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Effect of Wet Spinning Parameters on Bamboo Cellulose Nanofiber Filament Preparation

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Abstract

Cellulose nanofibers (CNF) are materials synthesized from wood-based fibers having excellent mechanical properties due to their high crystallinity. In this research, the cellulose nanofibers were synthesized from bamboo fibers, as the abundant natural fibers available worldwide. The cellulose nanofibers had been synthesized using chemical processes of bleaching with acetic acid and sodium chloride, followed by alkaline treatment with sodium hydroxide, and acid hydrolysis with sulfuric acid combined with mechanical process in ultrasonic bath. Isolation of nanocellulose from bamboo scrap raw material was confirmed by different analysis methods. The morphology of CNF was characterized by using a scanning electron microscope (SEM). Fourier transform infrared spectrometer (FT-IR) and X-Ray Diffractometer (XRD) examined chemical structure and identified the crystallinity of nanocellulose materials. The monofilament of cellulose nanofibers was prepared by using a wet spinning process. The effects of coagulating solvent, water, methanol, acetone, and calcium chloride ($CaCl_2$) on monofilament formation were studied. Morphology study of CNF monofilament was carried out using a digital camera to observe the spinnability of the monofilament and the relationship with wet spinning process conditions. The thermal properties of the nanocellulose spun in methanol and acetone as coagulation solvent were analyzed by differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA). Also, the measuring of degradation temperature of nanocellulose spun compared with the nanocellulose extracted.

Keywords: Cellulose nanofibers, Bamboo fibers, Wet spinning, Monofilament

1. Introduction

Recently, materials that are friendly to the environment and biodegradable have become more useful and more acceptable in worldwide application (Qiu et al., 2021). It is noteworthy to mention microplastic accumulation problems would affect different living organisms specially human and aquatic animals (Svensson, Ferreira, Hakkarainen, Adolfsson, & Zamani, 2021). The microplastic problems arise from plastic petroleum waste which have difficulty in decomposing and degrade for turning to nature (Chaichi, Hashemi, Badii, & Mohammadi, 2017; Nazrin et al., 2020). The effects reinforce the research and development of a new material, a composite material based on natural resources or agricultural wastes which can replace the plastic industry (Liminana, Garcia-Sanoguera, Quiles-Carrillo, Balart, & Montanes, 2018; Wu, Misra, & Mohanty, 2020). The general structure of plant fibers consists of cellulose, hemicellulose, lignin and other substances. The cellulose is the main constituent which is an organic substance that gives strength for the cell wall. The natural fiber is used in many applications, for example in the textile industry, in the pharmaceutics and in food packaging (Rostamitabar et al., 2021; Vanitha & Kavitha, 2021). Bamboo plant is popular and cultivated in many countries in Asia. The bamboo can grow rapidly and acclimate to several weather conditions. The bamboo has a great surface area and a high cross-linking property (Qian, Zhang, Yao, & Sheng, 2018). In addition, bamboo has a good property of high productivity and biomass density. The bamboo also shows anti-bacterial and UV protective

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properties (Singla et al., 2017). Due to their ability, bamboo is applied in many industries including papermaking, furniture, construction materials, textile, musical instruments, etc. (Abdul Khalil et al., 2012; Silva, Menis-Henrique, Felisberto, Goldbeck, & Clerici, 2020). In addition, the bamboo is a popular plant that has a great deal for usage in the polymer composite sector (Abdul Khalil et al., 2012). Synthesis of cellulose from plant cell wall or agricultural residues can be prepared in different solutions such as mechanical, biological, chemical or combination techniques. The mixed chemical and mechanical are commonly used with application different treatment techniques. The treatment is necessary to remove non-cellulosic material (Joy et al., 2016). Many studies have been done to separate cellulose filaments such as, Homogenization, grinding, micro-fluidization, oxidation and acid hydrolysis. The cellulose fiber can be spun in suitable condition (Kafy et al., 2017). The usage of cellulose nanofibril and cellulose nanocrystals are more applicable in medical sectors (Benini, Voorwald, Cioffi, Rezende, & Arantes, 2018; Gupta, Revagade, Anjum, Atthoff, & Hilborn, 2006) and also in polymer nanocomposite (Qian et al., 2018). There are various types of polymers fiber forming process such as dry spinning, wet spinning and melt spinning (Gupta et al., 2006). In the wet spinning process, the polymer is injected in the coagulation bath which help solidification the spun fiber solution or gel to the spun fiber filament. This (The wet spinning) technique is suitable to produce small quantity samples for medical devices (Lundahl, Klar, Wang, Ago, & Rojas, 2017). In this research, the preparation of nanocellulose fiber from the bamboo using chemical and mechanical process was studied. The bamboo was treated with bleaching and alkaline treatments follow by acid hydrolysis process with ultrasonic to obtain nanocellulose. The chemical properties, physical properties, and morphology of prepared nanocellulose from bamboo were characterized by FTIR, XRD and SEM, respectively. The mono-nanocellulose filaments were prepared from extracted nanocellulose and formed by wet spinning process. We studied the effect of coagulants including methanol, water, calcium chloride and acetone. The mono-nanocellulose filaments were analyzed the spinnability and their thermal properties.

