

**INVESTIGATION OF SURFACE PROPERTIES
FOR SELF-CLEANING COATING ON CERAMIC
WALL TILES**

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UNIVERSITI SAINS MALAYSIA

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**INVESTIGATION OF SURFACE PROPERTIES
FOR SELF-CLEANING COATING ON CERAMIC
WALL TILES**

by

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for the degree of
Master of Science
(Materials Engineering)**

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled “Investigation of Surface Properties for Self-cleaning Coating on Ceramic Wall Tiles”. I also declare that it has been not previously submitted for the award for any degree or diploma or other similar title for any other examining body for university.

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LIST OF SYMBOLS

C_t	Absorption of dye after reaction at t time
Å	Angstrom
C_B	Conduction band
$^\circ$	Degree
$^\circ\text{C}$	Degree Celcius
e_{cb}^-	Electrons at conduction band
eV	Electrovolt
g/mol	Gram per mole
g/cm ³	Gram per centimeter cube
h_{vb}^+	Holes at valence band
C_o	Initial absorption of dye
J/m ²	Joule per meter square
R_{\max}	Maximum reflectance value in wavelength
R_{\min}	Minimum reflectance value in wavelength
M	Molar
ppm	Parts per million
$h\nu$	Photon energy
rpm	Revolution per minutes
R_q	Root mean square
θ	Theta
V_B	Valence band

LIST OF ABBREVIATIONS

ALE	Atomic Layer Epitaxy
APTES	(3-aminopropyl) triethoxysilane
BPA	Bisphenol A
CA	Contact angle
CNT	Carbon nanotubes
CVD	Chemical Vapour Deposition
EDX	Energy Dispersive Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transmission Infrared
GA	Glutaraldehyde
HCl	Hydrochloric acid
ICDD	International Centre for Diffraction Data
IR	Infrared
MO	Methyl orange
PDMS	Polydimethylsiloxane
PEG	Polyethylene glycol
PEI	Polyethylenimine
PMMA	Poly(methyl methacrylate)
PRW	Petroleum Refinery Wastewater
PVA	Poly(vinyl) alcohol
PVC	Poly(vinyl) chloride
PVD	Physical Vapour Deposition
ROS	Reactive Oxygen Species
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
THF	Tetrahydrofuran
UV	Ultraviolet
UV-VIS	Ultraviolet Visible Spectroscopy
WCA	Water contact angle
XRD	X-ray Diffraction

**KAJIAN TENTANG SIFAT PERMUKAAN BAGI SALUTAN
PEMBERSIHAN-KENDIRI KE ATAS JUBIN DINDING SERAMIK**

ABSTRAK

Kajian ini memberi tumpuan pada sifat permukaan jubin dinding seramik bersalut kuprum dop titanium dioksida (Cu dop TiO_2) dalam menghasilkan sifat-sifat salutan pembersihan-kendiri dan antibakteria. Serbuk Cu dop TiO_2 dihasilkan melalui kaedah hidroterma diikuti dengan pengkalsinan pada $300\text{ }^\circ\text{C}$. Transformasi fasa dari struktur tetragonal TiO_2 tulen kepada TiO_2 heksagonal ditunjukkan dalam hasil difraksi sinar-X (XRD), diikuti oleh transformasi morfologi dari bentuk bulat ke nano tiub seperti yang dibuktikan dalam Mikroskop Imbasan Elektron (SEM) dan Mikroskop Transmisi Elektron (TEM). Pengabungan Cu^{2+} ion dopan ke dalam TiO_2 membuktikan jurang tenaga TiO_2 mengurang dari 3.2 eV kepada 2.07 eV di mana juga mempamerkan kecekapan dalam aktiviti degradasi fotokatalitik. Kemudian, PVA/GA mempamerkan pelekatan salutan yang lebih baik disebabkan peranan glutaraldehid (GA) berfungsi sebagai agen silang polimer. Lima kitaran salutan ditentukan sebagai lapisan yang sesuai pada jubin dinding seramik disebabkan faktor penampilan fizikal, kepekatan serbuk dan kekasaran permukaan. Sifat permukaan jubin dinding seramik (licau dan tidak licau) bersalut dengan teknik salutan yang berlainan (teknik celupan dan teknik semburan) dikaji dalam ujian fungsi. Jubin bersalut dengan teknik semburan memaparkan permukaan salutan yang sekata berbanding dengan jubin bersalut dengan teknik celupan. Dalam ujian kekerasan pensil, lapisan salutan melalui teknik semburan pada jubin tidak licau mempunyai rintangan calar yang paling tinggi. Dalam ujian rendaman, jubin bersalut dengan teknik celupan dan semburan menunjukkan lapis salutan terkupas daripada permukaan jubin semasa direndam di

