ALTERNATIVE PULPING PROCESS FOR PRODUCING DISSOLVING PULP FROM JUTE

M. Sarwar Jahan, Sabina Rawsan, D. A. Nasima Chowdhury, and A. Al-Maruf

Dissolving pulps are the raw materials of cellulose derivatives and of many other cellulosic products. Jute is a very good source of cellulose and worthy of consideration for the production of dissolving pulp. In this investigation jute fiber, jute cuttings, and jute caddis were used as raw materials to prepare dissolving pulp by a formic acid process. A very high bleached pulp yield (49 to 59%) was obtained in this process. The α-cellulose content was 93 to 98%, with a high pulp viscosity. Also a good brightness (81 to 87%) was achieved in totally chlorine free bleaching. Jute fiber showed the best and jute caddis showed lowest performance in producing dissolving pulp via the formic acid process. R18-R10 values were much lower than for conventional dissolving pulp.

Keywords: Jute fiber; Jute cutting; Jute caddis; Dissolving pulp; TCF bleaching; α-cellulose

*Contact information: Pulp and Paper Research Division, BCSIR Laboratories, Dhaka, Dr. Qudrat-I-Khuda Road, Dhaka-1205, Bangladesh *Corresponding author: m_sarwar@bdonline.com*

INTRODUCTION

Forest resources are diminishing in Bangladesh, while consumption of paper and allied materials is continuously increasing (FAO 2003). So it is important to seek strategies that will compensate for diminishing forest resources and also provide local economic value. It is therefore crucial to explore nonwoods as potential raw materials for pulp production. Jute used to play an important role in the socio-economic development of Bangladesh. A significant portion of the total export earnings was dependent on jute and related products in the sixties (Jahan et al. 2007). The chemical and morphological characteristics of jute favor it as a pulping raw material (Nahar 1987). Therefore, many studies have been done on the pulp from jute at home and abroad (Akhtaruzzaman and Shafi 1995; Jahan 2001; Roy et al. 1998; Jahan 2008). Retted jute fiber contains a very high α-cellulose and low hemicellulose content as compared to wood or other nonwood (Nahar 1987). So it may be used in producing dissolving pulp. But the price of jute fiber cannot compete with wood. The bottom part of jute, which is called jute cuttings, is discarded in jute mills. About 15% of jute cuttings and 3% of caddis are produced in processing of jute fiber in conventional jute mills. Bangladesh produced 8,10,000 MT of jute fiber in 2004-2005 (Anon 2007). So about 120,000 metric tons of jute cuttings and 24,300 metric tons of caddis remained left over in jute mills in one year. These residual materials have the potential to be used as pulping raw materials.

Most of the dissolving pulps are produced from wood using the prehydrolysis kraft or acid sulfite processes (Biermann 1993; Hinck et al. 1985). Dissolving wood pulp is a chemically refined bleached pulp composed of more than 90 percent pure cellulose.

The end uses of dissolving pulp include cellophane and rayon, cellulose esters (acetates, nitrates, etc.), cellulose ethers (carboxymethyl cellulose, etc.), and graft and cross-linked cellulose derivatives (Sjöström 1981).

Pulp quality of dissolving pulp is essential for cellulosic products such as carboxymethyl cellulose, viscose, cellulose film, and sausage skin, etc. The dissolving pulp quality depends both on properties of the raw materials and the pulp processing. The reactivity of cellulose pulp can refer to its capacity to participate in diverse chemical reactions. The two secondary hydroxyl groups on carbons two and three are more reactive than the primary hydroxyl group on carbon six (Krässig 1993). Dissolving pulp should have special properties, such as a high level of purity, uniform molecular-weight distribution, and good reactivity and accessibility of the cellulose to chemicals (Krässig 1993). To achieve maximum reactivity of pulp, acid hydrolysis, mechanical and swelling treatments, enzyme treatment, etc. is done (Engström et al 2006; Tang et al 2002). For example, endoglucanase preferably degrades amorphous rather than crystalline cellulose and cleaves the cellulose randomly within the chain (Rabinovich et al. 2002; Henriksson et al. 1999). Since less ordered or amorphous regions occur on the surface and between the microfibrils (Wickholm 2001; Vietor et al. 2002) endoglucanse treatment leads to a swelling of the cell wall and thus an increase in accessibility to solvents and reagents.

