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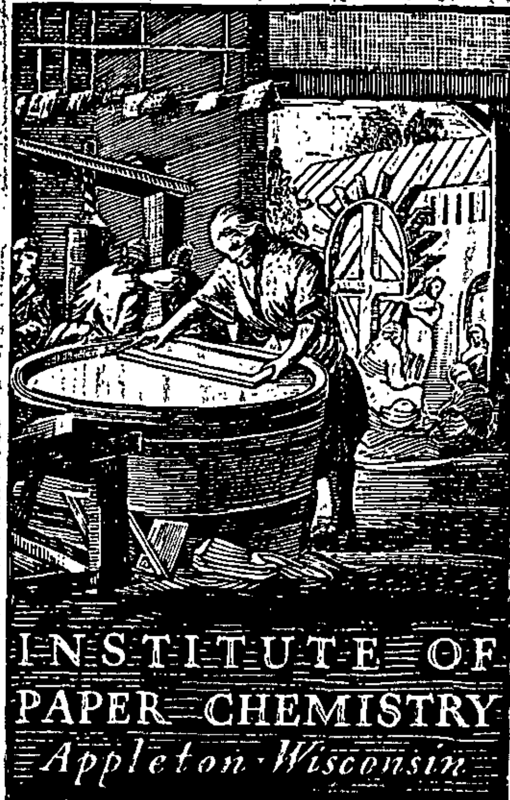

SELECTIVE DELIGNIFICATION OF WOOD AND OTHER FIBROUS MATERIALS:

CHLORINE DIOXIDE-ALKALI PULPING OF LOBLOLLY AND BLACK SPRUCE

Research Grant

Project 2500

Report Thirteen
A Progress Report
to
THE GRANTORS

September 7, 1971

THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

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SUMMARY

Progress is reported on investigations into producing chlorine dioxide-alkali pulps from softwood, particularly unbleached semichemical pulps from loblolly pine and black spruce. The process steps include chip fiberization with or without alkali pretreatment and delignification by chlorine dioxide and alkali extraction followed by primary refining.

In chip fiberization, which included water-impregnated chips, either an Asplund or Bauer pressurized refiner system was used. Assessment of fiberization conditions was by consideration of Bauer-McNett classifications and chlorite-alkali pulps. Fiber length measurements, microscopic examination, and handsheet properties are presented. These are discussed in reaching the conclusion that at higher temperatures, available in a pressurized refiner system compared with nonpressurized equipment, there is significantly less fiber damage including no apparent reduction in fiber length distribution. A further conclusion is that chlorite-alkali pulps from chips fiberized at 133 and 158°C. beat faster for the case of the lower temperature. Power consumption for chip fiberization was less than 5 h.p.-days for a ton of chips.

Delignification was based on the consumption of 6 and 9% chlorine dioxide with and without pretreatment with sodium hydroxide at 70 and 90°C. After alkali extraction, the yield range was 78-90%. Klason lignin analyses showed alkali pretreatment resulted in greater lignin removal and that delignification was selective. Less lignin was removed for a given amount of chlorine dioxide than found before for

hardwoods, presumably because softwood lignin is present in greater quantity and differs in chemical structure.

Semichemical pulps obtained by primary refining in a Sprout-Waldron after chlorine dioxide-alkali delignification have been beaten in a PFI mill and handsheet properties determined. A 59% high-yield kraft pulp that had been refined similarly was used as a basis for comparison. The 78-90%-yield chlorine dioxide-alkali pulps beat at a rate about comparable to the reference pulp. Handsheet densities at similar levels of beating were above as well as below that of the reference pulp. Other handsheet tests included sheet smoothness, breaking length, stretch, burst factor, tear factor, and ring crush. Black spruce pulps obtained at 80-85% yield using 9-6% chlorine dioxide gave the best strength results for the conditions investigated. At 85% yield for comparable low sheet density, handsheet drainage time was essentially the same, smoothness greater, breaking length greater, stretch about 10% lower, burst factor slightly lower, ring crush very significantly higher, and tear factor considerably lower than for the reference pulp. Consideration of the results for loblolly pulps led to the conclusion that it might be possible to achieve somewhat comparable results at about 80% yield through further research.

Approximate costs for 80%-yield chlorine dioxide-alkali pulps have been estimated using a $20-$40/cord range for wood costs and assuming about 6-9% chlorine dioxide consumption. These costs have been compared with estimates for a 55% high-yield kraft pulp. It has been concluded from comparison of these costs that consideration of chlorine dioxide-alkali softwood pulps as an alternative to high-yield kraft pulps for linerboard base sheet is a realistic possibility, especially where wood costs are high.

Consideration is given to possible future work.

INTRODUCTION

In an earlier report, initial studies on chip fiberization of loblolly pine were described (1). Using a chlorite-alkali pulp from small chips as a reference material, it was found that fiberization resulted in significant fiber damage with lowering of handsheet strength properties and a reduction in fiber length. In addition, microscopic studies revealed appreciable fibrillation of the fiber walls and other morphological changes had occurred during fiberization. These initial studies were interpreted to mean that the loblolly chips were not in a desirable viscoelastic state at the time of fiberization.

This report describes subsequent investigations on loblolly pine and black spruce. As a first objective it was established that loblolly chip fiberization could be carried out with fewer of the changes observed previously by the use of higher fiberization temperatures. In pursuing a further objective of producing desirable chlorine dioxide-alkali pulps from softwoods, the investigations have been focused on the possibility of preparing pulp which might be suitable for linerboard.

DISCUSSION AND RESULTS

HIGHER FIBERIZATION TEMPERATURES

In a previous discussion, consideration was given to factors to be taken into account in chip fiberization (2). These factors included the influence of higher temperature in achieving a more plasticized state during fiberization. The following work is primarily concerned with the prospect of obtaining better results on softwoods when higher fiberization temperatures are used.

Loblolly Preparation

Loblolly pine chips were fiberized in a 20-in. Asplund Defibrator equipped with a steam preheating vessel illustrated in a previous report (2). Details of the conditions employed are set out in Tables I and II along with Bauer-McNett classification data that grossly characterize the products. Fiberization temperatures were in the range 133-162°C. compared with about 100°C. previously (1).

A noticeable increase in the amount of the coarsest fraction of material in AB-8 through AB-11 (Table I) reflects a trend increase in the setting for the disk gap during fiberization. The Bauer-McNett classification of products made with a 0.014-0.016-in. disk gap were about comparable with the classification of the product SP-3 made with a disk gap of 0.025 in. when using the IPC Bauer machine at an earlier date. Such a difference in disk gap settings for about comparable classifications was also observed for aspen and red maple (2).

Maintaining the preheating temperature during chip fiberization (Table II) instead of relieving the pressure (Table I) led to no obvious difference in the Bauer-McNett classification data. However, a difference in the quality of the on-6-mesh materials was observed. This is illustrated in Fig. 1 from which it can be seen that the material SP-3 made in the IPC Bauer at probably about 100°C. had

TABLE I

LOBLOLLY FIBERIZATION AT 28 p.s.i.

Code	SP-3[a]	AB-8	AB-9	AB-10	AB-11
Chip impregnation	([b])	([c])		([d])	
Initial moisture content, % wet basis	53	48-51			
Final moisture content, % wet basis	63	65		68	
Preheating and fiberization	([e])	Asplund OVP Defibrator (batch-fed preheater and continuously fed defibrator)			
Preheater steam pressure, p.s.i./°C.	80/163	71/158			
Time to pressure, sec.	--	45			
Time in preheater, min.	3.0	3.0			
Defibrator steam pressure, p.s.i./°C.	--	28/133			
Feed period, min.	--	0-2.5	2.5-5.0	0-2.5	2.5-5.0
Disk gap, 0.001 in.	25	8	14	16	20
Bauer-McNett classification[f], % o.d.f.c.					
On 6 mesh	12.8	2.1	17.2	14.2	37.5
On 12 mesh	21.6	16.7	25.0	27.2	20.2
On 35 mesh	41.2	51.6	36.5	37.5	25.7
On 65 mesh	8.2	15.3	8.0	7.3	4.4
Through 65 (by difference)	16.2	14.3	12.9	13.8	12.2

[a] Fiberization carried out in the IPC no. 185 Bauer, see Progress Report Ten.

[b] Steamed twice at 15 p.s.i. for 2 min., then cold-water impregnated at 100 p.s.i. for 30 min.

[c] Steamed twice at 14 p.s.i. for 2 min., then cold-water impregnated at 78-86 p.s.i. for 30 min.

[d] Steamed twice at 14 p.s.i. for 2 min., then cold-water impregnated at 78-86 p.s.i. for 60 min. followed by further soaking at 14 p.s.i. for 16 hr.

[e] No. 185 Bauer with open feed inlet.

[f] Water temperatures: 21-22°C. Hot-water solubility (TAPPI Standard Method T 207 m-54): AB-8, AB-11, AB-14; 3.1, 3.2, and 3.6%, respectively.

