

Prehydrolysis kraft pulping of jute cutting and caddis mixture for rayon production

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ABSTRACT: Jute cutting, jute caddis, and cutting-caddis mixtures were prehydrolyzed by varying time and temperature to get about 90% prehydrolyzed yield. At the conditions of 170°C for 60 min of prehydrolysis, the yield for 100% jute cutting was 76.3%, while the same for jute caddis was only 67.9%. But with prehydrolysis at 150°C for 60 min, the yield was 90% for jute cutting, where 49.94% of original pentosan was dissolved and prehydrolysis of jute caddis at 140°C in 60 min yielded 86.4% solid residue. Jute cutting-caddis mixed prehydrolysis was done at 140°C for 30 min and yielded 92% solid residue for 50:50 cutting-caddis mixtures, where pentosan dissolution was only 29%. Prehydrolyzed jute cutting, jute caddis, and cutting-caddis mixtures were subsequently kraft cooked. Pulp yield was only 40.9% for 100% jute cutting prehydrolyzed at 170°C for 60 min, which was 10.9% lower than the prehydrolysis at 140°C. For jute cutting-caddis mixed prehydrolysis at 140°C for 45 min followed by kraft cooking, pulp yield decreased by 3.3% from the 100% cutting to 50% caddis in the mixture, but 75% caddis in the mixture decreased pulp yield by 6.7%. The kappa number 50:50 cutting-caddis mixture was only 11.3. Pulp bleachability improved with increasing jute cutting proportion in the cutting-caddis mixture pulp.

Application: This research demonstrates that jute cutting-caddis mixed pulp can be used in rayon production.

Both paper and dissolving grade pulps are produced from wood, which come from the forest. There is extensive interest in utilizing cellulose fibers from annual plants or short rotation trees in forest-deficient countries such as Bangladesh [1-7]. Historically, jute is the most important natural fiber in Bangladesh. The chemical and morphological characteristics of jute make it an excellent raw material for pulp production [2]. Utilization of jute has social and economic importance in Bangladesh. To increase the utilization of jute as a pulp producing material, many studies have been carried out on jute pulping [1-2,4-9].

Dissolving pulp is a chemically refined bleached pulp composed of more than 90% α -cellulose. Dissolving pulp is used for the production of rayon and cellophane, cellulose esters (acetates, nitrates, etc.), cellulose ethers (carboxymethyl cellulose, etc.), and graft and cross-linked cellulose derivatives [10]. Conventionally, dissolving pulp is produced by prehydrolysis kraft and acid sulfite processes. Mild autohydrolysis is an environmentally friendly technology, allowing the fractionation of lignocellulosic material (LCM), where LCM and water are contacted at temperatures in the range of 150°C–220°C. In these kinds of treatments, hemicelluloses are depolymerized and acetyl groups are cleaved, whereas little alteration is caused in lignin, and cellulose remains almost untouched. Purity of dissolving pulp depends on the severity of prehydrolysis [10]. Jahan et al. [11] showed that prehydrolysis temperature and sulfuric acid (H_2SO_4) concentration had

great influences on the properties of dissolving pulp from jute. It was also observed that prehydrolysis temperature affected overall pulp yield. Testova et al. [12] also showed that increasing prehydrolysis intensity resulted in pulps of higher purity but lower yield and degree of polymerization. It has been established that high purity dissolving pulp can be produced from jute fiber using the prehydrolysis kraft process [13] of 90%+ purity, which can be used for rayon grade dissolving pulp [10]. In this investigation, we tried to reduce biomass dissolution by around 10% by reducing prehydrolysis temperature and time.

Recently, the Bangladesh government is interested in producing rayon from jute fiber. Presently, rayon is being produced from wood. The price of jute fiber (US\$600) cannot compete with wood (US\$120). In jute mills, the bottom part of jute fiber is discarded (due to inferior properties) and is commonly known as jute cutting; in addition, jute caddis is obtained as rejects in jute mills. Compared with the price of jute fiber at around US\$600 per ton, the price of jute cutting and caddis is about US\$500 and US\$60 per ton, respectively [14]. Therefore, prehydrolysis of jute cutting, jute caddis, and cutting-caddis mixtures were optimized by varying time and temperature to get prehydrolysis yields of about 90%. The prehydrolyzed jute cutting, jute caddis, and cutting-caddis mixtures were subsequently cooked using the kraft process. The resulting pulps were bleached with $D_0E_pD_1$ bleaching sequences. Bleached and unbleached pulps were evaluated in terms of yield, viscosity, purity and reactivity.

