EVALUATION OF ADDITIVES IN SODA PULPING OF JUTE

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ABSTRACT

Seven additives to the soda pulping process – ethylenediamine (EDA), monoethanolamine (MEA), hexamethylene tetramine (HMTA), nitrobenzene (NB), anthrone, anthraquinone (AQ) and dihydro dihydroxy anthracene (DDA) were evaluated for making paper from jute fiber and whole jute plant. These pulps were tested for kappa number, yield, pentosan and α-cellulose. Pulp from soda-DDA process showed the lowest kappa number and the highest yield for both jute fiber and whole jute plant. Handsheets were tested for breaking length, burst index, teat index, double fold number and apparent density. Jute pulp from soda-EDA process exhibited the best tear index and soda-DDA process gave the pulps highest breaking length, burst index and double fold number. These pulps were also bleached by three stage bleaching sequences, namely CEH. Bleached pulp produced from soda-EDA process showed highest brightness in both raw materials. Jute pulp from soda-HMTA process lost maximum strength properties.

Jute is the internationally recognized name of the bast fiber obtained from the stalks of plants of the genus *Corchorus* in the Tiliaceae family. Traditionally it is being used for bags, sacks, feswas, hassien etc. Jute in Bangladesh was once popularly known as "golden fiber" for its contribution to the national economy of Bangladesh. Over the last few years its markets have declined due to the introduction of synthetic fibers. Therefore, it is worthwhile to look at the alternative uses of jute. Its chemical (*1*) and morphological (*2*) characteristics favor for using as pulping raw material. Therefore, some studies on jute pulping have been done for last few years in home (*3,4*) and abroad (*5,6*). But each of the processes have some limitations. These limitations include alkaline peeling reaction, lumping of the cooked materials, causing deficiency in digester blowing, washing, disintegration, environment, etc. In some cases pulp severely degraded on bleaching (*2, 7, 8*). Therefore, it is imperative to search an alternative process for better pulping. In soda pulping of white birch (*9*) and kraft pulping of Scandinavian pine (*10*), there is a trend to higher residual lignin content pulps prepared in the presence of borohydride than with hydroxylamine, which has a rather more favorable effect on delignification than borohydride. This suggests that the addition of additives in the cooking liquor is one of the important ways of better pulping.

The effect of additives on pulping, however, is different with various raw materials. Kubes *et al.* (*11*) indicated that Black spruce, Western hemlock, Douglas fir, Southern pine and Canadian mixed hardwoods were delignified at a rate significantly greater by soda-40% ethylenediamine (EDA) (on o.d. wood) liquor than that of kraft liquor. They showed that higher tear strength was achieved in softwood soda-EDA pulping but hardwood soda-EDA pulp did not

show improved tear strength. There is also a marked difference in the action monoethanolamine (MEA) on Aspen and on Sitka spruce (*12*). Aspen can be delignified almost completely by MEA alone in 5 to 6 hours at 170°C, whereas Sitka spruce still retains about 50% of its lignin after the same treatment.

The mechanism of pulping is different from one process to another. When using amines or their derivatives as additive, it is suggested *(13)* that the mechanism is influence by: 1) a redox process leading to the stabilization of the carbohydrates, 2) the interaction of free radicals, that suppresses the process of lignin condensation and facilitates its dissolution, as well as its removal from the reaction medium, 3) some surface effects connected with soaking or dissolving of the products.

The objective of the present investigation is to evaluate the seven additives - ethylenediamine (EDA), monoethanolamine (MEA), hexamethylene tetramine (HMTA), nitrobenzene (NB), anthrone, anthraquinone (AQ) and 1,4-dihydro 9,10-dihydroxyanthracene (DDA) in soda pulping of jute fiber and whole jute plant. The detail discussions of each soda-additive process are presented in separate manuscript.

