

Studies on the effect of prehydrolysis and amine in cooking liquor on producing dissolving pulp from jute (*Corchorus capsularis*)

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Abstract Dissolving pulps are the raw materials for the production of many different end-products. Jute is a very good source of cellulose. In this investigation, jute fiber was subjected to pulping in soda process in order to produce dissolving pulp under different prehydrolysis conditions and compared with prehydrolysed kraft pulp from jute. An increase of the prehydrolysis temperature or H_2SO_4 in prehydrolysis liquor increased the α -cellulose content and decreased the viscosity of pulp. The effect of ethylenediamine in soda liquor was also investigated when producing dissolving pulp. Jute fiber produced pulp having 90–97% α -cellulose. Ethylenediamine in soda liquor produced pulp of higher yield, viscosity and higher α -cellulose content than that of prehydrolysis soda or kraft pulp. The α -cellulose content and viscosity were increased with the increase of amine in soda liquor. The kappa number of dissolving pulp from jute was very low (9–5), which indicated that less bleaching chemicals are required for bleaching. The bleachability of soda-ethylenediamine pulp was lower than prehydrolysed soda and kraft pulp in ECF bleaching sequences. The bleachability of soda-ethylenediamine pulp was improved at the sacrifice of pulp yield when prehydrolysis was done prior to pulping. The alkali solubility S_{10} and S_{18} were 4–9 and 2–4%, respectively.

Introduction

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 those days (Jahan et al. 2007). The chemical and morphological characteristics of jute favor it as pulping raw material (Nahar 1987).

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Therefore, many studies have been done on paper grade pulp from jute at home and abroad (Akhtaruzzamen and Shafi 1995; Jahan 2001; Roy et al. 1998). Retted jute fiber contains a very high α -cellulose and low hemicelluloses as compared to wood or other nonwood (Nahar 1987). So, it may be used in producing dissolving pulp.

Most chemical cellulose or dissolving pulp comes 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% pure cellulose. The end uses of dissolving pulp include cellophane and rayon, cellulose esters (acetates, nitrates, etc.), cellulose ethers (carboxymethyl cellulose, etc.), graft and cross-linked cellulose derivatives (Sjöström 1981). When producing dissolving pulp for making products such as carboxymethyl cellulose, viscose, cellulose film and sausage skin, determining the pulp quality is essential. The dissolving pulp quality depends on both properties of the raw wood material 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). For derivatization reactions, it is important to note that reactions with the hydroxyl groups on carbons two and three are kinetically favorable, while substitution on carbon six is thermodynamically more stable (Schlotter 1988; Krässig 1993). Both celluloses I and II have been found in pulp. Cellulose II is more thermodynamically stable than cellulose I. This may make the dissolving pulps with large proportions of cellulose II more resistant to heating than pulps with large proportions of cellulose I (Lennholm and Iversen 1995a, b). Dissolving pulp should have special properties, such as a high level of purity, uniform molecular-weight distribution and the reactivity and accessibility of the cellulose to chemicals (Krässig 1993). The structure and morphology of the fibers determine these properties. 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) endoglucanase treatment leads to a swelling of the cell wall and thus an increase in accessibility to solvents and reagents. Even though dissolving pulp is a highly purified pulp, it possesses some disadvantages, for example, a broad molecular weight distribution and a low viscosity at a given purity level (Sixta et al. 2004). Many attempts have been made to find a correlation between the hydroxyl group reactivity and the microstructure of the cellulose. Both X-ray diffraction and iodine sorption measurements are commonly used to determine the crystallinity and accessibility, respectively. The iodine sorption method measures the amorphous part or the accessible hydroxyl groups, whereas X-ray diffraction measures the crystalline fraction (Hessler and Power 1954; Racz et al 1996). It is not clear, how well these methods correlate with the reactivity in industrial production.

In recent years, various innovative pulping methods have been developed primarily in response to environmental considerations (Sixta et al. 2004; Vila et al. 2004; Kirci and Akgul 2002). New acidic pulping processes, such as acetosolv,

formacell, milox, promise to have superior potential regarding purification selectivity, e.g., expressed by the viscosity–pentosan relationship and specific investment costs (Pulps et al. 1999; Sixta et al. 2004). It has been reported in our earlier studies (Jahan and Farouqi 2000, 2001) that ethylenediamine (EDA) in soda liquor increased delignification and hemicelluloses dissolution of jute. MacLeod et al. (1984) also observed that the soda-EDA process dissolved the greatest amount of xylan from spruce. Therefore, EDA in soda liquor can produce high α -cellulose containing dissolving pulp from jute.

