

Analysis of Structural Changes in Jute Fibers after Peracetic Acid Treatment

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ABSTRACT

Peracetic acid (PAA) is one of the most commonly used bleaching agents in the textile industry. The structural changes of jute fibers after PAA treatment are important in creating industrial applications. An investigation was carried out to determine the structural changes resulting from the removal of noncellulosic materials from jute fibers by PAA. The surface features of jute fiber were studied using a scanning electron microscope. PAA was effective in separating individual cellulose fibers from raw jute. However, fibers were damaged when the treatment time reached 80 minutes, PAA concentration was 10 wt. % and the pH was in the 4~5 range. The delignification by PAA treatment was confirmed by Fourier transform infrared spectroscopy. CP/MAS ^{13}C -NMR spectroscopy showed that the PAA did cause some macromolecular structure changes in the cellulose fibers. X-ray diffraction technique demonstrated that there were four stages of crystallinity change, due to the removal of noncellulosic materials. These four stages include attack at the surfaces of cellulose crystallites, the dissolution of amorphous zones, attack of the inner cellulose crystallite and oxygen interference, respectively.

Keywords: Jute Fibers; Peracetic acid; Scanning electron microscope; Fourier transform infrared spectroscopy; CP/MAS ^{13}C -NMR; X-ray diffraction

INTRODUCTION

Bast fibers have become the focus of intense interest in recent years. The use of bast fiber materials such as jute, hemp, ramie and flax provides several advantages including low density, low cost, good specific mechanical properties and biodegradability [1]. Jute fiber is the second most common natural fiber in the world and it is extensively grown in Bangladesh, India and China [2]. *Corchorus capsularis* L. (white jute) and *Corchorus olitorius* L. (tossa jute), which belong to the family Tiliaceae *Corchorus*, are commonly cultivated for fiber [3].

Normally jute is used for traditional purposes such as sacks, bags, hessian cloth, burlap, yarn, carpet backing and composite reinforcement [4].

Figure 1 depicts the jute stem and transverse section. Jute fiber, which is composed of a number of ultimate cells, including cellulose, hemicellulose and lignin. Cellulose is the main chemical component and thus contributes largely to the mechanical properties of fibers (*Table I*) [5]. Many researchers focused on the selective removal of non-cellulosic components for industrial uses [6-8]. Thus, it is important to investigate the structural changes in jute fibers after different chemical treatments.

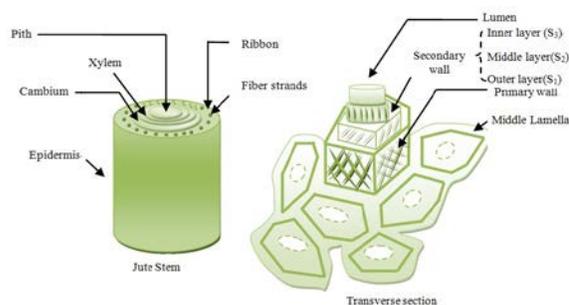


FIGURE 1. Scheme of the jute stem and transverse section [5,9,10].

TABLE I Approximate chemical composition of bast fibers (%) [5].

Fiber	Cellulose	Hemicellulose	Pectin	Lignin	Fat/Wax
Flax	62-71	16-18	1.8-2.0	2.0-2.5	1.5
Hemp	67-75	16-18	0.8	2.9-3.3	0.7
Ramie	68-76	13-14	1.9-2.1	0.6-0.7	0.3
Jute	59-71	12-13	0.2-4.4	11.8-12.9	0.5

The conventional bleaching process for jute is based on sodium hypochlorite technology. However, wide adoption of this technique could cause serious environmental problems because of the formation of toxic absorbable organic halides (AOX) during the

bleaching process [11]. Peracetic acid is most often used as bleaching agent in textile industry and has a special delignification effect on the lignocellulosic biomass [12]. Non-toxic and antimicrobial decomposition products are specific benefits of use of peracetic acid. The treated fibers have high whiteness but low damage after peracetic acid bleaching. Peracetic acid decomposes as shown in *Figure 2* during bleaching. The reactive oxygen [O] and peroxy radical [OOH] are the major oxidizing agents [13]. Two main mechanisms of the reaction of peracetic acid with lignin are electrophilic addition and substitution and nucleophilic reactions [14].

