



UNIVERSITI PUTRA MALAYSIA

***PROPERTIES OF SUGAR PALM/GLASS FIBRE-REINFORCED
THERMOPLASTIC POLYURETHANE COMPOSITES***

ATIQA H BINTI MOHD AFDZALUDDIN

IPTPH 2018 4



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By

ATIQA H BINTI MOHD AFDZALUDDIN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

April 2018

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DEDICATION

To

My parents

The most patience and supportive persons in my life who's always believe in her daughter's potential to finish for what she had started.

My beloved husband

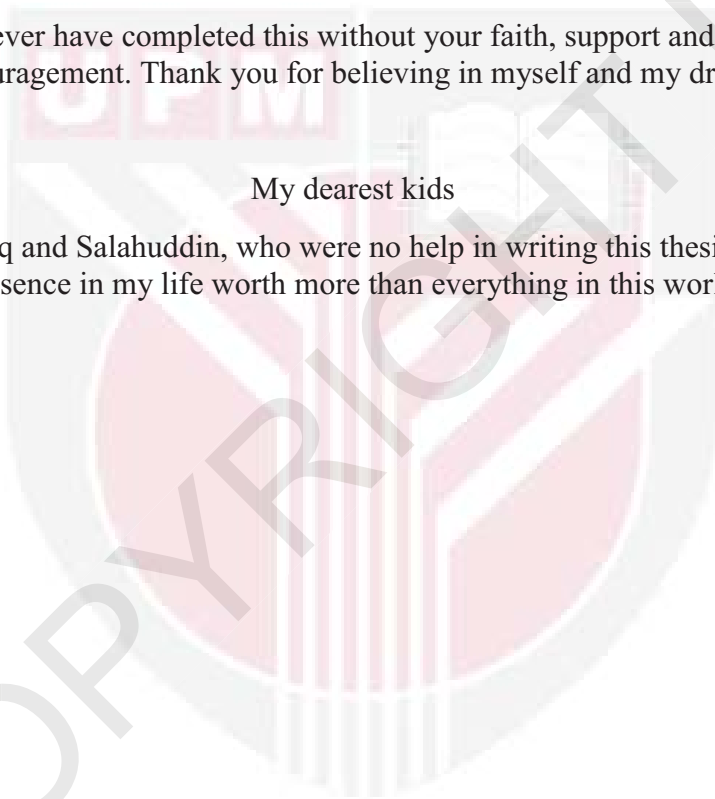
I could never have completed this without your faith, support and constant encouragement. Thank you for believing in myself and my dream.

My dearest kids

Fateh, Tariq and Salahuddin, who were no help in writing this thesis but their presence in my life worth more than everything in this world.



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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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THERMOPLASTIC POLYURETHANE COMPOSITES**

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ATIQA BINTI MOHD AFDZALUDDIN

April 2018

Chairman : Mohammad Jawaid, PhD
Faculty : Institute of Tropical Forestry and Forest Products

Sugar palm fibres are abundantly available in Malaysia and South Asian countries. In addition to the wide availability and the ease to grow and harvest the sugar palm, the properties of its fibres, such as high wear resistance, low cost of raw material, biodegradability, environmental friendliness are certain advantages. However, despite the advantages, they also have some drawbacks, for example, high water absorbance, low thermal stability and debonding between the polymer matrix and natural fibre, which cause limitations in utilizing natural fibres for different applications. Nonetheless, such drawbacks can be overcome by several modification methods were employed to strengthen the properties of natural fibres composites i.e (1) surface modification by using of 6% alkaline, 2% silane, and combined 6% alkaline-2% silane treatment, (2) fiber treatment and the reinforcement of thermoplastic polyurethane, (3) hybridization of sugar palm (SP) with glass (G) fibre, and (4) surface modification of treated sugar palm with glass fibre reinforced TPU hybrid composites. The findings show that the mechanical, morphological and structural properties of SP fibre were 2% silane treatment were compatible with TPU matrix. The influence of SP fibre at varying loading (0-50 wt%) on the physical, mechanical and thermal properties of TPU composites were evaluated. In term of physical properties, the density, water absorption and thickness swelling increases with increasing fibre loading. Conversely, the 2% silane treated were enhanced the physical properties of SP/TPU composites. Improvement in tensile, flexural, and impact properties of composites were reported for 2 % silane treated at higher SP fiber loading. Moreover, the treated SP fiber exhibited satisfactory values of storage modulus and thermal degradation. In addition, the effect of hybridization of SP with G fibres were also investigated. Hybridization of SP/G at weight ratio (30/10, 20/20, 10/30, and 0/40) were developed by melt-mixing compounding followed by hot pressing moulding. The findings indicated that hybridization of SP with G fibres enhanced the tensile, flexural, and impact properties of hybrid composites. The thermal stability were also improved in the SP/G/TPU hybrid composites. Lastly, the fiber treatment based SP/G/TPU hybrid composites

