

PHOTOCATALYSIS APPLICATION OF  
TiO<sub>2</sub> AND Fe<sub>2</sub>O<sub>3</sub> NANOSTRUCTURES

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**PHOTOCATALYSIS APPLICATION OF  
TiO<sub>2</sub> AND Fe<sub>2</sub>O<sub>3</sub> NANOSTRUCTURES**

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with Honours.

by

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfilment of the requirement for the Bachelor Degree of Manufacturing Engineering with Honours. The member of the supervisory committee is as follow:

.....  
Dr. Syahriza Binti Ismail

## ABSTRAK

Kajian tentang aplikasi fotokatalisis telah dikaji secara meluas kerana prestasi fotokatalisis yang cemerlang dalam aspek pemulihan alam sekitar. Contoh-contoh aplikasi fotokatalisis melibatkan degradasi bahan pencemar organik dan bukan organik, permukaan “self-cleaning”, permukaan “anti-fogging”, produk pembersihan dan aplikasi anti-bakteria. Tujuan kajian ini adalah untuk menentu dan mengukur kecekapan fotokatalisis bahan fotokatalis  $\text{TiO}_2$  dan  $\text{Fe}_2\text{O}_3$  yang mempunyai bentuk nanostruktur dengan menggunakan degradasi metil oren (MO). Spektrometer Raman (RAMAN), mikroskopi imbasan elektron (FESEM) dan difraksi Sinar-X (XRD) digunakan untuk mengifatkan sampel kepingan and sampel serbuk bahan  $\text{TiO}_2$  dan  $\text{Fe}_2\text{O}_3$ . FESEM analisis telah menunjukkan bentuk nanostruktur sampel-sampel bahan. Tambahan pula, RAMAN dan XRD analisis telah menyokong kepada kewujudan fasa anatase and fasa rutile dalam sampel bahan  $\text{TiO}_2$  manakala wujudnya fasa  $\alpha\text{-Fe}_2\text{O}_3$  dalam sampel bahan  $\text{Fe}_2\text{O}_3$ . Beberapa faktor atau parameter yang penting seperti jarak antara sumber cahaya dan cecair MO, jarak waktu, dan kepekatan cecair MO akan dikawal. UV-Vis spektrometer akan digunakan untuk menganalisis hasil degradasi cecair MO. Hasil daripada ujian fotodegradasi, jarak yang dekat (10cm), jarak waktu yang panjang (5 jam) dan kepekatan cecair MO yang rendah (10ppm) dapat meningkatkan prestasi fotokatalisis. Berdasarkan keputusan yang diperolehi, kesimpulan yang boleh dipaparkan ialah,  $\text{TiO}_2$  mempunyai prestasi fotokatalisis yang lebih unggul daripada  $\text{Fe}_2\text{O}_3$ . Di samping itu, sampel serbuk lebih berkesan daripada sampel kepingan.

## ABSTRACT

Photocatalysis applications have been studied extensively due to its excellent photocatalysis performance in environment remediation. The photocatalysis applications involves degradation of organic and inorganic pollutants, self-cleaning surfaces, anti-fogging surfaces, purification products and anti-bacterial applications. The aim of this study is to determine and quantify the photocatalytic efficiency of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  based photocatalyst materials by using methyl orange (MO) degradation. RAMAN spectrometer, field emission scanning electron microscope (FESEM) and X-ray diffractometer (XRD) were employed to characterize  $\text{TiO}_2$  thin film,  $\text{TiO}_2$  powder,  $\text{Fe}_2\text{O}_3$  thin film and  $\text{Fe}_2\text{O}_3$  powder. FESEM images have shown the nanostructure of the samples. RAMAN and XRD have confirmed there are anatase and rutile phases exist in  $\text{TiO}_2$  samples while  $\alpha\text{-Fe}_2\text{O}_3$  phase is observed in  $\text{Fe}_2\text{O}_3$  samples. The materials are then embedded into MO solution for photocatalytic testing. A few important factors or parameters such as distance between the light source and MO solution, time interval, and concentration of MO solution were being control. UV- Vis spectrometer was used to analyse the degradation result of MO solution. The data obtained from the UV-Vis analysis shows that at 10cm from the light, 5 hrs exposure time under UV light and 10ppm MO solution are giving the better efficiency of MO photodegradation. Based on the result obtained, a conclusion of  $\text{TiO}_2$  based photocatalyst has better photocatalytic performance in photocatalysis activity of MO solution than  $\text{Fe}_2\text{O}_3$  based photocatalyst is made. At the same time, the photodegradation rate of material with powder form is more effective than thin film material.

