THE EFFECT OF NI-TIO_2 COATING ON DIFFERENT SUBSTRATES

MUHAMMAD FAIQ AQIL BIN MOHD FAIZAIRI

B051510235

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2019

THE EFFECT OF NI-TIO_2 COATING ON DIFFERENT SUBSTRATES

Submitted in accordance with the requirement of the University Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Hons.)

By

MUHAMMAD FAIQ AQIL BIN MOHD FAIZAIRI

B051510235

961108-91-5003

FACULTY OF MANUFACTURING ENGINEERING

2019

DECLARATION

I hereby, declared this report entitled "The effect of Ni-TiO_2 coating on different substrates" is the results of my own research except as cited in reference.

Signature : ……

Author's Name : MUHAMMAD FAIQ AQIL BIN MOHD FAIZAIRI

Date : 31 May 2019

APPROVAL

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

……

(Professor Madya Dr. Jariah binti Mohamad Juoi) – Signature & Stamp

ABSTRACT

Common ceramic tiles do not have any self-cleaning property which can reduce the growth of bacteria that are harmful to humans. After several years, ceramic tiles with self-cleaning property are gaining ranks in the market of ceramic tiles. This was possible after years of research and development to improve the existing ceramic tiles. The improve self-cleaning ceramic tiles can be used in many areas especially areas that require high level of hygiene such as in food industries, pharmaceutical industries and hospitals. The aim of this study is to investigate the effect of Ni addition on the microstructure of TiO_2 coating on different ceramic substrates and to determine the photocatalytic properties of Ni-TiO_2 coating. Firstly, TiO2 sol is made by using sol – gel dip coat method. Nickel was added to the pure TiO_2 sol creating a Ni-TiO_2 sol. Glass, unglazed and glazed ceramic substrates were used as the substrates. The dip – coat process was done in five dipping time. The surface morphology and thickness of coated substrates are determined by using the SEM analysis while the phase formation is determined by using XRD analysis followed with the photocatalytic testing. It was observe that cracks was produced on the unglazed ceramic tile, meanwhile on glass and glazed ceramic tile, even coating was observed. Thickness of coating was vary depend on the type of substrates as unglazed ceramic tile had thicker coating followed by glazed and glass. In XRD analysis, anatase presence was higher compared to rutile for all the substrates that would lead to good photocatalytic properties. Result from photocatalytic testing indicate that unglazed ceramic tile had the better rate of methylene blue degradation among other substrates. In conclusion, the addition of Nickel into pure TiO_2 sol had a different effect on the surface morphology, thickess, phase formation and photocatalytic property of the coating depending on the type of substrates.

ABSTRAK

Jubin seramik yang biasa tidak mempunyai sebarang sifat pembersihan sendiri yang dapat mengurangkan pertumbuhan bakteria yang mungkin akan membahayakan manusia. Setelah beberapa tahun, jubin seramik yang mempunyai sifat pembersihan sendiri sedang menaiki tangga dalam pasaran dunia. Semua ini berhasil setelah beberapa tahun bergiat dalam pembangunan dan penaiktarafan jubin seramik yang sedia ada. Jubin ini boleh digunakan dalam pelbagai sektor terutama sektor yang mengutamakan kebersihan seperti sektor makanan, sektor perubatan serta hospital. Tujuan kajian ini adalah untuk mengenal pasti efek tambahan Ni keatas mikrostruktur salutan TiO_2 di atas substrat yang berbeza dan menentukan sifat *photocatalytic* salutan Ni-TiO_2. Pertama, sol TiO_2 dibuat daripada teknik celupan sol–gel. Ni-TiO_2 dihasilkan apabila Nikel diletakkan pada sol TiO_2 yang tulen. Kaca, jubin seramik *glazed* dan *unglazed* merupakan substrat yang digunakan. Proses celupan dilakukan pada kadar lima kali celupan. Morphology permukaan dan ketebalan ditentukan menggunakan analisis SEM manakala pembentukan fasa ditentukan menggunakan analisis XRD diikuti dengan ujian *photocatalytic.* Daripada pemerhatian, keretakan terhasil pada jubin seramik *unglazed* manakala pada jubin *glazed* serta kaca, salutan yang rata dihasilkan. Ketebalan salutan berbeza bergantung kepada jenis substrat. Jubin seramik *unglazed* mempunyai ketebalan salutan yang tebal diikuti dengan *glazed* dan kaca. Analisis XRD menunjukkan kewujudan anatase lebih tinggi berbanding rutile untuk kesemua substrat. Keputusan ujian *photocatalytic* menunjukkan jubin seramik *unglazed* mempunyai kadar degradasi *methylene blue* yang baik berbanding substrat yang lain. Konklusinya, penambahan Nikel kedalam TiO_2 tulen mempunyai efek yang berbeza dari segi morfologi permukaan, ketebalan, pembentukan fasa dan sifat photocatalytic bergantung pada jenis substrat.

