UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Mechanical Engineering

HYDROPHOBICITY PERFORMANCE OF POLYETHYLENE TEREPHTHALATE, THERMOPLASTIC POLYURETHANE AND ALUMINIUM SUBSTRATES USING SELF-FABRICATED CONTACT ANGLE TOOL

Solehah binti Jasmee

Master of Science in Mechanical Engineering

2019

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SOLEHAH BINTI JASMEE

A thesis submitted
in fulfillment of the requirements for the degree of Master of Science
in Mechanical Engineering

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2019

DECLARATION

I declare that this thesis entitled "Hydrophobicity Performance of Polyethylene Terephthalate, Thermoplastic Polyurethane and Aluminium Substrates Using Self-Fabricated Contact Angle Tool" 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.

Signature	:	....
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Date	:	....

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 Mechanical Engineering.

Signature	:	....
Supervisor Name	:	....
Date	:	....

DEDICATION

To my beloved mother, father, siblings and my best friend

ABSTRACT

Electronic components have become increasingly important and critical especially when it comes to humid environment that potentially lead to corrosion which causes many damages including electrical stress, thermal expansion, mechanical vibration and reduction in production. Moisture that resides at the interface of the electronic components can result in interface delamination. Recent attention on Flexible Printed Circuit (PFC) to replace Printed Circuit Board (PCB) using Polyethylene Terephthalate (PET) and Thermoplastic Polyurethane (TPU) based substrate to avoid corrosion however, understanding on its behaviour is still at infant stage. In addition, the use of good water repellent coating using Titanium Dioxide ($TiO_{2)}$ coating is expected to further improve moisture resistance of electronic components due to its hydrophobicity behaviour. The performance of hydrophobicity can be analysed through contact angle analysis. This thesis is focus on the design and development of self-fabricated contact angle measurement tool for hydrophobicity analysis; to study the effect of temperature on hydrophobicity performance of PET, TPU and Aluminium (Al) substrates and the effect of ceramic coating (TiO_2) on PET and TPU. The self-fabricated contact angle measurement tool was developed using Process Design and Development (PDD) involved Pugh Method followed by One-Sample T-Test to verify the accuracy measurement of the selected design. The polymer substrates had undergone thermal analysis by using Differential Scanning Calorimetry (DSC). The TiO_2 was deposited by spin coating method and verified by using Fourier Transform Infrared Spectroscopy (FTIR) before heated at the selected heating temperatures (RT, 40ºC, 60ºC and 80ºC). The result on self-fabricated contact angle measurement tool found that this tool is reliable, consistent and precise for contact angle measurement for different surface conditions of hydrophilic (73.25°), hydrophobic (107.25°) and superhydrophobic (158.33°) surfaces. Moreover, the image of contact angle captured from this tool shows a very clear edge of water droplet which help to measure the contact angle accurately. The TPU, PET and Al have different hydrophobicity properties behaviour. PET and Al are hydrophilic meanwhile TPU as hydrophobic which is mainly due to the differences in surface energy. However, surface morphology also influenced the hydrophobicity behaviour when the substrates were modified through heat treatment that changed the geometrical structure or surface roughness of the surface in term of peak and valley (P-V) height, distance between peak-to -peak (D) and width of roughness protrusion (W). The changes of P-V, D and W has affected the formation of air trap between the solid-liquid interface which is the principle of Wenzel and Cassie-Baxter theory. TPU increase in contact angle (98° to 103°) while PET (75° to 70°) and Al (89° to 72°) decrease after thermally aged. The contact angle and surface roughness also show high correlation; PET (0.95), TPU (-0.90) and Al (-0.94) by using Pearson R Test. The TiO_2 coating has further improved the hydrophobicity of the PET and TPU (between 94° to 113°) with no evidence of P-V, D and W geometrical structure modification. The knowledge gained would be beneficial to extend the use of TiO_2 coating in electronic applications.

