CHARACTERIZATION OF VINYLTRIETHOXYSILANE-TETRAETHOXYSILANE-TETRABUTOXYTITANATE HYDROPHOBIC SOL-GEL COATING FOR PHOTOVOLTAIC PANELS

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DEDICATION

This thesis is dedicated to my sweet and loving parents, whose affection, love, encouragement and prays of day and night make me able to get such success and honour. It is also dedicated to my beloved husband who always stand by me and hold my hand to help me make it through. I give special thanks to my best friend, who always believed in me and being my best cheerleader throughout this journey.

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ABSTRACT

Surfaces that simultaneously exhibit hydrophobicity, high contact angle and high transmission of visible light are of interest for many applications such as optical devices, photovoltaic (PV) panels and self-cleaning windows. The fabrication of such surfaces is challenging due to the complex preparation using various compositions and thermal treatment with the requirement of a controlled environment. In this study, the main goal was to develop and characterize hydrophobic coating using sol-gel method in ambient condition suited for PV application. A solution was prepared using three different materials including vinyltriethoxysilane (VTES), tetraethoxysilane (TEOS) and tetrabutoxytitanate (TTBU) called VTT (VTES-TEOS-TTBU) sol as the organic-inorganic hybrid sol. Then, this sol was applied onto glass substrates using spin-coating method for laboratory-scale working samples. Coated samples were produced into two sets with different composition to obtain silica-titania sol using Post-addition of Water to Titanium Alkoxide (PWTA) and Chemically Modified Titanium Alkoxide (CMTA). The effects of different composition were examined with respect to the contact angle, surface roughness, and transmittance. Besides, the VTT coating has also undergone different thermal treatments to study its hydrophobicity effect. Based on the results obtained, VTT sol still showed hydrophobic properties (CA>90^{o}) even when fabricated under ambient condition. The increase of temperature in the pre-bake and post-bake process increased the hydrophobicity of VTT coating. Moreover, CMTA method showed better hydrophobicity compared to the PWTA method. The best sample obtained was the CMTA sample with the highest post-bake thermal treatment (150°C) which exhibited the highest contact angle (110.01°), surface roughness (0.452 nm) and transmission percentage (96% centered at 571 nm of wavelength). All coating samples were prepared in ambient condition with promising performance, eliminating the need for controlled condition based on previous research to suit the varied temperature and humidity for different coating preparation.

ABSTRAK

Permukaan hidrofobik yang mempunyai nilai sudut sentuhan dan dalam masa yang sama mempunyai kepancaran cahaya nampak yang tinggi telah menjadi tumpuan kajian bagi pelbagai aplikasi seperti peranti optik, panel fotovolta (PV) dan tetingkap swacuci. Pembuatan permukaan seperti itu amat mencabar kerana penyediaan yang rumit yang melibatkan pelbagai komposisi dan perawatan haba dengan keperluan persekitaran terkawal. Dalam kajian ini, matlamat utama ialah untuk membangunkan dan mencirikan lapisan hidrofobik menggunakan kaedah *sol-gel* dalam keadaan ambien yang sesuai untuk aplikasi PV. Sejenis larutan disediakan dengan menggunakan tiga bahan yang berbeza termasuk *vinyltriethoxysilane* (VTES), *tetraethoxysilane* (TEOS) dan *tetrabutoxytitanate* (TTBU) yang dipanggil VTT (VTES-TEOS-TTBU) sol sebagai sol hibrid organik-inorganik. Kemudian, sol ini diletakkan pada substrat kaca menggunakan kaedah pelapisan secara putaran bagi sampel kerja berskala makmal. Sampel bersalut dihasilkan dalam dua set komposisi berbeza untuk mendapatkan sol silika-titania menggunakan kaedah *Post-addition of Water to Titanium Alkoxide* (PWTA) dan *Chemically Modified Titanium Alkoxide* (CMTA). Kesan komposisi yang berbeza dikaji berdasarkan sudut sentuhan, kekasaran permukaan, dan kepancaran. Selain itu, lapisan VTT juga telah menjalani perawatan haba berbeza untuk mengkaji kesan hidrofobiknya. Berdasarkan dapatan kajian, lapisan VTT masih menunjukkan ciri hidrofobik (CA>90^{o}) walaupun dibuat dalam keadaan ambien. Peningkatan haba sewaktu proses pra bakar dan pasca bakar meningkatkan juga hidrofobik lapisan VTT. Tambahan lagi, kaedah CMTA menunjukkan hidrofobik yang lebih baik berbanding kaedah PWTA. Sampel terbaik yang telah diperolehi ialah sampel CMTA dengan perawatan haba pasca-bakar paling tinggi (150°C) yang menunjukkan nilai yang tertinggi bagi sudut sentuhan (110.01°), kekasaran permukaan (0.452 nm) dan peratusan kepancaran (96% berpusat pada panjang gelombang 571 nm). Semua sampel lapisan yang dibuat dalam keadaan ambien menunjukkan prestasi yang baik mampu menghapuskan keperluan untuk persekitaran terkawal yang digunakan dalam penyelidikan sebelum ini supaya dapat disuaikan dengan suhu dan kelembapan yang bervariasi untuk penyediaan lapisan yang berbeza.