EXPERIMENTAL

Raw material

Whole jute plant (WJP) and jute fiber (Jute) of *Chorcorus capsularis* variety were collected from Jute Research Institute, Dhaka. Dried whole jute plant and jute fiber were chopped separately to small pieces about 2-3 cm in length and foreign materials, such as grass, roots etc., were removed. The moisture content of jute and whole jute plant was determined separately according to TAPPI Standard Methods (T18m-53). After determination of the moisture content of air-dried whole jute plant and jute fiber were preserved a polyethylene bag for subsequent cooking experiments.

Cooking Liquor

The cooking liquor was prepared by using technical grade NaOH and EDA, MEA, NB, HMTA, AQ, anthrone or DDA for soda-EDA, soda-MEA, soda-NB, soda-HMTA, soda-AQ, soda-anthrone and soda-DDA processes respectively. In case of kraft (control) process the cooking liquor was prepared by using technical grade NaOH and Na_2S.

Cooking

All pulping experiments were performed in an autoclave having 20 liters capacity, made of stainless steel, rotating at 1 rpm and fitted with thermostat. The following parameters were maintained:

- Total alkali charge as sodium hydroxide was 18% for whole jute plant in all processes except in soda-quinone processes, where alkali concentration was 16% on o.d. WJP and 16% for jute.
- Liquor to fiber ratio was 6:1 for whole jute plant and 5:1 for jute.

- 15 minutes were required to raise the temperature to 70°C from room temperature and 90 minutes was required to raise the temperature from 80°C to 170°C.
- Cooking time at 170°C was varying 1-4 hours.
- Variation of additive on o.d. raw materials was 10-40% EDA and MEA for soda-EDA and soda-MEA processes, respectively, 1-10% NB for soda-NB process, 0.05%-0.5% HMTA for soda-HMTA process and 0.05 & 0.1%, anthrone, AQ and DDA for soda-anthrone, soda-AQ and soda-DDA processes, respectively.
- Sulfidity 25% for kraft (control).

After disintegration, pulps were washed thoroughly to remove the residual chemicals. After washing, pulp was disintegrated. Hard cooked materials were defibrated in a Laboratory Model Sprout Waldron disc refiner where applicable. After defibration or disintegration the pulp was screened on a flat vibratory screen with 0.38-mm slots. After the screening, the pulp was passed on a screw press to remove excess water. The dewatered pulp was shredded and mixed thoroughly and then weighed. A portion of the pulp was dried at 105±2°C to a constant weight and the moisture content of the pulp was calculated according to TAPPI standard methods (T210-58). The yield was determined as percentage on o.d. raw material.

Determination of Chemical Characteristics

The percentage of α-cellulose (T203os-61), pentosan (T223m-58) and kappa number (T236-60) in pulps were determined according to TAPPI Standard Methods.

Determination of fiber dimension of the unbleached pulps

Unbleached pulp obtained by optimum conditions in each process was used for the determination of fiber dimension. About 0.05 gm pulp for each process was taken in a test tube with water and shaken well for dispersion of fiber into individual one. Taking one drops of it on a slide and examined under the microscope. The length and breadth of 200 fibers were measured using objective 3.5:1 for length and 10:1 for breadth.

Evaluation of pulps

Both jute fiber and whole jute plant pulps were beaten in a Valley Type Laboratory Beater. The samples were collected at different freeness and handsheets about 60 gm/m^2 were made in a Rapid Kothon Sheet Making Machine according to German Standard Methods number 106. The sheet were tested for tensile (T-404os 61), burst (T-403m 53), tear (T-414m-49), double fold (T-423m-50) and apparent density (T-411m-44) according to TAPPI Standard Test Methods.

Bleaching

Soda-additive pulps correspond to optimum conditions were bleached by a three stage bleaching sequences (CEH) which comprises chlorination, alkali extraction and hypochlorite treatment stages. The temperature was maintained at 25°C for 60 minutes with consistency of 3.5%. In chlorination stage chlorine-water was used as bleach liquor for 70% of the total chlorine demand. In the extraction stage 2% NaOH (on o.d. pulp) was used for extracting the chlorinated pulp. In this stage temperature, time and consistency were 70°C, 60 minutes and 10% respectively. After completing the extraction, the pulp was washed with distilled water and was

taken in sealed polyethylene bag for hypochlorite treatment at 45°C for 60 minutes with 3.5% consistency in a thermostatic water bath. The sodium hypochlorite was used in this stage. The fully bleached pulp was then washed by distilled water.

