PRODUCTION AND CHARACTERIZATION OF FIBRILLATED NANOCELLULOSE FIBER DERIVED FROM SPENT TEA LEAVES

ANGELINE WO WENG WEI
B051210079

UNIVERSITI TEKNIKAL MALAYSIA MELAKA
2016

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PRODUCTION AND CHARACTERIZATION OF FIBRILLATED NANOCELLULOSE FIBER DERIVED FROM SPENT TEA LEAVES

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

by

ANGELINE WO WENG WEI

B051210079

920119-08-5294

FACULTY OF MANUFACTURING ENGINEERING

2016

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **Production and Characterization of Fibrillated Nanocellulose Fiber Derived from Spent Tea Leaves**

SESI PENGAJIAN: 2015/16 Semester 2

Saya **ANGELINE WO WENG WEI**

mengaku membenarkan Laporan 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:

_________________________ _________________________

Alamat Tetap:

44, JALAN THEATRE,

30300, IPOH, PERAK.

Tarikh: 18th JUN 2016

Cop Rasmi:

Tarikh: _________________________

** 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 “Production and Characterization of Fibrillated Nanocellulose Fiber Derived from Spent Tea Leaves” is the results of my own research except as cited in references.

Signature	:	………………….
Author’s Name	:	ANGELINE WO WENG WEI
Date	:	07th JUNE 2016

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirement for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory committee is as follows:

………………………………………………

(Supervisor)

(PROF. DR. QUMRUL AHSAN)

ABSTRAK

Kajian ini memberi tumpuan kepada fibrilasi dan pencirian gentian nanoselulosa dari hampas daun teh (HDT). HDT dikenali sebagai salah satu bahan buangan lignoselulosa yang boleh dilupuskan dan tidak memberi kesan negatif kepada alam sekitar. Objektif kajian ini adalah untuk menentukan daya maju mengekstrak gentian nanoselulosa dari HDT dan untuk mencirikan gentian nanoselulosa dengan mikroskop optik, mikroskop imbasan elektron, mikroskop pelepasan bidang imbasan elektron, X-ray pembelauan, Fourier spektroskopi inframerah dan kaedah pengemparan. Kajian ini menyimpulkan pengeluaran dan pencirian gentian nanoselulosa berasal daripada HDT. Dua gred HDT akan digunakan dalam kajian ini, iaitu BHE-BM dan BHE-SW dari tangkai tumbuhan teh. Sampel nanoselulosa diekstrak adalah tertakluk kepada pemerhatian mikroskopik, pencirian fisikal dan kimia, dan hasil fibrilasi keazaman. Dari kajian ini, keputusan pencirian dan analisis telah menunjukkan bahawa HDT gred yang lebih halus iaitu BHE-SW menyediakan hasil fibrilasi yang lebih baik sebanyak 15.22% berbanding dengan gred kasar iaitu BHE-BM yang berhasil fibrilasi sebanyak 14.68%. Tambahan pula, kesan parameter fibrilasi seperti kelajuan pengadunan dan masa pengadunan dibandingkan dalam kedua-dua gred HDT dalam kajian ini.

ABSTRACT

This research focuses on the fibrillation and characterization of nanocellulose fibers derived from spent tea leaves. Spent tea leaves (STL) are known as one of the renewable lignocellulosic wastes that are biodegradable and no negative impact to the environment. The objectives of this research are to determine the viability of extracting nanocellulose fiber from STL and to characterize the extracted nanocellulose STL fibers by Optical Microscopy, Scanning Electron Microscopy, Field Emission Scanning Electron Microscopy, X-ray Diffraction, Fourier Transform Infrared Spectroscopy and centrifugation method. This research deduces the production and characterization of nanocellulose fiber derived from STL. Two grades of STL are used in this research, which are BHE-BM and BHE-SW from the stalk of the tea plant. The extracted nanocellulose samples are subjected to microscopic observations, physical and chemical characterization, and fibrillation yield determination. From this research, the characterization and analysis results have shown that the STL of finer grade BHE-SW provides better fibrillation yield of 15.22% than the coarser grade BHE-BM with fibrillation yield of 14.68%. Furthermore, the effect of fibrillation parameters such as blending speed and blending time are compared within both grades of STL in this research.

DEDICATION

Dedicated to
my beloved father, Wo Chee Seng
my appreciated mother, Fong Li Li
and my adored siblings Kevin Wo Kin Choong and Winnie Wo Weng Nie
for giving me moral support, cooperation, encouragement and also understanding.