2. Materials and Methods

2.1 Materials

Bamboo fibers used for preparation of nanocellulose were received from Kanchanaburi province, Thailand. The fibers were sieving to size of 20-40 mesh. Acetic acid was purchased from VWR Chemical company. Sodium chloride, sodium chlorite and sodium hydroxide were purchased from Ajak Finechem. Sulfuric acid, acetone and methanol were obtained from RCI Lab Scan Limited.

2.2 Preparation of nanocellulose

The bamboo was first bleached with 10 ml acetic acid and 60 g of $NaClO_2$ at 75°C for 1 h. This step was repeated for three times until the color of bamboo became white or very light yellow. In the alkaline treatment process, the bleached bamboo was treated in 5 M NaOH solution for 24 h (change the NaOH solution for each of 4 h) at room temperature. The residual was filtered and rinsed several times until its pH became neutral. The acid hydrolysis process, 50 wt% H_2SO_4 was added in a sample that was subjected to an ultrasonic generator at 50°C for disintegration of nanocellulose fiber (Kwak, Lee, Lee, & Jin, 2018). After 1 h, the reaction was interrupted by adding 250 ml of cold deionized (DI) water. Finally, the residue was centrifuged and dialyzed with water until neutral pH was reached. The preparation steps of nanocellulose from raw material bamboo was presented in Figure 1.

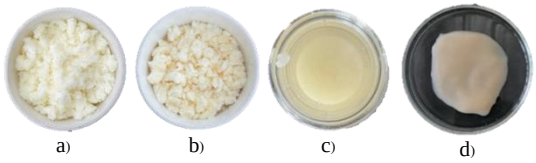

Figure 1. Products from nanocellulose extraction a) bleaching process b) alkaline treatment process c) acid hydrolysis process and d) nanocellulose.

2.3 Wet-spinning process of the bamboo nanocellulose

The nanocellulose suspensions were injected into the different coagulation baths (methanol, water, calcium chloride and acetone). The nanocellulose extracted previously was added in a 10 ml syringe, 14.5 mm of inner diameter equipped with 18G needle (1.2 mm of diameter). The syringe

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was installed with a single syringe pump, NE-1000 model that was parameter volumetric flow at 3 ml/min. The flow rate value assumes a minimum continuous flow of the nanocellulose suspension. The different coagulation baths were filled in a glass plate with a 25 ml solution. The spun fiber was left in the solution for a while before collecting. Figure 2 shows the wet spinning process.

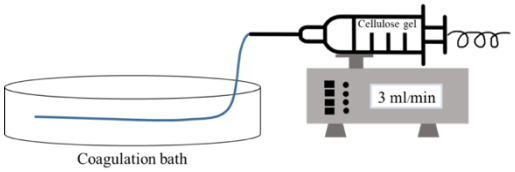

Figure 2. Wet spinning process.

2.4 Characterization

2.4.1 Chemical property of nanocellulose

The FTIR spectra of original bamboo, bleached bamboo, alkalied bamboo and nanocellulose were recorded using a Thermo scientific model Nicolet 6700 FTIR spectrometer with wavelength of 4,000-500 cm^{-1} at 1 cm^{-1} resolution and a frequency of 32 scans for each sample.

2.4.2 Crystallinity of nanocellulose

The crystallinity of the nanocellulose was characterized using Panalytical X'pert PRO, PW 3040/60 X-Ray diffractometer operated at Cu Kα radiation (λ= 1.54 ˚A), 30 kV and 20 mA, analyzed at 2θ from 5-80˚.

