# PREPARATION AND CHARACTERIZATION OF HEMATITE IRON OXIDE/CNT FOR PHOTOCATALYST

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

by

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# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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TAJUK: **PREPARATION AND CHARACTERIZATION OF HEMATITE IRON OXIDE/CNT FOR PHOTOCATALYST**

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# APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Material Engineering) (Hons.). The members of the supervisory committee are as follow:

(DR. SYAHRIZA BINTI ISMAIL)

# ABSTRAK

Penyelidikan ini untuk sintesis oksida besi dengan disaluti tiub nano karbon di permukaan, dengan cara salutan berpusing. Oksida besi dihasilkan daripada cara pengoksidaan haba dengan menggunakan parameter terbaik, 400°C dan 90 minit. Tiub nano karbon telah melalui rawatan asid dengan 3M $HNO_3$ untuk pembersihan dan menggurangkan pengumpulaan dari tiub nano karbon asal. Dalam projek ini, tiub nano karbon telah disuraikan dengan acetone, SDBS (Sodium Dodecyl Benzene Sulphonate) and DMF (Dimethylformamide) untuk mendapatkan sebaran tiub nano karbon yang terbaik dalam morfologi. Pelepasan bidang imbasan mikroskop elektron (FESEM) digunakan untuk menyelidik morfologi pembentukan oksida besi/tiub nano karbon, manakala X-ray pembelauan (XRD), raman spektroskopi, fotoluminesen spektroskopi (PL) and fourier mengubah inframerah spektroskopi (FTIR) digunakan untuk menyelidik pencirian struktur oksida besi/tiub nano karbon. Fotopemangkin uji kaji dilakukan dengan mengunakan lampu UV. Manakala, UV uji kaji untuk menetukan keupayaan oksida besi/tiub nano karbon sebagai pemangkin yang baik. Keputusan menunjukkan ciri-ciri oksida besi sebagai pemangkin telah bertambah baik dengan kehadiran tiub nano karbon dipermukaannya. Fotopemangkin uji kaji membuktikan oksida besi/tiub nano karbon telah menigkatkan kadar kemerosotan fotopemangkin.

# ABSTRACT

The purpose of this study is to synthesis hematite iron oxide nanowires decorated with MWCNTs by spin coating method. The substrate hematite iron oxide nanowires were produce by thermal oxidation method with optimum parameter, 400°C and 90 minutes. MWCNTs were treated with 3M $HNO_3$, for purification of the catalyst and to reduce agglomeration from the raw CNT. In this work, MWCNTs were dispersed in acetone, SDBS (Sodium Dodecyl Benzene Sulphonate) surfactant and DMF (Dimethylformamide) solution to get the best dispersion in morphology. Field emission scanning electron microscopy (FESEM) was used to investigate the morphology of iron oxide nanostructure/CNT formed, while X-ray diffraction (XRD), Raman spectroscopy, Photoluminescent spectroscopy (PL) and Fourier Transform Infrared Spectroscopy (FTIR) were used to investigate the structure characterization of hematite iron oxide nanostructure/CNT formed. Photocatalysis testing also had done using the UV lamp. While, UV visible to determine the ability of hematite iron oxide nanostructure/CNT as the good catalyst. The results were show the properties of hematite iron oxide as a catalyst were improved by decorated CNT on its surface. Photocatalysis testing was proved the degradation rate was increased by using sample hematite iron oxide/CNT.

# DEDICATION

This report is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time. Last but not least, dedicated to my brothers that always encourage and give me a moral support to finish what I have started.

ACKNOWLEDGEMENT

In the name of Allah, the most gracious, the most merciful, with the highest praise to Allah that I manage to complete this final project successfully without difficulty.

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| | | |
|---|---|---|
| CNT | - | Carbon Nanotube |
| SWNT | - | Single walled nanotube |
| MWNTs | - | Multi walled nanotube |
| SDBS | - | Sodium Dodecyl Benzene Sulphonate |
| DMF | - | Dimethylformamide |
| XRD | - | X-ray diffraction |
| PL | - | Photoluminescent spectroscopy |
| FTIR | - | Fourier Transform Infrared Spectroscopy |
| FESEM | - | Field Emission Scanning Electron Microscopy |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| K | - | Kelvin |
| °C | - | Degree Celsius |
| e.V | - | Electron Volts |

# CHAPTER 1
# INTRODUCTION

This chapter describes the introduction of the project. In this project, to determine the characteristic of iron oxide when decoration with carbon nanotube. This chapter also includes the background, problem statement, objectives, scope and project significance of this study.

## 1.1 Background Research

The genuine environment contamination and energy crisis due to the broad usage of fossil energizes are considered as the two noteworthy difficulties in the 21st century. Understanding the significance of protecting our planet in clean, scientists are effectively working for eco-accommodating elective advances for all regions of day by day life. Supportable vitality generation and poison eradication are two of the area that research is being excessively done. Semiconductor photocatalysis is an alternative for contamination eradication and hydrogen generation (clean fuel) creation by water part. Photocatalysis is a"catalytic reaction involving the production of a catalyst by absorption of light". The fitting situating of valence (VB) and conduction (CB) groups in semiconductors makes them reasonable materials for the absorption of light and photocatalytic activity (Manoj A. Lazar, 2012).

