UNIVERSITI TEKNIKAL MALAYSIA MELAKA

FORMATION OF IRON OXIDE NANOSTRUCTURE BY THERMAL OXIDATION

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Material) with Honors.

by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Material) (Hons). The Member of the supervisory is as follow:

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(Project Supervisor)

ABSTRAK

Oksida ferum nanostruktur telah disintesis pada kepingan Fe tulen dengan pendekatan proses pengoksidaan terma. Konvensional, oksida ferum nanostruktur boleh dihasilkan oleh teknik-teknik seperti; proses *sol-gel*, kaedah templat, penguraian terma, proses hidroterma dan lain-lain. Walau bagaimanapun, teknik ini adalah mahal dan rumit. Oleh itu, projek ini bertujuan untuk menghasilkan oksida ferum dengan menggunakan pengoksidaan terma. Beberapa parameter telah disiasat seperti kesan masa pengoksidaan, suhu pengoksidaan dan syarat ke atas substrat. The morfologi dan fasa Fe_2O_3 nanostruktur disifatkan. Kesan suhu pengoksidaan membentuk Fe_2O_3 nanostruktur. Beberapa fasa oksida yang dapat diperhatikan seperti α-Fe_2O_3 dan Fe_3O_4. Tambahan pula, pemerhatian FESEM menunjukkan bahawa Fe_2O_3 struktur telah berjaya ditubuhkan pada kepingan Fe tulen. XRD dan spektroskopi Raman mengesahkan fasa: α-Fe_2O_3 dan Fe_3O_4 berlaku selepas pengoksidaan. Raman spektroskopi telah digunakan untuk menyokong morfologi dan fasa diperolehi oleh SEM dan XRD. Walaupun, sampel optimum (pengoksidaan pada 400°C, 90 minit dalam udara) adalah tertakluk kepada pencirian elektrokimia dalam 250ml KOH elektrolit dan dalam ujian photodegradation bawah keadaan cahaya UV. Hasil daripada degradasifoto ini menunjukkan bahawa, sebagai meningkatkan masa larutan *methyl orange* telah berubah warna, analisis UV kelihatan digunakan. Berdasarkan keputusan yang diperolehi, dapat disimpulkan bahawa larutan itu berlaku proses degradasi berkesan apabila masa pendedahan kepada cahaya secara semakin meningkat.

ABSTRACT

Iron oxide nanostructure was synthesized on pure Fe foil by thermal oxidation process approach. Conventionally, iron oxide nanostructure can produced by others techniques such as; sol-gel process, template method, thermal decomposition, hydrothermal process and others. However, these technique are costly and complicated. Hence, this project serves to fabricate the iron oxide by using thermal oxidation. Several parameter were investigated such as the effect of oxidation time, oxidation temperature and condition on the substrate. The morphologies and phases of Fe_2O_3 nanostructure was characterized. The effect of oxidation temperature formed Fe_2O_3 nanostructure. Several phase oxide were observed such as α-Fe_2O_3 and Fe_3O_4. Furthermore, FESEM observation showed the ordered Fe_2O_3 nanostructure structure was successfully formed on pure Fe foil. XRD and Raman spectroscopy confirmed variant phase: α-Fe_2O_3 and Fe_3O_4 occurred after oxidizing. Raman spectroscopy were used to support the morphologies and phases observed by SEM and XRD. While, the optimum sample (oxidized at 400°C, 90min in air) was subjected to electrochemical characterization in 250ml KOH electrolyte and in photodegradation testing under UV light conditions. The result obtain from this photodegradation shows that, as increase the time of methyl orange solution has changed color, UV visible analysis is used. Based on the results obtained, it can be concluded that the solution was effective degradation process when the time of exposure to light progressively increase.

DEDICATION

Dedicated to my beloved family members especially my parents, lecturers, and also to all my friends.

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First of all, I would like to thank to Allah, for giving a great opportunity, strength and wealthy to complete my Project Sarjana Muda (PSM) titled “Formation of iron oxide nanostructure by thermal oxidation”. A grateful to my supervisor, Dr. Syahriza Binti Ismail who had given knowledge, advice and guidance throughout the entire project. Besides, without the moral support and understanding from my family especially my father, En. Abu Bakar Bin Hamid and my mother, Pn.Salbiah Binti Abd Rahman, it would be impossible for me to complete this research. Special thanks to my entire friend give cooperation, support and guidance provided all this while. I am greatly touched by the commitments and dedications they have shown. Finally, I wish to say that I treasure very much the friendship of my friends who have been very supportive in providing all necessary help and advice during journey of completing this research.

