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

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MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

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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.
Titanium dioxide (TiO\textsubscript{2}) thin film electrodes were prepared using two techniques which were cathodic electrodeposition and thermal oxidation of titanium plates. The characteristic of TiO\textsubscript{2} electrodes were analysed using X-Ray Diffractometry (XRD), Field Emission Scanning Electron Microscopy (FESEM) and UV/Vis Spectroscopy. TiO\textsubscript{2} anatase and rutile phase structure was found in electrodeposition TiO\textsubscript{2} thin film after heat treatment whereas only rutile phase was observed for thermal oxidation TiO\textsubscript{2} thin film. Electrodeposited and thermally oxidized TiO\textsubscript{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\textsubscript{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 TiO$_2$ thin film electrodes achieved better removal of CSB dye than in photocatalytic system. Thermal oxidized TiO$_2$ electrode gave faster removal rate compared to electrodeposited TiO$_2$ 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 TiO$_2$ thin film electrodes. The kinetic data at different applied potential fitted well to first-order kinetic model.

TiO$_2$ thin film electrodes showed its best photoelectrocatalytic degradation under illumination of UV light. The removal percentages of 5 times repeated usage for thermal oxidized TiO$_2$ electrode showed insignificant differences. However, the removal percentages of CSB decreased when electrodeposited TiO$_2$ 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 TiO$_2$ 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 PENGGELEKTROENAPAN

Oleh

ALVIN CHONG JING KAI

May 2009

Pengerusi: Profesor Zulkarnain bin Zainal, Ph.D
Fakulti: Sains


Penyingkiran fotoelektrokimia pewarna telah dijalankan dengan sistem reaktor 3 elektrod di mana elektrod kerja adalah filem nipis TiO₂ 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
kepokatan awal pewarna, keupayaan elektrik dan elektrolit penyokong. Kesu ulangan penggunaan, sumber cahaya dan sistem penyinkiran juga telah diselidik.

Sistem fotoelektromangkinan bagi kedua-dua jenis elektrod filem nipis TiO$_2$ memberi penytingkiran pewarna CSB yang lebih baik daripada dalam sistem fotomangkinan. Elektrod TiO$_2$ pengoksida terma memberi kadar penyeringkiran yang lebih cepat berbanding dengan elektrod TiO$_2$ elektroenapan. Penyeringkiran 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.

ACKNOWLEDGEMENTS

I would like to take this opportunity to express my sincere gratitude and heartfelt thanks to my project supervisor, Professor Dr. Zulkarnain Zainal for his extraordinary patience, kindness, invaluable guidance, constructive criticisms, advice, continuous supervision and suggestion throughout the duration of the study. My appreciation also goes to my co-supervisor, Associate Professor Dr. Abdul Halim Abdullah for the advice and consistent support throughout the completion of this thesis. I wish to thanks to all my lab mates especially Chee Siong, Sook Keng and Sook Liang who help me a lot in my Master research.

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.

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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:

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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:
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>DEDICATION</td>
<td>ii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>ABSTRAK</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>vii</td>
</tr>
<tr>
<td>APPROVAL</td>
<td>viii</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>x</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xiii</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xiv</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS AND SYMBOLS</td>
<td>xxi</td>
</tr>
</tbody>
</table>

## CHAPTER

1. **INTRODUCTION**
   1.1 Objectives

2. **LITERATURE REVIEW**
   2.1 Theory of Semiconductor
   2.2 The Semiconductor-Electrolyte Interface
   2.3 Properties of Titanium Dioxide
      2.3.1 Structural Properties
      2.3.2 Optical Properties
   2.4 Titanium as Supporting Materials
   2.5 Preparation of Titanium Dioxide Thin Films
      2.5.1 Preparation of Titanium Dioxide by Thermal Oxidation
      2.5.2 Preparation of Titanium Dioxide by Electrodeposition
   2.6 Photodegradation Process on Titanium Dioxide
   2.7 Photoelectrocatalysis of TiO₂ Semiconductor
   2.8 Effect of Various Removal Conditions using TiO₂ Electrode
   2.9 Electrochemical Studies
      2.9.1 Voltammetry
      2.9.2 Chronoamperometry
   2.10 Properties of Dyes
   2.11 Kinetic Study