MATERIALS AND METHODS

Raw materials

Jute cutting and jute caddis were collected from the Bangladesh Jute Mills Corporation (BJMC), and cut to 2–3 cm in length. After determination of the moisture content, air dried raw material equivalent to 200 g oven dried (o.d.) material was weighed separately in polyethylene bags for subsequent prehydrolysis and cooking experiments. Jute cutting and jute caddis contain 61.1% and 54.6% α -cellulose, 17.8% and 16.1% pentosan and 15.5% and 15.6% lignin, respectively, with other minor ingredients.

Prehydrolysis

Prehydrolysis of a jute cutting and caddis mixture in different ratios was carried out in an electrically heated digester of 5 L capacity. The digester was rotated at 1 rpm. Water prehydrolysis was carried out at 140°C, 150°C, and 170°C for 30 min, 45 min, and 60 min. The raw material-to-liquor ratio was 1:5 (g/mL). The time required to raise max temperature from room temperature (30°C) was 35 min. After completing prehydrolysis, pressure was released by venting the valve and the liquor was separated from the solid mass by filtration and kept in a refrigerator for analysis.

Solid contents

The total solid content in the prehydrolysis liquor (PHL) was gravimetrically determined by drying a 10 ml sample at 105°C to constant weight.

Pulping

Pulping of prehydrolyzed jute cutting and jute caddis mixtures was carried out by the kraft process in the same digester as in prehydrolysis under the following cooking conditions:

- Fiber to liquor ratio: 1:5 g/mL
- Active alkali charge: 18% on prehydrolyzed residue.
- Sulfidity: 27%
- Temperature: 170°C
- Cooking time: 120 min at maximum temperature

Pulp yield was determined gravimetrically. Pentosan dissolution was calculated as:

% of residual pentosan in pulpX% yield of pulp/% of pentosan in original raw material

$D_0E_pD_1$ bleaching

Pulps were bleached by $D_0E_pD_1$ bleaching sequences in plastic bags. In the first stage (D_0) the chlorine dioxide (ClO_2) charge was 1.5%. The initial pH was adjusted to 2.5 by adding dilute H_2SO_4 . In the alkaline extraction stage, the sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2) charges were 2% and 0.5% (on o.d. pulp), respectively. The temperature was 70°C for 60 min and pulp consistency was 10%. In the D_1 stage, the end pH was 4. The ClO_2 charge in the D_1 stage was 0.5%.

Evaluation of pulps

Pulp tests were performed according to TAPPI Standard Test Methods: kappa number (T 236 cm-85 “Kappa number of pulp”); brightness (T 452 om-92 “Brightness of pulp, paper, and paperboard [directional reflectance at 457 nm]”); viscosity (T 230 om-89 “Viscosity of pulp [capillary viscometer method]”); α -cellulose (T 203 om-88 “Alpha-, beta- and gamma-cellulose in pulp”); and alkali solubility S_{10} and S_{18} (T 235 cm-85 “Alkali solubility of pulp at 25°C”).

RESULTS AND DISCUSSION

Prehydrolysis

It is established that high purity dissolving pulp can be produced from jute fiber using a prehydrolysis kraft process [13]. But prehydrolysis of jute fiber at 170°C reduced yield to around 80%, consequently resulting in decreased overall pulp yield and pulp viscosity [11]. The rayon grade dissolving pulp requires 90% purity. Therefore, we tried to reduce biomass dissolution to around 10% by reducing prehydrolysis temperature and time. **Table I** shows the prehydrolysis yield of jute cutting, jute caddis, and cutting-caddis mixtures with varying temperature and time. Dissolved biomass in jute cutting was only 3%–5% after prehydrolysis at 140°C. With prehydrolysis at 150°C for 60 min, jute cutting yielded 90% solid residue, where residual pentosan content was 9.88%. This indicates that 49.94% of the original pentosan was dissolved during prehydrolysis at these conditions. Makishima et al. [15] separated 82.2% of the xylan fraction as a mixture of xylose, xylo-oligosaccharides, and higher-xylo-oligosaccharide from corncob at 200°C for 10 min in semi-pilot scale in a continuous flow-type hydrothermal reactor. Rissanen et al. [16] extracted hemicelluloses from spruce sapwood at 90°C and 110°C with water and showed that two hemicellulose fractions (pentose and hexose) have different susceptibility towards hydrolytic degradation. This fact has been reported for xylan degradation in hydrothermal treatments [17], and it can be ascribed to differences in accessibility or in chemical structure between different zones of xylan [17–19].

