Pulping and TCF Bleaching of Canadian Seed Flax Straw

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ABSTRACT

A large amount of cereal crops, including seed flax, are grown in the Canadian Prairies. Because of the bast fiber in flax straw, incorporation of the straw back into the soil is not possible so it is either burned off or removed from the fields. This study evaluated seed flax straw as a raw material for papermaking pulp production. Trials were carried out on samples processed in a Tornado Pulper: whole flax straw, separated bast fiber, and separated bast fiber treated with enzymes. These were compared to commercially-available dry decorticated bast fiber. Pulping was done using the soda-AQ process followed by TCF bleaching using a QPpZ sequence for a target brightness of greater than 85% ISO. Strength properties of the commercially available dry decorticated bast fiber and of the enzymatically treated materials were significantly higher than the Tornado-processed materials indicating that the Tornado-processed flax straw had not been properly retted prior to processing.

Keywords: Linum usitatissimum, flax straw, pulping, bleaching, pulp properties

INTRODUCTION

Seed flax, *Linum usitatissimum,* is an important rotational crop in the Canadian Prairies where it fits well in crop rotations in all soil zones. Canadian flax seed production accounts for about 40% of global production (1).

Figure 1 Flax field in bloom

Most seed flax is grown in Saskatchewan with smaller amounts in Manitoba and Eastern Alberta. Harvested area varies from year to year as shown in Table 1 (2). Seeded area is actually larger but not all of the seeded area is typically harvested for various reasons.

Table 1 Harvested seed flax area (000's ha)

Year	Alberta	Saskatchewan	Manitoba	Total
2009	16.2	489.7	107.2	613.1
2010	15.4	271.1	62.7	349.2
2011	27.5	213.3	50.6	291.7
2012	22.3	275.2	60.7	358.2
2013	36.4	331.8	43.7	411.9

2014	54.6	526.1	48.2	628.9
2015	48.2	530.1	48.6	626.9
2016	29.1	279.2	33.2	341.5
2017	34.4	360.2	24.3	418.9
2018	37.1	287.5	14.8	339.4

For the past ten years, the total harvested seed flax area varied from a low of 291,700 hectares to a high of 628,900 hectares. Using Atchison's estimate of collectable seed flax straw per hectare of 1.1 to 1.7 metric tons per hectare (3), the annual amount of flax straw varied from 320,000 to 1,070,000 metric tons. This estimate is similar to that of the Flax Council of Canada, which estimates availability of 500,000 to 1,000,000 metric tons annually depending on planted acreage and rainfall (4).

Flax is a dicotyledon, or dicot, and contains two types of fiber: long bast fibers and short core fibers called shive. As shown in Figure 2, the bast fibers are in bundles running lengthwise in the outer part stem between the epidermis and the shive (5).

From a pulp and paper perspective, it is the long bast fibers that are desirable. Individual bast fibers have (6):

- fiber length ranging from 10,000-45,000 microns, average 27,000 microns,
- fiber diameter of 16-30 microns, average of 22 microns, and
- aspect ratio of 1250:1.

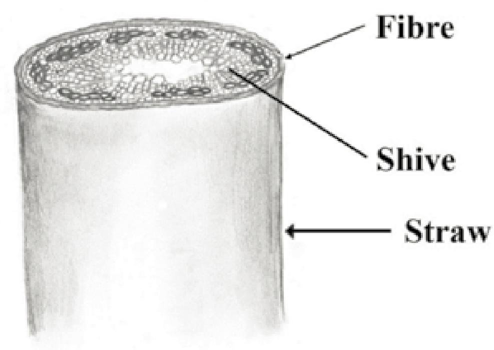

Figure 2 Flax stem cross-section

As reported by Tschirner and Satyavolu (7), the bast and shive have significant differences in chemical compositions as shown in Table 2.

Table 2 Typical composition of flax and wood fibers

	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives (%)
Flax Straw	63.33	8.16	14.66	2.22
Clean Bast Fiber	78.23	6.16	5.04	1.63
Clean Shives	53.12	13.16	24.01	1.52
Hardwood	43-47	25-35	16-24	2-8
Softwood	40-44	25-29	25-31	5

Flax straw usually contains between 15-25% bast fiber depending on a variety of factors including the flax variety, soil conditions, farming practices and rainfall (4). Others have reported that it can be as low as 10% and as high as 30%.

Historically, the largest users of Canadian seed flax straw have been Schewitzer-Mauduit Canada (SMC) and P. H. Glatfelter, both of which primarily used extracted bast fiber for making pulp for cigarette burning tube paper. With the decline in cigarette consumption as well as the switch to a wood-based burning tube paper, flax use has also declined. Only SMC continues to decorticate flax straw for the production of paper for cigarettes and some plastic composite products. There are several much smaller buyers and processors.

SMC uses both a fixed decortication facility as well as mobile systems. SMC purchases flax straw standing in the fields, and arranges for baling in round bales and transport to various locations for storage. Fields are selected for high bast fiber content as well as low trash (plastic litter etc.). The bales are transported to storage areas where they are put into piles five bales high containing about 600 metric tons. Piles are spaced far apart to avoid fire risks from lightning strikes. The flax is left in storage for 12–18 months to allow natural retting to take place. Once retted, the bales are retrieved from storage and processed through a specially designed decortication system to extract bast fiber. The decorticated bast fiber with a bast content of about 60-65% is baled and shipped to their pulp mill in New Jersey. Reject shive is collected and returned to farm fields.

