Faculty of Resource Science and Technology

ISOLATION OF CELLULOSE NANOFIBERS FROM AGRICULTURAL WASTES (SAGO PITH WASTE)

Then Yuan Zu (58154)

Bachelor of Science with Honours
(WS48 Resource Chemistry)
2019

Grade: A

Please tick (√)
Final Year Project Report ☑
Masters ☐
PhD ☐

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ISOLATION OF CELLULOSE NANOFIBERS FROM AGRICULTURAL WASTES (SAGO PITH WASTE)

Then Yuan Zu
(58154)

An undergraduate thesis dissertation is submitted in partial fulfilment of the requirements for the degree of Bachelor of Science with Honours (Resource Chemistry)

Supervisor: Associate Professor Dr. Chin Suk Fun

Resource Chemistry
Department of Chemistry
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2019

Grade: _______________

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Final Year Project Report ☑
Masters ☐
PhD ☐

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Isolation of Cellulose Nanofibers from Agricultural Wastes (Sago Pith Waste)

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ABSTRACT

The isolation of cellulose nanofibers from sago pith waste was performed by using combination of chemical treatment and homogenization. The purpose of this study is to isolate the cellulose nanofibers which are useful in various fields from the by-products of sago starch extracting process which are known might create environmental pollution. Besides, this research studies the surface morphology and diameter of the cellulose fibers and nanofibers at different stages of the chemical treatments and also after the homogenization process. Moreover, the changes in the functional groups of the cellulose fibers before and after each chemical treatment were studied. Furthermore, the percentage yield of the cellulose nanofibers was also determined by applying the proposed formula. The chemical treatments processes involve alkaline treatment, bleaching and acid hydrolysis whereas the mechanical treatment involves homogenization. The surface morphology and diameters of the cellulose fibers and nanofibers was determined by using Scanning Electron Microscope (SEM). Fourier Transform Infrared (FTIR) was used to study the changes in the functional groups of the raw fibers and the chemically-treated fibers. The combination of chemical treatment and homogenization successfully isolate the cellulose microfibers with diameter ranged from 16μm to 1.3μm. Moreover, the surface morphology, diameter and changes in the functional group of the cellulose nanofibers were successfully determine by SEM and FTIR respectively.

Keywords: cellulose nanofibers, sago pith waste, chemical treatments, homogenization

ABSTRAK

Pengasingan gentian selulosa bersaiz nanometer daripada sago hampas telah dilakukan dengan menggunakan cara gabungan antara rawatan kimia dengan homogenisasi. Tujuan kajian ini adalah untuk mengasingkan gentian selulosa bersaiz nanometer yang mempunyai banyak kegunaan dalam pelbagai bidang daripada produk sampingan dari proses pengekstrak kanji sagu yang akan menyebabkan pencemaran alam sekitar. Selain itu, kajian ini juga mengkaji permukaan morfologi dan diameter gentian selulosa dan gentian selulosa bersaiz nanometer selepas setiap rawatan kimia dan juga selepas proses homogenisasi. Tambahan pula, perubahan dalam kumpulan fungsional gentian selulosa sebelum dan selepas setiap rawatan kimia telah dikaji. Di samping itu, peratusan hasilan gentian selulosa bersaiz nanometer juga telah dikira dengan menggunakan formula yang dicadangkan. Proses rawatan kimia melibatkan rawatan alkali, pelunturan dan hidrolisis asid manakala rawatan mekanikal melibatkan homogenisasi. Morfologi permukaan dan diameter gentian selulosa dan gentian selulosa bersaiz nanometer ditentukan dengan menggunakan Mikroskop Pengimbasan Elektron (SEM). Fourier Transform Infrared (FTIR) digunakan untuk mengkaji perubahan dalam kumpulan fungsional gentian mentah sago hampas dan gentian sago hampas yang telah melalui rawatan kimia. Kombinasi rawatan kimia dan homogenisasi berjaya mengasingkan gentian selulosa bersaiz micrometer yang berdiameter dari 16μm hingga 1.3μm. Selain itu, permukaan morfologi, diameter dan perubahan dalam kumpulan fungsional gentian selulosa telah berjaya ditentukan oleh SEM dan FTIR masing-masing.