In this article, an effort was exerted to produce dissolving pulp from jute by prehydrolysed soda and kraft processes. The effect of ethylenediamine (EDA) in soda liquor on producing dissolving pulp was also assessed. The effect of acid and temperature on the prehydrolysis soda pulp was studied.

Experimental

Raw materials

Retted jute fiber was collected from the BJRI, Dhaka. It was sun-dried and cut to 2–3 cm in length. The moisture content of the raw materials was determined according to TAPPI Standard Methods (1953) (T 18 m-53). After determination of the moisture content of air-dried raw materials equivalent to 250 gm oven-dried (o.d.) was weighed separately in a polyethylene bag for subsequent cooking experiments.

Prehydrolysis

The prehydrolysis was carried out in an electrically heated stainless steel digester of 5 l capacity, rotating at 1 rpm. Water prehydrolysis was carried out either with or without the addition of sulfuric acid at 150 and 170°C. The jute to liquor ratio was 1:5. The time required to raise maximum temperature was 60 min. The liquor was drained after prehydrolysis. The prehydrolysate contained high amounts of sugars. It may be a valuable source of ethanol or other valuable chemicals (Rath et al. 2005). This will be studied in our next projects.

Cooking

All pulping experiments were performed in the same digester. The following parameters were kept constant in the soda process:

- active alkali charge was 18% on o.d jute
- liquor to fiber ratio was 5:1
- temperature was 170°C
- cooking time was 60 min

Two sets of experiments were carried out in soda-EDA process, one with prehydrolysis at 150°C and 0.25% H₂SO₄ and the other one without prehydrolysis.

The prehydrolysed jute was subjected to the soda-EDA pulping. The proportions of EDA in soda liquor were 10, 20, 30 and 40% (v/w), respectively. All other parameters were kept constant.

During prehydrolysis kraft cooking, the active alkali charge was adjusted to 18% Na₂O on oven dry wood and 25% sulfidity at 170°C for 60 min. Material to liquor ratio was 1:5.

Bleaching

Pulps were bleached in DoED₁ bleaching sequences. The bleaching was done in polyethylene bags. The kappa factor 0.22 was used in D₀ stage. The pH of bleach liquor was 2–2.5 in D₀. The bleaching continued for 60 min at 70°C. Alkaline extraction was carried out with 2% NaOH at 70°C for 60 min. The consistency was 5 and 10 in chlorine dioxide and extraction stage, respectively. In the final stage (D₁), half of the ClO₂ applied in the D₀ stage was used. The temperature was 70°C for 120 min.

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); carbohydrate (T249 cm00); α -cellulose (T 203 om-88); and alkali solubility S_{10} and S_{18} (T 235 cm-85). Alpha-cellulose is the pulp fraction resistant to a treatment in an aqueous solution containing 17.5% sodium hydroxide and indicates undegraded, high molecular weight cellulose content in pulp. Alkali solubilities S_{10} and S_{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 hemicelluloses (S_{10}) whereas hemicelluloses are soluble in an 18% sodium hydroxide solution (S_{18}). All pulp properties were analyzed in duplicate.

Results and discussion

Pulping

Soda process

To determine the optimum prehydrolysis temperature and H₂SO₄ concentration for getting high purity dissolving pulp, various experiments were carried out as shown in Table 1. A simple prehydrolysis (without H₂SO₄ and 60 min at 150°C) with water is insufficient for the thorough removal of hemicelluloses from jute in a simple soda process. Under identical cooking conditions, the cellulose content was raised when more severe prehydrolysis was applied (Table 1). Addition of H₂SO₄ in prehydrolysis regardless of temperature increased the α -cellulose content in pulp.

Table 1 Effect of H₂SO₄ in prehydrolysis and its temperature on soda process of jute

H ₂ SO ₄	Pulp yield (%)	Kappa number	Brightness (%)	Viscosity (mPa s)	α -cellulose
150°C					
0	52.9	9.0	36.1	9.0	91.3
0.25	52.6	6.1	36.2	8.3	92.5
0.50	46.9	5.8	41.9	7.2	93.3
0.75	45.4	5.3	42.3	6.8	95.3
1.0	45.3	4.7	43.2	6.3	95.4
170°C					
0	37.8	5.6	37.4	4.7	93.9
0.25	35.4	5.4	37.4	4.3	95.1
0.50	34.2	5.1	39.4	4.0	95.4
0.75	33.8	4.7	41.1	3.7	95.8
1.0	31.7	3.9	41.3	3.4	96.2

Pulping condition: NaOH 18%, temperature 170°C, time at max temp 60 min and liquor ratio 5

At a given temperature (150 or 170°C), the addition of 1% sulphuric acid increased the alpha-cellulose content by 4.1 and 2.3% (from 91.3 to 95.4 and from 93.9 to 96.2) at the expense of a severe pulp yield drop.