Figure 3 Retrieving flax bales from storage

Figure 4 Dry decortication & baled flax bast fiber

Proper retting is critical to good pulp properties. During the retting process, not only is there a loosening of the bond between the bast and core fiber fractions, but bast fiber containing raw materials also undergo a significant chemical change resulting from microbiological action on the fiber. This chemical change alters the physical properties of the final pulp product.

Flax bast fiber is notoriously difficult to process using traditional methods. The extreme fiber length leads to hydro-entangling, roping, and plugging of piping, pumps, and other equipment. Some kind of pretreatment must be done on the bast-containing materials in order to produce a pumpable slurry that can be processed further.

This study was carried out to:

- determine if whole flax straw could be wet processed into a uniform slurry suitable for whole straw pulping,
- determine if the whole flax straw slurry could be hydraulically separated into its bast and shive fractions,
- establish pulping and TCF bleaching conditions for whole flax straw and separated bast fiber to produce paper grade pulps, and
- evaluate the pulp properties.

EXPERIMENTAL

Initial Materials

Large round bales of Manitoba seed flax straw weighing about 500 kg were provided by a third party.

Wet Preparation

a) Whole flax straw pretreatment and processing

The Tornado pulper (Fig. 5) was developed by Bolton Emerson Americas to process high wet strength materials for recycling and to prepare cotton linters and staple fiber before chemical pulping. The Tornado pulper is a side entering unit equipped with a rotor and stator that have cutting elements that reduce the size of materials in the tank. Everything entering the tank must pass through the rotor and stator to exit. There are two commercial sizes: 24" & 36". The flow rate through the 36" Tornado is 3,000 gal/min (11,536 l/min).

The 36" Tornado located at the Herty Foundation in Savannah, GA was used for whole flax straw processing. It is equipped with a 400 HP (298 kW) motor.

The Tornado was filled with warm water, and large slabs of whole straw were introduced into the pulper tank to a consistency of 5%. The Tornado was operated in recirculation mode until the straw had been reduced to a uniform and pumpable slurry.

Several 45-gallon drums of processed whole flax were collected, treated with a small dose of biocide, and shipped to North Carolina State University (NCSU) for cold storage prior to further testing.

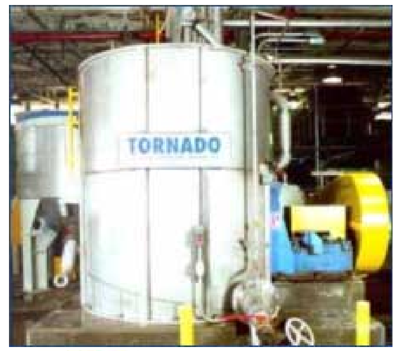

Figure 5 Tornado pulper

b) Hydraulic bast fiber separation

The apparatus used to separate the bast fiber was based on hydrocyclone cleaners. While the work was done at Herty, it was done using a semi-portable wheel mounted unit delivered from NCSU and operated by NCSU personnel.

This unit included a 760 liter tank equipped with a pump capable of developing about 480 Kilopascals head and plumbed with 32 mm sanitary piping and fittings. It is equipped to handle 1 forward and 1 reverse commercial hydrcyclone cleaner. For the trials, the unit was setup with a Beloit Posiflow (forward) and a Beloit Uniflow (reverse) cleaner. Electrical requirements were 220 VAC with 100 amperes service for the 18.6 kW pump motor.

6 OD kg of Tornado processed whole flax straw was dispersed in 600 liters of tap water in the test stand tank. Initial separation was carried out with the Posiflow cleaner at about 1.0 % consistency. Accepts from this separation were collected, diluted to about 0.4 % consistency and

run through the Uniflow cleaner. Rejects from the initial separation were collected, diluted to about 0.75% consistency and run through the Posiflow cleaner a second time.

Bast fiber accepts from the Uniflow cleaner were collected, treated with a small dose of biocide, and shipped to NCSU for cold storage prior to further testing.

Cooking & Bleaching

Cooking and bleaching work was carried out at the North Carolina State University Department of Forest Biomaterials laboratory under the direction of HurterConsult. All tests were performed in accordance with TAPPI Test Methods.

a) Cooking

The cooking process tested was soda-anthraquinone (soda-AQ). Anthraquinone is a well-known pulping additive that typically produces higher yield and better pulp properties than for pulping using only sodium hydroxide (soda). HurterConsult established a target Kappa number in the range of 10 - 15.

For Tornado processed whole flax straw, three cooks were carried out using 3-liter stainless bombs placed on a rotating rack in a heated-air oven.

For hydraulically separated bast fiber, four cooks were carried out in the same 3-liter bombs.

Table 3 provides the cooking conditions for the two Tornado processed materials. These were selected based on cooking conditions typically used for dry decorticated bast fiber with 60 – 65% bast.

Table 3 Cooking conditions

	Range	**Optimum**	
		Whole straw	**Separated bast**
Sample size	350 od grams	350 od grams	350 od grams
NaOH on OD fiber	20%	20%	20%
AQ on OD fiber	0.1%	0.1%	0.1%
Liquor-to-fiber ratio	5:1	5:1 start	5:1 start
Maximum temp.	160 - 170 ^{0}C	168 ^{0}C	160 ^{0}C
Time to max temp.	60 min	60 min	60 min
Time at max temp.	120 min	120 min	120 min
Post cooking treatment	mixed, washed and screened	mixed, washed and screened	mixed, washed and screened

b) Bleaching

Unbleached pulps from the cooking trials were bleached using a QPZ sequence with a target brightness of 85% ISO with conditions established by HurterConsult.

