ABSTRACT

Neutral sulfite anthraquinone pulping produces kraft-like pulp with a higher yield. This results in a spent liquor of low solids content. Recirculation of the pulping spent liquor substantially increases the solids content. As such, the fuel value of the liquor is enhanced, resulting in reduced fuel cost in the evaporator section. However, the reuse of spent liquor for pulping retards delignification with a minor change in pulp yield and quality.

Application:

Increasing the recirculation of spent liquor leads to a higher kappa number with only a minor change in pulp yield.

Effect of spent liquor recirculation on NS-AQ pulping of jute

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JUTE (CORCHORUS CAPSULARIES) HAS traditionally been a major source of exports for the jute-producing countries. However, the use of jute has been steadily declining in recent years, mainly due to its substitution by synthetic fibers in developed countries. The major decline in jute exports from developing countries has accelerated research to diversify the use of jute for value-added products.

The conservation of forests and increasing environmental awareness have increased research interest in new renewable fibrous resources and less toxic pulping and bleaching processes. A wide variety of nonwood plant fibers, i.e., bamboo, bagasse, and straw, are used for papermaking. Research works on the chemical pulping of jute plants *(1)*, bast fiber *(2)*, and core *(3)* have identified jute bast fiber as a promising fibrous raw material for pulp manufacture by the neutral sulfite anthraquinone (NS-AQ) process.

The NS-AQ process produces pulp with 6–12% higher yield on oven-dry (o.d.) wood compared to the kraft process *(4, 5)*. The papermaking properties of NS-AQ pulp are equivalent to its kraft counterparts *(4, 5)*. The higher yield results in spent liquor of low solids content and, thus, an increased cost of fuel for heating. However, reuse of the spent liquor for pulping is an approach used to increase the solids content of the liquor.

Mattson observed that there is an adverse effect of recycling on permanganate number and unbleached pulp brightness *(6)*. However, the recycling of black liquor did not appear to have a deleterious effect on the strength properties of kraft pulp from southern pine *(7)*. Sjoblom *et al. (8)* compared the results of a continuous-flow process and the corresponding batch process for pine, and concluded that the selectivity could be substantially affected by the dissolved organic matter in the cooking liquor. The increased concentration of dissolved lignin and hemicellulose in the black liquor increases the chance of redeposition of these materials on the fiber surface. In addition, lignin condensation may occur due to the higher lignin amount present.

Although information is available regarding the effect of recycling the spent liquor on the pulp quality of softwood and hardwood, the information related to nonwood fibers is scarce. Therefore, it is necessary to study the effects of reusing spent liquor on the delignification of nonwood fiber and the strength development characteristics of the pulps.

EXPERIMENTAL

Raw materials

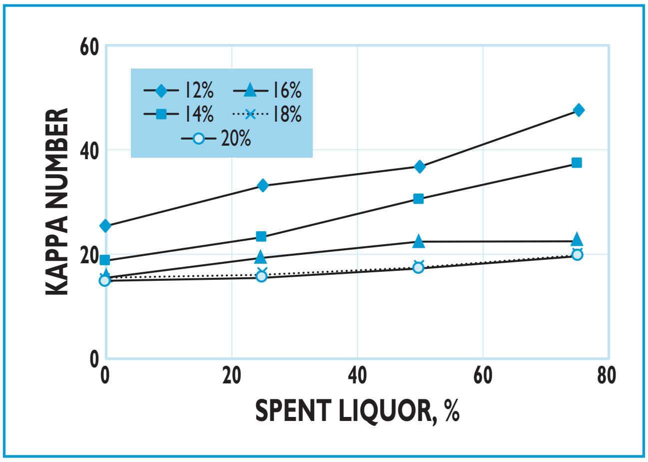
Air-dried jute cuttings (bast fiber) from white-B jute *(Corchorus capsularies)* were used as the fibrous raw material. The material was contaminated with unretted scales. The scales were allowed to remain in the raw materials, as it is not possible to remove them in an industrial setting with existing technology. The jute cuttings were mixed thoroughly to eliminate variability in the raw materials.

