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Microfibrillated Cellulose Extraction from Bagasse Using a Modified Kitchen Blender

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Keywords: Cellulosemicrofiber, microfiber extraction, blender, sugarcane bagasse

Abstract. The extraction of microfibers from plant fibers can be obtained through specialized, expensive equipment that demands high energy input but delivering low production yields, resulting in highly costly microfibers. This situation confines the use of cellulose microfibers to the laboratory and not for industrial applications. The goal of this study is to extract microfibers from sugarcane bagasse (SCB) by using a kitchen blender. Earlier studies have demonstrated that paper sheets made of blender-extracted microfibers after 10 min blending have mechanical properties modulus comparable with commercially available cellulose microfibers extracted by a high-pressure homogenizer. By reducing the volume of aqueous suspension, resulting in higher tensile modulus to those of sheets made from commercially available cellulose microfibers. The FTIR analysis demonstrated that the treatments resulted in the gradual removal of lignin and hemicelluloses from the fiber. Morphological characterization identified that the diameter of the fibers varied between 20 nm to 12 µm. Finally, the high enough strength and comparable mechanical properties (modulus) of SCB microfibers to those of commercially available cellulose microfibers, confirming their suitability in the manufacturing biomaterial composites.

Introduction

Recently, attention is devoted to the preparation and utilization of microfibrillated cellulose (MFC) to produce high-performance nanocomposites given their high mechanical properties, renewability, and high surface area to volume ratios. Furthermore, with the combination of bioplastic matrix, these materials can be made entirely environmentally friendly.

Cellulose fiber is obtained from a variety of sources, which include grasses, leaf fibers, wood, seed fibers, bast fibers, marine animals (tunicate), fungi, algae, invertebrate and bacteria [1]. Accordingly, Cellulose fiber can be converted into different nanostructures with various physical properties, depending on the cellulose source and the production method. The primary source of MFC has been wood pulp fibers, which is subjected to mechanical treatment in order to produce nanofibers. MFC production methods consist of several operational stages by specialized devices requiring high energy consumption. Notably, less content lignin makes non-wood plants receiving increasing attention as potential sources of cellulose and consequently, purification processes of the fiber are much easier and avoid damage to the cellulose. Moreover, it can reduce energy consumption for fibrillation of such cellulose.

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3 Cellulose microfibrils are basic structural units of all plants, which has Young's modulus of around 140 GPa [2] and its tensile strength is 1,7 GPa [3]. For these reasons, it is of great interest to examine the possibilities and limitations of their extraction method and processing conditions.

In this study, modifying an original 2-liter container (bottle) by reducing its capacity by half (i.e., 1-liter) as introduced by Nakagaito et al. [4] and using stainless steel with six cutting blades rotating at the bottom of the blender bottle is proposed to optimize the fibrillation of bagasse fiber. The tensile test was used to determine the tensile properties. The effect of the agitation time on surface topography and morphology was characterized using SEM, chemical, and physical FTIR spectroscopy. It was observed that cellulose microfibrils were obtained by using the combination of chemical and mechanical treatments which would be suitable for manufacturing biocomposites for various applications.

Experimental Methods

Materials. Sugarcane bagasse (SCB) was obtained from Madu Baru sugar factory (Yogyakarta, Indonesia). Sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2) solutions were used for pre-treatment and bleaching of the fibers. A Philips HR2096 800 W (21.000 rpm) household blender was used for mechanical fibrillation.

Fiber pre-treatment. Bagasse fibers (SCB) were cut for 2 mm in length and treated two times with a NaOH solution 5 wt.% at $90^\circ C$ under constant agitation for 1,5 h to remove hemicelluloses and lignin before drying at $105^\circ C$ in an oven for 6 h. The weight ratio of the NaOH solution and bagasse was 30:1. The resulting pulp fibers were then washed using distilled water until the water reached the neutral pH level. Then, bleaching was performed at $90^\circ C$ by adding the pulp fibers obtained previously, into preheated 5 wt.% H_2O_2 under an alkaline condition (by adding the NaOH solution until the pH level was 11) followed by 45 min of mechanical stirring. The weight ratio of the H_2O_2 solution and pulp fiber was 40:1. After that, the bleached pulp fibers were washed with distilled water to achieve neutrality.

Fibrillation process. A suspension of pulp fibers was agitated using a household blender (Philips HR2096 800 W) with an operating speed of around 21 000 rpm, in which the stainless steel with six cutting blades rotates in a recessed section at the bottom of a modified bottle, with a capacity of 500mL.

