Faculty of Manufacturing Engineering

INVESTIGATION OF CELLULOSE FIBER FROM BANANA WASTE BY CHEMICAL TREATMENT

MOHAMAD HANIFAH BIN ABD GHANI

Master of Manufacturing Engineering (Manufacturing System Engineering)

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MOHAMAD HANIFAH BIN ABD GHANI

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Signature : …………………………………….

Supervisor Name : Dr. Syahriza bt. Ismail

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DEDICATION

To my beloved mother Latifah bt. Musa for her endlessness loves and support and to my late father Abd Ghani bin Hassan even could not see the completion of this thesis as well as my lovely wife Normala bt. Miskam.

ABSTRACT

Nowadays, cellulose knowledge from natural plant is one of the major contributions to the new and advance product development since it has no harm to the environment. Microcrystalline cellulose (MCC) possesses the merits of cellulose which has a lots of potential to be used in all manufacturing areas and various studies on the isolation of MCC from different sources using various procedures was held in order to define its characterization so that the potential of MCC as fiber phase in composite will be beneficial to user. In this work, fibres from banana pseudostem appear as an interesting source for cellulose fiber due to its readily available in Malaysia. The aim of this project was to evaluate the effects of chemical treatments over structural modifications on the fibre by hydrolysis analysis and mechanical treatment. MCC were determined through fiber chemical reaction which involves alkali treatment, bleaching and acid hydrolysis treatment. Scanning micron electroscope (SEM) and X-ray diffraction analysis was used in order to see the morphology before and after the treatment. The results showed the highest crystallinity index is banana pseudostem treated with 60% H2SO4 concentration with 90 minutes concentration at value 79.53%. The addition of sulfuric acid concentration has given effect through the SEM image which is clearly seen that the fibers have raptured. Measurement on SEM and PSA images proved that the reduction of raw fibers was from 280.20µm to 58.16µm and 171.13µm respectively. This research specifies chemical treatment (hydrolysis process) is the main factor of cellulose presence meanwhile the mechanical treatment (sonication process) will catalyst the presence in terms of diameter size and surface morphology.

ABSTRAK

Kini, pengetahuan selulosa dari tumbuhan semulajadi merupakan salah satu sumbangan besar kepada pembangunan produk baru dan maju kerana ia tidak menjejaskan alam sekitar. Selulosa mikrokristalin (MCC) mempunyai merit selulosa yang mempunyai banyak potensi untuk digunakan di semua bidang perkilangan dan pelbagai kajian mengenai pengasingan MCC dari pelbagai sumber menggunakan pelbagai prosedur untuk menentukan sifatnya supaya potensi MCC sebagai fasa serat dalam komposit akan memberi manfaat kepada pengguna. Dalam kajian ini, serat dari pisang pseudostem muncul sebagai sumber menarik untuk serat selulosa kerana ia mudah didapati di Malaysia. Tujuan projek ini adalah untuk menilai kesan-kesan rawatan kimia terhadap pengubahsuaian struktur pada serat oleh analisis hidrolisis dan rawatan mekanikal. MCC ditentukan melalui reaksi kimia serat yang melibatkan rawatan alkali, pelunturan dan rawatan hidrolisis asid. Pengesan mikroskopik elektron (SEM) dan sinar-X telah digunakan untuk melihat morfologi sebelum dan selepas rawatan. Hasil menunjukkan bahawa indeks crystallinity terbaik adalah 79.53% apabila fiber batang pisang dirawat dengan kepekatan 60% asid sulfuric, H2SO4 dengan masa sonikator 90 minit. Penambahan kepekatan asid sulfuric telah memberikan kesan melalui imej SEM yang dilihat dengan jelas menunjukkan serat telah pecah. Pengukuran terhadap imej SEM dan PSA membuktikan bahawa pengurangan serat mentah adalah masing-masing dari 280.20µm hingga 58.16µm dan 171.13µm. Kajian ini memperincikan rawatan kimia (proses hidrolisis) adalah faktor utama kepada kehadiran selulosa sementara rawatan mekanikal (proses sonikator) menjadi pemangkin dari segi saiz diameter dan morfologi permukaan.

