UNIVERSITI PUTRA MALAYSIA

MICROMECHANICS OF OIL PALM MESOCARP FIBRES AND BIOCOMPOSITES

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By

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

July 2018

DEDICATION

Dedicated to my family

for their endless love, support and encouragement

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

MICROMECHANICS OF OIL PALM MESOCARP FIBRES AND BIOCOMPOSITES

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July 2018

Chairman : Mohd Afandi P Mohammed, PhD
Faculty : Engineering

Investigation was conducted on non-linear mechanical behaviour of oil palm mesocarp fibres (OPMF) and their biocomposites, with focus on the interface of the fibres (filler) and matrix. Viscoelastic with damage was observed from tensile tests conducted under cyclic mode, as reported from the unloading-reloading results of the cyclic tests at larger deformations (2 and 3mm deformation). This behaviour was related to the lignocellulosic components of the fibres, as well as geometry of the fibres consisting of silica bodies and cellular structure. On the other hand, mechanical tests comparison of the processed fibres mentioned before with fresh mesocarp fibres showed different viscoelastic behaviour of the latter fibres, which was due to moisture within the fibres containing palm oil, as well as the effect of oil palm processing that altered the processed fibres. The tests results were modelled through a viscoelastic model available in finite element software, Abaqus, which consisted of hyperelastic model with Prony series and a stress softening function. Good agreement was reported from the fitting of the model to the mechanical tests results, highlighting the viscoelastic behaviour of oil palm fibres. Emphasis was then given to the effect of silica bodies towards integrity of the oil palm fibres, where a cohesive zone modelling (CZM) was included to model the interface between silica bodies and fibres. The results showed minimal effect of silica bodies towards integrity of the fibres as a whole, which was due to the silica bodies were only partly embedded on the outer surface of the fibres. The fibres were then used for biocomposites development as filler, and LLDPE was used as matrix. The interface between the filler and

matrix was improved using anhydrate (maleic anhydride and itaconic anhdride). In addition to the interface improvement using chemical method (anhydrate to strengthen the filler-matrix interface), it is hypothesised that the geometric effect of the fibres consisting of silica bodies on the surface can also improve the filler-matrix interface. Therefore, the fibres were not chemically treated (with alkali or acid as conducted before in previous literatures) to preserve the silica bodies and fibres integrity. Improvement of the biocomposites with both anhydrate and silica bodies was reported from a series of experiments, namely mechanical tests, FTIR, and microscopy analyses. In particular, SEM image showed that silica bodies left craters after being pull during tensile testing, suggesting that the silica bodies prevent sliding between the filler-matrix interface. Likewise, evidence of OH bond between the silica bodies and matrix was shown, similar to the filler-matrix improvement due to addition of anhydrate. Biocomposites finite element model geometry was generated using Digimat software, but the modelling analysis was terminated before any results can be obtained. The results from both mechanical behaviour of fibres and biocomposites interface highlighted that oil palm mesocarp fibres behaved as a viscoelastic material with damage due to deformation, and the fibres used for biocomposites application can be obtained directly without chemical treatment.

