

PREPARATION OF BAMBOO DISSOLVING PULP FOR TEXTILE PRODUCTION. PART 1. STUDY ON PREHYDROLYSIS OF GREEN BAMBOO FOR PRODUCING DISSOLVING PULP

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Prehydrolysis was used as the first treatment of bamboo materials for producing high dissolving pulp for textile applications. In this paper, green bamboo (*Dendrocalamopsis oldhami*) was hydrolyzed in water, and the hydrolysis process was optimized. The morphology and super-molecular structures of the raw material and prehydrolyzed material were studied by SEM and XRD, respectively. Based on the optimum conditions, a study of the kinetics and mechanism showed that pentosan dissolution during the hydrolysis process was a first order reaction. When cooking temperature was 150 °C, 160 °C, 170 °C, and 180 °C, the corresponding reaction rate constants were 0.00411h⁻¹, 0.00495h⁻¹, 0.00730h⁻¹, and 0.00925h⁻¹, respectively. The activation energy was 44.94 kJ/mol based on the Arrhenius empirical equation. SEM results revealed that after the pre-hydrolysis process, the bamboo structure became loose because of hemicellulose dissolution, which created a favorable environment for chemical penetration in the subsequent treatment. Furthermore, the degree of crystallinity was increased from 45.43% to 57.06% during the prehydrolysis. Both the treated and the untreated fibers were assumed to be cellulose I.

Keywords: Green bamboo; Prehydrolysis; Kinetics; SEM; XRD

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INTRODUCTION

The development of efficient, safe, and profitable technologies for lingo-cellulosic utilization materials is a common objective for many research groups (Abad and Alonso 1997). Dissolving pulp is an important starting material for production of cellulose derivatives and regenerated cellulose (Wan Rosli et al. 2004). The traditional two major resources for production of dissolving pulp are cotton linters and wood pulp. With increasing demand and cost of pulpwood, new alternatives for the production of dissolving pulp are being investigated. Non-wood raw material is one of alternatives and is popular especially in developing countries where pulpwood stocks are not sufficient. Among the non-wood materials, the morphology and chemical composition of bamboo material shows better pulp properties, which could replace typical softwood such as spruce or pine to produce high-grade dissolving pulp for use in production of textile raw materials. In addition, bamboo grows fast, and therefore, it can be considered a rapidly renewable and “sustainable” fiber resource (Kelly and Brinsko 2010; Wang 2010; Ajoy and Sarkar 2009; Yang and Zhang 2009). China is known for the world’s most abundant bamboo resources, the largest area of bamboo forest, and the largest country in bamboo production, factors that help justify the term “bamboo kingdom.” Furthermore, recently

attempts have been made to produce textile fibers from bamboo. Bamboo fibers possess many excellent properties when used as textile materials such as high tenacity, excellent thermal conductivity, resistance to bacteria, and high water and perspiration adsorption, while bamboo fiber is an environmentally friendly fiber. Consequently, research and development of bamboo fiber as new textile fiber could not only open a new way to produce cellulosic fiber, but also play a significant role in solving the wood fiber shortage problem and also improve the textile fiber production technology and product quality (Liu and Hu 2008).

However, pulps used in textile fiber production differs from those were used in paper manufacturing. Bleached pulp with high purity is required, free of hemicellulose and lignin, with strict control of ash, metal ion content, and degree of polymerization (Jahan 2009). Dissolving pulp quality depends on both the properties of the raw material and pulp processing. In order to maximize the removal of hemicellulose, lignin, and other impurities and also enhance the reaction ability of bamboo, it is critical to apply a proper process (Ibrahim et al. 1996). In most instances, prehydrolysis kraft or acid sulfite processes followed by the conventional bleaching sequences have been used to produce chemical cellulose or dissolving pulp. The introduction of prehydrolysis prior to alkaline pulping process helps to produce pulp with a satisfactorily high content of cellulose and low hemicellulose content. Prehydrolysis could also open up the cellulose fibers for further reactions, increase the extractability of lignin during the subsequent alkaline pulping process, and thus substantially reduce the lignin and ash contents (Garrote et al. 2003; Lavarack et al. 2000).