3. **METHODOLOGY**
   3.1 Preparation of Titanium Dioxide by Thermal Oxidation
   3.2 Preparation of Electrodeposition Bath
   3.3 Preparation of Titanium Dioxide by Electrodeposition
   3.4 Preparation of Dyes Solution
   3.5 Determination of Wavelength at Maximum Absorption (λ<sub>max</sub>) and Construction of Standard Calibration Curve of Dye
   3.6 Characterization of Titanium Dioxide Thin Films
      3.6.1 Field Emission Scanning Electron Microscopy (FESEM)
      3.6.2 X-Ray Diffractometry (XRD) Analysis
3.6.3 Diffuse Reflectance Study 33
3.6.4 Voltammetry Studies 34
3.7 Photoelectrocatalytic Removal of Chicago Sky Blue 6B 35
  3.7.1 Effect of Calcination Temperature 36
  3.7.2 Effect of Various Removal Method 36
  3.7.3 Effect of Applied Voltage 37
  3.7.4 Effect of Initial Concentration 37
  3.7.5 Effect of Supporting Electrolyte 37
  3.7.6 Effect of Different Light Source 37
  3.7.7 Effect of Repeated Usage 38
3.8 Photoelectrocatalytic removal of Methyl Orange and Methylene Blue 38

4 RESULTS AND DISCUSSION 39
  4.1 Preparation of Titanium Dioxide Thin Film Electrodes 39
  4.2 Surface Morphology of Titanium Dioxide Thin Film Electrodes 42
  4.3 EDX Analysis 46
  4.4 X-Ray Diffractometry Studies 49
  4.5 Optical Study 53
  4.6 Electrochemical Characteristic 55
  4.7 Effect of Calcination Temperature 58
  4.8 Effect of Various Removal Method 61
  4.9 Effect of Applied Voltage 68
    4.9.1 Kinetic Order of Photoelectrocatalytic Removal of CSB 72
  4.10 Effect of Initial Concentration 77
  4.11 Effect of Supporting Electrolyte 84
    4.11.1 Effect of Anions 84
    4.11.2 Effect of Cations 92
  4.12 Effect of Light Source 97
  4.13 Effect of Repeated Usage 103
  4.14 Photoelectrocatalytic Removal of Methyl Orange and Methylene Blue 108

5 CONCLUSION AND RECOMMENDATIONS 114
  5.10 Conclusion 114
  5.11 Recommendations 116

REFERENCES 119
APPENDICES 127
BIODATA OF THE STUDENT 138
# LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Crystallographic properties of rutile and anatase.</td>
<td>15</td>
</tr>
<tr>
<td>4.1</td>
<td>Atomic percent of Ti and O element in TiO₂ thin films.</td>
<td>49</td>
</tr>
<tr>
<td>4.2</td>
<td>Comparison of d (Å) values for TO-TiO₂ and ETiO₂C with standard JCPDS.</td>
<td>52</td>
</tr>
<tr>
<td>4.3</td>
<td>Percentage of rutile phase and crystallite size of each phase in TO-TiO₂ and ETiO₂C electrodes.</td>
<td>52</td>
</tr>
<tr>
<td>4.4</td>
<td>The Langmuir-Hishelwood first order kinetic parameters for the effect of applied potential in TO7-TiO₂ and ETIO₂C6 electrodes.</td>
<td>75</td>
</tr>
<tr>
<td>4.5</td>
<td>The Langmuir-Hishelwood first order kinetic parameters for the effect of initial dye concentration by TO7-TiO₂ and ETIO₂C6 electrodes.</td>
<td>79</td>
</tr>
<tr>
<td>4.6</td>
<td>The Langmuir-Hishelwood first order kinetic parameters for the repeated usage for TO7-TiO₂ and ETIO₂C6 electrodes.</td>
<td>107</td>
</tr>
</tbody>
</table>
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Change in the electronic structure of a semiconductor compound as the number N of monomeric units present increases from unity to clusters of more than 2000.</td>
<td>5</td>
</tr>
<tr>
<td>2.2</td>
<td>Band structure of a dielectric, semiconductor and metal. The shaded regions represent energy level filled with electrons.</td>
<td>6</td>
</tr>
<tr>
<td>2.3</td>
<td>Energy band of n-type (a) and p-type (b) semiconductor lattices.</td>
<td>7</td>
</tr>
<tr>
<td>2.4</td>
<td>Relative dispositions of various semiconductor band edge positions shown both on the vacuum scale and with respect to the SHE reference in aqueous medium of pH ~1.</td>
<td>8</td>
</tr>
<tr>
<td>2.5</td>
<td>Energy levels in a semiconductor (left-hand side) and a redox electrolyte (right-hand side) shown on a common vacuum reference scale. $\chi$ and $\varnothing$ are the semiconductor electron affinity and work function, respectively.</td>
<td>9</td>
</tr>
<tr>
<td>2.6</td>
<td>The formation of a junction between an n-type semiconductor and a solution containing a redox couple O/R (a) before contact in the dark, (b) after contact in the dark and electrostatic equilibration and (c) junction under irradiation.</td>
<td>10</td>
</tr>
<tr>
<td>2.7</td>
<td>A schematic representation of different types of PEC cells; (a) photovoltaic cell, (b) photoelectrolytic cell and (c) photocatalytic cell.</td>
<td>11</td>
</tr>
<tr>
<td>2.8</td>
<td>Typical correlations between electronic energy states in semiconductors and redox electrolytes.</td>
<td>12</td>
</tr>
<tr>
<td>2.9</td>
<td>Energy diagram for the semiconductor-electrolyte interface at equilibrium for different concentrations.</td>
<td>13</td>
</tr>
<tr>
<td>2.10</td>
<td>Structure of rutile TiO$_2$.</td>
<td>14</td>
</tr>
<tr>
<td>2.11</td>
<td>Structure of anatase TiO$_2$.</td>
<td>14</td>
</tr>
<tr>
<td>2.12</td>
<td>View of hydroxylation of the (001) surface of anatase TiO2 via dissociation of water on surface adsorption. Note the two distinct OH surface groups.</td>
<td>16</td>
</tr>
<tr>
<td>2.13</td>
<td>Direct and indirect energy band transition in semiconductor.</td>
<td>17</td>
</tr>
<tr>
<td>2.14</td>
<td>Illustration of the major processes occurring on a semiconductor particle following electronic excitation.</td>
<td>22</td>
</tr>
</tbody>
</table>
2.15 Energy scheme depicting a photoelectrochemical cell containing a photoanode and a metal counter electrode during the process of energy conversion.