Organosolv pulping processes have been suggested as an alternative pulping route. Organosolv delignification followed by Totally Chlorine Free (TCF) bleaching is an environmentally friendly approach towards the production of high-purity cellulose pulp (Sixta et al. 2004). The susceptibility of organosolv pulp towards TCF bleaching has been claimed to be one of their most favorable features (Dapia et al. 2003).

New acidic pulping processes such as the formic acid process have potential to remove lignin and hemicellulose at the temperature of boiling (Jahan et al. 2007a). The objectives of this study were to produce dissolving pulp from jute fiber, jute cuttings, and jute caddis by use of a formic acid process at atmospheric pressure, followed by totally chlorine-free bleaching (TCF). A schematic flow diagram for this process is shown in Appendix 1.

EXPERIMENTAL

Materials

Jute fiber, jute cuttings and caddis were collected from Jute Mill in Narayangong, Bangladesh. The jute fiber was very clean and free from scaling. These were chopped to 2-3 cm in length. Chemical analysis of these raw materials is given in Table 1, which is published in elsewhere (Jahan et al 2007).

Chemical Analysis

The extractives (T204 om88), Klason lignin (T211 om 83), and pentosan (T223 cm-84) were determined in accordance with TAPPI Test Methods. Holocellulose was determined by treating extractives-free wood meal with $NaClO_2$ solution. The pH of the solution was maintained at 4 by adding CH_3COOH-CH_3COONa buffer. The α-cellulose content was determined by treating the holocellulose with 17.5 % NaOH.

Formic Acid Treatment (F)

Jute fiber, jute cuttings, and jute caddis were cooked in a flask with an aqueous solution of formic acid (90 %v/v) at boiling temperature (107 ^{o}C) under atmospheric pressure for 1 h. The cooking was carried out in batches of 30 g dry of samples in a 500 L, flat-bottom, and wide-mouth boiling flask equipped with a condenser. After cooking, the spent cooking liquor was separated and collected by filtration. Pulp was washed with 80% formic acid, followed by hot water.

Peroxyformic Acid Treatment (Pf)

Formic acid treated pulp was further delignified with peroxyformic acid (PFA) at 80 ^{o}C. The reaction was carried out in a thermostatic water bath. The peroxyformic acid was prepared by adding 90% formic acid with varying proportions of H_2O_2 (2, 4, and 6% of o.d. jute fiber, jute cuttings, and caddis). The time was held constant at 120 min for each peroxide concentration. After completion of the pulping, a pulp was filtered off and washed with 80% fresh formic acid, and finally with hot water. Pulp yield was determined gravimetrically on raw material. The kappa number of the pulps was determined by TAPPI standard methods (T 236). In one experiment, peroxyformic acid treatment as a first step was carried out for jute fiber, jute cuttings, and jute caddis, followed by formic acid treatment and again peroxyformic acid treatment.

Bleaching

Bleaching experiments of unbleached pulp (10g) were carried out at 10% pulp consistency. The pH was adjusted to 11 by adding NaOH. The hydrogen peroxide was 2% on o.d pulp. First, 0.1% $MgSO_4$, 2% NaOH, and 0.1 % $NaSiO_2$ were added to the required amount of distilled water in a beaker, followed by H_2O_2 addition to the mixture. This pulp was preheated to a desired temperature in a water bath and mixed with bleaching liquor. The bleaching temperature was 70 ^{o}C for 1 h. A similar procedure was followed in the 2nd stage of peroxide bleaching.

Evaluation of Pulps

Pulp tests were performed according to the Standard Methods of the Technical Association of the Pulp and Paper Industry (TAPPI, Atlanta, GA): kappa number (T 236 cm-85); brightness (T 452 om-92); viscosity (T 230 om-89); pentosan (T 223 cm 84), α-cellulose (T 203 om-88); alkali solubility R10 and R18 (T 235 cm-85) and carboxyl content (T 237 cm 98).