## **DEDICATION**

I am dedicating this work to my beloved parents, Tan Chee Chin and Yap Bee Cheng who always support and encourage me to achieve a success in everything I do.

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## LIST OF ABBREVIATIONS

AOPs	-	Advanced Oxidation Processes
ECs	-	Emerging Contaminants
FESEM	-	Field Emission Scanning Electron Microscope
FTO	-	Fluorine Doped Tin Oxide
MB	-	Methylene Blue
MO	-	Methyl Orange
NO <sub>x</sub>	-	Oxides of Nitrogen
PC	-	Photochemical cell
PEC	-	Photo-electrochemical cell
ROS	-	Reactive Oxidizing Species
SEM	-	Scanning Electron Microscope
TEM	-	Transmission Electron Microscope
TOC	-	Total Organic Carbon
UV	-	Ultraviolet
UV-Vis	-	Ultraviolet–visible
VOCs	-	Volatile Organic Compounds
XRD	-	X-ray Diffractometer

## LIST OF SYMBOLS

$\lambda$	-	Wavelength
$A$	-	Absorbance
$A_0$	-	Initial Absorbance
$c$	-	Speed of Light
$C$		Final Concentration
$C_0$		Initial Concentration
cm	-	Centimetre
$D$	-	Distance
$e_{CB}^-$	-	Electron in Conduction Band
eV	-	Electron volt
$E$	-	Energy
$E_g$	-	Band Gap
g	-	Gram
hr		Hour
$I$	-	Intensity
$I_D$	-	Intensity of D Band Peak
$I_G$	-	Intensity of G Band Peak
$I_0$	-	initial intensity of Light
$H$	-	Planck's Constant
$h_{VB}^+$	-	Positive Hole
hv	-	Energy of Light

log	-	Logarithm
L	-	Litre
mL	-	Millilitre
mm	-	Millimetre
mins	-	Minutes
nm	-	Nanometre
ppm	-	Part per Million
t	-	Time
T	-	Transmittance

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Photocatalysis is the combination of two words which are “photo” and “catalysis”. Photo is the energy in the form of light and catalysis is a process which a compound or substance is added to increase the rate of chemical reaction. In other word, photocatalysis is the process that use of light activated substances to speed up chemical reaction. Photocatalysis can convert the harmful organic compound and bacteria into harmless substance which does not pollute the environment. A completion can be make for photocatalysis process which is that in the present of light, photocatalyst can creates strong oxidising agent and electron-hole pair to breakdown organic compound into carbon dioxide and water. Photocatalysis has seem to become popular and growing interest recently, not only because it widely used application for environmental remediation, but also because its sustainability and environmentally friendly. Photocatalysis is said to be sustainable because the light source used for it can be obtain easily from sunlight and it is renewable.

Metal oxides or semiconductors are abundant in nature, used of metal oxides as photocatalyst application have been started since several decades ago due to their chemical stability, light absorption properties and ability to produce photo-generated electron- hole pair (Hoffmann, Martin, Choi, & Bahnemann, 1995; Khan, Adil, & Al-Mayouf, 2015; Hisatomi, Kubota, & Domen, 2014; Wang et al., 2014). Among the numerous metal oxides, researches about photocatalytic activity of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  were mostly being studied. According to the researches, metal oxides can be used as photocatalyst for environmental purification, decomposition or sterilization of pollutant and organic compounds, deodorization of smell, self- cleaning and anti-fogging of glass.

At 1972, A. Fujishima and Honda had first demonstrated that possibility of water photo-splitting into hydrogen and oxygen by using electrochemical photocell with  $\text{TiO}_2$  electrode (Pelaez et al., 2012; Fujishima & Zhang, 2006). However, due to  $\text{TiO}_2$  has the wide band gap,  $E_g$  ( $\sim 3.0 - 3.2\text{eV}$ ),  $\text{TiO}_2$  can only absorb the UV light which has short wavelength ( $\lambda < 400\text{nm}$ ) (Liu, Tian, Tan, Li, & Chen, 2017; Fujishima & Zhang, 2006). UV light is only occupied less than 5% of the solar energy from the sunlight. In addition, the common light source use for photocatalysis is a fluorescent lamp with phosphor coating. The phosphor coating absorbs the harmful UV-B and UV-C light rays and only allows the emission of UV-A light ray (Fujishima & Zhang, 2006). This situation has reduced the content of UV light, there is a low efficiency rate of photocatalytic activity under UV illumination. Therefore, to overcome this deficiency, a lot of efforts were carried out by the scientific community. The use of visible light for photocatalysts is being investigate because visible light make-up the largest part of solar energy. Another metal oxide semiconductor,  $\text{Fe}_2\text{O}_3$  metal oxide is selected as the photocatalyst under visible light illumination due to its lower  $E_g$  ( $\sim 2.2\text{eV}$ ) compare to  $\text{TiO}_2$ . The low  $E_g$  of  $\text{Fe}_2\text{O}_3$  allows it to absorb the light which the  $\lambda$  up to  $\sim 600\text{nm}$  (Karunakaran & Senthilvelan, 2006).