RESULTS AND DISCUSSION

Chemical Characteristics

It is seen from the **Figures 1** and **2** that the delignification rate of jute fiber and whole jute plant is increased with the addition of additive in soda liquor up to certain charge and beyond that increasing rates are reduced in most cases except soda-NB pulp where delignification rates are retarded after certain charge of additive. On the other hand,10% EDA, 20% MEA, 0.1% HMTA or 2% NB, 0.05% DDA, 0.05% AQ or 0.05% anthrone in soda liquor reduce the kappa number remarkably and increase of the additive doses beyond these ranges kappa number is not reduced significantly. Therefore, these doses of the above additives are considered as optimum for jute fiber and whole jute plant pulping.

In this study, kappa number 19 for jute and 30 for whole jute plant are considered as optimum kappa number. Optimum conditions and chemical characteristics soda-additive jute and whole jute plant pulps are listed in **Table 1**. The addition of EDA, MEA, NB or quinone in soda liquor reduces the cooking time from 4 hours to 1 hour whereas HMTA reduces cooking time to 2 hours. The higher pulping rate in soda-additive processes is possibly due to the higher redox potential of black liquor (*14*). In the case of jute pulping, the pulp yields increased for soda-EDA, soda-MEA, soda-HMTA, soda-NB, soda-AQ, soda-anthrone and soda-DDA processes are 4.4, 8.2, 4.5, 4.4, 8.0, 7.9 and 8.6 respectively whereas that in the case of whole jute plant pulping are 4.2, 6.6 4.0, 4.0, 10.0, 9.4 and 10.4 respectively. Small amount of quinone additive reduces the cooking time from 240 minutes to 60 minutes and saves 2% NaOH (on o.d. whole jute plant). Similar results are observed in the case of soda-AQ pulping from muli bamboo (*15*). Therefore, it is seen that quinone additives are more effective for whole jute plant pulping than jute pulping. Pentosan content in soda-EDA pulp is lower than the other soda-additive pulps for both raw materials. But other soda-additive pulps contain higher pentosan than the soda pulp. The dissolution of pentosan in soda-EDA process is also evident during black spruce pulping (*16*). This lower pentosan in soda-EDA pulp also causes the lower pulp yield.

