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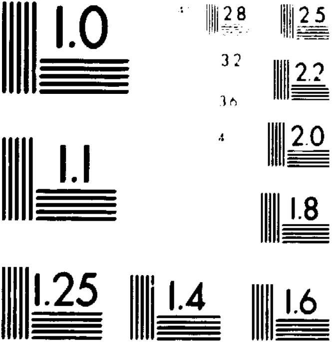
UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org



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JUTE PRODUCTS RESEARCH

DP/BGD/75/013

BANGLADESH

Technical report: Chemistry of Lignin, with Special Reference to the Jute Fibre and the Photochemical Yellowing upon Exposure to Sunlight *

Prepared for the Government of Bangladesh
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of K.V. Sarkanen,
Consultant in Organic Chemistry

United Nations Industrial Development Organization
Vienna

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CONTENTS

MISSION SUMMARY

The Consultant visited the Technology Wing, BJRI, first March 20 to 31, 1984 and, the second time, July 1 to September 11, 1984. The results of these visits are summarized in the following:

1. A novel chemical modification method for long jute and jute yarn was developed, based on pressure treatment with sodium sulfite solution. It was demonstrated that the treatment improved substantially the softness, flexibility, color, bleachability and weaving properties of jute fibres without impairment of their dry and wet strength characteristics. Color stability studies of the modified fibres could not be completed, however, in the short time available. Work on the chemically modified jute will continue at BJRI under the direction of Dr. A. Rahman. The suitability of the modified jute for apparel grade textiles is of particular interest, either as such or in blends with cotton and synthetic fibres.

2. In discussions with the Institute personnel, it became obvious that the current industrial practice in the piling of jute cuttings is unsatisfactory and needs to be modified in order to maintain optimal and uniform temperatures and to eliminate moisture gradients. A piling chamber design was proposed for achieving these goals, and a research program outlined for its implementation.

3. Visits to two pulp and paper mills demonstrated that the pulp industry is the most natural outlet for jute waste, caddies and surplus cuttings. A joint exploratory research program with BCSIR was initiated to determine the potential of semi-alkaline sulfite - anthraquinone pulping of jute cuttings. The results suggest that this pulping method is probably more advantageous than the currently used soda pulping for the manufacture of newsprint reinforcement pulp.

4. Recommendations are made concerning bleaching, mercerization and cross linking studies at the Institute.

5. This consultant feels that continued UNIDO assistance to BJRI is warranted in the form of a limited Phase II program, focused mainly on filling the gap between laboratory studies and industrial implementation.

I. INTRODUCTION

The purpose of the mission was "to assist the Bangladesh Jute Research Institute (BJRI) with its research program on the chemistry of lignin, with special reference to the jute fiber and the photochemical yellowing which occurs on prolonged exposure to sunlight".

This Consultant proposed the mission to be carried out in two parts:

a. A ten day planning trip to Dhaka in March 1984 and
b. A two and half month residence in Dhaka in July 1 - September 11,1984.

The proposed arrangement was approved by UNIDO, and the Consultant arrived to Bangladesh on March 20 after one-day briefing with Dr. H.P. Stout in Edinburgh. During this period, discussions were held with members of the Technological Wing of the BJRI, available equipment and chemicals were reviewed and, on this basis, a reasonably detailed research plan was drawn for the July-September period, 1984. The research plan was set forth in the First Technical Report (Final version: DP/ID/SER.A/511, dated 2 May, 1984). The proposed plan is briefly summarized as follows:

It was recognized that the photodiscoloration and - degradation of jute fibres is connected with the presence of lignin and other polyphenolic materials. In contrast to cotton, lignin cannot be completely removed from jute fibres without seriously impairing the mutual adhesion of the short intrinsic fibres in the composite fibre strands. Consequently, the photochemical yellowing tendency of jute fibres cannot, in all likelihood, be prevented without drastic reduction in dry and, particularly, wet strength. Realistically, then, the yellowing tendency can be only reduced but not eliminated. Two approaches were proposed for the reduction of yellowing:

i. Incorporation of an inorganic or organic component in the jute fibres that may either quench the photon-excited aromatic groups in lignin or to scavenge the radical intermediates in the oxidative chain process initiated by photon-excitation.

ii. Controlled, partial removal of the lignin component and/or its conversion to a photochemically more resistant form. Obviously, the degree of delignification should not be extended beyond the point where the dry -

and, particularly the wet-strength of the fiber is reduced below acceptable values. Also, the partial delignification should improve rather than detariorate the original color of the fiber. It would also be desirable to improve the softness of the fibre in the chemical delignification treatment.

Since it did not seem advisable to launch studies in too many directions at the beginning, it was proposed that efforts during the initial stages be concentrated on the second alternative. In this area, the Consultant had already performed exploratory experiments at the University of Washington together with Mr. Hossein Resalati, a graduate student from Iran. These experiments had demonstrated that alkaline sulfite - (Na_2SO_3 plus NaOH) and semialkaline sulfite - (Na_2SO_3 plus Na_2CO_3) methods appeared promising for controlled delignification of jute fiber, - yarn and - fabric. Both methods have been known and utilized in the pulp- and paper industry for the production of long-fibered reinforcing pulp from softwoods. Also, Na_2SO_3 is utilized in converting wood chips to high-yield chemi-mechanical pulps (CMP). Specifically for jute, alkaline sulfite methods have the following advantages in comparison with alternative delignification methods:

- Relatively inexpensive chemical charge. The liquors after the delignification treatment can be discarded. Thus, a chemical recovery system is not necessary.

- The color of the fibers is not darkened at intermediate stages of delignification, and can be readily improved by simple bleaching, e.g. with hydrogen peroxide.

- The loss of the hemicellulose components in the process is less than in soda - or kraft delignifications (which also degenerate the color of the fiber).

- The lignin component remaining in association with the fibres is converted to a solid lignin sulfonate, a polyelectrolyte. This transformation can be expected to improve such textile properties as fiber flexibility and softness. The effect on light stability is not known, but can be anticipated to be positive.

The first report also outlined a schedule for the implementation of the research plan consisting of two phases:

A. First Period, April 1 to June 10, 1984.

A broad preparatory program, to be separately carried out by Dr. A. Rahman and Mohammad Ali at BJRI and by the Consultant and Mr. Hossein Resalati at the University of Washington.

B. Second Period, July 1 to September 11, 1984.

Joint research by the Consultant with the BJRI scientists.

