UNIVERSITI PUTRA MALAYSIA

MECHANICAL AND THERMAL PROPERTIES OF THERMOPLASTIC POLYURETHANE REINFORCED WITH ROSELLE AND SUGAR PALM HYBRID COMPOSITES

MOHD RADZI BIN ALI

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By

MOHD RADZI BIN ALI

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

May 2019

DEDICATION

To my beloved father and mother

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To my beloved wife

&

To my beloved daughters and son

&

To my awesome 2 siblings

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

MECHANICAL AND THERMAL PROPERTIES OF THERMOPLASTIC POLYURETHANE REINFORCED WITH ROSELLE AND SUGAR PALM HYBRID COMPOSITES

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May 2019

Chairman : Mohd Sapuan Bin Salit, PhD
Faculty : Institute of Tropical Forestry and Forest Products

Recently, with the increasing environmental concerns of many researchers conduct various research activities and efforts to address environmental issues by replacing the conventional fibres (synthetic fibres) to natural fibres as more environmentally friendly in polymer composites. The advantages of natural fibres in polymer composites are low cost, light weight, biodegradable and have good mechanical properties. In Malaysia, there are about 800-1200 hectare roselle trees that have been planted. With the size of this plantation, dumping cut trees will cause problems for farmers for disposal. The easiest way to dispose for them is to do open burning. The disposal of roselle trees will cause the effects of natural pollution. Therefore, roselle trees are selected for use in composites to reduce environmental problems. Additional, this research focuses on roselle and sugar palm plant to produce hybrid composites. Hybridization two types of natural fibres due to RF has high tensile strength and low fibre cost. SPF has good mechanical properties especially in terms of impact. These two criteria are combined to further enhance the mechanical and thermal properties of TPU composites. In additional, this research aims on capability performances and sustainability of roselle and sugar palm fibre as the reinforcement materials for polymer hybrid composites. Hence, several modifications were carried out to improve the mechanical and thermal properties of RF and SPF reinforced thermoplastic polyurethane hybrid composites. The purposes of this research consist of four parts. In the first part, roselle fibre (RF)/reinforced thermoplastic polyurethane (TPU) composites were prepared with different fibre sizes i.e. 125 μm and lower, 125–300 and 300–425 μm using the internal mixer and hot compression. The finding shows that mechanical properties were improved with increasing of the fibre sizes. The thermal properties were improved as the fibre size increased. The second part, the influence of fibre varying contents (0 – 50 wt. %) on the mechanical and thermal stability of RF/TPU composites were investigated. From the results, the effects of fibre contents show improved in tensile, flexural and impact test. Besides that, the thermal properties of RF/TPU composites

with different fibre contents had improved the thermal stability. The third part, the effects of hybridization of RF/sugar palm fibre (SPF)/TPU composites. Hybridization of RF/ SPF at weight ratio 75:25, 50:50, and 25:75 were performed using hot compression. Obtained results indicated that hybridization of RF/SPF/TPU hybrid composites increased the impact strength with increasing of sugar palm fibre contents. Otherwise, it gave effects on reduction of the tensile and flexural properties. In addition, the thermal properties of RF/SPF/TPU hybrid composites were improved following with incorporation of sugar palm fibres. Finally, the effect of surface treatment RF/SPF/TPU hybrid composites on mechanical and thermal properties. Roselle and sugar palm fibres are treated with alkalization agent. From the results, the treated RF/SPF/TPU hybrid polyurethane composites fibres shows increased in the mechanical properties of TPU composites compared to untreated. The highest tensile is 14.26 MPa, flexural strength is 14.05 MPa and impact strength is 23.76 kJ/m^2 was obtained from treatment 6 % NaOH concentration on RF/SPF hybrid composites. Physical properties showed lower water uptake of the treated thermoplastic polyurethane hybrid composites. The lowest water uptake is 7.97 % and thickness swelling is 6.49 % obtained from 9 % NaOH concentration after soaked in water for 7 days. The thermal properties also improved the thermal stability. Overall, RF/SPF/TPU hybrid composites have the potential to be an alternative material in polymer composites products to compete with the conventional fibres. Besides, it can reduce the problem of dumping and open burning on the environment. In addition, this hybrid composite can be applied to the automotive parts especially as a battery holder and liner.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SIFAT MEKANIKAL DAN TERMAL KOMPOSIT HIBRID POLIURETANA TERMOPLASTIK DIPERKUAT GENTIAN ROSEL DAN IJUK