ABSTRAK

Komponen elektronik menjadi semakin penting dan kritikal terutamanya apabila berkaitan dengan persekitaran lembap yang berpotensi membawa kepada kakisan yang menyebabkan banyak kerosakan termasuk tekanan elektrik, pengembangan haba, getaran mekanikal dan pengurangan pengeluaran. Kelembapan yang terletak di antara komponen elektronik boleh menyebabkan pelekangan antara muka. Kini, perhatian pada Litar Bercetak Fleksibel (PFC) untuk menggantikan Papan Litar Tercetak (PCB) menggunakan substrat berasaskan Polyethylene Terephthalate (PET) dan Thermoplastic Polyurethane (TPU) untuk menghindari pengaratan namun kefahaman mengenai sifatnya masih lagi sedikit. Di samping itu, penggunaaan salutan dengan sifat tidak telap air yang baik menggunakan salutan Titanium Dioksida (TiO_2) dijangka akan meningkatkan lagi halangan kelembapan pada komponen elektronik kerana tingkah laku hidrofobisitinya. Prestasi hidrofobisiti boleh dianalisis menggunakan analisis sudut sentuhan. Tesis ini memberi tumpuan kepada reka bentuk dan mencipta alat pengukuran sudut sentuhan sendiri untuk hidrofobisiti analisis; untuk mengkaji kesan haba pada prestasi hidrofobiciti PET, substrat TPU Aluminium (Al) dan kesan salutan seramik (TiO_2) pada PET dan TPU. Alat pengukuran sudut sentuhan diri dibangunkan dengan menggunakan Proses Reka Bentuk dan Perkembangan (PDD) yang melibatkan Kaedah Pugh diikuti oleh One-Sample T-Test untuk mengesahkan ukuran ketepatan reka bentuk yang dipilih. Substrat polimer telah menjalani analisis termal dengan menggunakan Differential Scanning Calorimetry (DSC). TiO_2 disadurkan menggunakan kaedah putaran bersalut dan disahkan menggunakan Fourier Transform Infrared Spectroscopy (FTIR) sebelum dipanaskan pada suhu pemanasan yang dipilih (RT, 40ºC, 60ºC dan 80ºC). Hasilnya, alat pengukuran sudut sentuhan sendiri mendapati alat ini boleh dipercayai, konsisten dan tepat untuk pengukuran sudut sentuhan untuk permukaan hidrofilik (73.25°), hidrofobik (107.25°) dan superhidrophobik (158.33°C). Selain itu, imej sudut sentuh yang ditangkap dari alat ini menunjukkan bentuk titisan air yang sangat jelas yang membantu untuk mengukur sudut kenalan dengan tepat. TPU, PET dan Al mempunyai sifat tingkah laku hidrofobik yang berlainan. PET dan Al sebagai hidrophilik sementara TPU sebagai hidrofobik yang sebahagian besarnya disebabkan oleh perbezaan dalam tenaga permukaan. Walau bagaimanapun, morfologi permukaan juga mempengaruhi tingkah laku hidrofobisiti apabila substrat diubahsuai melalui pemanasan haba yang mengubah struktur geometri atau kekasaran permukaan dari segi ketinggian puncak dan lembah (PV), jarak antara puncak hingga puncak (D) dan lebar kekasaran penonjolan (W). Perubahan P-V, D dan W telah menjejaskan pembentukan perangkap udara antara muka pepejal-cecair yang merupakan prinsip teori Wenzel dan Cassie-Baxter. TPU meningkat pada sudut sentuh (98° hingga 103°) manakala PET (75° hingga 70°) dan Al (89° hingga 72°) berkurangan selepas dipanaskan. Sudut sentuhan dan kekasaran permukaan juga menunjukkan korelasi yang tinggi; PET (0.95), TPU (-0.90) dan Al (-0.94) dengan menggunakan Pearson R Test. Lapisan TiO_2 telah meningkatkan lagi hidrofobisiti PET dan TPU (antara 94° hingga 113°) tanpa sebarang pengubahsuaian struktur geometri P-V, D dan W. Pengetahuan yang diperoleh akan memberi manfaat untuk memperluaskan penggunaan salutan seramik dalam aplikasi elektronik.

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful

Alhamdulillah, all praises to Allah for the strengths and His blessing to me in completing this thesis. First and foremost, I would like to express my special appreciation and sincere gratitude to my supervisor, Prof. Dr. Ghazali Bin Omar from Faculty of Mechanical Engineering Universiti Teknikal Malaysia Melaka (UTeM) for his invaluable supervision, guidance, kindness, encouragement, and financial support throughout my study. I am also very thankful to my co-supervisor, Puan Anita Akmar Binti Kamarolzaman and Dr. Azmmi Bin Masripan for their advices, suggestion and motivation in evaluation of hydrophobicity study.