The chelation stage (Q) was done in an open container placed into a heated water bath. Pulp was mixed with sulfuric acid to achieve a ph of 5. DTPA was then added, and the mixture was mixed periodically. After reaction, the pulp was washed thoroughly with distilled water.

Pressurized peroxide bleaching (Pp) was done in 3-liter bombs lined with Teflon to reduce peroxide degradation. The bombs were placed on a rotating rack inside of a heated-air oven. Pulp was mixed with chemicals using an industrial kitchen mixer fitted with a kneading spade.

Low-consistency bleaching using ozone (Z) was carried out by sparging ozone gas into a mixing vortex of pulp slurry.

After each stage, the pulp was tested for the following: brightness, Kappa number, freeness (CSF), yield, and chemical consumption.

The same bleaching conditions and chemical charges were used for both unbleached pulps per Table 4.

Table 4 Bleaching conditions

Sequence			QPZ
Q stage	consistency	%	4.0
	temperature	^0C	55
	time	minutes	35
	pH		5
	DTPA	% on od fiber	0.5
P stage	consistency	%	12.0
	temperature	^0C	105
	time	minutes	90
	H_2O_2	% on od fiber	5.0
	NaOH	% on od fiber	4.0
	DPA	% on od fiber	0.2
	$MgSO_4$	% on od fiber	0.5
	Na_2SiO_3	% on od fiber	0.5
Z stage	consistency	%	3.0
	temperature	^0C	20
	pH		1.6

Pulp Testing

The bleached pulps were refined using a PFI mill and then tested for Canadian Standard Freeness.

Standard handsheets, with a target basis weight of 65 g/m2, were made using a British sheet mould, according to TAPPI standards.

RESULTS AND DISCUSSION

Wet Preparation

a) Whole flax straw processing

The Tornado pulper running in recirculation mode for 30 – 35 minutes was able to cut all of the bast fiber to lengths of 8 – 12 mm allowing the whole straw slurry to be pumpable.

a) Hydraulic bast fiber separation

Separation of the bast and shive was excellent. Visual inspection of the shive fraction showed less than 5% bast content and the bast fraction was shive free.

However, on a mass balance, the bast fraction was only 12.5% by weight of the total fed to the separation system. This could have been the result of:

- the flax straw provided had a low bast fiber content – as mentioned about it typically ranges from 15 – 25% (10 – 30%),
- recirculation through the Tornado multiple times sized reduced some of the bast to the extent that it was separated with the shive, and
- a combination of the above.

Unfortunately, all of the flax straw bales were processed so it was not possible to determine if a low bast content in the straw was the primary reason.

Cooking

Table 5 provides the cooking results.

The lower yield for whole straw versus separate bast was expected due to the higher lignin content of the shive.

However, the 4.1 Kappa number for separated bast was unexpectedly low, especially with an alkali consumption of only 62.6% and given that the cooking conditions used were similar to those for dry decorticated bast. It is believed that this occurred because the hydraulically separated bast fiber was virtually shive free versus 35 – 40% shive in dry decorticated bast fiber.

For future work on hydraulically separated bast fiber, the absence of shive will require adjustments to the cooking conditions.

Table 5 Cooking results

		Whole straw	Separated bast
Kappa number		15.2	4.1
Brightness	% ISO	35.3	52.2
Screened yield	%	46.3	60.6
Screened pulp freeness	CSF, ml	350	390
Alkali consumption	%	85.0	62.6
Black liquor terminal pH		11.6	12.8

Bleaching

Table 6 provides the QPZ bleaching results.

Table 6 Bleaching results

			Whole straw	Separated bast
Sequence			QPZ	QPZ
P stage	Brightness	% ISO	79.7	85.1
	Reversion tests	% ISO, 1/3 hr		84.8 / 84.5
	Kappa number		5.9	2.3
	Freeness	CSF, ml		
	Pulp yield	%	94.5	96.6
	Initial / final pH		na / 11.4	na / 12.5
	H_2O_2 consumption	% on od fiber	99.9	94.6
Z stage	Brightness	% ISO	86.3	89.4
	Reversion tests	% ISO, 1/3 hr	83.8 / 82.8	87.2 / 86.3
	Kappa number		1.3	0.8
	Freeness	CSF, ml	129	114
	Pulp yield	%	91.0	91.3
	Initial / final pH		1.7 / 1.6	1.7 / 1.7
	O_3 consumption	% on od fiber	0.8 – 1.2	0.5 – 0.7
Bleached Pulp				
	Viscosity	cp	4.97	4.13
	Total bleaching yield	%	86.0	88.2

QPZ bleaching achieved the 85% ISO minimum target brightness for both whole flax and separated bast fiber, 86.3% ISO and 89.4% ISO respectively.

However, the bleached whole flax and separated bast fiber pulps had very low freeness, 129 and 114 ml CSF respectively, and low viscosity, 4.97 and 4.13 respectively.

These indicate poor quality pulp and likely poor papermaking capability.

Pulp Testing

Tables 6 provides selected testing results for the bleached whole straw and separated bast fiber pulps. Given the low initial freeness of both pulps, only tests were carried out for only 2 data points.

