PREPARATION AND CHARACTERIZATION OF BANANA TRUNK DERIVED NANOCELLULOSE PREPARED BY ACID HYDROLYSIS METHOD

NURSYUHADA ISMAH BINTI MUHAMMAD ALI
B051310119

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PREPARATION AND CHARACTERIZATION OF BANANA TRUNK DERIVED NANOCELLULOSE PREPARED BY ACID HYDROLYSIS

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

by

NURSYUHADA ISMAH BINTI MUHAMMAD ALI

B051310119

930823-12-6730

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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 Bachelor of Manufacturing Engineering (Engineering Materials) (Hons). The members of the supervisory committee are as follow:

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ABSTRAK

Serat lignoselulosa menjadi tumpuan ramai terutama dari golongan pengkaji di masa kini kerana sifat-sifat semulajadinya yang mengagumkan. Pada masa kini, serat lignoselulosa dimanfaatkan untuk aplikasi-aplikasi seharian sebagai contoh, kraftangan, industri kain dan sebagainya. Oleh itu, tujuan kajian ini adalah untuk menyediakan bahan nanocellulosa yang diekstrak daripada serat batang pokok pisang menggunakan 40 % asid sulfurik. Secara kimia, penyediaan bahan nano ini adalah dengan menggunakan cara asid hidrolisis dimana asid sulfurik sebagai agen hidrolisis sehingga campuran asid dan serat tersebut mempunyai pembentukan kehabluran yang tinggi. Kajian ini juga adalah untuk menilai dan mengkaji keadaan optimum nanoselulosa bagi penghasilan struktur kristal yang tinggi dengan kesan masa dan suhu kepada hidrolisis asid. Komposisi unsur kimia dikenalpasti menggunakan mesin Fourier Mengubah Spektrometer Inframerah (FTIR) manakala, indeks penghabluran dan saiz kristalit dikenalpasti menggunakan X-ray Pembelauan Spektrometer (XRD). Keberkesanan rawatan alkali dinilai melalui sifat morfologi permukaan struktur nanoselulose serat batang pisang menggunakan Imbasan Elektron Mikroskopi (SEM). Oleh yang demikian, sellulosa nanokristal dapat dihasilkan dalam keadaan optimum 90 ˚C untuk 30 minit dengan indeks penghabluran 31.73 % dan 5.47 nm saiz kristallit dimana batang pisang bertidak sebagai sumber yang mampan kerana murah, mesra alam, mudah didapati dan dapat mengelakkan daripada pencemaran alam yang disebabkan oleh sisa batang pisang.

ABSTRACT

Lignocellulosic fibers have been received an intense attention to many researchers due to their tremendous advantages. Today, the lignocellulosic fibers have been value added through many ways such as handicraft, textile industry and so on. The aim of this study is to prepare nanocellulose in term of cellulose nanocrystals that derived from banana trunk fiber by using 40 wt% sulphuric acids (H_2SO_4) via acid hydrolysis method. Despite of this, the effect of hydrolysis time and temperature correlation is studied in order to find the optimum conditions of cellulose nanocrystals of banana trunk fibers which are possessed high crystallinity in their crystal structure by using acid hydrolysis method. The elemental chemical composition for different chemical stages is determined by using Fourier Transform Infrared (FTIR) while the crystallinity index and crystallite size is determined and evaluated by using X-ray diffraction spectrometer (XRD). The effectiveness of the alkali treatment of 6 wt% NaOH is evaluated through the surface morphology by using Scanning Electron Microcopy (SEM). Thus, the cellulose nanocrystals has been successfully produced with the optimum condition at 90 ˚C for 30 minutes by the 31.73 % crystallinity index by 5.47 nm of the crystallite which act as a sustainable source due to the cheap, eco-friendly and its avaibility while preventing pollution to the environment that caused by banana trunk wastes.

DEDICATION

To :

My beloved parents,

For their understanding and support

My friends,

For all those time that we spend time together to finish this final year project

ACKNOWLEDGEMENT

The final year project opportunity I had in my final year was a great chance for learning and professional development. Therefore, I consider myself as a very lucky individual as I was provided with an opportunity to be a part of it. I am also grateful for having a chance to meet wonderful people who led me through this project period. Bearing in mind, I am using this opportunity to express my deepest gratitude and special thanks to Dr. Rose Farahiyan Binti Munawar and Dr. Jefferie Bin Abd Razak whose in spite of being extraordinarily busy with their duties, took time out to hear, guide and keep me on the correct path and allowing me to carry out my duty. I express my deepest thanks to Dr. Mohd Zulkefli Bin Selamat, Dr Lau Kok Tee, Dr Edeerozey and Dr Nona Mery who are pleasurely shared their knowledge in my thesis. I choose this moment to acknowledge their contribution gratefully. It is my radiant sentiment to place on record my best regards, deepest sense of gratitude to my parents for their careful and precious guidance which were extremely valuable for my study. I perceive as this opportunity as a big milestone in my career development. I will strive to use gained knowledge in the best possible way, and I will continue to work on their improvement.