TABLE II

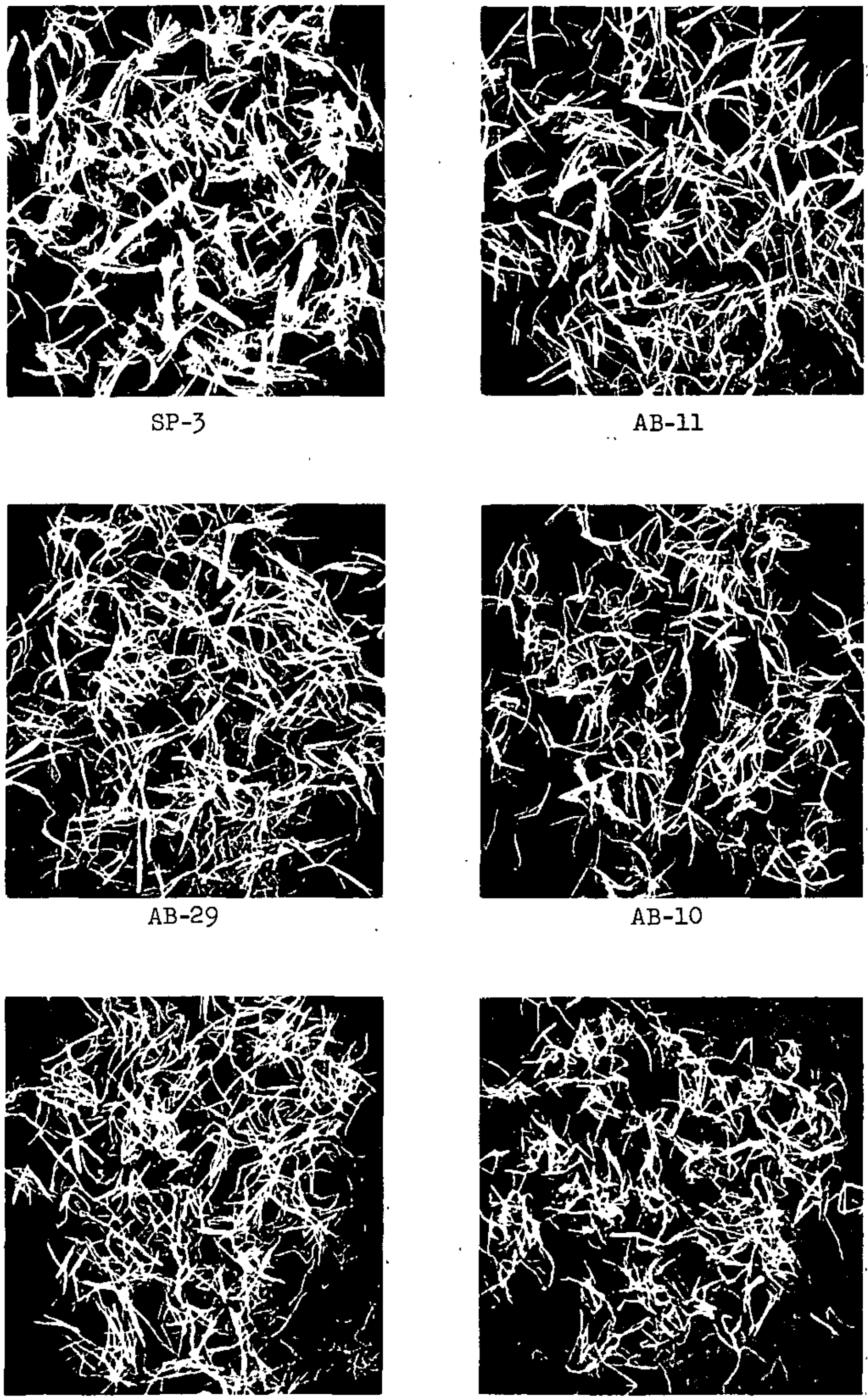
LOBLOLLY FIBERIZATION AT 71 AND 78 p.s.i.

Code	AB-29	AB-30	AB-12	AB-14
Chip impregnation	Steamed twice at 14 p.s.i. for 2 min., then cold-water impregnated at 78-86 p.s.i.			
Initial moisture content, % wet basis	48-51	48-51	48-51	48-51
Final moisture content, % wet basis	63[a]	63[a]	67[b]	67[b]
Preheating and fiberization	Asplund OVP Defibrator (batch-fed preheater and continuously fed defibrator)			
Steam pressure, p.s.i./°C.	71/158	71/158	78/162	78/162
Time to pressure, sec.	45	45	50	50
Time at pressure, min.	1.5-3.0	3.0-4.0	2.0-3.5	4.8-6.1
Time to feed, min.	1.5	3.0	2.0	4.8
Feed time, min.	1.5	1.0	1.5	1.3
Disk gap, 0.001 in.	16	16	12	12
Bauer-McNett classification[c], % o.d.f.c.				
On 6 mesh	24.4	25.9	21.9	19.3
On 12 mesh	24.2	25.4	23.7	22.6
On 35 mesh	32.3	34.3	34.6	33.8
On 65 mesh	5.8	6.3	6.3	5.3
Through 65 (by difference)	13.3	8.1	13.5	19.0

[a] Impregnated for 30 min.

[b] Impregnated for 60 min.

[c] Water temperatures, 21-22°C.



SP-3 AB-11

AB-29 AB-10

AB-14 AB-8

Figure 1. On-6-Mesh Bauer-McNett Fractions of Fiberized Loblolly, Magnification 1X

significantly coarser bundles than found when fiberization was at 133°C. or above. Fewer of the coarser bundles would be expected to be a positive factor in seeking uniform lignin modification with minimum screen rejects.

Assessment of Fiberization Conditions

To assess whether the fiberized conditions given in Tables I and II covered a range resulting in less fiber damage than found in the initial studies, which involved SP-1 and SP-3, chlorite-alkali pulps were prepared as included in Table III. This practice follows from previous work on aspen, covered in Report Six (3), in which an indication of the absence of fiber damage was obtained by comparing chlorite-alkali-delignified pulps prepared from both fiberized and "pin" chips.

The average fiber length and Bauer-McNett classification data for the pulps identified in Table III are given in Table IV. In addition, fiber length distributions of these pulps and photomicrographs of on-65-mesh fractions of fiberized chips are shown in Fig. 2 and 3. When these determinations of fiber length were made, broken as well as whole fibers were measured.

It is evident from Table IV that the average fiber lengths vary appreciably for the range of fiberization conditions covered, with the highest value (Pulp AB-14C) equal to that obtained for pulp from small chips or so-called "pin" chips. Significantly, the amount of through-100-mesh material was greatest for the lowest average fiber length value.

The fiber length histograms in Fig. 2 and 3 provide a picture somewhat similar to the data in Table IV. Pulps AB-11C and AB-14C are of interest since they apparently underwent no significant fiber shortening as a result of the fiberization step. On the other hand, Pulp AB-29/30C had an appreciable increase in the

TABLE III

CHLORITE DELIGNIFICATION[a] OF LOBLOLLY FIBERIZED CHIPS

Code	SP-1[b]	SP-3[b]	AB-8	AB-10	AB-11	AB-29/30[c]	AB-14
Alkali conditioning[d], yield %	--	96.6	93.1	97.1	95.4	96.0	93.6
Chlorite oxidation[e], yield %	72.3[f]	73.5	67.9	73.1	69.8	74.6	67.5
Alkali extraction[g], yield %	65.3	63.5	60.4	64.9	61.1	64.0	61.2
Brightness GE (ethanol)	56.7	54.0	--	60.5	56.8	--	56.9
Chlorite-alkali pulp code	SP-1	SP-3	AB-8C	AB-10C	AB-11C	AB-29/30C	AB-14C

[a] All reactions carried out at 8-10% consistency.

[b] From Progress Report Ten, SP-1 chlorite-alkali pulp from pin chips, SP-3 chlorite-alkali pulp from IPC Bauer-fiberized chips.

[c] Fiberized Chips AB-29 and AB-30 combined in the weight ratio of 3:2; starting material 1.4 kg. oven dry.

[d] 10% NaOH for 60 min. at 50°C. (45 min. from ambient).

[e] 100% $NaClO_2$ for 25 hr., temp. at 60°C. at 4 hr. from ambient, initial pH 4.4, final pH 4.1.

[f] 100% $NaClO_2$ for 72 hr.

[g] 5% NaOH for 60 min. at 25°C.

