

Dilute Acid Hydrolysis of Jute Bast Fiber for Selected Xylan Removal

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ABSTRACT

Jute fiber was proposed as an alternative raw material for dissolving pulp production. Jute fiber had cellulose ($71.8\% \pm 0.1\%$), xylan ($10.8\% \pm 0.1\%$), lignin ($13.7\% \pm 0.2\%$), and extractives ($3.8\% \pm 0.0\%$). In this study, jute fiber was hydrolyzed with H_2SO_4 to remove xylan. To achieve the best conditions for removing hemicellulose and obtaining a high cellulose content, acid hydrolysis pretreatment was performed at various acid concentrations (0.25–1 N), reaction times (60–120 min), and temperatures ($100^\circ C$ – $120^\circ C$). The results showed that the optimum condition of acid hydrolysis for higher dissolving pulp raw material yield within less than 5.0% xylan was cellulose (60.5 ± 0.2) and xylan (2.9 ± 0.2) at 1 N H_2SO_4 , $100^\circ C$ for 90 min. However, by increasing the reaction temperature to $120^\circ C$ for 120 min, the chemical used was reduced to 0.25 N H_2SO_4 and the highest purity of cellulose was produced, with cellulose and xylan content of $56.1\% \pm 0.1\%$ and $1.3\% \pm 0.1\%$, respectively.

Keywords: Jute, acid hydrolysis, cellulose, xylan, lignin.

1. Introduction

The production of high-purity cellulosic fibers, also known as dissolving pulp, has been gained significant interest over the past decade owing to the strong growth of market demand. [1–3] The technologies for producing this pulp have also been

developed. [4,5] Dissolving pulp contains high alpha-cellulose (more than 90%), high brightness with low extractives, hemicelluloses, and ash content. [6–8] Dissolving pulp is an important starting material for manufacturing rayon fibers and various cellulose-related materials, such as viscose, lyocell, cellulose acetate, nitrocellulose, car-

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boxymethyl cellulose, and cellulose ether. [2,4,9,10] These materials are commonly applied in various areas for food, pharmaceutical, and industrial products (such as textile, coatings, tobacco, and paints). [1,7,11–13]

Traditionally, cotton linters and wood pulp are the main raw materials for dissolving pulp production. [1] However, the availability of cotton linters and wood pulp was limited and the price was high. On the other hand, a stable supply of raw materials is an important factor in achieving an ecosystem capable of sustainable reproduction. Therefore, the alternative raw material for dissolving pulp production that can be continuously developed is needed. The utilization of non-wood materials such as bamboo, [7–14] bagasse, [15] jute, [16] and corn stalk [17] can be an alternative to overcome the problem. Among the non-wood materials, jute fiber has good advantages for chemical pulp production due to its high cellulose content and low lignin content. [18] In addition, jute is a fast-growing plant, so it can be used as a renewable and sustainable source of fiber. [19]

Several studies have recently been carried out to produce dissolving pulp from jute including jute fibers, jute cuttings, caddis, and sticks. [16,20] For example, dissolving pulp produced from jute fiber using 90% formic acid and reacted at 107°C for 4 h showed high pulp yield (57.6 to 63.5%) and high α -cellulose content (96 to 97%) after bleaching. [21] Research and development of jute fibers as raw materials to produce dissolving pulp could play a significant role in overcoming wood fiber deficiency and improving the production of cellulosic products technology and quality of products.

Dissolving pulp quality depends on the raw material and pulp-making process. [4] The raw materials quality can be checked by analyzing fiber dimensions and chemical components such as α -cellulose content. [5,14] The pulp-making process including the preparation of raw material,

pre-hydrolysis, cooking, and bleaching, also affects the quality of dissolving pulp. [7]

Dissolving pulp is commonly produced by two methods (pre-hydrolysis kraft pulp and/or acid sulfite) followed by conventional bleaching. Pre-hydrolysis kraft pulp and acid sulfite resulted in dissolving pulp with α -cellulose content of 96% and 93%, respectively. [22] These hydrolyses are an effective method in removing hemicellulose. [23] Furthermore, hemicellulose and lignin could also be removed simultaneously by using acid sulfite hydrolysis. Therefore, we are focused on developing the optimum acid hydrolysis condition for jute fiber as raw materials for xyolan removal to obtain high cellulose content effectively. In particular, the effect of acid concentration, temperature, and reaction time on the acid hydrolysis pretreatment was also studied.

