TCF BLEACHED SISAL MARKET PULP: POTENTIAL REINFORCING FIBRE FOR COMMODITY PAPERS - PART 1

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Presented at 1997 TAPPI Pulping Conference, October 19 - 23, 1997, San Francisco, California (published in Conference Proceedings, Book 1, 501-511)

ABSTRACT

Sisal pulp has physical characteristics superior to softwood kraft pulp. Depending on the furnish components and paper quality requirements, sisal pulp can replace softwood kraft at a rate of up to 2.8:1. This offers many opportunities for sisal pulp. For example, sisal pulp may be used as a reinforcing fibre in high recycle content papers, or its use may permit basis weight reductions while maintaining product quality. Sisal pulp as a value added replacement to softwood kraft in commodity papers is considered a viable alternative market.

Part 1 of this paper reviews the laboratory work to establish conditions for producing TCF bleached sisal pulp, and discusses the results of a pilot scale trial and tests on pulp samples which were distributed to paper companies for testing in various furnishes. Part 2 reviews a sisal estate plan to provide pulping fibre, tentative flowsheets for fibre preprocessing stations and a sisal market pulp mill using processes and equipment which are currently available on the market, and the estimated capital and manufacturing costs and economic analysis for a 50,000 metric ton per year sisal market pulp mill.

Keywords: sisal, line fibre, bole fibre, TCF bleaching

INTRODUCTION

Agave sisalana and its hybrids (commonly called sisal) traditionally have been cultivated for "line fibre" which is used in the manufacture of natural ropes, twine, sacking and carpet backing. Development of synthetic fibres has eroded the traditional sisal markets.

Sisal line fibre also is used as feedstock for high quality specialty pulp used in specialty paper products such as tea bags, plug wrap and surgical gauze. The market for specialty pulps is limited due to their high cost (US$2,400-2,600/admt) and the small size of specialty paper markets.

The aim of the work described in this paper was to develop a new product line which would revitalize the sisal industry in Tanzania, East Africa. The results presented are a condensation of a detailed feasibility study carried out during 1992/93 which established that there is a potentially large market for sisal pulp as a reinforcing fibre in commodity grade papers. The caveat is that the sisal pulp must be cost competitive with softwood kraft pulp based on the physical properties of the respective pulps.

THE PLANT

The sisal plant looks somewhat like an overgrown pineapple with a pineapple-like bole from which the leaves extend. For a mature plant, the bole is about 50 cm in height and about 20 cm in diameter, and the leaves can attain lengths of up to 2 m, but are usually about 1.0-1.2 m long. The leaves are about 12 cm wide, and are tipped with a sharp, highly lignified spine about 1.0-1.5 cm long.

Mature sisal field

The outside of the sisal leaf is covered with a well-developed epidermis with a waxy surface. The epidermis contains cutin, waxes and carbohydrates. The long fibre bundles that are extracted for line fibre are arranged under the epidermis in three or four parallel rows and another row of thinner fibres runs across the median of the leaf. The center of the leaf consists of pithy material containing plant juices and further randomly embedded fibre bundles.

Although the leaf fibre bundles extracted for cordage are very long (70-130 cm), the ultimate pulping fibre length ranges from 1.5-4 mm with an average of about 3 mm. The pithy material consists of amorphous cellulose, pectic material and protein. Juices contained in the pit cells are acidic (3.5-6 pH) and contain sugars, malic and citric acids, ascorbic acid, amino acids and sapogenins.

Sisal leaf cutting

In the bole, the ultimate pulp fibres are weaker than those from the leaves. The bole fibres are not in the form of fibre bundles except in the leaf butt ends attached to the boles. The base of the bole is woody and fibrous, but the ultimate pulp fibres are shorter and the actual fibre content is low. The upper portion becomes increasingly pithy and contains almost no fibrous material toward the growing tip. The bole is covered with a highly lignified cortical layer to which the leaves are attached. The bases of the leaves which remain attached to the bole when leaves are cut contain leaf fibre.

Bole fibre is less desirable for pulping because of the lower fibre content, the lower fibre strength, the highly lignified cortical layer and dirt trapped between the leaf butts and the bole. Nevertheless, pulping studies (1) have shown a good quality pulp can be produced if leaves and boles are pulped together provided that the amount of boles does not exceed 23% of the total green weight of leaves and boles. For the highest quality sisal pulp, only leaves would have to be used.

Table 1 provides the average composition of the sisal plant at 40 months as selected for pulping purposes (1).

Table 1. Average composition of sisal plant at 40 months

		Leaves	Boles	Total
Green Weight	kg	27	8	35
Fibre Content	%	6.0	4.8	5.7
Pith	%	6.1	8.7	6.7
Epidermal & cortical material	%	2.2	3.0	2.4
Solubles	%	2.5	7.3	3.6
Total Solids	%	16.8	23.8	18.4
Moisture	%	83.2	76.2	81.6

Notes: 1. Leaves include "spike" leaves.
2. Flowering poles excluded.