TABLE IV

AVERAGE FIBER LENGTH AND BAUER-McNETT CLASSIFICATION DATA
OF LOBLOLLY CHLORITE-ALKALI PULPS

Pulp code	SP-1[a]	SP-3[b]	AB-8C	AB-10C	AB-11C	AB-29/30C	AB-14C
Average fiber length, mm.	1.78	1.41	1.31	1.60	1.69	1.63	1.82
On 12 mesh, %	43.0	34.2	29.2	--	41.0	--	43.3
On 35 mesh, %	19.0	17.2	13.9	--	13.8	--	12.6
On 65 mesh, %	33.6	36.2	40.2	--	34.4	--	32.7
On 100 mesh, %	1.6	2.6	2.9	--	2.0	--	2.2
Through 100 mesh, %, by difference	2.8	9.8	13.8	--	8.8	--	9.8

[a] Pin chip pulp, Report Ten.

[b] IPC Bauer pulp, Report Ten.

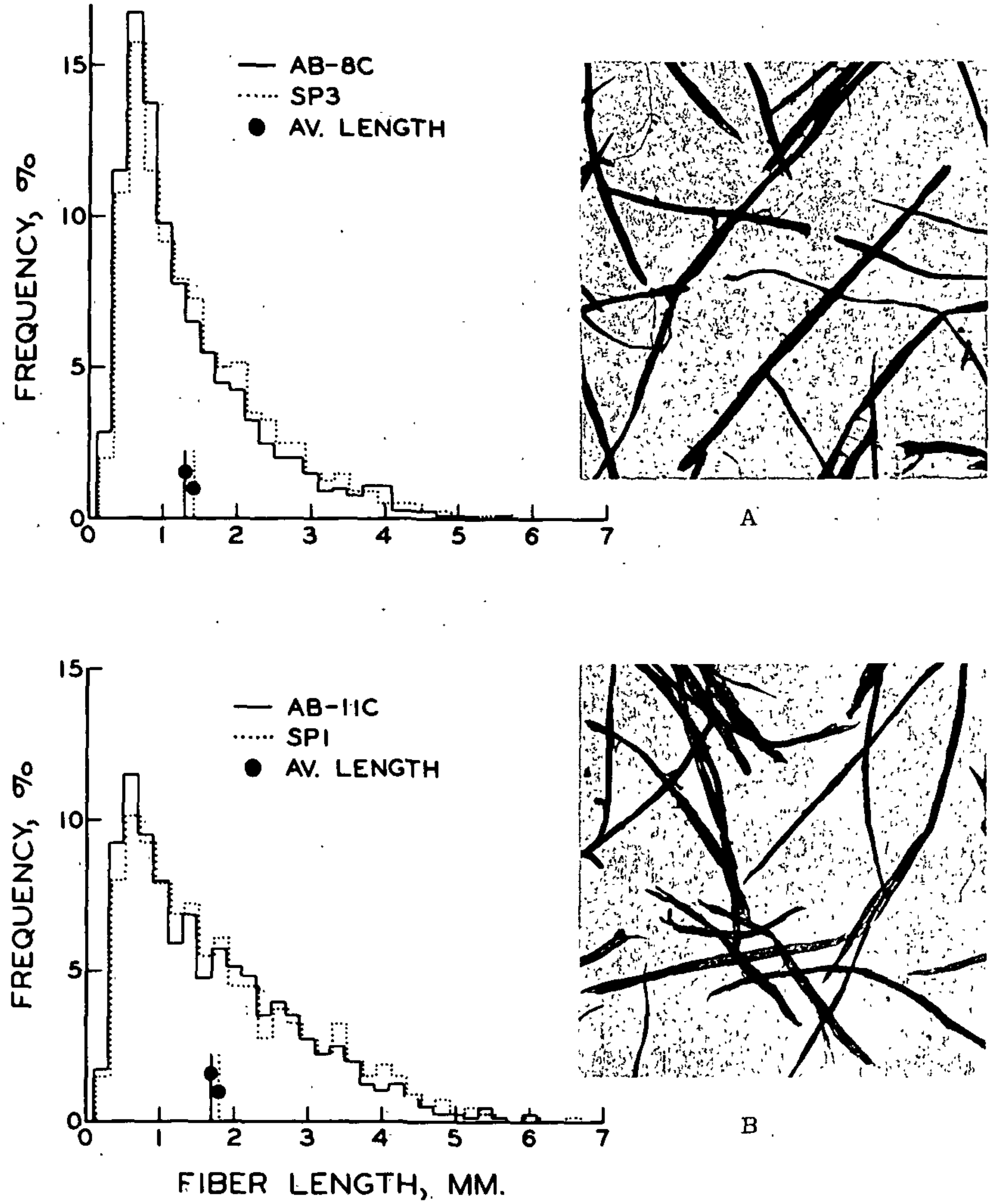


Figure 2. Fiber Length Distribution of Loblolly Chlorite-Alkali Pulps and Corresponding On-65-Mesh Bauer-McNett Fractions of Fiberized Chips.
A: AB-8, B: AB-11; Both 35x Magnification

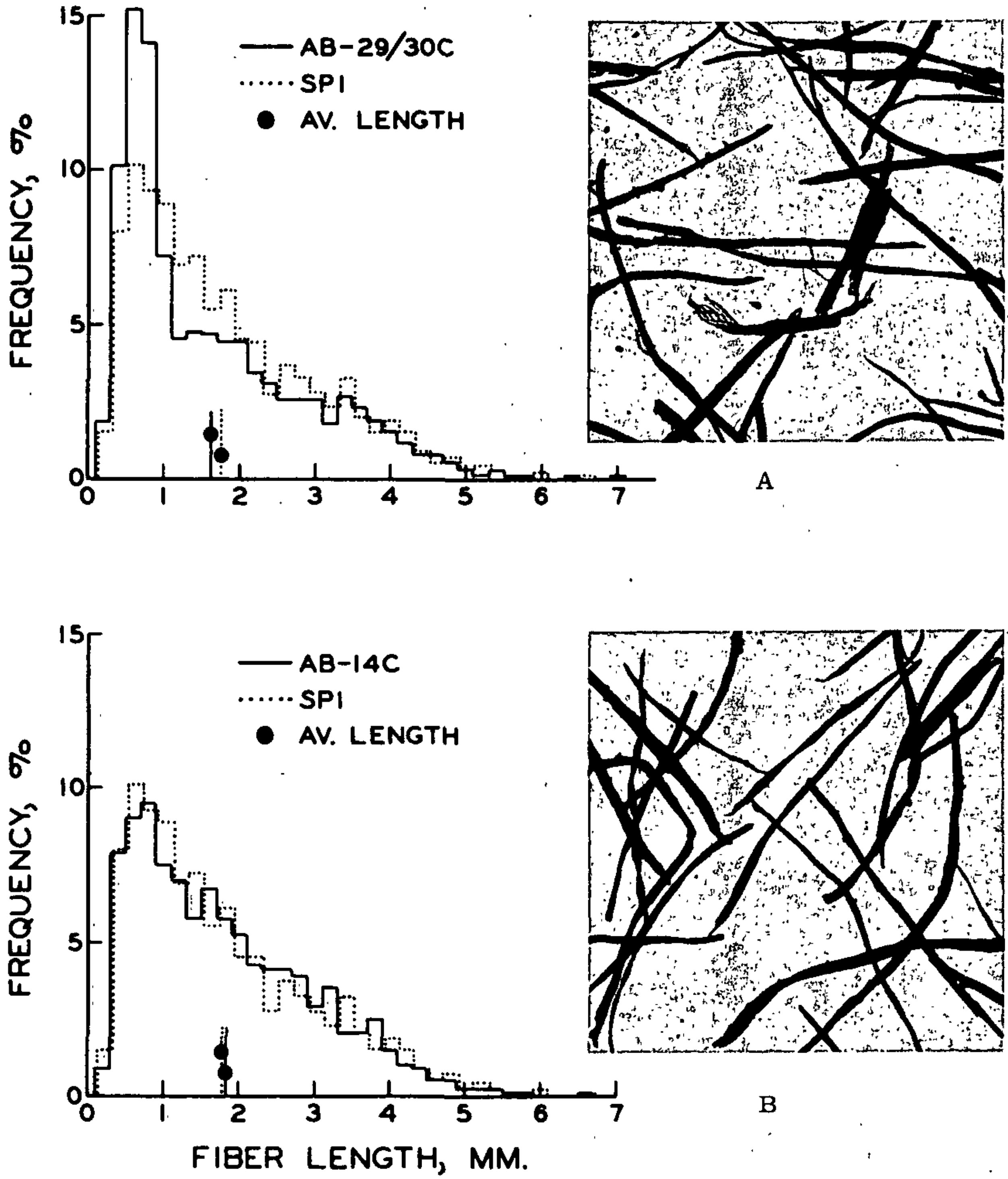


Figure 3. Fiber Length Distribution of Loblolly Chlorite-Alkali Pulps and Corresponding On-65-Mesh Bauer-McNett Fractions of Fiberized Chips. A: AB-29, B: AB-14; Both 35x Magnification

about-1-mm. fiber length fraction, as did Pulps SP-3 and AB-8C when compared with SP-1. The increase in the amount of about-1-mm. material for Pulp AB-29/30C compared with the case of Pulp AB-14C (Fig. 3) presumably arises from inclusion of the material heated for a shorter time before fiberization (AB-29).