***Kata kunci**: gentian selulosa bersaiz nanometer, sago hampas, rawatan kimia, homogenisasi*

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LIST OF ABBREVIATIONS

α	Alpha
BC	Bacterial nanocellulose
β	Beta
BOD	Biochemical Oxygen Demand
CNC	Cellulose nanocrystals
CNF	Cellulose nanofibers
CNW	Cellulose nanowhiskers
°C	Degree Celsius
D	Dextrose
DO	Dissolved Oxygen
FTIR	Fourier Transform Infrared
g	gram
kHz	kilohertz
mL	milliliter
SPW	Sago pith waste
SEM	Scanning Electron Microscope

CHAPTER 1: INTRODUCTION

Malaysia is a tropical country which has lavish natural resources (Yacob *et al.,* 2018). Aside from being known as the world main supplier of palm oil and rubber, Malaysia is also producing sago starch (Yacob *et al.*, 2018). It was reported that the state of Sarawak in Malaysia is one of the world's biggest exporter of sago starch (Awang- Adeni *et al.*, 2010). Besides, sago palm (*Metroxylon sagu*) was also exploited as one of the staple food source for Sarawak coastal Melanau communities (Hafizan *et al*., 2016).

Sago starch was derived from the sago pith which lined beneath the sago bark. The bagasse produced in the sago starch extraction process is known as the sago pith waste or sago '*hampas*' by the locals (Zainab *et al.,* 2014). According to Awang- Adeni *et al.* (2010), the sago pith waste (SPW) is often used as a feedstuff for swine in the farms nearby the sago processing compounds aside from being used as the compost for the cultivation of mushrooms and as a raw material for particleboard manufacture (Lai *et al.,* 2013).

The generation of the sago wastes increases as the production of sago starch increases. The residues which are not needed will be simply washed off into the rivers nearby together with the sago processing wastewater. Mohd *et al.* (2001) revealed a shocking data that the amount of starch disposed as sago pith waste in Sarawak alone accounted for nearly half of the annual imports of starch in Malaysia. As reported by Subair *et al.* (2017), the presence of lignocellulosic fibrous materials and starch residue in the SPW is a strong pollutant as it is insoluble in water as well as the starch remained within the sago pith waste. Some of the bacteria in the river is capable of decomposing the SPW but a long period of time will be needed to decompose the massive amount of SPW produced and its decomposition by the bacteria will also lead to the increase in the Biochemical Oxygen Demand (BOD) level of the river water. As a consequence, the Dissolved Oxygen (DO) level in the river water will decrease and causes the aquatic animals to receive insufficient oxygen and eventually die. Yacob *et al.* (2018) reported that an estimation of 7 tons of sago hampas are produced from a single sago starch processing mill daily. The massive generation of the sago waste is worrying and alternative ways to utilize or modify these wastes into the materials which are environmentally and economically valuable can help in solving the disposal problems (Zainab *et al.*, 2014).

Nanotechnology is a scientific technology which is conducted at nanoscale. As stated by Khan *et al.* (2017), nanofibers are fibers with diameter less than 100 nm. Unique chemical and physical properties can be found on nanofibers due to their high surface area and nanoscale size (Khan *et al.*, 2017). Abdul *et al.* (2012) state that nano-sized cellulose can be classified into 3 types including cellulose nanocrystal (CNC), cellulose nanofibers (CNF) and bacterial nanocellulose (BC). Based on the study of Subair *et al.* (2017), the formation of cellulose nanofibers is resulted from the application of high shearing forces of disintegration leading to a high degree of fibrillation, which yields highly interconnected fibrils. Tonoli *et al.* (2012) reported that the interest in cellulosic nanofibers isolated from renewable sources for several industrial applications had grown drastically due to their promising properties of biodegradability, nontoxicity and biocompatibility. Besides, the growing energy demands and environmental awareness encourage the researchers to explore and produce environmental friendly and low cost materials from the renewable agricultural wastes to provide an economic solution for the waste management system at the industrial areas in addition to reduce the polluting effects caused by the waste (Li *et al.*, 2014). Cellulose nanofibers can be isolated from various natural plant fibers such as wood and cotton. However, wood is widely used for the purpose of construction and the demand of wood for constructions and cotton in textile industries had increased in the past decade so this has merged the interest of many researchers towards other agricultural crops (He *et al.,* 2013) and their by- products such as sugar palm fibers (Ilyas *et al.,* 2017).

