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Research Article

Synthesis and Characterization of Cellulose from Green Bamboo by Chemical Treatment with Mechanical Process

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Bamboo cellulose was prepared by chemical process involving dewaxing, delignification, and mercerization process. Four samples namely, green bamboo fiber (GBF), dewaxed bamboo fiber (DBF), delignified bamboo fiber (DLBF), and cellulose fiber (CF) had been analysed. FTIR and TGA analysis confirmed the removal of hemicellulose and lignin at the end stage of the process. FTIR results reveal that the D-cellulose OH group occurred at 1639 cm^{-1} region. SEM micrograph showed that mercerization leads to fibrillation and breakage of the fiber into smaller pieces which promote the effective surface area available for contact. Barrer, Joiyner, and Halenda (BJH) method confirmed that the effective surface area of CF is two times larger compared to GBF. CF showed the highest activation energy compared to GBF. It indicates that CF was thermally stable.

1. Introduction

In recent years, many research works have been performed on the use of cellulose fibers as a reinforcing material for composites. This is mainly due to their high strength and stiffness combined with low weight and biodegradability. Application of cellulose nanofibers in polymer reinforcement is a relatively new research field [1]. Cellulose fibers are the most abundant renewable raw material for composite fabrication. According to Habibi et al. (2010), annual production of cellulose is more than 7.5×10^{10} tons [2]. Regardless of the sources, cellulose consists of high molecular weight homopolymer of β-1,4-linked anhydro-D-glucose units. The repeat segment is a dimer of glucose called cellobiose [3].

Cellulose fibers have certain disadvantages including quality variations, moisture absorption, and poor compatibility with the hydrophobic polymer matrix. Lack of good interfacial adhesion, low melting point, and poor resistance towards moisture limit the usage of plant cellulose fiber. Therefore, pretreatment is needed to modify the fiber surface. Chemical pretreatment limits the moisture absorption process and increases the surface roughness [4]. Among the various pretreatments available, acetylation, mercerization, peroxide, benzoylation, graft copolymerization, and bacterial cellulose treatment are the best methods for surface modification of fiber. For instance, mercerization leads to breaking down fiber bundle into smaller fibers. This treatment also effectively removes lignin and hemicellulose. Mercerization increases the number of possible reactive sites and allows for better fiber wetting [4]. As a result, mercerization had long lasting effect on the mechanical properties of fibers, mainly on fiber strength and stiffness [5].

Surface area analysis is done by BJH method, developed by Barrer, Joiyner, and Halenda. This method is widely used to obtain mesopore volume and mesopore size distribution [6]. This method determines pore area and specific pore volume by using adsorption and desorption techniques. An adsorption isotherm is obtained by measuring the amount of gas adsorbed across a wide range of relative pressures at constant temperature (77 K) using liquid nitrogen. Conversely,

desorption isotherms are achieved by measuring gas removed as pressure is reduced.

In Borneo Island, bamboo is a fast-growing species and a high yield renewable resource. Bamboo is inexpensive, fast-growing, and easily available, having comparable physical and mechanical properties to wood and can be processed by existing technologies [7]. The fast growth characteristic of bamboo is an advantage for its utilization. Asia and Oceania region is the richest bamboo producer with about 65% of total world bamboo resources which also include 80% of bamboo species in the world [8, 9].

The aim of this study is to focus on the preparation of cellulose fiber from green bamboo by mechanical process and chemical treatment. The product from each stage was characterized and analyzed. The effects of each preparation steps have also been carefully examined and compared to obtain optimize synthesis outcome.

2. Materials and Methods

2.1. Material. Bamboo fibers were used as a raw material for this study. Commercial cellulose was obtained from Sigma Aldrich Malaysia. The chemicals used to produce cellulose nanofibers were toluene (Sigma Aldrich, USA), ethanol (Sigma Aldrich), hydrogen peroxide (Qeric), acetic acid glacial (Ensure), titanium (IV) oxide (JT Baker), and sodium hydroxide (Merck KgaA). All chemicals used were of analytical grade.