RESULTS AND DISCUSSION

Pulping

A good delignification medium must have nucleophilic species to promote the cleavage of lignin and to dissolve the lignin fragments. The addition of water is able to promote the delignification reaction but reduces the ability of the solvent to dissolve the lignin generated in the process. This is because lignin is a hydrophobic biopolymer, and the hydrophobicity induces adsorption of lignin fragments onto the surface of a pulp

fiber. To prevent lignin reprecipitation in formic acid liquor, a suitable concentration of formic acid is critically important. It has been shown that a formic acid liquor having a 90% (v/v) concentration had a positive effect on delignification (Jahan et al. 2007, 2007b). If the formic acid charge was less than this in the reaction mixture, the extent of delignification decreased significantly.

Formic acid (FA) treatment at boiling temperature removed 79.6, 80.1, and 79.7 percent of the pentosan from jute fiber, jute cuttings, and jute caddis, respectively (data are not shown), which in very important for dissolving pulp. After this FA treatment, 62.9, 51.7, and 52.5 percent delignification was achieved from jute fiber, jute cuttings, and jute caddis, respectively. Peroxyformic acid delignification was carried out after formic acid treatment, and the pulp compositions are given in Table 2. The peroxyformic acid was prepared by adding H_2O_2 to 90 % formic acid. Such a mixture of FA, H_2O_2, and peroxyacid proved to be efficient in delignification of unbleached pulp through the combined action of the peroxyacids as oxidizing agent and formic acid as solvent for the lignin (Kham et al. 2005). A high pulp yield (52 to 69%) was obtained in all these raw materials. Jute cutting possessed the highest pulp yield (62-69 %), and jute caddis gave the lowest pulp yield (52-63 %) among these raw materials. This may be explained by higher impurities and foreign materials in caddis, which are dissolved during pulping, and also higher α-cellulose in jute fiber (Table 1). These yields are quite similar to the results reported for paper grade kraft pulping and better than for dissolving pulp in prehydrolysis kraft process (Jahan et al. 2007; Jahan 2008). Shahin and Young (2008) obtained over 70% pulp yield from jute fiber in acetic acid pulping, but kappa number was above 40. Easier pulping of these raw materials is attributed to lower lignin content (Table 1) and higher syringyl to guaiacyl ratio (Islam and Sarkanen 1993).

Table 1. Chemical Characteristics of Jute Fiber, Cuttings and Caddis (Jahan et al. 2007)

	Jute fiber	*Jute cutting*	*Jute caddis*
Klason lignin, %	12.7	14.0	14.7
Holocellulose, %	87.6	87.9	87.7
α-cellulose, %	63.1	60.0	58.6
Pentosan, %	13.5	14.1	14.0
Extractives (DCM), %	0.25	0.43	3.86

DCM- Dichloromethane

The kappa number analysis was included in this study to measure the reagent consumption in bleaching. The best results in term of kappa number were found for jute fiber. Kappa number of jute cuttings was 29.5 at 2% peroxide charge, which was rapidly dropped to 16.9 with increasing peroxide charge to 4%. A very high kappa number in jute cutting pulp at 2 % peroxide charge may be due to adhering scales of jute cuttings that consumed most of the peroxyformic acid. Kappa numbers of pulps were decreased with the increase of peroxide during peroxyformic acid treatment. Kappa number was again decreased if an additional peroxyformic acid treatment was included at the beginning.

Kappa number was reduced from 17.8 to 6.9 for jute fiber, 29.5 to 10.2 for jute cuttings, and 20.1 to 16.2 for jute caddis from FP_f to P_fFP_f stage pulping under similar peroxide charge. Pulp yield and kappa number were better than those of hardwoods (Parthasarathy et al. 1995).

Table 2. Properties of Jute Fiber, Jute Cutting, and Jute Caddis Pulp Obtained From FP_f, P_fFP_f Process

Stage	H_2O_2	Pulp yield, %	Kappa number	Viscosity	Brightness
Jute fiber					
FP_f	2	63.5	19.4	14.2	29.4
FP_f	4	61.3	17.8	13.8	31.3
FP_f	6	59.5	8.7	13.5	37.9
P_fFP_f	4	57.6	6.9	13.1	43.8
Jute cutting					
FP_f	2	69.2	29.5	19.9	19.3
FP_f	4	68.6	16.9	19.5	26.9
FP_f	6	65.9	11.2	16.4	27.3
P_fFP_f	4	62.3	10.2	16.2	34.3
Jute caddis					
FP_f	2	62.5	28.8	14.3	17.9
FP_f	4	58.8	20.1	12.8	19.2
FP_f	6	54.0	20.8	10.5	23.6
P_fFP_f	4	52.4	16.2	7.5	29.4

The viscosity of jute cutting pulp (16-20 mPa.s) was higher than that of jute fiber (13-14 mPa.s) and jute caddis (14-8 mPa.s). Viscosity values were decreased with peroxide charge in perpxyformic acid treatment. Jute cutting is obtained from the lower part of the plant. So this part is more mature, which is the reason for the higher viscosity.