The results obtained during both time periods are reviewed in the Section that follows:

II. FIRST PERIOD, April 1 to June 30, 1984

A. At the University of Washington, the first goal was to clarify, to what extent jute fibre could be delignified without impairing its dry-, and, particularly, wet-strength properties. In addressing this question, the following should be observed:

The lignin component in jute fiber strands in distributed between the middle lamellae and the secondary walls. The concentration of lignin in the middle lamella is high (80-90 per cent), but only about one-fifth of the total lignin is present in this region. The bulk of lignin (∿80 per cent) is located in the secondary wall. It has been shown by Whiting and Goring (Figure 1) that in common delignification processes, lignin is more rapidly removed from the secondary wall than from the middle lamella. This removal does not, however, weaken the bond between intrinsic fibre. In contrast, elimination of middle lamella lignin (as it occurs in chemical pulping of wood chips for example) destroys the interfiber strength and the delignified tissue can be readily desintegrated to intrinsic fibers in water-soaked condition.

If a similar delignification behavior is valid for the bast fibers of jute, as seems very likely, the delignification process should not be carried out beyond the point where lowered concentration of lignin in the middle lamella regions significantly deteriorates the wet-strength properties of the fibre.

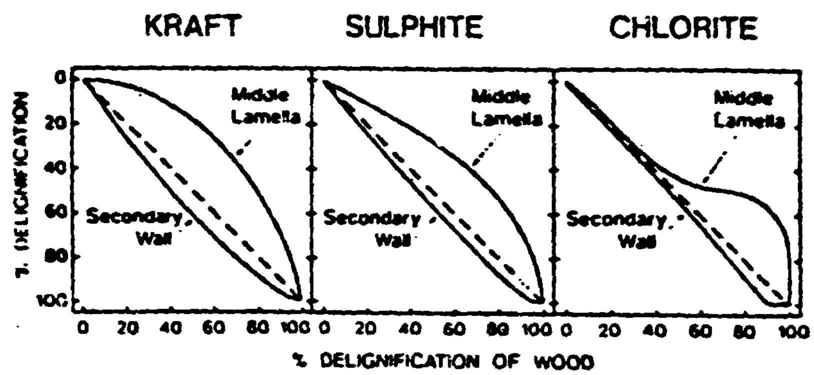


FIGURE - 1 Plot of percent lignin removed from the middle lamella and secondary wall of black spruce versus percent lignin removed from the whole wood. The dashed 45° line represents whole wood versus whole wood.

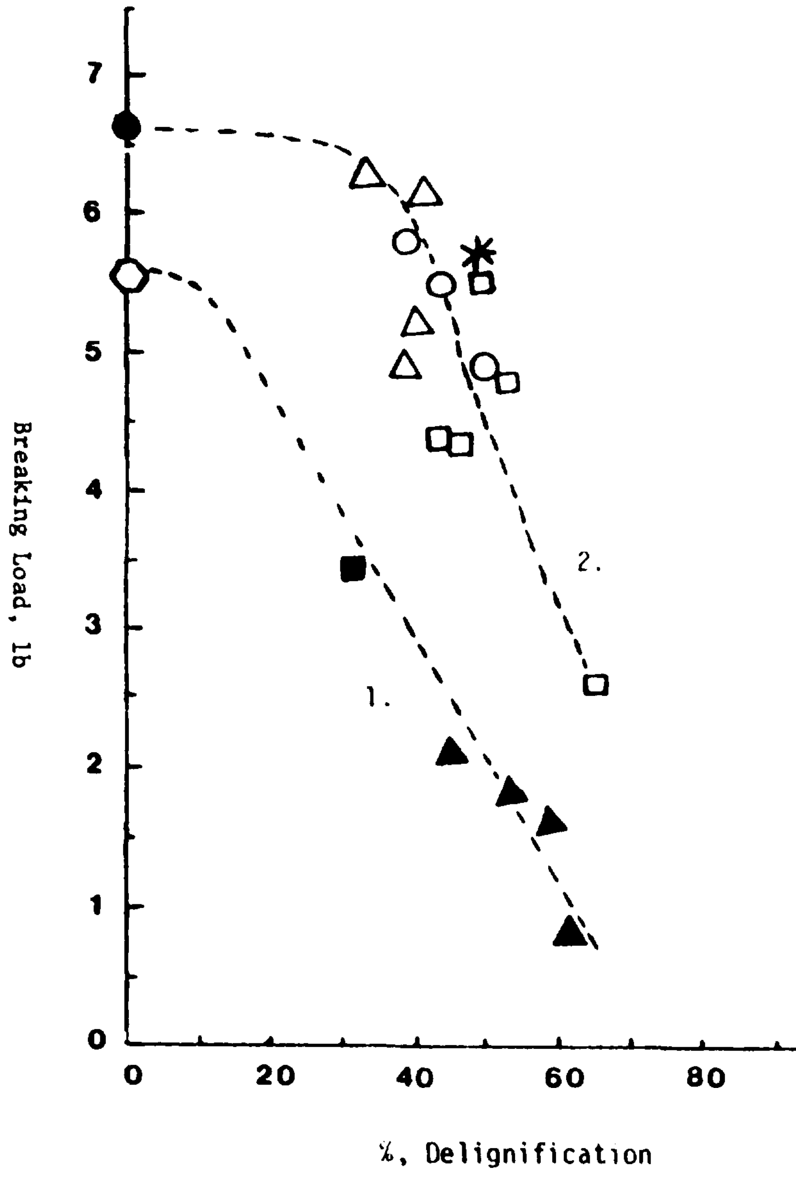
To determine this point, jute yarn obtained from BJRI was subjected to progressive delignification by alkaline sulfite-AQ method at 165°C, and the wet-strength values of the delignified samples were determined. The wet-strength values obtained are shown as a function of the degree of delignification in Figure 2.

At first glance, these results appear quite discouraging. At 50 per cent delignification level, the wet strength has been reduced by 65 per cent - obviously an unacceptably low value. However, at the time these experiments were performed, it was not recognized that the high pH of the alkaline sulfite delignification strongly promotes the deterioration of wet-strength. When semi-alkaline sulfonation ($Na_2SO_3 + Na_2CO_3$) is applied as delignification method, the pH of the medium remains in the range 11 to 7, and the wet-strength values are substantially higher. This was demonstrated in parallel work carried out during the first period at BJRI.

The experience gained in alkaline sulfite delignification gave raise to further studies on the relationship between delignification and wet-strength. Sodium hypochlorite and chlorine dioxide were used in these studies as delignification chemicals.

The results obtained are illustrated by Curve 2 in Figure 2. It can be seen that using these common bleaching agents, approximately forty-five percent of lignin can be removed from unscoured jute yarn without significant loss in wet-strength. After 50 per cent lignin removal, however, the wet strength degenerates precipitously. Chlorine dioxide appears to have no advantages in comparison with hypochlorite. An interesting observation was made in this connection on the effect of alkali on jute yarn. If the yarn is moistured with 1% NaOH at ambient temperature and washed with water prior to hypochlorite delignification, the resulting wet strength is only one-half of the value obtained without alkali pretreatment. This observation suggests that even brief exposure of jute to aqueous alkali causes irreversible changes in the fiber and this effect merits further studies.