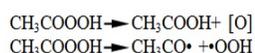


FIGURE 2. Chemical reaction of peracetic acid decomposition during bleaching process [13].

Recently, several researchers have studied the use of peracetic acid for bleaching jute. Mazumder *et al.* [15] bleached tossa jute with various concentrations of PAA in the laboratory. Weight loss and handle were improved following the PAA bleaching. Cai and David [16] demonstrated that 100% jute fabric and jute/cotton blends could be effectively bleached with peracetic acid in the presence of a phosphate buffer and tetrasodium pyrophosphate (TSPP). They also found [17] that peracetic acid-TSPP and $\text{H}_2\text{O}_2\text{-Na}_2\text{SiO}_3$ combined system improved the bleaching effects for 100% jute fabric and jute/cotton blends. This combined bleaching process could be regarded as a chlorine replacement study.

survey of previous literature indicates that no one investigated the effects of peracetic acid on the structural changes of jute fibers. To that end, this paper presents the results of experiments to analyze and compare the structural changes of jute after treatment with PAA as a function of treatment time. Fourier transform infrared (FTIR) was used to investigate chemical changes, CP/MAS ^{13}C -NMR spectroscopy was employed to characterize the structural change of the macromolecular cellulose, and X-ray diffraction was used to evaluate cellulose degradation by monitoring the changes in the relative degree of crystallinity.

EXPERIMENTAL

Materials and Methods

The jute used in this study was *Corchorus capsularis* L. (Tiliaceae), which was supplied by Wuxue Xinji Jute Textile Co., Ltd.(CHINA). Acetic acid, hydrogen peroxide and sulfuric acid were purchased from the Sinopharm Chemical Reagent Co., Ltd., (CHINA). Dimethyl sulfoxide was provided by

Shanghai RichJoint Chemical Reagents Co., Ltd., (CHINA). All the reagents were of analytical purity and used without further purification.

Peracetic acid is a strong oxidizing agent and is easy to decompose. It was prepared by reaction of 30% (w/w) hydrogen peroxide and anhydrous acetic acid in situ, with a volume ratio of 1.5:1 at room temperature for 48h. As shown in *Figure 3*, 1.5% (w/w) of sulfuric acid was added as a catalyst. This method was described in Zhao *et al.* [18]. The determination of peracetic acid concentration was in accordance with Chinese standard GB/T 19108-2003. The PAA concentration in the equilibrium mixture according to above methods was 10-12 wt. % (pH=4~5).

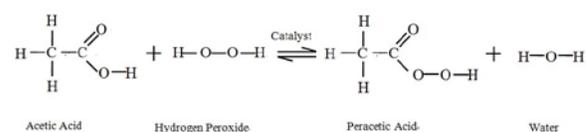


FIGURE 3. Preparation of peracetic acid.

All the samples were thoroughly combed. The chemical composition (w/w) of raw materials is 0.75% wax, 16.70% Klason lignin, 12.57% hemicelluloses, 58.23% cellulose and 3.9% pectin. The jute fibers (cut to 3-4 cm in length) were treated with 0.1 % sulfuric acid (1:20) at 50°C for 1 h. The fibers were washed with distilled water several times. The samples were sequentially treated with dimethyl sulfoxide (DMSO) in a flask and washed. After pre-treatment the sample was labelled as Pre-Jute.

The jute fiber samples (5.0~6.0 g) were immersed in peracetic acid at a liquor ratio of 20:1 for 10 to 160 minutes (*Table II*). The reaction temperature was 60°C. After the reaction, the excess reagent was pressed out and the fiber washed with distilled water several times until the pH of the wash water was neutral. The residue was then dried in an oven at 60°C for 24h. The sample designations and immersion times are listed in *Table II*.

TABLE II Sample designations and immersion times.