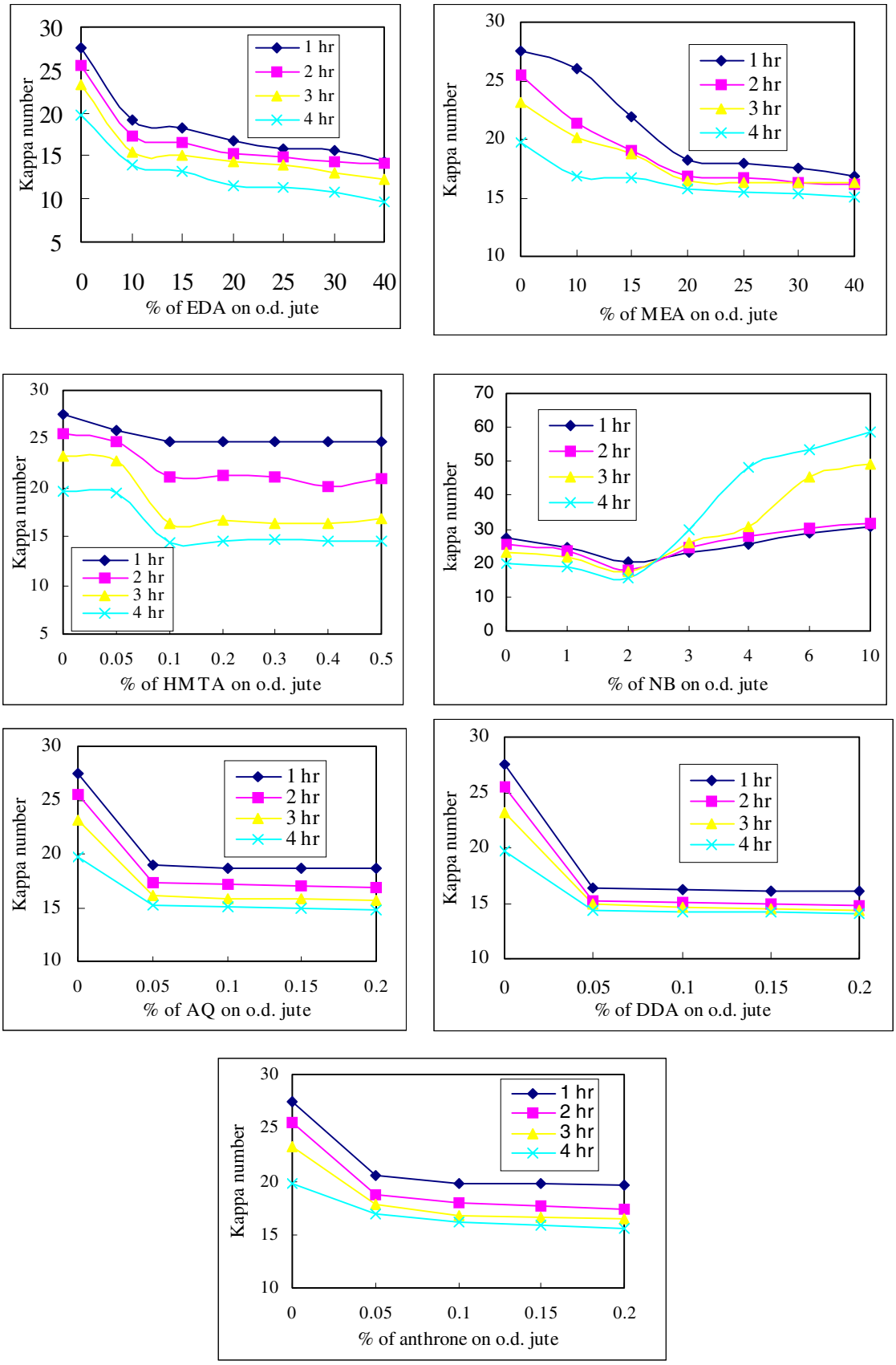

Figure 1. Effect of additive on the delignification of jute (-◆- 1 hour, -■- 2 hours, -▲- 3 hours and -✕- 4 hours).