2.16 Potential-time excitation signal in linear sweep voltammetry and cyclic voltammetry experiment.

2.17 Typical (a) linear sweep voltammetry and (b) cyclic voltammogram for a reversible single electron transfer reaction.

2.18 Current versus time response in chronoamperometric experiment.

3.1 The three electrode system for electrodeposition process.

3.2 Experiment set up for the photoelectrochemical cell.

4.1 Cyclic voltammogram of Ti plate in 50 mL of 0.02 M hydrolyzed TiCl₄, 0.03 M H₂O₂ and 0.10 M KNO₃ solution.

4.2 Current-time curve for electrodeposition of peroxotitanium hydrate onto Ti plate in 50 mL of 0.02 M hydrolyzed TiCl₄, 0.03 M H₂O₂ and 0.10 M KNO₃ solution. Inset shows the amplified image of the curve for the first 40 s.

4.3 FESEM micrographs of Ti plate with 15000 x magnification.

4.4 FESEM micrographs of TO7-TiO₂ with (a) 15000 x magnification and (b)100000 x magnification.

4.5 FESEM micrographs of ETiOP thin film with 15000 x magnification.

4.6 FESEM micrographs of ETiO₂C6 with (a) 15000 x magnification and (b) 100000 x magnification.

4.7 EDX analysis for (a) Ti plate, (b) TO7-TiO₂, (c) ETiOP and (d) ETiO₂C6.

4.8 XRD patterns of TO-TiO₂ thin film electrodes at various calcination temperatures.

4.9 XRD patterns of ETiO₂C thin film electrodes at various calcination temperatures.

4.10 UV-Vis absorbance spectra of TO-TiO₂ thin film electrodes at various calcination temperatures.

4.11 UV-Vis absorbance spectra of ETiO₂C thin film electrodes at various calcination temperatures.
4.12 Current-potential curves for TO-TiO$_2$ thin film electrodes at various calcination temperatures in CSB under illumination. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.13 Current-potential curves for ETiO$_2$C thin film electrodes at various calcination temperatures in CSB under illumination. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.14 Photocurrent-potential curve obtained for TO$_7$-TiO$_2$ and ETiO$_2$C$_6$ thin film electrodes in CSB under intermittent illumination. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.15 Effect of photoelectrocatalytic degradation of CSB by TO-TiO$_2$ at various calcination temperatures under illumination of light. [Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.16 Effect of photoelectrocatalytic degradation of CSB by ETiO$_2$C at various calcination temperatures under illumination of light. [Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.17 Effect of different removal method in CSB by TO$_7$-TiO$_2$ electrode. [Conditions: 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.18 Effect of different removal method in CSB by ETiO$_2$C$_6$ electrode. [Conditions: 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.19 Removal percentage of 10 ppm CSB in various method by TO$_7$-TiO$_2$ and ETiO$_2$C$_6$ electrodes.