were fabricated and characterized its properties. In terms of physical properties, the obtained results showed that treated hybrid composites with combined 6% alkaline-2% silane exhibited the lower density, thickness swelling and water uptake. Mechanical properties (tensile, flexural and impact) of treated hybrid composites enhanced as compared to untreated hybrid composites. However, treated 30/10 SP/G hybrid composites showed moderate range of storage modulus, loss modulus, damping factor and good thermal stability. Thus, treated hybrid 30/10 SP/G/TPU showed better physical, mechanical and thermo mechanical properties over untreated hybrid composites and it is suitable for light weight density and structural applications which can be recommend for automotive parts.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SIFAT-SIFAT POLIURETANA TERMOPLASTIK BERTETULANG GENTIAN ENAU/KACA

Oleh

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Gentian Pokok Enau mudah didapati di Malaysia dan negara-negara Asia Selatan. Selain mudah ditanam dan dituai, sifat gentian pokok enau seperti tahan haus, kos bahan mentah yang rendah, keterbiodegradasikan dan mesra alam juga merupakan kelebihanannya. Namun terdapat juga kekurangan; gentian ini memiliki kadar serapan air yang tinggi, kestabilan terma yang rendah dan penyahikatan antara matriks polimer dan gentian semula jadi, yang mengehadikan penggunaan gentian semula jadi tersebut dalam pelbagai aplikasi. Walau bagaimanapun, kekurangan ini boleh diatasi melalui beberapa kaedah pengubahsuaian bagi mengukuhkan sifat komposit gentian semula jadi. Antaranya, (1) pengubahsuaian permukaan menggunakan rawatan 6% beralkali, 2% silana dan gabungan 6% alkali-2% silana, (2) rawatan gentian dan pengukuhan poliuretana termoplastik, (3) penghibridan gentian enau (SP) dengan gentian kaca (G), dan (4) pengubahsuaian permukaan gentian enau terawat dengan komposit hibrid TPU bertetulang gentian kaca. Dapatan menunjukkan bahawa sifat mekanik, morfologi dan struktur gentian enau (SP) dengan rawatan 2% silana serasi dengan matriks TPU. Pengaruh gentian SP pada bebanan yang berlainan (0-50 wt%) terhadap sifat fizikal, mekanik dan terma komposit TPU dinilai. Daripada segi sifat fizikal, ketumpatan, penyerapan air dan pengembangan ketebalan bertambah dengan peningkatan bebanan gentian. Sebaliknya, rawatan 2% silana mempertingkatkan sifat fizikal komposit SP/TPU itu. Peningkatan dalam sifat tegangan, lenturan dan impak komposit dilaporkan selepas rawatan 2% silana pada bebanan gentian SP lebih tinggi. Tambahan pula, gentian SP terawat mempamerkan nilai modulus simpanan dan degradasi terma yang memuaskan. Di samping itu, kesan penghibridan SP dengan gentian G juga dikaji. Penghibridan SP/G pada nisbah berat (30/10, 20/20, 10/30 dan 0/40) dibangunkan dengan penyebatian pencampuran lebur yang diikuti dengan pengacuan kempa panas. Dapatan menunjukkan bahawa penghibridan SP dengan gentian G mempertingkat sifat tegangan, lenturan dan hentaman komposit hibrid itu. Kestabilan terma juga dipertingkatkan dalam komposit hibrid SP/G/TPU. Akhir sekali, komposit hibrid rawatan gentian SP/G/TPU ini dicipta dan sifatnya

