UNIVERSITI TEKNIKAL MALAYSIA MELAKA

COMPARISON OF EXPERIMENTAL AND THEORETICAL STUDIES OF NANOCELLULOSE JUTE FIBER IN THE POLYLACTIC ACID (PLA) COMPOSITE

Submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

TANG PEI CHIN

B051610041

961227-01-6414

FACULTY OF MANUFACTURING ENGINEERING

2020

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: **COMPARISON OF EXPERIMENTAL AND THEORETICAL STUDIES OF NANOCELLULOSE JUTE FIBER IN THE POLYLACTIC ACID (PLA) COMPOSITE**

Sesi Pengajian: **2019/2020 Semester 2**

Saya **TANG PEI CHIN (961227-01-6414)**

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. *Sila tandakan (√)

☐ SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysiasebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)

☐ TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/ badan di mana penyelidikan dijalankan)

☑ √ TIDAK TERHAD

Disahkan oleh:

peichin

Alamat Tetap:

660, Parit Payong Jalan Abdul
Rahman, 84000 Muar Johor
Tarikh: 28TH August 2020

Cop Rasmi:
PROF. DR. QUMRUL AHSAN
Profesor
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka

Tarikh: 01/09/20

*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared this report entitled "Comparison of Experimental and Theoretical Studies of Nanocellulose Jute Fiber In The Polylactic Acid (PLA) Composite" is the result of my own research except as cited in references.

Signature : ....peichin....................................

Author's Name : TANG PEI CHIN

Date : 28TH AUGUST 2020

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

....

(Professor Dr. Qumrul Ahsan)

ABSTRAK

Pengembangan komposit polimer bertetulang nanoselulosa telah menarik perhatian kerana nanoselulosa dapat memberikan komposit dengan kekuatan yang lebih tinggi dan kekakuan tegangan. Pengukuhan asid polilaktik (PLA) menggunakan serat nanoselulosa Jute telah menarik minat penyelidik dengan tujuan menghasilkan bahan komposit hijau. Penyelidikan ini bertujuan untuk mengkaji kesan rawatan alkali terhadap pencirian serat jute, untuk menganalisis pengaruh pemuatan nanoselulosa pada modulus Young dan morfologi penyebaran dalam komposit berdasarkan kajian eksperimen yang dilaporkan dan untuk menilai perbandingan hasil modulus Young yang diperoleh dari kajian eksperimen yang dilaporkan dan persamaan teori. Hasil projek dibahagikan kepada 2 jenis, yang berdasarkan hasil makmal dan berdasarkan kajian tinjauan kritikal. Pertama, serat rami dirawat secara kimia, dan serat yang tidak dirawat dan dirawat alkali dianalisis di bawah FTIR. Selepas itu, modulus Young dari nanocomposites PLA 5 wt% dan 10 wt% dikumpulkan dari tiga kajian eksperimen yang dilaporkan. Semasa menambahkan 5 wt% JNF ke matriks, JNF / PLA meningkat sebanyak 217.30%, tetapi menurun setelah 5 wt% pemuatan serat. Walau bagaimanapun, apabila kandungan nanofillers meningkat, komposit SCNF / PLA dan NFCHL / PLA menunjukkan peningkatan dalam modulus Young. Teori Halpin-Tsai dan Percolation telah digunakan untuk meramalkan modulus nanokomposit dan hasilnya dibandingkan dengan modulus eksperimen yang dilaporkan. Hasil modulus yang diramalkan dinyatakan setuju dengan hasil modulus eksperimen komposit dengan muatan nanofiller hingga 5 wt% dan menunjukkan penyimpangan pada 10 wt%. Penyimpangan hasil modulus dapat dijelaskan oleh keterikatan dan pengagregatan nanofiber yang terbentuk pada pemuatan serat yang lebih tinggi. Peratusan ralat minimum dan maksimum yang terdapat pada nanokomposit adalah 14.17% dan 82.08% untuk teori Percolation dan 26.93% dan 74.12% untuk model Halpin-Tsai.