In order to utilize and enhanced the sensitivity of photocatalyst to visible light, several approaches have been proposed (Pelaez et al., 2012; Fujishima & Zhang, 2006):

- a. Doping of photocatalysts with metal ions
- b. Doping of photocatalysts with non-metal atoms
- c. Preparation techniques

## 1.2 Problem Statement

Nowadays, the industrialization development and technology innovation are said to be achieved at the global level. Technology innovation and industrialization development had make human life better. The advance in the living standard or the improved in quality of life has led to the growth of urbanization. Industrialization, technology development and urbanization had create numerous environmental issues ranging from the local to the global scale (Kim & Baik, 2005; Cui & Shi, 2012), including increased air and water pollution and decreased water supply (J. Liu & Diamond, 2005), climate change and increased in energy consumption (Zhou et al., 2004).

Deforestation activity is increased to clear the land for the residential housing and office building and factory purposes. Industrial revolution had increased the emission of harmful gases and chemical such as sulphur dioxide, oil, lead and mercury. Industrial waste is one of the main factor which will cause the water and air pollution. For example, sulphur dioxide has acidic properties and may contribute to acid rain. The acid rain will cause the death of aquatic life and corrosion of building. The high energy demands had increases the rate of electricity generation which mainly lead to air pollution. In addition, most of the energy sources such as fossil fuels, natural gas and coal are non- renewable sources. The consumption or combustion of these sources will release the Greenhouse gases which cause global warming. Global warming lead to the rising of temperature which causing the melting of polar ice and rise in sea levels.

The poor quality of air and water will affect the health of human, animal and other living organisms. Air pollution will cause the respiratory system diseases and skin diseases. Water pollution will cause the toxicity problems if human consume the polluted water or the aquatic life.

In order to solve the issues, semiconductor photocatalysis which using solar energy (sunlight) as the source of driving force (solar- driven) is one of the alternative solution (C. Wang & Huang, 2016). In this research, the photocatalytic activity of  $\text{TiO}_2$  and  $\alpha\text{-Fe}_2\text{O}_3$  are being investigated. Metal oxides are used as the photocatalyst application because of their

promising arrangement of electronic structures, light absorption properties and capability to generate charge carrier (Khan et al., 2015).

### 1.3 Objectives

The objectives of this researches are:

1. To characterize TiO<sub>2</sub> thin film, TiO<sub>2</sub> powder and Fe<sub>2</sub>O<sub>3</sub> thin film and Fe<sub>2</sub>O<sub>3</sub> powder.
2. To measure and compare the photocatalytic activity pure TiO<sub>2</sub> thin film, pure TiO<sub>2</sub> powder and pure Fe<sub>2</sub>O<sub>3</sub> thin film and pure Fe<sub>2</sub>O<sub>3</sub> powder by using methyl orange (MO) degradation.
3. To investigate the effect of the photocatalytic parameter of MO degradation such as the distance between the light source and MO solution, time interval and concentration of MO solution.

### 1.4 Scope

In this research, the photocatalytic mechanism and photocatalytic properties of TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> will be studied. The working principle of the photocatalysis applications will also be discussed. RAMAN spectroscopy and X-ray diffractometer (XRD) are selected for phase identification of the TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> materials samples. Field emission scanning electron microscopy (FESEM) is used to observe the nanostructure of the sample. Degradation of methyl orange (MO) under light irradiation will be used to measure and evaluate the photocatalytic activity of the TiO<sub>2</sub>, and Fe<sub>2</sub>O<sub>3</sub> samples. The different condition of light illumination has been considered during the photocatalysis process. Degradation result of MO is analysed by using the UV-Vis spectroscopy method. UV- Vis spectrometer is used to measure the temporal changes of MO solution concentration.

## 1.5 Overview

This thesis consists of five chapters. Chapter 1 presents the general context of the project, a short background of photocatalysis and explained the factors lead to the development of photocatalysis technology, and finally the statement of objectives. Chapter 2 describes the photocatalysis mechanism, photocatalysis materials, photocatalysis applications and enhancement of the photocatalysis efficiency. Chapter 3 summarizes and discusses the procedure of the material preparation, characterization approaches and experiments details. Chapter 4 focuses on the result and discussion of the project. Lastly, Chapter 5 includes conclusion and recommendation.