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"Bismillahirrahmanirrahim"

"In the name of Allah, The Most Beneficent, The Most Merciful"

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CHAPTER 1

INTRODUCTION

1.0 Introduction

This chapter presents the introduction of the best advantages in cellulose presence towards the manufacturing knowledge. In part, the briefing of the background, problem statement, objective, scope of the project and significance of the study are discussed.

1.1 Background

Nowadays, the usage of cellulose characteristics is broadly implemented and contributed to the nanocellulose technology development of composite. Hence, various research efforts are being carried out in this cellulose field in line with the needs of people who want more sophisticated products that can simplify their daily lifestyle. In 1953, the Cellulose Research Institute which located in campus of the State University of New York was established by objectives is to provide fundamental knowledge about cellulose, which should lead to its increased utility.

Microcrystalline cellulose (MCC) possesses the merits of cellulose which has a lots of potential to be used in all manufacturing areas such as pharmaceutical, cosmetic, food and polymer composites industries. In the powder form, it is employed, for instance as a binder and filler in food, medical tablets, and particularly as reinforcement agent in the development of polymer composites. MCC is also one of the attractions in the field of manufacturing because of its green production due to increasing demand of alternatives to non-renewable and scarce fossil materials. D. Trache et al. (2016) state that MCC has a lots of advantages and widely used in various fields, such as food, pharmaceutical, medical, cosmetic and polymer composites industries.

Cellulose is potential to be studied because of its unique properties in physics, chemistry and biology. Lin & Dufresne (2014) stated that the stiffness and modulus of cellulose nanocrystals (CNC) with more crystalline regions are higher than those of cellulose nanofibrils and bacterial cellulose fibrils with both crystalline and amorphous structures. Dufresne (2013) already stated that the average value of CNC is around 130 GPa, that is, much higher than for cellulose microfibrils as expected. It was also stated that impressive mechanical properties make cellulose nanoparticles ideal candidates for the processing of reinforced polymer composites. Data from Chemical Economics Handbook (2016) state that China and Western Europe lead the global growth of cellulose opportunities. The largest consumption will grow at about 1.5% (Europe regions) and 4.5% (China) on an average annual basis. Meanwhile, North America and South America will grow faster than Europe, at a rate of 2.5% per year.

1.2 Problem Statement

Natural fiber reinforced composites is more preferable than synthetic fiber composites since it offers so many advantages either in term of cost or mechanical properties. They are two classes of fiber which are natural fibers and man-made fibers. Some examples of popular natural fibers include bamboo, coir, kenaf, wool, cotton, linen and silk are widely used in various applications. Natural fibers come out from nature source such as from animals and plant is more preferable compared with another one. Jauhari et al. (2015) stated that natural fibers are of interest for low-cost engineering applications and can compete with artificial glass fibers (E-glass fiber) when a high stiffness per unit weight is desirable. Besides, it is well known as environmental friendly so that make it is sustainable for the next generation. The popularity of natural fiber composite and the effort of Malaysia's research can contribute to the realization of our nation vision that wants to bring agriculture as business for the people.

Despite the natural fiber composite advantages, there are still challenges and disadvantages of it. Ramesh (2016) stated that plant fibers is exposed to biological decay and most of them darken and weaken with age and exposure to light. The composite consists of two phases which are fibers (discontinuous phase) used to carry the load meanwhile matrix (continuous phase) used to bind and transmit the load to the fibers. Therefore, these two phases has an important role in determining the micromechanical properties of the composite and gives opportunity to the researchers to development new fibre/matrix interface and good dispersion with increased mechanical properties (Pickering et al., 2016).

Furthermore, the cellulose which contains in the plant based fiber is actively studied by the other countries. Malaysia, especially for the plastics manufacturing industry should take this current opportunity in developing biodegradable products. Ramesh (2016) also stated that cellulose has been proven to be effective in reducing cost yet product quality composite product and the rising price petroleum based products. Eichhorn et al. (2010) stated that the principle reason to utilize cellulose nanofibers in composite materials is because one can potentially exploit the high stiffness of the cellulose crystal for reinforcement. Recently, advances production of fibers in nano size (1-100nm) have given another nanocomposite material for manufacturers (Souza et al., 2010).