Sample Label	Immersion Time(min)
D1	10
D2	20
D3	40
D4	60
D5	80
D6	100
D7	120
D8	140
D9	160

Characterization of Jute Fibers

Chemical Composition Analysis

The wax, pectin, cellulose, hemicellulose and lignin levels in samples were analyzed according to the China National Standard GB5889-86 (1986).

Scanning Electron Microscopy (SEM)

SEM (TM-1000, Hitachi, Japan) was used to study fiber morphology changes. The samples were coated with gold using a sputtering technique. Representative electron micrographs are presented in order to show the important surface features. All images were taken at an accelerating voltage of 15 kV.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR (NICOLET-5700, Waltham, MA, USA) was used to examine composition changes during the peracetic acid treatment process. The samples were ground into powder and palletized with KBr for testing. Spectra were obtained in the range of 700-4000 cm^{-1} with a resolution of 4 cm^{-1} .

Solid-state ^{13}C Nuclear Magnetic Resonance (NMR)

Solid-state ^{13}C NMR spectra of jute raw fibers and peracetic acid treated jute fibers were recorded using Avance 400 Bruker spectrometer (Bruker Inc., Switzerland) with a cross polarization/magic angle spinning (CP/MAS) unit at room temperature.

X-ray Diffraction (XRD)

The crystallinity changes of the jute fibers were measured with an X-ray diffractometer (D/Max-2550 PC, Rigaku, Japan) using $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at 40 kV and 200 mA. Scattered radiation was detected in the range of $2\theta = 5^\circ - 60^\circ$ at a scan rate of 4 $^\circ/\text{min}$. The relative degree of crystallinity was calculated according to the following Segal empirical method [19]:

$$C_r I = [(I_{002} - I_{\text{am}}) / I_{002}] \times 100\% \quad (1)$$

where I_{002} is the peak intensity from the 002 lattice plane and I_{am} is the peak intensity of the amorphous phase.

RESULTS AND DISCUSSION

Chemical Analysis of Jute Fibers

Figure 4 shows the changes in content (wt. %) of cellulose, lignin and hemicellulose from jute after peracetic acid treatment. The cellulose content increased with the removal of hemicellulose and lignin. The dramatic reduction of lignin content is due to the reaction between PAA and lignin, which results in electrophilic hydroxylation and formation of quinone, lactones and muconic acid [20-22]. According to the literature, the initial reaction between PAA and lignin is the electrophilic hydroxylation of active positions of the aromatic ring, ortho- and para oxygenated groups, as shown in Figure 5a. In Figure 5b, via an intermediate hemiacetal, aromatic ring hydroxylation results in demethoxylation, which leads to quinone formation. Quinones are more easily oxidized than the initial compounds. During the final oxidation stages of the lignin aromatic structures, the lactose and muconic acids form as a result of the oxidative cleavage of the rings from quinones as seen in Figure 5c. It has been reported that peracids do not possess high reactivity towards polysaccharides [23], so the cellulose resulting from peracid bleaching relatively high in delignification. The PAA equilibrium mixture used is slightly acidic, and hemicellulose is more easily susceptible to acid hydrolysis [24].

Compared with other bleaching agents used for jute, which require relatively high temperatures and complicated bleaching procedures [7,8,25], PAA has been shown to be an effective and efficient bleaching agent for jute fibers. PAA has been shown to be effective in bleaching lignocellulosic biomass such as sugarcane bagasse [26].

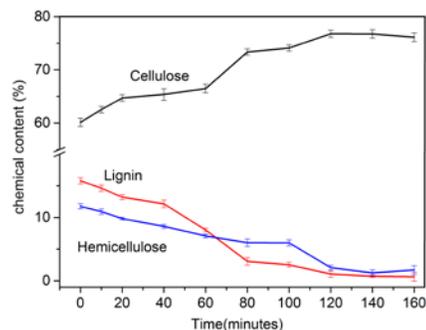


FIGURE 4. The effect of PAA immersion time on the chemical composition of jute fibers.