Cellulose is a polysaccharides and can be found abundantly in nature especially in the plant cells. Cellulose can be isolated from plants and it is capable of replacing the non-biodegradable petroleum- based materials to reduce negative environmental impacts (Anna *et al.,* 2016). As a readily available natural polymer, cellulose can be used to synthesize various potential reinforcing biomaterials.

Isolation of cellulose fibers from agricultural wastes requires the removal of non-cellulosic components before the fibers can be further refined to a smaller size. A pretreatment step is necessary to separate the cellulose fibers from the tightly bonded polymeric constituents such as hemicellulose and lignin. The treatments separate the cellulose fibers from the non-cellulosic components by solubilizing the non-cellulosic parts and to be removed during the reaction with the chemicals. This also helps in defibrillation of the cellulose fibers as the binding materials in the plant cells such as hemicellulose and lignin are removed. As reported by Lee *et al.* (2014), chemical treatments are the most popular pretreatment employed in the isolation of cellulose fibers to nanofibers from lignocellulosic biomass. On the other hand, physical treatment is also needed to provide high shear force to disintegrate the cellulose nanofibers from the cellulose fiber bundles. According to Nechyporchuk *et al.* (2016), refining, homogenization and grinding are the most common physical treatments applied to the cellulose fibers isolated to obtained nano-sized cellulose fibers.

For the problem statement of this study, the conservation and preservation of environment have become a rising issue due to the increase in environmental problems and depletion in natural resources recently. The increase in the generation of sago pith waste results in more environmental pollution as most of the SPW was disposed into the environment without any treatment. The significant amount of organic substances left within the sago pith waste encourages the proliferation of bacteria in the rivers. This results in the depletion of the quality of the river water. Besides, some of the sago pith waste was used as the fodder for livestock and as the medium for the cultivation of mushroom but large amount of it was not utilized to the maximum. This leads to the wastage in nature resources.

On the other hand, the production of cellulose nanofibers using only mechanical disintegration requires high input of energy to generate sufficient shear forces to make the cellulose nanofibers to detach from the cellulose fiber bundles. The introduction of chemical pretreatments prior to mechanical disintegration helps to solve this problem by enhancing fibrillation and reducing the energy demand. Besides, most of the current researches utilizes concentrated acid in the acid hydrolysis step to remove the residual hemicellulose. The concentrated acid possesses toxic, hazardous and corrosive properties and required extreme care in handling it. In addition, the cost for concentrated acid is also high and hence the concentrated acid needs to be recovered after the pretreatment to make the acid hydrolysis process economically and environmentally feasible. The choice of utilizing dilute acid in acid hydrolysis can minimize these problems by properly investigating the optimum conditions for maximum removal of non-cellulosic materials in the sago pith waste.

In conclusion, a study on the isolation of cellulose nanofibers from sago pith waste for its maximum utilization is required in order to prevent wastage and also the pollution it caused to the environment.

The objectives of this study are to isolate cellulose nanofibers from sago pith waste by using chemical pretreatment and homogenization method, to characterize the cellulose fibers and nanofibers isolated by using Fourier Transform Infrared Radiation (FTIR) and Scanning Electron Microscope (SEM) and finally to determine the yield of cellulose nanofibers isolated from the sago pith waste.