DEDICATION

Only

My beloved father, Mohd Faizairi Mohd Nor

My beautiful mother, Nor'Azura Ahmad

My lovely siblings

For giving me moral support, money, cooperation,

Encouragement and understanding

Thank you very much

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 year project successfully without any difficulty.

My respected supervisor Associate Professor Dr. Jariah binti Mohamad Juoi. Her kindness, unwavering patience and mentorship guided me through the process, her easily understood explanations and open mind allowed me to grow and learn in such a way that I am now a better researcher. I would like to thank for her time that she had given me to help in completing this project.

Not to forget, I would like to give special thanks to my friends who game me much motivation and cooperation in completing this project. They had given me their critical suggestion and comments throughout my research. Thanks for the great partnership and let us succeed together.

Finally, I would like to thank everybody who was important to this FYP report, as well as expressing my apology that I could not mention personally each one of you.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AFM	-	Atomic force microscope
ASTM	-	American society for testing and material
SEM	-	Scanning electron microscope
SOP	-	Standard operation procedure
UV	-	Ultraviolet
MB	-	Methylene blue
RB	-	Rhodamine B
ART	-	Anatase rutile transformation
TEM	-	Transmission electron microscope
TTiP	-	Titanium tetraisopropoxide
EDX	-	Energy dispersive X-ray
GA-XRD	-	Glancing angle X-ray diffraction
ISO	-	International organization for standardization

LIST OF SYMBOLS

cm	-	Centimeter
m	-	Meter
%	-	Percent
g/cm^3	-	Gram per centimeter cube
wt. %	-	Weight percentage
mm	-	Millimeter
^oc	-	Degree Celsius
K	-	Kelvin
nm	-	Nanometer
kg	-	kilogram
g	-	gram
mm/min	-	Millimeter per minute
kN	-	Kilo newton
Ag	-	Silver
Ni	-	Nickel
$^oc/min$	-	Degree Celsius per minute

CHAPTER 1

INTRODUCTION

1.1 Background Study

In this world, it is vital if our environment is free from the growth of microbial that can be harmful to human being. The practices to keep all the microorganisms and germs are surely hard but there are ways and technologies that may help to reduce the growth. Organisms and germs are an ordinary risk in our daily lives, and the control of microbial development and contaminants is a basic advance towards a more beneficial condition.

Classic applications of ceramic tile have included wet zones and areas requiring large amounts of sanitation, such as baths and kitchens. These areas sort of became the reason for growing microscopic gardens because of obvious reasons, for example, high moisture and increased surface contamination from sustenance and human contact (Ariss, 2009).

In construction of buildings, ceramic tile was also one of the widely used materials. Environmentally friendly construction was highly on demand. The law enactment highlighted strict environmental requirements rise in the functional tiles. To ensure a healthy environment, the normal tiles can be change into the antibacterial tiles where we can control the growth of these harmful microorganisms. These are important to many organizations and industries, such

as healthcare, food and drink, water treatment and military industries to ensure a healthier environment(Hasmaliza et al., 2016).

Transparent titanium dioxide (TiO_2) photocatalyst thin film have been used to keep surfaces clean by utilizing solar energy.(Alzamani et al., 2013)claimed that TiO_2 thin films possesses two unique tasks. The first is the photocatalytic function, in which there is a high recombining ratio of photoinduced electron-holes in the film that move to the surface and interact with daylight to breakdown the organic dirt. The second is the hydrophilic capability which implies that instead of forming droplets, water hits the TiO_2 coated surface and spreads evenly, running off and bringing the loosened dirt with it.