THE OBJECTIVES

The objectives of the 1992/93 feasibility study were to:

- establish markets for sisal pulps including estimated market size, technical preferences and pricing
- develop a process design to satisfy the market demand
- establish an overall concept from sisal cultivation to mill design which would meet market demand

THE MARKET

In 1992, we already had a significant amount of information concerning markets and technical parameters for sisal pulp, information which had been developed during a 1969/70 study by Alfred M. Hurter for Tanzania and a 1989 study by HurterConsult for the Dominican Republic. However, both of these studies had focussed on sisal pulp for the specialty pulp market. Thus, a new market survey was initiated to investigate the market potential for sisal pulp as reinforcing fibre in commodity papers.

As the potential market is global, it was impossible to conduct the market survey in person. Rather, using technical data developed during the earlier studies, a specific questionnaire and technical package were developed and mailed to 750 mills which could use sisal pulp either as specialty pulp (140 possible locations) or as a reinforcing fibre in commodity grades (610 possible locations). Response rate to the questionnaire was 27%.

Table 2 provides the combined results of the sisal pulp market survey both as specialty pulp and reinforcing fibre.

Table 2. Sisal pulp market potential

	Brightness	admt/year
Unbleached Pulp		53,000 - 55,000
Bleached Pulp	80-85% ISO	81,000 - 105,000
	85-90% ISO	82,000 - 108,000
	90-92% ISO	70,000 - 95,000
Total Sisal Pulp		**286,000 - 363,000**

Other key findings of the market survey were:

- specialty pulp users would be willing to pay high prices for both bleached and unbleached sisal pulps as they had been accustomed to the high pricing of currently available specialty pulps
- the potential market for sisal pulps as a reinforcing fibre would pay lower prices, prices which would be relative to softwood kraft pulps and the properties of the two pulps
- preference was given to TCF bleached pulps

The results of the market survey established that several changes had to be made to the study program:

- a laboratory and pilot plant test program was initiated to produce TCF bleached sisal pulp
- to establish a lower selling price in the reinforcing fibre market, the traditional sisal cultivation and processing system had to be redesigned to produce lower cost pulping fibre

LABORATORY EXPERIMENTAL WORK

The laboratory experimental work on Tanzanian sisal line fibre was carried out at the University of Quebec, Three Rivers, Canada under our direction.

Experimental Equipment, Procedures & Targets

a) Fibre Preparation

The sisal line fibre was cut to 5 cm lengths prior to cooking, and the fibre dryness was evaluated.

b) Cooking

Cooking was carried out in a double digester with indirect heating and forced liquor circulation. The capacity of each digester was 6.4 litres, and the liquor flow rate was 4 litres per minute. Each digester was charged with 700 grams o.d. sisal fibre and 4 litres of liquor which provided a liquor to fibre ratio of 5.7:1. Trials using both vapour and liquid phase impregnation were attempted. Liquor temperature was increased at a constant rate until the maximum temperature was reached. After the time at maximum temperature, the pressure was relieved over a 20 minute period and then the black liquor was removed. A black liquor sample was taken before pressure relief to permit evaluation of the residual alkali. After cooking, sisal line fibre retains much of its original appearance. Gentle agitation in a pulper was sufficient to break up the line fibre pieces into the ultimate fibres. The resulting pulp was washed and thickened before testing. A flat screen with 0.006" slots was used to measure rejects.

Regarding rejects, properly cooked and disintegrated sisal pulp should have little if any rejects. Also, if a blow tank is included, the action of blowing the pulp is sufficient to reduce the line fibre pieces to their ultimate fibres.

The target Kappa number after cooking was set at 14-18.

c) Oxygen Delignification

For oxygen delignification, a Quantum reactor was used. The dryness of the cooked pulp was measured, and the sodium hydroxide solution was standardized. The reactor bowl was preheated with water, and when the temperature was reached, the pulp and chemicals were added in the reactor. To eliminate air in the reactor, the inside pressure was decreased below zero, oxygen was added and the pressure was again decreased below zero. After air elimination, oxygen was added and the pressure was maintained at the set value. During the reaction time, the pulp was mixed at regular intervals. When the reaction was complete, the pressure was released. The pulp was filtered and the liquor kept for measuring residual alkali and pH. Then, the pulp was thoroughly washed. A flat screen with 0.006" slots was used to measure rejects.

Target Kappa number after delignification was set at 7-9.

d) Peroxide Bleaching

The pulp was pretreated with EDTA. After pretreatment, the pulp was washed twice at 2% consistency. Then, bleaching liquor was added to the pulp. After bleaching, the pulp was neutralized at pH 5.5 using sodium metabisulphite, and then washed once at 3% consistency. The target bleached pulp brightness was set at 80-85% ISO brightness or higher.

Cooking Results

Cooking conditions for both soda and soda-AQ cooks and selected results are provided in table 3. Complete test results are provided in table 6.

a) Soda Cooks

The first three cooks (tests NSV30, NSV45, NSV60) used vapour phase preimpregnation followed by cooking at

temperature for times varying from 30 to 60 minutes; however, the lowest Kappa number achieved was 27.9.