ABSTRACT

Development of nanocellulose reinforced polymer composites has attracted significant attention since the nanocellulose able to provide the composite with higher strength and tensile stiffness. The reinforcement of polylactic acid (PLA) using nanocellulose Jute fiber has gain interest of researchers with the goal of producing green composites material. This research was to investigate the effect of alkaline treatment on the characterization of jute fiber, to analyze the effect of nanocellulose loading on the Young's modulus and dispersion morphology in the composite based on the reported experimental studies and to evaluate the comparison of Young's modulus results obtained from reported experimental studies and theoretical equations. The project result was divided into 2 types, which are based on the laboratory results and based on the critical review. First, the jute fiber was chemically treated, and the untreated and alkali-treated fibers were analyzed under FTIR. After that, Young's modulus of 5 wt% and 10 wt% PLA nanocomposites were collected from three reported experimental studies. When adding 5 wt% of JNF to the matrix, modulus of JNF/PLA increased by 217.30%, but decreased after 5 wt% of fiber loading. However, as the content of nanofillers increases, SCNF/PLA and NFCHL/PLA composites show an increasing trend in Young's modulus. Halpin-Tsai and Percolation Theory have used to predict the modulus of the nanocomposite and the results compared with the reported experimental modulus. The predicted modulus results revealed agreed with the experimental modulus result of composites with nanofiller loading up to 5 wt% and showed deviation at 10 wt%. The deviation of the modulus result could be explained by the entanglements and agglomeration of nanofiber formed at a higher fiber loading. The minimum and maximum errors percentage found on the nanocomposite were 14.17 % and 82.08 % for the Percolation theory and 26.93 % and 74.12 % for the Halpin-Tsai model.

DEDICATION

This report is dedicated to my beloved parents,
Who educated me and enable me to reach this level.
To my honoured supervisor,
Proffesor Dr Qumrul Ahsan
for his advices during completion of the project
and to all staff and technicians,
for their advices and cooperation to complete this project.
Thank You So Much

ACKNOWLEDGEMENT

My respected supervisor, Prof Qumrul Ahsan, thank for very much for your patience in guiding me throughout the project. I would like to thank him for his advice, supervision and interpretation of the material knowledge and theory throughout the research process.

I would like to thank my friends who supported, suggested and encouraged me in completing this project. They provided their suggestions and comments to improve my project. Thank for my great friendship.

I would to thank the technicians who guided me to use the equipment and machines in the laboratory. Without your guidance, I may not be able to complete the research. Thank you for your patience while guiding me throughout my research. Besides, I would also like to thank the lecturers who provided me with machines and equipment throughout the research process.

Finally, I would like to thank everyone who help me to complete my FYP project, as well as apologies that I could not mention personally each one of you. Without your help, I would not be able to complete my project. Thank You.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF EQUATIONS

LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
FESEM	-	Field Emission Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscope
HCL	-	Hydrochloric Acid
JNF	-	Jute Nano fiber
MFC	-	Microfibrillated Cellulose
NaOH	-	Sodium Hydroxide
NCFHL	-	Nanocellulose Fibrils with High Lignin
NFC	-	Nano Fibrillated Cellulose
NFC-PLA	-	Nano fibrillated cellulose reinforced polylactic acid
PE	-	Polyethylene
PHB	-	Poly(3-hydroxybutyrate)
PLA	-	Polylactic Acid
PP	-	Polypropylene
PPP	-	Poly(p-phenylene) (PPP)
PTFE	-	Polytetrafluoroethylene
SCNF	-	Spherical Nanocellulose Formats
SEM	-	Scanning Electron Microscope
UTM	-	Universal Testing Machine

LIST OF SYMBOLS

wt. %	-	weightage percent
°C	-	Degree Celsius
rpm	-	Revolutions per minute
min	-	minute
MPa	-	Megapascal
GPa	-	Gigapascal
g	-	gram
%	-	percentage
g/cm^{-3}	-	Gram per Cubic Centimeter

CHAPTER 1

INTRODUCTION

This chapter discussed the research background, problem statements, objectives, scope, significant of the research and thesis organization.

1.1 Research Background

Disposal of waste polymer material had raised many problems related to the environmental safety. Environmentally-friendly materials have gained attention in recent years, especially due to increased environmental awareness, the expansion of global waste issues and the unsustainable petroleum consumption. Therefore, development of composites with natural fiber and biodegradable polymer is an alternative way to reduce the use of petroleum derived product (Kian et al, 2019). Owing to low cost, low density, renewability and biodegradability, natural fibers are generally considered as ideal candidate reinforcement in green composites.