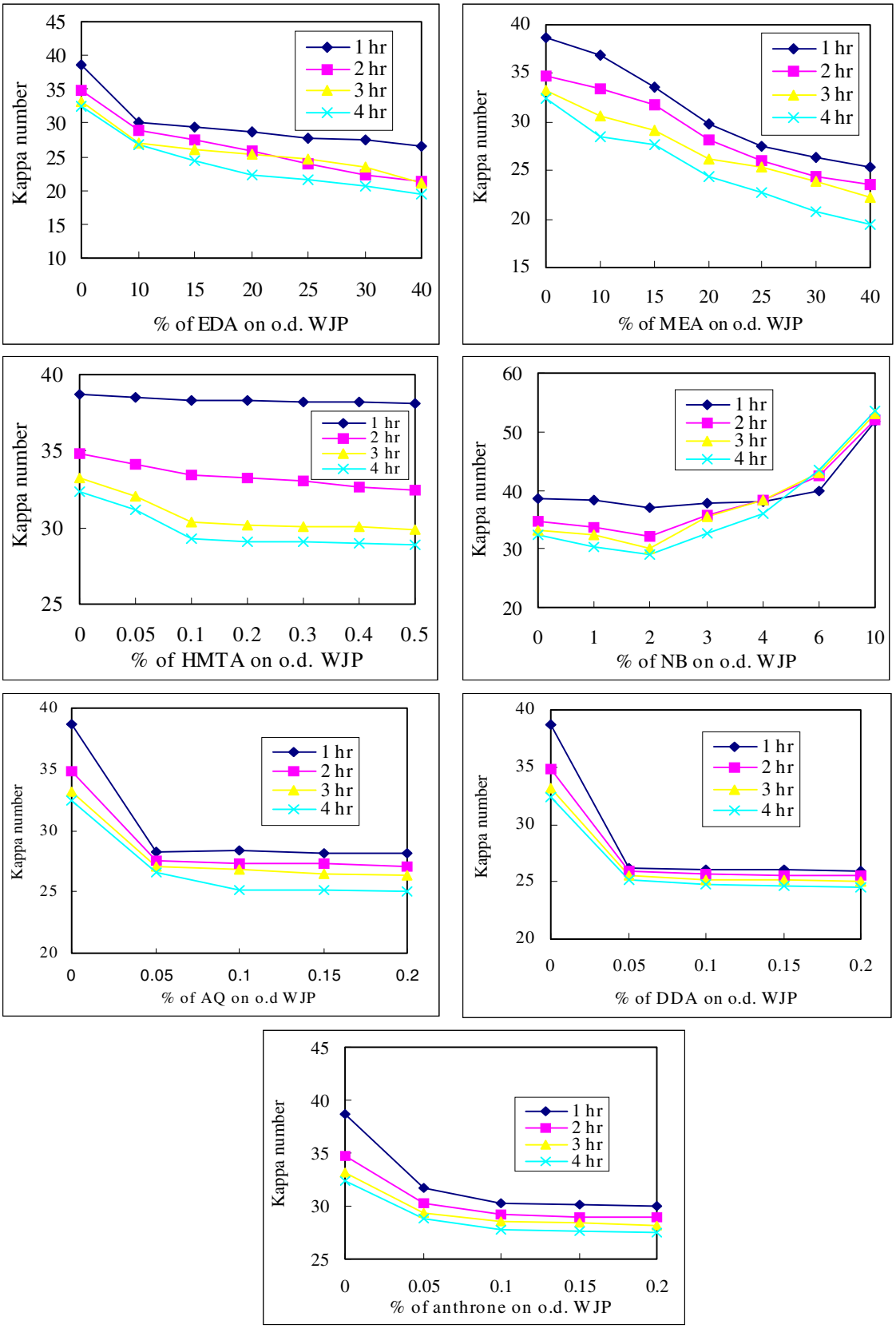

Figure 2. Effect of additive on the delignification of whole jute plant (-◆- 1 hour, -■- 2 hours, -▲- 3 hours and -✕- 4 hours).

Name of raw material	Name of the process	Cooking time, hour	NaOH, % on o.d. raw material	Additive, % on o.d. raw material	Pulp yield, %	Kappa number	α-cellulose in pulp, %	Pentos-an in pulp %
	Soda	4	16	0	56.3	19.7	86.1	9.7
	Soda-EDA	1	16	10	60.7	19.1	86.2	9.6
	Soda-MEA	1	16	20	64.5	18.2	85.2	11.5
	Soda-HMTA	2	16	0.1	60.8	21.1	85.6	11.1
Jute	Soda-NB	1	16	2	60.7	20.3	84.9	11.5
fiber	Soda-AQ	1	16	0.05	64.3	18.9	86.1	11.1
	Soda-anthrone	1	16	0.05	64.2	20.5	85.8	11.3
	Soda-DDA	1	16	0.05	64.9	16.4	86.1	11.3
	Kraft	1	16	25% sulphidity	59.0	20.8	85.1	11.1
	Soda	4	18	0	46.2	32.4	76.2	13.9
	Soda-EDA	1	18	10	50.4	30.1	80.7	14.4
	Soda-MEA	1	18	20	52.8	29.8	80.3	16.5
Whole	Soda-HMTA	3	18	0.1	50.2	30.4	76.3	15.6
jute	Soda-NB	3	18	2	50.2	30.1	78.2	16.2
plant	Soda-AQ	1	16	0.05	56.2	28.3	77.3	16.3
	Soda-anthrone	1	16	0.1	55.6	30.33	75.6	16.8
	Soda-DDA	1	16	0.05	56.6	26.2	78.8	16.5
	Kraft	2	18	25% sulphidity	50.3	29.6	77.8	15.7