Bamboo was demonstrated to be a very good source of cellulose for producing dissolving pulp (Salmela et al. 2008); but bamboo material is difficult to penetrate by liquor during the pulping processes. The compact structure, along with the thicker cell wall, high hybrid cell content, and wax are the major reasons for the difficulty of the liquor penetration. In addition, high hemicellulose content puts the pretreatment at a more important status. Undoubtedly, a kraft pulping process modified by incorporating a pretreatment of the bamboo prior to pulping is the most effective way to solve the problems. Indeed, acid prehydrolysis kraft pulping generally has been used as an efficient way to produce dissolving pulp (Zhang et al. 2009). Although in acid media the temperature would be lowered, a higher liquor ratio required by the prehydrolysis process can be expected to cause difficulties in the actual production process. It is important for actual production that the prehydrolysis process can be compatible with the original pulping process with respect to the corrosion resistance of the equipment and the effectiveness of waste water treatment.

In our study, bamboo material was subjected to pulping in prehydrolysis kraft cooking and conventional bleaching processes. Prehydrolysis of bamboo materials was achieved at elevated temperature with low liquor ratio. In this part, the objective was to optimize the prehydrolysis condition and study the kinetics and mechanism of hemicellulose solubilization during bamboo hydrolysis. Besides, a kinetic model of dissolving of hemicellulose is presented for describing hemicellulose extraction. The model assumes first order kinetics with Arrhenius-type temperature dependence of the reaction rate constants. The model is calibrated and validated with experimental data. Furthermore, the morphology property changes and super-molecular structure of bamboo fiber were further studied by SEM and XRD analysis.

EXPERIMENTAL

Materials

Green bamboo (*Dendrocalamopsis oldhami*) was provided by one of the forestry centers in Fujian Province. Bamboo chips were screened to particles with a size of 20×20×1 mm. The chips were air-dried and homogenized in a single lot to avoid differences in composition. The chemical composition was analyzed according to TAPPI standards.

Prehydrolysis Process

The prehydrolysis was carried out in an electrically heated stainless steel digester of 15L, rotating at 1 rpm. Water prehydrolysis was carried out at elevated temperature (150 to 180 °C) with the liquor ratio range from 1:2 to 1:5, cook time up to 120 min, and the heating rate range was 5 to 12 °C /10min. Subsequently, the optimum condition was determined according to the series of the experiments.

Kinetic Mechanism of Prehydrolysis

The study of the kinetic mechanism of prehydrolysis was carried out in a YYQ-10×1.25 oil bath cooking pot, in which there were 10 small sets. All bamboo materials tested were air dried and stored in a desiccator. In this section, the sample of bamboo 100 g was placed in one of the sets, and the hydrolysis conditions were selected from the former (prehydrolysis process) experiments. Moreover, during the entire hydrolysis, the concentration of substrate (measured by the liquid/solid ratio) was considered as constant. At given reaction conditions, the sets were taken from the reaction media and the reaction was ended by immersion in cold water. Then the samples were washed, air dried, and ground for testing.

Chemical Composition of Bamboo

Standard Methods of the Technical Association of the Pulp and Paper Industry (TAPPI, Atlanta, GA) were used to determine the chemical composition of the samples. Cold water extractives (T207cm-08), hot water extractives (T207cm-08), 1% NaOH extractives (T212om-07), solvent extractives (T204cm-07), acid-insoluble lignin (T222om-06), pentosan (T223cm-01), ash (T244.cm-99) were determined. Cellulose content was determined by the nitric acid-ethanol method.

Fiber Morphology

The morphology of the green bamboo was analyzed on the Morfi Compact fiber analyzer.

Scanning Electron Microscope (SEM) Analysis

An SEM-5310LV JEOL scanner was used to investigate the surface morphology of the bamboo.

Analysis by X-Ray Diffraction

X-ray diffraction (XRD) was used to investigate the supermolecular structure of bamboo fiber. X-ray diffraction data were obtained using a Japan MiniFlex2 instrument, and the samples were prepared as powder. Ni-filtered Cu-K α radiation generated at a

voltage of 30kV and current of 15mA was utilized. The scanning angle range was 5° to 45° with a scan speed of 5°/min. To calculate the crystallinity index, MDI Jade Version 5.0 software was applied.