Special thanks to Ministry of Higher Education (MOHE) and UTeM for their short term grant funding (PJP/2016/FKM/HI1/S01463) as well as Jabil Curcuit Sdn. Bhd for the financial support throughout this project (GLUAR/JABIL/2016/FKM-CARE/I00014). I would like to express my gratitude to material laboratory technician from Faculty of Technology Engineering and Faculty of Manufacturing Engineering, Mr. Azizul Ikhwan and Mr. Farihan and also technician from Chemistry Laboratory Faculty of Mechanical Engineering for their assistance, time spent and effort in all the lab and analysis works. Not to forget my fellow postgraduate colleagues and lectures in Advance Materials Characteristic Laboratory (AMCHAL) for their support, invaluable help and for providing an excellent research atmosphere during the course of my Master candidature. My sincere appreciation also extends to all friends especially Rabiatul Ardawiyah who have provided assistance at various occasions.

My deepest gratitude to my parents Jasmee Bin Zali and A'fifah Binti Mohamed, family members and my best friend Siti Syahirah Binti Che Othaman for their unconditional love, continuous support, encouragement and prayers during my life and throughout this study. Lastly, thank you to everyone who had been to the crucial parts of realization of this project. Not forgetting, my humble apology as it is beyond my reach not personally mentioned those who are involved directly or indirectly. Thank you and Assalamualaikum.

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LIST OF ABBREVIATIONS

AR	-	Aspect Ratio
DSC	-	Differential Scanning Calorimetry
FTIR	-	Fourier Transform Infrared Spectroscopy
IPA	-	Isopropyl Alcohol
LED	-	Light Emitting Diode
MYR	-	Malaysian Ringgit
MSL	-	Moisture Sensitive Level
MSDS	-	Material Safety Data Sheet
OSHA	-	Occupational Safety and Health Administration
PCB	-	Printed circuit Board
PFC	-	Printed Flexible Circuit
PET	-	Polyethylene Terephthalate
PDMS	-	Polydimethylsiloxane
PVD	-	Physical Vapor Deposition
PDD	-	Product design and development
RPM	-	Revolutions per Minutes
SMT	-	Surface Mount Technology
SHS	-	Superhydrophobic Surface
SiC	-	Silica Carbide
TPU	-	Thermoplastic Polyurethane
TiO_2	-	Titanium Dioxide

LIST OF SYMBOLS

C	-	Coated
D	-	Distance between peak to peak
f_{LA}	-	Geometrical area of the liquid-air interfaces under the droplet (air trap)
f_{SL}	-	Geometry areas of solid-liquid interfaces (contact area between droplet-surface)
L	-	Liquid
PV	-	Peak-valley height
R_f	-	Roughness factor
R_{rms}	-	Root mean square roughness
S	-	Solid
T_g	-	Glass transition temperature
T_m	-	Melting point
T_r	-	Temperature of recrystallization
UC	-	Uncoated
V	-	Vapor
W	-	Width of roughness protrusion
γ_{lv}	-	Liquid-vapor interfacial
γ_{sv}	-	Solid-vapor interfacial
γ_{sl}	-	Solid-liquid interfacial tension
θ_Y	-	Contact angle
θ_w	-	Contact angle on the rough surface
θ_o	-	Contact angle on the smooth surface
Δ	-	Different of contact angle before and after coated

LIST OF PUBLICATIONS

The research papers produced and published and awards during the course of this research are as follows:

Journals and Conference papers:

1. Jasmee, S., Omar, G., Masripan, N.A.B., Kamarolzaman, A.A., Ashikin, A.S and Che Ani, F., 2018. Hydrophobicity Performance of Polyethylene Terephthalate (PET) and Thermoplastic polyurethane (TPU) with Thermal Effect, *Material Research Express*, 5 (9). (Published)

2. Jasmee, S., Omar, G., Kamarolzaman,A.A., Masripan, N.A.B., Kusnan, N.I., and Che Ani, F., 2017 Effects of Thermal Aging on Hydrophobicity for Polymer and Metallic Surface, *5th International Conferences and Exhibition on Sustainable Energy and Advanced Material (ICESEAM).* Melaka, Malaysia, 16-19 October 2017. (Accepted)

Proceeding Papers:

1. Jasmee, S., Omar, G., Masripan, N.A.B., and Kamarolzaman, A.A., 2018. Wettability of Aluminum-alloys Surface with Various Surface Roughness and

Thickness Coating. *Proceeding of the Innovative Research and Industrial Dialogue'18 (IRID)*, Melaka, Malaysia, 18 July 2018. (Accepted)