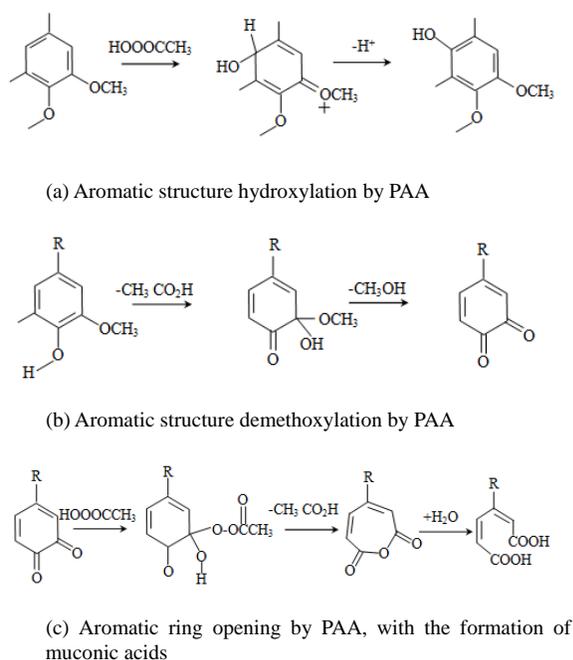


FIGURE 5. Reactions of PAA with lignin [21, 22].

Scanning Electron Microscopy (SEM) Analysis

Figure 6 shows the SEM micrographs of the raw jute and the samples after different stages of peracetic acid treatments. The photograph of raw jute (Figure 6a) shows a multicellular appearance. The surfaces of the individual cells are not clearly visible because the fibers were cemented using gummy materials. The measured diameter of raw jute was around 61 μ m. Some impurities and debris observed in the SEM image were considered to be lignin based crystals and deposition of calcium carbonate (CaCO₃) during bleaching [27-28]. After pretreatment, it can be seen that the gum of the raw jute bundle fibers has been partly removed. The crisscross network arrangement of the elementary fibrils could be observed in the primary layer (Figure 6b). Quite a few fibrils were observed showing parallel orientation along the axis.

Jute fibers D1 (Containing 14.64% Lignin) are clean and smooth due to the dissolution of noncellulosic materials (Figure 6c). The pictures (Figure 6d) show much more clear surface features for jute fibers D2 (Containing 13.23% Lignin) than for fibers D1. When the lignin content of Jute fibers D3 is 12.16% (Figure 6e), the surface appears clean, smooth and shows nodes. Some helically oriented fibrils ('Z' shape) are also observed in the secondary layer in jute, which appear to bind the straight fibrils as with a rope. As in previous research [29], there are too few

in the upper portion of the secondary layer to be identified after during chemical treatment. Nodes could be observed when ultimate cells were exposed. This occurred at irregular intervals. These nodes extend horizontally across the fiber and mostly remain in the outer layers of the fiber. When the outer layers are dissolved by peracetic acid treatment, the nodes disappear and the fibrils could be observed in parallel to the fiber axis with straight orientation. Occasional decementing action occurs starting with the multicellular structure when the lignin content of Jute fibers D4 is 8.03 % (Figure 6f). The removal of lignin from the middle lamellar region is significant.

Most of the noncellulosic cementing materials in the intercellular region had been removed from Jute fibers D5 (Containing 3.07% Lignin). All the ultimate cells (Figure 6g) are completely separated from the fiber strands, resulting in a diameter reduction. The separated cells showed a variety of shapes and sizes. The ultimate cells exhibit diameters around 7-21 μ m. The experimental results combined with the results of previous work by Rahman [27] show that the ultimate cell could be obtained when the lignin content was between 4.3% and 1.03%. Evidence suggests that there are two binding mechanisms of the middle lamella- physical and chemical bonding. Some of the lignin molecules infiltrate the cellulosic network of the cell walls and form strong bonds with some of the cellulose chains [30-31]. This explains why it is hard to remove the lignin during the bleaching process.

