



UNIVERSITI PUTRA MALAYSIA

**PHOTOELECTROCATALYTIC DEGRADATION OF DYES BY TITANIUM
DIOXIDE THIN FILMS PREPARED VIA THERMAL OXIDATION AND
ELECTRODEPOSITION**

ALVIN CHONG JING KAI

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By

ALVIN CHONG JING KAI

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

May 2009



DEDICATION

I would like to dedicate my work to my beloved parents, brother, sister and also my girlfriend for their support to carry out my Master Degree study.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

PHOTOELECTROCATALYTIC DEGRADATION OF DYES BY TITANIUM DIOXIDE THIN FILMS PREPARED VIA THERMAL OXIDATION AND ELECTRODEPOSITION

By

ALVIN CHONG JING KAI

May 2009

Chairman: Professor Zulkarnain Zainal, Ph.D

Faculty: Science

Titanium dioxide (TiO_2) thin film electrodes were prepared using two techniques which were cathodic electrodeposition and thermal oxidization of titanium plates. The characteristic of TiO_2 electrodes were analysed using X-Ray Diffractometry (XRD), Field Emission Scanning Electron Microscopy (FESEM) and UV/Vis Spectroscopy. TiO_2 anatase and rutile phase structure was found in electrodeposition TiO_2 thin film after heat treatment whereas only rutile phase was observed for thermal oxidation TiO_2 thin film. Electrodeposited and thermally oxidized TiO_2 electrodes showed the highest photosensitivity after calcination at 600 °C and 700 °C respectively when analysed using Linear Sweep Photovoltammetry (LSPV) technique.

Photoelectrochemical degradation of dyes was carried out in a 3 electrode system reactor where the working electrode was TiO_2 thin film under illumination of a light source for 2 hours. The removal of dyes was investigated by monitoring dyes decolourisation rates using UV/Vis Spectroscopy. The photoelectrochemical degradation studies of Chicago Sky Blue 6B (CSB) dye was studied varying the initial

dye concentrations, applied potentials and supporting electrolytes. The effect of repeated usage, light sources and changing removal methods were also examined.

Photoelectrocatalytic degradation system for both TiO₂ thin film electrodes achieved better removal of CSB dye than in photocatalytic system. Thermal oxidized TiO₂ electrode gave faster removal rate compared to electrodeposited TiO₂ electrode in photoelectrocatalytic degradation of CSB dye. The removal of CSB increased with the increased of external applied potential from 0 V to 1.5 V versus Ag/AgCl reference electrode in both TiO₂ thin film electrodes. The kinetic data at different applied potential fitted well to first-order kinetic model.

TiO₂ thin film electrodes showed its best photoelectrocatalytic degradation under illumination of UV light. The removal percentages of 5 times repeated usage for thermal oxidized TiO₂ electrode showed insignificant differences. However, the removal percentages of CSB decreased when electrodeposited TiO₂ electrode was used repeatedly for 5 times. The photoelectrocatalytic removal efficiency of CSB was compared with two different dyes which are Methyl Orange (MO) and Methylene Blue (MB). The removal of MO was higher than in CSB whereas the removal of MB was the lowest for both TiO₂ electrodes.

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

**DEGRADASI FOTOELEKTROMANGKINAN PEWARNA MENGGUNAKAN
FILEM NIPIS TITANIUM DIOKSIDA YANG DISEDIAKAN MELALUI
PENGOKSIDAAN TERMA DAN PENGELEKTROENAPAN**

Oleh

ALVIN CHONG JING KAI

May 2009

Pengerusi: Profesor Zulkarnain bin Zainal, Ph.D

Fakulti: Sains

Elektrod filem nipis titanium dioksida (TiO_2) telah disediakan dengan dua teknik iaitu pengelektroenapan katod dan pengoksidaan terma kepingan titanium. Ciri-ciri elektrod TiO_2 telah dianalisis menggunakan Pembelauan Sinar-X (XRD), Mikroskop Pengimbasan Elektron Pancaran Medan (FESEM) dan Spektroskopi Ultra Lembayung Nampak (UV/Vis). Filem nipis TiO_2 daripada pengelektroenapan didapati berfasa anatase dan rutil selepas rawatan haba manakala hanya fasa rutil telah diperolehi bagi sampel yang disediakan melalui kaedah pengoksidaan terma. Elektrod TiO_2 dengan kaedah pengoksidaan terma dan pengelektroenapan masing-masing menunjukkan kefotopekaan yang tertinggi selepas masing-masing dipanaskan pada suhu $600\text{ }^\circ\text{C}$ dan $700\text{ }^\circ\text{C}$ apabila dianalisis menggunakan Fotovoltammetri Pengimbasan Linear.