However, the jute was satisfactorily delignified under any prehydrolysis conditions. Pulp obtained after prehydrolysis at 150°C and without H₂SO₄ followed by pulping achieved a kappa number of 9.0. The kappa number was reduced to 6.1 when 0.25% H₂SO₄ was added in prehydrolysis under similar conditions. The delignification degree of pulping was increased with increasing H₂SO₄ concentration in prehydrolysis (Table 1).

The pulp yield dropped sharply from 52.9 to 45.3% when H₂SO₄ concentration was increased from 0 to 1.0% at 150°C during prehydrolysis. The pulp yield severely dropped (31.7–37.8%) when the prehydrolysis temperature was 170°C. The delignification selectivity was lower at 170°C (Fig. 1). The pulp yield was 11% lower at 170°C than at 150°C at kappa number 5. The dissolving pulp yield from jute by acidic prehydrolysis soda process was better than the corresponding pulp yield from bamboo or jute stem at the same level of purity and viscosity (Bhowmic 1999). The main target parameter in dissolving pulp production besides purity is viscosity as a measure of the degree of polymerization (D.P.) of the cellulose. In dissolving pulp production, the viscosity must always be considered in relation to purity, i.e. cellulose content of the pulp. In this case, a cellulose content of 91.3% was obtained under mild prehydrolysis conditions (without H₂SO₄ and 60 min at 150°C), which is the lower limit of the satisfactory range. Figure 2 shows the viscosity versus the cellulose content. In each case, the prehydrolysis conditions were variable and pulping was carried out under identical conditions (Alkali 18% as NaOH, cooking temperature 170°C, cooking time 60 min and liquor ratio 1:5). High cellulose content can be achieved at the expense of a severe degradation of molecular weight at a higher temperature of prehydrolysis. The effect of prehydrolysis on the purification efficiency or on the removal efficiency of short-chain carbohydrates, etc.

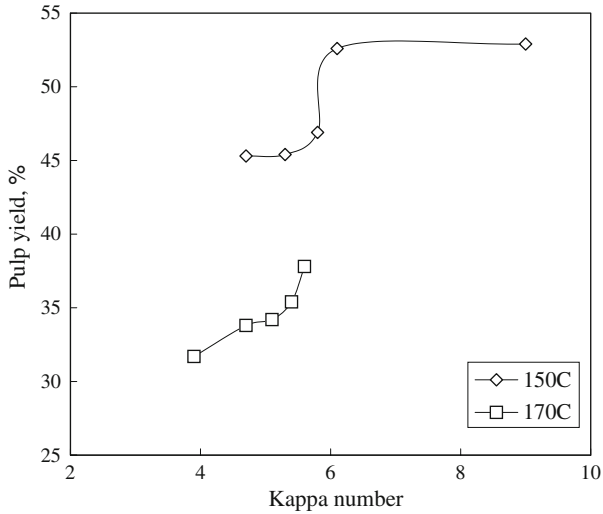


Fig. 1 Effect of prehydrolysis temperature on the delignification of jute

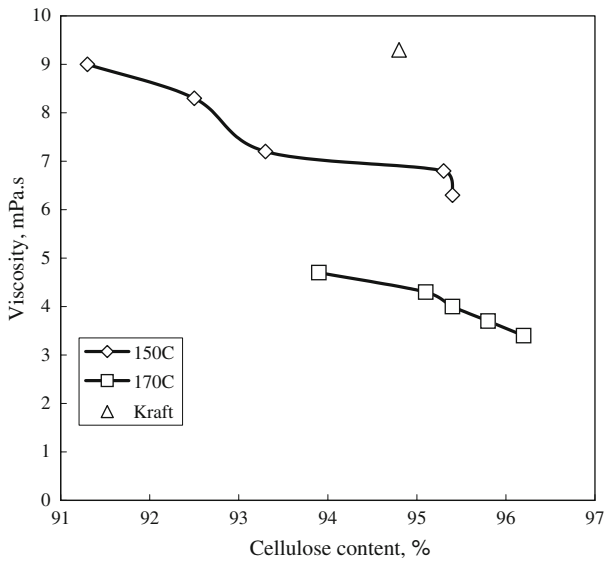


Fig. 2 Viscosity versus cellulose content in soda and kraft pulp

is determined by the intensity of prehydrolysis, which may be calculated by using the *P*-factor concept, which is expressed by the prehydrolysis time and temperature as a single variable and is defined as

