



**Faculty of Manufacturing Engineering**

**EFFECTS OF Ag ON TiO<sub>2</sub> DIP-COATING ON UNGLAZED  
CERAMIC SUBSTRATES FOR ANTI-MICROBIAL COATING**

**NurHamizah Binti Ahmad Rusli**

**Master of Science in Manufacturing Engineering**

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SUBSTRATES FOR ANTI-MICROBIAL COATING**

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**A thesis submitted  
in fulfillment of the requirement for degree of Master of Science  
in Manufacturing Engineering**

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**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2015**

## DECLARATION

I declare that this thesis entitle “Effects of Ag on TiO<sub>2</sub> dip-coating on unglazed ceramic substrates for anti-microbial coating” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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## **APPROVAL**

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering

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## ABSTRACT

Ag-TiO<sub>2</sub> coating has been of interest for the purpose of antimicrobial application because silver (Ag) is well-known as antimicrobial agent and TiO<sub>2</sub> possess as self-cleaning agent. In this research, it is aims to investigate the antimicrobial performance of Ag-TiO<sub>2</sub> coating on unglazed ceramic substrate where the effects of additives and number of layers on the characteristic of Ag-TiO<sub>2</sub> coating were investigated. The effect of PEG 2000 and Degussa P25 as additives on the characteristic of Ag-TiO<sub>2</sub> coating was reported. The TiO<sub>2</sub> coatings were produced via sol gel method using titanium tetraisopropoxide as a precursor from solutions that contains additive and without additive. Seven layers of TiO<sub>2</sub> coating were deposited on unglazed ceramic substrate produced from waste glass. All coatings were deposited using dip coating technique and were sintered at 500°C. The TiO<sub>2</sub> coating was then immersed in an Ag nanoparticles solution before dried at 90° C in an oven to form Ag-TiO<sub>2</sub> coating. Besides, the effects of number of layers on Ag-TiO<sub>2</sub> coating with additive (Degussa P25) were characterized at five, seven and ten layers using dip coating technique. TiO<sub>2</sub> coating at different layers was then sintered at 500°C before immersed in Ag nanoparticle solution. All coatings were evaluated using GAXRD and SEM analysis to characterized crystalline phase and coatings (surface morphology and thickness), respectively. Furthermore, Ag-TiO<sub>2</sub> coating with two concentrations of silver nanoparticles (50 ppm and 1000 ppm) were prepared and antimicrobial analysis according to McFarland standard against E.coli, S.aerues, Fungus and MRSA were carried out and zone of inhibition are measured. Ag-TiO<sub>2</sub> coating with additive (Degussa P25) shows anatase phases and homogeneous coating (morphology and Ag distribution) with the highest thickness (8.80 µm) which gives advantages to the coating for antimicrobial application as compared to others (without additive and with additive-PEG 2000). Besides, Ag-TiO<sub>2</sub> coating with Degussa P25 at 10 layers shows the best result where the coating exists in anatase phases and homogenous coating structure with the highest coating thickness (11.80 µm) compared to 5 layers (5.85 µm) and 7 layers (8.80 µm). As a result, Ag-TiO<sub>2</sub> coating with Degussa P25 as an additive with 10 layers at high concentration of Ag (1000 ppm) showed good performance in inhibiting E.coli growth on unglazed ceramic with a zone of inhibition of 8 mm.