2.4.3 Morphology of nanocellulose

The morphological structure of original bamboo, bleached bamboo, alkalied bamboo and nanocellulose were investigated using a JSM 5410-LV Scanning Electron Microscope (JEOL, Japan). Samples were placed on carbon tapes and then coated with platinum (Pt) using a sputtering coater. The analysis of fiber surface characterized on vacuum system, 15 kV power at 100X and 500X magnification.

2.4.4 Thermal properties

The Thermal properties were analyzed with differential scanning calorimeter (DSC), Mettler Toledo model DSC 3 plus star system and thermogravimetric analysis (TGA), Mettler Toledo model TGA/DSC 3 plus. The thermal analysis was carried out by using STARe software. The sample tested their thermal properties with 20 ml/min nitrogen flow and heat rate of 10°C/min. The range of temperature study was between 25°C to 500°C which covers the cellulose decomposition temperature range.

2.4.5 Spinnability of fiber in coagulation bath

The spun nanocellulose monofilament in different coagulation baths was recorded with a digital camera OLYMPUS OM-D E-M10 Mark II which was used to observe and describe the spinnability of cellulose fiber.

3. Results and Discussions

3.1 Chemical structure analysis

Modification of chemical structure in different process treatments appears in Figure 3 which represents the relationship between wavelength and functional groups. The range of 2800-3500 cm^{-1} and 500-1800 cm^{-1} were two important absorbance peaks. Peaks at 3340 cm^{-1} represented a stretching vibration hydroxy group (O–H). The hydroxy group is generally found in cellulose, hemi-cellulose and lignin (Kwak et al., 2018) that can be observed in all samples. Also, the absorbance peak at 2900 cm^{-1} refers to -CH_2 that presents in the bamboo raw material and the treated bamboo (Xie et al., 2016). The absorbance peak at 1650-1700 cm^{-1} and at 1200 cm^{-1} are corresponded to carbonyl ring observed in hemi-cellulose and methoxyl groups (Theivasanthi, Anne Christma, Toyin, Gopinath, & Ravichandran, 2018), respectively. In cellulosic samples, curve after acid hydrolysis process, we could observe absence of peak 1600 cm^{-1} and reduction of peak 1230 cm^{-1} that represented chemical bond of lignin and lower content of hemi-cellulose after alkali treatment and acid hydrolysis. The peak at 1700 cm^{-1} became more obvious after bleaching and alkali treatments. It was observed obviously in the acid hydrolysis step which refers to the elimination of contaminants and lignin and partial hemi-cellulose.

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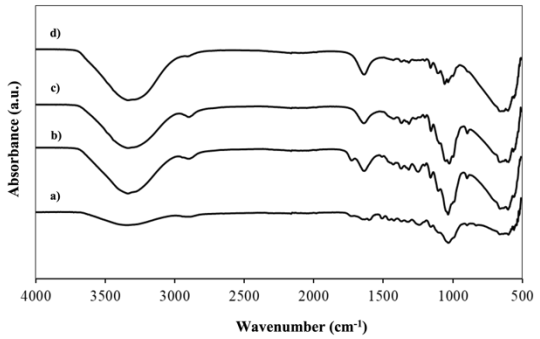

Figure 3. FT-IR results of original bamboo a), bleached fiber b), alkali-treated fiber c), and acid-hydrolyzed fiber d).

Table 1. The relationship between wavenumber and chemical function group.

Wavenumber (cm^{-1})	Function group	Reference
1200-1230	Methoxyl	Theivasanthi et al., 2018; Xie et al., 2016
1600	Methyl	Jiang et al., 2020
1650-1700	Carbonyl ring	Theivasanthi et al., 2018
2900	Methylene	Theivasanthi et al., 2018
3340	Hydroxy group	Kwak et al., 2018; Xie et al., 2016

3.2 Crystallinity

X-ray diffractogram of residue for each process was shown in Fig. 4. All diffractograms contained two 2 intense peaks, first at 16° and second at 22°. These peaks presented significance of cellulose crystalline structure. The peak at 2θ = 16° corresponded to crystallographic planes that is characteristic of crystallographic semi-crystalline materials such as lignocellulosic (Xie et al., 2016). The 2θ = 22° peak verified characteristic of crystalline cellulose. Increasing peaks in cellulose samples present dissolution and removal of non-cellulosic materials such as lignin and extractives from the amorphous regions. The crystallinity of cellulose increases with the next steps of the treatment process (Singh, Gaikwad, Park, & Lee, 2017). From the diffractogram, the peak of the acid hydrolysis process could present more evidence due to the reduction of the amorphous part of hemi-cellulose and lignin.