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LIST OF ABBREVIATIONS

AcAc	-	Acetylacetonate
AFM	-	Atomic Force Miscroscope
AlAA	-	Aluminium Acetylacetonate
CMTA	-	Chemically Modified Titanium Alkoxide
EDS	-	Electrodynamic screen
EtOH	-	Ethanol
LBL	-	Layer-By-Layer
PDMS	-	Polydimethylsiloxane
PV	-	Photovoltaic
PWTA	-	Post-Addition of Water to Titanium Alkoxide
SEM	-	Scanning Electron Microscope
TEOS	-	Tetraethoxysilane
TiO_2	-	Titanium Dioxide
TTBU	-	Tetrabutoxytitanate
VTES	-	Vinyltriethoxysilane
VTT	-	VTES-TEOS-TTBU
ZnO	-	Zinc Oxide

LIST OF SYMBOLS

f_g	-	Fractions of the gas areas on the surface
f_s	-	Fractions of the solid areas on the surface
P	-	Power
r	-	Surface roughness factor
T	-	Transmittance
γ_{lg}	-	Interfacial tensions of liquid-gas
γ_{sg}	-	Interfacial tensions of solid-gas
γ_{sl}	-	Interfacial tensions of solid-liquid
θ	-	Contact angle
θ_{CB}^*	-	Cassie-Baxter contact angle
θ_g	-	Liquid contact angle on gas
θ_s	-	Liquid contact angle on solid surface
θ_W^*	-	Wenzel contact angle
λ	-	Wavelength

CHAPTER 1

INTRODUCTION

1.1 Problem Background

Malaysia is in the tropical climate region along the Equator where sunshine is found abundantly all year round contributing to various developing strategies of solar energy to reduce the dependence on fossil fuels. Based on Sustainable Energy Development Authority (SEDA), the amount of renewable energy generated has increased over the past five years with solar generation shown the highest generation compared to other renewable energy sources such as biogas, biomass, and small hydro. Solar generation started with 4720.16 MWh in 2012, these figures change rapidly through the years up to 397715.22 MWh in 2017. From the total of 116 sites of photovoltaic (PV) system monitored in Malaysia, the most packed area is in Kuala Lumpur. Correspondingly, Malaysia has grown bigger in utilizing solar energy as the SEDA Feed-in-Tariff program as of July 2016, about 95% of approved applications are from solar PV alone. These figures show that there is increasing interest in installing PV modules in building in Malaysia as an alternative for electricity.

The rising demand for PV energy comes with increasing drawbacks to maintain the technology efficiency, one of the main causes is the soiling issues [1]. Sand dust and pollution particles deposition are the main causes of dirtiness in the panels' surface. These effects are translated into a decrease of about 40% in solar power conversion for each 4 gram of dust per square meter [2]. The solar panels can lose incident light through reflection by the cover glass and through scattering or absorption by particulates on the surface of solar panels, which in turn decreasing its power conversion efficiency. This reduction in PV power output can be anywhere between 2% and 50% depending on a local climate, dust composition and concentration, as well as whether a mitigation strategy is employed [3]. There is the need for regular maintenance to maintain the efficiency and the overall performance of the PV solar

panels. Cleaning of solar panels from contaminants to maintain the optimum solar harvesting capabilities is time-consuming and expensive. Since the last decade, self-cleaning coatings have attracted attention in the scientific community and industrial exploitation. These coatings have been made artificially by mimicking the biological surfaces, such as lotus leaf, gecko, and water striders [4]. Coatings with water-repelling (hydrophobic) or water-dispersing (hydrophilic) qualities are called self-cleaning [1]. Some of the self-cleaning coatings applications can be seen in the textile industry, automobile industry, and optical industry.