The fibrillation in the secondary cell wall is now visible in most of the jute fibers D6 (Containing 2.53% Lignin). It can be seen that the individual ultimate (Figure 6h) cells are separated. The degradation of the cellulosic materials in the secondary cell wall was noted in the delignified jute fibers. The fiber cell walls (S2) become thinner with the gradual reduction of lignin. The nodes were eroded, and some holes and transverse crack were observed in Figures 6g-j. The fibers seem easily damaged by the charging effect.

It can be observed that individual ultimate cells (Figure 6k) have collapsed surface depressions are observed. Degradation of the secondary cell wall for cellulosic materials was obvious for jute fibers D9 (Containing 0.64% Lignin). This may be due to the high vacuum to which they are exposed during sample coating and examination in the SEM.

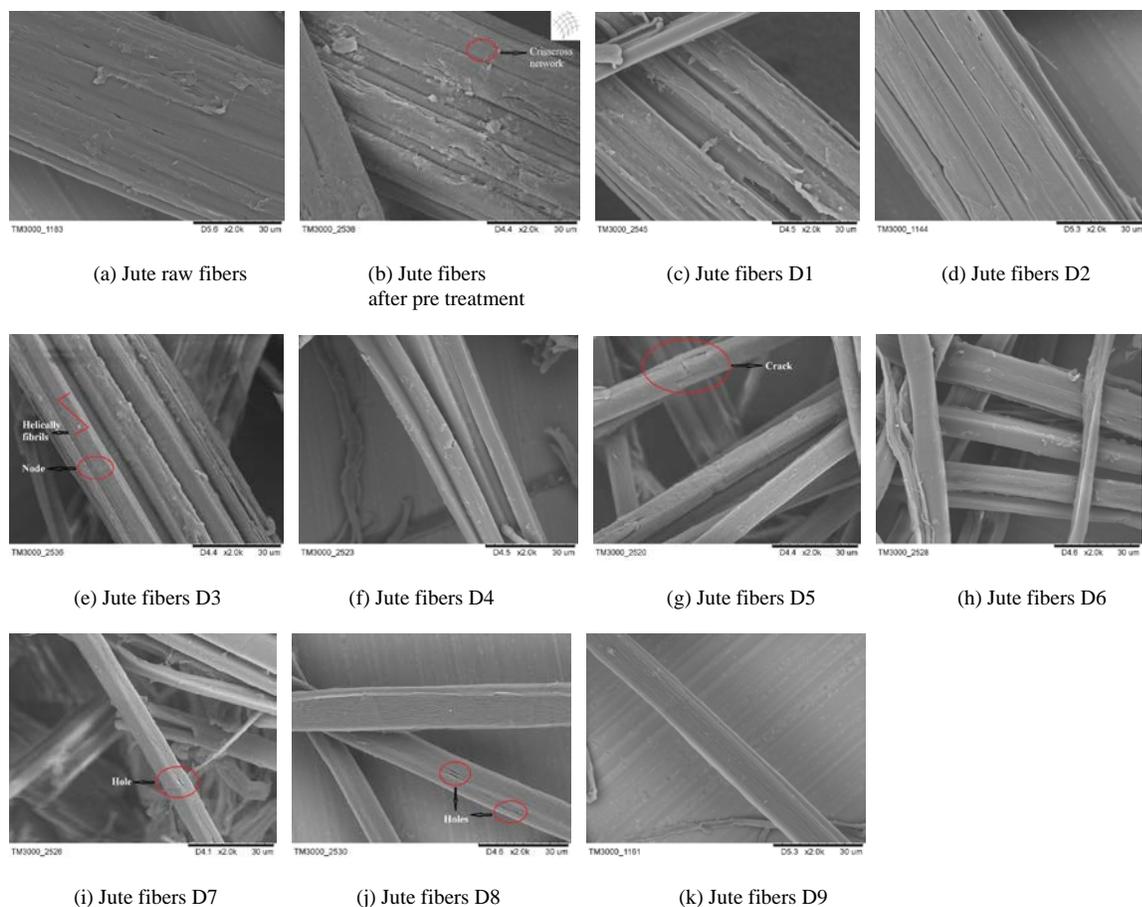


FIGURE 6. SEM images of the microstructure of untreated, pretreated and PAA treated jute fibers.