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SEM	-	Scanning electron microscopy
XRD	-	X-Ray Diffraction Microscopy
FESEM	-	Field Emission Scanning electron Microscopy
0D	-	Zero Dimension
1D	-	One Dimension
2D	-	Two Dimension
3D	-	Three Dimension
CVD	-	Chemical Vapor Deposition
CV	-	Cyclic Voltammetry

CHAPTER 1

INTRODUCTION

This chapter describes the introduction of the project. In this project, thermal oxidation process was used to form nanostructures of iron oxide. This chapter also includes the background, problem statement, objectives, scope and project outline of this study.

1.1 Background

In the last decade, nanoscience and nanotechnology have much interest in fundamental research and industrial applications. As the foundation of nanoscience and nanotechnology, nanostructured materials used on multiple research fields. Normally, nanostructures are defined as those structures with at least one dimension less than 100 nm (Xia *et al*., 2003). In this dimension, the number of atoms is countable, making the properties of nanostructures different from those of their bulk counterparts or single atoms, despite the fact that they share the same chemical compositions. Within the structure of countable atoms, the combination of quantum effects and multi-body interaction may contribute to several properties. Based on these properties, various future applications of nanostructures can be applied, such as in chemical and biological sensors, optical devices and so on. Since the revolutionary discovery of carbon nanotubes in 1991, one-dimensional (1D) nanostructures such as nanowires, nanobelts, and nanotubes become interesting due to the confinement of the other two dimension perpendicular to longitudinal direction (Xia *et al*., 2003). Due to the combination of quantum confinement in the nanoscale dimensions and the bulk properties in another dimension, a host of interesting properties and applications can be expected based on a wide variety of 1D nanostructures. Since the nanostructures is studied, some important which are to control of morphology, size and growth direction. In addition, the different morphologies obtained will affect the resulted properties with unique application.

Recently, nanostructure have been synthesized by various methods which from three phases. From the liquid phase (hydrothermal, electrodeposition), from the gas phase (Chemical vapor deposition) and from the solid phase (thermal oxidation). Nevertheless, thermal oxidation in various oxidizing atmosphere is a most simple, cheap, and direct procedure to form hematite (α-Fe_2O_3) (Grigorescu *et al*., 2012). In thermal oxidation, the oxide layer will be formed. Under typical conditions, layer of oxide will be formed, as hematite, magnetite, and wustite. Hematite (Fe_2O_3) is formed at the outer layer while magnetite (Fe_3O_4) at intermediate layer and wustite (FeO) layer form on an iron substrate (Marciu *et al*., 2012). The growth of iron oxide by thermal oxidation technique can be varied by different condition such as temperature, oxidation time and additive.

Furthermore, the characteristics of these oxide compounds include mostly the trivalent state of the iron, low solubility and brilliant colors (Cornell & Schwertmann, 1996). All the iron oxides are crystalline, except Schwertmannite and ferrihydrite which are poorly crystalline sixteen pure phases of iron oxides, i.e., oxides, hydroxides or oxy-hydroxides are known to date. These are Fe $(OH)_3$, $Fe(OH)_2$, $Fe_5HO_8.4H_2O$, Fe_3O_4, FeO, five polymorphs of FeOOH and four of Fe_2O_3 (Mohapatra and Anand, 2010). In Fe_2O_3, most frequent polymorphs alpha and gamma have been found in nature as minerals hematite and maghemite. Furthermore, hematite (α-Fe_2O_3) is one of the most stable iron oxide under ambient conditions and exhibits various interesting application. The application of iron oxide can be seen in many areas such as photoanode, pigments, catalyst and so on (Wen *et al*., 2005). However, the research focusing on the photocatayst application. Figure 1.1 shows the application of iron oxide in nano-scale (Liu *et al*., 2008).