Oleh

MOHD RADZI BIN ALI

Mei 2019

Pengerusi : Mohd Sapuan Bin Salit, PhD
Fakulti : Institut Perhutanan Tropika dan Produk Hutan

Baru-baru ini, dengan peningkatan isu alam sekitar ramai penyelidik menjalankan pelbagai aktiviti penyelidikan dan usaha untuk menangani isu alam sekitar dengan menggantikan serat konvensional (gentian sintetik) kepada serat semulajadi sebagai bahan utama yang lebih mesra alam di dalam komposit polimer. Kelebihan gentian semulajadi ini adalah rendah kos, ringan, biodegradasi dan mempunyai sifat mekanikal yang baik. Di Malaysia, terdapat kira-kira 800-1200 hektar pokok roselle yang telah ditanam. Dengan saiz ladang ini, lambakan pokok pelupusan akan menyebabkan masalah bagi petani untuk dilupuskan. Cara paling mudah untuk membuangnya ialah melakukan pembakaran terbuka. Oleh itu, proses pelupusan pokok roselle akan menyebabkan kesan pencemaran alam. oleh itu, pokok roselle dipilih untuk dijadikan composites untuk mengurangkan masalah persekitaran. Tambahan, penyelidikan ini memberi tumpuan kepada pokok roselle dan Enau untuk dihasilkan komposit hibrid. Hibridisasi kedua-dua jenis serat semulajadi ini adalah kerana RF mempunyai kekuatan tegangan dan kos bahan yang rendah dan SPF mempunyai sifat mekanikal yang baik terutamanya kekuatan impak. Kedua-dua sifat mekanikal ini digabungkan untuk meningkatkan kekuatan mekanikal dan terma ke atas komposit TPU. Tambahan, penyelidikan ini bertujuan untuk menilai keupayaan dan kelestarian serat roselle (RF) dan Ijuk (SPF) sebagai bahan pengukuhan untuk komposit hibrid polimer. Oleh itu, beberapa pengubahsuaian telah dilakukan ke atas RF dan SPF untuk meningkatkan sifat-sifat mekanikal dan terma atas poliuretana termoplastik komposit hibrid. Tujuan penyelidikan ini terdiri daripada empat bahagian. Pada bahagian pertama, RF/ diperkuat poliuretana termoplastik (TPU) komposit kesan terhadap pelbagai saiz serat seperti 125 μm dan lebih rendah, 125-300 dan 300-425 μm dengan menggunakan pengadun dalaman dan pembentukan mampatan. Hasil kajian menunjukkan bahawa sifat-sifat mekanik bertambah baik dengan peningkatkan saiz serat. Ciri-ciri termal bertambah baik apabila saiz gentian meningkat. Bahagian kedua, kesan perbezaan kandungan serat roselle (0 - 50 wt.%) pada sifat mekanik dan kestabilan termal diperkukuhkan dengan poliuretana termoplastik. Dari keputusan, kesan kandungan serat menunjukkan peningkatan tegangan, lenturan dan impak. Selain itu, sifat termal komposit RF/TPU dengan kandungan serat yang berbeza telah meningkatkan

kestabilan terma. Bahagian ketiga, kesan hibridisasi komposit RF/SPF/ TPU. Hibridisasi RF/ SPF pada kadar berat 75:25, 50:50 dan 25:75 telah disediakan. Hasil yang diperolehi menunjukkan bahawa, hibridisasi komposit RF/SPF telah meningkatkan kekuatan impak dengan peningkatan peratusan kandungan SPF. Malah, ia memberi kesan penurunan ke atas sifat tegangan dan lentur. Di samping itu, sifat terma hibridisasi komposit RF /SPF/TPU bertambah baik berikutan penggabungan dengan SPF. Akhir sekali, kesan rawatan pemukaan gentian hibrid komposit RF/ SPF ke atas sifat mekanikal dan termal. RF dan SPF dirawat dengan agen alkali. Dari hasilnya yang diperolehi, komposit hibrid RF/SPF/TPU yang dirawat menunjukkan peningkatan sifat mekanik berbanding dengan tidak dirawat. Ujian tegangan tertinggi ialah 14.26 MPa, kekuatan lenturan adalah 14.05 MPa dan kekuatan impak ialah 23.76 kJ/m^2 diperoleh daripada kepekatan 6% NaOH pada komposit hibrid RF / SPF. Sifat fizikal menunjukkan penyerapan air yang lebih rendah daripada komposit hybrid TPU yang dirawat. Penyerapan air terendah ialah 7.97% dan ketebalan bengkak adalah 6.49% diperolehi daripada kepekatan NaOH 9% selepas rendaman selama 7 hari. Ciri-ciri terma juga menunjukkan kestabilan yang lebih baik. Secara keseluruhannya, komposit hibridisasi RF/SPF/TPU mempunyai potensi untuk dijadikan bahan alternatif di dalam komposit polimer untuk menyaingi dengan serat konvensional. Selain itu, ia dapat mengurangkan masalah pembuangan dan pembakaran terbuka terhadap alam sekitar. Tambahan juga, komposit hibrid ini boleh diaplikasikan ke produk automotif khususnya sebagai pemegang dan pelapik bateri