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

MIKROMEKANIK GENTIAN MESOKAP KELAPA SAWIT DAN BIOKOMPOSIT

Oleh

SUHAIZA HANIM BINTI HANIPAH

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Penyelidikan ini dijalankan terhadap tingkah laku mekanikal bukan linear gentian mesokarpa kelapa sawit (OPMF) dan biokomposit, dengan tumpuan pada antara muka gentian (pengisi) dan matriks. Kerosakan viskoelastik ini diperhatikan dari ujian tegangan yang dijalankan di bawah mod kitaran, seperti yang dilaporkan dari keputusan pemuatan semula, ujian kitaran berubah bentuk kepada yang lebih besar (ubah bentuk 2 dan 3mm). Tingkah laku ini berkaitan dengan komponen lignoselulosik serat, serta geometri serat yang terdiri daripada badan-badan silika dan struktur selular. Sebaliknya, perbandingan ujian mekanikal gentian yang diproses yang disebut sebelum ini dengan gentian mesokarp segar menunjukkan kelakuan viskoelastik yang berbeza daripada serat kedua, yang disebabkan oleh kelembapan dalam serat yang mengandungi minyak kelapa sawit, serta kesan pemprosesan kelapa sawit yang mengubah keadaan gentian yang diproses. Keputusan ujian dimodelkan melalui model viskoelastik yang terdapat dalam perisian unsur terhingga, "Abaqus", yang terdiri daripada model hiperelastik dengan siri "Prony" dan fungsi pelekapan tekanan. Keseragaman keputusan telah dilaporkan dari melalui model kepada hasil ujian mekanikal, menonjolkan kelakuan viskoelastik gentian kelapa sawit. Penekanan kemudiannya diberikan kepada kesan badan silika ke arah integriti gentian mesokarpa kelapa sawit, di mana pemodelan zon kelekatan (CZM) dimasukkan untuk memodelkan antara badan silika dan gentian. Hasilnya menunjukkan kesan minima badan silika terhadap integriti serat secara keseluruhan, yang disebabkan oleh badan-badan silika hanya sebahagiannya tertanam di permukaan luar serat. Gentian kemudian digunakan untuk pembangunan

biokomposit sebagai pengisi, dan polietilin linear kurang tumpat (LLDPE) digunakan sebagai matriks. Antara muka antara pengisi dan matriks dipertingkatkan menggunakan anhidrat (malik anhadrida dan itakonik anhadrida). Selain penambahbaikan antara muka dengan menggunakan kaedah kimia (anhidrat digunakan untuk menguatkan antara muka pengisi-matriks), hipotesis bahawa kesan geometrik gentian yang terdiri daripada badan-badan silika di atas permukaan juga boleh meningkatkan kekuatan antara muka-matriks dan pengisi. Oleh itu, serat tidak dirawat secara kimia (dengan alkali atau asid seperti yang dilakukan sebelum dalam literatur terdahulu) untuk mengekalkan badan silika dan integriti serat. Peningkatan biokomposit dengan kedua-dua badan anhidrat dan silika dilaporkan dari satu siri eksperimen, iaitu ujian mekanikal, transformasi Fourier inframerah, dan analisis mikroskopi. Secara khususnya, imej pengimbasan mikroscop elektron menunjukkan bahawa badan-badan silika meninggalkan kawah selepas di tarik semasa ujian tegangan, mengimplikasikan bahawa badan-badan silika mencegah gelongsor antara antara muka dan matriks pengisi. Begitu juga, keterangan mengenai ikatan hidroksil di antara badan-badan silika dan matriks ditunjukkan, sama dengan peningkatan pengisi-matriks kerana penambahan anhidrat. Model geometri biokomposit melalui elemen tak terhingga dihasilkan dengan menggunakan perisian "Digimat", tetapi analisis pemodelan telah ditamatkan sebelum sebarang keputusan boleh diperolehi. Hasil dari kedua-dua tingkah laku mekanikal serat dan biokomposit antara muka menyerlahkan bahawa gentian kelapa sawit mesokarpa berkelakuan sebagai bahan viskoelastik dengan kerosakan akibat ubah bentuk, dan gentian yang digunakan untuk aplikasi biokomposit dapat diperoleh secara langsung tanpa rawatan kimia.