Pulping

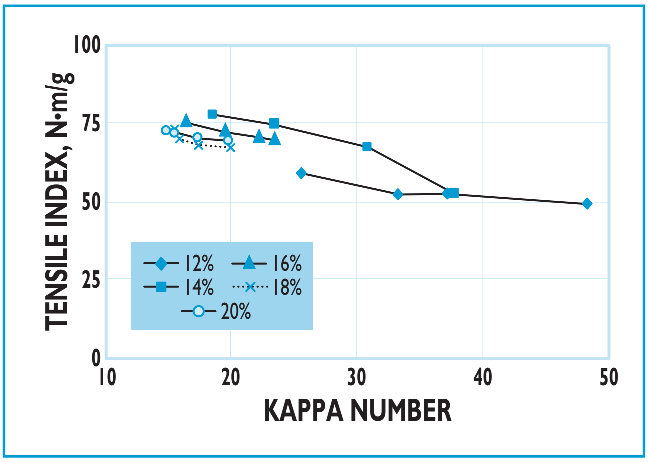
Pulping experiments were carried out with 250 g of jute on an o.d. basis in 2 L stainless steel autoclaves heated in a temperature-controlled air bath. Analytical grades of Na_2SO_3, Na_2CO_3, and anthraquinone (AQ) were used. The following parameters were kept constant:

- liquor-to-fiber ratio: 5:1
- AQ dose: 0.1% on o.d. jute
- alkali ratio: 0.83
- temperature profile: ambient to 80°C in 30 min, 80°C to 173°C in 90 min
- cooking at maximum temperature (173°C): 180 min
- total alkali: 12–20% as NaOH on o.d. jute.

Five different doses of alkali (12, 14, 16, 18, and 20%) were used to obtain different degrees of delignification. The spent liquors from these cookings were collected for subsequent pulping. Three series of cooks were done at each alkali dose in which the cooking liquor contained



1. Change in kappa number vs. percent spent liquor recycled



2. Tensile index as a function of kappa number (450 mL CSF)

25, 50, and 75% (v/v) spent liquor. The alkali doses were held constant after makeup with fresh chemicals. The pH of all the cooking liquors was measured at room temperature.

At the end of the cook, the autoclaves were cooled as quickly as possible. The cooked materials were thoroughly washed and then disintegrated or mechanically refined. The defiberized pulps were screened on a flat vibratory screen with 0.38 mm slots. The screened pulps were pressed to remove excess water and then stored in a refrigerator at 4°C in polythene bags.

Analyses

The following properties were determined: pulp yield, kappa number, sulfite consumption, and the pH and solids content of the spent liquors. The pulps were beaten to different freeness levels in a PFI mill with 30 g of o.d. pulp. The papermaking properties of the handsheets made from the pulps were evaluated using SCAN standard methods after conditioning the sheets at 50±2% relative humidity and 23±1°C.

Total alkali, %	Spent liq. recycled, % (v/v)	pH of spent liquor	Total yield, %	Kappa no.	Sulfite cons. (based on o.d. material), %	Solids content, %
12	0	7.2	61.4	25.5	8.44	5.9
12	25	6.9	63.2	33.2	8.46	7.2
12	50	7.0	62.1	37.0	7.98	9.4
12	75	7.1	64.5	48.2	7.98	10.4
14	0	7.6	60.9	18.5	9.52	10.5
14	25	7.7	60.7	23.4	8.92	13.9
14	50	7.7	60.2	30.8	7.96	14.9
14	75	7.8	64.4	37.7	7.72	15.5
16	0	7.7	60.8	16.2	9.23	11.3
16	25	7.8	60.1	19.6	10.00	13.4
16	50	7.7	62.8	22.8	10.20	15.9
16	75	7.9	62.5	23.4	10.60	17.4
18	0	7.7	61.5	15.3	9.62	13.5
18	25	7.6	63.4	15.9	10.40	15.0
18	50	7.5	60.2	17.4	10.60	16.4
18	75	7.9	60.0	20.0	10.90	17.9
20	0	6.9	59.7	14.8	11.30	14.9
20	25	7.2	60.1	15.5	11.80	15.9
20	50	7.5	61.1	17.4	12.10	17.7
20	75	7.2	61.5	19.8	12.50	19.4

I. Yield and kappa number of NS-AQ jute pulps with recycled spent liquor

RESULTS AND DISCUSSION

Pulp yield and delignification

The results (**Table I**) show that, in general, pulp yield increased slightly when >50% spent liquor was used. Pazukhina *et al.* also observed no change in yield during soda-sulfite pulping of aspen *(9)* and spruce wood *(10)* with recycling of spent liquor. However, Grankin *et al. (11)* found that recycling the spent liquor increased the pulp yield during pulping of hardwoods by the modified bisulfite method. The results of Aravamuthan *et al. (12)* also indicated a definite increase in yield with increased recycling of black liquor for both kraft and soda-AQ pulping of mixed hardwood chips. This yield increase was thought to be due to better retention of carbohydrates. A similar research by Patterson, quoted in Ref. 12, supported this contention by showing that there was an increase in hemicellulose content of pulp with an increase in recycled liquor volume.