The brightness of the yarns delignified by hypochlorite was generally low (∼50%) when the hypochlorite charge (5 to 6% active chlorine on O.D. fiber) was applied in a single stage. If the same hypochlorite charge, however, was divided between two or three successive stages, final brightness increased to the level of 60 per cent and above.



Breaking Load, lb
0
1
2
3
4
5
6
7
0
20
40
60
80
1.
2.
%, Delignification

● Untreated yarn

△ Hypochlorite (H)

✱ Chlorine Dioxide (D)

○ H - D

□ D - H

■ 1% cold NaOH - H

▲ Alkaline sulfite - AQ

⬡ 1% cold NaOH

Figure 2: Wet Tensile Strength of Jute Yarn as a Function of Delignification by Alkaline Sulfite (Curve 1) and by Hypochlorite and Chlorine Dioxide (Curve 2).

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B. At BJRI, successful experiments were performed by Mr. Mohammad Ali using semialkaline sulfite delignification (Na_2SO_3 plus Na_2CO_3) at 145°C (maximum temperature for available pressure equipment). Regardless of some initial operational difficulties, the small high temperature dyeing apparatus in the pilot plant was found to perform in a satisfactory manner in these experiments. The behaviour of crude jute fiber, jute yarn, Hessian fabric and cuttings were tested. It was found that significant improvement of fiber, yarn and fabric could be achieved, if the reaction period at 145°C was extended to three hours. The improved qualities included better color and whiteness, as well as much improved softness and flexibility. It was also estimated that only approximately 30 per cent of the original lignin dissolved in the treatment, while the total reduction in fiber weight was approximately 15 per cent, indicating that the dissolved material consisted mainly of hemicelluloses.

In contrast to these encouraging results there were some disappointments. Replacement parts for the second Xenotest instrument were not received. Consequently, this instrument could not be placed at the disposal of the Chemistry Department of BJRI, as specifically recommended in the First Report. The rotatory digestor, presumably ordered prior to March, 1984, was not received. A later check reveiled that the digestor order had actually not been processed by UNIDO in August, 1984.

III. SECOND PERIOD, July 1 - September 11, 1984

Initially, exploratory experiments using the small high-temperature dyeing apparatus were continued in the temperature range 145 - 155°. It was found that the sulfonation process was not attractive for the treatment of cuttings because of color problems and incomplete removal of pectins. Consequently, improvement of yarn quality and chemical modification of raw jute by sulfonation became the primary targets of the investigation. At this point, the gaskets in the apparatus started leaking, and the mechanics of BJRI were unable to repair the equipment.

A. Effect of Sulfonation on Jute Yarn Quality. The use of the large high-temperature dyeing apparatus was modified in such a manner as to allow the concurrent run of several experiments. This was accomplished by filling the equipment half-full with water. A large wire basket containing individual sulfonation experiments in cotton-plugged erlenmeyer flasks was hung above the water level, to be heated by pressurized steam.

The results obtained are shown in Table 1. It can be seen that an increase of the sulfite charge from 5 to 20 per cent had no effect on residual lignin content of the yarn, although it improves the brightness values. It should be noted, however, that many of the brightness values quoted in Table 1 are actually too low, because at that time, the importance of rapid washing of the yarns with hot water was not recognized. In general, the brightness values of well-washed sulfite treated yarns lie above 50. An attempt was made to determine the photodiscoloration of the yarns by exposing them to mercury lamp radiation. None of the sulfonated yarns developed noticeable yellowing after 96 hours of exposure. The equipment used by us was not very satisfactory for the purpose, however, and more detailed studies ought to be carried out on photochemical yellowing of sulfonated yarns in the future.

Table 2 shows that, at a constant chemical charge, higher reaction temperatures produced more extensive delignification but not improved brightness values. Sulfonation may be performed at as low temperatures at 95°C, but requires, in our experience, excessively long (~20 hrs) reaction times.

It was also possible to demonstrate that sulfonation improves the bleachability of yarns as shown in Table 3. A hydrogen paroxide charge corresponding only to one per cent of the weight of the yarn increases the brightness to over the 60 per cent level.

The most notable change caused by sulfonation is a conspicuous <u>improvement in softness</u>. This property is reflected in flexural rigity values which may be reduced by as much as 50 per cent as shown in Table 4. At the same time, reductions in dry tensile strength values are only minor. Wet tensile strength values are reduced more, but do remain on a level which ought to be acceptable for textile applications.

In order to test the weaving properties of sulfonated yarn, a batch of 3 kg of yarn was subjected to the treatment. This could not be done at BJRI, because delays in the repair of the two steam boilers paralyzed our activities there. With the kind help of Dr. Amirul Islam, we were able utilize the rotatory digestor at the BCSIR for the purpose. A union fabric

<u>Table 1 :</u> **Effect of Sulfonation conditions on the lignin content and brightness before and after exposure to mercury lamp radiation. In all experiments, 8.0 count jute yarn was utilized and the reaction time and temperature were 2 hrs. and 140°C, respectively.**

Chemical charge, %			Lignin	Origin	Brightness, after UV-radiation for		
Na_2SO_3	Na_2CO_3	EDTA	% of fiber	brightness %	12 hrs.	44 hrs.	96 hrs.
Original	sample		14. 0	41. 9	40. 7	-	42. 7
5	0	-	-	45. 7	-	42. 4	43. 6
5	4	-	10. 4	-	-	-	-
10	0	-	-	46. 7	-	43. 2	44.4
10	4	-	-	47. 2	-	44. 0	45. 8
20	0	-	10. 4	48. 3	47. 7	48. 4	-
20	0	0. 015	10.4	58. 8	54. 3	53. 4	54. 3
20	4	-	10. 7	48. 7	47. 2	48.0	-

Table 2: Effect of Sulfonation Temperature on the Lignin Content and Brightness of Jute Yarn. Chemical charge: 10% Na_2SO_3 and 4% Na_2CO_3. Reaction times: 20 hrs. at 95°C, 2 hrs. at 140°C and 1 hr. at 160°C.

Reaction temperature °C	% Lignin in fiber	Brightness, %
95°	11. 3	51. 5*
140°	10. 4	47. 2
160°	8. 5	51. 7*

*) 0.015% EDTA was added to Sulfonation.

Table 3: Effect of H_2O_2 - Charge on the Bleaching of Sulfonated Jute Yarn. (Count:80) Conditions of Sulfonation: 10% Na_2SO_3, 4% Na_2CO_3, 0.015% EDTA, 20 hrs. at 95°C.

Sample	Applied H_2O_2, % of yarn	Brightness, %
Original yarn	None	42.0
Sulfonated yarn	None	51.3
Bleached sample	0.5%	56.2
Bleached sample	1.0%	62.8
	2.0%	68.3

Table 4: Dry and Wet Tensile Strengths and Flexural Rigities of Sulfonated Jute Yarns.

