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Isolation of cellulose fibers from kenaf using electron beam

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ABSTRACT

Cellulose fibers were isolated from a kenaf bast fiber using a electron beam irradiation (EBI) treatment. The methods of isolation were based on a hot water treatment after EBI and two-step bleaching processes. FT-IR spectroscopy demonstrated that the content of lignin and hemicellulose in the bleached cellulose fibers treated with various EBI doses decreased with increasing doses of EBI. Specifically, the lignin in the bleached cellulose fibers treated at 300 kGy, was almost completely removed. Moreover, XRD analyses showed that the bleached cellulose fibers treated at 300 kGy presented the highest crystallinity of all the samples treated with EBI. Finally, the morphology of the bleached fiber was characterized by SEM imagery, and the studies showed that the separated degree of bleached cellulose fibers treated with various EBI doses increased with an increase of EBI dose, and the bleached cellulose fibers obtained by EBI treatment at 300 kGy was separated more uniformly than the bleached cellulose fiber obtained by alkali cooking with non-irradiated kenaf fiber.

1. Introduction

Kenaf (*Hibiscus cannabinus*) is one of the non-wood fibers that grow in tropical and sub-tropical areas. Recently, kenaf has drawn much attention as a replacement wood because it grows rapidly, reaching 12–18 feet in just five months, and also has good fiber quality (Seo et al., 2008; Nishino et al., 2003; Adamson and Bagby, 1975).

Kenaf cell walls in common with wood consist of stiff fillers, called cellulose fibers, embedded into soft matrix substances such as hemicellulose and lignin. Cellulose fibers of semicrystalline is a linear polymer with high aspect ratio and excellent mechanical properties including a high Young's modulus and a very great potential for use as reinforcement in composite, pulps for producing paper, and converting into a wide variety of derivative products such as rayon and an alternative fuel source. On the other hand, lignin and hemicellulose of a perfectly amorphous structure are softer than cellulose and act as glue between celluloses fibers by virtue of the interaction or linkages between the cellulose and the matrix (Yang et al., 2007). And then, in order to efficiently isolate cellulose fibers, it is essential to remove the lignin and hemicellulose as much as possible. Cooking process is done to remove the lignin and hemicellulose in the chip. This process use a combination of high temperature and chemicals such as alkaline sulfate and acid sulfite to break or weaken the chemical bonds by chemical action so that the cellulose fibers can be separated (Turbak et al., 1983; Charkraborty et al., 2005). However, the chemical cooking processes are still encountered with a large amount of high-efficiency, cost-effective, and environment-friendly problems (Dey and Gupta, 1992).

Electron beam irradiation (EBI), without adverse effects, has been used to decrease cell wall constituents, or degrade and delignify cellulose based fibers. The basic advantages of EBI includes the ability to promote changes in reproducibly and quantitatively, without the introduction of chemical reagents and without the need for special equipment to control temperature, environment, and additives (Van Soest et al., 1984; Mason et al., 1988; Masri and Zarkawi, 1994; Sandev and Karaivanov, 1977).

The aim of this study was to provide a progressive and convenient process for cellulose isolation from EBI kenaf bast fiber using only water without chemicals during cooking treatment. The obtained cellulose fibers were characterized using a chemical composition, FT-IR, SEM, and XRD.

2. Experimental

2.1. Materials

Five-month-old kenaf fibers were obtained from an internal source in Korea, i.e., Advanced Radiation Technology Institute, Korea Atomic Energy Research Institute. In this study, the obtained kenaf was separated into bast and core by hand and then air-dried. The dried bast fibers were used for this experiment.

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2.2. Methods

2.2.1. Electron beam irradiation of kenaf bast fibers

The Electron beam irradiation of the kenaf bast fibers were carried out at EB tech of Korea. The EBI treatments were conducted using the scanned beam of conveyor type at 1.14 MeV (beam current 7.6 mA, irradiation width 110 cm, distance between window and sample 20 cm, dose rate 6.67 kGy/sec) at room temperature in an air atmosphere. Dosimetry was performed using cellulose tri-acetate (CTA; ISO/ASTM 51650 (2005)(E)).

2.2.2. Isolation of cellulose fibers from kenaf bast fiber

Cooking treatments for removing the lignin and hemicellulose were carried out in an autoclave under following condition (Table 1).

In addition, for increasing the cellulose content by removing the residual lignin in kenaf fiber, a two-step bleaching process of all samples cooked was carried out as shown in Table 2.