2. Materials and Methods

2.1 Materials

The material used in this study was dried jute provided by Korean Textile Machinery Convergence Research Institute from Bangladesh. Chemical used for bleaching was sodium hypochlorite (NaClO) purchased from Sungju Industrial, Korea. Acetone from Samchun Pure Chemical Co., Ltd., Korea was used for acetone extraction. Sulfuric acid (H_2SO_4) from Samchun Pure Chemical Co., Ltd. Korea was used for acid hydrolysis and lignin determination. Deuterium oxide (D_2O) from Sigma-Aldrich, U.S. and α -L(+)-rhamnose monohydrate from Wako Pure Chemical Industries, Ltd. Japan, were used for NMR analysis.

2.2 Bleaching

Before acid hydrolysis treatment, dried jute was cut and ground into small size using an electric blender, then treated by bleaching. Bleaching was

performed two times by soaking 100 g of sample in 100 g of 4% NaClO solution for 18 h. After bleaching, the sample was washed and dried in the oven.

2.3 Acid hydrolysis

Acid hydrolysis was done by using various concentrations of H_2SO_4 to remove xylan. Briefly, 3.0 g of bleached jute was subjected to 30 mL of different H_2SO_4 concentrations (0.25, 0.5, 1 N) and reacted in the autoclave at various conditions of temperature (100, 110, 120°C) and time (60, 90, 120 min). The samples were then filtered using a glass filter (P4 type), washed several times with distilled water to remove the acid, and dried at 80°C for 24 h. All treatment conditions were performed 3 times.

2.4 Analysis of chemical constituents

The composition of jute was analyzed for cellulose, xylan, and lignin content following the methods described below. All the experiments were performed 3 times and the results were presented as mean \pm standard deviation.

2.4.1 Determination of acetone and water extractives

Semi-bleached and unbleached jute extractives were carried out using acetone following by the boiling water extraction. First extraction, about 5 g of each sample was macerated with 50 mL acetone and let at room temperature for 4 h. The sample was then filtered using filter paper to separate filtrate and residue. The filtrate was dried to evaporate acetone and the extractive was weighed. The residue was also dried at room temperature for further boiling water extraction. Second extraction, about 3 g sample was soaked in distilled water and placed in an autoclave at 100°C for 1 h. Then, the sample was filtered using filter paper and dried. Acetone and water extractives were calculated by following formula 1:

$$\text{Extractives (\%)} = \frac{(A-B)}{A} \times 100$$

A: dry weight of the sample before extraction (g)

B: dry weight of the sample after extraction (g)

2.4.2 Determination of polysaccharide composition

Dried jute was analyzed using a Bruker AVANCE NMR spectrometer (500 MHz) to determine the polysaccharide composition in different acid hydrolysis treatment conditions. The sample was prepared by diluting 0.04 g jute with 72% H_2SO_4 and reacted at 30°C for 1 h. Then, 3 mL of D_2O was added to the reaction and incubated at 100°C for 1 h. The solution was filtered using filter paper and put into an NMR tube for NMR analysis. α -L(+)-rhamnose monohydrate was used to determine the sugar peaks.

2.4.3 Determination of lignin content

Briefly, a 0.275 g sample was digested with 4.5 mL of 72% H_2SO_4 and placed in a shaking incubator at 30°C for 1 h (first hydrolysis). Then, 145 mL of distilled water was added into the diluted sample and placed in the autoclave for 1 h at the temperature of 120°C (second hydrolysis). The sample was filtered using a P4 glass filter and washed several times with hot water. Finally, it was dried at 103°C for 24 h and weighed. Lignin content was calculated by using the following formula 2:

$$\text{Lignin content (\%)} = \frac{(A-B)}{A} \times 100$$

A: dry weight of the sample before hydrolysis (g)

B: dry weight of the sample after hydrolysis (g)