Chemical charge %		Reaction temperature °C	Tensile Strength % of original		Flexural Rigidity, dyne/cm^2
Na_2SO_3	Na_2CO_3		Dry	Wet	Untr. yarn: 668
5	0	140°	82	73	423
5	4	140°	90	58	423
10	0	140°	86	63	435
10	4	140°	107	54	410
20	0	140°	89	48	266
20	4	140°	103	67	322
10	4	95°	90	50	411
10	4	160°	103	46	323
10*	4	160°	94	53	293*

*) 4.5 count yarn, flexural rigidity of untreated yarn 389 dyne/cm^2

with cotton warp and sulfonated jute weft was woven at the Institute. The weaving properties of the sulfonated yarn were good. A reference fabric was woven using untreated jute yarn. For both fabrics, measured tensile strengths and bending lengths are given in Table 5. The results obtained for the weft direction indicate clearly that the sulfonated yarn imparts better flexibility and equal strength to the fabric.

B. Carding and Spinning of Sulfonated Jute Fibre. The experiments in the previous section demonstrated that sulfonation improved significantly the softness and flexibility of jute yarns. It was therefore of interest to determine whether or not improved structural characteristics, such as better uniformity, lower count and increased twist, could be imparted to yarns, if the sulfonation treatment were applied to crude fiber prior to carding and spinning.

In the initial trial in this direction, approximately 10 kg of crude fiber were sulfonated for 1 hour at 165°C in the rotatory digestor at BCSIR. The sulfonated fibres had some excess moisture before the softening treatment. As a consequence, too much oil emulsion (25%) was added to the moist fibres. Satisfactory performance was achieved in the breaker card, but the second card became clogged.

In the second trial which came out to be succesful, 25 kg of crude fiber were loaded in the large high-pressure dyeing apparatus and treated with 10 per cent Na_2SO_3 and 4% Na_2CO_3 at 145°C for two hours. After cooling and drainage, the fibers were washed with hot water, centrifuged, washed again with cold water, centrifuged and dried. Oil emulsion (15%) was sprayed on the fibers from a watering can and the fibers conditioned at ambient temperature for 24 hours. This time, the performance in both carding machines and in the first and second drawing frames was completely satisfactory. Two separate batches of yarns, with counts 5.39 and 5.75, respectively, were spun from the material. Testing the yarns gave the following results:

	Count	T.S.	Q.R.	C.V.
First yarn	5.39	5.67	105	20.6
Second yarn	5.75	6.47	113	19.0

Table 5: Properties of Union Fabric woven from Sulfonated Jute Yarn. Warp: Cotton yarn, 62 per inch; weft: 4.5 count sulfonated jute yarn, 21 per inch.

	Union fabric with	
	treated yarn	Untreated yarn
Warp (Cotton):		
Bending length, cm	1.7	1.6
Strength, kg/inch	78.7	76.5
Weft (Jute):		
Bending length, cm	4.0	4.9
Strength, kg/inch	59.0	61.1

The results indicated that the Quality Ratios are higher than those obtained for untreated jute yarns. While these results are definitely encouraging, extensive further work will required to determine to what degree it will possible to improve the structural characteristics of jute yarns by chemical modification of crude fiber.

C. Conclusions and Recommendations

In the past, the following chemical treatments have been applied for the chemical modification of jute fibers:

- Mercerization with NaOH or liquid ammonia
- Bleaching with hypochlorites, chlorites and peroxides
- Cross-linking with epoxy - and halogenated compounds, urea-formaldehyde etc.

In general, these modifications have been applied in connection with fabric finishing.

Our work has demonstrated the feasibility of a novel type of modification based on the use of inexpensive sodium sulfite. The main advantage of this treatment is a drastic reduction in fiber stiffness, in addition to improvements in brightness and bleachability. We have also demonstrated that the treatment may be applied to yarns and crude jute fiber, in order to achieve improved structural characteristics for jute textiles as well as textile blends with cotton and synthetic fiber. It is possible, although not proven as yet, that chemical modification will ultimately lead to new processing technology in the jute field, with the improvement of existing jute fabrics and the generation of novel textile products. It seems therefore appropriate to suggest that chemical modification ought to occupy a significant part in the future research programs of BJRI.

IV. COMMENTS AND RECOMMENDATIONS ON ONGOING RESEARCH PROGRAMS AT TECHNOLOGICAL RESEARCH WING, BJRI

At the request of Dr. Bhuyan, Director of the Technological Wing, the consultant became aquainted with a number of ongoing research programs at the Institute. In the following, comments and recommendations are presented on these programs. Individual projects are identified by the Objective- and Project numbers, as they appear in the "Technological Research Progamme of BJRI (TRW) for the year 1984". For example, the notation 7 - 19 refers to Objective No. 7, Project No. 19.

A. Photochemical Discoloration and Loss of Strength

The importance of these characteristics of jute and jute products is recognized at the Institute. Significant progress in this area was made by Dr. A.B.M. Abdullah, S.S.O., in connection with his Ph.D. work in the United Kingdom. Dr. Abdullah was able to demonstrate that.

i. Sodium borohydride reduction of jute fibres significantly reduces yellowing tendency by the elimination of carbonyl functions.

ii. In dyed jute fibres, complexed nickel ion reduces yellowing by quenching excited singlet states of aromatic groups,

Both of these observations are of substantial practical interest and deserve to be pursued vigorously.

At BJRI, progress in photochemical yellowing research has been slow. There are several reasons for this circumstance:

a. A quantitative measure for photochemical yellowing ("yellowing index") needs to be developed on the basis of the visible reflectance spectra of photoexposed samples. Changes in brightness are often worthless in this respect, since yellowing and photochemical bleaching may occur concurrently.

b. The capacity of available xenotest instruments is not sufficient for photochemical yellowing studies. Appropriate mercury lamps with filters need to be aquired for accelerated yellowing studies.

c. Under the present system, all photochemical yellowing studies have been assigned to the Physics Department. The record of Annual Reports demonstrates that the Physics Department has been generally unable to perform even such simple measurements as whiteness determinations (of bleached and mercerized samples) within a period of six months. It would seem appropriate to propose that investigators needing optical data should have access to the appropriate instruments in order to carry out the measurements on their own samples.

The project 7 - 19 of the 1984 Programme carries the title "Study and Prevention of Photochemical Changes in Jute". This Consultant obtained the impression that the project was moving ahead very slowly for reasons mentioned above. It is of importance that the project be reinvigorated. We need to know, as precisely as possible, how different chemical treatments in bleaching, in mercerization and in sulfonation affect the photochemical stability of jute fibre.