The kappa number of the pulps increased gradually with an increase in the fraction of spent liquor, and the change was larger at lower alkali doses (**Fig. 1**). Similar observations were reported by Aravamuthan *et al. (12)* for recycling of kraft and soda-AQ black liquor. The increase in

Total alkali, %	Spent liquor recycled, % (v/v)	Total yield, %	Kappa no.	CSF, mL	PHYSICAL PROPERTIES Density, kg/m³	Tear index, mN·m²/g	Burst index, kPa·m²/g	Tensile index, N·m/g
12	0	61.4	25.5	450	477	22.6	5.60	59.0*
				250	558	17.8	8.94	84.5*
12	25	63.2	33.2	450	515	24.5	5.60	53.0
				250	590	18.6	8.87	81.0*
12	50	62.1	37.0	450	496	23.5	5.22	53.0*
				250	562	18.2	8.50	80.0*
12	75	64.5	48.2	450	535	22.3	5.01	50.0*
				250	608	17.9	8.39	75.0*
14	0	60.9	18.5	450	510	19.7	7.30	78.0
				250	577	16.8	9.68	94.0
14	25	60.7	23.4	450	503	21.0	7.43	74.6*
				250	594	16.8	9.20	88.0*
14	50	60.2	30.8	450	500	20.1	6.10	67.5*
				250	590	18.2	9.65	81.2*
14	75	64.4	37.7	450	505	22.8	5.70	53.0*
				250	589	18.0	8.28	79.3*
16	0	60.8	16.2	450	509	20.8	7.10	74.8
				250	582	16.9	9.30	90.0
16	25	60.1	19.6	450	457	20.0	6.52	72.5
				250	543	17.5	8.98	88.2
16	50	62.8	22.8	450	507	21.4	7.10	70.8
				250	577	17.0	9.84	87.5
16	75	62.5	23.4	450	539	20.3	6.50	69.8
				250	589	17.8	8.80	86.9
18	0	61.5	15.3	450	494	21.8	7.20	72.9
				250	555	16.8	9.65	80.0
18	25	63.4	15.9	450	490	20.0	7.13	70.0
				250	569	17.0	9.40	89.2
18	50	60.2	17.4	450	495	18.6	7.54	68.0
				250	583	15.9	9.58	88.0
18	75	60.0	20.0	450	500	19.6	7.38	67.3
				250	557	17.4	8.96	87.5
20	0	59.7	14.8	450	519	21.0	7.30	72.4
				250	589	17.2	9.61	89.8
20	25	60.1	15.5	450	513	18.5	7.10	71.5
				250	579	14.7	8.90	89.0
20	50	61.1	17.4	450	482	18.7	7.10	70.4
				250	570	15.5	9.05	88.6
20	75	61.5	19.8	450	490	19.6	7.30	69.6
				250	586	15.1	8.93	87.8

*Mechanically refined

II. Physical properties of NS-AQ pulp handsheets

kappa number might be due to a redeposition of dissolved lignin on the fiber surface. There were still residual chemicals, yet the kappa numbers increased. The poorer delignification could be due to lignin condensation, which made residual lignin resistant to pulping chemicals.

Sulfite consumption

At lower alkali doses, there was a gradual decrease in sulfite consumption as the volume of spent liquor increased (Table I). However, the kappa number increased simultaneously. Grankin *et al. (11)* also reported a reduction in chemical consumption on recycling spent liquor in modified bisulfite pulping of hardwoods. The residual chemicals were unable to reduce the lignin content, which became resistant to the pulping chemicals.

The sulfite consumption increased with an increase in active alkali. However, there was still an increase in kappa number, but the change in kappa number was less than it was with the lower alkali dose. The presence of higher alkali might have kept the lignin in the dissolved phase and thus retarded the redeposition of dissolved lignin onto the fiber surface.

Solids content

Table I shows that the solids content of the spent liquor increased on recycling, and it was the highest with 20% total alkali. The increment in solids content was, however, virtually the same for all alkali doses. This increase in solids content would reduce the additional fuel requirements in the evaporator section.

Papermaking properties

There was no noticeable change in both tear and burst indices (**Table II**). However, the tensile index decreased with an increase in spent liquor percentage. This might be due to a redeposition of dissolved lignin on the fiber surface, which had made the surface less hydrophilic and in turn reduced the chance of bonding between fibers. The variations in tensile index with kappa number (**Fig. 2**) clearly support this hypothesis.

CONCLUSIONS

An increased recirculation of spent liquor leads to a higher kappa number with only a minor change in pulp yield. The change in lignin content was higher at a low alkali dose. At higher alkali doses, the kappa number still increased, but the increase was smaller than it was with lower alkali doses. Sulfite consumption decreased gradually at a lower alkali charge, and the trend was reversed with higher alkali. The spent liquors contained unused chemicals at both the lower and higher alkali doses.

There was no change in either tear or burst indices. However, the tensile index decreased with an increase in spent liquor percentage. The decrease in tensile index might be due to poorer bonding between fibers caused by the redeposition of dissolved lignin. **TJ**

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