$$P = \text{Int} (t_0 - t) k_{\text{rel}} dt$$

where k_{rel} is the relative rate of acid-catalyzed hydrolysis of glycosidic bonds. The activation energy was increased when the prehydrolysis temperature increased.

The α -cellulose content was increased with the increase of H_2SO_4 concentration in prehydrolysis (Fig. 2). The addition of strong mineral acid contributes to the prehydrolysis intensity, meaning that at a given P -factor, the addition of e.g., H_2SO_4 enhances the prehydrolysis efficiency (Masura 1987). Consequently, to obtain a high degree of polymerization (D.P.) of cellulose a lower degree of purity of cellulose has to be accepted. Pulp of this kind is only suitable for dissolving grade pulp, where the viscosity requirements are in the low to medium range (e.g., for rayon stable, rayon filament, CMC technical use, cellophane, etc.) (Sixta et al. 1994). Compared to a reference pulp produced by prehydrolysis kraft process, prehydrolysis soda pulp showed poorer properties (Fig. 2). The pulp yield and kappa number were also better (data not shown).

Soda-EDA process

The jute was prehydrolysed with 0.25% H_2SO_4 at 150°C for 60 min and cooked with varied proportion of ethylenediamine (EDA) in 18% soda liquor. The pulp yield and kappa number were decreased with increasing EDA in soda liquor (Table 2). Julien and Sun (1979) also observed better delignification when EDA was added in soda liquor. The presence of amine in soda liquor altered the molecular weights of the dissolved lignin fraction and delignification is associated with negative reduction potential of pulping additive (Kubes and Bolker 1978). The viscosity of pulp was improved when EDA was used. The α -cellulose content was also increased with the addition of EDA. At 40%, EDA in soda liquor produced pulp of 95.3% α -cellulose content with viscosity of 11.7 mPa.s. Helmy and State (1991) observed increasing pulp yield, α -cellulose content and the D.P. and decreasing pentosan of bagasse pulp with the increase of EDA in soda liquor. Slight increase in viscosity and the substantial increase in alpha-cellulose could also be attributed to the selective removal of hemicelluloses, e.g., xylan that is rather more likely than a “stabilization” of the alpha-cellulose as indicated by Kubes and Bolker (1978) and

Table 2 Effect of amine and prehydrolysis on the soda process of jute

EDA	Pulp yield (%)	Kappa number	Brightness (%)	Viscosity (mPa s)	α -cellulose
Without prehydrolysis					
10	65.3	15.6	35.8	15.4	90.8
20	64.4	15.1	36.7	16.0	91.7
30	64.0	14.8	36.4	15.9	92.7
40	63.4	14.0	36.8	16.3	93.1
Prehydrolysed at 150°C H_2SO_4 0.25%					
10	50.0	6.7	36.8	9.4	93.7
20	49.8	6.1	37.2	9.6	94.1
30	48.6	5.8	36.9	10.0	94.7
40	48.3	5.4	37.7	11.7	95.3

Pulping condition: NaOH 18%, temperature 170°C, time at max temp 60 min and liquor ratio 5

Kubes et al. (1978). The slightly decreasing trend in pulp yield with increasing EDA addition may be explained by the dissolution of hemicelluloses (Jahan and Farouqui 2000). Table 2 also shows the effect of EDA in soda liquor on jute pulping without prehydrolysis. The pulp yield was 63–65% that was about 15% higher than prehydrolysed soda-EDA process. The high yield combined with high alpha-cellulose content has to be attributed to high cellulose yield, high viscosity and possibly the preservation of a high amount of high-molecular weight xylan. The α -cellulose content in pulp was increased with increasing EDA in soda liquor. Pulp yield was decreased slightly with increasing EDA. This may be attributed to the dissolution of hemicelluloses. The viscosity of soda-EDA pulp without prehydrolysis was 15–16 mPa s, which was 3–6 mPa s higher than prehydrolysed soda-EDA pulp.