B. Improved Utilization of Jute Cuttings

Jute cuttings which may sometimes amount to up to 25 per cent of total fiber harvest consist of low quality fiber with inferior market value. Currently there is a balanced market demand for products from cuttings (carpet backing, sacking etc.), but this situation may not continue. An excess production of non-marketable jute cuttings is a foreseeable possibility. Consequently, there is pressing need in the jute industry to improve the quality of cuttings as fibre raw material. At BJRI, improvements in cutting technology are being sought in the form of two research projects (2-3 and 2-4).

During the visit, the Consultant evaluated and made recommendations concerning the following three areas of cuttings research:

a. Improvements in the current practice of softening and piling of jute cuttings.

b. Chemical depectination as an alternative to current practice.

c. Increasing and improving the use of jute cuttings as a raw material for the pulp and paper industry.

The recommendations made in each of these areas are summarized in the section that follows:

a. Improvements in Current Practice (2-4)

Mr. Ataur Rahman, S.S.O., is currently working on this problem as a part of his Ph.D. dissertation research, under the supervision of Professor Jabbar Mian, Chemistry Department, Dhaka University. In the past, Mr. Rahman has carried out studies on the maturation of jute cuttings during piling under industrial conditions. Generally, the maturation time in pile, after the oil emulsion has been distributed to cuttings, varies from eight to twelve days. During the first four days, the temperature rises from ambient (30 - 35°C) to its maximum.

It is generally agreed that the purpose of piling is to achieve:

a. Uniform distribution of moisture in the material.

b. Complete enzymatic hydrolysis of pectin remaining in cuttings because its removal remains incomplete in retting.

These objectives are only partially realized in the conventional piling procedure. As shown by Ataur Rahman, the material on top of the pile can be nearly dry, whereas the moisture content at the bottom can be excessive (50 per cent). The peak temperature also varies across the pile, being often 70 - 80°C in the middle and about 45°C on the sides. It is obvious that material under these conditions can not become uniformly softened. Also, temperatures above 65°C are likely to terminate the action of pectinase - a fact apparently overlooked in earlier studies.

To correct these shortcomings, it is proposed that the moisture- and temperature gradients be eliminated by continuous, forced recycling of pile atmosphere. A laboratory apparatus for this purpose is shown in Figure 3. The prevailing temperature can be rapidly raised to an optimum, either by external steam heating or, by controlled intake of fresh air to boost aerobic activity. The maximum temperature should probably not exceed 65°C to allow thermophilic bacteria with pectinase activity to remain active. It is believed that laboratory data obtained with the apparatus described could be used to design analogous industrial piling chambers.

b. Chemical Depectinization

The researchers at BJRI have demonstrated that satisfactory softening of jute cuttings can be achieved by treatments with pectin solvents, such as hot dilute ammonium oxalate solutions. The advantage of chemical softening in comparison with the conventional piling consists of potentially more rapid and more uniform depectinization. On the other hand, the drawbacks of chemical softening include the cost of the chemical required and the necessity of washing and drying the cuttings before the addition of oil emulsion.

c. Potential Outlet for Jute Cuttings in the Pulp and Paper Industry

In order to determine the current use and future potential of jute cuttings in the pulp and paper industy, Dr. A. Rahman arranged a special visit to Karnafully Paper Mills and to Sylhet Pulp Processing. A Report of these visits is included as Annex I. The conclusions of these visits can be summarized as follows:

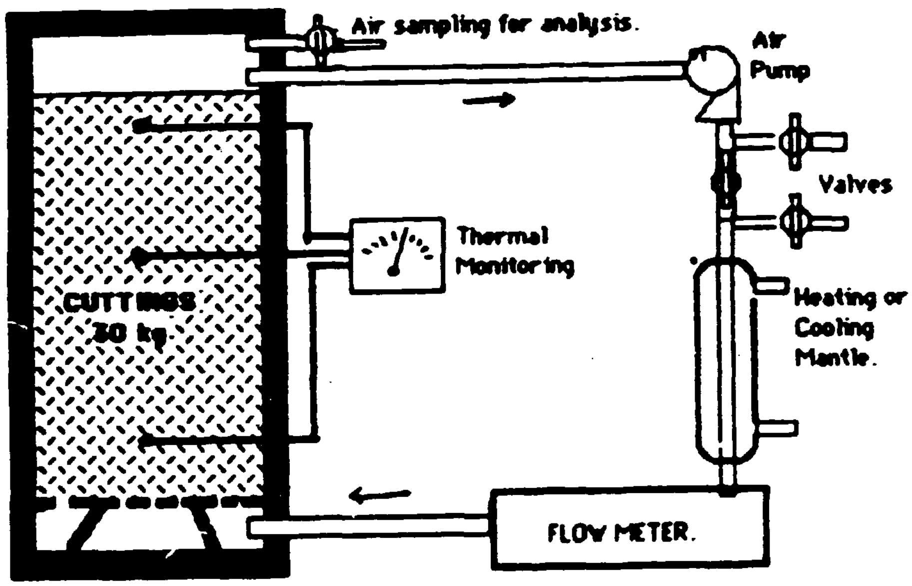

Figure 3. Proposed laboratory apparatus designed to eliminate moisture- and temperature gradients from piled jute cuttings and maintain an optimum temperature (65 °C) for pectinase activity.

i. Pulp superior to bamboo- and tropical hardwood pulps can be obtained from jute cuttings as well as from caddies and gunny sack waste.

ii. Fluctuating market price of cuttings and difficulties in their mechanical handling have so far limited their use as pulping raw material.

iii. Currently the soda process, combined with CEH bleaching, is utilized for pulping cuttings. An alternative pulping method, resulting in higher pulp yields and better brightness, would definitely increase the attractiveness of cuttings as pulping raw material.

Concerning the last-mentioned point, the Consultant formulated an exploratory research project to be carried out jointly with the Pulp and Paper Research Division, BCSIR (See Annex II). The plan was approved by Dr. M.A. Islam, C.S.O., and the project performed under the supervision of Mr. M.A. Khan, R.O. The study was carried out according to the conditions specified in the plan, with the following results:

Yield of unbleached pulp	65%
Brightness	56%
Kappa number	12.7

Unfortunately, the latter part of the plan, concerning the strength properties of the pulp and its combinations with groundwood, had to be delayed, because of inadequate beating equipment.

Although the study could not be completed, the results obtained so far are highly encouraging. The yield of unbleached pulp is 9 per cent higher than obtained in the soda process (56%). The brightness value is close to that required of newsprint reinforcement pulps. Consequently, a single stage peroxide bleaching would probably be adequate. The low kappa number (12.7) suggests that the pulping time used in the experiment was excessive and could be substantially shortened. If the strength properties of the peroxide bleached are found to be of the same order of magnitude as those of imported long-fibered reinforcement pulps, semialkaline anthraquinone pulping of jute cuttings appears to be an attractive alternative to soda pulping and deserves continued studies.