Using liquid phase preimpregnation, an effective alkali charge of 11% Na_2O and a maximum temperature of 165^0C resulted in a Kappa number of 28.0 (test NSL60). Increasing the effective alkali charge to 15% and the maximum temperature to 170^0C resulted in a Kappa number of 16.8 which was within the target range. As the target Kappa number had been achieved, this latter test identified as NS60 was selected for further testing and oxygen delignification.

b) Soda-AQ Cooks

The first soda-AQ cook, NS60AQ-15, used the same cooking conditions as the NS60 test with the exception that 0.1% AQ was added. The result of adding the AQ was a decrease in Kappa number from 16.8 to 10.8, a decrease in yield from 64.1% to 63.3%, a drop in viscosity from 29.6 cp to 18.9 cp, and a decrease in the rejects from 4.5% to 0.3%.

Since the above test overshot the target Kappa, a second test identified as NS60AQ-11 was carried out using 11% effective alkali. For this test versus the NS60AQ-15 test, the Kappa number increased from 10.8 to 16.1, the yield increased from 63.3% to 68.5% and the viscosity increased from 18.9 cp to 36.4 cp. However, the rejects also increased from 0.3% to 11.7%.

Oxygen Delignification Results

Oxygen delignification conditions are presented in table 4, and test results are presented in table 6. As the NS60AQ-15 cook had achieved a Kappa number of 10.8, delignification was not attempted on this sample.

For sample NS60, the Kappa number from cooking was 16.8. Initial oxygen delignification tests resulted in Kappa numbers of 14.8 and 14.3. Increasing the sodium hydroxide from 2.5 to 3.5%, the oxygen pressure from 35 to 80 psig, the temperature from 90 to 100^0C, the time from 50 to 120 min and the consistency from 10 to 20% resulted in a Kappa number of 8.1 which was within the target range. For the oxygen delignified pulp versus the cooked pulp, the brightness increased from 35.6 to 61.5% ISO, and the viscosity dropped from 29.6 to 17.6 cp.

The NS60AQ-11 sample was delignified at the same conditions as the NS60 sample, and a Kappa number of 8.4 was achieved. Compared to the NS60 sample, the brightness was lower (51.3% ISO versus 61.5% ISO) but the viscosity was higher (23.0 versus 17.6 cp).

EDTA-Peroxide (QP) Bleaching Results

EDTA-peroxide (QP) bleaching conditions are provided in table 5 for cooks NS60 and NS60AQ-11, both of which had been subjected to oxygen delignification, and for cook NS60AQ-15 which had not been subjected to oxygen delignification. Test results are summarized in table 6.

The QP bleaching resulted in a brightness of 87.0% ISO for the NS60 sample but the NS60AQ-15 and NS60AQ-11 samples only achieved 79.7 and 80.5% ISO, respectively. Viscosities of the three samples ranged from a low of 15.9 cc for the NS60 sample to a high of 16.9 cp for the NS60AQ-11 sample. CSF freeness of the bleached pulps are all in the same range and bleaching yields are similar.

Table 3. Cooking conditions - lab trials

Cooking Codes			NSV30	NSV45	NSV60	NSL60	NS60	NS60AQ-15	NS60AQ-11
Number of Cooks			1	1	1	1	3	1	1
Cooking Conditions									
Effective Alkali		$\%Na_2O$	11	11	11	11	15	15	11
Liquor to Sisal Ratio			5.7:1	5.7:1	5.7:1	5.7:1	5.7:1	5.7:1	5.7:1
Anthraquinone		%						0.1	0.1
Cooking Cycle									
Preimpregnation	Phase		vapour	vapour	vapour	liquid	liquid	liquid	liquid
	Pressure	psig	100	100	100				
	Temperature	^0C				70	70	70	70
	Time	min	20	20	20	15	15	15	15
Time to Cooking Conditions		min				95	100	100	100
Temperature Increase Rate		^0C/min	1	1	1	1	1	1	1
Maximum Temperature	^0C		165	165	165	165	170	170	170
Time at Temperature	min		30	45	60	60	60	60	60
Time to Relieve Pressure	min		20	20	20	20	20	20	20
Pulper (post cook disintegration)	min		30	30	30	30	30	30	30
SELECTED RESULTS (see table 6 for additional results)									
Kappa number			37.7	31.3	27.9	28.0	16.8	10.8	16.1
Residual Effective Alkali	g/l Na_2O		14.7				17.31	17.39	12.80
Residual pH			12.23				13.49	13.49	12.32

Table 4. Oxygen delignification conditions - lab trials

Sample		NS60			NS60AQ-11
Test Number		1	2	3	1
Delignification Conditions					
Preimpregnation	min			15	15
Consistency	%	10	10	20	20
NaOH	%	2.5	3.5	3.5	3.5
O_2 Pressure	psig	35	35	80	80
$MgSO_4$	%	0.3	0.3	0.3	0.3
Temperature	^0C	95	95	100	100
Time	min	60	120	120	120
SELECTED RESULTS (see table 6 for additional results)					
Kappa number		14.8	14.3	8.1	8.4
Residual Caustic	%			1.14	1.06
Residual pH				12.48	12.39

