



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

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by

TANG PEI CHIN

B051610041

961227-01-6414

FACULTY OF MANUFACTURING ENGINEERING

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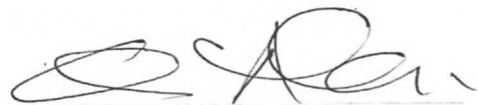
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Disahkan oleh:

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

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Author's Name : TANG PEI CHIN
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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:



.....
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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 diperolehi 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.
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TABLE OF CONTENTS

ABSTRAK	i
ABSTRACT	ii
DEDICATION	iii
ACKNOWLEDGEMENT	iv
TABLE OF CONTENTS	v
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF EQUATIONS	xii
LIST OF ABBREVIATIONS	xiii
LIST OF SYMBOLS	xiv
CHAPTER 1: INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	3
1.3 Objective	4
1.4 Scope of the Research	4
1.5 Significant of Study	5
1.6 Thesis Organization	5
CHAPTER 2: LITERATURE REVIEW	6
2.1 Natural Fiber	6
2.1.1 Jute fiber	8

2.1.2 Cellulose	9
2.2 Biopolymer	9
2.2.1 Polylactic acid (PLA)	10
2.3 Nanocellulose	11
2.4 Extraction of Nanocellulose	12
2.4.1 Chemical Pre-treatment	12
2.4.1.1 Physical Properties of the Untreated and Chemical treated Fiber	14
2.4.1.2 Chemical Properties of the Untreated and Chemical treated Fiber	15
2.5 Compression Moulding for NFC-PLA Matrix Composite	16
2.6 Nanotechnology	17
2.6.1 Nano Fibrillated Cellulose (NFC)	18
2.6.2 Effect of NFC dispersion in composite	19
2.7 Composite	20
2.7.1 Polymer Matrix composites	21
2.7.2 Natural Fiber Reinforced composite	22
2.8 Properties of Composite	23
2.8.1 Physical Properties of Polymer Matrix Composite	23
2.8.2 Mechanical Properties of Polymer Matrix Composite	24
2.8.3 Elastic Modulus Prediction from The Theoretical Model	26
2.9 Application of Nanocomposite	27
CHAPTER 3: METHODOLOGY	28
3.1 Overview	28
3.2 Raw Materials	30
3.2.1 Filler	30

3.2.2 Matrix	30
3.3 Preparation of Jute Fiber	31
3.4 Pretreatment of Jute Fiber	31
3.4.1 Chemical Pretreatment	31
3.4.2 Fibrillation Process	33
3.4.3 Sample Preparation of Fibrillated Nanocellulose Jute Fiber	33
3.4.3.1 Vacuum filtering	33
3.4.3.2 Oven Drying	34
3.5 Characterization	34
3.5.1 Fourier Transform Infrared Spectroscopy (FTIR)	34
3.5.2 Scanning Electron Microscopy	35
3.6 Preparation of the NFC-PLA composite	36
3.6.1 Internal Mixer	36
3.6.2 Hot pressing Moulding	36
3.7 Mechanical Testing	37
3.8 Theoretical Model	38
CHAPTER 4: RESULTS AND DISCUSSION	40
4.1 Characterization of Jute fiber	40
4.1.1 Fourier Transform Infrared Spectroscopy (FTIR)	40
4.1.2 Scanning Electron Microscopy (SEM)	44
4.2 Nano Fibrillated Cellulose Reinforced Polylactic Acid (NFC-PLA) Composite	46
4.2.1 Young's Modulus	46
4.2.2 Characterization of NFC-PLA composite	49
4.3 Theoretical Model of Young's Modulus	50

CHAPTER 5: CONCLUSION AND RECOMMENDATION	56
5.1 Conclusion	56
5.2 Recommendations	58
5.3 Sustainable Design and Development	58
5.4 Complexity of the study	59
5.5 Life Long Learning	60
REFERENCE	61
APPENDICES	
A Gantt Chart of FYP I	69
B Gantt Chart of FYP II	70