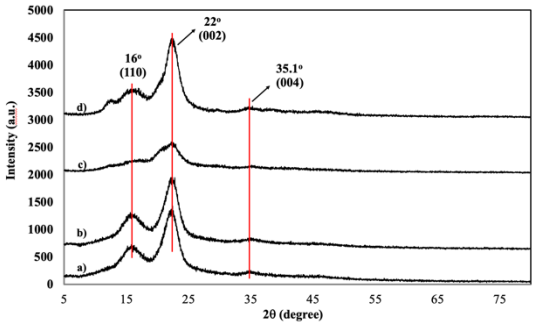

Figure 4. XRD results of original bamboo a), bleached fiber b), alkali-treated fiber c), and acid-hydrolyzed fiber d).

Table 2. The result from XRD data.

Peak position (degree)	Characteristic	Reference
16 (110)	semi-crystalline	Gong, Li, Xu, Xiang, & Mo, 2017; Theivasanthi et al., 2018; Xie et al., 2016
22 (002)	crystalline	

3.3 Morphology of nanocellulose

The scanning electron microscopy (SEM) in Fig.6 shows the morphology of different steps to isolate nanocellulose fiber from raw bamboo fiber. Fig.5 a), the original bamboo had a large size and rough surface. After the bleaching step, the impurities such as wax were eliminated. The surface area became more clear and smooth (Xie et al., 2016). Sizing of bleached fiber was not observed. The morphology of treated fibers with alkali solution 24 h shows in Figure 5 c), presented considerably smaller and longer fiber. The hemi-cellulose and lignin were removed from cellulose fibril. The morphology of residue from the acid hydrolysis process shows in Figure 5 d) with magnitude 500X suspension of nanocellulose diluted in water (Figure 5 d). Chemical and mechanical under ultrasonic technique induced the elimination of non-cellulosic compounds from original bamboo. The strong acid hydrolysis was used to fibrillate the micro cellulose sizing to nanocellulose (Kwak et al., 2018).

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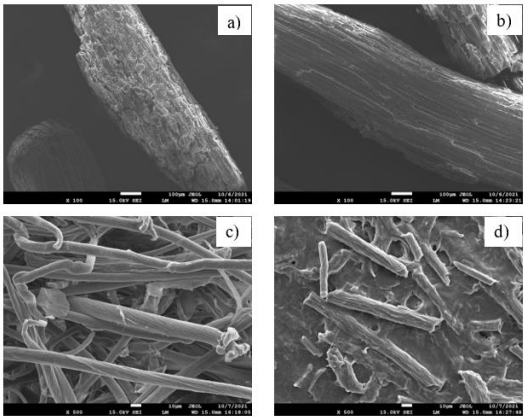

Figure 5. SEM images of original bamboo a), bleached fiber b), alkali-treated fiber c), and acid-hydrolyzed fiber d).

3.4 Spinnability of monofilament in different coagulation bath

Figure 3 shows spun fiber of different coagulation solvent selections. The spun cellulose filament in the $CaCl_2$ coagulation bath can spin easily differently from the other bath. The fiber injection in $CaCl_2$ solution, methanol solution and water as coagulation bath have a smooth and a uniform filament. However, the fiber in acetone was not a uniform filament. The acetone caused the cellulose suspension to dehydrate, encouraging the aggregation of cellulose spun and their retention in fibrous structures (Geng, Chen, Peng, & Kuang, 2017). The role of calcium chloride is ionic effect. The cellulose fiber created a crosslinking with Ca^{2+} ions and was solidified in filament (Kafy et al., 2017; Wang et al., 2019). In Figure 6, the fiber in methanol and $CaCl_2$ have continuity comparison to acetone bath, the fiber in acetone breaks easily and is discontinued. Furthermore, after leaving spun fiber in the solution, filament in water was dissolved in the water solution. The methanol and the acetone are completely evaporated and remaining only fibers.

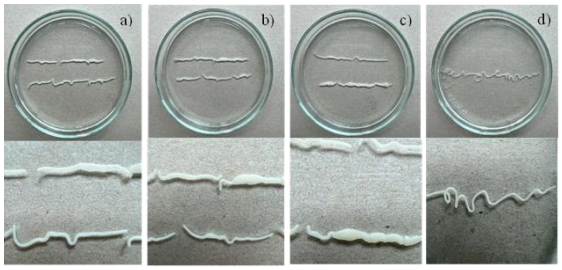

Figure 6. Spun cellulose monofilament in water a), methanol b), acetone c) and calcium chloride d).