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LIST OF ABBREVIATIONS

AGU	-	Anhydroglucopyranose
ASTM	-	American Society for Testing and Materials
BC	-	Bacteria Cellulose
CNCs	-	Cellulose Nanocrystals
CNFs	-	Cellulose Nanofibrils
CNWs	-	Cellulose Nanowhiskers
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
MCC	-	Microcrystalline Cellulose
MFC	-	Microfibrillated Cellulose
NCC	-	Nanocrystalline Cellulose
NFC	-	Nanofibrilated Cellulose
SCB	-	Sugarcane Baggase
SEM	-	Scanning Electron Microscop
XRD	-	X-ray Diffraction Spectrometer

LIST OF SYMBOLS

wt. %	-	Weight Percentage
C	-	Degree Celsius
Λ	-	Lambda
pH	-	Potential of hydrogen
g	-	gram
min	-	minute
hr	-	hour
mg mL^{-1}	-	volume per unit mass

CHAPTER 1

INTRODUCTION

1.0 Introduction

This chapter describes the background of study, problem statement, objectives, research scopes and project significance of this research.

1.1 Background of Study

Banana plant (Musaceae) become the second largest crop in the world which contribute about 16 % of the world's fruit production and being cultivated over 130 tropical countries. There are three most common species of banana plant that are widely cultivated over the world are based on their genomes which are composed of two wild genomes, A (Musa acuminata) and B (Musa balbisiana) while the hybrid of these genomes are known as AB (Musa paradisiaca).

Instead of its delicious fruit, other parts of this plant also play roles in many applications such as its male buds can be used as vegetable source, the leaves can be as food wrapping or plate, the banana stem sap can be used in medical application. Banana fiber is mainly extracted from its trunk, leaves, and rachis. However, the main focused in this study for fiber extraction is from its trunk which is rich in cellulose content after its leaves. Banana fiber has attracted the attention due to its useful contribution in many applications such as in paper making, textile, biodegradable ropes, bags and natural sorbent (Mohapatra *et al*, 2010). Moreover, there is study that considered making the banana fiber as the reinforcing material in polymer matrix due to its good mechanical properties (Santosh *et al*., 2014).

―Nanocellulose" is the cellulosic extracts or a processed material which can be obtained from the abundance biopolymer cellulose and could be derived from plants, tunicates, algae and some special bacteria. Nanocellulose can be classified into three main types namely cellulose nanocrystals (CNCs), cellulose nanowhiskers (CNWs) and bacteria cellulose (BC) (Abitbol *et al*., 2016). In recent years, lignocellulosic fiber has gained the attention and become the main source in producing the nanocellulose due to their tremendous advantages properties such as renewable and low cost that making them as the source in producing the nanocellulose (Chiyaril *et al*., 2014). Banana trunk fiber is one of the lignocellulosic fiber that been used for many applications. However, the utilization of this lignocellulosic as the starting material for nanocellulose has not been much exploited. Therefore, this opens up the opportunity to maximize the renewable sources of nanocellulose instead by using other common lignocellulosic fibers such as sisal, kenaf, hemp, flax, grass and many more.

The production of nanocellulose differs based on its cellulose sources, types and application. Cellulose nanocrystals can be obtained by using chemical treatment or by the combination, mechanical and chemical method. Acid hydrolysis is the famous used method that used in producing a stable colloidal suspension of cellulose nanocrystals. In the process, the acid help in degrading the amorphous regions of hemicellulose and lignin thus, the crystals cellulose become the domain structure (George and Sabapathi, 2015). The crucial parameters such as temperature, time of hydrolysis and acid concentration must be considered in the acid hydrolysis because it could affect the result of nanocellulose obtained (Bondeson, 2006). In order to make the cellulose more accessible and an effective hydrolysis, chemical pretreatment is always been done prior the hydrolysis method. The most common pretreatment used is the alkali pretreatment in which, the cellulose fiber is subjected into bases solutions (Kumar *et al*., 2009).

The objective of this current work is to prepare and characterize the cellulose nanocrystals (CNCs) that derived from banana trunk fiber using acid hydrolysis (H_2SO_4) method by different hydrolysis time and temperature. This is due to lack of previous studies that using banana fiber trunk as the starting material for cellulose nanocrystals extraction with a complete characterization has been done. The cellulose nanocrystals obtained will be further analyzed and characterized by using the selected tools such as Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction microscopy (XRD) and

Transmission Electron Microscopy (TEM). Therefore, a well-define cellulose crystals structure and crystallinity can be evaluated due the optimum condition of hydrolysis focusing on the effect of hydrolysis time and temperature.