2. Jasmee, S., Omar, G., Nordin, M.N.A., Masripan, N.A.B. and Kamarolzaman, A.A., 2018, April. Hydrophobicity Performance of Thermoplastic Polyurethane Coated with Tio2 Under Thermal Aging Effect. In *1st Colloquium Paper: Advanced Materials and Mechanical Engineering Research (CAMMER'18)* (Vol. 1, p. 65). Penerbit Universiti, Universiti Teknikal Malaysia Melaka. (Published)

3. Jasmee, S., Omar, G., Kamarolzaman,A.A., Razali, N., Masripan, N.A.B., and Mansor M.R., 2018. Design and Development of Contact Angle Measurement Tools for Hydrophobicity Analysis. *Proceedings of Mechanical Engineering Research Day 2018 (MERD18)*, Melaka, Malaysia, 3 May 2018. (Published)

Awards;

1. Silver Award in *Innovation Carnival UTEMEX2017* for Project Title 'Development of Contact Angle Measurement Tools for Hydrophobicity Analysis'.

2. Best Poster Award for *Mechanical Engineering Research Day 2018 (MERD18)* for Proceeding Paper Tittle 'Design and Development of Contact Angle Measurement Tools for Hydrophobicity Analysis'.

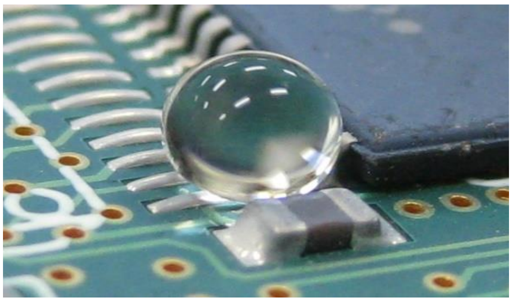
CHAPTER 1

INTRODUCTION

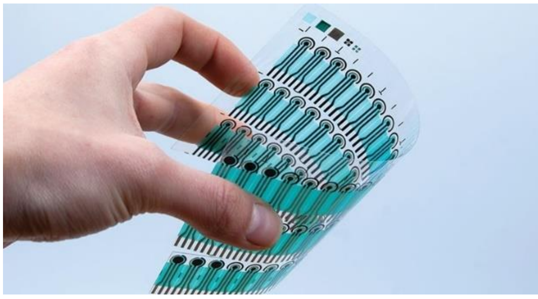
1.1 Research background

Electronics components and technology have become increasingly important in daily life and with such demand, the durability of the electronic component has become more crucial especially when it meets water/humidity in its operating environment. Electronic device is very well known for its sensitivity to water either vapor or liquid, where high absorption of water can degrade the performance of the electronic device (Walter et al., 2010). One of the main reasons is due to the moisture that can cause corrosion (Davis, 1999; Ghali, 2010). Moisture that resides at the interface of the Printed circuit board (PCB) or other electronic components can result in interface delamination (Yip and Jen, 2012). PCB is one of the elements that can be found in the electronic device, which connects the electronic or electrical component of the device.

Studies show that replacing PCB to become more flexible and versatile printed circuit called Printed Flexible Circuit (PFC) becomes the current attention among researchers in electronic industry (Ko et al., 2007; Rida et al., 2009; Wong and Salleo, 2009). Besides, researches also believe that combining the flexibility and stretchability of PFC such as Polyethylene Terephthalate (PET) and Thermoplastic Polyurethane (TPU) with good water repellent characteristics on coating known as the superhydrophobic coating can improve the functionality of printed circuit for electronic packaging (Mates et al., 2015). Water repellent characteristic is due to high hydrophobicity, which exhibits the self-cleaning effect called the lotus effect. The superhydrophobic coating also becomes one of the research areas that has been particularly interesting to protect electronics components from corrosive operating environments especially the temperature during the surface mounting process. The coating can be derived from many different materials. In the case of coating for water repellency, many researchers have found that the suitable candidates are from the family of ceramic such as Titanium dioxide (TiO_2) (Wang et al., 2014; Ortelli et al., 2015; Navid et al., 2016).



(a)



(b)

Figure 1.1: Example of (a) water repellent board (b) printed flexible circuit (PFC) (Mates et al.,

2015)

Sanjay et al. (2014) stated that there are two categories of technique to make superhydrophobic coating; making a rough surface from a low surface energy material and modifying a rough surface with a material of low surface energy. Roughening a coating enhances its hydrophobicity not only due to the increase in the solid-liquid interface but also when air is trapped on a rough surface between the surface and the liquid droplet (Adel et al., 2015). Bhushan and Jung (2011) also stated that the contact angle depends on several factors, such as surface energy, surface roughness, and its cleanliness.