ACKNOWLEDGEMENT

First of all, I would like to express my highest gratitude to my beloved supervisor, Prof. Dr. Qumrul Ahsan for his advices, valuable information and guidance, which helped me in completing the Final Year Project on time and fulfill the requirements.

Besides, a special thanks is also dedicated to my academic advisor, Dr. Noraiham Binti Mohamad for her nurturing guidance and valuable advises, which helped me to cope with the pressure of my project and academic assignments.

Last but not least, I would like to show my deepest appreciation to my family members for their moral support, understanding and encouragement throughout this project.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ATR	-	Attenuated Total Reflection
BHE-BM	-	coarse spent tea leaves grade
BHE-SW	-	fine spent tea leaves grade
BNC	-	Bacterial Nanocellulose
cm^{-1}	-	reciprocal centimeter
CNC	-	Cellulose Nanocrystals
CNF	-	Cellulose Nanofibrils
CTE	-	Coefficient of Thermal Expansion
FAO	-	Food and Agriculture Organization
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
H_2SO_4	-	Sulphuric acid
HCl	-	Hydrochloric acid
kV	-	Kilovolt
LCW	-	Lignocellulosic Waste
min	-	minute
ml	-	milliliter
mm	-	millimeter
$NaClO_2$/AA	-	Sodium Chlorite/Acetic Acid
NaOH	-	Sodium hydroxide
nm	-	nanometer
OM	-	Optical Microscopy
pH	-	potential hydrogen
PTFE	-	Polytetrafluoroethylene
rpm	-	revolution per minute
SEM	-	Scanning Electron Microscopy
STL	-	Spent Tea Leaves
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

^{o}C	-	degree celcius
Ø	-	diameter
µm	-	micronmeter
%	-	percent
λ	-	wavelength
wt%	-	weight percent

LIST OF NOMENCLATURES

BM-AR	-	as-received coarse STL
BM-T	-	pretreated coarse STL
BM-T-52-1	-	pretreated coarse STL at blending speed 5200 rpm for 1 min
BM-T-52-5	-	pretreated coarse STL at blending speed 5200 rpm for 5 min
BM-T-52-10	-	pretreated coarse STL at blending speed 5200 rpm for 10 min
BM-T-135-1	-	pretreated coarse STL at blending speed 13500 rpm for 1 min
BM-T-135-5	-	pretreated coarse STL at blending speed 13500 rpm for 5 min
BM-T-135-10	-	pretreated coarse STL at blending speed 13500 rpm for 10 min
BM-T-235-1	-	pretreated coarse STL at blending speed 23500 rpm for 1 min
BM-T-235-5	-	pretreated coarse STL at blending speed 23500 rpm for 5 min
BM-T-235-10	-	pretreated coarse STL at blending speed 23500 rpm for 10 min
SW-AR	-	as-received fine STL
SW-T	-	pretreated fine STL
SW-T-52-1	-	pretreated fine STL at blending speed 5200 rpm for 1 min
SW-T-52-5	-	pretreated fine STL at blending speed 5200 rpm for 5 min
SW-T-52-10	-	pretreated fine STL at blending speed 5200 rpm for 10 min
SW-T-135-1	-	pretreated fine STL at blending speed 13500 rpm for 1 min
SW-T-135-5	-	pretreated fine STL at blending speed 13500 rpm for 5 min
SW-T-135-10	-	pretreated fine STL at blending speed 13500 rpm for 10 min
SW-T-235-1	-	pretreated fine STL at blending speed 23500 rpm for 1 min
SW-T-235-5	-	pretreated fine STL at blending speed 23500 rpm for 5 min
SW-T-235-10	-	pretreated fine STL at blending speed 23500 rpm for 10 min

CHAPTER 1

INTRODUCTION

This chapter describes the background study, problem statement, objectives and the scope of study.

1.1 Background Study

According to Wang *et al.* (2015), the most abundant renewable natural biopolymer on earth is cellulose, the main constituent that forms the cell walls of all plants and trees. Scientifically, cellulose is an insoluble long chain of linked sugar molecules. In nature, cellulose appears in the form of complex matrix called lignocellulose. The lignocellulosic biomass is mostly wasted in the form of agricultural losses and wastes of food processing industries. This kind of lignocellulosic waste (LCW) can be utilized for the production of value-added products (Mtui *et al.*, 2009). Lignocellulosic biomass primarily consists of cellulose, hemicellulose and lignin. Cellulose and hemicellulose in lignocellulosic biomass is one of the largest renewable reservoirs on earth that contains carbohydrates content of 60-70 % to the total volume, which is high enough for fiber extraction (Yang *et al.*, 2013). This makes lignocellulosic biomass an attractive feedstock as natural and renewable resource to the functioning of modern industrial societies (Anwar *et al.*, 2014). The concept of sustainability is most being encouraged in today's development and environmental management.