dalam rendaman asid (pH 4), tetapi lapis salutan tidak terkupas semasa dalam rendaman air (pH 7) dan alkali (pH 9).

INVESTIGATION OF SURFACE PROPERTIES FOR SELF-CLEANING COATING ON CERAMIC WALL TILES

ABSTRACT

This study emphasized on the surface properties of Cu doped TiO₂ coated ceramic wall tiles in producing self-cleaning and antibacterial coating properties. Cu doped TiO₂ powder was synthesized via hydrothermal method followed by calcination at 300 °C. The phase transformation from tetragonal structure of pure TiO₂ to hexagonal TiO₂ was shown in X-ray diffraction (XRD) result, followed by morphology transformation from near-spherical shape to nanotubes as evidenced in Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) micrographs. The incorporation of Cu²⁺ metal ion dopant into TiO₂ was proved in reducing the band gap energy of TiO₂ from 3.2 eV to 2.07 eV. Also, Cu doped TiO₂ exhibited greater photocatalytic degradation than undoped TiO₂. Next, PVA/GA was found to have better coating adhesion due to the role of glutaraldehyde (GA) as polymer chains crosslinking agent. Five coating layers were determined as the appropriate coating cycles on ceramic wall tiles due to the factors of physical appearance, powder concentration and surface roughness. The surface properties of the coated ceramic wall tiles (glazed and unglazed) by different coating techniques (dip coating and spray coating) were investigated in functionality characterizations. Spray coated tiles displayed homogeneous coating surface compared to dip coated tiles. In pencil hardness test, spray coated unglazed tiles has the highest scratch resistance. In solution immersion test, both dip and spray coated tiles experienced coating peeled off in acid (pH 4) and immersion, but no peeled off surface was seen when immersed in water (pH 7) and alkaline (pH 9) solution.

CHAPTER 1

INTRODUCTION

1.1 Research background

Tile is one of the most well-known types of materials used in household, clinical, as well as industrial environments. The development of ceramic tiles have gained increasing attention from due to its various benefits such as durability, colour permanence resistance, as well as its hygienic factor. Basically, ceramic tiles are categorised into floor tiles, wall tiles and porcelain tiles, only wall tiles are focussed in this research. The current available ceramic wall tiles in the market is lack of self-cleaning properties. They are easily to get dirty such as dirt, dust, oil, split food, drinks or even chemicals can be simply stick on their surface. Therefore, it needs frequent cleaning such as use strong disinfectant to prevent bacteria, fungal or viruses growth on the tiles. Otherwise, the skin contact with these pathogenic microorganisms have high possibility to cause disease or even death. The potential health risks such as hospital infections and some dermatomes which caused by pathogenic microorganisms were highlighted in Onaizi & Leong (2011). However, water contamination issue was triggered by the cleansing agent that used in tiles cleaning process.

In fact, most cleansing agents consist of different chemical components for instance bleaches, surfactants, fillers, builders, optical brighteners, enzymes, anti-redeposition agents, dye and perfume. These chemical components will flow towards into drainage system and contributed to ground level water pollution (Goel & Kaur, 2017). Also, almost one in every three commercial cleaning products consists of harmful chemicals which are related to asthma, respiratory diseases, skin and major organ damage, reproductive disorder and even cause cancer (Sabharwal, 2015). For example, phosphate is a water-softening mineral which is widely used in most of the

detergents. The high percentage of phosphates (approximately 35-75 %) in cleansing agent has triggered the root cause of water contamination by inhibiting the biodegradation of organic substances (Kogawa et al., 2017; Sabharwal, 2015). Besides, dye component is known to be toxic and takes long time to degrade, which caused coloration as well as affecting water quality. Additionally, detergent contains oxygen reducing substances which caused severe impact to aquatic ecosystem. The flowing of detergent into rivers leads to eutrophication which is a situation when the water body becomes enriched in dissolved nutrients such as magnesium, phosphate and calcium. As a result, the water enriched in dissolved nutrients encourage the growth of aquatic plant life, hence leading to oxygen depletion (Kogawa et al., 2017).