Spectroscopic Characterization

Figure 7 shows the FTIR spectrum of the jute fibers before and after peracetic acid treatments. The band at $3700\text{-}3100\text{ cm}^{-1}$, corresponds to O-H stretching vibrations of hydrogen bonded hydroxyl (-OH) group. The peak at 2900 cm^{-1} corresponds to the aliphatic saturated C-H stretching vibration in cellulose and hemicelluloses. The reduction of this peak indicates the removal of hemicellulose [32]. The peak at 1733 cm^{-1} in the jute fibers corresponds to the acetyl and uronic ester groups of the hemicelluloses or the ester linkage of the carboxylic group of the ferulic and p-coumeric acids of lignin and/or hemicelluloses [33-34]. The gradual reduction of this peak in the jute fibers is due to the removal of most hemicelluloses and lignin during the peracetic acid treatment process. The peak at 1618 cm^{-1} in the fibers is associated with absorbed water molecules in

carbohydrates. The decrease in this peak intensity in the chemically treated jute fibers is due to the partial removal of hemicelluloses [35]. The C=C stretch of aromatic ring in lignin results in peaks at 1420 cm^{-1} . The intensity of this peak decreases significantly in peracetic acid treated fibers due to partial removal of lignin. The sharp peak at 1370 cm^{-1} reflects C-H asymmetric deformations [34]. The intensity of the peak at 1250 cm^{-1} is sharply weakened after peracetic acid treatment because of removal of the hemicellulose [32]. The bands in the region $1200\text{-}1056\text{ cm}^{-1}$ represents the C-O stretching vibrations of aliphatic primary and secondary alcohols in cellulose, hemicellulose, lignin, and extracts [36]. The peak at 900 cm^{-1} is related to β -glycosidic linkages in the glucose ring of cellulose [37].

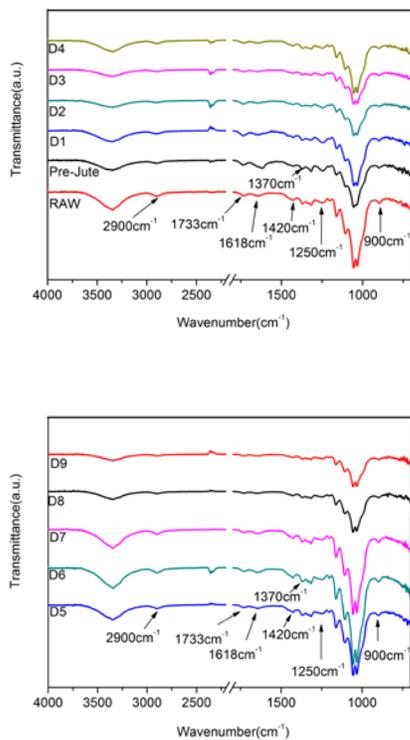


FIGURE 7. FTIR spectra of untreated, pretreated and PAA treated jute fibers.

CP/MAS ^{13}C -NMR Spectrum Analysis

CP/MAS ^{13}C -NMR spectroscopy of the jute fibers and peracetic acid treated samples are shown in Figure 8. CP/MAS ^{13}C -NMR spectroscopy has proved particularly useful for examining changes in the nature of cellulose subjected to degradation processes. The typical signals of cellulose appear at 105.00 ppm (C-1), 88.69 ppm and 83.97 ppm (C-4), 74.71 ppm and 72.04 ppm (C-2, C-3, and C-5), and 62.51 ppm and 64.88 ppm (C-6). The two peaks at 62.51 ppm and 64.88 ppm are assigned to C-6 crystalline cellulose and amorphous cellulose respectively. The cluster of resonances from 88.69 ppm and 83.97 ppm related to C-4 crystal-interior cellulose and crystal-surface cellulose, respectively. Figure 8 (a,b) indicated that the jute samples treated by peracetic acid had a similar structure to the raw jute fibers. It is apparent that there are some differences in supermolecular structure between the two samples. The accessible fibril surface (C-4, 83.97 ppm) and amorphous cellulose (C-6, 64.88 ppm) were partly damaged for sample D5 according to the spectrum [38,39].