In order to address new founding in the MCC potential as fiber phase, continuing studies need to be done. New material composites from cellulose fiber can be commercially produced rather than letting it harm and be an agricultural waste. A.B.M. Sharif Hossain (2014) stated that advancements in the use of waste materials could also significantly improve the economics of the biopolymer, biomaterial products, paper and pulp industries by leading to new sources of raw materials and other innovations. On the other hand, A. Rubio-Lopez et al. (2015) noted that a new class of monocomponent composites based on cellulosic materials, called all-cellulose composites (ACCs) have established which resulting a fully biodegradable material with perfect compatibility between matrix and reinforcement. A lot of studies have been made from different cellulosic sources and their use in high-performance applications. Moreover, M. Jonoobi et al. (2015) and D. Trache et al. (2016) state that the isolation different sources using various procedures, its characterization, and its application in bio-composites will be gained due to its their abundance, renewability, high strength and stiffness, eco-friendliness and low weight.

Therefore, the cellulose studies in natural fibers have become a popular trend in the recent years to study various since there are still have many opportunity to be develop. As reported by D. Trache et al. (2016) that ecofriendly bio-composites have the potential to be new products of the current century and partial remediation of several environmental problems for next generation. In Malaysia. current knowledge on the isolation of MCC from different sources using various procedures to define its characterization should be discuseed more. This project will investigate the characterization of cellulose from banana pseudostem by diameter measurement, X-Ray diffraction (XRD) and scanning electron microscopy (SEM) analysis.

1.3 Objective

The objectives of this study are:

i. To obtain the cellulose in banana pseudostem fibers by the acid hydrolisis process and sonication process.

ii. To characterize and analyze the cellulose properties before and after the treatment through size measurement, X-Ray diffraction (XRD), scanning electron microscopy (SEM) and particle size analyser (PSA).

1.4 Scope

Producing cellulose from fibers is normally by using the pretreatment, chemical and combination with mechanical methods. The pretreatment is important in order to isolate cellulose from lignin, hemicellulose and other impurities component. The most important method that will be focused is the extration of microcellulose using acid hydrolysis. Acid hydrolysis is the preferred method in order to remove amorphous region so that high crystallite particle or microcrystalline cellulose can be obtained. It involves high concentration of sulphuric acid as a medium.

The comparison of XRD pattern from the raw banana pseudostem fiber (before acid treatment) and MCC (after treatment) will determine the degree of crystallinity in cellulose. Besides, the scanning electron microscopy images of the raw fibers and microstructure obtained after acid treatment will explain the effectiveness of the downsizing the fiber from macro to the microrange.

1.5 Significant of study

This present study would be a contribution to the knowledge in relation to nanotechnology's achievement in Malaysia. Result obtained by this study could be highly significant and beneficial specifically to the following:

Students: The findings of this research such as literature review and basic knowledge will inspire and enlightened to this group. Surely more students will get interest and involve in exploring the cellulose technology since the chemistry and mechanical knowledge involvement during the extraction of natural fibers.

Researchers: Future researchers especially in Malaysia such as undergraduate students can take some useful information from this study in helping them to achieve their research targets in the field of cellulose. They also may modify the extraction procedure and increase the potential of banana pseudostem as ACCs. Increasing academic expertise in this field especially in Malaysia is beneficial so that Malaysia could be one of the sustainable product's manufacturers.

Industries: The potential of banana pseudostem in Malaysia can be a new potential contribution for new industries to come out with a new idea generation for a sustainable product. The industry can use the opportunities available from this study in reducing the cost of raw materials in producing their product by using cheap material which is from agriculture waste. At the same time, they can get better product in quality, good mechanical properties, recyclable and biodegradable.

1.6 Research Planning

Research activities of this study is outlined in a Gantt chart (refer Appendix A).

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

This chapter presents the review study related by other researchers. There are basically containing five main sections in this chapter, such as natural fiber composite, microcrystalline cellulose, cellulose extraction, characterizaton of banana pseudostem fibers and MCC.

2.1 Natural Fibre Composite

Since late 1980's, natural fiber have received more attention and various kind of research been conducted in order to gain its advantages as the reinforcement for polymeric matrix. Fiber-Reinforced polymeric (FRP) composites already known with its excellent mechanical properties such as high specific strength but it is lack in term of cost and biodegradability. These disadvantages of FRP can be replaced with natural fiber that offers much benefit although it also has some disadvantages as in Table 2.1. In spite of the disadvantages, N. Saba et al. (2015) also reported that it can be improved and overcome by hybridization with either natural or synthetic fibre.