2.2. Methods

2.2.1. Bamboo Fiber Preparation. The bamboo was obtained from the Forest Research Institute, Sarawak, Malaysia. The green bamboo culm with 1 meter length was prepared. It was then ground using a planner machine to produce chips and powders form excluding the internode. These chips and powders mixtures were put into an oven at 70°C for 72 hours to dry. The oven dried sample was ground and sieved using 600 μm size siever. The 600 μm mesh size fibers were used for the synthesis of cellulose fiber. This sample is labeled as green bamboo fiber (GBF).

2.2.2. Preparation of Cellulose from Bamboo Fiber

Dewaxing of Bamboo Fiber. 400 mL toluene and 200 mL ethanol were filled into a round flask to produce toluene-ethanol of 2 : 1 ratio. The round flask is placed onto a heating element. A Soxhlet extractor was placed on top of the boiling flask and fixed tightly using a retort stand. 10 g of GBF was scooped into a membrane tube and then placed into the extraction thimble. A Liebig condenser is placed on top of the extractor and fixed tightly. The temperature of the heating element was observed using a digital thermometer and maintained at 250°C.

The extraction process was continued until the color mixture disappears. The process was 2 hours with approximately 10–12 cycles of extraction. The extraction thimble is taken out using a tweezer. The product is poured out into a beaker and stirred using a glass rod while adding toluene-ethanol mixture. The final product is filtered using a filter funnel with filter paper. It was distributed evenly using glass rod on a filter paper. It is then placed into an oven set at 70°C for drying overnight and was kept for delignification processes. The dried sample is identify as dewaxed bamboo fiber (DBF).

Delignification of Bamboo Fiber. The delignification solution was prepared using 82.3 g of 35 wt% hydrogen peroxide (H_2O_2) and 106.2 g of 99.8 wt% acetic acid (CH_3COOH) in the present of titanium (IV) oxide catalyst. 30 g of dry DBF sample was weighed and immersed into delignification solution in round bottom flask. The flask is placed onto the heater element and heated up to 130°C.

After 2 hours, the heater was turned off and cooled to room temperature. The treated product was then filtered using Buchner flask and rinsed with deionized (DI) water until the pH level reached 7 and dried at 70°C for 24 hours. The dried sample was placed into a bottle and kept in a dark and cool place for alkaline treatment. The sample is identified as delignified bamboo fiber (DLBF).

Mercerization. DLBF was finally immersed into alkaline solution to dissolve the pectin and hemicelluloses. 6 wt% of sodium hydroxide was used to treat the DLBF in a flask at room temperature. The mixture was stirred using autoshaker at 150 rpm, heated to 80°C for 2 hours, and stopped after 8 hours of stirring. The mixture was rinsed continuously with DI water until the product reached pH 7. The treated product was then filtered using Buchner flask, rinsed with DI water until the pH level reached 7, and freeze-dried at −85°C for 48 hours.

2.2.3. Preparation of Cellulose by Mechanical Fibrillation. The cellulose was then ground using a shear grinder for 30 min. This fine powder was called the cellulose fiber (CF). Figure 5 show the summary of cellulose preparation flow.

2.2.4. Characterization of Cellulose

Fourier Transform Infrared Spectroscopy (FTIR). The infrared spectra were obtained using a Shimazu FTIR Spectrometer model IRAFFINITY-1 CE. The spectra were taken at a resolution of 4 cm^{-1}, with a total of 60 scans for each sample. The transmittance range of the scans was 600–4000 cm^{-1}.

Scanning Electron Microscopy (SEM). The bamboo fiber samples were vacuum-dried for 24 h at 70°C, pressed onto a carbon tape adhered to a sample holder surface, and sputtered with titanium. Imaging of each sample was done using Hitachi M-3030 scanning electron microscope. All images were taken at an accelerating voltage of 5 kV with a magnification of 1500 time.

Barrer, Joiyner, and Halenda (BJH) Analysis. The bamboo fiber samples were dried for 24 h at 70°C and inserted into a capillary tube. Outgas duration was approximately 7 hours with final outgas temperature of 350°C. After outgas process, the sample was analysed using Nova Quantachrome 4200e