Penyingkiran fotoelektrokimia pewarna telah dijalankan dengan sistem reaktor 3 elektrod di mana elektrod kerja adalah filem nipis TiO_2 yang disinari dengan satu punca cahaya selama 2 jam. Penyingkiran pewarna telah diselidik melalui penilaian pelunturan warna menggunakan Spektroskopi Ultra Lembayung Cahaya Nampak. Kajian telah dijalankan terhadap Chicago Sky Blue 6B (CSB) dengan mengubah

kepekatan awal pewarna, keupayaan elektrik dan elektrolit penyokong. Kesan ulangan penggunaan, sumber cahaya dan sistem penyingkiran juga telah diselidik.

Sistem fotoelektromangkinan bagi kedua-dua jenis elektrod filem nipis TiO_2 memberi penyingkiran pewarna CSB yang lebih baik daripada dalam sistem fotomangkinan. Elektrod TiO_2 pengoksida terma memberi kadar penyingkiran yang lebih cepat berbanding dengan elektrod TiO_2 elektroenapan. Penyingkiran CSB bertambah dengan bertambahnya keupayaan luar yang diaplikasi dari 0 V ke 1.5 V dibanding dengan elektrod rujukan Ag/AgCl bagi kedua-dua jenis elektrod filem nipis TiO_2 . Data kinetik pada keupayaan luar yang berbeza mematuhi model kinetik pertama.

Elektrod filem nipis TiO_2 menunjukkan penyingkiran fotoelektromangkinan terbaik dibawah sinaran cahaya ultra lembayung. Peratus penyingkiran bagi elektrod TiO_2 pengoksida terma dengan penggunaan 5 kali berturut-turut tidak menunjukkan perbezaan penyingkiran yang ketara. Bagaimanapun, peratus penyingkiran CSB berkurangan apabila TiO_2 yang dielektroenapan digunakan 5 kali berturut-turut. Kecekapan penyingkiran fotoelektromangkinan CSB dibandingkan dengan dua pewarna yang berlainan iaitu Metil Jingga (MO) dan Metilena Biru (MB). Penyingkiran MO lebih tinggi berbanding dengan CSB manakala penyingkiran MB adalah yang terendah pada kedua-dua elektrod TiO_2 .

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I would like to thank my family members for their unconditional support, patience and help in ensuring me to have a comfortable atmosphere to write my thesis. Thanks to my mother again for the delicious and healthy food that you cook for your son.

Lastly, to my beloved girlfriend, Guat Eng who always been a source of inspiration and strength throughout my study. Thank you for your love, support and understanding whenever I need it.

I certify that an Examination Committee met on 12th May 2009 to conduct the final examination of Alvin Chong Jing Kai on his Master of Science thesis entitled “Photoelectrocatalytic degradation of dyes by titanium dioxide thin films prepared via thermal oxidation and electrodeposition” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the Master of Science.

Members of the Examination are as follows:

Anuar Kassim, PhD

Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Mohd Zobir Hussein, PhD

Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Tan Wee Tee, PhD

Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Musa Ahmad, PhD

Professor
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
(External Examiner)

BUJANG KIM HUAT, PhD

Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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 are as follows:

Zulkarnain Zainal, PhD

Professor

Department of Chemistry

Faculty of Science

Universiti Putra Malaysia

(Chairman)

Abdul Halim Abdullah, PhD

Associate Professor

Department of Chemistry

Faculty of Science

Universiti Putra Malaysia

(Member)

HASANAH MOHD GHAZALI, PhD

Professor and Dean

School of Graduate Studies

Universiti Putra Malaysia

Date: 17 July 2009



DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.

ALVIN CHONG JING KAI

Date:

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LIST OF ABBREVIATIONS AND SYMBOLS

CSB	Chicago Sky Blue 6B
CV	Cyclic Voltammetry
E_C	Conduction band
EDX	Energy Dispersion X-ray
E_F	Fermi energy level
E_g	Band gap energy
E_V	Valence band
ETiO ₂ C	Titanium Dioxide Prepared by Electrodeposition
ETiO ₂ C4	Electrodeposited Titanium Dioxide Calcined at 400°C
ETiO ₂ C5	Electrodeposited Titanium Dioxide Calcined at 500°C
ETiO ₂ C6	Electrodeposited Titanium Dioxide Calcined at 600°C
ETiO ₂ C7	Electrodeposited Titanium Dioxide Calcined at 700°C
ETiOP	Electrodeposition of Peroxotitanium Hydrate
FESEM	Field Emission Scanning Electron Microscopy
HOMO	Highest Occupied Molecular Orbital
JCPDS	Joint Committee of Powder Diffraction Standard
LUMO	Lowest Unoccupied Molecular Orbital
LSV	Linear Sweep Voltammetry
LSPV	Linear Sweep Photovoltammetry
MB	Methylene Blue
MO	Methyl Orange
TiO ₂	Titanium Dioxide
TO-TiO ₂	Titanium Dioxide Prepared by Thermal Oxidation
TO7-TiO ₂	Titanium Dioxide Prepared by Thermal Oxidation at 700°C

XRD X-ray Diffractometer

