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Studies on the spent liquor recirculation during soda-amine pulping of jute

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Spent liquor of soda-monoethanolamine and soda-ethylenediamine processes were recirculated with white liquor in different proportions and its effect was studied on pulp properties and solid content in spent liquor. Pulp yield, kappa number, and solid contents in spent liquor were increased with increasing spent liquor recirculation. These increases were higher in soda-ethylenediamine process than that in soda-monoethanolamine process. There was not much significant change in strength properties up to 35 per cent of the spent liquor recirculation. The bleachability of the spent liquor recirculated pulp did not change much.

Keywords: Spent liquor, Jute, Soda-amine pulping, Pulping

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Introduction

Jute is the internationally recognized name of the bast fibre obtained from stalks of plants of the genus *Corchorus* of the Tiliaceae family. It is the most important long vegetable fibre worldwide.

Once jute was known as golden fibre of Bangladesh because of its contribution in its economy. Nowadays, traditional uses of jute have declined domestically and abroad due to the introduction of synthetics. Therefore, many studies have been done on alternative uses of jute[1]. The major alternative use of jute is pulping. Various studies on the pulping of jute based raw materials have been done for the past two decades, within the country and abroad[2-4]. Laboratory studies have shown few problems in jute pulping by conventional processes. These problems include lumping of the cooked materials, causing difficulties in digester blowing, washing, etc. Thus, efforts have been made on the alternative pulping processes for jute.

During the past two decades, many new and modified chemical pulping processes have been devolved[5] and some are already in commercial use[6]. In fact, most of the processes might be termed "soda-additive and categorized as containing inorganic additive, organic additive or a combination of both.

The additive can change the rate and degree of delignification and can also influence the mechanical and optical properties of the pulps.

Ethylenediamine (EDA) or monoethanolamine (MEA) in soda liquor increased the delignification and produced pulps better than that of kraft pulp.

Accelerated delignification in the presence of amine in soda liquor was associated with an apparent decrease in the molecular weight of the dissolved lignin[7]. The magnitude of its effect was related to its negative reducing potential. The rate of delignification coorelated with the redox potentials of the spent liquors[8].

Soda-amine processes produce higher pulp yield from jute. The tear index of soda-amine pulps is high. Due to high yield, the solid content in soda-amine spent liquor is lower, thus cost of fuel for heating increases.

Ogiyama *et al.*[9] have described that soda-amine spent pulping liquor had been recycled up to five times without change in pulp quality but no data of paper making properties is reported.

Information is available on the effect of recycling the spent liquor on the pulp quality of softwood and hardwood. No information is available on the non-wood pulping by soda-amine processes. Therefore, efforts were made on reusing spent liquor of soda-ethylenediamine (EDA) and soda-monoethanolamine (MEA) pulping of jute and its impact on pulp quality and bleachability.

Experimental Procedure

Raw Material

Jute fibre of *Chorcorus capsulares* variety was collected from the Bangladesh Jute Research Institute,

Dhaka. The dried jute was chopped by hand to small pieces of about 2-3 cm length and foreign materials such as, grass roots were removed by hand. The moisture content of jute was determined separately, according to TAPPI Standard Methods (T 18m-53). After determination of the moisture content of air dried jute fibre equivalent to 1000g oven dried (od) was weighed separately in a polyethylene bag for subsequent cooking experiments.

Cooking Liquor

The cooking liquor was prepared by using reagent grade NaOH and ethylenediamine (EDA) or monoethanolamine (MEA).

Cooking

All pulping experiments were performed in an electrically heated autoclave of 20 liters capacity, made of stainless steel, rotating at 2 rpm, and fitted with thermostat. The following parameters were maintained:

- EDA or MEA, 40 per cent on od jute fibre.
- Liquor to fibre ratio, 5:1.
- 15 min to raise the temperature to 70°C from room temperature and 90 min to further raise the temperature from 70 to 170°C.
- Cooking time at 170°C, 1h.