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Thank You

I certify that a Thesis Examination Committee has met on 10 July 2018 to conduct the final examination of Suhaiza Hanim binti Hanipah on her thesis entitled "Micromechanics of Oil Palm Mesocarp Fibres and Biocomposites" 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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LIST OF ABBREVIATIONS

ADF	Acid detergent fibre
ADL	Acid detergent lignin
BPO	Benzoyl peroxide
CPO	Crude palm oil
CZM	Cohesive zone modelling
DCP	Dicumyl peroxide
DMF	Dimethylformamide
EFB	Empty fruit bunch
FFB	Fresh fruit bunch
HDPE	High density polyethylene
LLDPE	Linear low density polyethylene
LLDPE-g-UT	LLDPE graft Untreated
LLDPE-g-T	LLDPE graft Treated
MAH	Maleic anhydride
MAPP	Maleic anhydride polypropylene
MDI	Methylene diphenyl isocyanate
NDF	Neutral detergent fibre
OPEFB	Oil palm empty fruit bunch
OPF	Oil palm fibre
OPMF	Oil palm mesocarp fibre
OPMF-b-LLDPE	OPMF blend LLDPE
OPMF-g-LLDPE	OPMF graft LLDPE
PE	Polyethylene

POME	Palm oil mill effluent
PP	Polypropylene
RVE	Representative elementary volume
SiO_2	Silica dioxide (silica body)
Si-O-C	Silica-oxygen-carbon linkages
TGA	Thermogravimetric analysis

LIST OF SYMBOLS

Symbol	Unit	Descriptions
σ	MPa	true stress
ε	mm/mm	true strain
E	dimensionless	Young's modulus
λ_m	dimensionless	locking stretch
μ	dimensionless	instantaneous initial shear
a	dimensionless	global interaction parameter
W	J	Strain energy potential
$g\ (t)$	dimensionless	step strain loading in function of time
gi	dimensionless	Constant
g_∞	dimensionless	Constant
G_i	dimensionless	Modulus of ith spring
G_o	Pa	Instantaneous modulus
G_∞	dimensionless	Modulus of infinite lone spring
G_c	J/m^2	Energy release rate
$\mathbf{t}$	MPa	Traction separation vector
t	s	time
s	s	time
P_o	MPa	True stress
ξ_i	dimensionless	Relaxation time constant
η	dimensionless	Damage variable
U_{dev}^m	dimensionless	Deviatoric part of strain energy

n	dimensionless	Nominal traction stress vector
K	dimensionless	Tensor coefficient
δ	dimensionless	Separation vector

CHAPTER 1

INTRODUCTION

1.1 Overview of oil palm mesocarp fibre mechanics and bio composite

Malaysia has been recognised as the second largest producer of palm oil products such as oil palm, olein, stearin and other derivatives in the world (Sabil et al., 2013). With such a huge development, residual materials from the industry become an environmental issue. In palm oil processing, three types of side-products are produced, mainly oil palm empty fruit bunch, oil palm mesocarp fibre and oil palm trunk fibres. Numerous scientific works on empty fruit bunch and palm fibres (mixture of oil palm empty fruit bunch and oil palm stalk fibres) have been extensively reported (Gunawan et al., 2009; Isroi et al., 2012; Law et al., 2007). Nevertheless, to the best our knowledge, very few research works on the mechanics of oil palm mesocarp fibres (OPMF) has been reported. In principle, OPMF is obtained after milling process of oil palm fruit bunches in an oil palm mill. In natural, all fibres are cellulosic material containing cellulose, hemicellulose and lignin. However, detailed investigation of the mechanics of oil palm fibres bundle is not yet available, such as complex mechanical behavior under different loading conditions.

Oil palm fibre usually was used for biocomposites (Shinoj et al., 2011; Rozman et al., 2003; Then et al., 2015) and biocomposting (Nordin et al., 2012; Mohammed et al., 2013). The former application focusses on the creation of new material with intention on superior mechanical strength, while in the latter application, works were conducted on finding the best biological method in production of the compost, as well as the quality of composts produced. The recent study on biodegradation of oil palm fibres by Omar et al. (2017) suggested the importance of studying mechanics of oil palm fibres, especially towards understanding the effect of microbial activity towards volume reduction of fibre from biodegradation process. For oil palm mesocarp fibres biocomposites, the interaction between fibre and matrix, fibre and protrusion (silica bodies) as well as the non-linear mechanics of fibre are very important in order to further understand the mechanics of oil palm biocomposites system. For the fibre – matrix interface, improvement of the interface need to be considered, for example the use grafting process to improve the fibre(filler)-matrix interface using anhydrate chemicals. In addition, the effect of fibre's outer geometry (such as silica bodies) which can acts as a sliding-prevention mechanism between the fibre-matrix under deformation is important and not yet clearly studied. Thus this research will focus with the study of fibre-silica body-matrix interface, in addition to the nonlinear mechanical behavior of oil palm mesocarp fibres.