A particular point regarding the fiber length data is that at fiberization temperatures higher than that used before for SP-3 (1) fewer across-fiber breaks were encountered.

Examination of photomicrographs of on- and through-65-mesh Bauer-McNett fractions of the fiberized chips coded AB-8, AB-10, AB-29/30, AB-12, and AB-14 revealed significant differences. These are illustrated by example in Fig. 2 and 3. Fractions from AB-8 were the most fibrillated. There was significantly less fibrillation in fractions from AB-10 obtained by fiberization after heating for a longer time. Fractions from AB-14 showed the least fibrillation. For this case, the fiberization temperature was 162°C. compared with 133°C. for AB-10. Less fibrillation is thus associated with the higher temperature. Fractions from AB-12 showed slightly more fibrillation than those from AB-14 providing an indication that the shorter heating time was probably not quite sufficient to bring the chips to temperature. The observations on fibrillation tend to correlate with fiber length data.

Since the fiberization conditions given in Tables I and II cover a range within which fiber damage varies to the point where little fibrillation is evident and there is no apparent reduction in fiber length, significant differences might be expected in handsheet properties. Handsheet data based on Valley beater evaluations of the chlorite-alkali pulps are given in Table V.

TABLE V

PRIMARY HANDSHEET DATA FOR LOBLOLLY CHLORITE PULPS MADE FROM FIBERIZED CHIPS

Pulp	AB-80					AB-100					AB-110				
Beating time, min.	2	4	6	8	12	2	4	6	8	10	2	5	7	9	11
Handsheet drainage time, sec.	--	--	--	--	--	4.5	4.7	5.1	6.0	7.4	--	--	--	--	--
Canadian freeness, ml.	635	560	500	420	285	620	525	395	250	185	665	575	460	355	270
Density, g./cc.	0.571	0.589	0.591	0.608	0.641	0.528	0.545	0.588	0.599	0.615	0.519	0.542	0.570	0.580	0.587
Specific scattering coefficient, $cm.^2/g.$	171	160	159	152	137	172	158	139	137	123	169	159	153	145	137
Specific absorption coefficient, $cm.^2/g.$	10.3	9.9	10.7	10.2	11.2	5.79	5.38	4.94	5.46	5.22	10.4	10.2	10.2	10.2	9.9
Breaking length, km	6.99	7.67	8.38	8.75	9.76	6.98	7.26	7.99	8.99	9.61	7.11	7.70	7.90	8.20	9.32
Stretch, %	2.4	2.5	2.4	2.4	2.6	2.2	2.5	2.5	2.5	2.4	2.0	2.1	2.1	2.2	2.4
Tensile energy absorp., $g.cm./cm.^2$	59.5	82.0	82.0	85.8	107	62.7	72.5	80.8	90.8	94.3	56.7	67.2	67.0	74.3	91.7
Tensile stiffness, Et, kg /cm.	463	502	531	554	570	450	445	487	530	557	484	514	515	542	565
Burst factor	42.7	47.7	48.7	53.0	58.9	43.7	51.8	53.4	57.0	61.5	44.8	52.6	57.6	60.1	59.1
Tear factor (Elmendorf)	108	102	97.6	89.3	84.7	125	115	105	94.5	88.4	134	119	114	108	104
Tear factor × yield/65	100	95.0	90.7	83.0	78.7	125	113	105	94.3	88.3	126	112	107	102	98
In-plane tear, g.cm./cm.	95.4	93.6	94.2	93.4	86.7	104	104	102	102	96.5	104	100	105	104	109
Zero-span breaking length, km. × yield/65	14.8	14.7	15.1	15.0	16.2	15.7	16.1	17.4	16.7	17.0	14.7	15.2	16.2	15.4	15.8
M.I.T. fold	209	254	281	325	412	239	260	362	398	437	213	309	322	344	366
Bendtsen smoothness, ml./min.	1800	1750	1710	1640	1820	1900	1710	1860	1870	2130	2110	2090	2230	1910	2010

Pulp	AB-29/30					AB-140				
Beating time, min.	3	6	12	16	20	2	4	7	10	15
Handsheet drainage time, sec.	4.2	4.6	4.9	5.3	7.2	--	--	--	--	--
Canadian freeness, ml.	725	680	510	370	230	695	575	610	510	340
Density, g./cc.	0.467	0.489	0.537	0.566	0.592	0.510	0.523	0.542	0.562	0.595
Specific scattering coefficient, $cm.^2/g.$	167	161	147	138	135	179	175	170	159	146
Specific absorption coefficient, $cm.^2/g.$	13.1	12.2	12.1	13.6	12.3	8.60	8.19	8.20	8.05	9.15
Breaking length, km	6.04	6.84	7.97	8.82	9.10	6.36	7.59	7.49	8.23	8.96
Stretch, %	1.9	2.0	2.3	2.3	2.3	2.0	2.2	2.2	2.3	2.4
Tensile energy absorp., $g.cm./cm.^2$	46.4	59.6	74.1	84.1	83.6	62.8	67.4	66.3	77.0	86.3
Tensile stiffness, Et, kg /cm.	430	463	514	549	583	456	486	497	530	551
Burst factor	35.5	39.7	46.3	54.4	56.9	39.1	42.5	45.9	52.9	58.1
Tear factor (Elmendorf)	144	137	106	108	91.0	144	138	123	121	107
Tear factor × yield/65	142	135	104	106	89.8	136	130	116	114	101
In-plane tear, g.cm./cm.	103	97.2	103	100	98.6	109	112	115	109	99.8
Zero-span breaking length, km. × yield/65	13.2	13.4	14.4	14.6	13.9	13.1	14.4	14.9	14.3	14.6
M.I.T. fold	122	141	304	389	420	177	215	227	272	352
Bendtsen smoothness, ml./min.	2390	2350	2330	1910	2180	1850	2020	1840	1830	1860

An interesting aspect of the results in Table V is the lower density range of handsheets from Pulps AB-11C and AB-14C, for example, compared with Pulp AB-8C. The latter pulp was the most damaged and the former were apparently the least damaged during fiberization. This seems to be a factor influencing sheet density. Corroboration of the greater amount of fiber damage to Pulp AB-8C is provided by comparatively lower values for breaking length, burst factor, and tear factor at similar handsheet densities.

To illustrate that it is possible to fiberize loblolly chips with improvement in handsheet strength properties, comparisons have been made as in Fig. 4. In this figure, Pulp SP-1 is from "pin" chips and represents a reference material. Pulp SP-3 is from material found to be fiberized with significant fiber damage, as described in a previous report (1). The improved results for Pulp AB-11C, for example, are in accord with observations on fiber morphology (Table IV and Fig. 2).

Prior to undertaking this work on fiberization of softwoods at higher temperatures no particular basis was available to provide a precise guide on what targets to choose for the various parameters such as chip moisture content, plate setting, fiberization temperature, and time at temperature. In practice, the conditions set out in Tables I and II had to be decided upon on a somewhat arbitrary basis, which puts them in an exploratory category. This has meant that systematic optimization of the fiberization parameters has been left for the future.

The outcome of this assessment of using fiberization temperatures of 133-162°C. for loblolly is that less fiber damage is attainable than was found previously when using a fiberization temperature of about 100°C. A relative increase in handsheet strength properties and an increase in average fiber length are reflections of less fiber damage.