One experiment for each raw material was carried out starting with peroxyformic acid (P_f) followed by FP_f treatment. This was done to better understand xylan removal and delignification efficiency of formic acid treatment after P_f treatment. Selective removal of lignin in P_f treatment may enhance xylan removal during a subsequent formic acid step. However, no meaningful enhancement of xylan removal efficiency was evident in formic acid treatment after P_f reatment (Table 3). But additional Pf treatment in the starting mixture improved pulp deligninfication (Table 2).

As expected, unbleached pulp brightness of jute fiber pulp was the highest, followed by jute cutting, and finally jute caddis. Brightness was improved with increasing peroxide charge in peroxyformic acid treatment. An additional peroxyaformic acid treatment in the beginning improved pulp brightness. Brightness was increased from 31.3 to 43.8 for jute fiber, from 26.9 to 34.3 for jute cuttings, and from 19.2 to 29.4 for jute caddis from FPf to PfFPf stage pulping under similar peroxide charge.

Bleaching

All pulps were bleached by alkaline peroxide, and results are given in Table 3. The pulp yield losses on bleaching were 1 to 3.4% for jute fiber, 13.1 to 15.1% for jute cuttings, and 2.2 to 4.7% for jute caddis, which were either as expected or lower than the expected range (Sixta et al. 2003).

Table 3. Properties of Bleached Pulp Obtained from FP_f and P_fFP_f Processed Pulps of Jute Fiber, Jute Cuttings, and Jute Caddis

Stage	H_2O_2	Pulp yield, %	α-cellulose	R-10	R-18	Viscosity	Brightness	Carboxyl content, meq/100 g pulp
Jute fiber								
FP_f	2	60.1	95.7	97.8	98.1	12.7	78.4	6.30
FP_f	4	59.5	96.9	96.9	97.9	11.8	79.1	6.26
FP_f	6	56.8	97.2	97.3	98.4	12.3	85.0	4.86
P_fFP_f	4	56.6	96.6	96.6	98.2	9.5	87.1	4.34
Jute cutting								
FP_f	2	54.1	98.9	97.9	98.4	19.0	71.4	3.69
FP_f	4	53.5	98.5	97.9	98.5	18.8	79.2	3.64
FP_f	6	52.8	98.2	97.2	98.6	16.2	81.4	3.16
P_fFP_f	4	48.7	98.1	97.1	98.7	12.8	86.3	2.98
Jute caddis								
FP_f	2	58.3	91.5	93.2	97.0	13.1	59.1	6.92
FP_f	4	54.1	93.3	95.8	97.5	12.3	59.5	6.05
FP_f	6	49.6	93.1	94.9	97.5	9.0	61.9	5.60
P_fFP_f	4	50.2	93.7	97.0	98.7	7.2	70.1	5.07

The loss of pulp yield in jute fiber was the lowest, and in jute cuttings was the highest. The loss of yield on bleaching was caused by saponification, delignification, and solubulization of pentosan and low molecular weight cellulose. All dissolving pulps met the specifications typical for market sulfite and prehydrolysis kraft pulps (Sixta and Borgards 1999). The degree of purification expressed as R-values and cellulose content were comparatively high for these pulps. The dissolving pulp in jute cuttings was highly pure (α-cellulose content: 98-99 %). The α-cellulose content in dissolving pulp from jute fiber was 96-97 % and from jute caddis was 92-94 %. There was no significant difference between FP_f and P_fFP_f stages pulping in the purity of dissolving pulp. It was observed from our earlier investigation that the dissolving pulp produced from jute is not as pure as wood in the conventional processes (Jahan 2008). So a formic acid process is suitable to produce dissolving pulp from jute. Figure 1 shows viscosity vs. cellulose content. High cellulose content can be achieved at the expense of a severe degradation of molecular weight at a higher temperature of prehydrolysis. Jute caddis showed scattered viscosity and alfa cellulose data.