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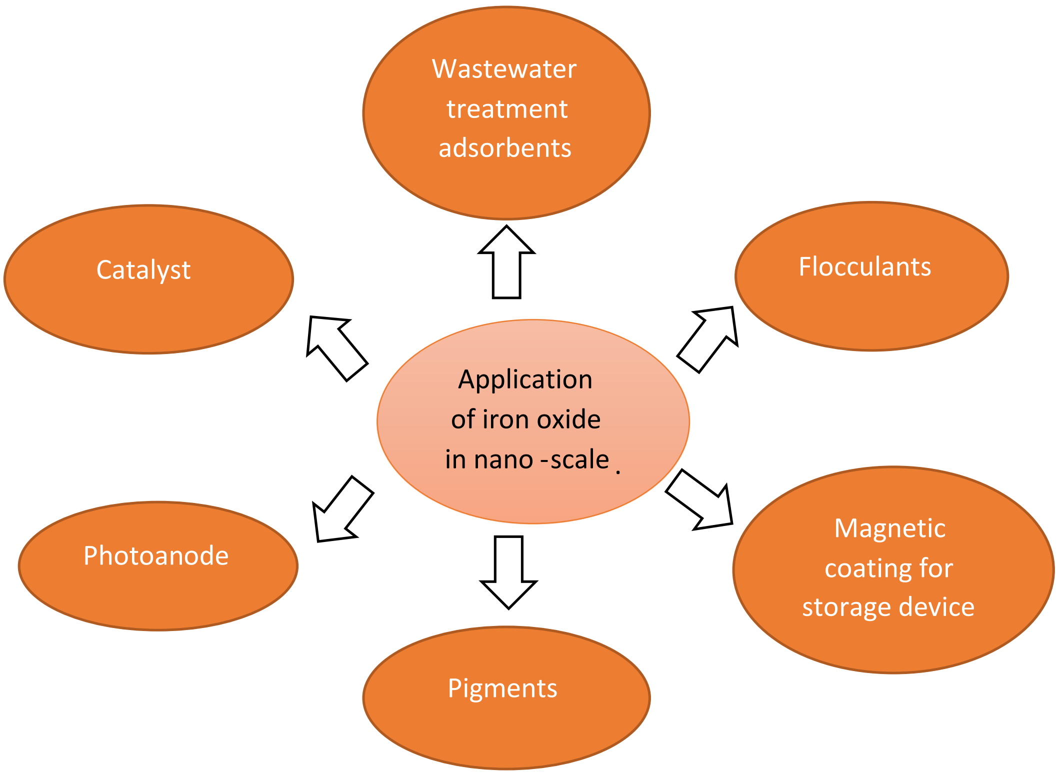

Figure 1.1: Application of iron oxide in nano-scale (Liu *et al*., 2008).

Photocatalytic reaction promoted by structured metal oxide have become interested in subject of recent research. The use of iron oxide in the solar photoelectrolysis cell was reported by Bakardijeva et al (2007), which indicate that iron oxide is a promising photocatayst. While, in photoelectrochemical, α-Fe_2O_3 has been used as photoanode since it has good electrochemical stability for water splitting. The details on this iron oxide application will be reviewed in chapter 2, section 2.6.

1.2 Problem Statement

Nowadays, due to population growth, rapid development of industrialization and long term droughts are contributed to a lot of contamination include inorganic compounds, organic pollutants, and many other complex compounds especially in a river. All contaminants release to the environment through wastewater which are harmful to humans and the environment. Therefore, removing contaminants is uncertain. Furthermore, existing method to eradicate the problem are filter and particle photocatalyst, but both method are less efficient and non-recyclable. To combat water pollution problem, a new treatment can be done by photocatalytic oxidation using the nanostructure iron oxide by thermal oxidation method. Thermal oxidation method is a most simple, direct produce hematite phase and low cost.

Recently, nanostructure metal oxide have been suggested as cost-effective and environment friendly alternative to existing treatment material in photocatalytic application. Besides that, the iron oxide nanostructure properties such as excellent magnetic properties, high stability against corrosion, large surface area and high surface modification flexibility which are not found in bulk-sized material. In water treatment technologies, four conditions must be considered: (1) treatment flexibility and final efficiency, (2) reuse of treatment agents, (3) environment security and (4) low cost (Xu *et al*., 2012).

Until now, ZnO and TiO_2 are used as photocatalyst for degradation of organic pollutants. Nevertheless, iron oxide is promising for wastewater treatment due to low cost, strong adsorption capacity, easy separation and enhanced stability (Xu *et al.*, 2012). According to Abe (2010), iron oxide with which are easy to prepare, can absorp at longer wavelengths and the high stability during irradiation are very attractive for photocatalytic application. Most important, iron oxide are abundantly available as waste metal in many industry. But, ZnO has a drawback related to unsatisfactory photostability in wide pH range of solution. Mostly, many semiconductor materials for photocatalysts are relatively wide band gap energy, such as for anatase and rutile TiO_2 contains 3.2eV and 3.02eV bandgap and 3.2eV for ZnO (Tyagil and Rajl, 2006). These semiconductors can only be excited by photons which are close to the UV region and utilize only 4-6% of solar light, which limits their practical applications. Furthermore,