UNIVERSITI TEKNIKAL MALAYSIA MELAKA

EFFECT OF NANO CELLULOSE INDUSTRIAL TEA LEAF FIBER ON THE PHYSICAL AND MECHANICAL PROPERTIES OF POLYLACTIC ACID (PLA) COMPOSITE

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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I hereby, declared this report entitled "Effect of Nano Cellulose Industrial Tea Leaf Fiber on the Physical and Mechanical Properties of Polylactic Acid (PLA) Composite" is the result of my own research except as cited in references.

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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:

……………………….................................

(PROFESOR DR. QUMRUL AHSAN)

ABSTRAK

Kajian ini bertujuan untuk mengkaji hasil selulosa nanofibrillated (NFC) selepas proses kimia dan proses fibrilasi serat ITL oleh kaedah pengemparan, untuk menganalisis morfologi pengedaran dan penyebaran NFC dalam NFC-PLA komposit menggunakan OM dan SEM, serta mengkaji kesan pengukuhan NFC terhadap sifat mekanik komposit NFC-PLA dengan menggunakan ujian mekanikal. Pertama, serat ITL telah dihaluskan kepada skala nano menerusi proses rawatan dan fibrillasi kimia. Dalam proses fibrillasi, pelbagai parameter suhu pengadunan digunakan untuk mendapatkan hasil selulosa nanofibrilasi yang tinggi dengan kaedah sentrifugal. Kemudian, pelet PLA dengan jumlah serat nanoselulosa yang berbeza dikenakan tekanan panas untuk membentuk komposit NFC-PLA. Teknik pencirian OM dan SEM dijalankan bagi mengkaji morfologi penyebaran dan penyebaran NFC dalam komposit NFC-PLA. Ujian lenturan dilakukan untuk komposit NFC-PLA untuk memeriksa kekuatan lenturan dan modulus lenturan. Dari kajian ini, hasil pencirian dan analisis menunjukkan bahawa serat ITL dicampur pada 20℃ dengan kelajuan 23000rpm dan masa 10 minit memberikan hasil fibril yang lebih baik (24.19%) daripada serat ITL yang dicampur pada 0℃ (16.26%) dan 80℃ (19.01%) dengan kaedah pengemparan. Peningkatan maksimum kekuatan lenturan sebanyak 7.43% dan modulus lentur sebanyak 55.30% diperolehi untuk komposit NFC-PLA dengan serat ITL fibril yang diperoleh daripada kelajuan campuran, masa dan suhu 23000rpm, 10minutes dan 20℃ berbanding komposit PLA tulen. Ciri lentur menunjukkan bahawa kekuatan dan modulus telah bertambah baik dengan kandungan nanofiber yang meningkat.

ABSTRACT

This research is to investigate the yield of nanofibrillated cellulose (NFC) after chemical treatment and fibrillation process of industrial tea leaves (ITL) fibers by centrifugal method, to analyze the distribution and dispersion morphology of NFC in nanofibrillated cellulose reinforced polylactic acid (NFC-PLA) composite by optical microscopy (OM) and scanning electron microscopy (SEM), and to investigate the reinforcing effect of NFC on mechanical properties of NFC-PLA composite by using mechanical testing. Firstly, ITL fibers were fibrillated to nanoscale by chemical treatment and fibrillation process. In fibrillation process, various parameter of blending temperatures were used to get the high yield of nanofibrillated cellulose by centrifugal method. Then, PLA pellet with different volume of nanocellulose fibers underwent hot pressing to form the NFC-PLA composite. Characterization technique of OM and SEM was implemented to study the distribution and dispersion morphology of NFC in NFC-PLA composite. The flexural test was conducted for the NFC-PLA composite to examine its flexural strength and flexural modulus. From this research, the results indicated that the ITL fibers blended at 20℃ with blending speed and time of 23000rpm and 10minutes provides better fibrillated yield (24.19%) than ITL fibers which blended at 0℃ (16.26%) and 80℃ (19.01%) by centrifugation method. The maximum increase in flexural strength of 7.43% and flexural modulus of 55.30% was observed for NFC-PLA composites with fibrillated ITL fiber obtained using blending speed, time and temperature of 23000rpm, 10minutes and 20℃ compared to pure PLA composite. The flexural properties indicated that the strength and modulus were improved with increased nanofiber contents.