Table 1. Optimum conditions and chemical characteristics of soda-additive jute and whole jute plant pulps.

Physical Characteristics

The physical characteristics of soda-additive unbleached jute and whole jute plant pulps by optimum conditions are presented in **Table 2**. It is clearly seen that the addition of additive to the soda liquor enhances strength properties and in some cases it is exceeded the kraft properties. But the additive is less effective for whole jute plant pulping. Thus the effects of addition of additive to the soda liquor are higher strength properties than soda pulp and somewhat higher than kraft pulp but having few exceptions. Soda-EDA process produces the best tear strength among all the processes for jute pulping. It is about 39.4% and 18% higher than the soda and kraft process, respectively. This is probably due to the lower density of soda-EDA pulp (Table 2). The higher tear strength of soda-EDA pulp was also observed in black spruce pulping (*17*). But soda-EDA does not increase tear strength in whole jute plant like jute. Table 2 also shows that double fold number is decreased in soda-HMTA pulping. This is in good agreement with the kraft-HMTA pulping of Doglas fir (*18*). But this effect is not evident on whole jute plant. Soda-NB jute pulp produces about 8.7, 13.4, 8.1 and 15.7% higher breaking length, burst index, tear index and double fold number, respectively than the soda process and about 5.2%, 5.5% and 15.5% higher breaking length, burst index and fold number, respectively than kraft pulp. On the other hand, whole jute plant pulps from soda-additive processes give slightly higher strength properties than the soda and almost similar to kraft pulp. Soda-AQ and soda-DDA produce much better strength properties except tear strength than the other processes in both jute and whole jute plant pulping. Soda-DDA process gives 17.8% and 13.9% higher breaking length of jute pulp and 12% and 8.5% of whole jute plant pulp than the soda and kraft processes respectively.

Name of the raw material	Name of the process	Breaking length, meters	Burst index, kPa.m^2/g	Tear index, mN.m^2/g	Double fold number	Apparent density, Kg/m^3
Jute	Soda	7288	6.7	12.4	1066	571.1
	Soda-EDA	7546	7.4	17.4	1178	500.3
	Soda-MEA	8128	8.1	16.0	1665	543.2
	Soda-HMTA	7422	6.9	13.5	793	553.6
	Soda-NB	7925	7.6	13.4	1233	546.6
	Soda-AQ	8370	7.7	14.5	1552	546.4
	Soda-anthrone	7522	7.2	15.9	1164	545.9
	Soda-DDA	8585	8.2	14.9	1852	550.3
	Kraft	7533	7.2	14.7	1070	561.4
Whole jute plant	Soda	6887	6.5	10.1	492	593.8
	Soda-EDA	7028	7.0	10.8	532	584.7
	Soda-MEA	7121	7.1	10.3	541	595.6
	Soda-HMTA	7019	6.8	11.2	521	592.3
	Soda-NB	7116	6.9	11.6	545	591.2
	Soda-AQ	7596	7.5	9.9	797	651.1
	Soda-anthrone	7126	7.0	9.6	629	624.5
	Soda-DDA	7723	7.6	9.9	872	656.2
	Kraft	7118	7.1	11.0	509	594.5

Table 2. Physical characteristics of soda-additive jute and whole jute plant unbleached (UB) pulps obtained by optimum conditions at 40°SR.