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Thank you.

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature: _____
Name of Chairman Supervisory Committee:
Professor Ir. Dr. Mohd Sapuan bin Salit

Signature: _____
Name Member of Supervisory Committee:
Dr. Mohammad Jawaid

Signature: _____
Name Member of Supervisory Committee:
Dr. Muhd Ridzuan bin Mansor

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BSC	Sugar cane bagasse
DEG	Ethylene glycol
DMA	Dynamic mechanical analyser
DSC	Differential scanning calorimetric
FTIR	Fourier Transform Infrared
KL	Kraft lignin
MDI	Diphenylmethylene diisocyanates
NaOH	Sodium hydroxide
PTMG	Polytetramethylene ether glycol
PU	Polyurethane
RF	Roselle fibre
RSO	Rubber seed oil
RSOPU	Rubber seed oil-based polyurethane
SEM	Scanning electron microscope
SPF	Sugar palm fibre
T	Thickness
TDI	Toluene diisocyanates
TG	Glass transition temperature
TGA	Thermal-gravimetric analysis
TPU	Thermoplastic polyurethane
UV	Ultra violet
W	Weight

CHAPTER 1

INTRODUCTION

1.1 Background

Recently, the development of environment friendly products has been growing rapidly due to accumulating of biodegradable waste on the landfill. Besides, polymer industries produced the product from synthetic fibres obtained from fossil fuels. Most of the conventional materials are non-degradable and non-renewable and they are hazardous upon disposal. Therefore, various efforts have been made to develop green materials either by recycling or from natural resources that can solve some problems associated with environmental pollution. Nowadays, the development of environmentally friendly materials is increasing and becoming the focus of researchers to replace the synthetic based fibres and composites. Natural fibre products are biodegradable and inexpensive, this composite contributes to the century due to environmental problems. Also, composite materials offer solution for waste disposal problems related to petroleum-derived plastics. Various types of natural materials have been used such as in the forms of fibres, celluloses and starch to be used as major constituents in composite materials. Of all these sources, natural fibre is the most widely used and it can be found in abundance (Mohammed et al.,2015; Sapuan et al.,2015).

Roselle fibres (RF) and sugar palm fibres (SPF) are natural fibres, which have the potential to be used as reinforcements in polymer composites. Both fibres can easily be found in abundance in Malaysia, Thailand and Indonesia. RF are used in the form of bast fibres, while SPF have their own natural fibres normally being wrapped around the trunk of the trees. Both fibres have good mechanical and thermal properties (Chandramohan & Marimuthu, 2011b; Ishak et al., 2013)

Therefore, this research presents the investigation of various aspects of RF and SPF reinforced thermoplastic polyurethane (TPU) hybrid composites such as the determination of mechanical, morphology and thermal properties. Both fibres were also treated with alkaline treatment (NaOH). The research also investigated the effect of different fibre treatments on mechanical and thermal properties using different approaches. The purpose of fibre treatment process is to improve mechanical and thermal properties of RF/SPF fibre hybrid composites.

1.2 Problem statements

Nowadays, producing and study of new materials focuses on the enhancement of mechanical and physical properties, especially in environmentally friendly materials.
This study involved composites using natural fibres with new effects and taking advantage of each properties to create better new material in generally.

Composite-based products are widely applied in various industries such as furniture, automotive and others. In addition, the manufacture of composite materials can conserve energy as well as demand where the materials produced need to have features such as high characteristics properties, improvement material design and lightweight conversion.