RESULTS AND DISCUSSION

Raw Material Composition and Morphology of Bamboo Fibers

The chemical composition of the bamboo is listed on Table 1. (based on average values of three replicates, error lower than 1% in all compounds, weight percent on dry basis):

Table 1. Chemical Composition of Bamboo Material

Extractives/%				Ash /%	Pentosan /%	Cellulose /%	Lignin /%
Cold water	Hot water	1% NaOH	Organic solvent				
4.45	6.75	26.91	5.65%	1.78	17.45	49.60	23.12

In general, non-woods differ somewhat from woods in their chemical composition and properties, which in turn have a direct influence on the pulping and bleaching processes. From the above data, in this respect, bamboo is a clear exception, since its lignin and hemicelluloses contents are similar to those in wood.

Additionally, bamboo is a kind of monocot grass with slender, spindle shape. The average fiber length is generally 1.5-2.5 mm, which is shorter than cotton fiber and softwood fiber, but close to the length of hardwood fiber (Jackson 1988). Green bamboo fibers are normally more than 1 mm in length, with an average length of 2.19 mm. The fiber width varied from about 6 to 25 μm , with an average of about 13.83 μm , giving a ratio of length to diameter of 158.26. The thickness of the cell wall was more than 3 μm , with an average of 6.16 μm , while the lumen diameter was included in the range of 1.8 to 5.7 μm , with an average of 3.49, giving a ratio of thickness to diameter of 1.76. Its close structural resemblance to softwoods is also the main reason why bamboo materials could be used as raw material in textile field. Besides, bamboo is an attractive long fibered material for chemical pulp, and it has strength properties comparable to softwood pulp (Salmela et al. 2008). Unfortunately, the thicker cell wall of bamboo would cause some difficulties in cooking liquor penetration.

Optimization of Prehydrolysis Process

To determine the optimum prehydrolysis conditions for maximum removal of the hemicellulose, various experiments were carried out as shown in Tables 2 through 5. Behin's study showed that extraction of hemicellulose can be readily accomplished to a great extent by water pre-hydrolysis (Behin and Zeyghami 2009). Wan Rosli also noted that, contrary to wood-based materials, prehydrolyzing of empty fruit bunches in the absence of acid was most effective (Wan Rosli. et al 2004). Furthermore, under the mild conditions, which involved no addition of mineral acid in the reaction media, acetic acid was liberated from the xylan of the bamboo materials in a sufficient amount and enabled the formation of the necessary acidity to catalyze the hydrolysis and thus the dissolution of a great part of the hemicelluloses. Thus, water prehydrolysis resulted in low

environmental impact, and adverse reactions leading to cellulose decomposition and lignin repolymerization were largely avoided. That was to say, the degradation of cellulose was controlled in order to preserve the degree of polymerization of cellulose (Garrote et al. 2001; Wan Rosli et al. 2003).

Table 2. Effects of Liquor Ratio on Prehydrolysis

Liquor ratio	Yield /%	pH	Cellulose/%	Pentosan/%
1:2	87.83	3.45	53.68	13.21
1:3	85.24	3.23	58.34	8.58
1:4	86.34	3.54	50.96	16.24
1:5	86.78	3.60	50.78	16.40

Heating rate: 7 °C/10 min; cooking time: 60 min; cooking temperature: 170 °C

Table 3. Effects of Heating Rate on Prehydrolysis

Heating rate/ °C/10min	Yield /%	pH	Cellulose/%	Pentosan/%
5	79.62	2.98	60.30	8.95
7	81.43	3.15	58.75	10.36
10	84.76	3.31	55.52	11.70
12	84.88	3.49	50.73	12.53

Liquor ratio: 1:3; cooking time: 60min; cooking temperature: 170 °C

Table 4. Effects of Cooking Temperature on Prehydrolysis

Cooking temperature/ °C	Yield /%	pH	Cellulose/%	Pentosan/%
150	94.06	4.06	53.55	16.82
160	90.38	3.78	54.73	15.09
170	83.87	3.50	57.00	8.58
180	81.15	3.25	57.56	8.25

Liquor ratio: 1:3; heating rate: 7 °C/10 min; cooking time: 60 min

Table 5. Effects of Different Cooking Time on Prehydrolysis

Cooking time/min	Yield /%	pH	Cellulose/%	Pentosan/%
30	87.14	3.68	50.41	16.20
60	85.59	3.40	51.02	12.77
90	76.55	3.24	53.71	9.88
120	75.78	3.16	54.94	6.12