Table 7 Bleached pulp properties

Test	Units	Whole straw		Separated bast	
PFI revolutions	rev	0	250	0	250
Freeness	CSF, ml	129	41	114	30
Basis weight	g/m^2	64.4	65.0	65.0	62.8
Density	g/cm^3	0.505	1.654	0.410	0.503
Bulk	cm^3/g	1.98	1.53	2.44	1.99
Tear Index	$mN \cdot m^2/g$	3.95	4.36	7.23	6.65
Tensile Index	Nm/g	46.23	52.81	34.03	43.27
Burst Index	$kPa \cdot m^2/g$	2.36	3.04	1.63	2.35
Stretch	%	1.98	2.02	1.81	2.77
TEA	J/m2	40.20	48.68	28.68	55.67
Gurley porosity	sec/100 ml	40.6	2093.0	250.5	151.5
TAPPI opacity	%	81.0	77.8	81.4	80.9
Printing opacity	%	71.0	69.1	72.0	71.3
Kajaani Fiber Length Distribution					
Arithmetic average	mm	0.48		0.77	
Length-weighted average	mm	1.48		2.45	
Weight-weighted average	mm	3.63		4.54	
P-fraction	%	34.6		30.3	

Table 7 shows anticipated differences between the two pulps. For example,

- the separated bast fiber pulp had higher tear strength and lower tensile strength than the whole straw pulp, and
- the separated bast fiber pulp had a higher average fiber length.

These were anticipated because shive is a shorter fiber than bast and the separated bast fiber was virtually shive free.

However, it was questioned why both pulps had inferior properties in general, and as compared to the properties of wheat straw and oat straw pulps that were tested in the same project (reported at PEERS 2018).

Several possibilities were considered:

- The bales of flax straw had not been properly retted. During the retting process, not only is there a loosening of the bond between the bast and core fiber fractions, but bast fiber containing raw materials also undergo a significant chemical change resulting from microbiological action on the fiber. This chemical change alters the physical properties of the final pulp product.
- Processing in the Tornado pulper damaged the fibers.
- Procedures used at NCSU damaged the pulp.

Additional Investigations

Given the above unexpected poor pulp properties exhibited by the bleached Tornado-processed whole flax straw and the separated flax bast fiber, additional tests were carried out in an attempt to determine what was causing the poor results.

1. <u>Confirmation cooks on Tornado-processed separated bast fiber</u>

   Two cooks on Tornado-processed separated bast fiber were carried out using the same test conditions as previous cooks. The brown stock was beaten using a Valley beater and tested for tear strength. In all cases and at various freeness levels, tear index was in the range of 7.35 – 9.32 mN·m^2/g, which was unacceptably low for flax bast fiber.

2. <u>Control cook on SMC dry decorticated flax bast fiber</u>

   To establish a control point, several bales of SMC dry decorticated flax bast fiber were processed through the Tornado pulper. The processed material was cooked using conditions based on actual mill scale conditions for SMC raw material:

   - 16% NaOH and 0.1% AQ on OD fiber,
   - 5:1 liquor to fiber ratio,
   - maximum cooking temperature of 165 ^{0}C,
   - 60 minutes to temperature, and
   - 180 minutes at temperature.

   Pulp yield was 41% on od fiber. The unbleached pulp was beaten using a Valley beater and tested for tear strength with the results shown in Table 8.

Table 8 SMC dry decorticated bast unbleached pulp

CSF, ml	**Tear Index**, mN·m^2/g
230	21.57
147	18.73

The SMC control exhibited expected tear index results which indicated that the test procedure used by NCSU was acceptable.

It also indicated that there could be two possible problems with the Tornado-processed whole flax straw and hydraulically separated flax bast fiber:

- the Tornado had damaged the fiber, and/or
- the flax straw raw material had not been properly retted and was, in fact, “green’ raw material.

It was decided to investigate the possibility that the flax straw was “green”, and an enzymatic retting process was established for treating the material prior to pulping.

3. Cook 1 on enzymatically retted Tornado-processed, hydraulically separated flax bast fiber

Tornado-processed, hydraulically separated flax bast fiber raw material was treated with Superace, a commercially-available cellulose-specific enzyme, with an application of 0.1% enzyme on od fiber.

The enzyme treated material was cooked using the same conditions used for the SMC control described above.

Pulp yield was 45% versus 60-64% yields from earlier cooks on material that had not been treated with enzymes. The brown stock was beaten using a Valley beater and tested for tear strength with the results shown in Table 9.

Table 9 Superace treated Tornado-process, hydraulically separated bast fiber unbleached pulp

CSF, ml	**Tear Index**, mN·m^2/g
327	18.63
242	17.46
100	17.65

Table 9 shows that the Superace treated material exhibited similar tear factors to the SMI control (Table 8).

This finding indicates that the original flax straw was not properly retted.

4. Another cook done on enzymatically retted Tornado-processed, hydraulically separated flax bast fiber

Tornado-processed separated flax bast fiber raw material was treated with another commercially- available enzyme, Viscozyme (developed specifically for textile flax retting), using the same application rate and process used for Superace above.

The enzyme treated material was cooked using the same conditions used for the SMC control described above.

Pulp yield was 48.6%. The brown stock was beaten using a Valley beater and tested for tear strength with the results shown in Table 10.

Table 10 Viscozyme treated Tornado-process, hydraulically separated bast fiber unbleached pulp	
CSF, ml	**Tear Index**, mN·m^2/g
364	16.87
255	16.87

Table 9 shows that the Superace treated material exhibited similar tear factors to the SMI control (Table 8).

The test results using Viscozyme treated material, while not as good as the previous test with Superace, further support the contention that the original flax straw was not properly retted.