The main causes of environmental pollution are open burning of the agricultural from farmers without supervision of the authorities. Dumping mature trees and there is no yield that need to be cut down and replaced with new trees. This plant will often be burned by farmers to clear their crops. One of the plants to be cut down and burned is the roselle tree. According to the website Anim Agro Technology, in Malaysia, there are about 800-1200 hectare roselle trees that have been planted. Therefore, roselle plants are planted around 7400 trees per hectare in the mineral (Nadlene et al.,2016a). With the size of this plantation, dumping cut trees will cause problems for farmers for disposal. The easiest way to dispose for them is to do open burning. Later, the combustion residue will normally be used as fertilizer for a new crop. Thus, the process of disposing of roselle trees will cause one of the effects of natural pollution.

In addition, these plastic materials cannot be dissolved separately when disposed into the sea or land. Uncontrollable abandonment will bring harmful to humans and animals and can cause odour and open burning. Additionally to the remnants of the tree cutting waste will also be burned on a large scale. Such behaviour will also bring pollution to air and odour. This problem has attracted the attention of many countries, where the government has banned the disposal and open burning of plastic waste as an effort to curb environmental pollution and to address such issues. Therefore, this research attempt to solve the environmental pollution issue by developing renewable on polymer materials. Indirectly, studies on new composites materials on enhancing mechanical and thermal properties and produce environmentally friendly materials.

The manufacture of hybrid composites product has been widely applied in the automotive and aircraft industries. The production of this material is to produce lightweight products and have good mechanical properties (Panthapulakkal & Sain, 2007). Currently, research on hybrid composite studies using natural fibres as a source has become a focus among researchers. These natural fibres source is easy to find is one of the reasons why it is used as a reinforcing agent in polymer materials. In addition, the advantages of this fibre are more environmentally friendly than conventional fibres. Besides, advantages of the natural fibres are the low cost of materials and manufacturing, environmentally friendly and good in mechanical and thermal properties (Essabir et al., 2016; Idiculaet al., 2005; Saw et al., 2012).

RF and SPF are the natural fibres that can easily found in tropical areas. Usually, these fibres used to produce sweepers, ropes and textile. Both types of this natural fibres have fruits that can be as a food sweeteners, sugar block (gula Enau) and also used in medicine (Ishak et al., 2013; Nadlene et al., 2016a). For roselle tree, once this tree reaches a year it will normally be cut off after the fruit was harvested and becomes waste disposal without proper management. The roselle stem can produce fibres and can be used as reinforcement in polymers composite. This method is an alternative to

increase the income of roselle trees farmers by using roselle tree waste that can be used as reinforcement to the composites. The sugar palm tree belongs to the 'Palmae' family. This tree is a multipurpose tree as the entire tree can be used i.e. trunk, leaves, fruit and fibres. It is the most widely used sugar palm for the harvest of sugar and fibres. Previous studies have shown that SPF has good tensile properties and appropriate as reinforcement agent in polymer composites (Huzaifah et al.,2017). Information for both of these fibres are very important for understanding and evaluating mechanical, physical and thermal properties before it is used in various engineering industries. The selection of RF and SPF in this study was to reduce environmental pollution such as open burning and additionally, this fibres is low cost compared to other natural and conventional fibres.

Hybridization is a combination of more than one fibre used to reinforce a matrix to increase its mechanical properties and to reduce the cost of producing fibre reinforced polymer hybrid composites. The hybridization of natural fibres with natural or synthetic fibres can improve thermal, strength, stiffness, ductility, and damping properties of polymer composites (Nunna, Chandra, Shrivastava, & Jalan, 2012). In this research, the hybridization of RF/SPF/TPU hybrid composites were performed.

The combinations of these two type of fibres as RF and SPF have different mechanical properties reinforced TPU composites. RF has good tensile strength and SPF has high impact strength. Therefore, RF/SPF hybrid to further enhance the mechanical and thermal properties.

1.3 Objectives

This study involved polymers composites using RF and SPF by providing characteristic effects on RF and SPF and taking advantage of each properties to create better new material in general.

The objectives of the research are as follows:

1. To evaluate the effect of fibre size on mechanical and thermal properties of RF/SPF composites
2. To analyse the effect of filler loading on mechanical, thermal and morphology properties of RF/SPF composites
3. To investigated the effect of the hybridization of RF/SPF/TPU on the mechanical, thermal & morphological properties.
4. To evaluate the effect of surface treatment of RF/SPF/TPU hybrid composites on morphological, mechanical, physical and thermal properties.

1.4 Significance of the research

1. The successful development of natural fibres such as RF and SPF will enhance living standards for farmers dealing with roselle and sugar palms in rural areas especially in Malaysia.
2. In addition, this study is expected to expose the potential of using RF and SPF in green or friendly product development.
3. The outputs and findings of the research are expected to enhance the knowledge of RF/SPF/TPU hybrid composites.
4. The development of hybrid composites with enhanced features is expected to provide alternative materials to conventional materials.
5. The development of hybrid composites is expected to reduce environmental pollution during disposal.