digambarkan. Daripada segi sifat fizikal, keputusan yang didapati menunjukkan bahawa komposit hibrid yang dirawat dengan gabungan 6% alkali-2% silana mempamerkan ketumpatan yang lebih rendah, pengembangan ketebalan dan penyerapan air. Sifat mekanik (tegangan, lenturan dan impak) komposit hibrid terawat bertambah bagus dibandingkan dengan komposit hibrid tidak terawat. Bagaimanapun, komposit hibrid SP/G 30/10 terawat menunjukkan julat sederhana modulus simpanan, modulus kehilangan, faktor redaman dan kestabilan terma yang baik. Dengan demikian, hibrid SP/G/TPU 30/10 terawat menunjukkan sifat fizikal, mekanik dan mekanik terma yang lebih baik berbanding komposit hibrid tidak terawat dan sesuai untuk kegunaan struktur dan ketumpatan ringan di bahagian automotif.



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I certify that a Thesis Examination Committee has met on 11 April 2018 to conduct the final examination of Atiqah binti Mohd Afdzaluddin on her thesis entitled "Properties of Sugar Palm/Glass Fibre- Reinforced Thermoplastic Polyurethane Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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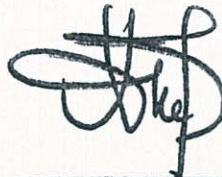
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LIST OF ABBREVIATIONS

ISO	Isosorbide
pMDI	Polymeric Methylene Diphenylisocyanate
RO	Rapeseed Oil
ASTM	American Society For Testing And Materials
BDO	1,4-Butanediol
DEA	Diethanolamine
DMA	Dynamic Mechanical Analysis
DTG	Derivative Thermogravity
E'	Storage Modulus
E''	Loss Modulus
EFB	Empty Fruit Bunch
FTIR	Fourier Transform Infrared Spectroscopy
G	Glass
GL	Glycerol
HMDI	Hexamethylene Diisocyanate
IFSS	Interfacial Shear Strength
MDI	4, 4' Diphenylmethane Diisocyanate
NCO	Isocyanate
NFC	Natural Fibre Composites
OH	Hydroxyl
OPEFB	Oil Palm Fruit Bunch
PAN	Polyacrylonitrile
PEG	Polyethylene-Glicol
PLA	Poly Vinyl Chloride
PU	Polyurethane
SEM	Scanning Electron Microscopy
SGF	Short Glass Fibre
SIPNs	Semi-Interpenetrating Polymer Networks
SOPEP	Soybean Phosphate Ester Polyol
SP	Sugar Palm
SPF	Sugar Palm Fibre

Tan δ	Tan Delta
TDI	Diisocyanate
TEA	Triethanolamine
T_g	Glass Transition Temperature
TGA	Thermogravimetric Analysis
TMP	Thermo Mechanical Pulp
TNSP	Treated 6 % Alkaline Sugar Palm
TNSSP	Combined Treated 6% Alkaline-2% Silane Sugar Palm
TPSO	Thevetiaperuviana Seed Oil
TPU	Thermoplastic Polyurethane
TSSP	Treated 2% Silane Sugar Palm
UTSP	Untreated Sugar Palm
VARTM	Vacuum Assisted Resin Transfer Molding