4. <u>Bleaching of pulp produced from enzymatically retted Tornado-processed, hydraulically separated flax bast fiber</u>

Given the brown stock test results with the two enzyme treatments, fully bleached pulp was produced from Tornado-processed separated flax bast fiber using a Superace treatment followed by cooking and QPZ bleaching.

Cooking conditions used were the same as for the SMC control cook above. Bleaching test conditions used were the same as in Table 4. Final pulp brightness was 88.5 %ISO.

The bleached pulp was beaten using a Valley beater and tested for tear strength with the shown in Table 11.

Table 11 Superace treated Tornado-process, hydraulically separated bast fiber QPZ bleached pulp	
CSF, ml	**Tear Index**, mN·m^2/g
269	8.43
187	7.72
98	8.61
58	9.12

The tear strength of the bleached pulp was lower than expected. Further investigations identified that ozone bleaching stage generally may have had a negative effect on bast fiber containing raw materials and that the high shear mixer used may have also been part of the problem.

5. <u>Modified bleaching of pulp produced from enzymatically retted Tornado-processed, hydraulically separated flax bast fiber</u>

Given the results in Table 11, the ozone bleaching stage was dropped. Fully bleached pulp was produced from Tornado-processed separated flax bast fiber using a Superace treatment followed by cooking and QP bleaching.

Cooking conditions used were the same as for the SMC control cook above. Bleaching test conditions used were the same as in Table 4. Final pulp brightness was 85.5 %ISO.

The bleached pulp was beaten using a Valley beater and tested for tear strength with the shown in Table 12.

Table 12 Superace treated Tornado-process, hydraulically separated bast fiber QP bleached pulp	
CSF, ml	**Tear Index**, mN·m^2/g
364	9.81
184	10.40
106	10.49

Tear strength of the QP bleached pulp was higher than for the QPZ pulp which indicated that the ozone stage did have a negative effect on pulp properties.

However, the tear strength of the QP bleached pulp was still much lower than that of the brown stock and of commercially available flax pulps.

This last test on bleached pulp indicated that there could be two possible problems areas which require further investigation:

- the Tornado pulper or equipment in the fiber separation system damaged the bast fiber in a manner that only became apparent on bleaching, and/or
- there was a procedural problem at NCSU during the chelation stage or peroxide bleaching stage which damaged the fiber.

Work on the Tornado-processed, hydraulically separated flax bast fiber was terminated at this point due to a lack of funding.

CONCLUSIONS

1. Proper retting of the whole flax prior to pulping is critical. During the retting process, not only is there a loosening of the bond between the bast and core fiber fractions, but bast fiber containing raw materials also undergo a significant chemical change resulting from microbiological action on the fiber. This chemical change alters the physical properties of the final pulp product.

2. The Tornado pulper can effectively size reduce whole flax into a pumpable slurry.

3. Bast fiber can be effectively hydraulically separated from pumpable slurry of whole flax material using hydrocyclones and the bast/shive fiber fractions can be adjusted as required.

4. A wet preparation system for whole flax straw based on the Tornado pulper could offer a lower fiber cost for pulping whole straw and separated bast than traditional dry decortication systems.

5. Additional research on the wet preparation system using properly retted whole flax straw is warranted.

REFERENCES

1. Saskatchewan Flax Development Commission, https://www.saskflax.com
2. Agriculture and Agri-Foods Canada, Agricultural Industry Market Information System (AIMIS), https://aimis-simia.agr.gc.ca/rp/index-eng.cfm?action=pR&r=243&lang=EN
3. Pulp & paper Manufacture, Vol. 3, Secondary Fibers and Non-Wood Pulping, 1987 - Page 7, Table 7
4. Flax Council of Canada, https://flaxcouncil.ca/growing-flax/chapters/flax-straw-and-fiber/
5. Flax Council of Canada, https://flaxcouncil.ca/wp-content/uploads/2015/02/FCOC-growers-guide-Chapter-12-Flax-Straw-and-Fibre.pdf
6. http://www.hurterconsult.com/2019/files/plant%20fiber%20characteristics.pdf
7. Tschirner, U and Satyavolu, J, Canadian Oil Seed Flax Fiber in Traditional Pulping, 1999 TAPPI Pulping Conference Proceedings

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October 27-30, 2019
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NC STATE UNIVERSITY

Agenda

- Introduction
- Experimental
- Results
- Additional Investigations
- Conclusions

Introduction

Textile flax

- Grown for linen (bast fiber)
- Taller plant - 90 to 120 cm
- Small amount of seed branches near top
- Skucthing tow and wastes used for pulp
- Very limited amount grown in North America

Seed Flax

- Grown for oilseed
- Shorter plant – 40 to 91 cm
- Large amount of seed branches
- Limited uses for flax straw