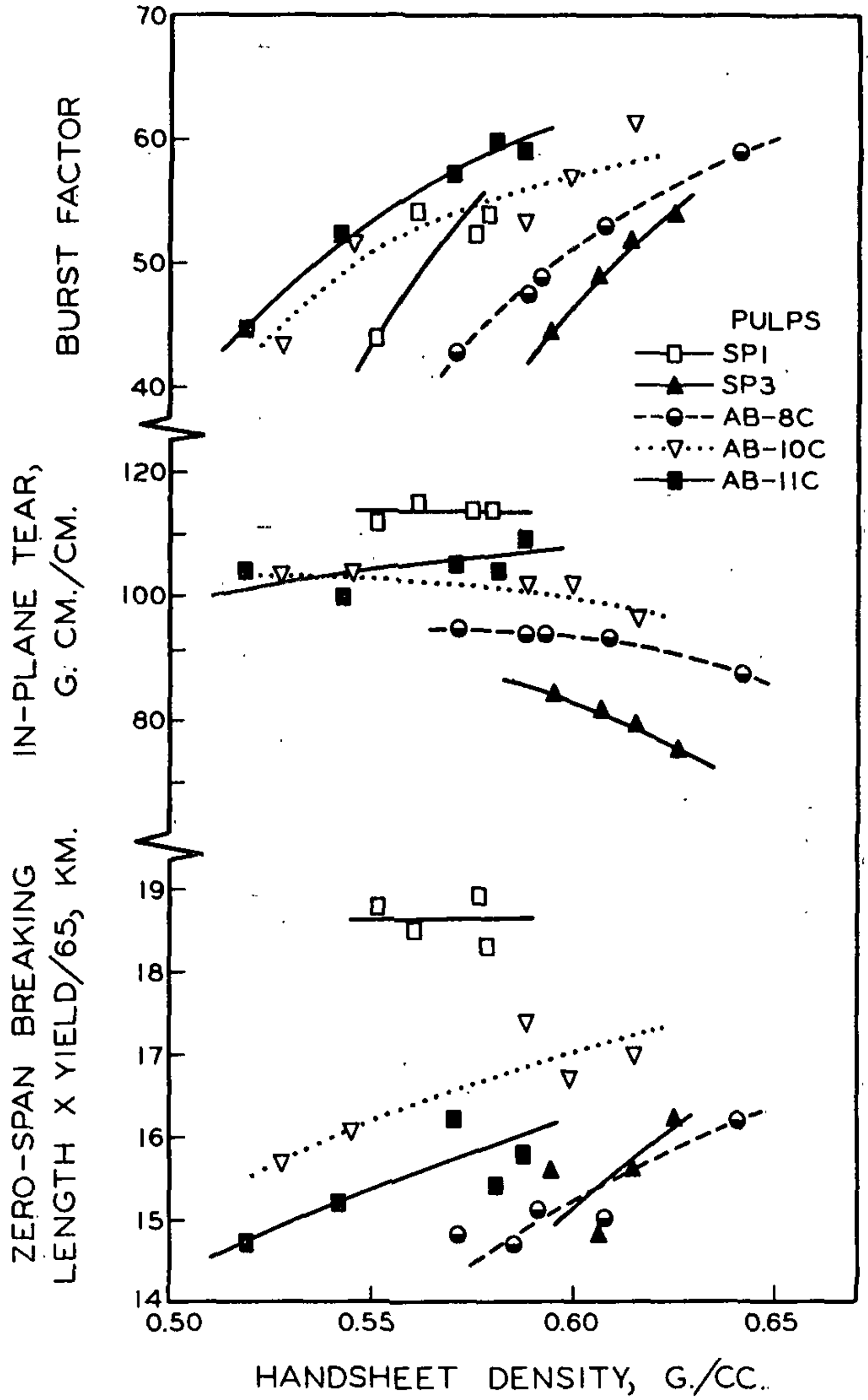


Figure 4. Loblolly Chlorite-Alkali Pulps — Burst Factor, In-Plane Tear, and Zero-Span Breaking Length x Yield/65 vs. Handsheet Density

CHLORINE DIOXIDE-ALKALI INVESTIGATIONS

Chip Fiberization in Bauer Pressurized Refiner System

For this part of the investigations on softwoods, loblolly and black spruce chips were fiberized in a Bauer pressurized refiner system illustrated in Fig. 5. The conditions used are set out in Table VI, which also includes Bauer-McNett classifications of the products. Further details are given in the experimental section. Use of a pressurized refiner system follows from the finding that a fiberization temperature of 133-162°C. was preferable to about 100°C.

For both species, about half of the chips had been impregnated with water and the Bauer-McNett classifications show that for black spruce a significantly coarser product was obtained when impregnation was omitted.

The selection of steam pressures was based partly on the range covered in earlier work on loblolly and black spruce (Appendix I) and partly by on-the-spot assessment of the product. For loblolly, there was an observable decrease in coarseness as the steam pressure was increased from 30 to 50 to 70 p.s.i., but with the black spruce an increase in steam pressure caused a less noticeable difference. These observations are similar to the trends seen in the Bauer-McNett classification data.

With one exception, time at pressure and plate gap were not varied. The exception was time at pressure for one lot of loblolly chips. These were held at the higher pressure for about three times as long in one case. This caused some change in the Bauer-McNett classification data and it is likely the longer time at pressure resulted in better between-fiber separation; however, this has not been pursued further. The plate gap was much greater than that used in earlier work on the Defibrator machine, which is to be expected on the basis of other experience.

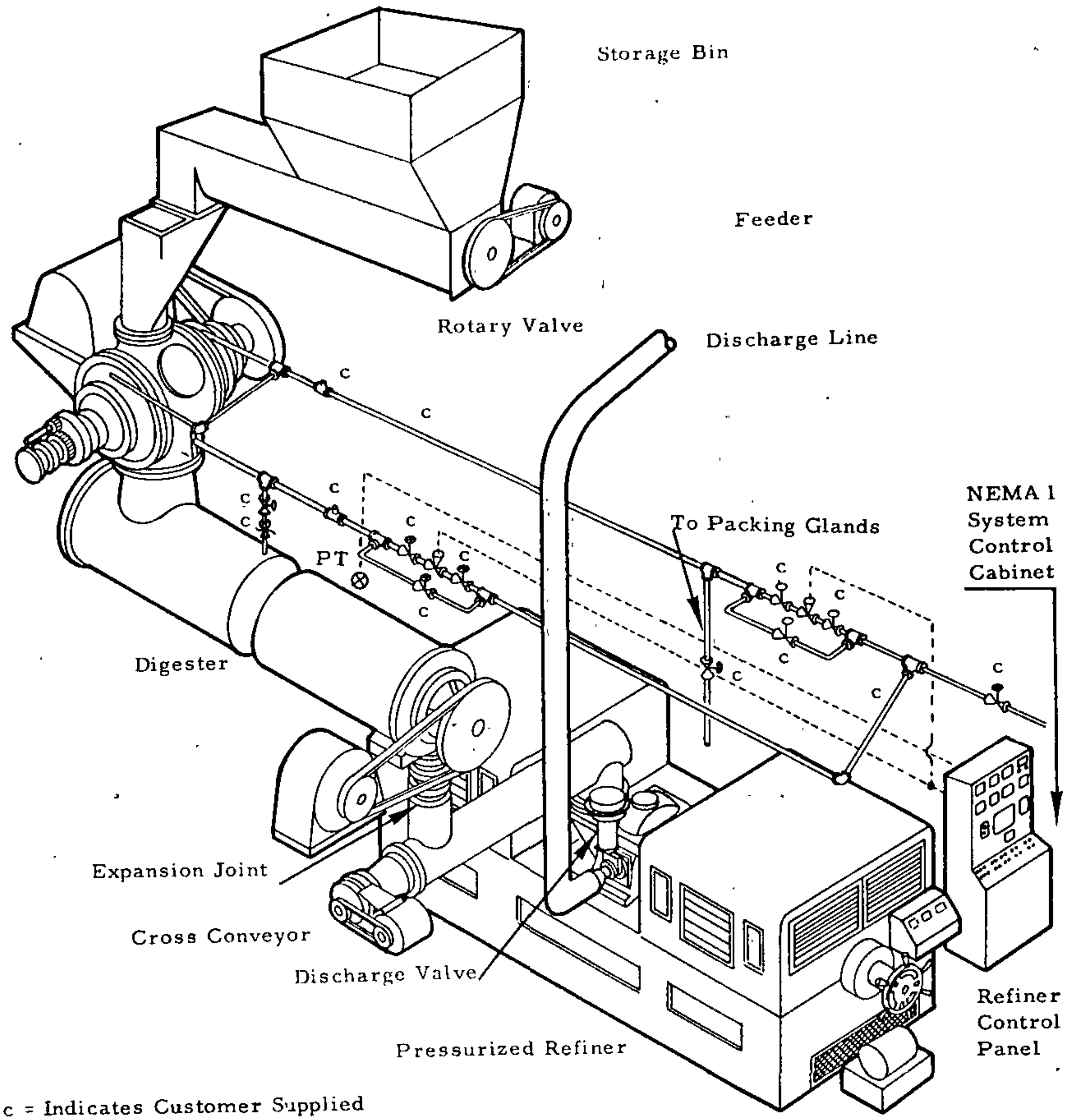


Figure 5. 418 Bauer Pressurized Refiner System

TABLE VI

NO. 418 BAUER FIBERIZATION DATA

	Loblolly						Black Spruce			
Code	A13	A17	A18	A14	A15	A16	A12	A9	A11	A10
Chip treatment[a]	R	R	R	I	I	I	R	R	I	I
Moisture content, % wet basis	35.0			55.9	56.5	55.5	23.1	22.6	64.9	63.7
Fiberization										
Steam pressure, p.s.i./°C.	30/135	70/158		30/135	50/147	70/158	30/135	70/158	50/147	70/158
Steaming time, min.	2.50		7.37	2.50			2.50		2.50	
Plate clearance, 0.001 in.	35			35			35		35	
Run time, min.	5.50	5.28	5.31	5.10	6.00	5.49	6.72	6.05	6.20	5.93
Feed rate, o.d. tons/day	6.87	6.27	6.23	6.92	6.78	7.17	4.56	4.32	4.18	4.30
Load, brake h.p. day/o.d. ton[b]	3.65	2.76	2.72	3.22	2.49	2.57	4.84	3.82	4.35	2.96
Bauer-McNett classification, %[c]										
On 6 mesh	43.0	23.3	21.8	38.5	31.5	24.0	66.8	57.0	34.7	37.9
On 12 mesh	21.6	20.2	18.4	18.1	18.9	19.3	15.1	10.0	20.9	21.1
On 35 mesh	25.1	37.4	43.3	23.9	31.2	36.9	12.5	21.0	23.6	29.1
On 65 mesh	4.4	5.7	7.3	4.1	5.2	5.8	1.8	7.0	4.9	6.0
Through 65 mesh (by difference)	5.9	13.4	9.2	15.4	13.2	14.0	3.8	5.0	15.9	5.9

[a] R = raw or untreated.
I = water impregnated.