1.2 Problem Statement

This study focus on the preparation and characterization of nanocellulose derived from banana trunk fiber using acid hydrolysis method which is high in crystallinity and stable colloidal suspension of cellulose nanocrystals. In the way to produce the nanocellulose from the banana trunk fiber, several chemical treatment procedures need to be conducted. The selection of banana trunk fiber as the starting material of nanocellulose source in this study is to help in reducing the potential of environmental problem that cause by banana trunk waste due to the current scenario, most of the farmers will abandon the banana trunk waste on the cultivation land right after the banana fruit being harvested. Therefore, in order to value added this wastage is by converting it into the nanomaterial despites of its availability, green and cheap material properties. In addition, the preparation of nanocellulose in this study is that, the banana trunk fiber is rich with the cellulose content with 69 % (Faradilla *et al*., 2016) which is promising a high yield of nanocellulose. Nevertheless, to date no studies have been discussed on the preparation of derived nanocellulose from banana trunk fiber by using acid hydrolysis method and its characterization with the effect of time and temperature on the crystallinity has not yet exploit. Despite the weakness, the preparation and characterization of nanocellulose derived from banana trunk is fully conducted in this study.

Two main phases of chemical treatment were conducted in this study which is involving the chemical treatments of banana trunk fiber in order to produce cellulose nanocrystals. The alkali pretreatment was used to extract the purified cellulose banana fiber. Then, acid hydrolysis process was used to convert the purified cellulose into the nanocellulose in term of cellulose nanocrystals (CNC). The acid hydrolysis is the main chemical treatment in this study and were conducted in several specific conditions with the selected parameters such as acid concentration, hydrolysis time and temperature. An experimental was designed conventionally according to the selected parameters in order to find the optimum condition for nanocellulose banana trunk fiber with the highest crystallinity. High potential of cellulose nanocrystals with the high crystallinity will give high strength and rigidity even will provide a good dispersion in polymer matrix that will be useful as the reinforcing potential material in biocomposites (Ng *et al*., 2015) if compared with cellulose nanofibrils cellulose.

1.3 Objectives

The objectives of this study are :

(a) To prepare the nanocellulose banana trunk fiber via a simplified acid hydrolysis method with selected parameters.

(b) To determine the elemental composition and crystal structure of the nanocellulose banana trunk fiber by Fourier Transmission Infrared spectroscopy (FTIR) and X-ray diffraction microscopy (XRD).

(c) To characterize the surface morphology and structure of nanocellulose banana fiber by using Scanning Electron Microscopy (SEM).

1.4 Research Scopes

The aim of this study is to prepare the nanocellulose derived from banana trunk and to study the influenced of morphological and crystallinity from obtained nanocellulose. The experiments were conducted in different condition with the selected parameters in order to find the optimum condition to produce high crystallinity and a well-define crystals of cellulose nanocrystals banana fiber.

For the first objective, the banana trunk fiber were treated in alkali treatment with sodium hydroxide (NaOH) to obtain a purified cellulose before subjected in acid hydrolysis that will conducted in different conditions according to the selected parameters such as acid concentration, hydrolysis time and temperature. The acid hydrolysis was conducted in order to obtain the nanocellulose derived from banana trunk fiber. In this process, Sulphuric acid (H_2SO_4) is used as the hydrolyze agent in acid hydrolysis as it can produce a stable colloidal suspension of cellulose nanocrystals as compared to other type of acids. The concentration of sulphuric acid was fixed at 64 wt % which is the optimum concentration in acid hydrolysis method. Moreover, the time and temperature were varied since they are correlated to each other (Bonjesson and Westman, 2015).

As to fulfill the objective number two and three, the selected characterization tools were used to characterize the crystals structure and the surface morphology of the cellulose nanocrystal banana fiber. There were three characterization tools that were used in this study namely Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction microscopy (XRD) and Scanning Electron Microscopy (SEM). The FTIR analysis will be used to determine the elemental of the raw banana fiber and two main chemical phases in this study, alkali treated and acid hydrolyzed fiber with the highest crystallinity index. The XRD analysis will be used to study the crystallinity index of different condition of hydrolyzed banana trunk fiber while SEM is used to characterize the surface morphology and measure the fiber of nanocellulose obtained of the different condition of hydrolysis.