4.20 Effect of applied potential in CSB removal by TO$_7$-TiO$_2$ electrode under illumination of light. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.21 Graph of the photocurrent versus time at various applied potentials by TO$_7$-TiO$_2$ electrode in CSB under illumination of light. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.22 Effect of applied potential in CSB removal by ETiO$_2$C$_6$ electrode under illumination of light. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]
4.23 Graph of the photocurrent versus time at various applied potentials by ETiO₂C₆ electrode in CSB under illumination of light.
[Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.24 Graph ln C/C₀ versus time for the effect of applied potential by TO7-TiO₂ electrode in CSB under illumination of light.

4.25 Graph ln C/C₀ versus time for the effect of applied potential by ETiO₂C₆ electrode in CSB under illumination of light.

4.26 The first order kinetic constant versus applied potential in TO7-TiO₂ and ETiO₂C₆.

4.27 Effect of initial concentration on CSB removal by TO7-TiO₂ electrode under illumination of light.
[Conditions: 300 W halogen lamp, 1 V and 124 mL of CSB containing 0.1 M KCl]

4.28 Effect of initial concentration on CSB removal by ETiO₂C₆ electrode under illumination of light.
[Conditions: 300 W halogen lamp, 1 V and 124 mL of CSB containing 0.1 M KCl]

4.29 UV-Vis adsorption spectra of CSB at various concentrations with 0.1 M KCl.

4.30 Graph of photocurrent versus time in various initial concentrations of CSB by TO7-TiO₂ electrode under illumination of light.
[Conditions: 300 W halogen lamp, 1 V and 124 mL of CSB containing 0.1 M KCl]

4.31 Graph of photocurrent versus time in various initial concentrations of CSB by ETiO₂C₆ electrode under illumination of light.
[Conditions: 300 W halogen lamp, 1 V and 124 mL of CSB containing 0.1 M KCl]

4.32 Amount of CSB removal at different initial concentrations by TO7-TiO₂ electrode.

4.33 Amount of CSB removal at different initial concentrations by ETiO₂C₆ electrode.

4.34 Effect of anion in CSB removal by TO7-TiO₂ electrode under illumination of light.
[Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]
4.35 Effect of anion in CSB removal by ETiO$_2$C$_6$ electrode under illumination of light.  
[Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.36 Removal percentage of CSB at different anions by TO7-TiO$_2$ and ETiO$_2$C$_6$ electrodes.

4.37 UV-Vis adsorption spectra of 10 ppm CSB containing 0.1 M of supporting electrolyte.

4.38 Current-potential curves for TO7-TiO$_2$ electrodes in CSB with various anions under illumination of light.  
[Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.39 Current-potential curves for ETiO$_2$C$_6$ electrodes in CSB with various anions under illumination of light.  
[Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.40 Effect of cation in CSB removal by TO7-TiO$_2$ electrode under illumination of light.  
[Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.41 Effect of cation in CSB removal by ETiO$_2$C$_6$ electrode under illumination of light.  
[Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.42 Removal percentage of CSB at different cations by TO7-TiO$_2$ and ETiO$_2$C$_6$ electrodes.

4.43 Current-potential curves for TO7-TiO$_2$ electrodes in CSB with various cations under illumination of light.  
[Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.44 Current-potential curves for ETiO$_2$C$_6$ electrodes in CSB with various cations under illumination of light.  
[Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M supporting electrolyte]

4.45 Effect of different light sources in CSB removal by TO7-TiO$_2$ electrode.  
[Conditions: 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.46 Effect of different light sources in CSB removal by ETiO$_2$C$_6$ electrode.  
[Conditions: 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]
4.47 Removal percentage of 10 ppm CSB under different illumination light sources by TO7-TiO2 and ETiO2C6 electrodes.