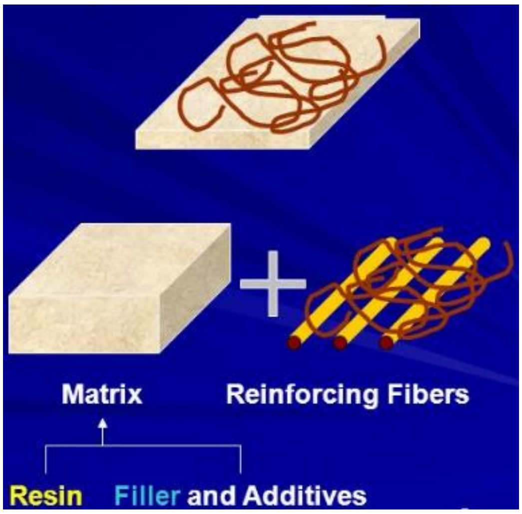

Figure 2.1: Analogy of Fiber-Reinforced Polymeric (FRP)

Table 2.1: Advantages and Disadvantages of Natural Fibers

Advantages	**Disadvantages**
Low specific weight	Lower strength
Renewable resources	Variable quality, influenced by weather
Friendly processing	Restricted maximum processing temperature
High electrical resistance	Lower durability
Biodegradable	Poor matrix adhesion

According to X. Li et al. (2007), natural fibres are composed of cellulose, hemicellulose, lignin, pectins, waxes and water soluble substances and the chemical composition can be seen in Table 2.2. Also, the absence of lignin, hemicellulose and waxes on the fibre surface are to enhance the compatibility between fiber and polymer matrix. Similar with J. Ch. Cintil et al. (2014) study which state that natural fiber basically constituted of cellulose, lignin and hemicellulose. Meanwhile pectin, pigments and extractives can be found in lower quantity.

Table 2.2: Chemical Composition in Common Natural Fibers

Type of fiber	Cellulose *(%)*	Lignin *(%)*	Hemicellulose (or Pentosan) *(%)*	Pectin *(%)*	Ash *(%)*
A) Bast fiber					
▪ Fiber flax	71	2.2	18.6 - 20.6	2.3	-
▪ Seed flax	43 - 47	21 - 23	24 - 26	-	5
▪ Kenaf	31 - 57	15 - 19	21.5 - 23	-	2-5
▪ Jute	45 - 71.5	12 - 26	13.6 - 21	0.2	0.5 - 2
▪ Hemp	57 - 77	3.7 - 13	14 - 22.4	0.9	0.8
▪ Ramie	68.8 - 91	0.6 - 0.7	5 - 16.7	1.9	-
B) Core fiber					
▪ Kenaf	37 - 49	15 - 21	18 - 24	-	2 - 4
▪ Jute	41 - 48	21 - 24	18 - 22	-	0.8
C) Leaf fiber					
▪ Abaca	56 - 63	7 - 9	15 - 17	-	3
▪ Sisal	47 - 78	7 - 11	10 - 24	10	0.6 - 1
▪ Henequen	77.6	13.1	4-8	--	-

Natural fibre composite materials are also commonly known as bio-composites and its major goal is to reduce the manufacturing cost by replacing the usage of expensive synthetic fiber composites industries. Automotive industry also very committed since early 2000 in developing these composites by seeking-out for materials that can be applied in the future. S.A Ariadurai (2012) stated one of the benefits of using natural fiber is the reduction in cost around 25-50% cheaper than using the glass fiber composites. Besides that, natural fiber also very useful since it is biodegradable or compostable materials in the packaging sector. Table 2.3 shows the various kinds of bio-plastics that are originated from the combination of polymer and natural fiber phases.

Although natural fiber are very useful in producing bio-plastics, but it cannot intimidate the packaging sector in the current domain due to the not-so superior properties of bio-plastics compared to synthetics. Natural fibers are fortunate due to its environmental impact and the advantages. M.F.M. Alkbir et al. (2016) justified that natural fibres such as hemp, kenaf, jute, sisal and bamboo have been studied due to their mechanical properties and their potential use in composite materials. Nowadays, the challenges, expectations and