1.5 Scope of study

In this research, the purpose of the study is to understanding of the natural fibre reinforced polymers composites. Natural fibres used in this research is RF and SPF reinforced TPU hybrid composites were characterized in terms of their mechanical, physical and thermal properties. RF were obtained from retted stems using water retting process, where they were immersed in water for 14 days, and then were cleaned using water tap and finally, the fibres were manually removed. SPF were extracted from the sugar palm tree and it was necessary to wash with water to remove impurities. Then, RF with different short fibre sizes and loading percentages were blended with TPU to form composites and their properties were determined. The evaluation of material composition properties is mechanical (tensile, flexural and impact properties) and thermal (TGA) properties. Hybridized composites made from RF/SPF reinforced TPU with different hybrid loading composites and determination material composition properties includes physical (density, water absorption and thickness swelling), mechanical (tensile, flexural and impact) and thermal (TGA) test. Lastly, both fibres were treated with chemical treatment to observe the effect of alkaline treatment on physical, mechanical and thermal properties of hybrid composites. The alkaline treatment was studied to analyse which NaOH concentration parameters contributes to improvement in mechanical, physical and thermal properties of RF/SPF hybrid composites. FTIR analysis were conducted for chemical composition analysis. The results of untreated and treated samples were compared. The potential of this composite product is for automotive parts (battery holder) evaluated through mechanical, physical and thermal testing

1.6 Structure of thesis

The structure of this thesis is in accordance with the alternative thesis format of Universiti Putra Malaysia, which is based on journal publication. Each research chapter (journal paper) represents a separate research on its own: ‘Introduction’, ‘Materials and methods’, ‘Results and discussion’, and ‘Conclusions’. The details of the thesis structure are presented as follows:

Chapter 1

Problem statements and objectives are presented in this chapter. The significance of the research work and the scope of research are also presented in this chapter.

Chapter 2

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

Chapter 3

The methodology used in this research for the preparation of materials, testing procedures, and data collection is presented in this chapter.

Chapter 4

This chapter presents the first article "**Mechanical and thermal performance of roselle fibres-reinforced thermoplastic polyurethane composites**". In this article, the effect of fibre size on RF/TPU composites on the mechanical and thermal properties was investigated.

Chapter 5

This chapter presents the second article "**Influence of fibre contents on mechanical and thermal properties of roselle fibre reinforced polyurethane composites**". In this article, the effect of fibre loading on RF/TPU composites on the mechanical and thermal properties was investigated.

Chapter 6

This chapter presents the third article "**Mechanical performance of roselle/sugar palm fibre hybrid reinforced polyurethane composites**". In this article, the effect of SPF fibre loading on mechanical properties of roselle composites was investigated.

Chapter 7

This chapter presents the fourth article "**Water absorption, thickness swelling and thermal properties of roselle/sugar palm fibre reinforced thermoplastic**

polyurethane hybrid composites." In this article, the effect of SPF fibre loading was studied relative to the physical and thermal properties of roselle hybrid composites.

Chapter 8

This chapter presents the fifth article "**Effect of alkaline treatment on mechanical, physical and thermal properties of roselle/sugar palm fibre reinforced thermoplastic polyurethane hybrid composites**". In this article, the effect of alkaline treatment on the mechanical, physical and thermal properties of RF/SPF/TPU hybrid composites was evaluated.

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BIODATA OF STUDENT

The student Mohd Radzi bin Ali was born on the 27th February 1981in Ipoh, Perak. He received his early primary education in 1993 at Sekolah Rendah Kebangsaan Senawang, Seremban, Negeri Sembilan and complete his secondary school in 1998 at Sekolah Menengah Senawang, Seremban, Negeri Sembilan. Then, he proceed to pursue his Diploma in Mechanical Engineering in 2002 at Politeknik Sultan Salahuddin Abdul Aziz Shah, Shah Alam, Selangor. Then, proceed to pursue his Bachelor's Degree in Metallurgy Engineering in 2007 and Master of Science Material Engineering in 2014 at Universiti Malaysia Perlis (UniMAP), Perlis. Thereafter, he served at Samsung Display Energy Malaysia Sdn. Bhd, Seremban as production engineer from 2012-2015. In 2015, he started his PhD study in the field of Technology Biocomposites at Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia (UPM).

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