1.2 Problem Statements

There are only a few literatures available on the complex mechanical behavior of OPMF (for example only one study on viscoelasticity of oil plam fibres by Sreekala et al. 2001). The complex mechanical behaviour in this case can referred to as viscoelastic and damage within the fibres due to deformation, this can be due to the lignocellulosic contents of the oil palm fibres, as well as complex geometry of the fibre containing silica bodies partly embedded on the surface and cellular structure within the cross section. Understanding these complex behaviour and microsctructure of oil palm fibres is important for biocomposites application, where it was reported that the fibres need to undergo chemical treatment before they are suitable for composites production. This chemical treatment can alter the mechanical behaviour of the fibres, causing inferior mechanical behaviour of the biocomposites produced. In addition, the interface between oil palm fibres (filler) and matrix need to be investigated, where the behaviour at the interface can be influenced by chemical interaction between filler-matrix and/or geometric effect (silica bodies on the surface).

1.3 Research Objectives

Based on the problems statement mentioned before, the following objectives are considered to embark this research project:

(a) To investigate the non-linear mechanical behavior of oil palm mesocarp fibres (empty fibres and fresh fibres), namely viscoelastic as well as damage due to deformation through complex mechanical tests (tensile, cyclic-tensile and tensile-relaxation modes).
(b) To simulate oil palm mesocarp fibres deformation using a finite element viscoelastic model with a damage function, which is developed using information from the complex mechanical tests in previous objective.
(c) To produce the bio-composite material using oil palm mesocarp fibres as filler and LLDPE as matrix, where the behaviour of the filler-matrix interface is studied using chemical methods (grafting) and fibre geometric effect (silica bodies).

(d) To simulate oil palm biocomposites interface behaviour using a finite element model, which focusses on the filler-matrix interface and silica bodies effect.

1.4 Research scope

The research scope of this work focusses on the mechanical behaviour of oil palm mesocarp fibres, and mechanics of oil palm fibres biocomposites interface. The former will involve complex mechanical characterisation and micromechanical modelling of oil palm fibres. The mechanical characterization will consider tensile tests under constant speed, cyclic tensile and tensile-relaxation modes. These tests will reveal viscoelastic behaviour of the fibres, with possibility of damage occurring within the fibres due to deformation. The biocomposites study on the other hand involves production of bio composites using in situ grafting method in internal mixer, which includes anhydride, polymer (LLDPE) and OPMF itself. Focus will be placed on the interfacial behaviour of the filler-matrix interface in terms of chemical bonding (from grafting) and microstructural effect (silica bodies effect on preventing filler-matrix sliding).

1.5 Thesis Outline

This thesis is organized into five chapters (Figure 1.1). The introduction and objectives are stated in Chapter 1, whilst the Chapter 2 deals with literature review on the oil palm fibres mechanical behaviour and their biocomposites application. In Chapter 3, the general material and methods involved in mechanical testing of the oil palm fibres and their biocomposites are explained in detail. Likewise, the procedures involved in the finite element model development is discussed in detail in this chapter. Chapter 4 showed experimental results of the oil palm fibres mechanical tests and microscopy analyses, as well as their biocomposites mechanical, chemical and microscopy (x-ray micro-tomography) analyses. This then proceeds with modelling results for the oil palm fibres and biocomposites. Conclusions and recommendations for further work are presented in Chapter 6.