Fiber length of the pulp

Fiber length of jute pulps is much higher than the whole jute plant pulp. It is seen from **Table 3** that average fiber length of soda-additive pulps are comparatively higher than soda pulp. Therefore, the addition of any of the above additives in soda liquor causes the prevention of α-cellulose from the alkaline degradation during pulping (*17-19,20*). Soda-EDA pulp posses the highest average fiber length in both raw materials. This is the possible reason of higher tear strength in soda-EDA pulp (Table 2).

Bleaching

The use of soda-additive processes is quite new for lignocellulosic raw material such as jute, so its response to bleaching chemical is completely unknown. Therefore, effect of bleaching on soda-additive jute and whole jute plant pulps is one of the most important factor. It is seen from **Table 4** that the loss of pulp yields on bleaching of soda-additive jute pulps are about 5.0, 6.5, 8.5, 6.2, 4.9 and 4.4% (on o.d unbleached pulp) for soda-EDA, soda-MEA, soda-NB, soda-HMTA, soda-anthrone, soda-AQ and soda-DDA, respectively. The TAPPI brightness of all pulps is around 80-85%. The soda-EDA pulp shows the highest brightness among these processes, which is 85.7%. The different characteristics during bleaching in different processes are probably due to different characters of residual lignin. It is also seen from Table 4 that whole jute plant pulps produce a bleached yield of about 89-94% on o.d. unbleached pulp from soda-additive processes. Among these additives DDA produces the highest yield and HMTA gives the lowest yield. Thus there is not a severe loss of carbohydrate on bleaching the soda-additive pulps. However, a dissimilar phenomenon is observed in bleaching the kraft pulp from whole

jute plant (*8*) and jute (*2*). Therefore, soda-additive processes are suitable for whole jute plant pulping and these pulps may be used in making papers, which require bleaching. It is also evident from the Table that the soda-EDA pulp shows the highest brightness as compared to other soda-additive pulps. Soda-DDA and soda-MEA pulps also response well on bleaching in respect to brightness and other pulps show almost similar brightness.

Name of the raw material	Name of the process	Length in mm			Diameter in mm		
		Max.	Min.	Avg.	Max	Min.	Avg.
Jute	Soda	3.6	1.52	2.17	0.03	0.01	0.02
	Soda-EDA	4.08	1.68	2.52	0.02	0.01	0.02
	Soda-MEA	3.84	1.6	2.51	0.03	0.01	0.02
	Soda-HMTA	3.6	1.2	2.05	0.03	0.01	0.02
	Soda-NB	3.65	1.28	2.18	0.02	0.01	0.02
	Soda-AQ	3.67	1.47	2.37	0.02	0.01	0.02
	Soda-anthrone	3.56	1.2	2.15	0.02	0.01	0.02
	Soda-DDA	3.85	1.58	2.41	0.02	0.01	0.02
Whole jute plant	Soda	2.6	0.6	1.45	0.04	0.01	0.02
	Soda-EDA	3.6	0.68	1.68	0.04	0.01	0.02
	Soda-MEA	3.64	0.64	1.67	0.04	0.01	0.02
	Soda-HMTA	3.0	0.64	1.57	0.04	0.01	0.02
	Soda-NB	2.92	0.6	1.47	0.04	0.01	0.02
	Soda-AQ	3.1	0.61	1.52	0.04	0.01	0.02
	Soda-anthrone	3.0	0.64	1.5	0.04	0.01	0.02
	Soda-DDA	3.1	0.62	1.54	0.04	0.01	0.02

Table 3. Fiber dimension of jute and whole jute plant pulps.