Three different doses of alkali (14, 16 and 18 per cent) were used to obtain different degrees of delignification. The spent liquor from these cooking was collected and used in the ratio of 25, 35, and 50 per cent (v/v) with the substitution of fresh liquor in subsequent cooking.

At the end of the cooking the internal pressure was reduced to atmospheric pressure by venting before opening the digester. Recovered pulps were washed thoroughly to remove the residual chemicals. After washing, pulps were disintegrated. Hard cooked materials were defibrated in a disc refiner. After defibration or disintegration the pulp was screened on a flat vibratory screen with 0.38mm slots. After the screening, the pulp was passed on a screw press to remove excess water. Then the pulp yield was determined as percentage on od raw material.

Bleaching

Pulps obtained from soda-amine processes, comprising different proportion of spent liquor (0, 25 and 35 per cent) were bleached in DED bleaching sequences the kappa factor being 0.20 and at 70°C for 2h in chlorine dioxide stage. The consistency was 7 per cent and pH at 3-5 by adding H_2SO_4. In the extraction stage, pulp was extracted by 2 per cent alkali at 70°C for 1h.

Evaluation of Pulps

Jute fibre pulps were beaten in a Valley beater. The samples were collected at different ^{0}SR and hand sheets of about 60g/m^2 were made in a Rapid Kothen Sheet Making Machine according to German Standard methods no. 106. The sheet were tested for tensile (T 404os 61), burst (T 403m 53), and tear strength (T 414m-49), according to TAPPI Standard methods. Viscosity of the bleached pulp pulps was determined according to T-230.

Results and Discussion

The effect of different proportions of spent liquor recirculation on pulp yield and kappa number is shown in Table 1. Pulp yield and kappa numbers were increased with increasing spent liquor recirculation. The increased concentration of dissolved lignin and hemicellulose in the black liquor may be the reason for higher pulp yield and kappa number. Pulp yield was higher in soda-EDA process than that of soda-MEA process. This may be attributed to the dissolution of more hemicellulose in soda-EDA process[10], dissolved hemicellulose was grafted during recirculation of spent liquor. At 35 per cent spent liquor recirculation in 16 per cent alkali, soda-EDA process produced 8.8 per cent higher yield whereas in soda-MEA process it was 1.5 per cent. Bose *et al.*[11] also observed higher yield in NS-AQ pulping of jute with recycling of spent liquor. Pazukina *et al.*[12] showed no change in yield during alkaline sulphite pulping of Aspen and Spruce. Aravamuthan *et al.*[13] have observed definite increase in yield with increased recycling of black liquor during soda-AQ and kraft pulping of hardwoods. Similar observation was reported for modified bisulphite pulping of hardwoods[14].

The kappa number of the pulps increased with increasing spent liquor recirculation. This increase was higher in the case of lower alkali charge in both soda-EDA and soda-MEA processes. Similar observation was reported in NS-AQ pulping of jute[11]. The increase in kappa number may be due to the redeposition of dissolved lignin on the fibre surface and lignin condensation. Fig. 1 shows the selectivity of jute delignification during soda-EDA and soda-MEA spent liquor recirculation in respect to spent liquor proportion at 16 per cent alkali. It is evident

Table 1— Pulping characteristics of soda-amine with recycled spent liquor

(a) Soda-EDA

Alkali, per cent	Spent liquor recycled, per cent (v/v)	Pulp yield, per cent	Kappa number	Solid content, per cent	Brightness, per cent
14	0	62.4	20.1	7.1	42.5
14	25	66.8	29.4	8.3	42.3
14	35	70.9	51.2	9.2	42.1
14	50*	75.1	64.1	10.1	41.5
16	0	60.7	16.1	9.3	46.5
16	25	65.1	20.5	10.9	46.2
16	35	69.5	28.9	11.2	46.1
16	50*	73.1	50.8	11.7	46.0
18	0	58.3	14.5	10.7	46.8
18	25	59.9	19.1	11.3	46.8
18	35	66.1	25.5	12.1	46.2
18	50*	68.5	33.7	12.8	45.7