3.5 Thermal properties

The thermal properties of monofilament injected in methanol bath, in acetone bath and in calcium chloride bath were compared with the cellulose extracted and presented in Figure 7 and Figure 8. The melting point of cellulose extracted was 210°C.

The DSC thermal curve of the cellulose and monofilaments were presented in Figure 7. The DSC curve present two temperatures observed in all samples; cellulose, C-Acetone (cellulose injected in acetone as coagulant), C-Methanol (cellulose injected in methanol as coagulant), and C-$CaCl_2$ (cellulose injected in calcium chloride as coagulant). The cellulose extracted from the bamboo with chemical and mechanical process had a degradation temperature at 210°C. The temperatures of spun cellulose were 221°C in acetone, 225°C in $CaCl_2$ and 228°C in methanol. The first peak of C-Methanol and C-$CaCl_2$ were observed at 108°C and 134°C, respectively, which presented the evaporation of moisture content in the sample.

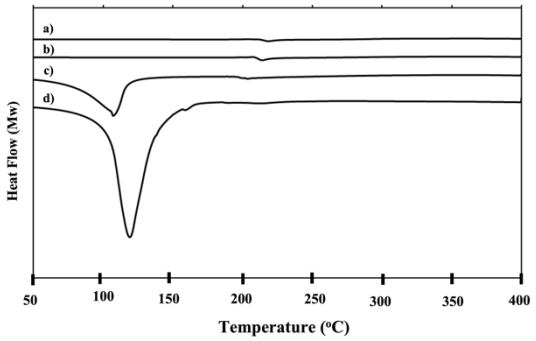

Figure 7. DSC thermal curves of cellulose a), monofilament injected in acetone bath b), in methanol bath c) and in $CaCl_2$ d).

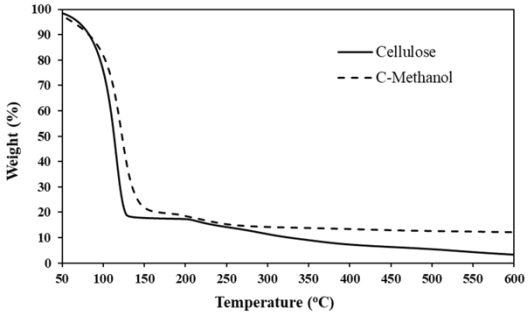

Figure 8. TGA thermal curves of the cellulose and the cellulose in methanol as coagulant.

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In addition, TGA measured the change in percent weight of sample during heating from 30 to 500°C. From Figure 8, it shows the first phase of the decline in TGA curve that refers to the evaporation of the moisture content in the sample. The degradation temperature of the cellulose extracted began more quickly than the cellulose spun fiber in methanol. The data in Table 3 shows detail of temperature and residue sample weight. When the cellulose fiber heated to 144°C, the mass about 82.1% dissipated dramatically. That indicates a large amount of moisture in the cellulose fiber. In the second part, the weight change of mass was slightly. At the temperature 264°C, the residue is 13.6%. For the spun fiber in methanol, the mass loss was 80.2% at 168°C indicating the lower water molecule content in the spun fiber. And when the temperature reaches to 212°C, the residue of the spun fiber remains 17.6%.

Table 3. The degradation temperature of cellulose and the cellulose in different coagulant.

Sample	Temperature (°C)	Residue (%)
Cellulose	264	13.6
C-Methanol	212	17.6

4. Conclusion

The cellulose nanofibers (CNF) were extracted from bamboo fibers using chemical and ultrasonic processes. The bleaching, alkali treatment and sulfuric acid reach to disintegrate and eliminate non-cellulosic. The monofilament of cellulose nanofibers was prepared using a wet spinning process. The effect of coagulating solvent consisting of water, methanol, acetone and $CaCl_2$ on monofilament formation was studied. The monofilament of cellulose nanofibers was produced in methanol and calcium chloride coagulating solvent. Morphology study of CNF monofilament verified the relationship with wet spinning process conditions. The CNF monofilament prepared in methanol as coagulant showed higher thermal properties due to the degradation at high concentration of acid sulfuric on acid hydrolysis step and high temperature of ultrasonic under exothermic reaction.

5. Acknowledgement

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