Polylactic acid (PLA) is a thermoplastic polyester with biodegradable and bioactive properties that derived from the natural resources such as sugar cane, potatoes and corn starch. PLA has high mechanical strength, good biocompatibility, transparency, easy processing, and fast decomposition rate, so it has attracted much attention among other biodegradable polymers (Baheti et al., 2013). When exposed to the environment, it naturally degrades into water and carbon dioxide without causing any harm to the environment. PLA is used in a wide variety of applications including degradable plastic bags, bottles, food packaging and automotive applications. PLA is very useful for short lifespan applications where biodegradation is highly recommended. Conventional plastics can take hundreds to thousands of years to degrade in the ocean, while PLA takes 6 to 24 months to degrade in the same situation. Nevertheless, the thermal and mechanical properties of PLA are unstable at a higher temperature because it will soften around 60 °C (Baheti et al., 2013) and PLA has a relatively low glass transition temperature. Moreover, high brittleness and poor flexibility of PLA can degrade product performance and processing become challenging. The problems limit PLA for high performance applications.

To improve PLA's mechanical and thermal properties, the inclusion of natural fiber in PLA composites is seen as a desirable solution because of its high firmness, durability and environmentally friendly. Natural fibers are mainly sourced from plants and animals. Natural fibers like jute, hemp, sisal, flax, kenaf and pineapple leaf fibers are the most common and commercially used as biodegradable reinforcing materials for the manufacture of composite materials. Natural fibers offer more benefits in terms of being recyclable and biodegradable, reusable and comparatively high in strength and rigidity compared with synthetic fiber. Jute fiber has been selected as reinforcing materials in PLA composites in this research study due to it is easily available in fabric and fiber forms with good thermal and mechanical properties (Hassan et al., 2009). It is evidenced from research work carried out by Plackett et al. (2003) found that the combination of PLA and jute fiber, the tensile strength become twice and even more than double tensile stiffness at heating temperature of 210 °C.

Jute is known as bast fiber that come from the inner bark of the plants. Jute fiber is one of the cheapest natural fibers, composed primarily of the plant materials pectin, lignin and cellulose. Jute fiber is fully biodegradable and recyclable materials, thus environmentally friendly, one of the most versatile natural fibers, high tensile strength and used in raw materials for packaging. The jute stem contains high cellulose content and can be obtained in 4-6 months, so it can also save the forest and meet the world's cellulose and wood needs. Nanocellulose that derived from any cellulosic source that made up of nano-sized cellulose fibrils with a high aspect ratio (length-to-width ratio). Typical length and diameter are 10 nm to 10 μm and 5 to 20 nm respectively (Carron et al., 2019). Nanofibrillated cellulose (NFC) will discuss in this project. NFC, consists of bundle of stretched cellulose chain molecule with long, soft and entangled cellulose nanofiber. NFC is an ideal reinforcement for nanocomposites as they are easy to prepare, high specific surface area, high strength and durability, low density and biodegradability. This bio-based nanomaterial is mainly used in nanocomposites due to its excellent potential for enhancement. Larsson et al. (2012) stated that NFC in the composite able to improve the thermal stability due to a strong NFC bond formed within the matrix. High quality of NCFs can be obtained at low cost through mechanical disintegration after chemical pre-treatments of natural fibers.

1.2 Problem Statement

Disposal of non-biodegradable composite after their intended life time has bring effects to the environment. The use of non-biodegradable substances that make a major contribution to pollution growth should be reduced to a remarkable level. Considering environmental concerns, biodegradable polymers have become the target of replacing petroleum-based polymers in many applications because biodegradable polymers are susceptible to degradation compared to non-biodegradable ones. However, most biodegradable polymers are very expensive and have weak structure strength compared to synthetic polymer (Kumar et al., 2017). Addition of the natural fibers to the biodegradable polymer able to improve the performance of the biodegradable composite as natural fibers are high strength and stiffness.