R-values (Retention in alkali) in jute fiber, jute cuttings, and jute caddis were also comparatively high. The retention in alkali data R_{10} and R_{18} provide information on the low molecular weight carbohydrates (degraded cellulose and hemicellulose) in pulp. A 10% sodium hydroxide solution dissolves both degraded cellulose and hemicellulose (Retention in 10% alkali, R_{10}) whereas hemicellulose is soluble in an 18% sodium hydroxide solution (Retention in 18% alkali, R_{18}). Degraded cellulose (R_{18} minus R_{10}) in jute cuttings was very low, as compared to jute fiber and jute caddis. Degraded cellulose (R_{18} minus R_{10}) in pulp increased with decreasing viscosity and increasing pulp brightness (Figs. 2, 3).

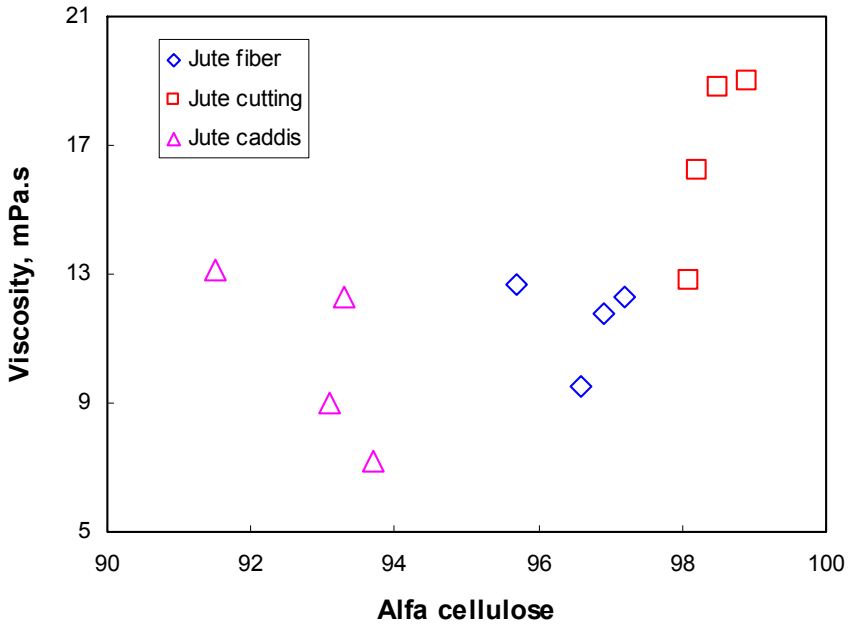

Fig. 1. Viscosity *vs* α-cellulose of jute fiber, cuttings, and caddis pulps by formic acid process

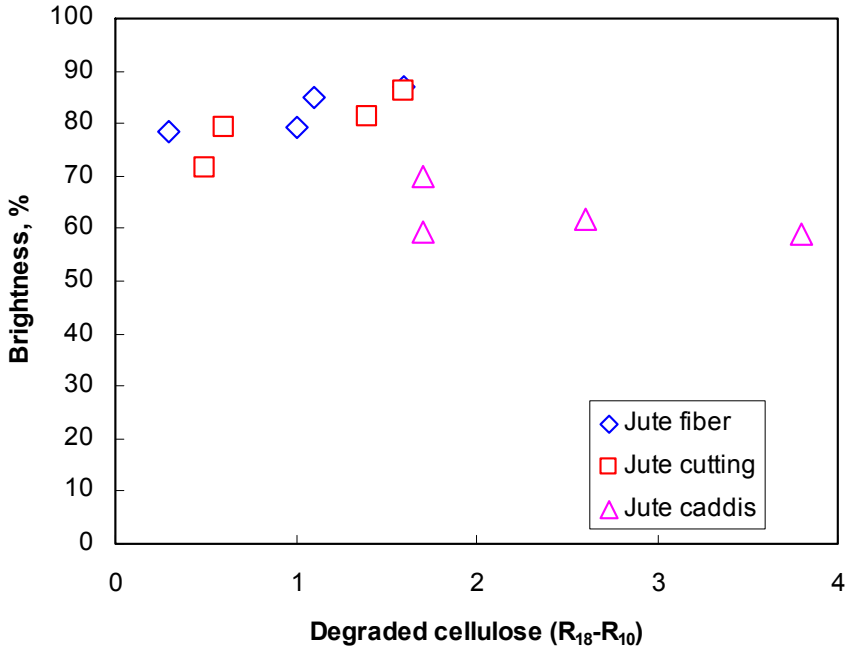

Fig. 2. Degraded cellulose *vs.* brightness of jute fiber, cuttings, and caddis pulps by formic acid process