The methods used for the synthesis of TiO_2 include alkali precipitation, thermal decomposition, sol-gel and many other routes. Among these, the sol-gel route remains one of the most versatile to prepare metal oxide films even at low temperatures, due to the material's control during preparation, good homogeneity, low processing cost, and the possibility of preparing powder or thin films(Alzamani et al., 2013; Andronic et al.,2013). Thin films are obtained using organic and inorganic precursors and the deposition on the substrate can be further done by dipping, spraying, spin coating or doctor blade techniques.

However, when the TiO_2 catalyst is immobilized onto a substrate, the photocatalytic activity of the TiO_2 decreases. According to Lopez et al., 2013, the nature of the substrate has been shown to play a significant role in the preparation of TiO_2 films. Substrates can control the microstructure of TiO_2 films and henceforth the effect on the photocatalytic action. There have been several studies on the preparation of TiO_2 film on a variety of substrates and ceramic surfaces is not an exception. When TiO_2 film is successfully deposited on glass substrates with less to no micro-cracks at all, the deposition of the film onto ceramic substrates creates large cracks and peeling off the film (Lopez et al., 2013). Therefore, this research is aimed to improve the microstructure of TiO_2 coating onto ceramic substrate hence its adhesion performance as well.

1.2 Problem Statement

Based on previous studies titanium dioxide film can hold better on a rougher surface substrates(Anastasescu et al., 2014; Lopez et al., 2013). Thus, when TiO_2 is coated by dip-coating technique on glazed ceramic tile with a smooth surface, some of the coating will drip from the tiles when remove from the solution. This is due to the smooth surface of the tile which causes little TiO_2 colloid to retain on the substrate before even putting in into the furnace for calcination. The low adhesion of TiO_2 film on ceramic substrates especially on glaze surface tiles is because the smooth surface of the ceramic tiles. This will also result in lower film thickness(Lopez et al., 2013).

However, when TiO_2 is dip-coated on a rougher surface, increasing in the number of dipping times, will increase the thickness of the coating. This is also same when increasing in deposition duration. The results are that the cracks are getting severe from three dipping times to five dipping times(Musa et al., 2017). This could be related to the thicker coating formation observed as well as the differences in linear thermal expansion between the coating and the substrate (Musa et al., 2017).Therefore, a study needs to be done to improve the film's microstructure on ceramic tile to reduce, or even better, eliminate cracks and peeling off the film from the substrate. A way to improve this problem is to modify the TiO_2 coating by adding additives to act as a binder to bind the coating with the substrate.

Barmeh et al.,(2018) use Ni-doped TiO_2 coating to study the effect of Ni to the TiO_2 coating as the author claims that ceramic tiles coated with Ni-doped TiO_2 have been rarely studied, as well as the photocatalytic and self-cleaning properties of them. The coating was studied on a glazed ceramic tile. The microstructure characterization was done on glazed ceramic tile. However, test on unglazed ceramic tiles was not done. Thus, further study will be done to identify the microstructure of TiO_2 coating with addition of Ni on unglazed and glazed ceramic tiles.

It is understood that the Ni dopant can inhibit Anatase Rutile transformation (ART), which is due to the introduction of Ni^{2+} into the TiO_2 lattice as interstitially (Choi et al., 2007; Vargas et al., 1999). Therefore, 4 wt% of Ni-doped TiO_2 can inhibit the Anatase Rutile transformation.

The study also shows that Ni-doped TiO_2 coating can reduce the band gap. This is shown when the Ni-doped TiO_2 coating have a low band gap that is 2.48 eV. Meanwhile, the undoped TiO_2 coating have a high band gap that is 3.06 eV. This indicates that the TiO_2 coating with the doped Ni can have its photocatalytic property perform under visible light. From the photocatalytic test, with a 55.5% of photocatalytic degradation of Ni-TiO_2 on glazed ceramic tile, this indicates that this tile can be used in indoor environment. However, the photocatalytic property was not tested on unglazed ceramic tiles. Further research will be done to evaluate the photocatalytic property of Ni-TiO_2 coating on glazed and unglazed ceramic tile.