CHAPTER 1

INTRODUCTION

1.1 Introduction and Background

Polymer composites materials with specific characteristics for a specific application, with an added value of environmentally friendly materials, have gained a lot of attention due to the limited availability of the resources (Shaniba et al., 2010; Väisänen et al., 2016). Due to the market demand, the research on composites using natural fibres as the reinforcing filler and biodegradable polymers as matrices are also gaining a lot of attention. At the present time, synthetic polymers were combined with various reinforcing fillers to improve the properties and to obtain the characteristics demanded in actual applications (Koronis et al., 2013). Polyurethanes (PU) is one of the most extensively used plastics both in developed and developing countries due to its advantages regards to the economical factor (price), the ecological (recycling behaviour), and technical requirements (higher thermal stability). On-going research is currently conducted to replace synthetic fibres with lingo-cellulosic fibres as reinforcing fillers. Compared to talc, silica, glass, carbon and other synthetic fibres, the lingo-cellulosic fibres (corn stalk, rice husk, rice straw, jute, abaca, sawdust, wheat straw, and grass) are lightweight, able to reduce wear on the machine used for their production, easily available, renewable, and inexpensive (Swolfs et al., 2014; Thakur et al., 2014b).

In the automotive industry, the definition of an environmental-friendly vehicle represents a vehicle with low carbon emission during operation. In-line with this development, there is a rising demand for light-weight vehicles where some of the metal components can be replaced with composites. Carbon fibre composites have been well-equipped in limited-edition cars, for instance. Composites originated from natural resources are highly in demand similar to other composites in the current market. It is to be expected in future, natural fibre composite (NFC) market reach \$ 3.8B and it implies that there is a healthy grow in NFC demand (Brief, 2011). Although the market performance is still lower than steel, the improvement on the materials will increase the growth of natural fibre composites in various applications. An automotive components inside a vehicle made of steels which have been proven to be reliable in terms of performance. However, due to aggressive efforts towards reducing the vehicle weight to meet more stringent automotive policies by the regulatory body worldwide, lighter and renewable materials such as natural-based polymer composite, is an excellent material substituting the traditional engineering material to develop a similar component. Natural-based polymer composite or biocomposite exhibits a significant weight reduction compared to steel, and while being a renewable material, biocomposite is able to meet the automotive policy which is to increase the recyclability percentage of components in a vehicle (Al-Oqla et al., 2016; Rwawiire et al., 2015). Thus, in this research, the potential of using natural-

synthetic hybrid composites to manufacture the automotive interior part will be explored.

Sugar palm tree is a promising source of natural fibres which are abundantly available in Southeast Asia, particularly in Malaysia (Sanyang et al., 2016). These fibres are used in many applications due to its superior strength and durability. Among the natural fibres, sugar palm fibres are well known for its high durability and their resistance to seawater (Mukhtar et al., 2016). These two characteristics are the main advantages of sugar palm fibres. Bachtiar et al. (2011) have developed the sugar palm fibre reinforced high impact polystyrene (HIPS) composites and studied its properties, resulting in an improvement on the properties after treatment conducted. Another investigation was carried out by Ishak et al. (2013) on sugar palm fibres, its polymers and composites, in which a number of advantages were observed in order to develop composites materials using the sugar palm fibre (Ishak et al., 2013). Research conducted by Bachtiar et al. (2012b) on untreated short sugar palm fibre/HIPs composites subjected to flexural, impact, and thermal properties, found that the impact performance of these natural fibre composites is poor. The development of the hybrid composites provides a new perspective to the manufacturing industry to reduce the dependency on synthetic fibres which often related to high potential hazard, particularly for human and the environment as well.

1.2 Problem statement

There are many advantages associated with the usage of natural fibres as the composite materials. Most natural fibre composites provide a healthier working condition than the synthetic fibre composites. The trimming, cutting, and mounting of synthetic fibre components produces dust in which causes skin irritation and respiratory diseases to human beings. Besides, natural fibres are less abrasive in nature compared to synthetic fibres. Therefore, natural fibres that offer good thermal and insulating properties are easily recyclable and biodegradable. However, natural fibre composites also have its own disadvantages such as low mechanical properties, low impact strength, poor moisture resistance, poor microbial and fire resistance, and low durability. In order to overcome these flaws, natural fibre can be combined with a stronger synthetic fibre in the same matrix to produce hybrid composites.