Jute caddis contained higher degraded cellulose, while jute fiber and jute cuttings contained very low degraded cellulose. These results were quite different from conventional pulping of jute (Jahan et al. 2007; Jahan 2008). The alkali resistance, R18 was also closely related with xylan content. The R_{18} values increased when peroxide charge in peroxyformic acid increased or with introduction of an additional peroxyformic acid treatment. FP_f to P_fFP_f processes responded very well in alkaline peroxide bleaching. Both jute fiber and jute cuttings yielded above 80% brightness. But jute caddis responded poorly. The maximum brightness achieved was 70% in the P_fEP_f process. This may be explained by higher impurities and foreign material in caddis. It can be mentioned that caddis is produced as waste in mills after treating jute with fatty materials. Jute fiber and jute cutting showed very promising results in this process.

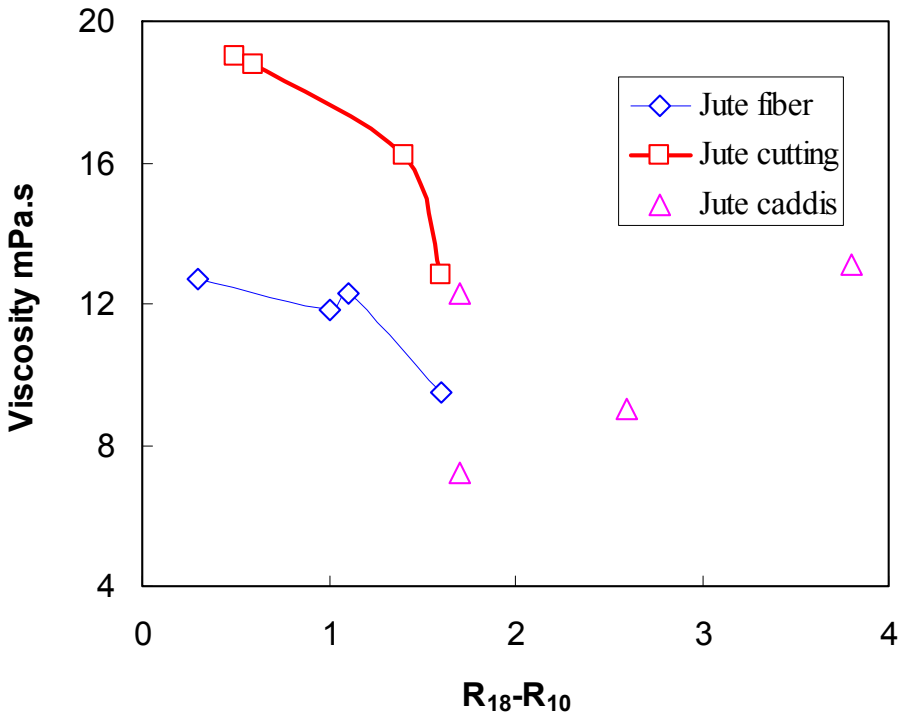

Fig. 3. Degraded cellulose *vs* viscosity of jute fiber, cuttings, and caddis pulps by formic acid process

The carboxyl group content was 4.3 to 6.3 meq per 100 g pulp in jute fiber, 3.0 to 3.7 meq per 100 g pulp in jute cuttings, and 5.1 to 6.9 meq per 100 g pulp in jute caddis pulp, depending on conditions. The lower value of carboxyl group content in jute cutting pulp indicates no or low oxidation in pulp, which is correlated with α-cellulose content and R18 - R10 values. The reduction of viscosity by means of drastic cooking conditions causes a decrease of carboxyl groups. Obviously this is due to the removal of xylan. Sixta (2000) observed a 3 mmole decrease of –COOH per kg of pulp, along with a 100 ml/g viscosity reduction. A similar observation was made in our previous investigation (Jahan et al 2008).