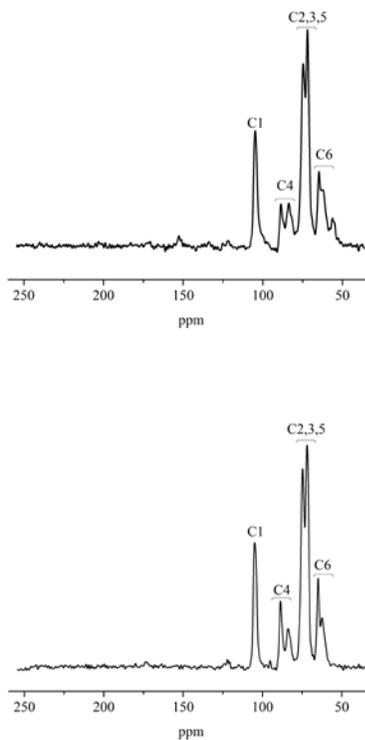


FIGURE 8. Solid-state ^{13}C -NMR spectra of (a) raw jute fibers and (b) PAA treated jute fibers D5.

XRD Analysis

The linear cellulose molecules are linked laterally by hydrogen bonds to form crystal and amorphous regions, giving rise to an elementary fibril, which aggregates into microfibrils, and then fibril structure [40]. The crystalline lattice of cellulose is monoclinic.

XRD analyses of jute fibers were done after peracetic acid treatment in order to study the crystalline behavior of the fibers and reveal the relationship between the structure and properties of the fibers. Figure 9 shows the X-Ray diffraction results for the jute fibers. It is noted that the results did not show any change in the cellulose I lattice pattern for the jute fibers. The major crystalline peak for each sample occurred at around $2\theta = 22.6^\circ$, which corresponds to the (002) lattice plane. The peak at $2\theta = 16^\circ$ corresponds to the (101) crystallographic plane, and the peak at $2\theta = 34.7^\circ$ corresponds to the (023) and (040) planes [41,42]. Figure 10 shows the crystallinity change of the jute fibers after peracetic acid treatment. The crystallinity values were

calculated to be 65.27%, 70.05%, 82.02%, and 76.61 % for the raw jute fibers, pre-jute fibers, D7 and D9, respectively. The schematic representation of the crystallinity change as illustrated in *Figure 11* [43]. The initial rapid increase in relative crystallinity is due to the efficient removal of noncellulosic materials. A slight decrease in crystallinity is noted in sample D5. This occurs because of glucose oxidation by PAA at the surfaces of cellulose crystallites, which causes random cleavage of glycosidic bonds. At the same time, the carbohydrate depolymerization and dissolution may occur with the treatment solution at low pH, so acid hydrolysis may also occur at the surface of cellulose crystallite [44].

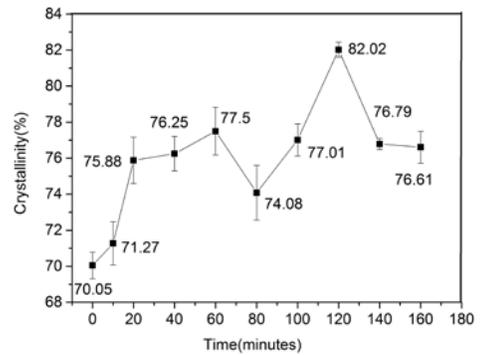


FIGURE 10. The effect of PAA immersion time on the crystallinity of jute fibers.