Sugar palm fibres (SPF) as compared to other natural fibres on the basis of cost, abundant, local issue were reported (Ishak et al., 2013; Sahari et al., 2012; Sanyang et al., 2016). It is known for its high durability and resistance to seawater. These two properties of using SPF as reinforcement are not based on excellent characteristics, but also on its fast-growing palm in topical regions especially in South East region of Asia. Though, SPF also like natural fibres, faces poor adhesion between hydrophobic polymers that lead to poor properties of the composites. Therefore, by proper fibre treatment before fabricating the composites can improve the properties of the material.

Hybridization of natural fibre with stronger and more corrosion-resistant synthetic fibre, for example, glass fibre or carbon fibre, can also improve the stiffness, strength, as well as the moisture resistant behaviour of the composite. Automotive industries are the main industries that incorporate the use of natural fibres because of the material properties which are lightweight, high strength-to-weight ratio, and minimum environmental impact. The advantages of using one type of fibre could complement what are lacking in using two or more types of different fibres in a hybrid composite. As a result, a balance in performance and cost could be achieved through a proper material design. However, only a few studies on the properties and characterisation of natural/synthetic fibre reinforced polymer hybrid composites are available today, and mostly, cases on surface treatment are not well-addressed.

A proper weight fraction of natural fibre (such as sugar palm fibre) and synthetic fibre (such as glass) can possibly develop a hybrid composite which can match the thermo-mechanical properties similar to the synthetic fibre reinforced composite with a significant amount of weight and surface treatment. The SPF treatment with NaOH and silane could significantly increase the interfacial bonding between fibre/matrix and ultimately improve the overall performance of hybrid composites. Biocompatibility and thermal stability are the main issues in which can inspire the new formulation of hybrid composites towards the benefits of incorporating biocomposites.

Therefore, the aim of this study is to develop an innovative formulation of untreated and treated sugar palm and glass (SP/G) fibre hybrid composites for a potential application in automotive parts. With this aim, the physical, mechanical and thermal properties of untreated and treated hybrid composites and hybrid composites were compared to find best suitable hybrid composites for automotive parts. Finally, it is expected that the utilisation and usage of SP/G/TPU hybrid composites would lead to the fabrication of biocomposites for automotive part applications.

1.3 Research Objectives

1. To characterise the effect of alkaline and silane treatment on physical, mechanical, and morphological properties of sugar palm fibres
2. To investigate the effect of sugar palm fibre loading on physical, mechanical, and thermal properties of thermoplastic polyurethane composites
3. To evaluate the effect of silane treatment on physical, mechanical, and thermal properties of sugar palm fibre/thermoplastic polyurethane composites
4. To evaluate the effect of surface modification on physical, mechanical, and thermal properties of sugar palm/glass reinforced thermoplastic polyurethane hybrid composites
5. To develop and investigate the effect of glass fibre loading on the physical, mechanical, and thermal properties of sugar palm/thermoplastic polyurethane hybrid composites

1.4 Significance of study

1. The findings from the current study are expected to improve the knowledge in developing composites from sugar palm fibre and thermoplastic polyurethanes.
2. The development of renewable materials with improved properties in this study is expected to aid in addressing the environmental problems regarding the alternative material for petroleum-based materials.
3. The problems associated with the properties of natural fibres could be improved by introducing treatment of the natural fibres.
4. In terms of hybrid composites, this study will explore the new potential materials of sugar palm/glass fibres reinforced thermoplastic polyurethane composites for different applications.
5. In addition, this study also employs fibre treatments for the development of hybrid composites. Thus, added a significance study for the treatment of natural fibres and its effect on the improvement of fibre/matrix interfacial bonding of composites.