Liquor ratio: 1:3; heating rate: 7 °C/10 min; cooking temperature: 170°C

Table 6. Results of Bamboo Prehydrolysis Under the Optimum Conditions

pH	Yield /%	Pentosan /%	Cellulose/%	Lignin/%
2.98	79.62	8.95	60.30	18.06

Liquor ratio: 1:3; heating rate: 5 °C/10 min; cooking temperature: 170 °C; cooking time: 60 min

Under the following conditions (heating rate: 7 °C /10min; cooking time: 60 min; cooking temperature: 170 °C), the cellulose content was increased first and then decreased when more water was added, and the pentosan content was first decreased and

then increased (Table 2). Typically, pentosan content was the most important criterion in determining the prehydrolysis process. As the prehydrolysis proceeded, it could also produce a significant level of cellulose degradation if the process conditions were not well selected; therefore, cellulose content was also used to determine the prehydrolysis process.

A high liquor ratio resulted in more energy and waste effluent, whereas a low liquor ratio would cause non-uniform penetration, thus resulting in incompletely reaction. A liquor ratio of 1:3 was selected as a suitable condition for the lowest pentosan content and highest cellulose content. In water prehydrolysis, the organic acid (acetic acid) liberated from the fiber by the natural deacetylation of polysaccharides lowered the pH below 4 (Neogi et al. 2001). Elevating of the heating rate in prehydrolysis decreased the cellulose content and increased the pentosan content in the pulp (Table 3). A high heating rate was unfavourable for uniform penetration and reaction because of the difficulty in penetration, while a low heating rate could extend the penetration time. The heating rate 5 °C /10 min was judged to be feasible because of the lowest pentosan content. The cooking time and cooking temperature were beneficial for removal of the hemicelluloses; thus, elevating the temperature and extending time could accelerate the removal of hemicelluloses. It is obvious from Table 4 and 5 that when drastic conditions were used, the yield and pentosan content was dramatically decreased and the cellulose content was increased. However, the difference of pentosan content between 170 °C and 180 °C was negligible, and 170 °C was selected as the best when energy consumption and lignin condensation was taken into account at higher temperature. Previous studies also showed the cooking temperature should not exceed 180 °C.

As for cooking time, a low heating rate and proper cooking time would ensure the liquor penetration and removal of appropriate hemicelluloses. Although extending the cooking time would enable more hemicellulose to separate from the bamboo materials, energy consumption was considerable and another unexpected result was the condensation of lignin, which must be avoided in the process. Otherwise, condensation reactions were supposed to be responsible for an observed re-increase in resistance to delignification during prehydrolysis and subsequent kraft pulping process when long hydrolysis treatments were conducted (Leschinsky et al. 2008). Furthermore, the soluble hemicellulose remaining in the bamboo material could be easily removed in the subsequent kraft pulping process. When considering all the conditions and results together, a cooking time of 60 min was reasonable. The optimal prehydrolysis conditions were as follows: liquor ratio: 1:3, heating rate: 5 °C/10min, cooking time: 60 min, and cooking temperature: 170 °C. Results corresponding to the optimum conditions are shown in Table 6. In the prehydrolysis process, apart from hydrolyzing about 50% of the hemicellulose molecules into soluble sugars or oligomers, also partially depolymerizing the lignin (23.12% vs 18.06%), presumably via homolytic cleavage of the β -O-4 ether bond, resulted in an increase of lignin dissolution during the subsequent alkaline pulping and produced pulp with relatively low kappa numbers (Leh et al. 2008). The optimum prehydrolysis conditions were used in the subsequent analysis of the kinetic mechanism.

Kinetic Model of Prehydrolysis

In the pre-hydrolysis process, the hydrolysis or degradation of raw material components occurred, and the part of hydrolysate was dissolved in the solution. Gray and

Casebier studied the pre-hydrolysis process of southern pine and black Bakelite and found that higher temperature led to a quicker reaction and more dissolved components (Gray and Casebier 1983). Cooking time also had a promoting effect on the reaction, and the dissolved product yield was enhanced when the temperature was increased. It was further shown that time and cooking temperature had a great impact on the pre-hydrolysis reaction (Starr et al 1976).

