



UNIVERSITI PUTRA MALAYSIA

***PROPERTIES OF SHORT KENAF FIBER-REINFORCED
THERMOPLASTIC POLYURETHANE COMPOSITES***

YOUSUF ALI GUMAAN EL-SHEKEIL

FK 2012 31

**PROPERTIES OF SHORT KENAF FIBER-REINFORCED
THERMOPLASTIC POLYURETHANE COMPOSITES**

By

YOUSUF ALI GUMAAN EL-SHEKEIL

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

August 2012

DEDICATION

This thesis is gratefully dedicated to:

My Beloved Father and Mother for their unlimited sacrifices, encouragements and support throughout my life

My Wife for her patience and understanding



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

**PROPERTIES OF SHORT KENAF FIBER-REINFORCED
THERMOPLASTIC POLYURETHANE COMPOSITES**

By

YOUSUF ALI GUMAAN EL-SHEKEIL

August 2012

Chairman : Mohd Sapuan Salit, PhD, PEng

Faculty : Engineering

This research was carried out to produce a new composite material from kenaf (*Hibiscus Cannabinus*) bast fiber and thermoplastic polyurethane (TPU/KF). The effect of processing parameters (i.e. temperature, time and speed in the internal mixer) on tensile strength were studied. The effect of different fiber sizes; namely, <125, 125-300, and 300-425 μm on tensile, flexural, and impact strengths and thermogravimetric analysis (TGA) were studied. Fiber loading (i.e. 20, 30, 40 and 50 % fiber by weight) was tested using tensile and flexural properties and impact strength. Further characterizations for the 30% fiber loading using hardness and abrasion resistance were performed. Effect of sodium hydroxide (NaOH) treatment on TPU/KF mechanical properties was determined. Effect of polymeric Methylene Diphenyl Diisocyanate (pMDI) additive on mechanical and thermal properties of TPU/KF composites was also investigated. Effect of pMDI chemical treatment was studied in two procedures. First, fibers were chemically treated with pMDI. Second, pMDI chemical treatment was used with NaOH pretreated fibers. Temperature, time and speed were optimum at 190 °C, 11 min, and 40 rpm, respectively.

Fiber size and fiber loading were optimum at fibers between 125 and 300 μm and 30% fiber loading. Tensile strength decreased by 37, 47 and 62% for 2, 4 and 6% NaOH concentrations; respectively. Flexural and impact strengths have also deteriorated. These results were supported by Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and TGA. It was noticed that the addition of 2, 4 and 6% pMDI did not show a significant change in tensile or thermal properties. These results were also confirmed by FTIR, TGA and Differential Scanning Calorimetry (DSC). pMDI chemical treatment has slightly increased tensile strength, while NaOH+pMDI chemical treatment showed increase in tensile strength and modulus by 30% and 42% respectively, however no significant change was noted in strain. This increase was evident by FTIR, which showed a better H-bonding and SEM which showed a better fiber-matrix adhesion.