Prehydrolysis of jute caddis at 140°C yielded 89.2% solid residue for 30 min, which was further decreased to 86.4% in 60 min of prehydrolysis. The result indicated that caddis dissolved biomass easily as it is exposed and the presence of dirt, grease, bath oil, etc., in caddis, which were dissolved in the prehydrolysis process. The pentosan dissolution was 36.8% in 30 min and 43.9% in 60 min of prehydrolysis at 140°C. The prehydrolysis of jute caddis was not carried out at 150°C as the yield was already down below 90% at 140°C. In this context, cutting-caddis mixed prehydrolysis was done at 140°C and yielded 92% solid residue for 30 min of prehydrolysis time for 50:50 cutting-caddis mixtures, where pentosan dissolution was only 29%. As expected, prehydrolysis yield increased with increasing jute cutting proportion in the cutting-caddis mixture (Table I).

For comparison, prehydrolysis at 170°C for 60 min was also carried out for different ratios of cutting-caddis mixtures fol-

Raw Material	Temperature, °C	Time, min	Yield (%) A	Solid Content (%) B	A + B	Pentosan, %
Jute cutting	140	30	97.85	2.91	100.76	16.22
		45	96.5	3.57	100.07	14.81
		60	95.53	4.04	99.57	13.75
	150	30	95.51	4.92	100.43	14.02
		45	93.67	6.69	100.36	11.95
		60	90.04	8.94	98.98	9.88
Jute caddis	140	30	89.2	9.58	98.78	11.41
		45	88.2	10.41	98.61	11.06
		60	86.4	12.33	98.73	10.46
Jute cutting (50%) + jute caddis (50%)	140	30	92	8.02	100.02	13.82
		45	91.8	8.56	100.36	12.97
		60	89.8	11.86	101.66	12
Jute cutting (75%) + jute caddis (25%)	140	45	92.95	6.22	99.17	12.91
Jute cutting (25%) + jute caddis (75%)	140	45	90.12	7.64	97.76	12.54

I. Prehydrolysis of jute cutting, jute caddis, and cutting-caddis mixtures at 140°C and 150°C.

Identification No.	Percentage of Jute Cutting	Percentage of Jute Caddis	Prehydrolysis Yield (%) A	Prehydrolysis Liquor		Pulping	
				Solid Content (%) B	A+B	Pulp Yield	Kappa No.
1	100	0	76.31	9.5	85.81	40.9	5.2
2	83.33	16.67	76.92	9.3	86.22	40.2	5.3
3	80	20	76.38	9.8	86.18	40.2	5.4
4	62.5	37.5	75.51	10.9	86.41	40	5.9
5	60	40	75.02	10.1	85.12	39.9	6
6	57.14	42.86	75.83	10.2	86.03	39.2	6.2
7	55.56	44.44	74.92	11	85.92	39.2	6.6
8	50	50	73.54	11.2	84.74	39.2	6.6
9	25	75	73.42	9.3	82.72	37.9	7.2
10	0	100	67.92	8.6	76.52	35.2	9.3

II. Prehydrolysis of jute cutting, jute caddis, and cutting-caddis mixtures at 170°C.

Identification No.	Percentage of Cutting	Percentage of Caddis	Pulp Yield, %	Kappa No.
1	100	0	51.8	9.8
2	75	25	48.7	11.2
3	50	50	48.5	11.3
4	25	75	45.1	12.1
5	0	100	41.7	13.7

III. Kraft pulping of prehydrolyzed jute cutting-caddis mixture (active alkali = 18%; time = 90 min; temperature= 170°C).

lowed by kraft cooking with 18% active alkali charge at 90 min of cooking at 170°C. The detailed results are shown in **Table II**. The prehydrolysis yield for 100% jute cutting was 76.3%, while the same for jute caddis was only 67.9%. These yields were much lower than those seen using prehydrolysis at 140°C and 150°C (Table I). The prehydrolysis yield decreased slowly with increasing caddis proportion in the cutting-caddis mixture. The sum of solid content in the prehydrolysis liquor (PHL) and prehydrolysis yield was supposed to be 100%. However, total mass balance reached to 85% up to 50:50 cutting-caddis mixtures (Table II). A further increase of caddis proportion decreased mass balance closure, but in the prehydrolysis at 140°C–150°C, mass balance was about 100% (Table I). At a higher temperature of prehydrolysis, most of the acetyl groups were liberated and accelerate hemicelluloses degradation, which generated furfural and other volatile compounds [20]. These were not accounted for in the solid content.