3. Results and Discussion

3.1 Analysis of the chemical composition of jute fiber

The chemical composition of raw jute fiber and

Table 1. Chemical composition of jute before acid hydrolysis

Samples	Yield (%)	Chemical composition of jute (%)				
		Cellulose	Xylan	Lignin	Extractives	
					Acetone	Hot water
Raw jute	100	71.8±0.1	10.8±0.1	13.7±0.2	0.1±0.0	3.7±0.0
Semi-bleached jute	91.9±0.0	68.1±0.0	10.0±0.0	9.9±0.2	0.0±0.0	3.9±0.1

semi-bleached jute were presented in Table 1. Jute fiber was found to contain cellulose (71.8%), xylan (10.8%), lignin (13.7%), and extractives (3.8%). The presence of 82.6% total carbohydrates makes jute fiber potential raw materials for dissolving pulp. The chemical composition of jute fiber is widely studied in the literature. As expected, jute fiber's main component was cellulose, followed by lignin, hemicellulose, and extractives, respectively. It was reported jute fiber from Bengal containing 65.4% cellulose, 17.6% hemicelluloses, 14.35% lignin, 1.86% fats, 0.61% waxes, and 0.13% pectin. [24] The variation in the chemical composition might be due to differences in geographical and climatic conditions. [25] For example, jute fiber obtained from China had 64% cellulose, 19% hemicellulose, 16% lignin, and extractives. [18]

The non-cellulosic part of the material such as hemicelluloses, lignin, pectin, waxes, oils, and other impurities in fibers can be reduced by bleaching treatment in order to improve the cellulose purity. [26] Effect of bleaching treatment on jute fiber was presented in Table 1. When bleaching was performed, about 0.8% point of xylan was fractionated, and 3.8% point of lignin disappear. However, cellulose content was also decreased by about 3.7% point.

The extractives removed with different solvents from jute are different of content. As shown in Table 1, hydrophilic extractives (as hot-water extraction, 3.7%) was more than hydrophobic extractives (as acetone extraction, 0.1%). This observation was similar to jute fiber from Spain in which hot water extraction showed higher extractives content than acetone. [27] It was

reported that the water-soluble substances in lignocellulosic material are tannins, pigments, alkaloids, carbohydrates, and some inorganic salts. [28] Otherwise, acetone extractives are mainly composed of fats, waxes, tannins, etc. Furthermore, the most lipophilic compounds present in jute fibers were waxes, free fatty acids, free fatty alcohol, and α -hydroxy fatty acids. [27] It also reported that extractive from jute fiber treated by dichloromethane was higher in content (0.52%) than acetone extractive obtained in this study (0.1%). [29]

3.2 Prehydrolysis for jute bast fiber for hemicelluloses removal

Jute fiber was pre-hydrolyzed with different acid concentration, time, and temperature to obtain maximum cellulose and less xylan content. Reaction temperature, time, and acid concentration are the key parameters that affect the sugars' release and their degradation. [30] Tables 2–4 showed that the solid yield decreased continuously from 85.9 to 62.0% with increasing temperature, time, and acid concentration, attributed to xylan removal. Cellulose content was also reduced along with xylan loss. This observation was similar with water hyacinth biomass in which an increase in the acid hydrolysis conditions lowering the yield (mass) and total reducing sugars increased as well. [31] It was also reported biomass treated with phosphoric acid and maleic acid resulted in greater yield than sulphuric acid. Furthermore, fermentative inhibitors, such as 5-HMF, furfural, formic acid, and acetic acid could also produce when treated fibers under certain acid hydrolysis conditions. [30]

Table 2. Chemical composition of jute fiber with different acid hydrolysis conditions at 100°C

Acid conc. (N)	Time (min)	Yield (%)	Chemical constituents (%)		
			Cellulose	Xylan	Lignin
0.25	60	85.9±0.2	65.2±0.7	9.2±0.7	11.6±0.2
0.50	60	81.6±0.2	63.1±0.1	7.1±0.1	11.4±0.2
1.00	60	76.2±0.2	61.4±0.3	4.2±0.3	10.7±0.7
0.25	90	84.6±0.2	64.0±0.3	8.6±0.3	12.0±0.2
0.50	90	79.8±0.8	61.8±0.2	6.7±0.2	11.3±0.1
1.00	90	74.0±0.6	60.5±0.2	2.9±0.2	10.7±0.2
0.25	120	83.7±0.4	63.0±0.2	7.5±0.2	13.2±0.1
0.50	120	77.4±0.5	60.1±0.3	4.6±0.3	12.7±0.5
1.00	120	72.8±0.1	58.1±0.1	2.4±0.1	12.3±0.2