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

**SIFAT- SIFAT KOMPOSIT POLIURETANA TERMOPLASTIK
BERTETULANG GENTIAN KENAF**

Oleh

YOUSUF ALI GUMAAN EL-SHEKEIL

Pengerusi : Mohd Sapuan Salit, PhD, PEng

Fakulti : Kejuruteraan

Kajian ini telah dijalankan untuk menghasilkan bahan komposit baru dari gentian kulit kenaf (*Hibiscus cannabinus*) dan poliuretana termoplastik (TPU/KF). Kesan parameter pemprosesan (iaitu suhu, masa dan kelajuan dalam pencampur dalaman) ke atas kekuatan tegangan telah dikaji. Kesan saiz gentian yang berbeza iaitu, <125, 125-300, dan 300-425 μm pada kekuatan tegangan, lenturan, dan hentaman dan analisis termogravimetri (TGA) telah dikaji. bebanan gentian (iaitu gentian 20, 30, 40 dan 50% mengikut berat) telah diuji menggunakan sifat tegangan dan lenturan dan kekuatan hentaman. Pencirian lanjut untuk bebanan gentian 30% menggunakan kekerasan dan rintangan lelasan telah dijalankan. Kesan rawatan natrium hidroksida (NaOH) ke atas sifat mekanikal TPU/KF telah ditentukan. Kesan bahan tambah polimer Metilena Diphenil Diisosiyanat (pMDI) ke atas sifat mekanikal dan termal komposit TPU/KF telah juga dikaji. Kesan rawatan kimia pMDI telah dikaji dalam dua prosedur. Pertama, gentian telah dirawat secara kimia dengan pMDI. Kedua, rawatan kimia pMDI telah digunakan dengan gentian terprerawat NaOH. Suhu, masa dan kelajuan adalah optimum masing-masing pada 190 ° C, 11 min, dan 40 rpm. Saiz gentian dan bebanan gentian adalah optimum pada gentian antara 125 dan 300 μm dan benanan gentian 30%.

Kekuatan tegangan menurun sebanyak 37, 47 dan 62% masing-masing untuk 2, 4 dan 6% kepekatan NaOH; kekuatan. Lenturan dan hemtaman juga telah merosot. Keputusan ini telah disokong oleh Spektroskopi inframerah transformasi Fourier (FTIR), eikroskopi elektron pengimbasan (SEM) dan TGA. didapati bahawa penambahan pMDI sebanyak 2, 4 dan 6% tidak menunjukkan perubahan yang ketara dalam sifat tegangan atau haba. Keputusan ini juga telah disahkan oleh FTIR, TGA dan kalorimeter pengimbasan perbezaan (DSC). Rawatan kimia pMDI telah meningkat sedikit kekuatan tegangan, manakala rawatan kimia NaOH + pMDI menunjukkan peningkatan dalam kekuatan tegangan dan modulus masing-masing sebanyak 30 dan 42%, namun tiada perubahan ketara telah dicatatkan dalam terikan. Peningkatan ini adalah dibuktikan oleh FTIR, yang menunjukkan ikatan-H dan keputusan SEM menunjukkan ikatan gentian-matriks yang lebih baik.