Pulping

The jute cutting-caddis mixture with prehydrolysis at 140°C for 45 min was cooked, and results are shown in **Table III**. Pulp yield from the starting raw material was 51.8% for 100% jute cutting with kappa number 9.8. Pulp yield decreased and kappa number increased slightly with increasing caddis proportion in the cutting-caddis mixture. Pulp yield decreased by 3.3% from the 100% cutting to 50% caddis in the mixture, but 75% caddis in the mixture decreased pulp yield by 6.7%. As jute caddis is cheaper than jute cutting, our target was to employ as much jute caddis in the cutting-caddis mixture as possible. The kappa number of cutting-caddis mixture (50:50) was only 11.3, which was supposed to be easily bleached [21]. Considering pulp yield and the kappa number, the 50:50 cutting-caddis blend was considered to be the optimum mixture for this work. In an earlier study, it was observed that prehydrolysis of jute cutting at 150°C for 60 min followed by kraft cooking with 18% active alkali (as sodium oxide, Na₂O) and 25% sulfidity for 60 min at 170°C yielded pulp of 49.9% with kappa number 11.2 [2]. In another study, prehydrolysis at 170°C for 60 min followed by soda pulping with 18% alkali charge for 60 min of cooking yielded only 37.8% pulp, which was much lower pulp yield than our present investigation [11].

As shown in Table II, pulp yield was only 40.9% for 100%

jute cutting prehydrolyzed at 170°C for 60 min, which was 10.9% lower than the prehydrolysis at 140°C–150°C (Table III). Pulp yield also decreased with increasing caddis proportion in the cutting-caddis mixtures (Table II). Pulp yield was only 35.2% in 100% caddis, 6.5% lower than the prehydrolysis at 140°C. The kappa numbers of cutting-caddis mixtures were 5–7, which were much lower than the pulp obtained from the prehydrolysis at 140°C–150°C (Table III). This may be explained by the same results seen in bamboo studies [22]. That study showed that bamboo structure became loose in the prehydrolysis process due to the removal of hemicelluloses, which creates a favorable environment for chemical penetration in the subsequent pulping process. Increase in prehydrolysis temperature improved delignification in a subsequent cooking process [6,11]. From the previously mentioned results, it is seen that pulp yield from mild prehydrolysis was quite high with a low kappa number.

Bleaching

The cooks described in Tables II and III were then bleached using a D₀EpD₁ bleaching sequence and results are shown in **Table IV** and **Table V**. As shown in Tables IV and V, increasing cutting proportion in the cutting-caddis mixture improved pulp bleachability. The final brightness of 100% jute cutting pulp obtained from prehydrolysis at 140°C for 45 min reached to 82.9%, while the same for caddis was only 77.46%. The final brightness of 50:50 cutting-caddis mixtures was 80.8% (Table IV). On the other hand, with prehydrolysis at 170°C for 60 min, final brightness of pulp blends fell between 87.82% for pure cutting and 81.68% for pure caddis (Table V). As shown in Table V, brightness decreased with increasing caddis proportion in the cutting-caddis mixture.

Table IV also shows that the α -cellulose content in the resulting bleached pulp was about 91.5% for 100% cutting in such mild prehydrolysis conditions and pentosan content was 3.34%. These results were better than the dissolving pulp produced from jute stick by the prehydrolysis at 170°C for 60 min followed by kraft pulping [23]. In a study, dissolving pulp was produced from rice straw with 93.6% purity at a given residual pentosan content of 5.1% [24]. The purity of bleached pulp was dependent on the severity of prehydrolysis and active alkali in cooking. Table V shows that the α -cellulose contents in the pulps obtained from the prehydrolysis at 170°C

Identification No.	Percentage of Cutting	Percentage of Caddis	Bleaching Yield, %	Brightness, %	Viscosity, mPa.s	α -cellulose, %	S_{10} , %	S_{18} , %	Pentosan, %
1	100	0	95.11	82.9	10.65	91.45	9.79	7.12	3.34
2	75	25	94.85	82.5	10.84	90.12	9.38	7.8	3.43
3	50	50	94.48	80.8	10.85	90.03	11.12	8.3	4.57
4	25	75	93.73	80.48	10.23	89.40	12.66	8.87	3.58
5	0	100	94.77	77.46	11.00	89.19	12.00	8.91	4.41