C. Bleaching of Jute

Jute is a composite fibre strand in which middle lamella lignin acts as an adhesive between intrinsic fibres and cannot be removed without drastic reduction in strength properties. Therefore, delignifying bleaching agents, such as sodium chlorite, have no place in jute technology. Consequently, attention should be focussed on non-delignifying agents such as:

- Hydrogen peroxide.
- Hypochlorites (under mild conditions only), such as bleaching powder and sodium hypochlorite.
- Reductive bleaching agents, such as hydrosulfites and, possibly, borohydrides or analogous carbonyl-reducing agents.

It should be also noted that it makes no sense to bleach jute to high brightness values (> 70 per cent), because the unavoidable photodiscoloration is conspicuous at these brightness levels.

Earlier bleaching studies at BJRI have admittedly been rather unfocussed. Like in other projects, no attention has been paid on critically important cost estimates. Practically no data are available on photodiscoloration sensitivity of bleached fabrics. It seems, however, that in the planning for future work these aspects have been adequately observed H (6-14).

D. Mercerization

It is well known that mercerization improves the softness, lustre, dry-and wet strengths of cotton, while these effects are absent in the mercerization of jute - the strength properties are actually significantly degenerated. Also, a part of jute dissolves in the mercerizing liquor. It is true that certain physical properties (elongation, woollenization effect) are improved, but overall, this Consultant feels sceptical about the merits of mercerization in the chemical modification of jute fabrics. Currently available data on this question appear to be insufficient, but will hopefully be complemented in connection with Project (5-13).

In the past, alternative bases for mercerization have been contemplated. Of these, relatively expensive chemicals, such as KOH, make no sense for obvious reasons. Liquid ammonia is of interest in preserving the strength characteristics of jute fibres. Its potential merits, perhaps, more thorough evaluation.

E. Cross Linking Studies

Imparting better resiliency to jute fabrics is obviously one of the most important research goals at the Institute. Laboratory experimentation with UF- and MF- precondensates has shown promising results (8-27) The project ought to move to a vigorous applied research stage. As far as epichlorohydrin concerned the following potential improvements are recommended:

- Current glass apparatus is inadequate for performing the cross-linking process and should be replaced by a much simpler autoclave set-up.
- Low alkali additions and increased reaction temperatures under pressure merit exploration.

F. Utilization of Jute Caddies and Wastes (8-33, 8-35)

Jute caddies and wastes have a natural and demonstrated raw material potential for the pulp and paper industry. Semialkaline sulfite-anthraquinone process would probably be the most attractive pulping method for these materials. It is proposed that Project 33 a would put the main emphasis on this alternative, possibly in cooperation with BCSIR.

The other contemplated alternative uses for jute wastes are, perhaps, less attractive. There could be market potential for non-woven products, but it may be expected to be a limited one (33b). Microbiological conversions of biomass materials are much in fashion today, but the results to-date have been disappointing.

G. Jute Reinforced Plastics

These products are curently manufactured in Bangladesh. It is unclear what specific advantages jute fiber has in comparison with glass fiber. Perhaps the Institute should not be involved in the rather specialized studies in this area, since the volume potential for jute in reinforced plastics is relatively minor.

H. Equipment Maintenance

The Institute has well-equipped machine shop and capable maintenance personnel which promptly respoads to needs for repair. The problem was that many jobs were not completed. During the Consultants visit, the follwoing equipment was partially repaired but remained inoperational at the time of departure:

- One Xenotest instrument.
- Small high-temperature dyeing apparatus.
- Two sterilization autoclaves.
- One high-pressure Hydrogenation autoclave.

VI. RECOMMENDATIONS

A. Equipment

The Institute needs the following laboratory equipment for general use:

1. Rotatory autoclave - order submitted to UNIDO, but not processed for unknown reason.

2. Freeze-drying apparatus. Essential for biological and lignin studies.

3. Capillary gas chromatograph, such as Hewlett-Packard 589DA (appr. Cost $7000) with integrator (HP-3392A), for sugar analysis and lignin characterization studies.

B. Acquisition of Information

Arrangements should be made for the library to process orders for copies of published papers and patents by Telex.

C. Reports

The quality of Semiannual and Annual Reports needs improvement. A more detailed List of Contents would be helpful to the reader. Most of the investigators need to improve their abilities to express themselves in English and to compose meaningful tables and diagrams.

D. Meetings and Seminars

It appears that investigators at the Institute rarely get together to discuss their research, generate new ideas and improve mutual cooperation, even if they work in related areas. It has been the experience elsewhere that this type of activity can be very beneficial, if conducted in an informal atmosphere. Periodic research review meetings in selected areas should be definitely encouraged.

Along the same line, the seminar program at the Institute could receive more emphasis. As it stands now, most seminars are given by visiting foreign experts. There is no reason why a weekly seminar program could not be initiated, with speakers from the Institute itself - those who have completed a significant research program, for example. In addition, selected seminar speakers could be invited from Jute Industry, Dhaka University or Bangladesh Council for Industrial and Scientific Research.

As far as the international visibility of the Institute and relations with foreign institutions are concerned, these could receive increased emphasis, if the Institute were to send representatives to international textile meetings to present papers. As it stands, the Institute does not have much contact even with its counterpart in Calcutta.

E. Pilot Plant Facilities

The facilities at the Institute are excellent and the machinery is modern. As a consequence, it has been possible to demonstrate the manufacturing potential of new products, based on novel methods of weaving, finishing and dyeing.

It seems likely, however, that the development of new chemical modification treatments for jute fibre will require the application of higher pressures and temperatures than can be reached with the currently available equipment, such as the High-Temperature Dyeing Apparatus. Examples of these types of treatments include sulfonation, discussed earlier in this report, and novel cross-linking procedures. Consideration should, therefore, be given to the acquisition of appropriate autoclaving equipment for use in the pilot plant facility.

F. Relationship Between the Institute and the Jute Industry

In general, the Institute maintains effective communication with the Jute Industry by frequent mill visits. On the other hand, it appears that industrial representatives do not visit the Institute often and are not well acquainted with its research program. Since the principal purpose of the Institute is to develop new improved technology for Jute mills, more intensive interaction with the jute industry is desirable, particularly in the formulation of its research program. This type of communication could be initiated by inviting selected industrial representatives to annual program preview meetings and by publishing a newsletter describing selected research projects underway at the Institute. These approaches have proven successful in many European and North American research laboratories. The research review committees could also include representatives of the pulp and paper industry, to provide advice specifically in chemical modification of jute fibres.

G. Future Directions of the Institute

The overall-all impression of the Technological Research Wing is quite favorable, reflecting in a very positive manner a successfully implemented UNIDO assistance program. Both the laboratories and the pilot plant are well-equipped and the training program has been largely successful. Many of the research programs are slowly producing results of obvious industrial application potential. The question which often remained unclear was how and when these results were going to be implemented on an industrial scale. Promising laboratory studies should obviously be followed by a realistic cost analysis and well-designed pilot plant studies. It appeared that in many cases, insufficient attention was paid to this crucially important phase.