## ABSTRAK

Salutan Ag-TiO<sub>2</sub> telah menjadi perhatian untuk penggunaan aplikasi antimikrob kerana perak (Ag) dikenali sebagai agen antimikrob dan TiO<sub>2</sub> mempunyai agen pembersihan diri. Dalam kajian ini, menjadi tujuan utama untuk menyelidik kebolehpayaan salutan Ag-TiO<sub>2</sub> ke atas substrat seramik tanpa licau iaitu kesan bahan tambahan dan bilangan lapisan keatas pencirian salutan Ag-TiO<sub>2</sub> telah dikaji. Kesan PEG 2000 dan Degussa P25 sebagai bahan tambahan terhadap perincian lapisan Ag-TiO<sub>2</sub> dilaporkan. Lapisan TiO<sub>2</sub> dihasilkan melalui kaedah sol gel menggunakan titanium tetraisopropoxide sebagai pemangkin dari larutan yang mengandungi bahan tambahan dan tanpa bahan tambahan. Tujuh lapisan salutan TiO<sub>2</sub> didepositkan menggunakan teknik salutan celupan dan dibakar pada 500°C. Salutan TiO<sub>2</sub> kemudian direndam dalam larutan nanopartikel Ag sebelum dikeringkan pada 90°C didalam pemanas untuk membentuk salutan Ag-TiO<sub>2</sub>. Kemudian, kesan nombor lapisan terhadap salutan Ag-TiO<sub>2</sub> dengan bahan penambahan (Degussa P25) diperincikan pada lima, tujuh dan sepuluh lapisan menggunakan teknik salutan celupan. Salutan TiO<sub>2</sub> pada lapisan berbeza kemudian dibakar pada 500°C sebelum direndam dalam larutan nanopartikel Ag. Kesemua salutan dinilai menggunakan analisa GAXRD dan SEM untuk diperincikan fasa kristal dan salutan (morfologi permukaan dan ketebalan), masing-masing. Tambahan pula, salutan Ag-TiO<sub>2</sub> dengan dua kepekatan partikel nano perak (50 ppm dan 1000 ppm) telah disediakan dan analisa antimikrob berdasarkan kepada piawaian McFarland menentang E.coli, A.aerues, Kulat dan MRSA dijalankan dan zon perencatan diukur. Salutan Ag-TiO<sub>2</sub> dengan bahan tambahan (Degussa P25) menunjukkan fasa anatase dan salutan yang seragam (morfologi dan taburan perak) dengan ketebalan yang tertinggi (8.80 µm) yang memberi kelebihan kepada salutan berbanding yang lain (tanpa bahan tambahan dan dengan bahan tambahan PEG 2000). Selain itu, salutan Ag-TiO<sub>2</sub> dengan Degussa P25 pada lapisan ke 10 menunjukkan hasil salutan yang terbaik iaitu salutan menghasilkan fasa anatase dan keseragaman struktur salutan dengan ketebalan yang tertinggi (11.80 µm) berbanding 5 lapisan (5.85 µm) dan 7 lapisan (8.80 µm). Sebagai kesimpulan, salutan Ag-TiO<sub>2</sub> dengan bahan tambahan Degussa P25 dengan 10 lapisan pada kepekatan Ag yang tinggi (1000 ppm) menunjukkan kebolehpayaan dalam menghalang pertumbuhan E.coli ke atas seramik tanpa licau dengan zon perencatan pada 8 mm.

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## LIST OF ABBREVIATIONS

AgTiO <sub>2</sub>	Silver doped titanium dioxide
EDX	Electron Dispersive X-Ray
eV	Electron volt
hν	Energy
GAXRD	Grazing Angle X-ray Diffraction
MRSA	Multi Resistant Staph Aureus
PEG	Polyethylene Glycol
ppm	parts per million
Degussa P25	Titanium Dioxide powder
SEM	Scanning Electron Microscopy
S.Aureus	Staph Aureus
TiO <sub>2</sub>	Titanium dioxide
UV	Ultraviolet radiation
XRD	X-Ray Diffraction

## LIST OF PUBLICATIONS

Zulkifli, M.R., Nur, H.A.R., Jariah, M.J. and Mazidah, Z., 2015. The Effect of Ceramic Substrates Roughness on the Deposition of AgTiO<sub>2</sub> Coatings. *Applied Mechanics and Materials*, 699, pp. 9-14.

M.R. Zulkifli., A.R.Nurhamizah, J.M. Juoi and Z.Shamsudin., 2015. Silver Nanaoparticles in TiO<sub>2</sub> Coatings for Potential Antimicrobial Coatings, *Advanced Materials Research*, 1087, pp. 20-24.

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of study

Tiles materials are used widely in building construction which commonly can be categorized into two types that are glazed and unglazed. Generally, ceramic tiles do not have antibacterial protection, causing microorganism to easily grow on its surface. Therefore, antimicrobial coating that can kill, or inhibit the growth of microorganism such as bacteria is beneficial (Hochmannova and Vytrasova, 2010). In this study, Ag-TiO<sub>2</sub> coating was deposited onto unglazed ceramic tile produce from waste glasses for the purpose of antimicrobial application.

Ag-TiO<sub>2</sub> coating is been of interest for the purpose of antimicrobial application because silver (Ag) are well-known antimicrobial agent and TiO<sub>2</sub> possess self-cleaning properties under UV radiation (Xubin et al. 2010). Anatase phase of titanium dioxide is known to possess electron of the valence band that reacts enthusiastically when exposed to UV light; where it produces surface oxidation to eliminate harmful from coated surface (Mohsen and Vajiheh, 2012). The additions of silver nanoparticles (Ag) into TiO<sub>2</sub> improve the TiO<sub>2</sub> self-cleaning properties under visible light or dark environment as reported by Robert et al. (2012).