The study of hydrophobicity involves the measurement of the contact angle that indicates the degree of wetting when a solid and liquid interact. There are three different wettability behaviors of surface known as hydrophilic (θ <<90º), hydrophobic (θ >>90º) and superhydrophobic (θ >>150º). Measurement of hydrophobicity surfaces plays an important role in many industrial processes due to their potential applications such as self-cleaning, nanofluidics, and electrowetting. The first invention of contact angle measurement tool named Ramè-hart contact angle telescope goniometer in the early 1960s was developed by W.A. Zisman (Yuan and Lee, 2013). Since then, there are more innovation of contact angle tools with more advance in technology and application such as Kruss DSA10, goniometer G1 and more. The invention also leads to more method to measure contact angle like sessile drop, capillary tube, drop shape analysis, and Young-Laplace to measure the hydrophobicity. However, the available tools to measure surface wettability are limited due to the high price and can be used in the lab only.

The focus of this study is to design and develop low-cost self-contact angle measurement tools with accuracy measurement validation. After completing the development and validation process, the tool is used to investigate the effect of temperature on polymer and metallic substrate and also the effect of ceramic coating TiO_2 on PFC polymer substrate which is PET and TPU. Characterizations of the heated and coated samples were carried out to determine the physical, thermal, chemical properties of the samples. The main hypothesis of this study is that the inclusion of TiO_2 on PFC would improve hydrophobicity performance of the surfaces. The knowledge that will be gained from this study is important to extend the application of PFC with water repellent coating.

1.2 Problem statement

Performance of hydrophobicity can be analyzed by characterizing the wetting behavior of the surface. It can be done by measuring the contact angle between liquid-liquid and solid-liquid interfaces. Contact angle measurement plays an important role in investigating the performance of wetting behavior. However, more detailed analysis is limited due to the high price of contact angle tools and most of the machines are complex to handle since equipped with high technology software and can be used in the laboratory only. Therefore, self-fabricated contact angle measurement tools equipped with advantageous features such as portability and easy to handle is needed in order to further understand the wetting behavior of different materials. This is because, different types of materials exhibit different types of wetting behaviors.

Current electronic industry is using PCB in electronic packaging, which consists of various types of metallic component including the PCB itself. Corrosion has caused many damages to these components including electrical stress, thermal expansion, mechanical vibration and reduction in production, which are mostly due to the heat and humidity. Recent attention is focusing on PFC to replace the PCB, which is polymer based that can avoid corrosion between the surface and electrical component. PET is the example of PFC that is widely explored by many researchers due to their flexibility attributes but has limitation to its rigidity stretchability. Because of that, the introduction of TPU in PFC industry has provided a lot of advantage especially due to its flexible and stretchable properties. However, the study of this PFC is still at infant stage which is important to have a better understanding of PET and TPU performance at any condition especially when heated. Apart from that, the used of PFC only cannot fully protect the surface under harsh environment particularly when contact with water or heat due to its low hydrophobicity. Because of that, the researcher also believes that combining the flexibility and stretchability of PFC with good water repellent characteristic on a coating that has good hydrophobicity can further improve the functionality of printed circuit for electronic packaging. The used of ceramic coating especially TiO_2 which has high hydrophobicity was recognized by many researchers to explore more its properties due to its outstanding performance in a wide range of application and chemical stability (Pawlowski, 2013). Compared to other ceramic coatings, TiO_2 also has low-cost production and able to create a layer with low electrical resistivity while maintaining the anti-corrosion in the electrochemical field (Berger, 2004). However, there are only a few studies dedicated on PFC combined with superhydrophobicity functionalities by using ceramic coating TiO_2 as a protective layer which can become more advantageous in the electronic field applications in the near future. Therefore, a comprehensive study is required to give an insight knowledge about the topic.

1.3 Objectives

The objectives of this study are:

1. To design and develop self-fabricated contact angle measurement tools for hydrophobicity analysis.
2. To study the hydrophobicity behaviour of polymer and metallic substrates with varying temperature.
3. To investigate the effect of ceramic coating Titanium Dioxide (TiO_2) on the hydrophobicity behaviour of the polymer substrates with varying temperatures.

1.4 Scope of research

The scope for this study is focusing on hydrophobicity performance of polymer (PET and TPU) and metallic (Al) substrates under a few test conditions.