Acid con., Acid concentration

Table 3. Chemical composition of jute fiber with different acid hydrolysis conditions at 110 °C

Acid conc. (N)	Time (min)	Yield (%)	Chemical constituents (%)		
			Cellulose	Xylan	Lignin
0.25	60	77.1±0.2	60.5±0.1	6.2±0.1	10.4±0.4
0.50	60	70.9±0.2	57.9±0.0	3.3±0.0	9.8±0.0
1.00	60	67.6±0.1	55.7±0.1	1.9±0.1	10.1±0.5
0.25	90	76.8±0.6	60.4±0.0	6.1±0.0	10.3±0.2
0.50	90	69.9±0.6	56.2±0.1	2.7±0.1	11.0±0.3
1.00	90	67.4±0.6	55.8±0.1	1.8±0.1	9.8±0.1
0.25	120	75.8±0.7	60.1±0.1	5.4±0.1	10.2±0.1
0.50	120	69.6±0.1	57.6±0.0	2.1±0.0	9.9±0.2
1.00	120	67.3±0.7	54.7±0.0	1.7±0.0	10.9±0.2

Acid con., Acid concentration

Table 4. Chemical composition of jute fiber with different acid hydrolysis conditions at 120°C

Acid conc. (N)	Time (min)	Yield (%)	Chemical constituents (%)		
			Cellulose	Xylan	Lignin
0.25	60	68.3±0.3	56.5±0.0	3.1±0.0	8.7±0.4
0.50	60	64.8±0.8	55.1±0.1	1.5±0.1	8.2±0.5
1.00	60	62.8±0.9	53.0±0.3	1.0±0.3	8.8±0.8
0.25	90	67.2±0.9	56.1±0.0	2.0±0.0	9.0±0.1
0.50	90	63.9±0.8	54.4±0.0	0.9±0.0	8.6±0.4
1.00	90	62.6±1.2	52.4±0.1	0.7±0.1	9.4±0.3
0.25	120	66.8±0.9	56.1±0.1	1.3±0.1	9.4±0.3
0.50	120	63.2±1.2	53.1±0.1	0.8±0.1	9.3±0.2
1.00	120	62.0±1.4	52.5±0.1	0.6±0.1	8.9±0.2

Acid con., Acid concentration

3.2.1 Effect of acid concentrations

Acid hydrolysis pretreatment of bleached jute was carried out using various concentrations of sulfuric acid ranging from 0.25 to 1 N. Ratio of solid and liquid was 1:15 (w/v). As expected, increasing the acid concentration increased the ability of chemicals to remove xylan. However, yield and cellulose content was also reduced with increasing acid concentration, as seen in Table 2. As a catalyst, sulfuric acid acted to breakdown the glycosidic linkages present in polymeric carbohydrates. [30] As evident, cellulose content decreased from 65.2 to 61.4%, and xylan was also reduced from 9.2 to 4.2% as sulfuric acid concentration increased (0.25–1 N). A similar effect was also observed: jute fiber treated by acid hydrolysis promoted xylan's release and changed the cellulose content. [32]

3.2.2 Effect of reaction time and temperature

Effects of reaction time and temperature in acid hydrolysis are presented in Tables 2, 3 and 4. The results showed that treating jute fiber with high temperature for a long time slightly lowering the solid yield and cellulose content. The hydrolysis of xylan also increased by increasing the reaction temperature from 100 to 120°C. Obviously, xylan content was decreased from 9.2 to 3.1% with increased temperature from 100 to 120°C. It can be observed that increased temperature enhanced the catalytic effect of sulfuric acid. Table 4 also implied that increased reaction time from 1 h to 2 h increased the ability of the chemical to remove xylan and cellulose content was also reduced. Dubey et al. observed that total reducing sugar (TRS) from waste paper increased as the reaction time increased from 1 h to 6. [33] Furthermore, lignin yield was slightly reduced with increased temperature. This indicated that a part of lignin was dissolved in the prehydrolysis process. The lower lignin content caused by disrupting the structure by acid which also meant at high reaction condition

resulted in the loss of polysaccharides.