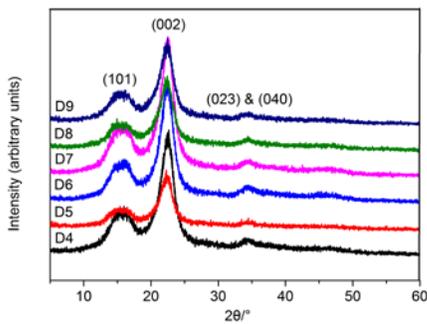
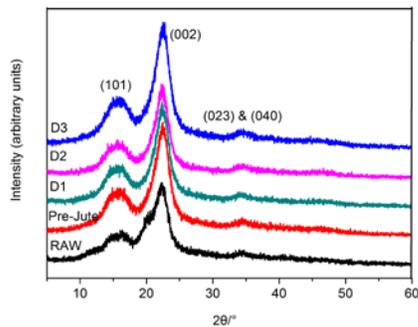


FIGURE 9. XRD curves of untreated, pretreated and PAA treated jute fibers.

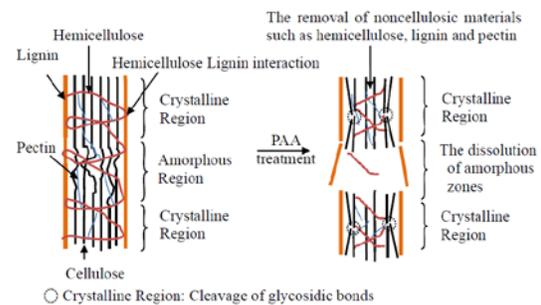


FIGURE 11. Schematic diagram of the crystallinity change.

There is a stable increase in crystallinity because of the dissolution of amorphous zones. The results also confirm that the crystalline regions are more stable than the amorphous zones during PAA treatment. When the fiber underwent chemical treatment for longer periods of time, the crystallinity began to decrease, as in the case of sample D8. This decrease may involve three aspects: (1) Glucose oxidation with PAA at the inner cellulose crystallite, and the chemical reaction of glucose oxidation with PAA as shown in *Figure 12*. (2) The weakening of the intramolecular hydrogen bonding system in cellulose due to oxygen interference. (3) Inner cellulose crystallite depolymerization and dissolution caused by acid hydrolysis when the PAA equilibrium mixture has a pH value lower than the 4 to 5 range [45]. Four stages of changes in crystallinity could be observed from this experiment. Zhao *et al.* [46] found that after alkali-PAA treatment, individual fibers with relatively higher cellulose crystallinity could be successfully obtained from sugarcane bagasse. Different technical approaches could result in different treatment effects.

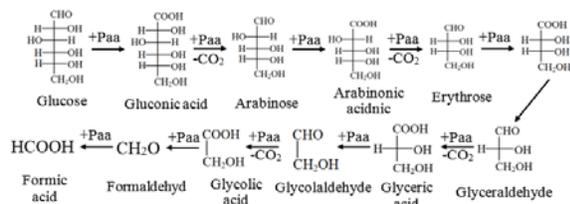


FIGURE 12. Proposed chemical reaction process of the glucose oxidation with PAA [42].

CONCLUSION

In this paper, the structural changes of jute fibers were studied in peracetic acid (PAA) solution at different treatment time. According to the SEM analysis, the outermost primary layer of the fibers was easily removed after the peracetic acid treatment, exposing the layers underneath the secondary layer. Fibrils parallel to the fiber axis with straight orientation were observed. The raw jute fibers split into ultimate cells when residual lignin and residual hemicellulose components were 3.07% and 6.03%, respectively. However, fibers were damaged when the treatment time reached 80 minutes, PAA concentration was 10 wt.% and pH value was 4~5. FTIR spectra confirmed that the lignin and hemicellulose continued to be removed as treatment time was increased. Investigation by CP/MAS ¹³C-NMR spectroscopy showed some changes in the macromolecular structure in cellulose fibers after PAA treatment. Crystallinity changes in four stages were observed during the treatment process. There was an initial crystallinity increase during the first 60 min due to the removal of noncellulosic materials. Attack at the surfaces of the cellulose crystallites and the dissolution of amorphous zones cause stages of fluctuating crystallinity. The final stage of crystallinity decline is due to glucose oxidation, oxygen interference and acid hydrolysis. PAA has great potential for wide use in industry because of excellent bleaching effects combined with compelling environmental friendly features.

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