron Oxide

Iron oxide contains iron (Fe) and oxygen (O) or hydroxide (-OH). Most common iron oxide phase are wuestite, magnetite and hematite. Wuestite is black iron oxide contains only divalent Fe with cubic closed packed anion structure. Magnetite is also black, opaque mineral that has inverse spinel structure contain Fe (II) and Fe (III). It is also known as black iron oxide, magnetic iron ore, iron $^{II,III}$ oxide and etc. Hematite is a type of iron oxide that can be found in rocks and soils. It is blood-red colour in fine particles but black and sparkling grey in large particles with hexagonal closed packed anion structure. The other common names for hematite are iron III oxide and ferric oxide (Cornell, R.M., Schwertmann, U, 2003).

### 1.1.3 Wastewater

Wastewater is used water that carried waste from numerous sources. Wastewater can be categorized into two types according to source of the waste. Domestic or sanitary wastewater is wastewater that comes from residential source such as toilet, sinks and laundry usually contain pathogenic that dangerous to human intestinal tract. Meanwhile, wastewater that comes from manufacturing and processes and commercial enterprises waste are known as industrial wastewater. Department for Environment Food and Rural Affairs (2012) stated that in the range of 624 200 kilometres, about 11 billion per day of wastewater managed to collect from homes, commercial and industrial premises, municipal and drainage. Untreated wastewater discharged into water bodies such as rivers, lakes, and oceans accumulates pathogenic microorganism, excess of nutrients and other chemical compounds threatened aquatic life and human health.

## 1.2 Problem Statement

Large amount of collected mill scale waste has become great potential of land pollution. Iron oxide contained in the mill scale waste could be extracted and can be recycled in other applications. However, extremely small sizes of iron oxide nanoparticles tend to agglomerate makes the application and method of preparation of the iron oxide become limited and restricted. Previous researchers stated that surface modification of iron oxide nanoparticles was needed to overcome the agglomeration problem. Somehow, only a few researchers reported about the properties of iron oxide nanoparticles before and after surface modification. Apart from that, production of wastewater has increased due to extensive growth development of Malaysia. Untreated wastewater has discharged into water bodies such as rivers, oceans and lakes cause water pollution that might threatened human health and environment.

## 1.3 Objectives

The main objective of this research is to study the properties of iron oxide nanoparticle (IONp) extracted from mill scale waste and the properties of the iron oxide nanoparticle after surface modification (IONpCTAB). Also, as a preliminary study, the other objective of this research is to study the potential of the extracted IONp and IONp modified with surfactant in wastewater treatment. Below are the work phase to achieve the objective of this research:

a) To extract iron oxide from mill scale waste using Magnetic Separation Technique (MST) and Curie Temperature Separation Technique (CTST) and convert the extracted iron oxide into different average particle size of iron oxide nanoparticle via mechanical alloying by using different milling time of 4, 6, 9 and 12 hours.
b) To study the effect of average particle size of extracted IONp and effect of surface modification of IONp on the morphological, physical and magnetic properties.
c) To evaluate the potential of the extracted IONp and IONp modified surfactant in wastewater treatment.

## 1.4 Limitation Study

The reader should bear in mind that the study is focused on the properties of the IONp extracted from mill scale and the properties of the extracted IONp after surface modification. Though this research includes the potential study of the extracted IONp in wastewater treatment, a full discussion about the application of IONp in the wastewater treatment lies beyond the scope of this study. This research does not engage with the optimization of the performance of the IONp in the wastewater treatment.

## 1.5 Thesis Outline

The overall structure of the study takes the form of six chapters, including this introduction chapter. Chapter 2 begins by finding of the research, focusing on the four key themes which is the preparation method of iron oxide nanoparticle, mechanical milling, surface modification and the application iron oxide in wastewater treatment. Chapter 3 starts by laying out the theoretical dimensions of the research such as mechanism of mechanical milling, crystal structure of iron oxide, fundamental of magnetism, magnetic properties of materials and iron oxide, surface modification and IONp in wastewater treatment. Chapter 4 is concerned with the methodology used in this research. Chapter 5 analyses and discusses the finding results and Chapter 6 gives a brief summary that concludes the finding and some suggested recommendations. At the end of the thesis, the list of publications, the author's biography and the bibliography is added.

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