Chirayil *et al.* (2014) stated that nanotechnology is the understanding and controlling of matter at dimensions of roughly 1-100 nm, where unusual properties are often encountered. Nanomaterial from lignocellulosic biomass also known as nanocellulose. It has unique combination of good physical properties, which can produce products with low impact on the environment. Deepa *et al.* (2015) presented that nanocellulose can be categorized into cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC) based on the morphology differences. These novel forms of cellulose have very high economical potential in replacing synthetic fibers.

Fiber mechanical properties are improved with the decrease in fiber diameter (Dzenis, 2004). Khalil *et al.* (2014) claimed that many applications were studied by different researchers. The most commom application is nanocomposite that exhibits superior strength but light in weight. Hassan *et al.* (2015) improved the properties of paper sheets using microfibrillated cellulose from softwood and bagasse. Paper sheets that obtaining improved strength properties, increased in density and reduced in porosity is a big leap in paper industry.

1.2 Problem Statement

Nanomanufacturing process can be categorized into two techniques, which is bottom-up method and top-down method. According to Dzenis (2004), most of the nanofibers are produced by synthetic bottom-up method, which is high in processing cost and low uniformity of self-assembly nanofibers. Hence, top-down method such as fibrillation is one of the alternative ways of producing nanocellulose fiber.

Tea is one of the most consumable low cost beverages, after water (Hicks, 2009). According to Food and Agriculture Organization of the United Nations (FAO), more than three billion cups of tea are consumed daily worldwide. Chandrasekaran M. (2012) stated that waste tea leaves from tea-producing factories are considered a new resource for biological research. During the tea production process, the roasted tea leaves are sent for rolling to crush the leaf cells into smaller particles. In the rolling process, the crushed tea leaves producing residues from the leaf cells. Once the tea leaves is roasted and leaf cells is separated with the leaf veins, the spent tea leaves (STL) becomes a waste and has no significant value. A large amount of spent tea leaves (STL) will be left over everyday, and it can be easily collected with very low cost.

According to Begum *et al.* (2013), the first synthetic fiber nylon was born in the year of 1938. It has been developed from the human civilization. Synthetic fiber with exclusive mechanical strength has diverse applications ranging from load-carrying elements to sports equipment. However, synthetic fibers have some serious drawbacks such as high cost, high density, poor recycling and non-biodegradable properties. For these reasons, over the last few years' natural plant fibers are increasingly gaining attention as viable alternative to synthetic fiber. Meanwhile, the extraction of nanocellulose fibers from natural resources are not commonly disclose to the public due to its financial potential. Hence, efforts have to be done by current researcher in order to explore more natural resources of nanofibers.

1.3 Objectives

(a) To evaluate the viability of extracting nanocellulose fiber from Spent Tea Leaves (STL) by chemical pretreatment and fibrillation method.

(b) To characterize and analyze the extracted nanocellulose STL fibers by using OM, SEM, FESEM, XRD, FTIR and determine the yield of fibrillation by centrifugation method.

1.4 Scope

The scope of this study will focus on the fibrillation of nanocellulose fiber derived from STL and the characterization of the extracted fibers. STL will undergo chemical pretreatment to degrade the intermolecular bonding between cellulose, hemicellulose and lignin follow by fibrillation to isolate the lignocellulosic contents mechanically. Vacuum filtering will use to filter the fibrillated fibers. Then, the extracted fibrillated fibers will carry out further characterization and analysis.

CHAPTER 2

LITERATURE REVIEW

A literature review on previous research work in various areas which is relevant to this research is presented in this chapter.

2.1 Lignocellulosic Biomass

Wang *et al.* (2015) described lignocellulosic materials including agricultural wastes, forestry residues, grasses and woody materials are potentially developed for bio-fuel production, cellulosic fiber extraction and value-added product applications. University of Gujrat, Pakistan experimentally found that most of the agricultural lignocellulosic biomass is comprised of about 10-25% lignin, 20-30% hemicellulose, and 40-50% cellulose. However, the composition of lignocellulose depends on the lignocellulosic sources.