Table 5. EDT-Peroxide bleaching conditions - lab trials

Sample		NS60	NS60AQ-15	NS60AQ-11
Pre-Bleach Conditions				
Kappa #		8.1	10.8	8.4
Brightness	% ISO	65.9	38.4	51.3
EDTA Pretreatment Conditions				
EDTA	%	1.2	1.2	1.2
Consistency	%	3.0	3.0	3.0
Temperature	^{0}C	50	50	50
Time	min	30	30	30
pH		5.8	6.2	6.3
Peroxide Bleaching Conditions				
Na_2SiO_3	%	3.0	3.0	3.0
$MgSO_4$	%	0.05	0.05	0.05
NaOH	%	3.0	3.0	3.0
DTPA	%	0.2	0.2	0.2
H_2O_2	%	4.0	4.0	4.0
Consistency	%	12	12	12
Temperature	^{0}C	90	90	90
Time	min	120	120	120
SELECTED RESULTS (see table 6 for additional results)				
Brightness	% ISO	87.0	79.7	80.5

Table 6. Summary of key results - lab trials

Sample		NS60	NS60AQ-15	NS60AQ-11
Kappa Number				
Cooking		16.8	10.8	16.1
Oxygen delig.		8.1	none	8.4
TCF bleaching		3.5	3.9	4.6
Washed Yield				
Cooking	%	64.1	63.3	68.5
Oxygen delig.	%	97.0	none	97.0
TCF bleaching	%	98.5	99.0	99.1
Overall yield	%	61.2	62.2	65.8
REJECTS				
Cooking	%	4.5	0.3	11.7
Oxygen delig.	%	none	none	none
TCF bleaching	%	none	none	none
Brightness				
Cooking	% ISO	35.6	36.2	27.0
Oxygen delig.	% ISO	61.5	none	51.3
TCF bleaching	% ISO	87.0	79.7	80.5
Freeness, CSF				
Cooking	ml	730	705	700
Oxygen delig.	ml	711	none	663
TCF bleaching	ml	666	697	665
Viscosity				
Cooking	cp	29.6	18.9	36.4
Oxygen delig.	cp	17.6	none	23.0
TCF bleaching	cp	15.9	16.5	16.9

Discussion of Test Results

The purpose of the laboratory work was to establish preliminary conditions for producing TCF bleached sisal pulp in a pilot plant operation. Process optimization was not considered essential.

Table 6 presents a summary of the key results of the laboratory trials for soda cook NS60 and for two soda-AQ cooks. The lowest overall yield was for the soda cook whereas the highest overall yield was for the soda-AQ cook at 11% effective alkali. Cooking rejects varied from a low of 0.3% for the NS60AQ-15 trial to a high of 11.7% for the NS60AQ-11 trial. However, the issue of rejects is a matter for further consideration as the method used, screening on a flat screen with 0.006" slots, likely gives an erroneous result. In actual practice, the use of a blow tank and agitation will disperse any properly cooked line fibre pieces into their ultimate fibres. Regarding brightness, the soda cook offered the highest brightness at 87% ISO which was well above the other trial pulps.

Table 7 provides the physical and optical test results for the trial pulps, NS60, NS60AQ-15 and NS60AQ-11. The data clearly shows that the soda cook, sample NS60, has the highest burst factor and index, tear factor and index, and breaking length and tensile index, and the lowest dirt count. Although the yield for the NS60 test was the lowest, it was decided that the test conditions established for NS60 would be used for the pilot plant work as this sample offered the highest strength characteristics. As noted previously, how the laboratory handled the “rejects” could have resulted in higher yields. Finally, earlier work by Hurter (1) showed that higher yields could be achieved.

Table 7. Physical and optical tests - laboratory sisal pulps

Sample		NS60				NS60AQ-15				NS60AQ-11			
PHYSICAL TEST DATA													
Freeness, CSF	ml	666	550	450	300	697	550	450	300	665	550	450	300
PFI mill	revolutions	0	4192	7838	13306	0	3986	6840	11121	0	3972	7589	13014
Burst Factor	$gf*cm^2*m^2/g$	24.1	52.1	65.9	80.5	20.8	52.1	63.2	75.2	26.3	45.4	56.7	68.2
Burst Index	$kPa*m^2/g$	2.36	5.11	6.46	7.89	2.03	5.11	6.20	7.37	2.58	4.45	5.56	6.69
Tear Factor	$100\ gf*m^2/g$	200	326	296	273	197	285	254	209	206	278	293	266
Tear Index	$mN*m^2/g$	19.61	31.97	29.03	26.77	19.32	27.95	24.91	20.50	20.20	27.26	28.73	26.09
Tensile, B.L.	km	3.65	6.76	8.12	9.48	4.20	7.25	7.81	8.34	4.03	6.46	7.56	8.61
Tensile Index	N*m/g	35.79	66.29	79.63	92.97	39.42	71.10	76.59	81.79	39.52	63.35	74.14	84.44
Elongation	%	2.01	4.08	4.32	4.70	1.59	2.65	3.25	4.16	2.07	3.08	3.76	4.78
Fold (M.I.T.)	# double folds, 1.5 kg	6	51	115	230	4	51	106	205	7	40	80	139
Bulk	cc/gm	2.13	1.96	1.86	1.70	2.38	2.06	1.91	1.69	1.98	1.90	1.84	1.75
Opacity	%	73.0	71.6	70.4	68.8	77.3	74.9	70.7	69.4	71.5	70.5	70.5	70.5
OPTICAL & CHEMICAL TEST DATA													
Brightness	% ISO	87.0				79.7				80.5			
Pulp Dirt Count	mm^2/m^2	13				72				20			
Sheet Dirt Count	mm^2/m^2	10				48				15			
% Air Dry	%	23.6				21.8				28.4			
CED 0.5% Viscosity	cp	15.9				16.5				16.9			
Micro Kappa Number		3.5				3.9				4.6			
Alpha Cellulose	%	94.5				95.0				95.0			
Pitch (alcohol)	%	0.18				0.42				0.14			
Ash	%	0.42				0.85				0.85			