Next, fibrillating 500 mL of bagasse fiber pulp aqueous suspension with a fiber content of 1 wt.% and agitation speed of 21 000 rpm were carried out. The agitation time was set for 3, 5, and 10 min, respectively. Paper-like sheets were obtained by filtering the fiber suspensions in a vacuum filter, followed by drying at $60^\circ C$ for 5 h in a cabinet dryer. The commercial cellulose nanofiber Celish KY-100G (Daicel Corporation, Japan) produced by a high-pressure homogenizer was used as control [4].

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Characterization of fibers. Fourier transform infrared spectroscopy (FTIR) spectra were obtained using a SHIMADZU IR Prestige 21 Fourier transform infrared spectrometer. The samples were analyzed in a spectral region between 4000 and 400 cm^{-1} , at a resolution of 2 cm^{-1} , performing 20 scans. The morphological structure of bagasse fiber before and after chemical and mechanical treatment was examined using a Scanning Electron Microscope (SEM), (JOEL Ltd., JSM-6510LA). The samples were placed on carbon tapes and coated with a thin layer of platinum using a Sputter Coater (JEOL Ltd., model JEC-3000 FC). The sample test pieces $70\text{ mm} \times 10\text{ mm}$ for the tensile test were cut from the fiber sheets and subjected to a tensile test with a MESDAN model TENS 300 at a strain rate 1.0 mm/min . Thick paper tabs were prepared to prevent damage to the ends of each specimen during handling. A specimen was glued to the paper tabs, which was then carefully gripped to a testing machine. The cross-sectional areas corresponding to the actual fracture sites were used to determine tensile strength and Young's modulus. In this study, averaged across five specimens were tested and analyzed for tensile properties.

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Results and Discussions

Effect of chemical treatment. The effectiveness of alkali and bleaching treatment of the fiber were analyzed using an FTIR spectroscopy. The FTIR spectra of untreated, alkali, and bleached fibers are depicted in Fig. 1, respectively.

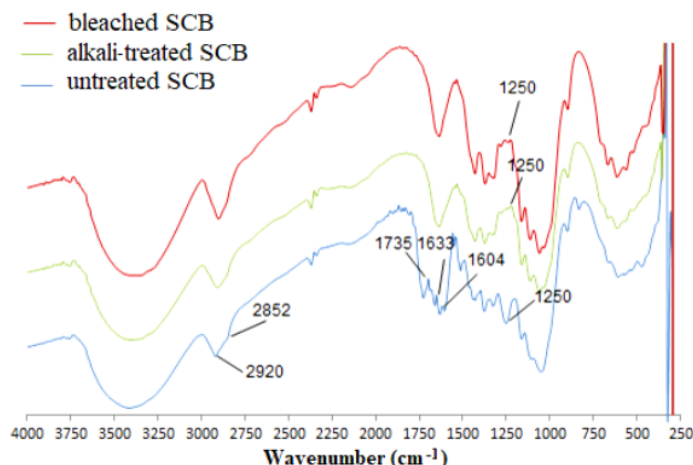


Figure 1. Fourier transform infrared (FTIR) spectra for untreated, alkali-treated, and bleached sugarcane bagasse (SCB)

The FTIR spectra of all samples have shown a wide band in the region between 3100 and 3600 cm^{-1} that specifies the free O-H is stretching intramolecular hydrogen bonds for cellulose [5]. The peak at 3386 cm^{-1} is characteristic of the O-H group present in lignin, and carbohydrates. There is a sharper peak change in O-H group bondage from before until after the chemical process (Fig.1). It can be interpreted as a reduction in the number of O-H groups due to alkali and bleaching treatments. The 2920 cm^{-1} peak may be associated with aliphatic C-H in CH_2 and CH_3 groups of cellulose, lignin, and hemicellulose [6]. The peak at 2852 cm^{-1} represents the vibration of the OCH_3 group, commonly present in lignin [6]. This group can also be characteristic derived from hemicellulose acetyl. The disappearance of the peak OCH_3 group after alkali treatment confirmed the removal of some part of lignin and hemicellulose follow by a change in the lignin methoxyl group into a phenol group at 1250 cm^{-1} . The peaks at 1604 cm^{-1} and 1633 cm^{-1} show the C-Ph and C = C groups, respectively [6]. The peaks are usually found in the aromatic structure of lignin. Both of these peaks disappear after alkali treated fibers. The peak at 1735 cm^{-1} represents the acetyl group present in hemicellulose. The peak also disappears after alkali treatment [7].