The deposition of Ag-TiO<sub>2</sub> coating on the unglazed ceramic substrates (produced from waste material) was carried out using sol gel method. Sol gel method offers several benefits such as homogeneity of coating, low cost and low processing temperatures compared to other method (Zhang, 2010). In this study, dip coating; a technique of evaporation-induced and self-assembled on the substrate as it is slowly withdrawn from the coating solution is utilized for the deposition of coating. Substrates were immersed into a titanium dioxide (TiO<sub>2</sub>) sol gel and silver nanoparticles (Ag) solution to produce an antimicrobial coating.

## **1.2 Problem Statement**

Ceramic tiles are widely manufactured in industry and generally used as construction materials for buildings. In addition, ceramic tiles have purposes such as to complete building appearances and to cover the concrete area. Nevertheless, they need a periodical cleaning process to maintain their cleanliness as well as their appearance. During utilization, walls and floors covering structure e.g. ceramic tiles are subjected to physical, chemical and biodegradation that leads to deterioration. Therefore, microorganisms can easily grow on their surfaces especially in existence of moist (Knetsch and Koole, 2011). It would spread infections and pose a threat to people or patients who have weak immune systems.

In general, Ag-TiO<sub>2</sub> coating possesses beneficial properties for application as antimicrobial agents. The deposition of AgTiO<sub>2</sub> coating on surface is explored by many researchers in order to improve efficiency of the antimicrobial under UV and visible light (Liu et al. 2008). It was observed that the performance of AgTiO<sub>2</sub> on antimicrobial is depending on the types of phases formed, its surface microstructure, the coating thickness and the silver presence in the deposited coating (Yu et al. 2011). Prior studies explored the

deposition of TiO<sub>2</sub> film on several kinds of substrates through the sol gel method such as ceramic tiles (Konkanok et al. 2008), glaze ceramic tiles (Sun et al. 2008), glass substrate (Hyeok et al. 2006), stainless steel (Kristopher et al. 2006), polymer (Piangjai et al. 2012), porous ceramic  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> (Ma et al. 2009), glass fibre roving (Ubonchonlakate et al. 2012), and wood (Kandelbauera and Widsten, 2009). However, limited study is carried out on the deposited of Ag-TiO<sub>2</sub> coating on unglazed ceramic tiles. Ceramic substrates used in this study were produced from mixture of glass waste made of transparent glass (85 wt%): ball clay (15 wt%) formed by hydraulic pressing and sintering process at 850°C. In addition, unglazed ceramic tiles contained porosity, less smoothness and do not have antimicrobial coating. Therefore, the deposition of Ag-TiO<sub>2</sub> coating on unglazed ceramic substrate is carried out to look for its potential for antimicrobial application hence improves the substrates properties.

In this research, the effect of additives in TiO<sub>2</sub> sol gel preparation and effects of layers on the coating were evaluated in order to produce titania phases which are anatase and homogeneous coating. Moreover, the effect of silver nanoparticle (Ag) concentration was also assessed against different microbes. Later on, the antimicrobial performances of the deposited Ag-TiO<sub>2</sub> coating were evaluated to look at its efficiency as antimicrobial coating. This is an innovative approach in producing antimicrobial tiles that have good resistance against microbial.

### **1.3 Objectives**

The aim of this research is to deposit Ag-TiO<sub>2</sub> coating on unglazed ceramic substrate for antimicrobial application. The specific objectives are:

1. To determine the effect of additive on the characteristic of Ag-TiO<sub>2</sub> coating.
2. To determine the effect of layers on the characteristic of Ag-TiO<sub>2</sub> coating deposited on unglazed ceramic substrate.
3. To evaluate the antimicrobial activities of the Ag-TiO<sub>2</sub> coating deposited on unglazed ceramic substrate through Gram positive (E.coli) and Gram negative (MRSA) microbes.

### **1.4 Significance of Research**

The outcome of this research will contribute to the development of bacteria prevention in both indoor and outdoor humidity conditions such as in toilets and roofing tiles on buildings. Furthermore, ceramic coated with antimicrobials coating are particularly useful in an environment that requires a high level of hygiene. Moreover, result from this research will be able to contribute towards enhanced deposition of Ag-TiO<sub>2</sub> coating by identifying the suitable additives and number of layers for better antimicrobial performance.