(b) Soda-MEA *Mechanical refining

14	0	65.3	22.4	6.3	44.2
14	25	67.9	24.1	7.1	44.1
14	35	70.1	38.6	8.9	43.6
14	50*	72.1	52.4	9.7	43.0
16	0	64.9	18.9	8.5	47.8
16	25	65.1	22.0	9.9	47.6
16	35	66.4	24.1	10.6	47.2
16	50*	69.3	48.4	11.0	46.3
18	0	61.6	16.7	8.9	47.9
18	25	63.7	20.8	10.5	47.3
18	35	65.1	22.3	12.1	47.0
18	50*	67.8	29.8	12.6	46.5

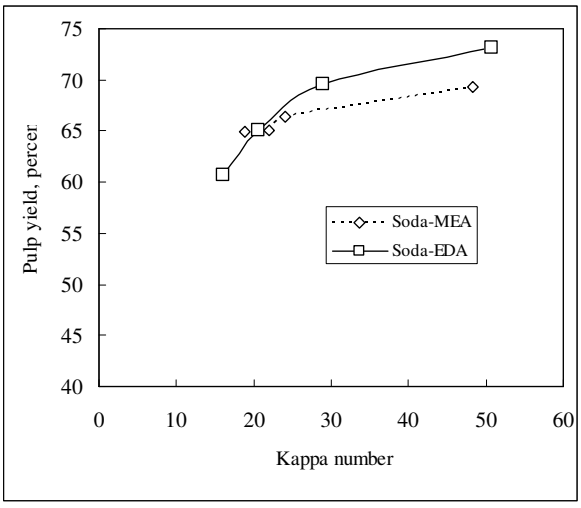

Fig. 1—Selectivity of delignification of jute in respect to recirculation of soda-amine liquor (16 per cent alkali)

that soda-EDA was more effective than that of soda-MEA liquor.

The solid content in the black liquor was increased with the increase of liquor recirculation (Table 1) in each of alkali charge. The solid content in soda-MEA spent liquor was lower than that of soda-EDA spent liquor. This may be explained by the higher yield in soda-MEA process. Solid content in the spent liquor of both of soda-amine processes were lower than that of NS-AQ pulping of jute[11]. There was no change of unbleached brightness on the spent liquor recirculation during soda-amine of jute (Table 1). But Mattson has observed[15] no adverse effect of spent liquor recycling on unbleached brightness. This difference may be attributed by the difference in structure of residual lignin of soda-amine pulp from soda, kraft or sulphite pulps.

Table 2 shows the effect of spent liquor recirculation on the papermaking properties of soda-amine pulps. No significant change was observed up to 35 per cent spent liquor recirculation. At 50 per cent liquor recirculation properties were detoriated. At lower alkali charge this effect was pronounced. Tear index of soda-amine pulp retained their superiority even after spent liquor recirculation (Fig. 2).

In order to determine the bleachability of pulps during recirculation of spent liquor, pulps were bleached by DED sequences. The kappa factor 0.16 was used in all pulps bleachings. Soda-EDA pulp