It is precisely this aspect of the Institute activity - filling the gap between results obtained on the laboratory scale and the industrial application of these results - which not only requires increased attention at the Institute, but could also benefit from continued UNIDO assistance activity. In this sense, instituting a Phase II Program, with the main focus upon reinforcing the application aspect in research and development activity, would appear meaningful.

VI ACKNOWLEDGEMENT

The Consultant is thankful to Dr. Bhuiyan, the director of the Technological Wing, for excellent cooperation during the visit. The successful completion of the sulfonation program within a short time span was entirely due to the continuous attention, enthusiasm and aggressive vigor of Dr. A. Rahman, Chief of the Chemistry and Biochemistry Division. The implementation of the relevant experiments was in the competent hands of Mr. Mohammad Ali, S.S.O. and his assistants, Mr. Momen Mia, S.O., Mr. Nural Huq, Research Fellow, Mr. Mujibur Rahman, AEO and Mr. Farid Ahmed, AEO who all did an excellent job.

REPORT
on
Visits to Karnafully Paper Mills Ltd., C.J.M.C and
Sylhet Pulp Processing

by
Dr. K.V. Sarkanen, UNIDO Expert at
BJRI.

A. Purpose of Travel: To determine the potential of jute cuttings and whole mesta (Kenaf) as raw materials for pulp production. In addition, the current technology of soda and kraft pulping of jute was demonstrated to us in full scale. We also discussed potential cooperation in order to determine the applicability of Neutral Sulfite-Anthraquinone method to pulping of jute cuttings.

B. Travel Schedule, See Annex I

C. Travel Team included the following representatives of BJRI: Dr. A. Rahman, Chief, Chemistry & Biochemistry Division, Mr. Mohammad Ali, S.S.O., Mr. Ataur Rahman, S.S.O., and Mr. Momen Miah, S.O. In Chittagong, duscussions were also held with Dr. Jabbar Mian, Processor of Chemistry, Dhaka University, who was at that time visiting the University of Chittagong.

D. Karnafully Paper Mills, Ltd. Dr. Shafiqur Rahman, the General Manager, is personally interested in jute pulping having conducted research in this area. He emphasized that converting jute cuttings to pulp presants no problems - on the contrary, both the yield (55%) and quality of the pulps are excellent. This was demonstrated by reports on research conducted at Karnafully Paper Mills of which we received copies.

Dr. S. Rahman pointed out that the real problems in pulping jute cuttings lie in the mechanical handling of the material. It cannot be cut to proper size in conventional chippers and tends to entangle itself to large aggregates in the digestor. The development of new types of machines for the mechanical handling is probably needed. Otherwise, Dr. S. Rahman saw continuously increasing use for jute cuttings at Karnafully and Sylhet Paper Mills. Should the BJRI initiate a research programme in this area, he offered the following forms of assistance and cooperation:

a. Personal advising to BJRI in formulating the research program.
b. Conduction of pulping and pulp evaluation experiments at the Mill laboratory
c. Provision of literature material unavailable at BJRI.

The team had the opportunity of touring the excellent library and laboratory facilities of the mill. A partial tour of the kraft mill was conducted. The production capacity is currently 25000 annual tons, mainly from bamboo, but also from mixed tropical hardwood chips. This capacity will soon be increased. The paper mill has the amazing capabiluty of producing twenty different kinds of paper - and board products. A visit was also made to a new and ultramodern chipping installation at Kaptai's - a thought-provoling experience in view of the mechanical handling problems of jute cuttings. A partial tour of the rayon - and chemical plants was also conducted. During the visit, board and lodging was cordially provided to the team by the Karnafully Paper Mills Guest House.

E. Chittagong Forest Research Institute.

The Director of the Institute, Dr. M. Omar Ali assured us that cooperation with BJRI would be entirely feasible. The pulping department has the necessary equipment for experimentation. During the brief visit, we also saw the wood impregnation department, headed by Dr. Latif, a former Ph.D. student of this Expert.

F. Sylhet Pulp and Paper Mills Ltd.

This mill was established in the seventies and the current annual production capacity is 15000 tons using bamboo, reeds and jute cuttings and - waste (used gunny sacks, caddies etc.). Plans to double the production by enlarged raw material base are being drawn, and besides jute cuttings, consideration is being given to whole mesta, grown on company land (30000 to 40000 acres)

The mill is unique in having two separate production lines - one for bamboo and reeds and another, for jute. The later was, of course, of specific interest to us. Conversion of jute cutting bales to pulpable material was shown to us. It is a labor-intensive operation requiring the use of a bale crusher and and a manually fed minichipper. The soda pulping of jute is performed in a rotary digestor. After pulping, jute fibres tend to form entangled aggregates and consequently, the pulps and spent liquor must be treated in a hollander-type beater before the pulp can be pumped to washing. After CEHH-bleaching, jute pulp has substantially better strength properties than bamboo pulp.

In summary, experience at the Sylhet Mill has demonstrated that

a. Jute materials, whether in the form of whole jute, cuttings, gunny sack waste or caddies can be converted to good quality pulps on an industrial scale.

b. Jute pulps have clearly superior properties in comparison with pulps produced from other domestic raw materials, such as bamboo, mixed hardwoods or reeds.

We had a thorough and productive discussion in the office of the General Manager, Mr. A.H. Din Mohammad, Briefly, the conclusions reached were the following:

1. For a pulp mill, purchasing bales of jute cuttings for pulping has the disadvantage that no adequate machinery is available for the disintegration of bales into pulpable form. Therefore, a better alternative would be the following:

Cuttings should not be separated as fiber. Instead, farmers should be encouraged to separate the l wer parts of whole jute plants upon harvesting (1 to 1.5 ft.). These parts would not be retted at all, but transported directly to the pulp mill. In contrast to cutting bales, this material is readily chipped in conventional bamboo clippers. In addition, the pulp from the stick part will augment the pulp yield available from cutting fibers alone. Mr. Din Mohammad felt that this idea was a practical one and indicated that his mill would test it in practice.

2. An alternative to sodapulping of jute materials, the Neutral Sulfite Anthraquinone pulping was discussed in light of this Experts experimentation at the University of Washington in Seattle. The NS-AQ process has the following advantages in comparison with soda pulping:

- Higher pulp yield, appr. 70 per cent in comparison with 55 per cent yield in soda pulping.
- Higher brightness (55.65), requiring no (or only superficial) bleaching, if this pulp is used as reinforcement pulp for newsprint.
- Original fiber strands may not become completely desintegrated in beating. On this account, the NS-AQ pulp may be superior to bleached soda pulp for newsprint reinforcement.