The absence of the peak at 1250 cm^{-1} after bleaching treatment indicating the removal of C-O (phenol group) derived from lignin. The lack of the phenol group followed by changes in the stronger absorption area of the OH group (sharper peak region) confirmed the removal of the remaining lignin content from the alkali treatment and leaving pure cellulose fibers. These results denote that treatment with alkali followed by bleaching is an effective way to remove hemicellulose and lignin from bagasse to obtain pure cellulose fibers.

Mechanical strength of SCB sheet. The original blender bottle (2-liter) is replaced with a smaller capacity (1-liter) as shown in Fig 2. The aim is to increase the probability of the fibers colliding with six rotating blades, resulting in greater impact and shear forces, that can segregate the cell walls of the fibers and fibrillate them into finer microfiber bundles.



Figure 2. Modified 1-liter blender bottle and the original 2-liter bottle

Evaluation of the quality of microfibers obtained by testing the tensile strength of the paper-like sheets of SCB. Assuming the cellulose molecule chain group contain O-H (hydroxyl groups), the more surface area of microfibers, the higher tensile sheet strength due to the increasing number of hydrogen bonds interconnecting the microfibers. At the same time, the remaining thick microfibers bundles which not completely microfibrillated have the potential to be defects which decrease the tensile strength of the sheets.

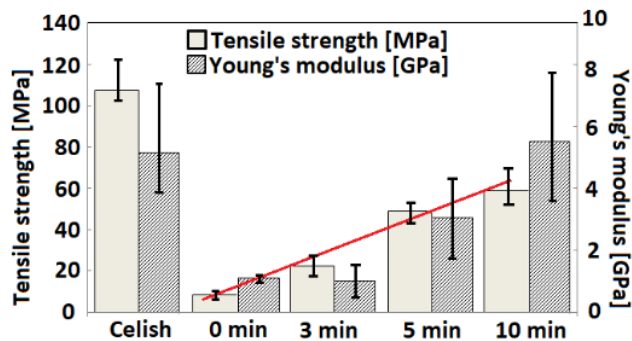


Figure 3. Tensile properties of 0, 3, 5, and 10 min microfibers sheets extracted by the blender in 500 mL aqueous suspension compared to a commercial nanofiber Celish sheet

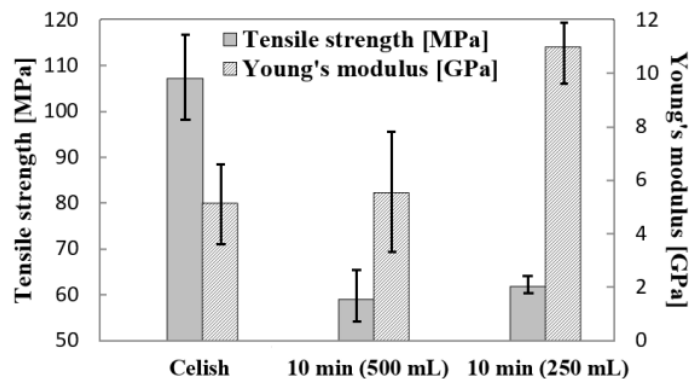


Figure 4. Tensile properties of microfiber sheets fibrillating in different volume of an aqueous suspension for 10 minutes compared to a commercial nanofiber Celish sheet

The tensile strength and Young's moduli of a paper-like sheet of SCB as a function of agitation time are presented in Fig. 3. The ultimate strength of the sheet increased linearly with the agitation time. The strength gain at agitation time at 10 min was approximately 59 MPa, or 6 times higher compared to the original sheet (0 min of agitation) and it was half of than sheets made from commercial Celish.

Similar to the ultimate strength, Young's modulus of the sheet agitated for 3 to 10 min increased linearly from the original sheet. At agitation time of 10 min, the modulus was about 5,5 GPa, or 8 times higher than that of the original sheet and comparable to sheets made from commercially available Celish [4], which are produced by high-pressure homogenization.

In order to increase the probability of the fibers colliding with the rotating blades, the volume of water solution was reduced to 250 ml. Figure 4 shows the tensile strength and Young's moduli of fibers fibrillating in 250 mL (SCB25) and 500 mL (SCB50) in aqueous suspension for 10 min. The average tensile strength of SCB25 (62 MPa) sheets increased slightly than SCB50 (59 MPa). However, a more homogenous distribution of strength with respect to SCB sheets was observed, or it means that the remaining thick microfibrils bundles which not completely microfibrillated decrease and more homogeneous distribution of finer microfibril bundle are obtained.