[b] These values should be increased about 20% to obtain the estimated total, including allowances for no load and motor efficiency.

[c] Water temperatures 16-18°C.

Power consumption figures are given for the first time in Table VI. Previously, an estimate of 5 horsepower-days per ton of wood was used for fiberization power (4). It appears there is no reason to believe this figure is an underestimate and while the previous estimate could be conservative, it is not clear at present how valid it might be to accept power consumption figures for the low chip feed rates applicable to Table VI as a basis for what could be achieved at production rates.

The loblolly and black spruce materials with Codes A-16 and A-10, respectively, were selected as being the most suitable for further experiments. It will be noted that these materials were made using the same fiberization conditions which included a fiberization temperature of 158°C., reflecting in part the conclusion reached in Appendix I.

Delignification

After exploratory experiments indicated chlorine dioxide-alkali delignification of fiberized loblolly or black spruce could give a very high-yield pulp capable of developing appreciable bonding properties, a plan was made for the preparation of pulps in sufficient amount for handsheet evaluation. This plan provided for the alternatives of proceeding to a chlorine dioxide reaction with or without pretreatment of the fiberized material with alkali.

The possible advantages of a pretreatment with alkali were envisaged as being at least twofold. One advantage could arise from the removal of substances capable of consuming chlorine dioxide, which would mean more lignin should be degraded for a given amount of chlorine dioxide. A second advantage would be the removal of tall oil components for recovery.

In arriving at the conditions used for alkali pretreatment, trial experiments were carried out as shown in Table VII. From these, it appeared that, provided there was sufficient alkali present to ensure a final pH above 11.0, the yield differences were positive yet not great. Yield fell within the range 91.0-93.6% (70°) and 86.0-91.2% (90°) for loblolly, or 90.4-93.2% (70°) and 88.3-92.9% (90°) for black spruce, with extraction times of from 15 to 60 min., indicating that species, temperature, and time each had some influence on yield.

The conditions used for chlorine dioxide-alkali delignification of fiberized loblolly and black spruce with and without pretreatment with alkali are given in Tables VIII and IX. From these, it can be seen the final yields are 78-90%, whereas for hardwoods the yield level would be about 65-70% for the same consumption of chlorine dioxide. The reasons for this situation are believed to relate to basic differences in the chemical composition of softwoods and hardwoods. In softwoods, not only is there more lignin than in hardwoods, but the lignin is different in structure. The expected outcome of there being more lignin is that more will remain after reaction with a certain amount of chlorine dioxide. However, in addition to this, the lignin analyses show the amount of lignin removed is about 1.5 times the amount of chlorine dioxide added, which is appreciably less than found for hardwoods. At the same time, carbohydrate loss through dissolution in alkali is much less than found for hardwoods. This reflects the greater resistance of softwood hemicelluloses to alkali. The net outcome is that in all cases in Tables VIII and IX delignification was selective with respect to the lignin and carbohydrate content of the wood.

Primary Refining and Handsheet Evaluation

At the very high yield levels obtained as in Tables VIII and IX, separation into individual fibers did not occur as for a chemical pulp. Pulps that by

TABLE VII

TRIAL ALKALI PRETREATMENTS OF FIBERIZED LOBLOLLY AND BLACK SPRUCE

Material	Loblolly (A-16)								Black Spruce (A-10)							
Consistency, %	10															
Temperature, °C.	70				90				70				90			
Sodium hydroxide, %	2	4	6	8	2	4	6	8	2	4	6	8	2	4	6	8
Final pH																
15 min.	10.7	11.6	12.3	12.4	10.3	11.3	12.0	12.3	10.5	11.5	11.9	12.2	10.5	11.3	11.8	12.0
60 min.	10.1	11.5	12.0	12.3	9.6	11.3	11.9	12.1	10.4	11.5	11.8	12.1	9.6	11.2	11.6	12.0
120 min.	9.9	11.5	12.0	12.3	9.2	11.1	11.7	12.1	10.2	11.5	11.7	11.9	8.9	11.0	11.6	11.9
Yield, %																
15 min.	93.8	92.7	93.5	93.6	91.3	91.0	91.2	87.8	94.0	91.0	92.6	93.2	94.8	92.9	92.1	91.8
60 min.	94.8	93.4	93.3	91.7	90.5	86.9	86.6	88.4	92.6	91.8	90.9	92.2	91.7	92.3	90.9	90.1
120 min.	94.1	91.8	91.3	91.0	91.6	86.4	86.2	86.0	92.8	91.8	91.8	90.4	93.7	90.7	88.3	89.9

TABLE VIII

LOBLOLLY CHLORINE DIOXIDE-ALKALI DELIGNIFICATION

Starting material: A16[a], Klason lignin 28.6%, acid-soluble lignin 0.0%

Alkali conditioning - 4.0% NaOH, 45 min., 10% consistency								
Temperature, °C.	70		90		--	--	--	--
Final pH	11.6		11.4		--	--	--	--
Yield, %	94.7		91.2		--	--	--	--
Klason lignin, %	26.3		25.6		--	--	--	--
Acid-soluble lignin, %	0.0		0.0		--	--	--	--
Lignin modification - 25→35°C. in 60 min., 8% consistency								
ClO_2, %	6.0	9.0	6.0	9.0	6.0		9.0	
Time, min.	55	110	60	115	55		115	
Final pH	2.1	2.0	2.1	1.9	2.1		1.9	
Yield, %	93.9	93.2	89.8	89.7	98.6		97.7	
Klason lignin, %	24.4	21.9	23.6	21.5	26.4		24.2	
Acid-soluble lignin, %	1.1	2.1	1.0	2.0	1.2		2.0	
Alkali extraction - 6.0% NaOH, 60 min., 12% consistency								
Temperature, °C.	70		90		70	90	70	90
Final pH	11.8	11.6	11.5	11.0	10.9	10.4	10.5	10.1
Yield, %	87.1	86.0	82.1	78.9	88.8	90.1	86.2	86.5
Klason lignin, %	19.2	14.0	17.5	13.0	20.7	20.9	16.9	16.9
Acid-soluble lignin, %	0.5	1.2	0.5	0.9	0.6	1.5	1.0	1.0
Total lignin removed, %	8.9	13.4(?)	10.6	14.7	7.3	7.2	10.7	10.7
Carbohydrate loss, %[b]	4.0	0.6	7.3	6.4	3.9	2.7	3.1	2.8
Code	A16-1	A16-2	A16-3	A16-4	A16-5	A16-6	A16-8	A16-9

[a] Table VI

[b] Calculated.

TABLE IX

BLACK SPRUCE CHLORINE DIOXIDE-ALKALI DELIGNIFICATION

Starting material: A10[a], Klason lignin 27.4%, acid-soluble lignin 0.0%

Alkali conditioning – 4.0% NaOH, 45 min., 10% consistency								
Temperature, °C.	—70—		—90—		--		--	
Final pH	—11.6—		—11.4—		--		--	
Yield, %	—94.7—		—91.2—		--		--	
Lignin modification – 25→35°C. in 60 min., 8% consistency								
ClO_2, %	6.0	9.0	6.0	9.0	—6.0—		—9.0—	
Time, min.	70	130	70	135	—80—		—110—	
Final pH	1.9	1.8	1.8	1.7	—1.7—		—1.7—	
Yield, %	93.0	90.5	91.1	91.6	—98.8—		—96.9—	
Alkali extraction – 6.0% NaOH, 60 min., 12% consistency								
Temperature, °C.	—70—		—90—		70	90	70	90
Final pH	11.9	11.8	11.8	11.8	10.6	10.2	10.4	10.1
Yield, %	85.2	77.7	81.5	79.2	89.6	87.0	83.4	85.0
Klason lignin, %	17.0	10.6	15.1	10.8	18.9	18.3	14.4	14.2
Acid-soluble lignin, %	0.9	1.4	0.6	1.5	1.7	1.7	0.8	1.3
Total lignin removed, %	9.5	15.4	11.7	15.1	6.8	7.4	12.2	11.9
Carbohydrate loss, %[b]	5.3	6.9	6.8	5.7	3.6	5.6	4.4	3.1
Code	A10-1	A10-2	A10-3	A10-4	A10-5	A10-6	A10-7	A10-8

[a] Table VI.