Bleaching

Pulps obtained under different conditions in soda and soda-EDA processes were subjected to DED bleaching. All pulps were bleached under identical conditions. Increasing H_2SO_4 concentration in prehydrolysis liquor or increasing prehydrolysis temperature improved pulp bleachability (Table 3). Pulp obtained under simple water prehydrolysis (without H_2SO_4 , 150°C) followed by pulping had a brightness of 78.3%, but increased to 82.2% when 0.25% H_2SO_4 was added during prehydrolysis. The solubility in 10% alkali (S_{10}) was increased with increasing H_2SO_4 in prehydrolysis liquor or prehydrolysis temperature. The higher S_{10} value indicated that the H_2SO_4 attacked both cellulose and hemicelluloses of jute. Therefore, the hemicelluloses content in pulp was decreased (S_{18}) with increasing H_2SO_4 . The viscosity of bleached pulp was decreased from 7.1 to 3.8 mPa s at 150°C and 6.1 to 3.4 mPa s at 170°C with increasing H_2SO_4 from 0 to 1.0% in prehydrolysis liquor.

Table 3 Effect of H_2SO_4 in prehydrolysis and its temperature on the bleaching of soda pulp

H_2SO_4	α -cellulose	S_{18}	S_{10}	Viscosity (mPa s)	Brightness (%)
150°C					
0	91.6	7	10.2	7.2	78.3
0.25	92.7	6.9	11.3	5.3	82.2
0.5	92.9	6.7	12.1	4.9	82.3
0.75	93.5	6.3	12.3	4.4	82.6
1	95.2	6.4	12.7	3.8	82.9
170°C					
0	93.6	5.1	13.1	6.1	80.3
0.25	94.8	4.7	13.7	4.8	81.7
0.5	95.7	4.1	13.7	4.4	83.4
0.75	96.1	3.8	13.9	3.9	84.9
1	96.1	3.1	14.1	3.4	85.5

Table 4 Effect of amine and prehydrolysis on the bleaching of soda-EDA pulp

EDA	α -cellulose	S_{18}	S_{10}	Viscosity (mPa s)	Brightness (%)
Without prehydrolysis					
10	92.4	5.3	8.1	14.1	72.3
20	93.2	5.1	7.8	14.5	73.4
30	93.9	4.6	7.3	14.7	72.8
40	94.2	4.4	6.9	15	72.6
Prehydrolysed at 150°C H ₂ SO ₄ 0.25%					
10	89.2	6.4	10.9	7.5	73
20	95.2	3.3	7.8	7.9	75.8
30	95	3.1	4.9	8.2	76.4
40	96.2	2.3	4.4	8.5	79.5

Table 4 shows the bleachability and dissolving pulp properties of soda-EDA pulp. α -cellulose and viscosity were increased and alkali solubility (S_{10} and S_{18}) decreased with increasing EDA in soda liquor. Soda-EDA pulp showed poor bleachability (Table 4). There was no change of brightness within the applied EDA in soda liquor. Prehydrolysed soda-EDA pulp showed a slightly better brightness than the one without prehydrolysed soda-EDA pulp. Lower bleachability of soda-EDA pulp is under investigation in our laboratory.

Figure 3 shows the comparison of soda (obtained from prehydrolysed at 150°C and 0.25% H₂SO₄ concentration) and soda-EDA (obtained at 20% EDA with and without prehydrolysis) process for producing dissolving pulp from jute. Soda-EDA pulp without prehydrolysis showed the highest and soda the lowest viscosity (14.5 and 5.3 mPa.s) among these three pulps. Prehydrolysed soda-EDA pulp had the highest and soda the lowest α -cellulose, which indicates the better solubility of hemicelluloses and stabilization of α -cellulose in soda-EDA process. S_{10} – S_{18} values indicate the content of degraded cellulose with a degree of polymerization between approximately 50 and 150 (Hinck et al. 1985). Soda-EDA pulp showed better S_{10} – S_{18} values. These results are consistent with the data of α -cellulose and viscosity. The main problem of soda-EDA process was bleachability. Unbleached brightness of soda-EDA pulp was lower, which indicates its lower bleachability. Using the same kappa factor, soda pulp gave 82.2% brightness that was 8.8% unit higher than soda-EDA pulp (with an EDA concentration of 20%).