- Focus of this presentation

Flax straw availability

Harvested seed flax area (000's ha)				
Year	Alberta	Saskatchewan	Manitoba	Total
2009	16.2	489.7	107.2	613.1
2010	15.4	271.1	62.7	349.2
2011	27.5	213.3	50.6	291.7
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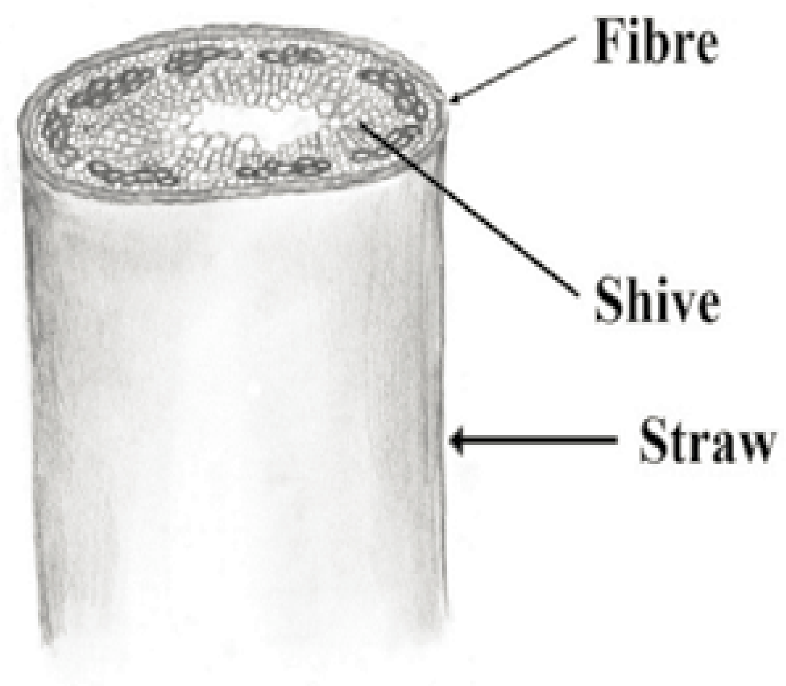
2016	29.1	279.2	33.2	341.5
2017	34.4	360.2	24.3	418.9
2018	37.1	287.5	14.8	339.4

- Rotational crop
- 291,700 ha to 628,900 ha
- 1.1 - 1.7 mt/ha collectable seed flax straw
- 320,000 - 1,070,000 mt/y straw

Anatomy of flax straw

- Dicotyledon, or dicot
  - Long bast fibers in bundles
  - 15 – 25% (10 – 30)
  - Short core fibers called shive

- Individual seed flax bast fibers:
  - Length: 10-45 mm, average 27 mm
  - Diameter: 16-30 microns, average 22 microns
  - Aspect ratio: 1250:1



- Bast fiber impedes plowing straw under
- Traditionally burned in fields
- Limited uses for flax straw
  - Fiber raw material for cigarette burning tube paper
  - Schweitzer-Mauduit Canada (SMC)

SMC Mobile Decortication


Retrieving flax bales from storage


Delivering bales to decorticator system


Feed conveyor with guillotine cutter


Modified tub grinder & baled decorticated fiber


Building new piles

- Flax straw sourced from specific fields
- Storage piles
  - approx. 600 mt
  - widely spaced for fire protection
- Natural retting for 12 – 18 months
- Decorticated fiber
  - approx. 60 – 65% bast


Shive & dust removal

- Traditional decortication is expensive.

- Purpose of this work was to determine if:
  - whole flax straw could be wet processed into a uniform and pumpable slurry suitable for whole straw pulping,
  - the whole flax straw slurry could be hydraulically separated into its bast and shive fractions, and
  - the wet processed materials would produce acceptable papermaking pulps.

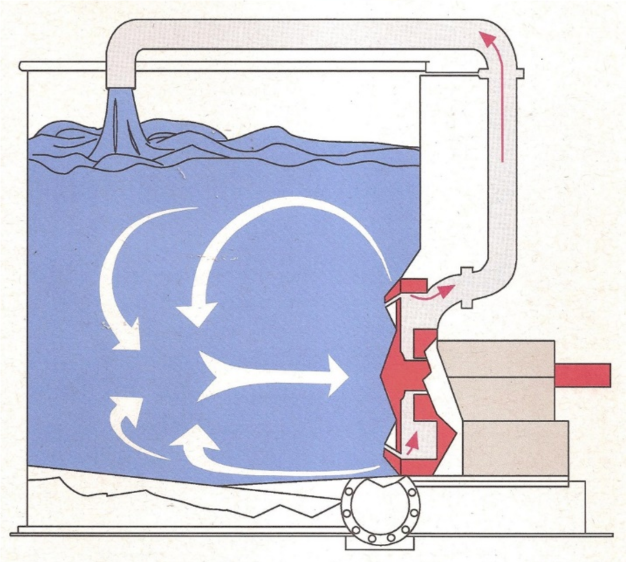
Experimental

Fiber Raw Material

- Seed flax straw
- Large round bales from Manitoba
- Approx. 500 kg each
- Provided by third party

Whole flax straw wet pretreatment

- Bolton Emerson Americas Tornado pulper
    - Rotor/stator with cutting elements
- 36” unit located at the Herty Foundation
    - 400 HP (298 kW) motor
- Pulper tank filled with warm water
- Large slabs of whole straw fed into pulper tank to 5% consistency.
- Operated in batch recirculation mode until straw reduced to a uniform and pumpable slurry



Whole flax straw hydraulic separation

Centri-cleaner based

- Initial separation
  - Whole flax straw slurry diluted to 1% consistency and processed through Posiflow
- Posiflow accepts
  - diluted to 0.4% consistency and processed through Uniflow
- Posiflow rejects
  - diluted to 0.75% consistency and processed through Posiflow 2nd time

Cooking

- Process: soda-AQ
- Target Kappa number: 10 - 15
- 3-liter rotating bombs, heated-air oven
- Whole straw: 3 cooks
- Separated bast: 4 cooks