ACKNOWLEDGEMENTS

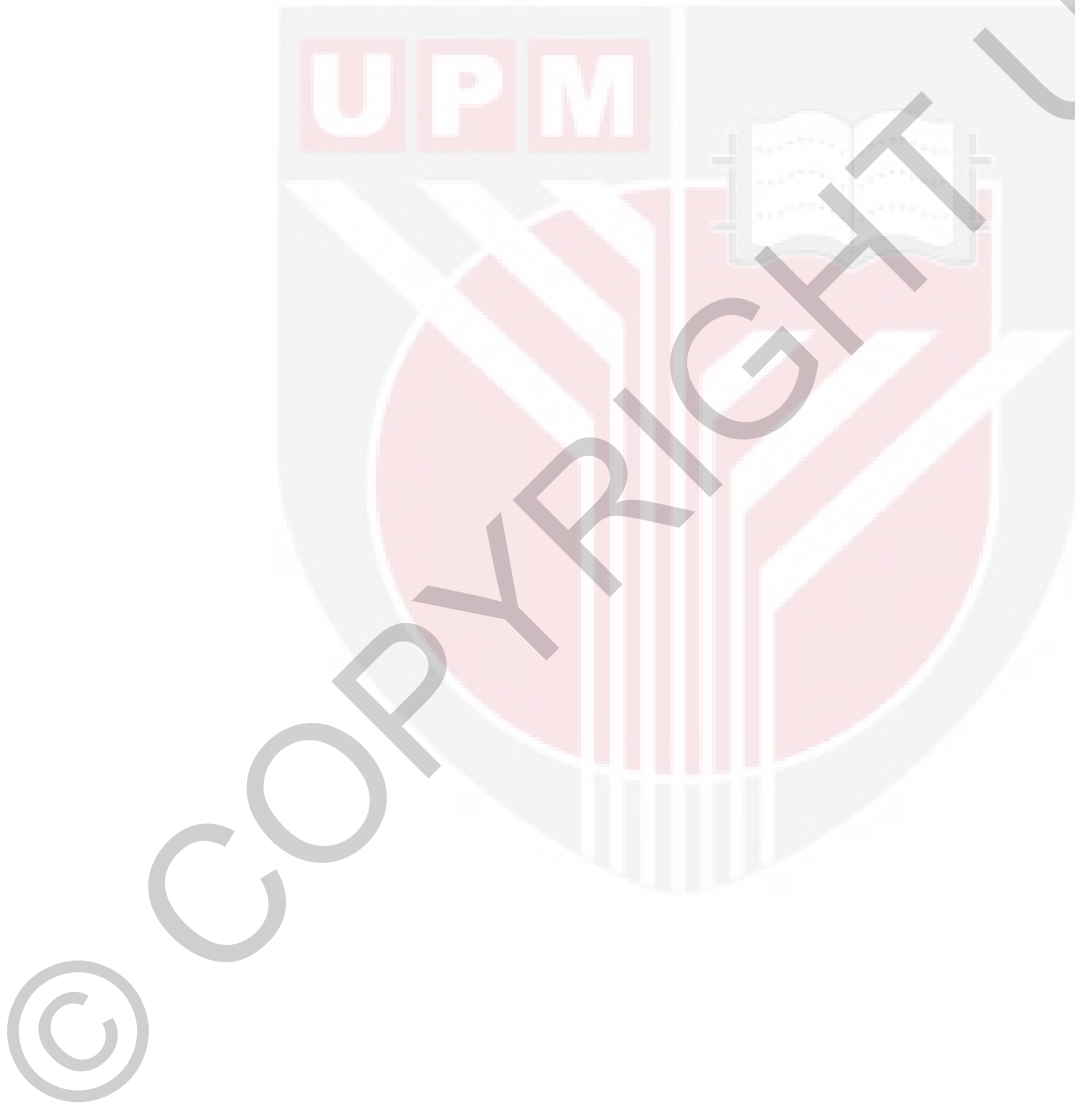
First, my praise to Almighty Allah for giving me the power and will to complete this project.

I particularly would like to express my appreciation and profound gratitude to the chairman of supervisory committee, Professor Mohd Sapuan Salit for his invaluable advice, guidance, effort, and support throughout the course of this study. I also would like to convey my thanks to the members of supervisory committee, Dr. Edi Syams Zainudin and Associate Professor Dr. Khalina Abdan, for sharing their expertise and experience and for continuous guidance, encouragement and suggestions that enriched the study.

Words cannot be enough to express my thanks to my family for their unconditional love and endless support that gave me the strength through the hardest times, especially the unlimited sacrifices of my parents. Special thanks are extended to my wife, for her understanding, patience and effort. It is worth to mention all my friends in for their support and cooperation.

Special thanks to Dr. Riza Wirawan for the fruitful discussions throughout the course of this study. Thanks to science officer, technical assistant and technicians in Faculty of Engineering UPM, Institute of Tropical Forestry and Forest Products (INTROP) and SIRIM Berhad including Nor Hasni Zahari, Maslinda Abdullah, Muhammad Wildan Ilyas Mohamed Ghazali, Ismail b. Abdul Ghani, and Rahmad b. Abd Shukor.

Ministry of Higher Education, Yemen is acknowledged for the financial support for my PhD study. Fundamental Research Grant Scheme (FRGS), Ministry of Higher Education Malaysia grant number (01-10-10-924FR) is acknowledged for support of this research. The author wish to thank Bayer Co. (Malaysia) Sdn Bhd, Petaling Jaya, Selangor, Malaysia for the material supply and the information provided.