However, nanofibers greatly blurred the crystal texture, it is difficult to evaluate the dispersion of NFC. Biopolymer, hydrophobic in nature and NFC as reinforcement is hydrophilic in nature have influenced the properties of the composites due to the dispersion and weak interfacial interaction problems. Mechanical properties of the polymer can improve when nano fibers combined with polymer matrices. It is fundamental for the nanofiber to be uniformly dispersed and distributed in the polymer matrix. Dispersion of NFC is important in the composite because it can affect the performance of the materials. The mechanical properties of the composite decrease when the weaker interface contact between the polymer matrix and the natural fiber. For this study, acid and alkaline hydrolysis have introduced in the chemical treatment process in order to improve the interfacial adhesion between the filler and the matrix.

By applying this method, the matrix and polymer particles can be better mixed in a dispersed state and aggregation can be avoided.

1.3 Objective

The objectives of the research are as follows:

a) To investigate the effect of alkaline treatment on the characterization of jute fiber.

b) To analyze the effect of nanocellulose loading on the Young's modulus and dispersion morphology in the composite based on the reported experimental studies.

c) To evaluate the comparison of Young's modulus result obtained from reported experimental studies and theoretical equations.

1.4 Scope of the Research

The research scopes are as below:

a) 3.7 wt. % HCL is used for acid hydrolysis and 5 wt. % NaOH is used for alkaline hydrolysis and analyzed under FTIR. The FTIR result was supported by the FTIR and SEM result from previous experimental studies.

b) The Young's modulus and surface morphology of the nanocomposites were analyzed via reported experimental studies under pure PLA and fiber loading of 5 wt% and 10 wt%.

c) Halpin-Tsai and Percolation equations are used to predict the modulus of the nanocomposites. The predicted modulus is compared with the reported experimental modulus under fiber loads at 5 wt% and 10 wt%. Error percentage is measured for the result comparison.

1.5 Significant of Study

The significant of the research are as below:

a) Biodegradable polymers are designed to degrade under action of organisms. The disposal of waste polymer materials can be solved.
b) Development of composites with natural fiber and biodegradable polymer in order to reduce the use of petroleum derived product.
c) Natural fiber in nano form level is able to bond the matrix firmly in order to increase the mechanical and physical properties of material in many applications.

1.6 Thesis Organization

The organization of this thesis consists of five chapter. Chapter 1 is about the research background, problem statement, objectives, scope, significant of research are delineated in order to better define the addition of NFC in PLA to have a better physical and mechanical properties addressed in this thesis. Chapter 2 literature review comprises previous study or research about the natural fiber, biopolymer, NFC, composite, physical and mechanical properties of the composite and the method to extract the nanocellulose. Chapter 3 methodology describes the raw materials and process used for completing the project. It includes methods for fiber characterization and theoretical models for predicting the modulus of composite materials. Chapter 4 is analyzed the results obtained by the methods stated in Chapter 3. In Chapter 5, conclusion and recommendation about this research are examined.

CHAPTER 2

LITERATURE REVIEW

This chapter describes previous research work in various fields related to the study that were defined and completed by several researchers a few years ago. This chapter focused on biodegradable polymers and nanofibrillated cellulose in biodegradable matrix composites. The properties of cellulose and polymers will be discussed as a reference for influencing the properties of composites. The morphology and mechanical properties of nanocellulose jute fiber in the composite will discuss as well.

2.1 Natural Fiber

Natural fiber is a renewable material that abundant on earth and produced by plant, animal and geological processes. Natural fibers such as kenaf, flax and jute have been widely used in diverse applications such as automotive industry and aerospace (Hassan et al., 2012). Table 2.1 shows the comparison of the mechanical properties of natural and synthetic fibers. Natural fibers have been used as fillers to improve the performance of composites as it has advantages over synthetic fiber like they are high specific strength, low density, low cost, high degree of flexibility, recyclable, biodegradable and sustainable (Hassan et al.,2012).

Table 2.1: Mechanical Properties of Natural and Synthetic Fiber (reproduced from Pickering et al., 2016)

Fiber	Density (g/cm^{-3})	Tensile Strength (MPa)	Stiffness /Young's modulus (GPa)	Specific tensile strength (MPa/gcm^{-3})	Specific Young Modulus (GPa/gcm^{-3})
Jute	1.3-1.5	393-800	10-55	300-610	7.1-39
Glass	2.5	2000-3000	70	800-1400	29