PILOT PLANT WORK

Pilot plant work was carried out at the Department of Wood and Paper Science, North Carolina State University (NCSU), Raleigh, North Carolina, under our direction.

Objectives

The objectives were: a) to evaluate the suitability and physical/chemical properties of Tanzanian sisal line fibre subjected to soda pulping and subsequent TCF bleaching using the cooking and bleaching conditions established for sample NS60 produced at the University of Quebec; and b) to generate 50-60 kg of the bleached, dried product for evaluation by potential clients.

Fibre Raw Material

Tanzanian sisal was extracted from all of the leaves of selected 42 month old sisal plants and of older plants. The leaves were decorticated using traditional methods, and the line fibre was air dried, baled and shipped to NCSU.

Equipment & Procedures

a) Fibre Preparation

The line fibre, approximately three feet long, was cut into lengths of 4-6 cm using a standard lever-arm paper cutter. Cut fibre was collected in heavy polyethylene bags, which were sealed and allowed to stand for several days. Samples were taken and analysed for moisture content.

b) Cooking

Cooking conditions for the pilot plant work are provided in table 8. These are the same conditions established for test NS60 conducted at the University of Quebec. The target Kappa number was 16-18.

Pulping was carried out in a 45 litre batch digester equipped with bottom to top sidearm liquor recirculation via an in-line pump with a rate of 35 lpm. The liquor was heated indirectly using a heat exchanger supplied with 150 psig steam.

Cut fibre was packed into the digester as tightly as possible giving an average batch size of about 7 kg (OD) of fibre. After packing the digester vessel, the soda cooking liquor made up of technical-grade sodium hydroxide and enough water to achieve the liquor-to-wood ratio were added. The digester was sealed, and the recirculation pump and heat exchanger were started. The preimpregnation temperature of 70^0C was reached in about 15 minutes, and the contents were held at this temperature for 15 minutes to complete the impregnation phase. The temperature was then increased at maximum rate in an effort to achieve the following conditions: maximum temperature 170^0C, 100 minutes to maximum temperature, 60 minutes at maximum temperature.

Occasionally, limitations in the heat exchanger only allowed a temperature of 168^0C to be attained. In these cases, the cook time was increased in order to maintain the same number of H-units.

At the completion of cooking, vessel pressure was relieved for 15-20 minutes until atmospheric conditions were achieved. The fibre was removed and transferred by bucket into a 680-litre stainless tank equipped with powerful agitation (2-HP Lightnin mixer). The fibre was diluted with hot tap water (54^0C) to a consistency of about 1% and agitated for thirty minutes to effect defibering. The final pulp was drained into a screen basket for dewatering and washing. After washing, the pulp was centrifuged, placed into sealed plastic bags, allowed to equilibrate for several days, and then tested for moisture content. The pulp was then tested for cooked yield, Kappa number, CED viscosity, and brightness.

Table 8. Cooking conditions - pilot plant

Cooking Conditions			
Effective Alkali		$\%Na_2O$	15
Liquor to Sisal Ratio			5.7:1
Cooking Cycle			
Preimpregnation	Temperature	^{0}C	70
	Time	min	15
Time to Cooking Conditions		min	100
Temperature Increase Rate		^{0}C/min	1
Maximum Temperature		^{0}C	170
Time at Temperature		min	60
Time to Relieve Pressure		min	20
Post Cook Disintegration		min	30

The first tests at these conditions resulted in Kappa numbers in the range of 10-12 which indicated that the NCSU equipment performed differently than equipment used at the University of Quebec. Due to time constraints, we instructed NCSU to proceed with cooking to 10-12 target Kappa numbers and to eliminate the oxygen delignification stage.

c) Bleaching

In preparation for bleaching, several bench-scale bleach tests were conducted in order to determine feasible conditions. Bleaching was done on 20-gram (OD) samples sealed in plastic bags and immersed in a water bath. Bench-scale bleaching conditions are provided in table 9.