CHAPTER 2: LITERATURE REVIEW

2.1 Sago Palm

Figure 1: *Metroxylon sagu sp*. (Diana, 2018)

Sago starch was extracted from sago palm. The word 'sago' originates from Javanese language which defines palm pith that contains starch. The accumulation of sago starch occurs in the pith core of the sago palm stem. The most common species of sago palm involved in the sago extraction process is the *Metroxylon sagu sp*. *Metroxylon sagu* is categorized in the genus of *Metroxylon* and the order of *Arecaceae*. In adidition, it belongs to the *Palmae* family which has about 181 genera with around 2600 known species (Ishak *et al*., 2013). According to Flach (1997), the scientific name *Metroxylon sagu* is derived from the phrase *metra*, meaning pith or parenchyma, and *xylon*, meaning xylem. *Metroxylon sagu* has a stem where the starchy pith is located while *cycas revoluta* is often cultivated only as an ornament plant. Sago palm can grow quickly and thus it is dominant in the tropical peat swamp rainforests, where other crop plants do not thrive (Ishak *et al.*, 2013). *Metroxylon sagu* can tolerate with low pH or highly acidic condition which coupled with concentrated metals in the soil that inhibit the growth of other plants species.

Sago palm is highly productive as one sago palm can yield around 150 kg sago starch (Kumoro *et al.*, 2008). Besides, as reported by Santoso (2018), *Metroxylon sagu* gives higher yield of starch as compared to other crops such as 3 to 4 times higher yield than rice and wheat or 17 times higher than the yield of cassava roots. From here, it can be concluded that sago palm is a potential plant in the starch extracting industry. Since sago contains high concentration of starch which acts as an energy source, this makes sago one of the important source of food in some areas in Southeast Asia. Aside from being as a food source, the importance of *Metroxylon sagu* as a socioeconomic crop in Southeast Asia increases from time to time. Sago starch can also be used to manufacture biodegradable plastics. However, studies on the sago palm and the uses of the other parts of the plant as well as the sago residues are relatively lesser as compared to others staple food crops like paddy and tapioca.

As reported by Karim *et al*. (2008), New Guinea is the center of diversity of *Metroxylon sagu* and it also occurs naturally from the South Pacific islands, extending westward through Melanesia into Indonesia, Malaysia, and Thailand. In Malaysia, about 25000 to 30000 ton of sago flour was exported annually and hence sago is proven to be economically important to people who live near the swampy areas which the growth of *Metroxylon sagu* are favoured. In Sarawak, *Metroxylon sagu* is the only crop which can tolerate harsh swampy peat condition and thus, it is reported that the area coverage of *Metroxylon sagu* reaches 12% which is equals to 1.5 million hectares in the year 2007 (Karim *et al*., 2008).

2.2 Sago Pith Waste (Sago *Hampas*)

As reported by Singhal *et al*. (2008), sago pith waste is a fibrous residue in which most of the starch have been extracted from the rasped sago pith which is formed from the sago starch extraction process. Linggang *et al.* (2012) stated that sago pith waste contains high amount (58%) of starch, 23% cellulose, 9.2% hemicellulose and 3.9% lignin on a dry weigh basis. It is necessary to remove the non-cellulosic materials including starch from the sago *hampas* in order to isolate the cellulose fibers. The three major components of a lignocellulosic biomass are cellulose, hemicellulose and lignin (Harmsen *et al.*, 2010). Awang- Adeni *et al.* (2010) claimed that there is a strong association between the lignin and the hemicellulose in the cell walls of sago palm. There are some researches had been carried out to isolate and characterize nanoparticles from the sugar palm fibers (Ilyas *et al.*, 2017) and sago seed shell (Subair *et al.*, 2017). However, no studies on the isolation of cellulose nanofibers from *Metroxylon sagu* pith waste has been found in the literature.