[b] Calculated.

definition would be semichemical pulps were obtained by mechanical action using a Sprout-Waldron refiner. Details of the procedures are described in the experimental part. A high-yield slash pine kraft pulp kindly provided by one of the Grantors was used to provide a basis for comparison.

The amounts of material available made it necessary to proceeed without optimization of plate clearance, and it seems in retrospect that some of the clearances used as shown in Table X were probably somewhat high. This applies particularly to the loblolly chlorine dioxide-alkali pulps. Also, freeness values make it apparent that the plate settings of 0.012 in. were too close. A general impression gained from working with the high-yield kraft pulp and the chlorine dioxide-alkali pulps was that there was more similarity in their behavior during primary refining than might have been expected on the basis of yield.

TABLE X

PRIMARY REFINING IN 12-IN. SPROUT-WALDRON

Reference: high-yield kraft							
Plate gap, nom. 0.001 in.					12	15	18
Canadian freeness, ml.					475	740	740
Loblolly: chlorine dioxide-alkali							
Code, A16	1	2	3	4	5	5	9
Plate gap, nom. 0.001 in.	20	18	20	20	12	15	20
Canadian freeness, ml.	770	760	780	790	435	770	775
Black spruce: chlorine dioxide-alkali							
Code, A10	1	3	4	5	5	6	8
Plate gap, nom. 0.001 in.	15	15	18	15	18	15	15
Cnadian freeness, ml.	725	730	740	745	765	730	720

The results obtained by beating in a PFI mill and testing handsheets are presented in Tables XI and XII. In these tables the pulp codes indicate whether 6 or 9% chlorine dioxide was added and what yield was obtained to the nearest percent.

For both species, the best strength results in Tables XI and XII correlate with the lowest yields, which were 79%. If the data are subdivided into groups related to chlorine dioxide addition, the results corresponding to the addition of 6% chlorine dioxide tend to fall below those for 9% chlorine dioxide. This appears to be associated primarily with the amount of residual Klason lignin, which is reflected in the yield levels, except that at comparable yields there is less lignin in the pulps derived using 9% chlorine dioxide.

In Tables XI and XII, the sets of evaluation data toward the left have the highest lignin contents and those toward the right have the lowest lignin contents. The latter are of particular interest because they tended to have the best strength properties. In terms of the process conditions in Tables VIII and IX used to obtain the corresponding products, each was prepared with the inclusion of an alkali conditioning step carried out at 90°C. Thus, for the various conditions considered in Tables VIII and IX using 6 and 9% chlorine dioxide, the conditions used in the third and fourth columns led to pulps with the best handsheet properties.

It should be noted that freeness does not provide a reliable guide to handsheet drainage time. For example, in Table XI for a handsheet drainage time of say 4.2 sec., freeness varied from 395 to 595 ml., which is far beyond the limits of experimental error.

The handsheet data are of particular interest when compared with similar results for a high-yield kraft pulp. Such a comparison is made in Table XIII, in which the black spruce data are for an 85%-yield pulp prepared using 6.0% chlorine dioxide. It will be seen from Table XIII that at this very high yield level the

TABLE XI

HANDSHEET DATA FOR HIGH-YIELD CHLORINE DIOXIDE-ALKALI LOBLOLLY PULPS

ClO_2, %/yield, %/code	6.0/89/A16-5					6.0/87/A16-1					6.0/82/A16-3					
Sprout refiner plate clearance, 0.001 in.	15					20					20					
PFI mill, counter revs.	200	300	400	500	650	400	500	600	700	800	300	400	500	600	700	800
Canadian freeness, ml.	660	525	395	320	195	595	480	430	260	205	705	640	520	435	345	255
Handsheet drainage time, sec.	--	4.1	4.2	4.6	6.1	4.1	4.5	4.5	5.0	6.6	4.0	4.1	4.2	4.3	4.5	5.9
Handsheet density, g./cc.	0.326	0.354	0.385	0.398	0.444	0.400	0.424	0.417	0.461	0.488	0.366	0.381	0.407	0.434	0.447	0.501
Bendtsen smoothness, ml./min.[a]	3280	3150	2830	2810	2610	2810	2680	2690	2710	2550	3040	2990	2850	2740	2650	2470
Burst factor	8.4	13.4	14.8	14.6	17.6	18.8	21.2	20.9	24.7	27.9	13.5	14.9	18.3	21.2	25.8	29.4
Breaking length, km.	2.02	2.76	3.06	3.41	4.14	3.68	4.10	4.03	4.75	5.39	2.94	3.12	3.93	4.18	4.72	5.66
Stretch, %	0.9	1.2	1.2	1.3	1.4	1.4	1.6	1.4	1.6	1.8	1.2	1.2	1.5	1.6	1.7	2.0
Tear factor	49.8	58.0	56.9	57.4	49.2	76.8	75.4	68.0	64.5	64.2	86.0	85.9	80.0	78.9	74.7	68.1
Modified ring crush, lb./in.	3.1	4.3	4.4	4.8	5.0	4.4	4.7	5.1	4.6	5.2	5.3	5.3	5.7	5.7	5.7	6.3

ClO_2, %/yield, %/code	9.0/87/A16-9					9.0/86/A16-2						9.0/79/A16-4				
Sprout refiner plate clearance, 0.001 in.	20					18						20				
PFI mill, counter revs.	250	400	500	600	700	300	400	500	600	700	800	300	400	500	600	700
Canadian freeness, ml.	745	570	465	350	285	540	455	360	315	255	200	595	530	360	270	205
Handsheet drainage time, sec.	4.1	4.2	4.5	4.8	5.3	4.1	4.2	4.5	5.0	5.5	6.6	4.2	4.3	4.8	5.4	7.4
Handsheet density, g./cc.	0.358	0.392	0.442	0.463	0.465	0.429	0.446	0.465	0.479	0.487	0.505	0.430	0.454	0.487	0.494	0.525
Bendtsen smoothness, ml./min.[a]	3180	2910	2720	2720	2620	2660	2590	2620	2780	2690	2590	2810	2780	2560	2800	2640
Burst factor	14.9	20.9	26.4	27.3	28.0	25.0	27.7	30.1	30.7	32.7	34.2	28.1	31.0	35.4	37.3	41.5
Breaking length, km.	3.13	4.17	4.86	5.22	5.52	4.48	4.80	5.28	5.44	5.74	6.19	4.90	5.45	5.38	6.47	6.84
Stretch, %	1.2	1.5	1.8	1.7	1.8	1.9	1.9	1.9	2.1	2.1	2.1	1.8	2.0	2.2	2.3	2.1
Tear factor	88.8	83.4	74.2	72.5	70.5	94.6	91.3	85.0	82.6	80.5	76.8	92.3	89.5	86.2	78.0	73.7
Modified ring crush, lb./in.	4.3	5.1	5.1	5.4	5.4	4.3	4.6	5.1	5.2	5.7	6.4	4.7	5.4	5.4	5.3	5.3

[a] Rough side of handsheet

TABLE XII

HANDSHEET DATA FOR HIGH-YIELD CHLORINE DIOXIDE-ALKALI BLACK SPRUCE PULPS

ClO_2, %/yield, %/code	6 0/90/A10-5				6 0/87/A10-6					
Sprout refiner plate clearance, 0.001 in	15				15					
PFI mill, counter revs	300	500	700	900	150	300	500	700	900	1200
Canadian freeness, ml	600	480	395	275	670	605	500	380	290	195
Handsheet drainage time, sec	4.1	4.3	4.8	6.0	4.0	4.2	4.3	4.7	5.7	6.9
Handsheet density, g/cc	0.435	0.495	0.542	0.583	0.419	0.467	0.498	0.527	0.566	0.584
Bendtsen smoothness, ml/min [a]	2020	1760	1850	1730	2140	2140	1840	1610	1740	1560
Burst factor	33.9	39.1	45.8	48.7	30.8	37.9	41.3	42.9	47.5	48.6
Breaking length, km	6.17	7.23	7.83	8.29	5.68	6.58	7.13	7.65	8.38	8.57
Stretch, %	2.0	2.1	2.2	2.1	1.9	2.1	2.2	2.2	2.3	2.4
Tear factor	73.5	59.5	56.1	50.4	79.2	66.7	66.2	60.5	56.6	55.1
Modified ring crush, lb/in	6.3	6.2	6.7	7.4	5.9	6.6	7.2	6.9	7.4	7.8