Later, the developing of nanostructure coating layer based on self-cleaning mechanism contributed in minimizing the chemical water contamination problem. Due to the health risk issue triggered by pathogenic microorganisms, the demands for anti-microbial wall and floor coating materials in domestic environment, clinical and industrial are expected to increase (Özcan et al., 2017). Accordingly, the researchers attempt to develop self-cleaning tiles using various nanomaterials with antibacterial properties such as silver and titanium dioxide. Nanomaterial is the root of nanotechnology where it refers to a method for treatment of a matter, with the aim of obtaining new functional materials as well as improved their properties. Generally, nanomaterial is defined as an object that has at least one of its dimension in nanometer scale which is around 1 nm to 100 nm (Feynman & Feynman, 2018).

Nanoparticles is an example of nanomaterials, which plays an essential role among the varieties of applications since they have unique properties and characteristics. Among the developed nanoparticles with antibacterial behavior, the

attention was focussed on titanium dioxide (TiO_2) nanoparticles. Since twentieth century, many works correlated to TiO_2 nanoparticles have been studied, which includes various applications besides large-scale production (Chen & Mao, 2007; Gupta & Tripathi, 2011; Lan et al., 2013). Among the oxide photocatalyst, TiO_2 nanoparticles is considered as the most well-known and widely used as photocatalyst materials. When compared to TiO_2 , other photocatalyst such as $\alpha\text{-Fe}_2\text{O}_3$ and ZnO have several disadvantages. For instance, $\alpha\text{-Fe}_2\text{O}_3$ has been reported to have lower photocatalytic activity than TiO_2 whereas, Zn^{2+} ions in ZnO structure can be easily released into the aqueous solutions when ZnO was used as photocatalyst (Lusvardi, Barani, Giubertoni, & Paganelli, 2017).

TiO_2 particularly crystalline anatase phase, succeeded to gain attention in the field of self-cleaning surface technology (Ambrus et al., 2008; Inagaki et al., 2001; ÖZCAN et al., 2017; Shakeri et al., 2018; Zhao et al., 2018). Besides its strong photochemical ability, TiO_2 offers various advantages such as non-toxic, abundant, inexpensive, hydrophilicity, chemical stability and anti-algae properties which makes TiO_2 as a favorable candidate for many industrial and environmental applications (De Niederhäusern et al., 2013; Hu et al., 2015). Later, the attention was shifted from anatase phase TiO_2 to hexagonal TiO_2 (h- TiO_2 is a new crystal structure of TiO_2) since anatase phase TiO_2 has been reported to have large band gap energy which has limited its ultraviolet light absorption.

Recently, hydrothermal method has been recently discovered to produce hexagonal TiO_2 nanostructure (Razali et al., 2014). The benefits of hydrothermal technique have been discussed in the past research compared to the conventional material processing techniques (Yoshimura & Byrappa, 2008). Hydrothermal

technology offers an excellent opportunity for fabricating of advanced materials neither it is in fine particles, bulk single crystals or even nanoparticles. Moreover, it is also an interesting technique to synthesis highly homogeneous and mono-dispersed nanoparticles besides nano-hybrid and nanocomposite materials (Byrappa & Adschiri, 2007; Xu et al., 2011) .