2.3. Characterization

All Samples for chemical analysis were reduced to powder by miller. Neutral detergent fiber (NDF) values were gained by refluxing the milled kenaf samples with sodium lauryl sulfate (pH 7.0) solution for 1 h. Hemicellulose contents were established by weight loss of the NDF residue after acid detergent treatment, and then calcined at 550 °C after 72% H_2SO_4 treatment to obtain cellulose and lignin contents. The same method was also used to gain acid detergent fiber (ADF). Table 3 shows chemical composition of raw kenaf bast fiber (Van Soest et al., 1991; Van Soest and Wine, 1967; Fulgencio et al., 1983).

Table 1
Condition of cooking process.

	Non-irradiated kenaf chips (0 kGy)	EB-irradiated kenaf chips
NaOH	12%	Use of only water without chemicals
AQ	0.15%	
Liquor-to-kenaf ratio	10:1	
Maximum temperature	120 °C	
Time to maximum temperature	120 min	

Table 2
Conditions of the bleaching process.

	Step 1	Step 2
Chemical charge	Sodium chlorite (2%) Acetic acid (3%)	NaOCl (1.2%)
kenaf consistency	10%	10%
Temperature	70 °C	Room temperature
Time	90 min	60 min

Table 3
Chemical composition of raw kenaf bast fiber.

Raw Kenaf bast fiber		
Cellulose (%)	Hemicellulose (%)	Lignin (%)
60.75	19.21	14.71

The FT-IR spectra were obtained by BRUKER, TENSOR 37 in order to determine changes in functional groups that may have been caused by each treatment. All samples were prepared as thin films, and mixed with KBr in sample/KBr ratio of 1/100(w/w). The spectra obtained were the results of 30 scans at a spectrophotometer resolution of 8 cm^{-1}.

The X-ray diffraction patterns of samples were recorded by RIGAKU, D/MAX-2500 with CuKα radiation generated at 40 kV and 30 mA.

Scanning electron microscopy (SEM) was used to observe the effect of the treatments on the fiber morphology. The samples were coated with gold using a vacuum sputter coater and observed with a Jeol JSM 5910 LV microscope.

3. Results

3.1. Chemical composition

The chemical compositions after cooking process are shown in Fig. 1(a). Non-irradiated kenaf fiber obtained after alkali cooking were found to be efficient in removing the lignin and hemicellulose: the lignin content decreased from 14.71 to 6.05 wt% while the amount of hemicellulose decreased from 19.21 to 8.62 wt%. EB irradiated kenaf fibers after water cooking were not found to significantly remove the lignin and hemicellulose, and then the cellulose contents were increased piecemeal compared to those of non-irradiated kenaf after alkali cooking.

The two-step bleaching process was used to increase the cellulose content and to remove most of the lignin and hemicellulose: an acidified sodium chlorite solution caused a high extraction of lignin and hemicellulose, and NaOCl rendered the removal of lignin more efficiently, thus we could obtain more pure and separated cellulose fibers than the previous process. Based on the chemical composition analysis after two-step bleaching as shown in Fig. 1(b), the content of the lignin and hemicellulose were found to be further reduced while the cellulose contents were more increased. The bleached cellulose fibers obtained by alkali cooking with non-irradiated sample did not show a significant difference for the decreased content of the lignin and hemicellulose. Meanwhile, the bleached cellulose fibers obtained by water cooking with EBI sample decreased the content of the lignin and hemicellulose with an increase of EBI dose. Especially, the value of chemical composition of the bleached cellulose fiber treated at 300 kGy showed a significant result: the lignin content was reduced from 8.64% to 0.68%, the amount of hemicellulose was reduced from 12.61% to 2.38%. The cellulose content was, therefore, increased remarkably from 68.24% to 93.45. These results showed that EBI treatment on kenaf bast fiber affected on the removal of the lignin and hemicellulose through water cooking and bleaching process, as evidenced by the increased cellulose content.

3.2. FT-IR analysis

FT-IR spectroscopy was used to illustrate the physical structure and functional groups of non-irradiated kenaf and EB-irradiated kenaf fibers obtained after each process. The FT-IR was presented in Fig. 2.

The stretching O–H bond and C–H bond for all samples were absorbed strongly in the 3400–3300 cm^{-1} region and in the 2900–2800 cm^{-1}. The peak at 1731 cm^{-1} in the raw kenaf bast fiber was due to the C=O stretching of the acetyl group in hemicellulose or the ester linkage of carboxylic group in the ferulic and p-coumeric acids of lignin or hemicellulose (Alemdar and Sain, 2008; Khalil et al., 2001; Sgriccia et al., 2008). This peak in the spectra of non-irradiated kenaf after alkali cooking disappeared completely, but the peak of EBI kenaf fibers obtained after water