According to Jedvert *et al*. (2012), cellulose is responsible for mechanical strength of plant cell walls and hemicellulose macromolecules are often repeated polymers of pentoses and hexoses. Lignin usually forms a protective seal around the cellulose and hemicelluloses. Figure 2.1 shows the structure of lignocellulosic biomass with cellulose, hemicellulose and lignin.

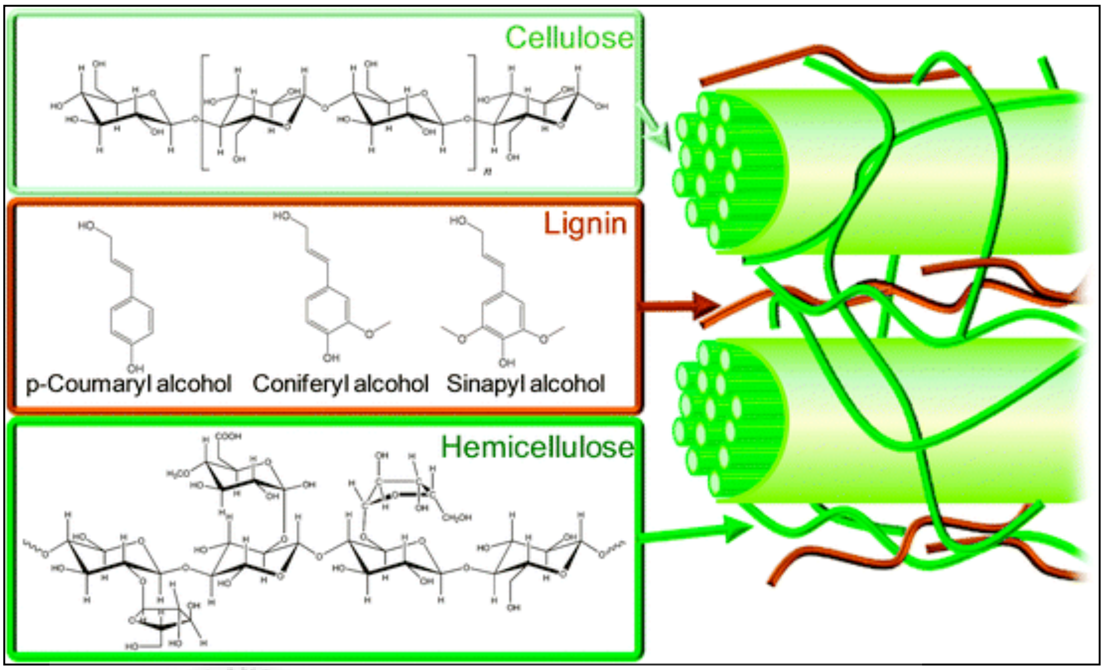

Figure 2.1: Structure of lignocellulosic biomass with cellulose, hemicellulose and lignin (Alonso *et al.*, 2012).

2.1.1 Cellulose

In 1838, cellulose from green plant is discovered and isolated by French chemist, Anselme Payen (Poletto *et al.*, 2013). Jedvert *et al.* (2012) stated that cellulose is a highly stable polymer consisting of glucose and attached with linear chains up to 12,000 residues. The repeat unit is shown in Figure 2.2.

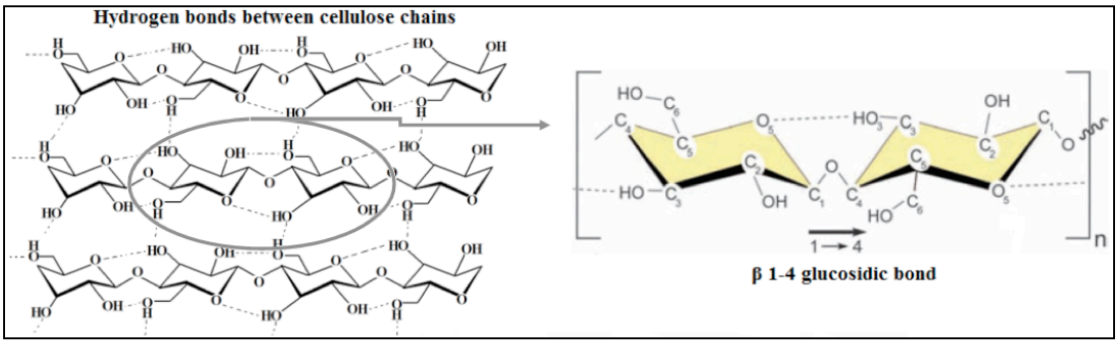

Figure 2.2: Molecular structure of a cellulose unit (Poletto *et al.*, 2013).