3.3 The optimum conditions

The optimum condition for high cellulose production with less xylan was obtained using 1 N sulfuric acids and reacted at 100°C for 90 min, in which cellulose and xylan content was 60.5 and 2.9%, respectively. However, sulfuric acid concentration could be reduced to 0.25 N with increasing reaction condition to 120°C for 120 min. As a result, 56.1% and 1.3% of cellulose and xylan were obtained, respectively. Dubey, et al. reported that the condition of 0.5 N sulfuric acid at 120°C for 120 min was the optimum condition for bioethanol production from waste paper. It was also reported that 97.6% of hemicellulose from sorghum biomass was converted into xylobiose, xylose, arabinose, and furfural by reacted sorghum biomass with 0.2 M sulfuric acid at 121°C for 120 min. [30]

Jute fiber treated at severe reaction conditions (high temperature or higher acid concentrations) caused the loss of cellulose and hemicellulose contents. [32] A decrease in hemicellulose content at severe reaction conditions might be due to the xylan's degradation to furfural. It was reported that increasing the acid concentration and reaction condition to 121°C increased the concentration of furfural. [30] At severe reaction conditions, furfural can be decomposed to form formic acid. [34] Furthermore, a decrease in cellulose content might be due to the cellulose's degradation into glucose and a further increase in the reaction condition glucose was easily decomposed to 5-HMF (dehydration product of glucose). It was reported that increase the acid concentration to 0.2 M at 121°C increased 5-HMF content. [30] Further increased in the reaction condition, 5-HMF could be decomposed into formic acid. [35] However, an increase in acid hydrolysis conditions was affected in improving cellulose purity.

Table 5 showed the purity of cellulose from dif-

Table 5. Cellulose purity of jute from the optimum conditions at different temperature

Acid conc. (N)	Temp. (°C)	Time (min)	Chemical constituents (%)		
			Lignin	Cellulose	Xylan
1.00	100	90	14.4±0.2	81.7±0.0	3.9±0.2
0.50	110	120	14.3±0.2	82.7±0.1	3.1±0.0
0.25	120	120	14.1±0.3	84.0±0.3	2.0±0.1

Acid con, Acid concentration

ferent reaction conditions. An increase in cellulose purity along with a decrease in yield. Cellulose purity from raw jute was 71.8%, which is improved continuously with increasing reaction time and temperature. Cellulose purity was 84.0% when reacted at severe conditions (120°C for 120 min), indicating that about 81.8% of xylan was dissolved. A reduction in the xylan content caused an increase in cellulose purity. However, the yield was also reduced by about 33.2%.

Hemicellulose composition in jute biomass mainly consists of xylan, mannose, galactose, arabinose, and rhamnose. [29] Dissolving pulp must be low in hemicellulose content. Hemicelluloses had detrimental effects on the manufacturing process and the properties of regenerated celluloses' end-products. [36] Hemicellulose increase the consumption of chemicals, causing false viscosity and poor filterability during the rayon production process. [37] Hemicellulose also lead to decrease strength properties of regenerated cellulose. [21] Cellulose purity can also be improved by reducing lignin through bleaching. Therefore, further bleaching is required in order to produce high purity dissolving pulp.

4. Conclusions

The presence of 82.6% of carbohydrates makes jute as potential and renewable raw materials for dissolving pulp. Acid hydrolysis pretreatment of jute fiber was carried out with various acid concentrations and reacted with different tempera-

tures and times for xylan removal. The results showed that increasing temperature, reaction time, and acid concentration resulted in higher cellulose purity. However, cellulose yield also decrease along with the loss of xylan. The optimum condition for acid hydrolysis of jute for cellulose production was 1 N H₂SO₄ at 100°C for 90 min. However, increased the reaction conditions to 120°C for 120 min could be reduced chemical used to 0.25 N H₂SO₄ with cellulose and xylan content was 70.1 and 1.7%, respectively. Severe reaction conditions started to degrade cellulose to glucose and reduce the yield. However, this condition could remove xylan completely and result in high cellulose purity. In mild reaction conditions, xylan degraded faster than cellulose and improved the yield in which cellulose was kept but some xylan was still remaining. Cellulose purity of jute fiber at the optimum condition was 81.7% with less xylan (3.9%). Further bleaching is needed for lignin removal in order to improve the cellulose purity for dissolving pulp production.

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