

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS AND
CHARACTERIZATION OF VISIBLE
LIGHT ACTIVE PHOTOCATALYSTS
BY SOLGEL METHOD**

ELVANA NERISSA BT MOHAMAD

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science


Faculty of Chemical Engineering

August 2015

AUTHORS DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledge as referenced work. This thesis has not been submitted to other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student : Elvana Nerissa bt Mohamad
Student I.D No : 2012289546
Programme : Master of Science (Chemical Engineering) by Research
Faculty : Faculty of Chemical Engineering
Thesis Title : Synthesis and Characterization of Visible Light Active
Photocatalysts by Solgel Method
Signature of student : .....
Date : August 2015

ABSTRACT

In the present study, synthesis and characterization of visible-light active photocatalysts needed for the photodegradation of organic pollutants was presented. Non metals like N, C and F were doped with titanium dioxide by solgel method. The effects of dopant concentration, calcination temperature, dopant element and titanium precursor on the synthesis were studied systematically. The photocatalytic activities of the surface modified titanium dioxide were tested using 10 ppm methylene blue solution. The properties were characterized by using XRD, UV-vis DRS, FESEM, BET and XPS analysis. Dopant concentration, calcination temperature, dopant element and titanium precursors gave significant effect on the properties of the photocatalysts and photocatalytic activity. Dopant concentration of 0.75% and calcination temperature of 600°C yielded the highest photocatalytic degradation efficiency as high as 91.3%. Each dopant element showed different crystal structure and properties of the photocatalysts. A combination of two elements (N-C codoped TiO₂) showed the best performance. Titanium tetraisopropoxide was found to be the best titanium precursors. XRD analysis confirmed the anatase phase of TiO₂. Activation of photocatalysts under visible light was confirmed by a reduction in band gap energy as observed from UV-Vis DRS. The surface morphology of the photocatalyst samples was analysed by FESEM. The particles formed were in almost spherical shape with a specific surface area ranging from 17.54 to 23.65 m²/g for 0.75% N-F-TiO₂ (600) and 0.75% F-TiO₂ (600) respectively. Details of chemical and electronic states of elements were shown by XPS analysis. The N-C codoped TiO₂ photocatalyst was identified as an effective photocatalyst suitable for the photodegradation of organic pollutants.

ACKNOWLEDGEMENT

First of all, I would like to thank to Allah S.W.T for His approval and blessing that made all this happened and came true. Nothing can be done without His permission. Alhamdulillah.

Special appreciation goes to my supervisor, Dr Jagannathan Krishnan for his guidance, patience and support. I am truly honoured and humble to have him as my supervisor because of his knowledge and expertise. This project cannot be done successfully without his guidance and full commitment given by him. Not forgotten, my appreciation to my co-supervisor, Dr Abdul Hadi for his knowledge and ideas.

I would like to express my appreciation to my parents and husband for the prayers, support and encouragement to complete this challenging journey successfully.

Finally, to all technicians, lab assistance, friends and course mates, thank you for the encouragement and kindness. Your kindness means a lot to me.

CHAPTER ONE

INTRODUCTION

This chapter gives a brief description of the introduction to this study. It contains background of study, problem statements, research objectives as well as scope and limitation. Background of studies covered the introduction about the photocatalysis, TiO₂ photocatalysts, TiO₂ modification technique and method to synthesize the photocatalyst.

1.1 BACKGROUND OF STUDY

As the industrial sectors are rapidly growing in Malaysia, many industrial zones have been approved by the government to be set up. Entailing this, thousands of trees are cut down daily to accommodate the building of large industrial factories. As a result, the oxygen supply for the ecosystem is decreasing. Moreover, these industrial areas are also emitting poisonous gases from various production processes. Additionally, the nearby rivers and their residents have also become polluted due to the industrial wastes that have been continuously discharged into the rivers. Most of the pollutants emitted are organic pollutants which are complex and highly stable structure. These pollutants can cause fatal harm to human health and the surrounding environment. Some of them are carcinogens and contain highly toxic compounds. Thus, these harmful pollutants need to be chemically treated before they can be safely released into the environment.

There are several methods reported on the removal of these pollutants. Among the available methods, photocatalysis is the most practicable method. Photocatalysis is a light activated reaction used to initiate the substance called catalyst, which modifies the rate of chemical reaction without being altered at the end of the process. As discovered by Dr Fujishima and Honda in 1972, the titanium metal could break the water molecule into oxygen and hydrogen gas after irradiated by UV light (Chen & Mao, 2007). Photocatalytic reaction was used to accelerate the cleaning and purifying process naturally using light as the energy source.