The amount of residual hemicelluloses (Table 5) reflects the degree of purity, which is crucial for the production of rayon fibers and products made from cellulose acetate. In the case of hardwood, the major hemicelluloses component is 4-*O*-methyl glucuronoxylan, which can be easily identified by xylose. Prehydrolysed-soda-EDA pulp showed the highest purity, when the residual xylose content was 3.0% (Table 5). The xylose content in jute was lower as compared to hardwood as shown in Table 5. However, the produced dissolving pulp from jute is not as pure as wood. This is maybe due to some part of hemicelluloses being entrapped within the cellulose matrix and some part was associated with the lignin–carbohydrate complex (Gübitz et al. 1998).

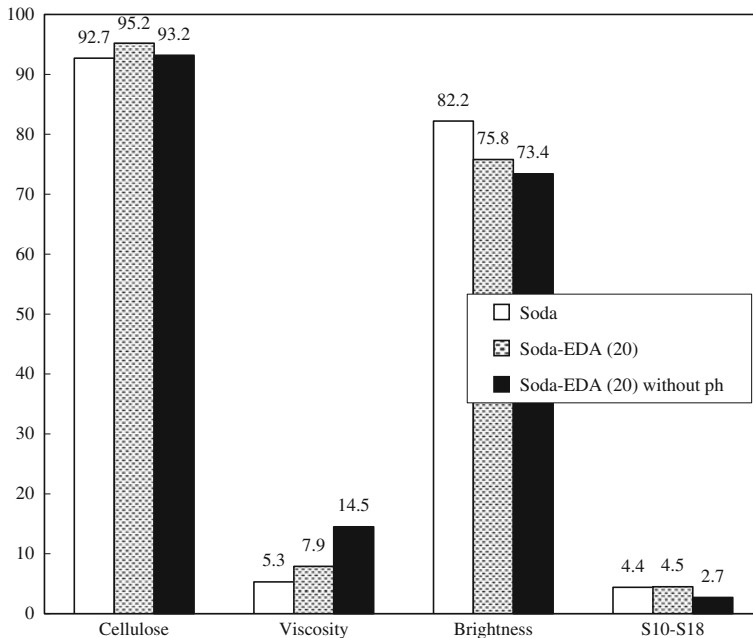


Fig. 3 Comparison of dissolving pulp produced by prehydrolysed soda, soda-EDA, without prehydrolysed soda-EDA process

Table 5 Carbohydrate composition of jute fiber and dissolving pulp

Sample	Neutral sugars					
	Rhamnose	Arabinose	Xylose	Mannose	Galactose	Glucose
Raw jute ^a	0.41	1.15	12.24	1.66	1.73	69.92
Soda	0.08	0.41	5.7	0.74	0.81	93.1
Soda-EDA	0.04	0.33	3.0	0.79	0.71	94.9
Soda-EDA without PH	0.04	0.31	4.6	0.78	0.68	96.5

^a Jahan et al. (2008)

Conclusion

The aim of the present study was to investigate the ability of extracting hemicelluloses of ethylenediamine (EDA) in soda liquor and the effect of prehydrolysis on soda-EDA pulp. Prehydrolysis temperature and H_2SO_4 had great influences on the properties of dissolving pulp. An increase of the prehydrolysis temperature or H_2SO_4 in prehydrolysis liquor increased the α -cellulose content and decreased the pulp yield and kappa number and viscosity of pulp. Jute fiber prehydrolysis at $170^\circ C$ had a pulp yield of 37.8% only. The pulp yield was further decreased to 31.7% when 1.0% H_2SO_4 was used in prehydrolysis. EDA in soda liquor had also a good effect on dissolving hemicelluloses. The α -cellulose content and viscosity were increased with the increase of amine in soda liquor. EDA in soda

liquor produced pulp of higher yields (63–65%), viscosity (14–16 mPa s) and α -cellulose (91–93%) content when prehydrolysis was not used. However, the bleachability of soda-EDA pulp was very poor. Soda-EDA pulp without prehydrolysis showed a brightness of about 72% only. The bleachability of soda-EDA pulp was improved with prehydrolysis. The α -cellulose content in prehydrolysed soda-EDA (40%) pulp was 96.2%, where the pulp brightness was 80%. EDA is a highly protective agent for α -cellulose and extracting agent for hemicelluloses, thus better viscosity was obtained. Degraded cellulose (S_{10}) in pulp was lower in soda-EDA pulp than that of soda pulp.

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