In PV application, hydrophobic coatings can provide a variety of conveniences including a reduction in maintenance cost, the extermination of dreary manual work as well as minimizing time spent on cleaning. Other than that, it can also be a cost-effective and alternative solution to reduce the efficiency losses from dust accumulation [5-7]. Coatings can be applied over a substrate in a continuous or discontinuous film through several application methods including sol-gel. Sol–gel method is advantageous in applying coating to a surface because it allows better control of chemical composition and micro-structure of materials, simple employment, and low-cost equipment as well as able to produce unique compositions that cannot be created by conventional methods [8].

Sol-gel is a process involving two chemical reactions: hydrolysis and condensation. In this study, the aim is to synthesis the commonly used multicomponent material systems based on silicon alkoxide by the integration of other metal alkoxide, which is titanium alkoxide. As an improvement towards existing studies [9], the materials synthesized in this research are prepared under ambient condition to prove if it possesses the self-cleaning characteristics as a comparison towards existing controlled condition practice. At the end of the sol-gel process, the Si – O – Ti bonds must be obtained from the sol prepared. However, it is important to consider the difference in reactions rate among various metal alkoxides. While the silicon alkoxide are less reactive compared to titanium alkoxide, this condition will result to phase separation of Si – O – Si and Ti – O – Ti bonds instead of a homogenous Si – O –Ti bonds upon completion of the reactions. This problem can be solved by reaction of pre-hydrolyzed silicon alkoxide with either Post-addition of Water to Titanium

Alkoxide (PWTA) or Chemically Modified Titanium Alkoxide (CMTA), as represented in Figure 1.1. It is important to consider both methods to provide comparison in terms of material development and self-cleaning characteristics of the resulting coatings.

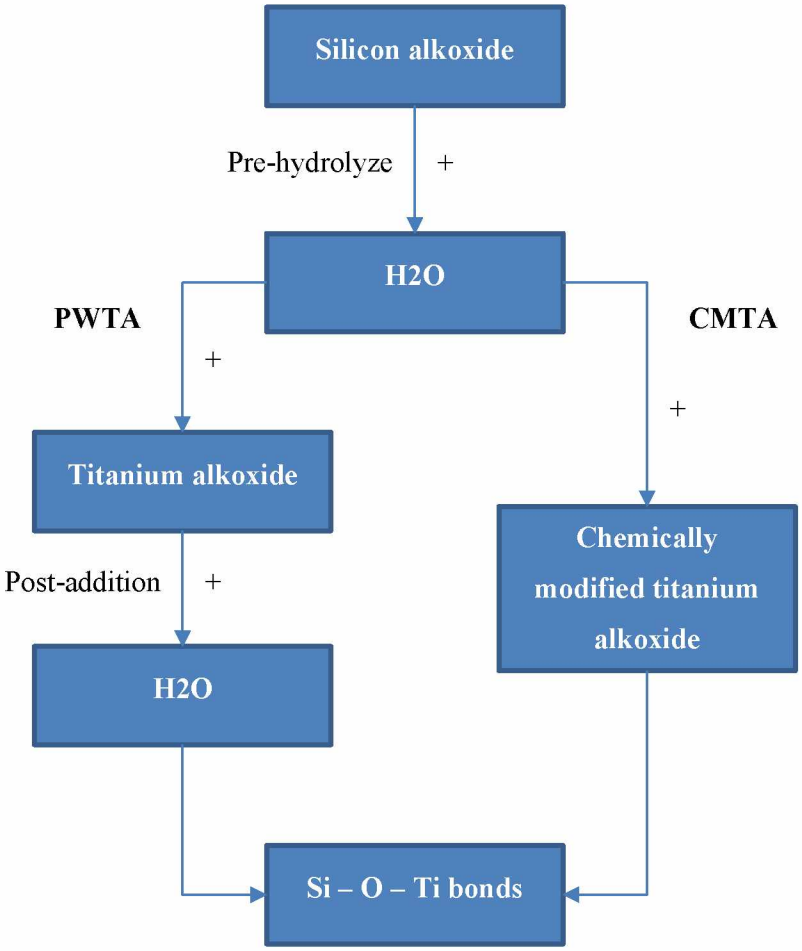

Figure 1.1 Integration of titanium alkoxide to silicon alkoxide using sol-gel

1.2 Problem Statement

Advancement of technology in enhancing PV performance to overcome soiling issues has been growing over the years. Maintaining PV performance from soiling issues using manual cleaning is costly and tedious which has been a major concern in deploying this technology. Dust accumulation and soiling issues can greatly influence the degradation of photovoltaic performance including its efficiency, reliability, and

cost of deployment. To address this issue, coatings play a crucial role in protecting PV panels' surfaces from the aggressiveness of environment. Moreover, they can also provide additional functionalities to the surface of the protected materials. Coatings can be applied over a substrate in a continuous or discontinuous film through several application methods including lithography, template method, one-pot synthesis, layer-by-layer, and sol-gel [10-14].