For the adhesion properties, the details explanation about Ni microstructure presence in TiO_2 and adhere to ceramic substrate are still lack of explanation. The only explanation is that the Ni-doped TiO_2 coating shows a form of is uniform thin film and have no cracks while undoped TiO_2 coating shows vice versa (Barmeh et al., 2018). The journal does not state whether the coating will produce cracks and peel off during handling. This indicates that the adhesion properties of Ni-doped TiO_2 coating was not further study in their work. Thus, further studies on adhesion properties such as testing the Ni-doped TiO_2 coating on different substrates such as glass, glaze ceramic tile and unglazed ceramic tile and number of dipping in Ni-doped TiO_2 is necessary.

1.3 Objectives

The objectives of this research are:

i. To analyze the effect of Ni addition on the microstructure of TiO_2 coating on different ceramic substrate.
ii. To determine the photocatalytic properties of Ni-TiO_2 coating.

1.4 Scope of Study

The study focused on using nickel (Ni) as additive in TiO_2 coating on ceramic substrate. The substrate used for the coating were glass, glazed and unglazed ceramic tile. The coating was dip-coated on glass substrate to investigate the effect of the additive on the improvement of the TiO_2 coating as benchmark. Optimized result then be used to deposit the coating on glazed and unglazed ceramic tile. Characterization analysis was conducted to analyze the effect of Ni addition on the microstructure of TiO_2 coating on the different ceramic substrate. Photocatalytic properties was also tested to identify the difference between Ni-TiO_2 coating and TiO_2 coating.

1.5 Significant of Study

The significant of the study is to identify the addition of Ni as additive on the ceramic tiles. This study purposely to evaluate the improvement of the microstructure, the photocatalytic property which is for better antimicrobial purpose .

Characterization analysis, photocatalytic test and adhesion test were done. This study helped to upgrade the available photocatalytic tiles in the market, so new and improve photocatalytic tile can be manufactured. By study the effect of additives to the pure TiO_2 coating it helped the production of antimicrobial tiles that can be manufactured and used for building residentials housing or industrial area especially industry in the pharmaceutical, foods and water treatment fields which required a safety and healthy environment.

1.6 Organizational of report

Chapter 1 (introduction) discuss on the background of the project. The problem statement and objectives of this project.

Chapter 2 covers literature review on previous studies conducted by researchers regarding the characteristic and performance of TiO_2 coating focusing on the effect of additives on the microstructure, photocatalytic performance and adhesion properties.Thus, the literature review is divided into 2 parts. Mainly, part 1 review study on pure TiO_2 coating and focusing on the microstructure, photocatalytic property, antimicrobial property and the adhesion property. Then, part 2 is dedicated to review on the effect of additives to pure TiO_2 coating on the microstructure, photocatalytic property and adhesion property.

Chapter 3 describing on the methodology of the project that involved.The method of preparing Ni-TiO_2 sol and the substrates, deposition of the coating onto the substrates preparing the substrate, characterization analysis and testing the performance of the coating(photocatalytic).

In chapter 4, content are based on the results of the testing that had been done on the coating to achieve project's objectives. First objective was to analyze the effect of Ni addition on the microstructure of TiO_2 coating on different ceramic substrates meanwhile for the second objective was to determine the photocatalytic property of Ni-TiO_2 coating.

Lastly, chapter 5 showed the conclusion and recommendation of the project. Recommendation can be used in future research that may improve the results of the finding.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter explains on the substrate utilized in this project that is ceramic tiles with an emphasis on aim to produce antimicrobial properties. Later, TiO_2 coating is discussed on its photocatalysis properties, self-cleaning and the antimicrobial ability. Lastly, the effect of the additives on the TiO_2 coating such as its deposition, morphology, photocatalytic property, antimicrobial property and adhesion is discussed given an attention to nickel (Ni) addition.

2.2 Ceramic Tiles

Aesthetic characteristics are among the reasons why ceramic tiles is widely used. Besides, from that, ceramic tiles have excellent technical properties, namely high chemical durability, anti-staining properties and good mechanical resistance, amongst others. Moreover, ceramic tiles also have high resistance to deep abrasion, very low (0.5%) water absorption, high hardness, and suitable resistance to thermal shock and frost. With all these characteristics, that is why ceramic tiles is used widely in the application such as in hospitals, commercial buildings, schools, and private houses (Maria et al, 2013).