The main objective of this part of the research was to establish a reaction scheme leading to useful kinetic equations to describe the experimental trends. The operational conditions of the sets were fixed based on the optimum prehydrolysis conditions. Unlike in the previous experiments, the bamboo samples were treated at 150 °C, 160 °C, 170 °C, and 180 °C, respectively. The pentosan content remaining in the solid residue at different cooking temperatures is shown on Table 7.

Table 7. Pentosan Content of Bamboo with Different Cooking Temperature

Time /min	Pentosan content p/%			
	150 °C	160 °C	170 °C	180 °C
10	23.34	20.91	17.26	15.88
20	23.05	19.91	16.88	15.80
30	20.87	19.04	15.93	13.54
40	20.35	16.75	14.73	12.99
50	19.69	16.29	13.88	12.70
60	18.76	15.28	13.06	10.98
70	17.88	15.12	11.64	9.89
80	17.36	14.52	10.33	8.64
90	16.85	13.94	9.85	8.24
100	16.29	13.50	9.54	6.90

The rate of reaction leading to a reduction of pentosan content can be expressed as:

$$r = -\frac{dp}{dt} = kp \quad (1)$$

The kinetic model represented by Eq. 2 was obtained from integration of Eq. 1,

$$\ln p = \ln p_0 - kt \quad (2)$$

where $-dp/dt$ denotes the rate of pentosan dissolution; k is a constant expressing the rate of reaction h^{-1} ; p_0 means the initial pentosan content; p means pentosan remaining in the solid residue; and t denotes time.

Results are plotted in Fig. 1 and the linear fit results are shown on Table 8, which is based on the use of Eq. 2. As can be seen from Table 8, the correlation coefficients of the curves were close to 1, which indicated that the linear correlation between fitting curves and experimental value was good under each cooking temperature. The results implied that the hydrolysis of bamboo was a first-order reaction.

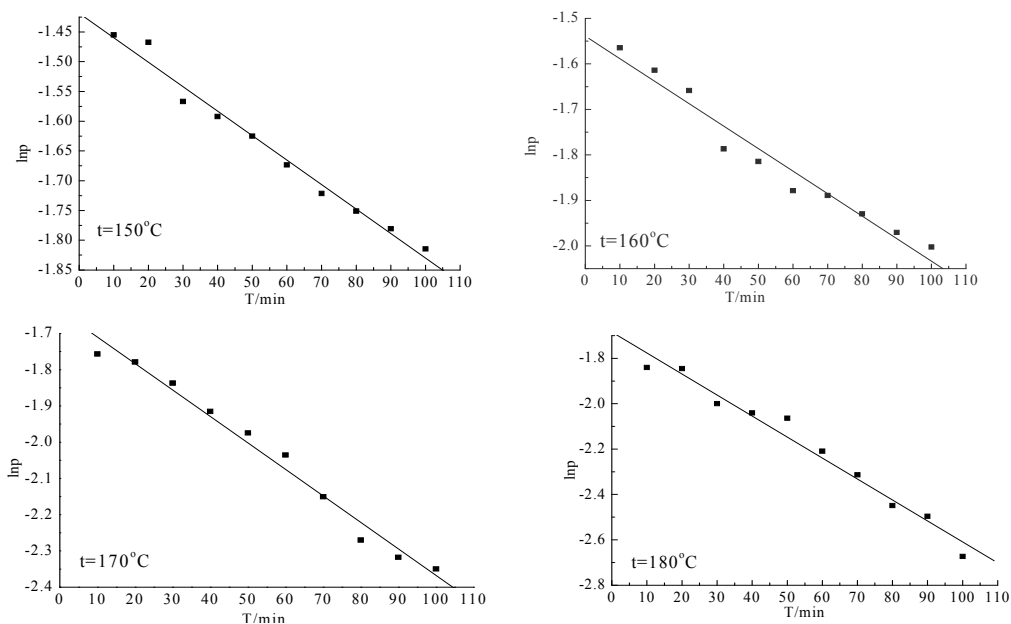


Fig. 1. The $\ln p$ - T diagram of pentosan stripping with different cooking temperature

Table 8. Fitting Results and Error Analysis

Temperature/ °C	k/ h^{-1}	$\ln p_0$	Correlation coefficient	Standard deviation
150	0.00411	-1.41857	-0.99139	0.01745
160	0.00495	-1.53891	-0.98007	0.03219
170	0.00730	-1.63734	-0.99065	0.03227
180	0.00925	-1.68413	-0.98647	0.04938