LIST OF TABLES

2.1	Mechanical Properties of Natural and Synthetic Fiber	6
2.2	Chemical Compositions of Different Bast Fibers	8
2.3	Comparison between cellulose crystals and common polymer reinforcement materials	11
2.4	Mechanical properties of untreated and treated kenaf fibers	14
2.5	Mechanical properties of PLA composites Reinforced with different types of fibers	16
2.6	Tensile properties of neat and nanocomposite PLA films composite	25
2.7	Mechanical characteristics of natural fibers reinforced	25
4.1	FTIR absorption bands of cellulosic materials	41
4.2	Young's Modulus of nanocellulose fiber reinforced PLA composite (GPa)	46
4.3	Modulus comparison of the previous experimental studies and Percolation Theory against nanofiller loading	50
4.4	Modulus comparison of the previous experimental studies and Halpin-Tsai model against nanofiller loading	50

LIST OF FIGURES

2.1	Classification of Natural Fibers	7
2.2	Structure of Plant Cell Wall in Lignocellulosic Biomass	8
2.3	Representation of cellulose molecule	9
2.4	Direct and ring-opening polymerization method of PLA	10
2.5	Process flow of extraction of nanocellulose	12
2.6	Extraction of nanocellulose from lignocellulosic biomass	13
2.7	a) Untreated jute fiber and (b) alkali treatment of jute fiber	14
2.8	Comparison of FTIR spectra between (a) untreated, (b) alkali treated jute fibers, (c) purified cellulose and (d) blender 20 times	15
2.9	Preparation of PLA composites reinforced with cellulose mat	16
2.10	Fiber size and associated manufacturing/ processing technologies	17
2.11	Extraction of nanocellulose from lignocellulosic biomass	18
2.12	Formation of nano fibrillated kenaf cellulose at 23000 rpm in (a) 5 min and (b) 15 min	19
2.13	Stress-strain curve of different types of filler	21
2.14	SEM image of fracture surface (a) 5 wt% JNF/PLA nanocomposites film. (b) 10 wt% JNF/PLA nanocomposites film.	23
2.15	SEM tensile fracture surface of (a) pure PLA (b) PLA/ NFKC at 7500 rpm, (c) PLA/ NFKC at 15000 rpm & (d) PLA/ NFKC at 23000 rpm for 15 min	24
2.16	Comparison between the experimental result and predictive mechanical models of JNF/PLA composites against volume fraction	26
2.17	Application of nanocellulose in various field	27
3.1	Project Process flow	29
3.2	Vibratory Sieve Shaker	31
3.3	Set Up of Chemical Treatment of Jute Fiber	32

3.4	The (a) treated jute fiber washed with distilled water and (b) the treated jute fiber at neutral pH	32
3.5	Vacuum Filtering	34
3.6	JASCO FT/IR-6100 FTIR analyzer	35
3.7	Zeiss SEM type Evo 50 series	35
3.8	Sputter Coater Machine	36
3.9	Test specimen dimensions defined by ASTM D638-10 (Type 1)	37
3.10	Universal Testing Machine (UTM)	37
4.1	FTIR spectra between as-received jute fiber and chemically treated jute fiber	41
4.2	FTIR spectra between as received (ITL-AR) and chemically treated industrial tea leave fiber (ITL-CT)	43
4.3	SEM image of (a) untreated jute fiber	44
4.4	SEM image of (a) chemical treated jute fiber and (b) Chemical treated kenaf fiber	45
4.5	Young's modulus of the nanocellulose fiber reinforced PLA composite	46
4.6	SEM image of (a) neat PLA and Reinforced PLA composites with NCFHL under (b) 5 wt% and (c) 10 wt%	49
4.7	Modulus Comparison between previous experimental studies and predicted theoretical models of JNF/PLA composite	51
4.8	Modulus Comparison between previous experimental studies and predicted theoretical models of SNCF/PLA composite	51
4.9	Modulus Comparison between previous experimental studies and predicted theoretical models of NCFHL/PLA composite	52
4.10	Error percentage between predicted theoretical models and experimental studies of JNF/PLA composite	53
4.11	Error percentage between predicted theoretical models and experimental studies of SCNF/PLA composite	54
4.12	Error percentage between predicted theoretical models and experimental studies of NCFHL/PLA composite	54

LIST OF EQUATIONS

3.1	Tensile Strength	38
3.2	Tensile Modulus	38
3.3	Halpin-Tsai Equation	38
3.4	Percolation model	39
3.5	Error Percentage	39

LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
FESEM	-	Field Emission Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
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