DEDICATION

Only

my beloved father, Lin Kee Yong

my appreciated mother, See Nyet Yuen

my adored sister and brother, Lin Wei Lin & Lin Wei Chien

for giving me moral support, money, cooperation, encouragement and also understandings

Thank You So Much & Love You All Forever

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LIST OF ABBREVIATIONS

ASTM	-	American society for testing and materials
BHE-SW	-	Fine fiber
CNF	-	Cellulose nano fiber
CO_2	-	Carbon dioxide
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier transform infrared spectroscope
H_2O	-	Water
HCI	-	Hydrochloric acid
ITL	-	Industrial tea leaves
NaOH	-	Sodium hydroxide
NFC	-	Nano fibrillated cellulose
NFC-PLA	-	Nano fibrillated cellulose reinforced polylactic acid
OM	-	Optical microscope
PLA	-	Polylactic acid
PTFE	-	Polytetrafluoroethylene
SEM	-	Scanning electron microscope

LIST OF SYMBOLS

%	-	Percentage
b	-	Width of specimen tested in millimeter
d	-	Thickness of specimen tested in millimeter
℃	-	Degree Celsius
cm^{-1}	-	Reciprocal centimeter
g	-	Gram
GPa	-	Gigapascal
kN	-	Kilo Newton
L	-	Support span in millimeter
m	-	Slope of tangent to the initial straight line portion of load deflection curve (N/mm)
min	-	Minute
ml	-	Milliliter
mm	-	Millimeter
μm	-	Micrometer
mol/L	-	Moles per litre
MPa	-	Megapascal
N	-	Newton
nm	-	Nanometer
P	-	Maximum load applied on test specimen (N)
Pa	-	Pascal
rpm	-	Revolution per minute
wt%	-	Weight percent
λ	-	Wavelength

LIST OF NOMENCLATURES

ITL-AR	-	as-received ITL fiber
ITL-CT	-	chemically-treated ITL fiber
ITL-T0	-	fibrillated ITL fiber at blending temperature of 0℃
ITL-T20	-	fibrillated ITL fiber at blending temperature of 20℃
ITL-T80	-	fibrilated ITL fiber at blending temperature of 80℃
NFC-PLA-T0-1	-	NFC-PLA composite with fibrillated ITL fiber at 0℃ and 1% fiber loading
NFC-PLA-T0-3	-	NFC-PLA composite with fibrillated ITL fiber at 0℃ and 3% fiber loading
NFC-PLA-T0-5	-	NFC-PLA composite with fibrillated ITL fiber at 0℃ and 5% fiber loading
NFC-PLA-T20-1	-	NFC-PLA composite with fibrillated ITL fiber at 20℃ and 1% fiber loading
NFC-PLA-T20-3	-	NFC-PLA composite with fibrillated ITL fiber at 20℃ and 3% fiber loading
NFC-PLA-T20-5	-	NFC-PLA composite with fibrillated ITL fiber at 20℃ and 5% fiber loading
NFC-PLA-T80-1	-	NFC-PLA composite with fibrillated ITL fiber at 80℃ and 1% fiber loading
NFC-PLA-T80-3	-	NFC-PLA composite with fibrillated ITL fiber at 80℃ and 3% fiber loading
NFC-PLA-T80-5	-	NFC-PLA composite with fibrillated ITL fiber at 80℃ and 5% fiber loading

CHAPTER 1

INTRODUCTION

This chapter discusses the research background, problem statements, objectives and research scope of this project.

1.1 Research Background

The disposal of waste polymer material has become a major worldwide environmental problem after the end of its useful life. Environmental problems and reduced petroleum resources around the world have prompted many researchers to developed composites with natural fiber and biodegradable polymer to reduce the use of petroleum derived product. Nowadays, biodegradable polymers have become the target of replacing petroleum-based polymers in many applications because of environmental problems and reduced fossil resources.

Polylactic acid (PLA) is thermoplastic aliphatic polyester with biodegradable and bioactive properties derived from renewable resources such as cassava and corn. PLA has the excellent commercial potential because of its biocompatibility, mechanical strength and easy to process (Huda et al., 2008). The great advantage of PLA as a biodegradable plastic is its versatility. When exposed to the environment, it naturally degrades into H_2O and CO_2 without causing any harm to the environment. PLA is used in a variety of applications such as degradable plastic bags, bottles, food packaging and automotive applications.