Table 9. Bench-scale bleaching conditions - pilot plant

Trial #	1	1A	2	2A	3
EDTA Pretreatment					
% Consistency	3		3	3	3
% EDTA	1.2		1.2	1.2	1.2
Temperature, ^{0}C	50		50	50	50
Time, min	30		30	30	30
Bleaching Conditions					
Time, min	120	120	120	150	150
% Consistency	4	4	12	4	4
% H_2O_2	4	2	4	4	5
%NaOH	3	1.5	3	2	3
% DTPA	0.2	0.2	0.2	0.2	0.2
% $MgSO_4$	0.5	0.5	0.5	0.5	0.5
Residual H_2O_2, ml	15.8	11.8	9.5	18.8	20.6
Residual NaOH, ml	0.6	0.45	1.65	1.15	0.65
Brightness, % ISO	76.2	80.9	81.3	81.7	78.8
Notes: Trail 1A is the second stage of trail 1 with an intermediate wash					

Based on the bench-scale results and equipment limitations which only allowed bleaching at a maximum consistency of 4%, it was determined that the large-scale bleaching would be carried out in two stages. Large-scale bleaching conditions are provided in table 10.

For bleaching, the total mass of brown stock (62 kg) was split into three batches. Each batch was processed in the same 680-litre tank used for brown stock defibering. Heating was accomplished using an immersion heater with 150-psig steam. Dewatering and washing were carried out by draining the pulp into a large screen box, manually pressing to remove filtrate, and diluting with wash water.

Prior to bleaching, each batch was subjected to an EDTA pretreatment. The pulp mass was agitated during the entire treatment. After the pretreatment, the pulp was first dewatered as much as possible prior to washing. Washing efficiency was monitored by checking the pH and the effluent colour. Dilution and washing were carried out with distilled water. After washing, the pulp was again dewatered as much as possible and returned to the tank.

Each stage of peroxide bleaching was carried out in similar fashion, with the only difference being the amount of chemical applied. Pulp from the EDTA pretreatment was diluted to 4% consistency with distilled water. The stock was agitated and heated to approximately 95^{0}C. Then, the heater was removed from the tank in order to prevent baffling of the pulp mass. DTPA and magnesium sulfate, each dissolved in a small amount of distilled water, were added and allowed to distribute evenly. An amount of 50% hydrogen peroxide solution (commercial reagent grade) was measured and mixed into the required amount of 10% sodium hydroxide solution. This mixture was added slowly to the agitated pulp, in order to avoid disturbance of the mixing process. No additional heating was supplied during the 2-hour reaction time; the final temperature was around 75^{0}C. The stock was agitated during the entire bleaching process.

At completion of each stage of peroxide bleaching, sodium metabisulfite solution was added to the pulp until the pH was approximately 5.5 (this specification was changed to 6.0 for the second and third batches when it was decided that a pH of 5.5 required excessive washing). The pulp was allowed to agitate for 10 minutes prior to draining, thickening, and washing with cold tap water. Washing was monitored by checking filtrate pH and was deemed complete when the pH was between 6.5 and 7.

After the final stage of bleaching, washed pulp from each batch was dewatered as much as possible and then stored in sealed plastic bags. Ambient room temperature in the storage area was approximately 21^{0}C. Pulp from the first batch was stored in this fashion for three days prior to being processed on the paper machine.

Table 10. Large-scale bleaching conditions - pilot plant

Stage	1	2
EDTA Pretreatment		
% Consistency	3	
% EDTA	1.2	
Temperature, ^{0}C	50	
Time, min	30	
Bleaching Conditions		
% Consistency	4	4
% H_2O_2	4	3
%NaOH	3	2.25
% DTPA	0.2	0.2
% $MgSO_4$	0.5	0.5
Temperature, ^{0}C	90	90
Time, min	120	120

d) Cleaning

The bleached pulp batches were combined in a 9500-litre agitated tank and diluted to 1% consistency. The pulp was allowed to agitate overnight to insure maximum fibre mixing. The pulp was then pumped through a single-body Beloit Posiflow forward cleaner. Feed rate was approximately 70 gpm,

inlet pressure was 30 psig, and outlet accept pressure was 10 psig. Reject flow was approximately 2 litres/minute. Cleaner accepts were returned to the feed tank, which was continuously agitated. Cleaning was continued for approximately three passes. Rejects were collected, dewatered, and saved for analysis.

The pulp was next pumped through a double-body Beloit Uniflow reverse cleaner, using process conditions as described above. The reject rate was excessive, yet no contaminants appeared to be present in the rejects. This cleaner run was aborted after several minutes.

e) Undried Pulp Testing

Three samples of the cleaned pulp (undried) were taken and beaten in a PFI mill to freenesses of 549, 453 and 325 CSF. Standard hand sheets were made from an unbeaten sample and each beaten pulp, and tested for physical properties. The unbeaten pulp was tested for optical and chemical properties. Test results are provided in table 12.

f) Pulp Drying

The pulp was formed and dried on a pilot Fourdrinier machine without any prior refining of the pulp. The pilot machine had the following attributes:

- open head box (consistency 0.2-0.5%)
- white water recycle
- table rolls
- vacuum boxes
- vacuum couch roll
- forward, reversing, and smoothing presses
- steam-heated dryer cans, serpentine run

The machine was run at approximately 12 metres/minute, with a deckle width of 30.4 cm. Because extreme feathering of the sheet edge was destroying the sheet in the press section, it was necessary to trim approximately 2.54 cm from each edge. The sheet was pressed very lightly and dried just until the moisture was between 15-20%.