Comparing these available methods, sol–gel method is advantageous in applying coating to a surface because it allows better control of chemical composition and microstructure of materials, simple employment, and low-cost equipment as well as able to produce unique compositions that can be tailored to suit an intended function. Processes like lithography and template method are costly due to higher overall cost of raw materials or other expensive process requirements so it cannot be used to generate large area substrates with nanostructured surfaces [15]. Although recent studies have pushed the advancement of self-cleaning surface to superhydrophobic and superhydrophilic [16, 17], most of these coatings are fluorine-based which can cause environmental risk and pollution. While other researchers used nanoparticles as an alternative for fluorine-based material such as nano silica to fabricate superhydrophobic coating, a controlled condition during fabrication process is required since nanoparticles are susceptible towards temperature changes. The composition developed are greatly dependent on certain base material under specific working temperature.

A hybrid sol–gel consists of inorganic silica and organically modified silica, which forms three-dimensional networks to impart self-cleaning nature to coated surfaces. As compared to inorganic coating such as silica, titania, or silica-titania; organic coating is more flexible, less fragile, and much easier to fabricate with most fabrication used controlled temperature at 20-30^{o}C [18-20]. The property of hardness is imparted by the inorganic network, while the flexibility and adhesion properties are taken care by the organic agents. This hybrid composite film developed by X. Zhang et al. present high transmittance value up to 99%, enhanced photocatalytic activity and more persistent self-cleaning properties [9]. With the lower reaction temperature, this

study proposed to develop hybrid organic-inorganic coating in ambient condition to see if the developed coating still possess hydrophobic properties.

In application such as coating for solar panels, there is a requirement of coatings that are transparent and anti-reflective. Most common used precursors are silica and titania. A study by L. Ye et al. and Ö. Kesmez et al. found that the use of both silica-titania multilayer coating has proven to have higher transmittance compared to silica- or titania-sole-based coating (97-98%) [18, 21]. To fabricate silica-titania coating for this study, the integration of these inorganic precursors can incorporate either post addition of water towards titanium alkoxide (PWTA) or chemically modified titanium alkoxide (CMTA) [22]. There is a very scarce information on the comparison between these two methods towards its hydrophobicity. Based on the research gaps found, this study pushed the limitation of previous literatures by developing hybrid organic-inorganic hydrophobic coating using VTT with improvement in terms of elimination of controlled condition during fabrication while providing comparison of two different integration of titanium alkoxide using PWTA & CMTA methods.

1.3 Research Objectives

The objectives of the research are:

(a) To synthesize a hydrophobic sol-gel based self-cleaning coating under ambient condition that fits for soiling mitigation technique in photovoltaic application.

(b) To characterize the hydrophobic sol-gel based self-cleaning coating in terms of contact angle and transmittance value.

(c) To study the effect of different heat treatment of the sol-gel based self-cleaning coating to its hydrophobicity.

(d) To study the effect of different composition of sol-gel based self-cleaning coating using PWTA and CMTA method to its hydrophobicity.

1.4 Scope of Research

This research will be focusing on:

(a) Identification of materials and composition suited for sol-gel synthesis with the abilities to exhibit self-cleaning properties.

(b) Synthesis of VTT material by sol-gel method – vinyltriethoxysilane (VTES), tetraethoxysilane (TEOS) and tetrabutoxytitanate (TTBU).

(c) Fabrication of VTT material into self-cleaning coating on laboratory scale working glass samples to meet the requirements of available characterization equipment and facilities.

(d) Characterization of hydrophobic on working glass samples – water contact angle, surface morphology and transmittance.

(e) Observation on effect of different composition of VTT sol using PWTA and CMTA methods towards its hydrophobicity.

1.5 Significance of Study

This study provides an understanding of hydrophobic coating as well as its importance in the PV application. In this study, the hydrophobic issue entails the investigation on the advantages of utilizing a self-cleaning coating in the PV field over traditional cleaning techniques in terms of convenience. Furthermore, this research sheds light on the evolution of hydrophobic coating solutions, including methods for obtaining organic-inorganic sol, coating deposition, and surface morphologies. Researchers in related fields will benefit from this study since it expands their

understanding of self-cleaning techniques using sol-gel generated hydrophobic coatings combined with manufacturing technology. The materials synthesized in this study are created in ambient conditions to see if they have self-cleaning properties while providing a result on different heat treatment can allow switchability of the coating properties, which is an advancement over previous studies.

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