IV. Bleaching of cutting-caddis mixed pulps prehydrolyzed at 140°C for 45 min (S_{10} and S_{18} = alkali solubility).

Identification No.	Percentage of Jute Cutting	Percentage of Jute Caddis	Brightness, %	Viscosity, mPa.s	α -cellulose, %	S_{10} , %	S_{18} , %	Pentosan, %
1	100	0	87.82	4.52	95.6	3.51	1.71	1.32
2	83.33	16.67	85.92	5.33	95.1	5.73	2.35	1.19
3	80	20	85.76	4.57	95.2	5.78	2.10	1.19
4	62.5	37.5	86.35	3.74	95.4	5.59	2.12	1.62
5	60	40	84.72	3.54	94.4	6.78	2.45	1.52
6	57.14	42.86	86.35	5.35	96.2	6.93	1.52	1.60
7	55.56	44.44	85.18	3.77	94.6	7.60	1.52	1.40
8	50	50	84.04	4.55	96.2	6.68	2.05	1.86
9	25	75	83.16	3.96	95.3	6.85	2.15	1.21
10	0	100	81.68	4.50	96.9	7.71	2.37	2.92

V. Bleaching cutting-caddis mixed pulps prehydrolyzed at 170°C for 60 min (S_{10} and S_{18} = alkali solubility).

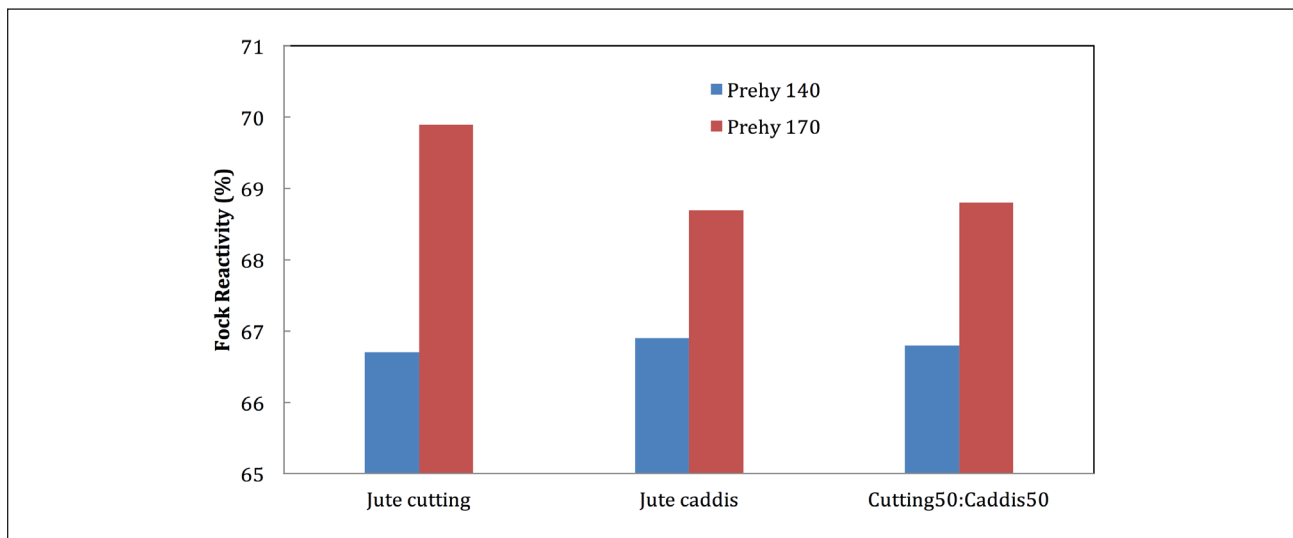
for 60 min were 94%–97%. The α -cellulose content was independent of cutting-caddis proportion.

Alkali solubilities S_{10} and S_{18} illustrate information on the low molecular weight carbohydrates (degraded cellulose and hemicellulose) and hemicelluloses in pulp, respectively [25]. As shown in Tables IV and V, low molecular weight cellulose (S_{10} – S_{18}) in the pulp obtained from prehydrolysis at 140°C was 2%–4% only, while the same was 2%–8% for from prehydrolysis at 170°C.

Dissolving pulp with high purity and controlled viscosity are required for viscose rayon production. A high viscosity creates problems with the filtration of viscose, and low viscosity results in poor physical strength of the resulting rayon products. Therefore, uniform molecular weight distribution, and good reactivity is usually preferred [10,26]. As shown in Tables IV and V, viscosities of dissolving pulps obtained from prehydrolysis at 170°C were 3–5 mPa.s, while the viscosities of dissolving pulps obtained from prehydrolysis at 140°C were 10–11 mPa.s. From the previous discussion, it can be seen that milder prehydrolysis produced dissolving pulp with acceptable

properties (purity, residual pentosan, viscosity, and brightness) for rayon production from jute cutting-caddis mixtures.