However, the high brittleness, less flexible and poor thermal stability of PLA lead to processing difficulties and lower product performance, which limits the wide application of PLA. Moreover, PLA can be reinforced by addition of natural fiber in PLA composites as an effective method to improve the physical and mechanical properties of PLA. Natural fibers are generally considered as ideal candidate reinforcement in green composites because of their low cost, low density, renewability and biodegradability. A simple definition of natural fibers is a non-synthetic or artificial fiber. Natural fiber can come from plants or animals. In this research study, industrial tea leaf (ITL) fibers have been selected as reinforcing materials in PLA composites due to its long sustainability and availability in low cost. ITL is known as one of the renewable lignocelluloses wastes that is biodegradable and has no negative impact to the environment. Recently, waste tea leaves from tea producing factories are considered a new resource for biological research due to its low density, high specific stiffness, renewability and biodegradability. Hence, tea leaf fibers are widely used in biodegradable polymers to prepare biodegradable composite materials. Adding ITL fibers to a PLA composite will not only improve the mechanical properties of the composite, but also reduce the cost of the final product because it is inexpensive and readily available.

Tea leaf is a lignocelluloses biomaterial and it consists of mainly cellulose, hemicelluloses and lignin. Scientifically, cellulose is an insoluble long chain of linked sugar molecules which helps to increase the stiffness of the composite material. Besides that, cellulose can utilize in more effective way by fibrillated the cellulose fiber into nanocellulose. Recently, nanocellulose has received great attention and it is combined with a biodegradable polymer matrix such as PLA by hot pressing to produce “green” environmentally friendly bionanocomposite material (Berglund *et al*., 2012). This is because nanocellulose is a natural material with excellent strength properties, biodegradable, biocompatible and possesses extraordinary enhancement potential.

It is evidenced from research work carried out by Kowalczyk *et al.* (2011) that the addition of cellulose nanofibers extracted from kenaf pulp to a PLA matrix increased tensile modulus and tensile strength by 24% and 21%, respectively. Nanocellulose is a cellulosic material having at least one dimension of nanometers, usually produced by decomposition of cellulose fibers, producing two different cellulose nanoparticles having different properties, which are nanocellulose whiskers and nanofibrillation cellulose (NFC).

However, in this project, nanofibrillated cellulose (NFC) will be the focus on this study due to their high mechanical properties, low density, economic value, biodegradability and renewability. NFC produced from natural cellulose can uses as a reinforcing agent in polymer composites because of its unique characteristics of high strength and stiffness, low weight and biodegradability. NFC consisted of longer and more flexible microfibers having a width of about 15-20 nm and a length of a few micrometers (Berglund *et al.*, 2012). NFC is usually a hydrogel with a viscosity and it able to form a transparent film after drying. Both of these key properties are related to their high specific surface area (at least 10 times greater than cellulose fibers) and their extensive hydrogen bonding capabilities.

Because NFC is more flexible, longer and has a larger surface area than any other fiber, it has been developed for many scales, ranging from addition in food, biodegradable food packaging and so on. Therefore, in this study, the industrial tea leaves were fabricated into nanofabricated cellulose by chemical-mechanical treatment and then filled into the PLA composite material, which not only can increase the value of ITL, but also can improve the mechanical properties of the PLA composite material.

1.2 Problem Statement

Biodegradable polymers have become the target of replacing petroleum-based polymers in many applications because of environmental concerns. However, the main challenges in using biodegradable polymers in industrial applications are mechanical performance limitations and production costs. Most biodegradable polymers are very expensive and have weak structure strength compared to synthetic polymer. There are several methods have been made to enhance the strength of the biodegradable composite, such as addition of natural fibers to the biodegradable polymer. This is due to the excellent properties of natural fibers such as low density, high specific strength and specific stiffness. The reinforcement of kenaf fibers increases the flexural strength of composite by 36% compared to the pure biopolymer (Sood *et al.*, 2017). But it is still new, and researchers are still concerned about the compatibility of composites. Moreover, natural fibers are hygroscopic, which causes the poor interactions between natural fibers and polymer matrix and lower durability because of their hydrophilic nature. Since there are differences in