Cooking

Soda-AQ cooking conditions			
	Range	Optimum	
		Whole straw	Separated bast
Sample size	350 od grams	350 od grams	350 od grams
NaOH on OD fiber	20%	20%	20%
AQ on OD fiber	0.1%	0.1%	0.1%
Liquor-to-fiber ratio	5:1	5:1 start	5:1 start
Maximum temp.	160 – 170 ^{0}C	168 ^{0}C	160 ^{0}C
Time to max temp.	60 min	60 min	60 min
Time at max temp.	120 min	120 min	120 min
Post cooking treatment	Mixed, washed and screened		

Bleaching

- Target brightness: >85% ISO
- Sequence: QPZ
- Q stage: open container in hot water bath
- P stage: 3-liter Teflon lined bombs
- Z stage: sparged ozone gas into mixing vortex of pulp slurry

Q stage conditions		
Consistency	%	4.0
Temperature	^{0}C	55
Time	minutes	35
pH		5
DTPA	% on od fiber	0.5

Bleaching

P stage conditions		
Consistency	%	12.0
Temperature	^0C	105
Time	minutes	90
H_2O_2	% on od fiber	5.0
NaOH	% on od fiber	4.0
DTMPA	% on od fiber	0.2
$MgSO_4$	% on od fiber	0.5
Na_2SiO_3	% on od fiber	0.5

Z stage conditions		
Consistency	%	3.0
Temperature	^0C	20
pH		1.6

Results

- Wet processing of whole flax straw
- Hydraulic separation of whole flax straw
- Cooking results
- Bleaching results
- Pulp properties
- All tests conducted according to TAPPI Test Methods

Results

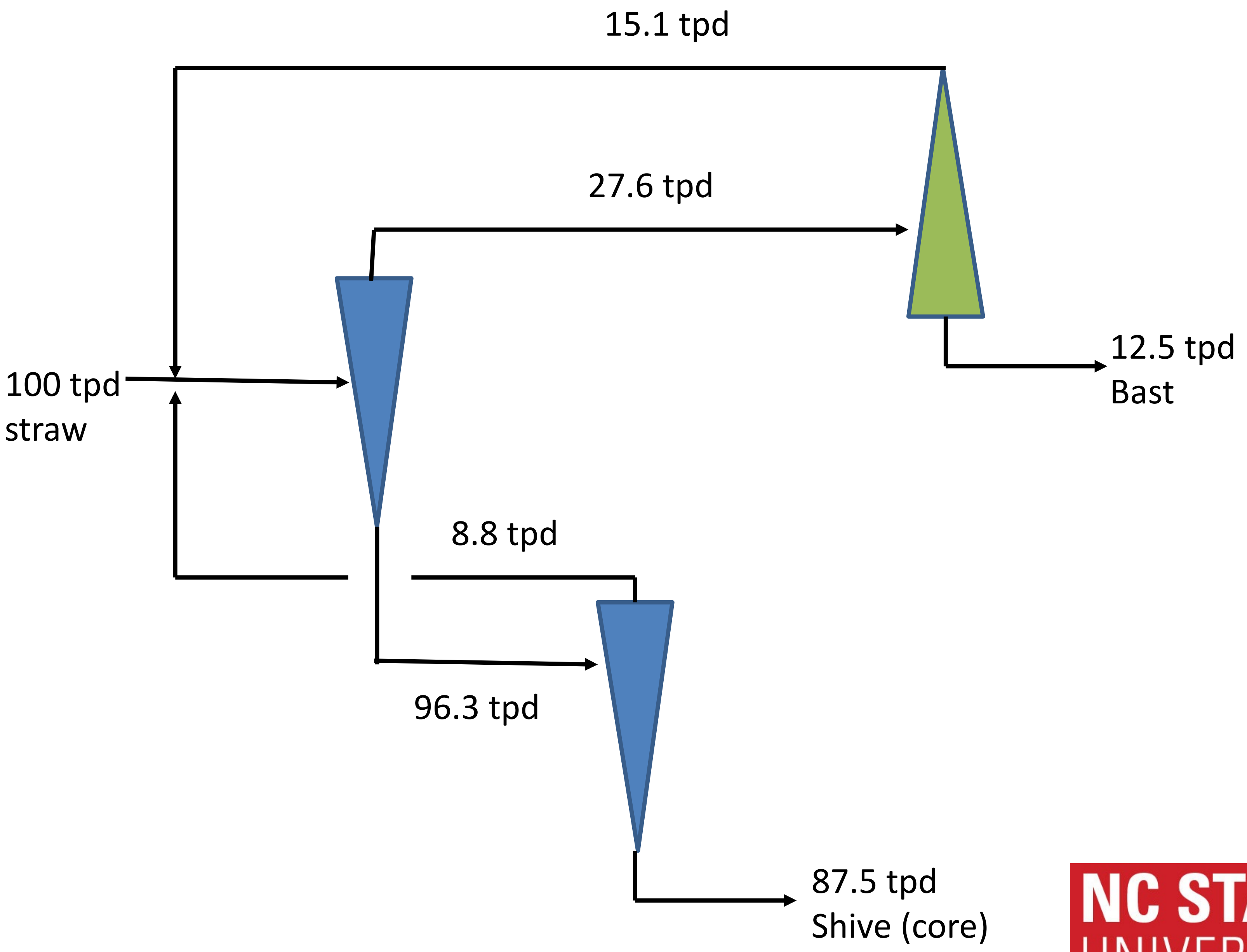
Wet processing of whole flax straw

- Operating 30 – 35 min in batch recirculation mode
- All straw cut to 6 - 12 mm
- Pumpable slurry
- In continuous operation mode, some of the flow will recirculate and some moved forward in the process - a Claflin refiner will be needed for size reduction