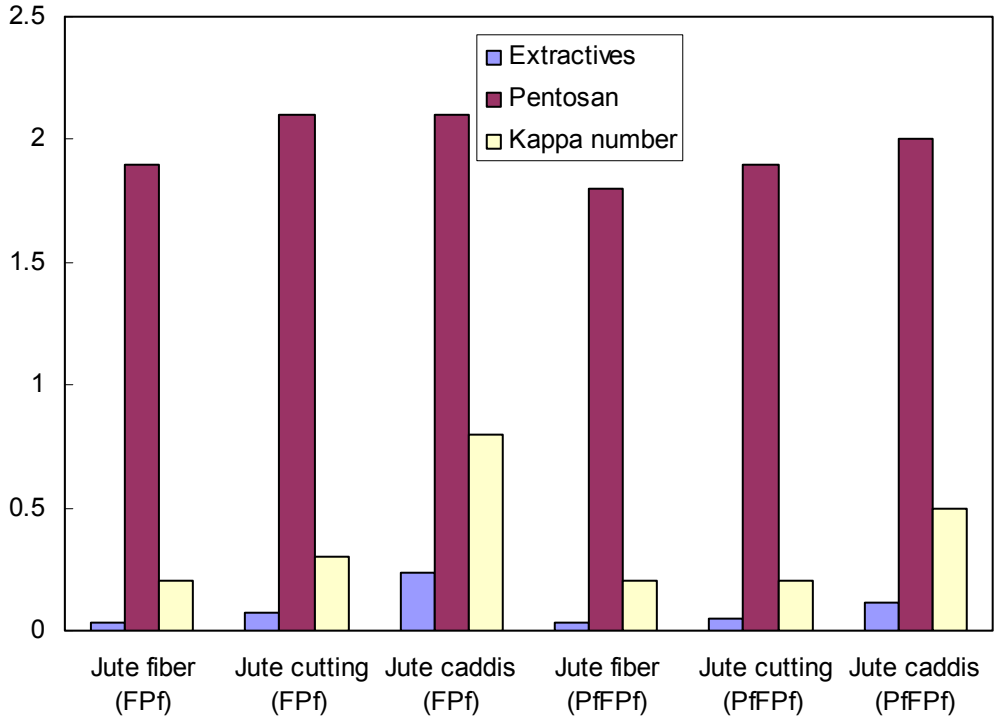

Fig. 4. Kappa number, extractives, and pentosan content of final pulps

Figure 4 shows the kappa number, extractives, and pentosan contents of the final pulps. Kappa number, extractives, and pentosan content were 0.2 to 0.8%, 0.03 to 0.24%, and 1.8 to 2.1 %, respectively. Jute caddis contained higher extractives than jute cutting and fiber. Extractives were reduced with the introduction a P_f stage at the beginning.

CONCLUSIONS

Jute fiber, jute cuttings, and jute caddis yielded pulp of high purity and low kappa number in a formic acid-peroxyformic acid process. Jute caddis showed inferior results as compared to jute fiber and cuttings. Kappa number and viscosity of the pulps were decreased and unbleached brightness increased with the increase of peroxide charge in peroxyformic acid. Introduction of additional peroxyformic acid in the beginning of pulping improved delignification and pup brightness with the sacrifice of pulp yield and viscosity. Alkaline peroxide bleaching responded very well for jute fiber and jute cutting pulps. The degree of purification expressed as R-values and cellulose content were comparatively high for these pulps. The dissolving pulp from jute fiber (α-cellulose content: 96 to 97%) and jute cuttings (α-cellulose content: 98 to 99%) was highly pure. Degraded cellulose (R18-R10) in jute fiber and jute cutting was very low as compared to conventional pulps. Jute caddis did not respond very well in terms of pulp brightness in this process. Introduction of an additional peroxyformic acid in the first stage improved final pulp brightness with the expense of pulp viscosity. Finally, we can conclude that FP_f

and PfFPf pulping processes followed by alkaline peroxide bleaching are suitable to produce dissolving pulp from jute fiber and jute cuttings.

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Appendix 1

Schematic Flow Diagram for the Production of Dissolving Pulp from Jute