Sheet basis weight varied between 115 and 180 g/m2. Basis weight and moisture for the sheet were determined continuously using a Measurex 2002 Scanner.

Three samples of the dried pulp were subjected to PFI beating to three different freenesses. Standard hand sheets were made from each beaten pulp and an unbeaten sample, and tested for tensile and tearing strength.

Cooking Results, Observations and Discussions

Table 11 provides the cooking and brown stock data for 31 cooks.

Table 11. Cooking and brown stock data - pilot plant

Cook #	Kappa #	Viscosity, cp	% Yield	H-Factor
1	10.0	23.4	70.0	1205
2	11.1	36.5	68.8	1215
3	10.8	35.2	67.0	1224
4	10.3		67.6	1229
5	9.7		67.1	1226
6	10.6	30.0	67.9	1216
7	10.0	29.6	67.6	1230
8	10.3	29.3	67.1	1203
9	12.2	34.4	67.2	1184
10	11.5	38.5	67.0	1197
11	11.0	35.5	67.9	1259
12	10.5	38.9	67.6	1250
13	9.8	42.0	67.6	1241
14	10.5	40.4	67.2	1253
15	11.4	35.2	66.3	1255
16	10.2	37.2	67.7	1244
17	10.7	38.6	68.2	1252
18	10.3	38.2	68.2	1251
19	10.6	45.3	68.2	1254
20	11.0	42.7	67.4	1252
21	10.3	31.9	67.3	1255
22*	11.7	33.0	66.6	1247
23	10.5	35.6	67.2	1243
24	10.8	32.0	67.5	1247
25*	10.9	32.8	67.1	1251
26*	10.0	35.0	66.7	1250
27*	10.9	32.6	66.7	1309
28*	10.2	34.2	67.8	1348
29*	10.5	29.5	66.8	1336
30*	9.9	30.0	67.0	1343
31*	11.5			1342

Note: The * indicates cooks using bale #2 fibre.

Overall, the cooked fibre had a pinkish-red colour, with an "oatsy" odour similar to that from other nonwood fibres. Except for the colour, the fibre appeared to be little changed as compared to the uncooked state. When the cooked fibre was agitated in hot tap water, it quickly disintegrated into individual fibres. The disintegrated pulp was light pinkish-gray in colour, with a smooth and soft feel. Some undefibered pieces were seen during disintegration, but they tended to be ragged out of the pulp by the mixer shaft and impeller. Otherwise, the pulp was remarkably free of shives and foreign materials.

There was some concern about the uniformity of a large batch of brown stock generated by multiple small batches. The process and test data in table 11 show that the Kappa numbers and yields for the batches were relatively uniform. There is a larger spread for the viscosities, but it is not known if this is due to some special cause or simply natural variability. The brightness of each batch of pulp was consistently in the 41-42 ISO range. Overall, the multiple-batch method appeared to produce pulp of uniform quality.

Most of the fibre used in cooking was taken from bales marked #3 and #4. The fibre from these bales was from plants of the same age, and yielded consistent cooking results at the same H-factor. However, it later became necessary to use some fibre from bale #2 which was from older plants. Cook #22 was the first batch using this older fibre, and the Kappa number was slightly higher at the same H-factor used for bales #3 and #4. When the H-factor was raised slightly, this difference was eliminated.

Black liquor from Cooks #2 and #3 was analysed and found to have residual effective alkali levels of 8.1 and 6.8 g/l, respectively.

Bleaching Results, Observations and Discussions

Results from the bench-scale exploratory bleaching studies are shown in table 9. For the large-scale bleaching, it was decided to use low-consistency bleaching with two stages of hydrogen peroxide. First-stage peroxide application was 4%, while the second stage application was 3%. These dosages were not optimal, but were set so as to ensure that the minimum brightness of 82% ISO was achieved.

When the pulp was dewatered after pretreatment with EDTA, the effluent had a deep yellow-brown colour. After several washes, the effluent became colourless.

In both stages, the pulp responded rapidly to the addition of peroxide. Brightening began almost immediately as the pulp turned from brown to yellow. Mottled areas in the pulp disappeared within five minutes, indicating that the chemical was being distributed evenly. A sample taken after the first bleaching stage had a brightness of 71.5% ISO.

The final brightness for the combined pulps was 82.3% ISO, so it appears that about 73% of overall brightening was achieved in the first stage. Each bleaching stage had an end pH of 11.0-11.4.

When sodium metabisulfite solution was added at the end of each bleaching stage, the pulp turned significantly whiter and brighter, and this brightness was maintained throughout washing.

First-stage bleaching was quite effective at removing the few shives observed in the brown stock. In fact, the pulp appeared to be shive-free prior to second-stage bleaching. Overall, very few natural contaminants were seen in the bleached pulp. Some foreign contamination was seen in the form of black rubber particles, rusted metal flakes, and what appeared to be silver solder. The source of the metal flakes was determined to be a mixer shaft. However, the sources of the other two contaminants remain a mystery.