It was agreed that if NS-AQ pulping would fulfil these expectations, the need for imported long-fibered pulp in newsprint manufacture could be substantiall reduced. At BJRI, discussions have been held with Dr. Aminul Islam of the BCSIR about joint work on the application of the NS-AQ method to pulping of jute cutting Mr. K.M.R. Islam Abu Wahed, Additional Chief Operations Manager at Sylhet indicate interest in conducting NS-AQ pulping experiments on jute in his mill laboratory.

3. The feasibility of growing whole jute or, preferably, mesta (kenaf) as r material for pulping was also discussed. Extensive work has been carri

out in this area by the Northern Regional Research Laboratory, Peoria, Illinois, USA. The results of these studies have been published and demonstrate that mesta pulps are far superior to hardwood pulps. A pulp mill based on mesta-raw material is in operation in Thailand. Sylhet Pulp Processing is considering initiating mesta growing for pulping in Bangladesh. During its visit, the team enjoyed the hospitality of the Sylhet Pulp Processing Guest House.

CONCLUSIONS ON THE EXPERIENCE GAINED DURING THE TRIP

Up to the present, not much attention has been given at the BJRI to the pulp and paper industry as a growing outlet for jute cuttings and waste. This situation clearly deserves re-evaluation, on the following grounds:

a. Jute cuttings, - waste and mesta appears to be the best domestically available raw materials for the production of high-quality pulps and the industry is eager to expand their use.

b. Currently there is balanced market demand for products from cuttings (carpet backing, gunny sack etc.) and from top part of jute. However, the demand for products from cuttings is continuously decreasing. As a consequence, in the foreseeable future, there will be excess production of non-marketable jute cuttings, unless additional outlets for this material are systematically developed.

It seems, therefore, that the BJRI ought to study and promote increased utilization of appropriate jute materials by the pulp and paper industry. This does not mean that the Institute should aquire apparatus for conducting pulping studies. Rather, it ought cooperate with the Bangladesh Council for Industrial and Scientific Research, already in possession of such equipment. The Institute's role in this cooperation could take several forms, for example:

a. Provisiom of appropriate jute materials for pulping experiments on the laboratory and pilot plant scale.

b. Development of harvesting methods in which the lower parts of jute plants would be directly separated for use by the pulp-and paper industry.

c. Development of productive and rapidly growing varieties of mesta to augment the raw material base of the rapidly growing pulp and paper industry.

Conversely, the BJRI stands much to gain from this type of cooperation in its efforts to develop new chemical modification methods for jute fiber and to improve current bleaching - and finishing practices. In these endeavors, constructive criticism by experts from the pulp and paper industry can be valuable. Furthermore, as experieance has already shown, in chemical fiber modification work, the Institute could (and should) utilize with great advantage the laboratory - and pilot plant equipment available at the BCSIR and at various pulp and paper mills.

Tour Programe to visit Chittagong and Sylhet

Date	Time	Departure	Arrival	Time
3 Aug, '84	7:30 A.M	Dhaka	Chandraghona, Chittagong Hill Tracts	9:00 P.M.
4 Aug, '84 & 5 Aug, '84		Halt at Chandraghona, Chittagong Hill Tracts to visit Karnafully Paper Mills, Rayon Complex, New bamboo chipper at Kaptai, Forest Research Laboratories & C.J.M. at Kalurghat, Chittagong		
6 Aug, '84	10.00 A.M.	Chandraghona	Chhatak, (7 Aug'84) Sylhet	2:15 A.M. (After midnight
7 Aug'84		Halt at Chhatak, Sylhet to visit Pulping Units.		
8 Aug, '84	7.00 A.M.	Chhatak, Sylhet	Dhaka	7:30 P.M.

FEASIBILITY OF PRODUCING LONG-FIBERED PULP FROM JUTE CUTTINGS FOR NEWSPRINT REINFORCEMENT BY THE NS-AQ PULPING METHOD.

Draft for a Cooperative Research Project between the Technology Wing, BJRI and the Pulp and Paper Department, BCSIR.

Purpose of the Study:

Curr.ntly, the newsprint mills in Bangladesh import relatively large quantities of long-fibered softwood pulp from North America and/or Nothern Europe, needed as reinforcement pulp in newsprint manufacture. Consequently, there has been a considerable interest, both in the pulp and paper industry as well as at the BCSIR, to reduce these imports by pulping long-fibered domestic raw materials such as jute cuttings. Specifically at BCSIR, a research program has been in progress for some time to produce long-fibered pulps from jute cuttings, whole jute or mesta by soda-AQ and kraft pulpings. These studies show considerable promise.

It was proposed by the UNIDO Expert at BJRI Dr. K.V. Sarkanen, that comparative studies should be conducted on jute cuttings using the novel neutral sulfite (or "semialkaline sulfite") - anthraquinone method. This method is used in Finland to prodice newsprint reinforcement pulps from softwoods. The pulps are obtained in high yields (7 pefcent higher than kraft) and possess satisfactofy brightness-and strength properties. Preliminary experiments at the University of Washington have indicated promise in applying this method to jute.

This proposal met with the approval of Dr. Amirul Islam, CSO, the Pulp and Paper Department of BCSIR and Dr. A. Rahman, Chief, Chemistry and Biochemistry Division, BJRI. The framework of the cooperative project is outlined briefly in the following:

A. Pulping

BJRI will supply a representative sample of jute cuttings (3.00 kg OD). The cuttings will be cut to "chips" of appr. 20 mm length at BCSIR.

a. Chemical charge, as per cent of OD jute:

.20 per cent Na_2SO_3 (as anhydrous Na_2SO_3)

8 ,, ,, Na_2CO_3 (as Na_2CO_3)

.05 ,, ,, anthraquinone

b. Pulping conditions:

- Temperature use : 90 minutes to 175 °C.
- Pulping time : 5 hrs. at 175°C

After pressure relief, excess liquor will be drained and the pulp washed and screened. Finally the pulp will be centrifuged, the centrifuged pulp weighed. From the moisure content, the yield will be estimated.

B. Pulp Evaluation

The pulp will be beaten to a convenient CSF by multiple passes through a Jordan mill. The conditions should be adjusted to promote hydration over cutting. About 20 hardsheets will be prepared from the pulp, for the following determinations:

- Kappa number
- Brightness
- Tensile, burst and tear strength
- Microscopy examination, to determine residual strand fragments.

The rest of the pulp will be collected to thick pulp sheets which will be air-dried and used to prepare appropriate mixtures with groundwood.

C. Newsprint Reinforcement Studies

Fifty-gram batches of mixtures of NS-AQ- and groundwood pulps will be prepared and converted to handsheets. The relative weight percentages of the power former pulp should be the following: 0, 5, 10, 15, 20, 25, 30, 50 per cent, respectively. The strength properties, brightness - and opacity values will be determined. In this manner comparison of NS-AQ pulp in newsprint reinforcement with long-fibered imported pulp, on one hand, and with alkaline jute cutting pulps, on the other, can be determined.