Fock reactivity of dissolving pulp is one of the most important parameters for viscose rayon production. The measurement procedures can simulate the commercial viscose-rayon process very well [27–28]. The influence of prehydrolysis conditions and jute cutting-caddis mixture on the Fock reactivity of the dissolving pulp is shown in **Fig. 1**. Fock reactivity for jute cutting, caddis and cutting-caddis mixed pulp obtained from prehydrolysis at 140°C was 66.7%, 66.9%, and 66.8%, respectively, Fock reactivity of pulps obtained from prehydrolysis at 170°C were 69.9%, 68.7%, and 68.8%, respectively. Lower hemicelluloses content in pulps at 170°C may explain the better reactivity as compared to pulp at 140°C (Tables IV and V). This can also be explained by the higher “ S_{10} – S_{18} ” value at 170°C, possibly indicating improved accessibility (i.e., the physical ability of reactants/solvents to reach the hydroxyl groups, and to overcome steric hinderance in the fiber structure) [29]. Many efforts have been made to increase Fock reactivity. Miao et al. [30] showed that Fock reactivity of the



1. Effect of prehydrolysis conditions on Fock reactivity of dissolving pulp.

pulp sample increased from 49.27% to 71.75% after 6 min of treatment by cellulase. In another study, the Fock reactivity of eucalyptus sulfite-based dissolving pulp was also increased from 65% to 82% after treatment with endoglucanase at the enzyme dosage of 30 ECU/g dry weight pulp for an hour [31]. It is suggested that these treatments can be used on cutting/caddis pulps. Based on literature data, acceptable Fock reactivity was obtained in mild prehydrolysis of jute cutting-caddis mixtures (50:50).

CONCLUSIONS

Jute cutting, jute caddis and cutting-caddis mixtures were prehydrolyzed in order to produce dissolving pulp for rayon production. Prehydrolysis at 170°C dissolved 22%–32% biomass, while prehydrolysis at 140°C dissolved 2%–10% biomass. Dissolved biomass increased with the increase of caddis proportion in cutting-caddis mixtures. The overall pulp yield was only 40.9% for 100% jute cutting prehydrolyzed at 170°C, while the yield was 51.8% for prehydrolysis at 140°C. Pulp yield also decreased with increasing caddis proportion in cutting-caddis mixtures. Prehydrolysis at 170°C showed improved delignification compared with prehydrolysis at 140°C. The final brightness (brightness 77%–83% at 140°C vs. 82%–88% at 170°C) and purity of pulps (α -cellulose 89%–92% at 140°C vs. 94%–97% at 170°C) obtained from the prehydrolysis at 170°C was better than the prehydrolysis at 140°C. The final brightness of 50:50 cutting-caddis mixed pulps reached to 80.8% with the consumption of 20 ClO₂ kg/ton of pulp with purity of 90% α -cellulose and 66.8% Fock reactivity. The cutting-caddis mixed pulp can be used in rayon production. **TJ**

ACKNOWLEDGEMENTS

The authors wish to thank the Ministry of Science and Technology Government of Bangladesh and BCSIR for providing

necessary fund to carry out this research.

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ABOUT THIS PAPER

Cite this article as:

Nayeem, J., Jahan, M.S., Popy, R.S., et al., *TAPPI J.* 18(5): 287(2019).

<https://doi.org/10.32964/TJ18.5.287>

DOI: <https://doi.org/10.32964/TJ18.5.287>

ISSN: 0734-1415

Publisher: TAPPI Press

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The Bangladesh government is going to install a dissolving pulp mill based on jute. To reduce the cost of raw materials, we carried out this study. Jute-based raw material can also be used in dissolving pulp, in addition to paper pulp, with higher yield and lower bleaching chemicals.

We found that mass balance was difficult for prehydrolysis at high temperature (170°C) due to the formation of volatile compounds. We also discovered that optimization of prehydrolysis conditions is very important for each raw material. It was interesting to find that prehydrolysis at lower temperature (140°C) is also possible for producing rayon grade dissolving pulp.

For mills, this research shows that a mixture of raw materials (jute cutting and caddis) can be used in same fiber line. Our next step is to study the processability and reactivity of dissolving pulp made of this mixture.

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