1.5 Project Significance

Since the nanocellulose that are extracted from the natural biomass of banana fiber trunk has no yet being exploited, it is significant to study the suitable method to produce nanocellulose in term of cellulose nanocrystals which derived from banana trunk fiber. Moreover, in order to produce high crystallinity of cellulose nanocrystals banana trunk fiber, the optimum condition of acid hydrolysis were studied. In addition, due to its availability, green material and cheap material thus, it provides another alternative raw material for cellulosic sources instead of other available cellulosic sources and this feature provide a sustainable cellulose source for the nanocellulos production. The isolation of nanocellulose from banana trunk fiber itself can help to save the environment problems that are potentially caused by the agricultural wasted since banana trunk is an agricultural waste after the fruit is harvested at the same time, can be served as a sustainable sources for the nanocellulose extraction sources. This study also provide the optimum acid concentration used in the acid hydrolysis method in order to obtain the highest crystallinity, high aspect ratio with a stable colloidal suspension of cellulose nanocrystal (CNC) that can be used in many applications for example as the bio-based reinforcement material.

CHAPTER 2
LITERATURE REVIEW

2.0 Introduction

In this chapter, the extraction of raw material for nanocellulose from various types of plants were investigated and discussed in detail. The major focus was emphasized into the structure of banana fiber since banana trunk (pseudostem) is the main source of cellulose that will be utilized in this study. The preparation methods for nanocellulose extraction from the plants and their potential applications were also accentuates. Recent advances in the nanocellulose characterization and related works deals with it by using various tools and procedures were systematically reviewed.

2.1 Nanocellulose Fiber

Cellulose as depicted in Figure 2.1 is arranged orderly in micro-fibrils embedded with other two main components, lignin and hemicellulose responsible for mechanical properties of plant cell wall which is consist of D – anhydroglucopyranose units (AGU) and bonded linearly by β-1,4 glycosidic oxygen bridge bond by condensation (Morán *et al*., 2008). It is composed of β-1,4-linked glycopyranose units which is formed a high-molecular-weight linear homopolymer where the monomer unit is corkscrewed at 180º to its neighbours. This repeating unit of polymer is a dimer glucose which known as cellobiose (George and Sabapathi, 2015).

It is the most abundant polymer that could be found in nature that were restocked by the process photosynthesis by plants and become the one of the main constituent in natural fibers. Today, cellulose become the main ingredient in nanocellulose which is can be used for many kind of applications in various fields (Moran *et al*., 2008).

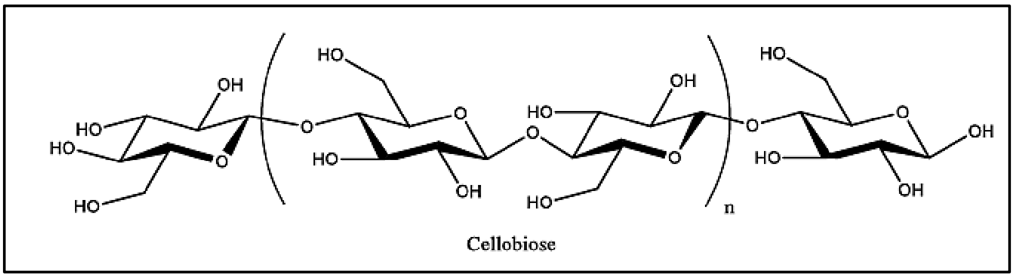

Figure 2.1 : Cellulose structure (George and Sabapathi, 2015).

2.1.1 Nanocellulose Fiber from Various Plant Resources

Moran *et al*., (2008) extracting the cellulose from sisal fiber to produce nanocellulose due to its high cellulose content of 50 – 74 % which is promising high yield of nanocellulose. According to Jonoobi *et al*.,(2015), the starting material of cellulose nanocrytsalline can be obtained from many types such as tunicate cellulose, bacterial cellulose, kraft pulp, microcrystalline cellulose (MCC), sugar beets, wood pulp, ramie, sisal, straw and cotton as the starting material. In addition, Beck-candanedo *et al*., (2005) had used the bleached softwood of black spruce, *Picea mariana* as the raw material in producing the suspension of cellulose nanocrystals.

Recently, Zain *et al*., (2014) had used Albedo, Pomelo (*Citrus grandis)* peel which is rich in cellulose as the material in producing nanocellulose. Other than that, Abraham *et al*., (2011) had successfully produced a stable colloid suspension of cellulose nanofibrils from three different types of natural fibers which are, banana trunk fiber, jute fiber and pineapple fiber leaf. In order to study the potential as the reinforcement fibers in biocomposite, cellulose nanofibers were extracted from durum wheat straw had been produced and characterized by (Leyva *et al*., 2011). Meanwhile, Deepa *et al.,* (2011) had successfully obtained the cellulose nanofibers by using banana fiber as the raw material.

Recently, Benini *et al*., (2016) had concluded that *Imperata Brasiliensis*, plant that originated from Brazil can be used as an alternative resource of nanocellulose since it has identical characteristic like other lignocellulosic fibers. While, Kallel *et al.,* (2016) were succeeded in obtaining the cellulose nanocrystals (CNCs) from the garlic straw. The