4.48 Graph photocurrent versus time of CSB removal under various illumination light sources by TO7-TiO2 electrodes. [Conditions: 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.49 Graph photocurrent versus time of CSB removal under various illumination light sources by ETiO2C6 electrodes. [Conditions: 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.50 Repeated usage of TO7-TIO2 electrode in CSB removal under illumination of light. [Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.51 Repeated usage of ETIO2C6 electrode in CSB removal under illumination of light. [Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.52 Graph ln C/C0 versus time for the repeated usage of TO7-TiO2 electrode in CSB removal.

4.53 Graph ln C/C0 versus time for repeated usage of ETiO2C6 electrode in CSB removal.

4.54 A plot of kinetic constant versus number of times for TO7-TiO2 and ETiO2C6 electrodes used.

4.55 Removal of various dyes by TO7-TiO2 and ETiO2C6 electrodes respectively under illumination of light. [Conditions: 300 W halogen lamp, 1 V and 124 mL of 10 ppm dye containing 0.1 M KCl]

4.56 Current-potential curves for TO7-TiO2 and ETiO2C6 electrodes in various dyes removal under illumination of light. [Conditions: 300 W halogen lamp and 124 mL of 10 ppm CSB containing 0.1 M KCl]

4.57 UV-Vis absorption spectra of the removal of 10 ppm CSB containing 0.1 M KCl by TO7-TiO2 at different time intervals.

4.58 UV-Vis absorption spectra of the removal of 10 ppm CSB containing 0.1 M KCl by ETiO2C6 at different time intervals.

4.59 UV-Vis absorption spectra of the removal of 10 ppm MO containing 0.1 M KCl by TO7-TiO2 at different time intervals.
4.60 UV-Vis absorption spectra of the removal of 10 ppm MO containing 0.1 M KCl by ETiO₂C₆ at different time intervals.

4.61 UV-Vis absorption spectra of the removal of 10 ppm MB containing 0.1 M KCl by TO7-TiO₂ at different time intervals.

4.62 UV-Vis absorption spectra of the removal of 10 ppm MB containing 0.1 M KCl by ETiO₂C₆ at different time intervals.
# LIST OF ABBREVIATIONS AND SYMBOLS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSB</td>
<td>Chicago Sky Blue 6B</td>
</tr>
<tr>
<td>CV</td>
<td>Cyclic Voltammetry</td>
</tr>
<tr>
<td>EC</td>
<td>Conduction band</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy Dispersion X-ray</td>
</tr>
<tr>
<td>EF</td>
<td>Fermi energy level</td>
</tr>
<tr>
<td>$E_g$</td>
<td>Band gap energy</td>
</tr>
<tr>
<td>$E_V$</td>
<td>Valence band</td>
</tr>
<tr>
<td>ETiO$_2$C</td>
<td>Titanium Dioxide Prepared by Electrodeposition</td>
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<tr>
<td>ETiO$_2$C4</td>
<td>Electrodeposited Titanium Dioxide Calcined at 400°C</td>
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<td>ETiO$_2$C5</td>
<td>Electrodeposited Titanium Dioxide Calcined at 500°C</td>
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<tr>
<td>ETiO$_2$C6</td>
<td>Electrodeposited Titanium Dioxide Calcined at 600°C</td>
</tr>
<tr>
<td>ETiO$_2$C7</td>
<td>Electrodeposited Titanium Dioxide Calcined at 700°C</td>
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<td>ETiOP</td>
<td>Electrodeposition of Peroxotitanium Hydrate</td>
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<tr>
<td>FESEM</td>
<td>Field Emission Scanning Electron Microscopy</td>
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<tr>
<td>HOMO</td>
<td>Highest Occupied Molecular Orbital</td>
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<tr>
<td>JCPDS</td>
<td>Joint Committee of Powder Diffraction Standard</td>
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<tr>
<td>LUMO</td>
<td>Lowest Unoccupied Molecular Orbital</td>
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<tr>
<td>LSV</td>
<td>Linear Sweep Voltammetry</td>
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<tr>
<td>LSPV</td>
<td>Linear Sweep Photovoltammetry</td>
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<tr>
<td>MB</td>
<td>Methylene Blue</td>
</tr>
<tr>
<td>MO</td>
<td>Methyl Orange</td>
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<td>TiO$_2$</td>
<td>Titanium Dioxide</td>
</tr>
<tr>
<td>TO-TiO$_2$</td>
<td>Titanium Dioxide Prepared by Thermal Oxidation</td>
</tr>
<tr>
<td>TO7-TiO$_2$</td>
<td>Titanium Dioxide Prepared by Thermal Oxidation at 700°C</td>
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XRD X-ray Diffractometer