Name of Raw Material	Name of Process	Bright-ness (% (tappi)	Yield, % on o.d.UB	Breaking length, meters	Burst index, $kPa.m^2/g$	Tear index, $mN.m^2/g$	Double fold number	Apparent density, Kg/m^3
Jute	Soda-EDA	85.7	95	6421	6.3	16.5	923	491.9
	Soda-MEA	83.4	93.5	7201	7.0	14.2	1275	546.2
	Soda-HMTA	82.3	91.5	6411	6.0	10.3	491	569.2
	Soda-NB	80.1	90.1	6850	7.0	12.9	520	549.5
	Soda-AQ	83.7	95.1	8107	8.0	12.7	1527	587.1
	Soda-anthrone	80.3	93.8	7436	7.2	13.8	1065	574.3
	Soda-DDA	84.2	95.6	8303	8.1	14.1	1801	601.5
Whole Jute Plant	Soda-EDA	83.4	92.1	6971	6.7	10.3	410	584.7
	Soda-MEA	82.1	91.6	6998	6.8	10.3	529	624.5
	Soda-HMTA	80.8	90.2	6606	6.5	9.1	175	689.5
	Soda-NB	79.2	88.8	6895	6.6	9.8	470	623.3
	Soda-AQ	81.1	93.5	7414	7.2	9.3	778	679.2
	Soda-anthrone	80.1	91.7	7168	6.8	9.8	679	637.1
	Soda-DDA	81.9	94.1	7618	7.4	9.4	811	688.9

Table 4. Bleached properties of soda-additive jute and whole jute plant pulps.

Table 4 also shows that tear index of jute pulp from soda-EDA process retains its superiority even after bleaching. About 2-6 points are higher than the other soda-additive jute pulps. On bleaching, soda-EDA jute pulp losses about 15% breaking length and burst factor whereas no appreciable losses is evident in whole jute plant. Soda-quinone bleached pulps also possess better strength properties than the soda-nitrogen containing additive pulps for both raw materials like in unbleached state. Huge loss of strength properties of soda-HMTA jute pulp is observed on bleaching. About 13% breaking length and burst index and 24% tear index are lost. But soda-HMTA whole jute plant pulp does not lose strength properties severely on bleaching. On bleaching, soda-NB jute pulp loses about 14% breaking length but its tear index does not lose severely. On the other hand, soda-NB whole jute plant pulp slightly lost strength properties on bleaching. The double fold number of soda-NB jute pulp is rapidly reduced on bleaching.

CONCLUSIONS

From the present investigations, clear evidence has been adduced that the presence of each of the additives EDA, MEA, DDA, AQ and anthrone in soda liquor accelerates the pulping reactions in both whole jute plant and jute fiber. On addition of 10% EDA, 20% MEA, 0.05% AQ, 0.05% DDA or 0.05% anthrone in soda liquor reduces the cooking time from 240 minutes to 60 minutes. However, the rate of pulping is increased significantly to a certain charge of additive on o.d. raw material and after that the increase of pulping rate is reduced. DDA, AQ, anthrone and MEA have a profound effect on the stabilization of all carbohydrates. EDA is also effective for the stabilization of α-cellulose but it can not protect lower molecular weight carbohydrates from the dissolution during jute and whole jute plant pulping. A remarkable observation is that the tear index of soda-EDA jute pulp is 17% higher than the control kraft pulp but soda-EDA pulp can not produce better breaking length, burst index and fold number as compared to soda-MEA, soda-quinone pulps. In respect to all physical strength properties, DDA is the most effective additive for jute pulping. For whole jute plant pulping DDA, AQ, and MEA show almost similar physical strength properties.

On bleaching with a three stage bleaching sequence (CEH), final brightness for jute pulps are 81-87% and for whole jute plant pulps are 79-84% by the soda-additive processes. The loss in pulp yield on bleaching is 5-10% for jute pulps and 6-11% for whole jute plant pulps on o.d. unbleached pulps. The physical properties loss is not severe especially for soda-MEA and soda-quinone pulps from both raw materials. Soda-EDA pulp gives very high tear strength even after bleaching, has 10% higher tear strength than the unbleached soda-MEA pulp. The strength properties of soda-HMTA jute pulp decrease rapidly on bleaching. Therefore, it is suggested that jute and whole jute plant (unretted) pulps from soda-quinone and soda-MEA processes may be used in the production of writing and printing papers, and soda-EDA jute pulp can be used where requiring higher tear strength.

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