I certify that a Thesis Examination Committee has met on August 8th 2012 to conduct the final examination of Yousuf Ali Gumaan El-Shekeil on his thesis entitled "Properties of Short Kenaf Bast Fiber-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.

Members of the Thesis Examination Committee were as follows:

Shamsuddin b. Sulaiman, PhD

Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Zulkifile b. Leman, PhD

Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Luqman Chuah Abdullah, PhD

Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Faiz Mohammad, PhD

Faculty of Engineering and Technology
Aligarh Muslim University
(External Examiner)

SEOW HENG FONG, PhD

Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Mohd Sapuan Salit, PhD, P.Eng

Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Edi Syams Zainudin, PhD

Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Khalina Abdan, PhD

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD

Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia
Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.

YOUSUF ALI GUMAAN EL-SHEKEIL

Date: 8/August/2012



TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	v
ACKNOWLEDGEMENTS	vii
APPROVAL	ix
DECLARATION	xi
LIST OF TABLES	xv
LIST OF FIGURES	xvii
LIST OF ABBREVIATIONS	xxiii
CHAPTER	
1 INTRODUCTION	1
1.1 Background of Study	1
1.2 Significance of Study	2
1.3 Problem Statement	3
1.4 Objectives	4
1.5 Scope and Limitations	4
2 LITERATURE REVIEW	6
2.1 Polymers	6
2.2 Advantages of Thermoplastics Compared to Thermosets	8
2.3 Disadvantages of Thermoplastics Compared to Thermosets	9
2.4 Natural Fibers	9
2.5 Composite Materials	14
2.6 Composite Mechanical Properties	14
2.7 Natural Fiber Composites	15
2.8 Kenaf Fibers	17
2.9 Kenaf Fiber Composites	21
2.10 Polyurethane	24
2.11 Polyurethane Natural Fiber Composites	26
2.12 Isocyanate Treatment	29

2.13	Alkali Treatment	30
2.14	Conclusions	32
3	MATERIALS AND METHODS	33
3.1	Introduction	33
3.2	Materials	33
3.3	Fibers Preparation	35
3.4	Composite Preparation	35
3.5	Alkali Treatment	36
3.6	pMDI additive	36
3.7	pMDI Chemical Treatment	36
3.8	Tensile Testing	36
3.9	Flexural Testing	37
3.10	Impact Testing	37
3.11	Abrasion Resistance Testing	37
3.12	Hardness Testing	38
3.13	Scanning Electron Microscope	38
3.14	FTIR Spectra Characterization	38
3.15	Thermogravimetric Analysis (TGA)	38
3.16	Differential Scanning Calorimetry (DSC)	39
4	DEVELOPMENT OF A NEW KENAF BAST FIBER-REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITE	40
	Article 1	40
	Acceptance Letter	57
	Copyright Permission	58
5	INFLUENCE OF FIBER CONTENT ON THE MECHANICAL AND THERMAL PROPERTIES OF KENAF FIBER REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITES	59
	Article 2	59
	Acceptance Letter	74
	Copyright Permission	75
6	EFFECT OF ALKALI TREATMENT ON MECHANICAL AND THERMAL PROPERTIES OF KENAF FIBER-REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITE	76
	Article 3	76

	Acceptance Letter	94
	Copyright Permission	95
7	EFFECT OF pMDI ISOCYANATE ADDITIVE ON MECHANICAL AND THERMAL PROPERTIES OF KENAF FIBER REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITES	96
	Article 4	96
	Acceptance Letter	109
	Copyright Permission	110
8	INFLUENCE OF CHEMICAL TREATMENT ON TENSILE PROPERTIES OF KENAF FIBER REINFORCED THERMOPLASTIC POLYURETHANE COMPOSITES	111
	Article 5	111
	Acceptance Letter	130
	Copyright Permission	131
9	DISCUSION	132
10	CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH	139
	REFERENCES	141
	APPENDICX	153
	BIODATA OF STUDENT	167
	LIST OF PUBLICATIONS	168