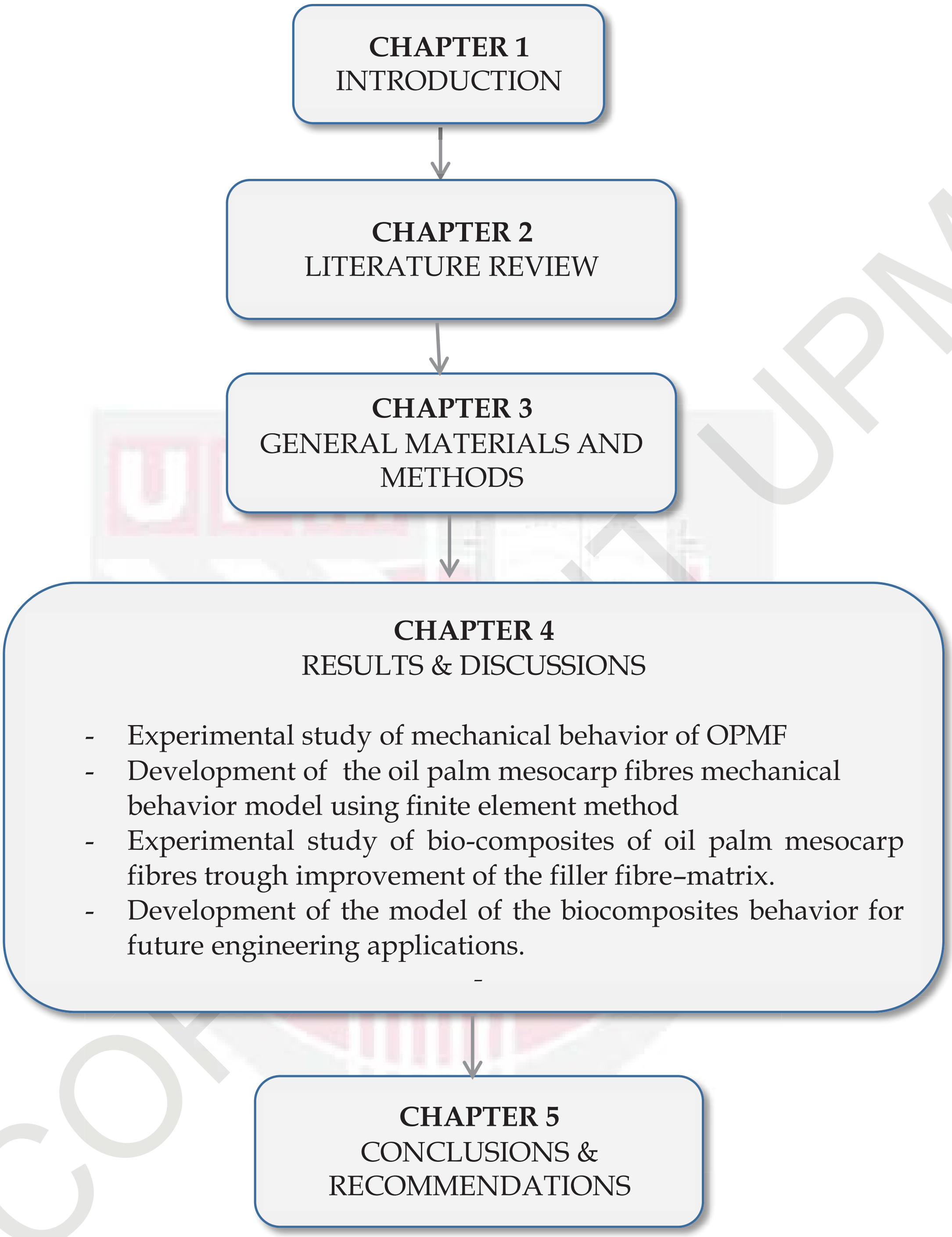

Figure 1.1 : Thesis outline

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