Cleaning & Pulp Drying Observations

During operation of the forward centrifugal cleaners, it appeared that a large portion of the foreign contaminants were rejected. Unfortunately, it also appeared that a higher amount of good fibre was being rejected than is usually noticed for either bleached softwood or secondary fibre pulps. The rejects tended to rope when discharging from the cleaner requiring frequent nozzle cleaning.

When the reverse cleaners were started, a large amount of fibre was rejected. The fibre appeared to contain few lightweight contaminants. It was decided that the risk of fibre loss outweighed potential cleaning benefits, and the process was stopped after several minutes.

Papermaking using the unrefined bleached sisal fibre was significantly more difficult than had been anticipated. Due to poor trim cutting due to the high sheet weight and inherent fibre strength, after several minutes of operation, it became necessary to reduce basis weight from approximately 180 g/m2 to 150 g/m2.

The final dried sheet appeared to be of high quality and cleanliness, with only occasional foreign contaminants.

Data from physical and chemical properties testing for the final, never-dried pulp is shown in table 12. The strength test data of the bleached pulp appears to be good and consistent with the findings from the University of Quebec.

It should be noted that the PFI mill used for this study had just been recalibrated using standard reference pulps. Some testing for selected properties was also done on the dried product and the results also are provided in table 12.

Table 12. Physical, optical and chemical properties for TCF bleached sisal pulp - pilot plant

Sample		Never-Dried TCF Beached Sisal Pulp				Machine-Dried TCF Bleached Sisal Pulp			
Physical Test Data									
Freeness, CSF	ml	599	549	453	325	624	565	486	385
PFI mill	revolutions	0	750	2500	4500	0	750	2500	5000
Burst Factor	$gf*cm^2*m^2/g$	27.6	42.0	59.6	76.1				
Burst Index	$kPa*m^2/g$	2.71	4.12	5.84	7.46				
Tear Factor	$100\ gf*m^2/g$	236	320	259	197	191	267	311	219
Tear Index	$mN*m^2/g$	23.14	31.38	25.40	19.32	18.73	26.18	30.50	21.48
Tensile, B.L.	km	3.96	6.11	7.80	9.78	3.52	5.18	7.17	8.98
Tensile Index	N*m/g	38.83	59.92	76.49	95.91	34.52	50.80	70.31	88.06
Elongation	%	2.62	4.14	5.17	6.41	2.28	3.42	4.71	5.88
Fold (M.I.T.)	# double folds, 1.5 kg	5	26	93	230				
Bulk	cc/gm	2.03	1.75	1.62	1.48	2.16	1.89	1.69	1.55
Opacity	%	75.6	73.4	70.7	67.7				
FIBRE FRACTIONATION (Bauer McNett)									
Screen #	28	48	100	200	P200				
% Retained	75.7	12.6	9.4	0.5	1.8				
OPTICAL & CHEMICAL TEST DATA									
Nominal Brightness	% ISO	82.3							
Reverted Brightness	% ISO	80.7							
CED 0.5% Viscosity	cp	18.0							
Kappa Number		6.4							
Alpha Cellulose	%	95.61							
Beta Cellulose	%	3.42							
Gamma Cellulose	%	0.975							
Ash Content	%	0.35							
Pitch in alcohol/benzene	%	2.1							

COMMENTARY ON RESEARCH WORK

Although the process parameters were not optimized, the experimental and pilot plant work clearly show that high quality bleached sisal pulp can be produced using either a soda or a soda-AQ cook followed by either oxygen delignification and peroxide bleaching or simply peroxide bleaching.

It appears that the final pulp brightness will be in the 80-84% ISO range if cooking is followed by peroxide bleaching only and that the brightness will increase to the 85-90% ISO range if cooking is followed by oxygen delignification and then peroxide bleaching. In both cases, it is anticipated that process optimization of all stages of the process - cooking, delignification and peroxide bleaching - will improve the overall results.

Samples of the unrefined TCF bleached sisal pulp produced at NCSU was distributed to various paper companies in North America, Europe and elsewhere for testing. Although the results of these tests are not available for general publication due to confidentiality issues with each company, the general findings were:

- sisal pulp has a tear strength twice that of softwood pulp and three times that of hardwood pulp

- minor refining is recommended to develop the tensile strength of sisal pulp without hurting the tear strength

- sisal pulp could be used as a reinforcing fibre in many commodity paper grades, including grades which contain a high recycled fibre content

- sisal pulp could be used to replace softwood or other expensive high strength pulps

- although the specialty paper market would pay high prices for sisal pulp, the potential reinforcing fibre market would pay lower prices for sisal pulps, and the price would be relative to softwood kraft pulp and the respective properties of the two pulps

ACKNOWLEDGEMENTS

The work cited in this paper could not have been carried out without the cooperation of the Tanzania Sisal Authority (TSA), Tanga, Tanzania which provided the sisal fibre and assisted in local data collection and the Canadian International Development Agency (CIDA) which co-funded the feasibility study.

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