2.3 Cellulose, Hemicellulose and Lignin

Cellulose is a homopolysaccharide with semi-crystalline structure that consists of many β-D-glucopyranose as its monomers. Each monomer of cellulose contains 3 hydroxyl (-OH) functional groups in which one is the primary hydroxyl group while the other two are secondary hydroxyl group (Kargarzadeh *et al.*, 2017) distributed evenly on both sides of the monomers and this allows the formation of hydrogen bonds between the hydroxyl group at the first carbon of one glucose molecule with the hydroxyl group at the fourth carbon of another glucose molecule. According to Nechyporchuk *et al.* (2016), two glucose molecules link together will form anhydroglucose units while two anhydroglucose units compose anhydrocellobiose. The chemical linkage between the anhydroglucose units is named as β-1, 4-glycosidic bond (Julie *et al.,* 2016). Besides, the anhydroglucose units are capable of forming internal hydrogen bonds between the C3 hydroxyl (-OH) group of one unit and the oxygen atom in the ring of the adjacent units (Kargarzadeh *et al.*, 2017). This makes the free rotation of the glucopyranosic rings around the glycosidic bonds being hindered and leads to the increase in the stiffness of the cellulose chains (Dufresne, 2012). Moreover, the poly(1, 4-β-D-glucopyranose) chains can form inter-chain hydrogen bonding with the neighboring chains and this contributes to the crystalline structure of cellulose (Hayati *et al.*, 2017). Besides, cellulose is less branching as compared to starch which is made up of α-D-glucopyranose monomers that do not allow the polymer chain to pack linearly and hence starch have a more amorphous structure. By having a strong system of intra- and intermolecular hydrogen bonds with all the hydroxyl groups in the chains, the resulting crystalline regions in cellulose is insoluble in water and some chemical reagents (Dufresne, 2012) such as diluted acid solution at low temperature. However, it becomes soluble at very high temperature as the heat energy provided is sufficient to break the hydrogen bonds that hold the crystalline structure of the cellulose (Harmsen *et al*., 2010). The amorphous regions of cellulose chains consists of weak hydrogen bonds and thus contributes to better hydrophilicity and accessibility of the cellulose (Dufresne, 2012). The inter- and intramolecular hydrogen bonds allow the cellulose molecules to be packed side by side in planar sheet and bundled into microfibrils (Lee *et al.,* 2014).

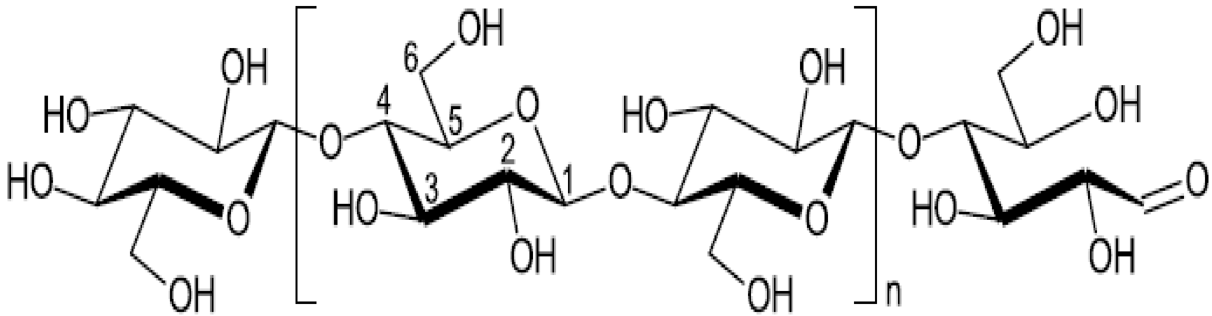

Figure 2: The structure of single cellulose molecule in chair conformation (Nechyporchuk *et al*., 2010)

As stated by Hayati *et al.* (2017), hemicellulose is made up of heterogeneous polysaccharide polymer that primarily comprised of xylan and intermixed with components such as glucose, xylose, arabinose, galactose, mannose, glucuronic acids and galacturonic acids. Due to the highly branched chains, hemicellulose has an amorphous structure. Besides, the presence of acetyl groups between the polymer chains also contributes to its amorphous property. Hemicellulose is easier to be hydrolysed by chemicals such as dilute acids and alkali under mild conditions as compared to cellulose which is relatively more recalcitrant (Lee *et al*., 2014). Hemicellulose is also insoluble in water at low temperature but the addition of alkali highly enhances its solubility in water (Harmsen *et al*., 2010). As reported by Lee *et al.* (2014), hemicellulose is one of the compounds that obstruct the accessibility of cellulose and a research suggested that the digestibility of cellulose can be extensively increased by the removal of a minimum of 50% of hemicellulose. Since hemicellulose is highly thermochemically sensitive, its degradation occurs easily to form unwanted coproducts such as furfurals and hydroxymethyl furfurals (Lee *et al*., 2014).