The reaction constant k shown in Table 8 was calculated using a least-squares method, showing differences with respect to temperature, generally increasing with an increase of temperature. Furthermore, activation energy was calculated according Arrhenius Eq. 3 using a least-squares method, (k constant expressing the rate of reaction h^{-1} , E_a activation energy, R gas constant $8.32 \text{ J/mol}\cdot\text{K}$, T temperature, A frequency factor).

$$\ln k = \ln A - E_a / RT \quad (3)$$

The fitting results and errors are shown in Table 9.

Table 9. Fitting Results and Error Analysis

k/ h^{-1}	T/K	$-E_a/R$	$E_a/kJ.mol^{-1}$	Correlation coefficient	Standard deviation
0.00411	423.15	-5405.04	44.94	-0.99036	0.06236
0.00495	433.15				
0.00730	443.15				
0.00925	453.15				

The activation energy of prehydrolysis of green bamboo was found to be 44.94 kJ.mol⁻¹. This represents the minimum energy required to start the hydrolysis reaction. The activation energy of green bamboo hydrolysis obtained in the present work may be compared with other bamboos and provide data for bamboo research; moreover, it could support the determination of the conditions used for the subsequent pulping process.

SEM and XRD Analysis

Cross-sectional SEM images of green bamboo are shown in Fig. 2. It can be seen that the vascular elements of the original bamboo in the direction of the cross-section were compact and smooth, and they were more regular than the bamboo after prehydrolysis. The cell wall became loose, subsequently, and the diameter of the vascular space was enlarged, making it easier for chemicals to penetrate after prehydrolysis. Also, it was implied that the primary wall of the bamboo was destroyed by prehydrolysis, and the linkages between lignin and carbohydrates were diminished. The dissolution of hemicellulose also created some holes in the material, which would result in efficient treatment in subsequent processes such as kraft pulping.

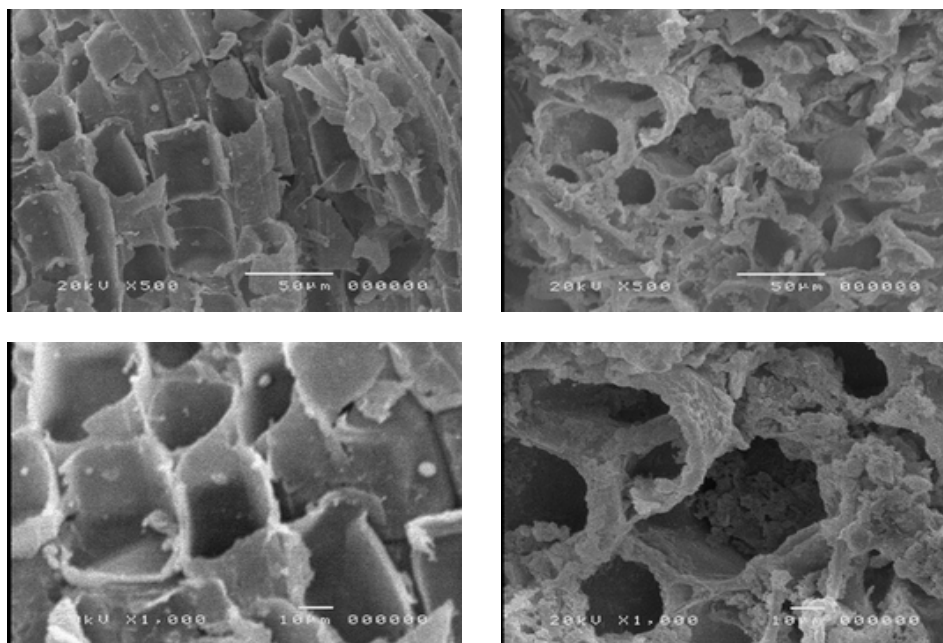


Fig. 2. SEM images of bamboo cross-section. Left: original; Right: prehydrolysis; Upper 500 times magnification; Lower: 1000 times