1.2 Problem statement

Currently, TiO₂ nanotubes in anatase form has been reported to have larger band gap energy (3.2 eV) which has limited its efficiency to absorb ultraviolet (UV) light (Xu et al., 2011; Zeng, 2011). Thus, anatase TiO₂ requires the activation from UV ray to stimulate the valence band electron to conduction band for conducting photocatalytic reaction (Štengl et al., 2010; Tada et al., 2011). Yet, solar light has only 5 % UV light (190- 380 nm) compared to 43 % visible light (380-760 nm) which had limited the efficiency for solar photocatalytic reaction. With the aim of overcoming the wide band gap problem of TiO₂, metal ion dopants such as Cu²⁺ can be doped into TiO₂ to reduce the band gap and with its additional properties like antimicrobial properties. The doping of metal impurities into TiO₂ has been found in reducing band gap energy concurrently increase the absorptions of TiO₂ to the visible light. Later, the findings by Razali et al. (2014) doped Cu²⁺ metal ion into TiO₂ nanotubes, proved in enhancing its photocatalytic power by gaining smaller band gap (2.05 eV). However, this Cu doped TiO₂ powder never been used as a self-cleaning agent.

When apply on tiles coatings, TiO₂ powder alone is not sufficient to fabricate self-cleaning tiles. One of the approach is to directly incorporate TiO₂ into glaze composition, but the difficulties in changing the glaze composition is not favourable by the industries. Therefore, it is preferable to develop a thin layer of coating on wall

tiles. Yet, the new challenge appeared which is the difficulties in fabricating a suitable medium to bind TiO₂ powder on surface of wall tiles. There were numerous studies in recent years reported on the combination of nanostructure photocatalyst and polymer binder coated on ceramic tiles had performed a great and expected self-cleaning behaviour (Khan et al., 2018; Yu et al., 2005). Based on the past research, polyethyleneglycol, poly (vinyl alcohol) and polyethylenimine are some of the polymer binders which often used as the coating medium.

Since TiO₂ has hydrophilic nature, there is a limited choice of polymer binder with hydrophilic properties. It was a critical decision to select a suitable polymer binder to incorporate with nanostructure photocatalyst in order to establish strong adhesion on tiles surface as well as not affecting its function in destroying organic pollutants. An economical polymer, poly(vinyl alcohol) was proposed as a base material to develop PVA/TiO₂ nanocomposites and makes it particularly useful in commercial applications. However, water soluble characteristic of PVA was considered due to it has affected the adhesion of self-coating layer on ceramic wall tiles (Khan et al., 2018).

Last but not least, the evaluation on surface properties from different self-cleaning coating methods are least reported in the literature. Therefore, this research propose in exploring the surface properties by comparing dip coated and spray coated self-cleaning ceramic wall tiles. Theoretically, spray coating provides an uniformly dispersion of TiO₂ particles on tiles surface compared to dip coating method. Hence, it will not affect the efficiency of self-cleaning behaviour due to the agglomeration possibility of TiO₂ nanoparticles has been minimised.

1.3 Research objectives

This research aimed to develop a new coating layer on the ceramic wall tiles with self-cleaning properties and to investigate the surface properties of the newly developed coating layer. In order to achieve these aims, the following research objectives are set:

- i. To synthesize Cu doped TiO₂ nanotubes with hydrophilic nature through hydrothermal method.
- ii. To determine the adhesion effectiveness of coating solution (PVA and PVA/Glutaraldehyde) on ceramic wall tiles.
- iii. To investigate the surface properties of self-cleaning ceramic wall tiles.

1.4 Scope of research

This study aimed to focus on the surface properties of self-cleaning ceramic wall tiles. Initially, the synthesizing of TiO₂ nanotubes via hydrothermal method was the first step carried out. With the aim of improving photocatalytic activity, Cu²⁺ ion was doped into TiO₂ structure.

In this study, two strategies were suggested to produce a stable covalent coating of Cu doped TiO₂ powder onto ceramic wall tiles to achieve self-cleaning proficiency. In the first approach, PVA/Glutaraldehyde (GA) polymeric coating solutions was employed to build a strong physical bond between Cu doped TiO₂ powder and the surface of ceramic wall tiles. The comparisons of using PVA and PVA/GA in coating Cu doped TiO₂ nanotubes to highlight the importance of glutaraldehyde as crosslinking agent. This section aimed to investigate the crosslinking efficiency of glutaraldehyde in PVA solution in the absence of acid catalyst.