1.5 Scope of study

In this research, fibre modification made of sugar palm fibre was performed with various treatment such as using alkaline 6%, silane 2%, and the combination of alkaline 6%-silane 2% for three hours. The characterisations of both physical and mechanical properties were then performed and the most effective fibre treatment was further investigated for the development of sugar palm/TPU composites. The development of SP/TPU composites were fabricated through melt-mixing compounding, followed by hot pressing moulding. The characterisation of their physical, mechanical, and thermal properties was performed. The best SP fibre loading were then used to be treated with the most effective fibre treatment in order to investigate the effect of fibre treatment on SP/TPU composites. Besides, the fibre loading for the SP/TPU was also used to be substituted with the hybrid composites. The physical, mechanical, and thermal properties of the untreated and treated SP/TPU composites were investigated. The hybridization of the composites was carried out by substituting sugar palm and glass fibre as the reinforcement and the thermoplastic polyurethane as the matrix. The physical, mechanical, and thermal properties were also investigated for comparison. The optimum properties of hybrid composites were then undergone further pre-modification of sugar palm fibre. The properties such as physical, mechanical, and thermal properties again were observed. The potential of the properties of the hybrid composite was evaluated as a new potential material to be used in automotive part applications.

1.6 Thesis organization

This thesis is organised in accordance with the alternative thesis format of Universiti Putra Malaysia, which is based on the publications of this study. Each research chapter represents a separated study inclusive of introduction, methodology, results and discussion, and conclusions. The details of the thesis structure are as follows:

Chapter 1

The problems that initiate this research and the research objectives were clearly highlighted in this chapter. The significance of this work and the scope of the study were also explained in details in this chapter.

Chapter 2

This chapter presents the a comprehensive literature review on the areas related to the topic of the thesis. Moreover, the research gaps obtained from the review were also clarified in this chapter.

Chapter 3

This chapter presents the methodology used in this study for the preparation of materials, testing procedure, and data collection.

Chapter 4

This chapter presents the second article entitled “Effect of Alkali and Silane Treatments on Mechanical and Interfacial Bonding Strength of Sugar Palm Fibres with Thermoplastic Polyurethane”. In this article, the properties of the sugar palm fibre for both untreated and treated composites were investigated.

Chapter 5

This chapter presents the third article entitled “**Moisture Absorption and Thickness Swelling Behaviour of Sugar Palm Fibre Reinforced Thermoplastic Polyurethane**”. The influence of fibre loading (0-50 wt%) of sugar palm fibre on physical properties (density, thickness swelling, and water absorption) of thermoplastic polyurethane was evaluated.

Chapter 6

This chapter presents the fourth article entitled “Mechanical and Thermal Properties of Sugar Palm Fibre Reinforced Thermoplastic Polyurethane Composites: Effect of Silane Treatment and Fibre Loading”. In this article, the effect of silane treatment and fibre loading (0-50 wt%) of sugar palm fibre reinforced thermoplastic polyurethane was investigated in terms of mechanical and thermal properties.

Chapter 7

This chapter presents the fifth article entitled “**Effect of Surface Treatment on Mechanical Properties of Treated Sugar Palm/Glass Fibre Reinforced Thermoplastic Polyurethane Hybrid Composites**”. In this article, the effect of sugar palm/glass fibre reinforced thermoplastic polyurethane treatment on mechanical properties were investigated.

Chapter 8

This chapter presents the “**Physical Properties of Treated Sugar Palm Fibre Reinforced Thermoplastic Polyurethane**”. The physical properties of silane treatment on sugar palm fibre reinforced thermoplastic polyurethane were characterise in this article.

Chapter 9

This chapter presents the “**Physical and Mechanical Properties of Sugar Palm/Glass Fibre Reinforced Thermoplastic Polyurethane Hybrid Composites**”. The effects of sugar palm/glass fibre loading (30/10, 20/20, 10/30, and 0/40 wt%) on physical and mechanical properties were evaluated.

Chapter 10

This chapter presents the “**Physical and Thermal Properties of Treated Sugar Palm /Glass Fibre Reinforced Thermoplastic Polyurethane Composites**”. The effect of surface modification on physical (density, thickness swelling, water absorption) and thermal (DMA and TGA) properties were characterised in this article.

Chapter 11

This chapter presents the overall conclusions from the whole study and future recommendations for further improvement of this study.

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