Generally, ZnO and $TiO_2$ are very famous photocatalyst for degradation of organic pollutants. But, iron oxide is also a good catalyst in photocatalytic activity. Hematite iron oxide nanostructure have a higher surface area, high conductivity and low cost. In this work, iron in a form of foil will be thermally  oxidize to react with oxygen to produce iron oxide. However, as the naked iron oxide is not enough to improve the process. So, hematite was  attached with nano materials to improve the properties as photocatalyst.

Carbon nanotubes have special physical and chemical properties that become attraction to the industry nowdays. One of the physical properties of carbon nanotubes is that the possiblity of making the thickness into single atomic layer with the magnetic properties of iron oxides combined with the unique properties of CNT would produce an effective photocatalyst materials especially for magnetic adsorbent. In this research, carbon nanotubes must deposit on iron oxide by spin coating method. Spin coating method is widely used for carbon nanotube and can create carbon nanotubes on various surface. Furthermore, iron will produce nanowires in thermal oxidation process. However, perfect nanowires structures can not be produced using spray coating process. This because by using spray coating, nanowires will fall during spraying. So the best method to decorate the nano iron oxide with carbon nanotube is using a spin coating method.

In this work, nanostructured iron oxide were prepared and characterized by X-ray diffraction (XRD), Raman Spectroscopy, Photoluminescent spectroscopy (PL), Fourier Transform Infrared Spectroscopy (FTIR) and field emission scanning electron microscope (FESEM).

## 1.2 Problem Statement

Nowadays, due to population growth, quick development of industrialization and long term deficiencies 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 simplest and direct method to produce hematite phase of iron oxide and low cost.

Recently, the nanostructure metal oxide has been suggested as an economical and environment friendly substitute to present treatment material in photocatalytic application. Besides that, the iron oxide nanostructure properties such as excellent magnetic properties, high stability against corrosion, extensive surface zone and high surface alteration adaptability which are not found in bulk-sized material. In water treatment technologies, four conditions must be considered: (1) treatment flexibility and final productivity, (2) reuse of treatment agents, (3) environment security and (4) low cost (Xu et al., 2012).

But, iron oxide nanostructure has low absorption coefficient due to its direct band gap transition that provide low photcatalysis efficiency. Besides that, iron oxide has low charge mobilities properties that cause high recombination rate in photocatalysis. This limitation can be overcome by hybridization of iron oxide with carbon nanotube (CNT) that is a good absorbent and excellent conductivity. Therefore, in this research iron oxide nanowires with CNT will be produced by spin coating.

## 1.3 Objective

The main objectives that have to be in consideration for this project, are as stated below:

i. To synthesis hematite iron oxide decorated CNT as metal hybrid composite
ii. To characterize the properties of hematite iron oxide/CNT in hybrid formed.
iii. To evaluate the degradation of Methyl Orange using hematite iron oxide/CNT for photocatalyst application.

## 1.4 Scope

This research covered the study on the formation of an oxide layer by thermal oxidation and decorated with carbon nanotube (CNT). For the synthesis of the nanostructure of iron oxide, the thermal oxidation technique was selected due to low cost method and is a direct method to produce hematite . The thermal oxidation was set up by using chamber furnace and conducted in

90 minutes. While, the oxidation temperature is 400°C. This is a optimize paeameter to growth nanowires in thermal oxidation process.

Then after growth nanowires, CNT were decorated on it by using spin coating. But before coating, CNT will be disperse in acetone, Sodium Dodecyl Benzene Sulphonate (SDBS) surfactant and Dimethylformamide (DMF). Then, the spin coating will set up with the range of speed, 3500–4500 rpm; and time, 30–120 seconds. This parameter will use to decorated the CNT on the nanowires structural in spin coating process.

Furthermore, the application and feasibility of iron oxide/CNT as a photocatalyst was also considered with degradation process of methyl orange. Phase and morphology characterization on the nanowires of iron oxide were examined by using X-Ray Diffraction (XRD), Raman Spectroscopy, Photoluminescent spectroscopy (PL), Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM). Meanwhile, the degradation analysis of methyl orange dye was evaluated by using UV visible spectroscopy.

## 1.5 Significant of the report

This research or experiment is important to achieve all the objectives in the end of the research and the results that can get from the experiment must have improvement from the previous research like from aspect of parameter of that use for the experiment. This research also important to solve the problem statement of the properties of iron oxide to be more stability and good properties when react with carbon nanotubes.

# CHAPTER 2
# LITERATURE REVIEW

## 2.1 Introduction

This section basically will discuss on the literature review that related to the general iron oxide and carbon nanotube (CNT). Summarized of other people investigation has been highlighted and discussed. Iron oxide nanostructure have a higher surface area, high conductivity and low cost. One dimensional nanostructure show extraordinary optical, electronic, attractive and mechanical properties as contrasted with those mass of materials (Zhou et al., 2008). But iron oxide is not the best absorbent material in photocatalysis. Carbon nanotube is owed to the tunable physical, chemical, electrical, and structural properties, this can motivate new technologies to address the water pollution harms. In addition CNT is a good absorbent and has high conductivity properties, that good in photocatalysis (Liu, Wang, Zhang, & Pan, 2013).So, to improve the properties of iron oxide as photocatalyst, was decorated with CNT that have good properties in photocatalysis.

## 2.2 Nanostructure materials

Practical nanostructures on metals, semiconductors, or polymers have been ceaselessly created for different applications, for example, photovoltaic tools, biomaterials, optical focal points and catalysis. The setup of nanostructures is considered as a regulating variable for