The diffraction peaks of pure cellulose fiber (Cellulose I) appear at 2 θ ~14.8°, 16.6°, and 22.7°, and for Cellulose II they appear at 2 θ ~12.3°, 20.2°, and 21.9°, which are in accordance with the reference data. Figure 3 shows patterns of the two types of fibers before and after hydrolysis. Sharp high peaks at 2 θ equal to 22.1° and 22.3°, respectively, and weaker peaks at 16.2° and 16.0° were assigned to cellulose I. Based on these results it was apparent that prehydrolysis treatment could not transform the molecular aggregation structure of the bamboo fiber. The intensity of the diffraction peak for cellulose increased after pre-hydrolysis; the calculated degree of crystallinity of pre-hydrolysis bamboo fibers was 57.06%, in contrast to 45.43% for the original fiber.

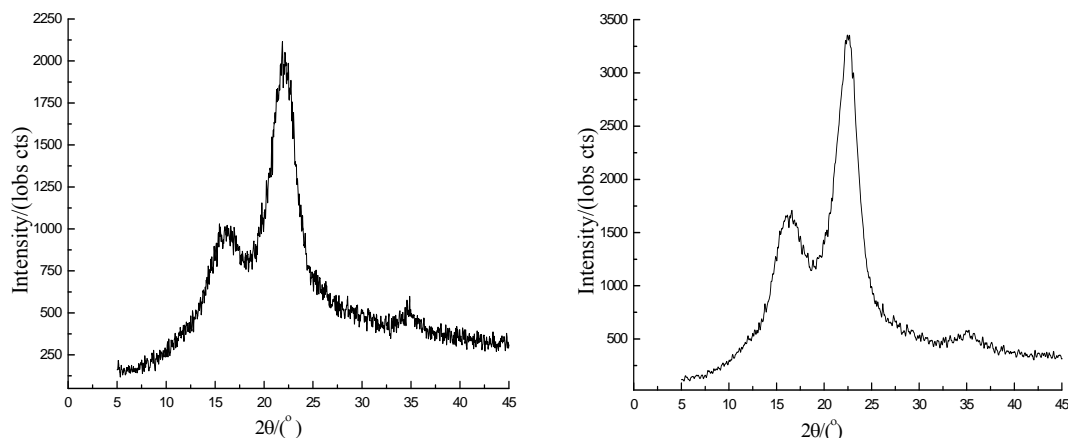


Fig. 3. X-ray diffraction pattern of Green bamboo fiber. Left: original; Right: prehydrolysis

It is well known that the cellulosic fibers undergo three distinct processes during mercerization: fiber swelling, disruption of the crystalline areas, and formation of new crystalline lattice after rinsing away mercerizing solution. Only the amorphous regions and crystal surfaces in the cellulose structure, that is, the cementing materials, can readily react with reagent and can be removed (Liu and Hu 2008). In this study, it was clearly evident from Fig. 4 that the crystallinity of the fiber increased following water treatment. From this, it is understandable that the crystalline cellulose content was increased, whereas the amorphous hemicellulose and lignin content decreased during the treatment (Venkata 2010; Zhao et al. 2007). It was revealed that the hemicellulose, cellulose, and lignin in the amorphous region were degraded and dissolved in the water during pre-hydrolysis. In this case, the interfibrillar regions were likely to be less dense and less rigid, making the fibrils more capable of rearranging by themselves. Consequently, the crystallinity index of fibers increased after prehydrolysis.

CONCLUSIONS

Based on the results of this work it appears that the best conditions for hydrolyzing green bamboo materials would be as follows: liquor ratio: 1:3, heating rate: 5°C /10 min, cooking temperature: 170 °C, cooking time: 60 min. During prehydrolysis about half of the hemicellulose was solubilized. A kinetic mechanistic study indicated that prehydrolysis was a first-order reaction. The reaction rate constants were 0.00411 h⁻¹, 0.00495 h⁻¹, 0.00730 h⁻¹, and 0.00925 h⁻¹, corresponding to the temperatures 150 °C, 160 °C, 170 °C, and 180 °C. The activation energy of prehydrolysis was 44.94 kJ/mol. SEM results revealed that, after the pre-hydrolysis process, the bamboo structure had become loosened as a result of hemicellulose dissolution, creating a favorable environment for cooking liquor to penetrate. Also, XRD results revealed that the degree of crystallinity increased from 45.43% to 57.06% after prehydrolysis.

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