In a case of tensile Young moduli of SCB sheets, the moduli of sheets increased from about 5,5 GPa (SCB50) to twice as high at value around 11 GPa (SCB25). Moreover, these microfibril sheets also stronger in moduli than that of sheets made from commercial Celish.

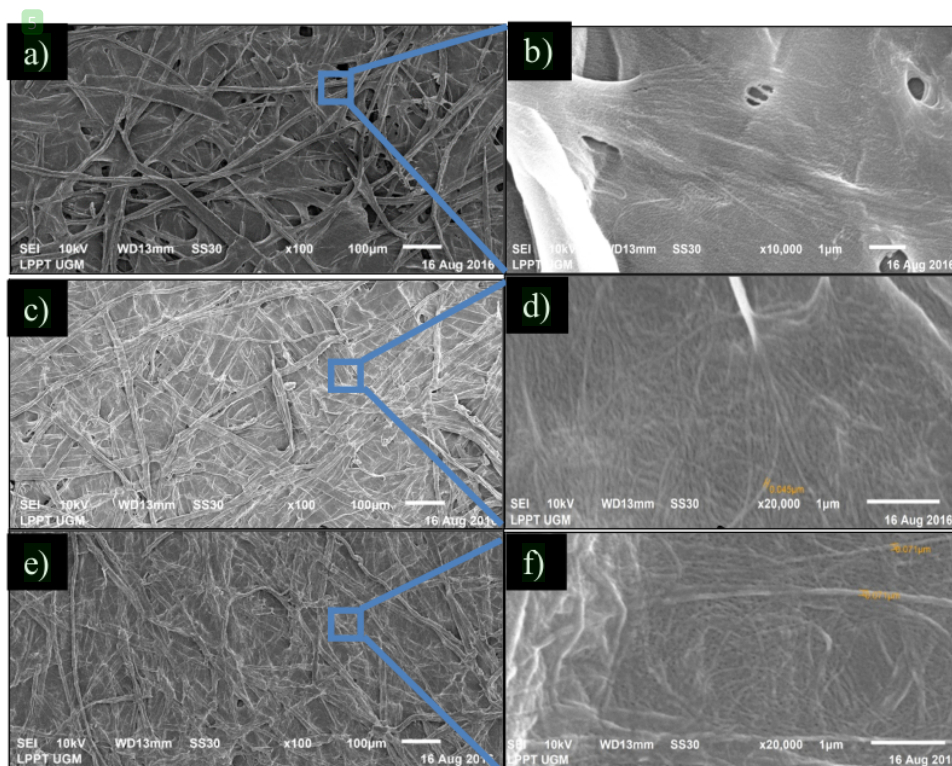


Figure 5. Morphological investigation. Scanning electron micrographs of cellulose fibers agitated by blender for (a and b) 3; (c and d) 5; (e and f) 10 min, (SEM, 100 μm scale and blue rectangles are shown the magnified detail of the fibers 1 μm).

Scanning Electron microscopy (SEM) analysis. Figures 5(a-f) show that the fiber fraction was reduced, and the fibrillated fraction increased with increasing agitation time. The fiber diameter extracted from the bagasse appeared to be less uniform from 20 nm to 12 μm . Microfibrils were observed in 5, and 10 min samples where fiber fraction was reduced, and the portion of microfibrils increased with increasing agitation time. The fibers are often aggregated, and the individual fibers diameter in the range of 7-20 nm. However, it is difficult to characterize the aspect ratio and this fibril orientation because of agglomeration and fiber's small dimensions. Future

research is necessary to ascertain the aspect ratio and the role of fibril orientation which are an important factor influencing the mechanical properties of sheets.

Summary

The goal of this study is to optimize the extraction process to isolate cellulose microfibrils from sugarcane bagasse using 800 Watt modified kitchen blender. By decreasing the volume of the blender bottle, the microfibril aggregation can be obtained in short treatment time, reducing the cost of fiber extraction. Moreover, it is important for future works to consider the effect of chemically pre-treatment on the fibrillation efficiency and energy consumption of the cellulose microfibrils. With the use of a properly modified kitchen blender with appropriate chemically pre-treatment, anybody interested in developing cellulose microfibrils in a simple and inexpensive way.

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