For the second approach, different coating techniques (dip coating and spray coating) were used to examine the dispersions of Cu doped TiO₂ powder on ceramic wall tiles. Since this study focused on the surface properties, hence microbiological test is not conducted, instead the work emphasized on the structural and functionality testing to examine the adhesiveness of self-cleaning coating layer on ceramic wall tiles. As a whole, the scope of research in this experiment was summarized in Figure 1.1.

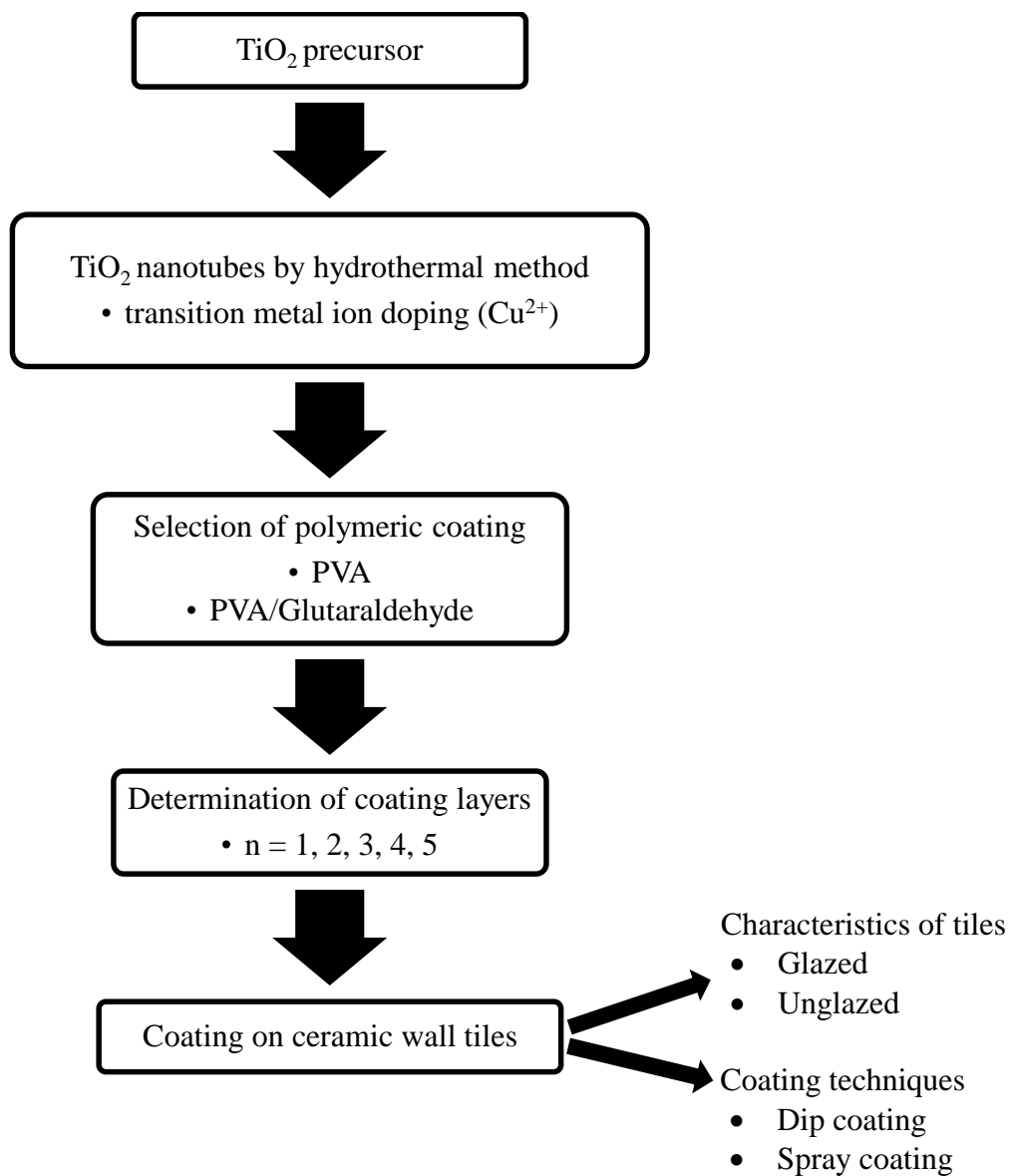


Figure 1.1: Scope of study in this research.