Table 2 — Physical properties of soda-amine pulps with recycled spent liquor

Alkali, per cent	Spent liquor recycled, per cent (v/v)	Breaking length, km	Tear index, kPa.m^2/g	Burst index, mN.m^2/g
(a) Soda-EDA				
14	0	7911	22.4	4.3
14	25	7581	20.9	4.1
14	35	7327	19.3	3.4
14	50	6523	18.1	3.1
16	0	8278	22.6	4.9
16	25	8191	20.8	5.3
16	35	8107	19.8	5.2
16	50	7729	18.7	4.3
18	0	8231	22.1	4.3
18	25	8109	20.9	4.4
18	35	8010	19.8	4.1
18	50	7734	18.8	3.7
(b) Soda-MEA				
14	0	8087	19.8	4.7
14	25	7668	18.3	4.4
14	35	7317	18.1	4.0
14	50	6629	17.6	3.1
16	0	8578	21.9	5.3
16	25	8298	20.0	5.1
16	35	8133	19.8	4.8
16	50	7824	18.9	4.5
18	0	8596	21.2	5.1
18	25	8371	19.5	5.0
18	35	8082	18.9	4.9
18	50	7853	18.1	4.7

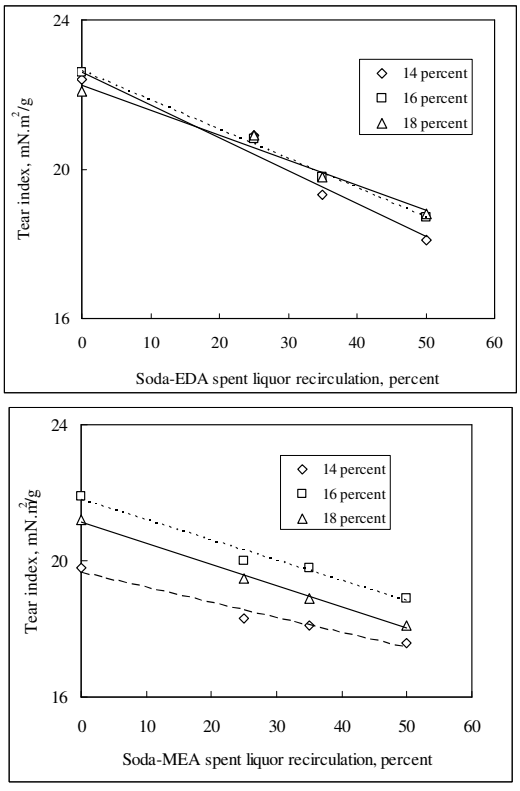

Fig. 2 — Effect of liquor recirculation in soda-amine pulping of jute on tear index

exhibited higher brightness value than that of soda-MEA pulp. Spent liquor recirculation did not adversely affect the brightness of soda-amine pulps. Soda-MEA pulp showed 82.02 per cent brightness when no liquor recirculation was used. When 35 per cent spent liquor was used, pulp produced of 80.54 per cent brightness. In soda-EDA pulp, no change of brightness was observed on liquor recirculation. Brightness of soda-EDA pulp was higher than that of soda-MEA pulp. This was may be due to more hemicellulose content in soda-MEA pulp than that of soda-EDA pulp. Spent liquor-recalculated pulp in soda-EDA showed little decrease in viscosity. This may be explained by the adsorption or grafting of lignin and hemicellulose in spent liquor recirculation. Removal of this lignin during bleaching, caused degradation cellulose. The soda-MEA pulp did not show any change in viscosity on spent liquor recirculation (Table 3).

Conclusions

Pulp yield and kappa numbers were found to significantly increase with spent liquor recirculation. The increase was lower at higher alkali charge. The solid content in spent liquor was increased with increasing spent liquor recycled. At higher alkali charge, there was no significant change in strength

Table 3 — Brightness and viscosity of soda-amine pulp with recycled spent liquor

Liquor recirculation per cent	Soda-MEA pulp		Soda-EDA pulp	
	Brightness, per cent	Viscosity, mPa.s	Brightness, per cent	Viscosity, mPa.s
0	82.02	30.3	85.02	35.1
25	81.15	30.1	85.00	32.1
35	80.54	30.4	85.01	32.2

properties during spent liquor recirculation. The brightness and viscosity of the recirculated spent liquor bleached pulp were almost similar to 100 per cent white liquor pulp. Soda-EDA process responded well on spent liquor recirculation than that of soda-MEA process.

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