Results

Hydraulic separation

- Uniflow bast fraction was shive free
- 2nd Posiflow shive fraction had <5% bast visually
- Low bast recovery may be result of low bast in straw



Results

Cooking results			
		Whole straw	Separated bast
Kappa number		15.2	4.1
Brightness	% ISO	35.3	52.2
Screened yield	%	46.3	60.6
Screened pulp freeness	CSF, ml	350	390
Alkali consumption	%	85.0	62.6
Black liquor terminal pH		11.6	12.8

Results

Bleaching results – P stage			
		Whole straw	**Separated bast**
Brightness	% ISO	79.7	85.1
Reversion	% ISO, 1/3 hr		84.8 / 84.5
Kappa number		5.9	2.3
Freeness	ml CSF		
Pulp yield	%	94.5	96.6
Initial / final pH		na / 11.4	na / 12.5
H_2O_2 consumption	% on od fiber	99.9	94.6

Results

Bleaching results – Z stage			
		Whole straw	**Separated bast**
Brightness	% ISO	86.3	89.4
Reversion	% ISO, 1/3 hr	83.8 / 82.8	87.2 / 86.3
Kappa number		1.3	0.8
Freeness	ml CSF	129	114
Pulp yield	%	91.0	91.3
Initial / final pH		1.7 / 1.6	1.7 / 1.7
H_2O_2 consumption	% on od fiber	0.8 – 1.2	0.5 – 0.7
Bleached pulp			
Viscosity	cp	4.97	4.13
Total bleaching yield	%	86.0	88.2

Results

Selected bleached pulp properties					
	Units	Whole straw		Separated bast	
PFI revolutions		0	250	0	250
Freeness	CSF, ml	129	41	114	30
Tear Index	$mN \cdot m^2/g$	3.95	4.36	7.23	6.65
Tensile Index	Nm/g	46.23	52.81	34.03	43.27
Arithmetic average	mm	0.48		0.77	
Length-weighted average	mm	1.48		2.45	
Weight-weighted average	mm	3.63		4.54	
P-fraction	%	34.6		30.3	

Results

Summary

- Tornado pulping – worked
- Hydraulic separation – worked
- Pulp properties
  - Freeness – significantly lower than expected
  - Strength – significantly lower than expected
  - **WHY?**
    - Fiber damage in wet processing and hydraulic fiber separation
    - Fiber damage in NCSU procedures
    - Retting issues

Additional investigations

Retting

- Loosens bond between the bast and core fiber fractions.
- Microbiological action causes fiber to undergo significant chemical change.
- Chemical change alters physical properties of final pulp product.

Additional investigations

Confirmation cooks on wet-processed separated bast fiber

- 2 cooks using same cook conditions
- Brown stock beaten in Valley beater
- Tear index range: 7.35 – 9.32 mN·m^2/g
- Unacceptably low for flax bast fiber

Additional investigations

Control cook on SMC dry decorticated flax bast

- Bales processed in Tornado pulper
- Cooking conditions adjusted to:
  - 16% NaOH and 0.1% AQ on OD fiber
  - 5:1 liquor to fiber ratio
  - 165 ^{0}C max. cooking temperature
  - 60 minutes to temperature
  - 180 minutes at temperature
- Yield: 41% on od fiber

Unbleached pulp	
CSF, ml	Tear Index, mN·m^2/g
230	21.57
147	18.73

Additional investigations

Cook 1 on enzymatically retted wet-processed separated bast fiber

- Superace – 0.1% on od fiber
- Cooking conditions same as SMC
- Yield: 45% on od fiber versus 60-64% per earlier cooks

Unbleached pulp	
CSF, ml	Tear Index, mN·m^2/g
327	18.63
242	17.46
100	17.65

Additional investigations

Cook 2 on enzymatically retted Tornado-processed separated bast fiber

- Viscozyme – 0.1% on od fiber
- Cooking conditions same as SMC
- Yield: 48.6% on od fiber versus 60-64% per earlier cooks

Unbleached pulp	
CSF, ml	Tear Index, mN·m²/g
364	16.87
255	16.87

Additional investigations

Bleaching enzymatically retted wet-processed separated bast fiber

- Superace
- Cooking conditions same as SMC
- QPZ sequence
- Final brightness: 88.5% ISO
- High shear mixer in ozone stage

QPZ bleached pulp	
CSF, ml	Tear Index, $mN{\cdot}m^2/g$
269	8.43
187	7.72
98	8.61
58	9.12

Additional investigations

Bleaching enzymatically retted wet-processed separated bast fiber

- Superace
- Cooking conditions same as SMC
- QP sequence
- Final brightness: 85.5% ISO
- Future testing – perhaps drop Q stage

QP bleached pulp	
CSF, ml	Tear Index, mN·m^2/g
364	9.81
184	10.40
106	10.49

Conclusions

1. Proper retting of flax straw prior to pulping is critical.

2. Wet preparation using a Tornado pulper effectively size reduces flax straw into a pumpable slurry.

3. Bast fiber can be hydraulically separated from a pumpable slurry of flax straw using hydrocyclones, and the bast/shive fractions can be adjusted as required.

4. A wet preparation system could offer a lower fiber cost for pulping whole straw and separated bast than traditional dry decortication systems.

5. Additional research on the wet preparation system using properly retted whole flax straw is warranted.

Questions?

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