

UNIVERSITI TEKNOLOGI MARA

**MODIFICATION AND
CHARACTERIZATION OF
IMMOBILIZED TiO₂/PEG DSAT FOR
PHOTODEGRADATION OF MB DYE**

NUR RAIHAN BTE ZAHARUDIN

Thesis submitted in fulfilment of
the requirements for the degree of
Master of Science
(Applied Chemistry)

Faculty of Applied Sciences

JUNE 2018

ABSTRACT

A photocatalysis mediated with immobilized titanium dioxide (TiO_2) with polymer has been considered as an interesting subject to degrade organic pollutants. This research is conducted to modify the immobilized TiO_2 using double sided adhesive tape (DSAT) to improve its adhesiveness between TiO_2 and glass as well as to measure its photoactivity and to characterize their properties. Immobilized TiO_2 with polyethylene glycol (PEG) is prepared via brush coating method using Degussa P-25 titania (particle size: 15-20 nm). The films were deposited on DSAT onto glass which acted as a support binder and annealed by a thermal treatment at 100 °C, known as TiO_2 /PEG DSAT. The photocatalytic activity of the sample was measured by photodegradation of methylene blue (MB) dye as model pollutant. It was found that washed TiO_2 /PEG DSAT film has the highest photoactivity at 0.10% of PEG on 0.30 g of photocatalyst loading named as TP2 sample. From the experimental parameters conducted, it was found that optimum aeration, pH and initial concentration of MB were found at 75 mL/min, pH 11 and 12 ppm respectively. Based on a pseudo first order kinetics, the study showed that the photoactivity of the washed TP2 is 1.8 times faster than TiO_2 suspension. The washed TP2 film turned out smooth, crack-free and doesn't leached out easily due to sufficient amount of TiO_2 to PEG ratio on DSAT. The washing process has influenced the photoactivity of the TP2 sample by 1.6 times faster than unwashed TP2 sample. Results showed that the enlarged number of surface area was 43% higher in the washed TP2 film, where the BET surface area of the photocatalysts: washed TP2, 88 m^2/g ; TiO_2 powder, 50 m^2/g). SEM analysis showed that the washed TP2 has larger pore depth than immobilized TiO_2 only. The potentiality of porosity (i.e. larger mesopores) as exhibited by the BET and SEM analysis accounts for numerous active sites that are often associated with high photodegradation rates (pore volume = 0.64 cm^3/g and pore size diameter = 1.71 nm). XRD analysis demonstrated the anatase and rutile phases of the immobilized samples. Furthermore, the XPS spectrum proved that the existence of C=O at 288.7 eV of binding energy in washed TP2 films, where it was not detected in unwashed sample. Since the C=O bond was previously reported as sensitizer for photocatalyst, this has led to a significant photoresponse under normal and visible light irradiation. Additionally, the presence of C=O in the washed TP2 is detected owing to the strong absorption by carbonyl group at 1705 cm^{-1} from the FT-IR spectrum. Through the washing process, the PEG becomes significantly oxidized and the C=O bond is generated. The highest photoluminescence intensity found in the washed TP2 film helped to prove that the C=O species is crucial to enhance the photocatalysis where it acted as an electron injector (sensitizer). Through the application of DSAT, it was found that the films' reusable capacity is up to 30 cycles. Ultimately, the washed immobilized TP2 film is efficient in obtaining a favourable photocatalytic activity performance

ACKNOWLEDGEMENT

Firstly, I wish to thank Allah for giving me the opportunity to embark on my research study and for completing this journey successfully. My gratitude and thanks go to my supervisor Dr Wan Izhan Nawawi bin Wan Ismail, and co-supervisor, Dr Zuliahani bt Ahmad for guiding me in doing my task and for being such a great friend. Thank you for sharing the time, dedications, knowledge, support, patience and invaluable ideas in assisting me with this project. Deepest gratitudes are also due to the photocatalyst team members and beloved friends, Saifulddin, Ain, Faezah, Hanani and Amani without whose knowledge, motivation and assistance this study would not have been enjoyable and meaningful. I would also like to express thanks to all FBERG members, PJI staffs, UiTM staffs, SIRIM, USM, UMT and UiTM Perlis Postgraduate Society for helping me with my studies, especially PM Dr Azlan, Dr Razif, Dr Mohd Ali, Ramlah, Syahida, Pn Zurita, Pn Rohana, Dr Shukor, Miss Nazifah, Dr Mad, Prof Khudzir and Miss Nasulhah.

Deepest love and thanks are also due to my siblings, home girls, colleagues, lab assistants, lecturers, juniors and friends for all of your contributions, efforts and prayers. May Allah increase all of you in your affairs for the better, amen. Finally, this thesis is dedicated to the heroes in my life, father Zaharudin Abdul Rahman and mother . for their understanding and endless love through the duration of my studies. I want to be all you think about. Everything and anything you dream about. This piece of victory is dedicated to both of you. Alhamdulillah.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	x
LIST OF FIGURES	xi
LIST OF SYMBOLS	xiv
LIST OF ABBREVIATIONS	xv
CHAPTER ONE: INTRODUCTION	1
1.1 Water Pollution	1
1.2 TiO ₂ as Photocatalyst	2
1.3 Development of Immobilized TiO ₂	3
1.4 Immobilized TiO ₂ with Water Base Polymer	7
1.5 Polyethylene Glycol	8
1.6 Double Sided Adhesive Tape	9
1.7 Cationic Methylene Blue Dye	10
1.8 Problem Statement	11
1.9 Research Objectives	13
1.10 Scope and Limitation of Study	13
1.11 Significance of Study	14
CHAPTER TWO: LITERATURE REVIEW	15
2.1 Titanium Dioxide Photocatalyst	15
2.2 Mechanism of TiO ₂ Photocatalyst	17
2.3 Potential of Polymer as Sensitizer	20
2.4 Immobilized TiO ₂ with Polyethylene Glycol (PEG)	23

CHAPTER THREE: METHODOLOGY	32
3.1 Reagents and Chemicals	32
3.2 Conceptual Framework	33
3.3 Instruments and Equipments	34
3.4 Preparations of MB Standard Solution	34
3.5 Preparation of Immobilized TiO ₂ with PEG and DSAT	34
3.6 Photocatalytic Degradation Test	36
3.7 Determination of Kinetic Equation	37
3.8 Control Test and Adsorption Study	38
3.9 The Optimization Study of Immobilized TiO ₂ /PEG DSAT	38
3.9.1 Effect of PEG Ratio	38
3.9.2 Effect of Photocatalyst Loading	39
3.10 The Effect of Operational Parameters on The Photocatalytic Efficiency of Immobilized TiO ₂ /PEG DSAT	40
3.10.1 Effect of Coating Method	40
3.10.2 Effect of Initial Dye Concentration	41
3.10.3 Effect of Aeration Flow Rate	41
3.10.4 Effect of Initial pH and Determination of pH of Zero Charge	41
3.10.5 Effect of Washing	42
3.10.6 Effect of Reusability	42
3.10.7 Effect of Adhesiveness	43
3.11 Characterization Test of Immobilized TiO ₂ /PEG DSAT	43
3.11.1 X-Ray Diffraction Analysis	43
3.11.2 SEM Analysis	43
3.11.3 BET Surface Area Analysis	44
3.11.4 FT-IR Analysis	44
3.11.5 XPS Analysis	44
3.11.6 UV-Vis DRS Analysis	45
3.11.7 Photoluminescence Analysis	45
3.12 Mineralization Study	45
3.12.1 Preparation of Chemical Oxygen Demand Reagent	45
3.12.2 Chemical Oxygen Demand Test	46