ClO_2, %/yield, %/code	6 0/85/A10-1				6 0/82/A10-3					
Sprout refiner plate clearance, 0.001 in	15				15					
PFI mill, counter revs	300	500	700	900	150	300	500	700	900	1200
Canadian freeness, ml	585	525	370	300	670	600	510	400	305	200
Handsheet drainage time, sec	4.1	4.3	4.9	6.0	4.0	4.0	4.5	4.8	5.6	7.3
Handsheet density, g/cc	0.467	0.494	0.546	0.582	0.485	0.524	0.559	0.598	0.610	0.649
Bendtsen smoothness, ml/min [a]	1850	1740	1680	1590	1910	1660	1800	1620	1500	1500
Burst factor	41.3	46.7	52.7	55.7	42.7	46.9	52.0	54.8	56.2	63.5
Breaking length, km	6.78	7.59	8.14	8.81	6.65	7.48	7.82	8.67	8.61	9.61
Stretch, %	2.2	2.3	2.4	2.4	2.3	2.4	2.5	2.5	2.4	2.6
Tear factor	74.6	70.1	64.5	56.8	74.6	66.3	64.7	59.2	57.5	53.1
Modified ring crush, lb/in	6.2	6.2	6.8	6.5	5.9	6.8	6.9	7.5	6.7	6.6

ClO_2, %/yield, %/code	6 0/90/A10-5					9 0/85/A10-8				9 0/79/A10-4				
Sprout refiner plate clearance, 0.001 in	18					15				18				
PFI mill, counter revs	300	500	700	900	1100	300	500	700	900	300	500	700	900	1200
Canadian freeness, ml	690	535	505	395	320	595	525	420	315	640	570	525	465	330
Handsheet drainage time, sec	4.0	4.2	4.3	4.7	5.1	4.1	4.4	4.8	5.9	4.1	4.2	4.4	4.8	5.2
Handsheet density, g/cc	0.364	0.429	0.450	0.509	0.533	0.480	0.527	0.552	0.593	0.523	0.563	0.577	0.610	0.648
Bendtsen smoothness, ml/min [a]	2460	2140	1960	1750	1630	1930	1910	1780	1860	2200	1910	1860	1810	1580
Burst factor	25.3	34.2	35.7	41.9	44.3	46.0	51.4	55.1	57.6	53.7	55.0	62.2	62.8	66.3
Breaking length, km	5.07	6.19	6.51	7.67	8.00	7.94	8.34	8.99	9.28	8.18	9.06	9.16	9.79	10.2
Stretch, %	1.5	1.8	2.0	2.1	2.0	2.3	2.2	2.3	2.3	2.3	2.7	2.5	2.6	2.7
Tear factor	81.7	70.4	68.3	60.7	56.5	70.8	63.9	59.7	55.6	71.2	72.7	63.4	62.8	54.8
Modified ring crush, lb/in	5.3	6.0	6.3	6.2	6.6	6.7	6.7	6.9	7.1	6.4	6.9	6.9	6.6	7.0

[a] Rough side of handsheet

TABLE XIII

COMPARISON OF HANDSHEET DATA FOR HIGH-YIELD KRAFT SLASH PINE PULP AND 85%-YIELD CHLORINE DIOXIDE-ALKALI BLACK SPRUCE PULP

Pulp	59%-Yield Slash Pine Kraft					85%-Yield Black Spruce ClO_2-Alkali (A10-1)			
Sprout refiner plate clearance, 0.001 in.	18					15			
PFI mill, counter revs.	300	500	700[a]	900	1100	300	500	700	900
Canadian freeness, ml.	745	670	475	320	225	585	525	370	300
Handsheet drainage time, sec.	4.1	4.2	4.4	5.1	7.5	4.1	4.3	4.9	6.0
Handsheet density, g./cc.	0.447	0.490	0.521	0.546	0.578	0.467	0.494	0.546	0.582
Bendtsen smoothness, ml./min.	2450	2220	2230	2200	2220	1850	1740	1680	1590
Burst factor	38.9	48.8	54.5	58.9	61.1	41.3	46.7	52.7	55.7
Breaking length, km.	5.71	6.66	7.93	8.39	8.93	6.78	7.59	8.14	8.81
Stretch, %	2.2	2.5	3.0	2.9	2.8	2.2	2.3	2.4	2.4
Tear factor	224	190	169	151	130	75	70	65	57
Modified ring crush, lb./in.	4.9	4.6	4.7	5.0	4.8	6.2	6.2	6.8	6.5

[a] On the basis of 9 beatings. 95% confidence limits of the mean were found to be: Canadian freeness ± 24 ml., handsheet drainage time ± 0.08 sec., handsheet density ± 0.032 g./cc.; Bendtsen smoothness ± 140 ml./sec., burst factor ± 5.6, breaking length ± 0.48 km.; stretch ± 0.2%; tear factor ± 14, and modified ring crush ± 0.4 lb./in.

handsheet properties indicate that the pulp has considerable potential strength and compares favorably with the high-yield kraft pulp except for tear factor. For a linerboard, the other properties are believed to be more important.

The two levels of smoothness values in Table XIII are believed to reflect a species difference. Both pulps were comparatively free of shives and the same comment applies to the best of the loblolly pulps (Table XI). There was an observable trend to an increased number of shives in the higher yield pulps, as indicated by a comparative decrease in smoothness in the case of Pulp A16-5, for example, even in the face of a smaller refiner plate clearance.

Considering again the results for loblolly (Table XI), it will be seen that the best set of results, which was for Pulp A16-4, has sheet density and ring crush values about comparable to those for the high-yield kraft reference pulp (Table XIII). Burst factor and breaking length values for the chlorine dioxide-alkali pulp are at lower levels. However, it should be noted that the primary refiner gap was 0.020 in. for the loblolly chlorine dioxide-alkali pulp. It is believed appreciably better results for this pulp could be obtained by primary refining with a smaller disk gap. This is supported, for example, by the handsheet data in Table XII for Pulp A10-5, which was primary refined with 0.018 and 0.015-in. plate gaps. If optimizing the primary refining conditions for loblolly chlorine dioxide-alkali pulps gave an improvement in handsheet properties similar to the difference for the two sets of results for Pulp A10-5, the data for loblolly would compare more favorably with the high-yield reference pulp such as used for linerboard base stock.

Another line of thought which could lead to better results for loblolly than those presented above follows from the trends already observed. Since the best strength values were associated with an alkali conditioning step at 90°C.

compared with either no alkali conditioning or 70°C., a more severe alkali pretreatment at a temperature above 90°C. could prove to be advantageous.

To provide a more complete picture concerning the possibility of chlorine dioxide-alkali pulps being of merit in linerboard production, for example, approximate costs are considered in the following discussion.

Approximate Cost Estimates

Approximate costs for producing pulps as in Tables VIII and IX will be considered with reference to the costs provided in Report Eleven (4). For the purpose of discussion here the costs from that report for the case of pulping aspen with aqueous chlorine dioxide in a closed system will be treated as being composed of:

(a) wood costs,

(b) other costs (excluding capital), and

(c) capital costs.

Other costs as covered in this list include make-up chemicals, utilities, and labor.

To aid in the present discussion, relevant costs from Report Eleven (4) are given in Appendix II.

Wood costs used in the example presented in Table XIV are based on information obtained for southern pine. On the basis of information obtained for black spruce, one rough cord yields 2120 lb. ovendry wood, which is a lower yield than that applied in Table XIV, implying the wood costs for black spruce would be relatively higher for the same cost per cord. The range of wood costs considered in Table XIV is believed to be realistic in terms of today's costs in North America and Europe (5).

TABLE XIV

WOOD COSTS PER AIRDRY TON OF PULP ($)

Cord Cost	80% Yield[a]	55% Yield[a]	Difference
20	20.4	29.8	9.4
25	25.5	37.2	11.7
30	30.6	44.7	14.1
35	35.7	52.1	16.4
40	40.8	59.6	18.8

[a] Basis: One rough green cord of southern pine pulpwood provides 2200 lb. of ovendry chips. Therefore, 2250/2200 or 1.02 cords per ton of airdry pulp at 80% yield (1800 lb. = 1 ton a.d. pulp), and 3270/2200 or 1.49 cords per ton a.d. pulp at 55% yield.

Other costs (excluding capital) are set out in Table XV. In this table the costs per ton of pulp for make-up chemicals, etc., have been reduced directly in proportion to lowered wood input and any lowered chlorine dioxide requirement. Changes in utility costs also reflect the same factors to the extent applicable, and labor costs have been assumed to be the same per ton of pulp as estimated previously (Appendix II).

The basis used in Report Eleven (4) for calculating the costs in Appendix II was for chemical usages of

9% ClO_2,

10% NaOH, and

3% NaOCl.

Thus, the cost of producing sodium hypochlorite, the use of which is not applicable to the bleached softwood pulps being considered in this report, is included in Table XV. Exclusion of the cost of hypochlorite should lower the upper total cost figure about 5-10%. On the other hand, it should be noted that when 6% ClO_2 was

TABLE XV

OTHER COSTS (EXCLUDING CAPITAL) PER AIRDRY TON OF PULP ($)

	80% Yield			
	9% ClO_2		6% ClO_2	
A. Make-up chemicals, etc.				
$\frac{3.60}{3000}$ (Appendix I) x 2250 for 9% ClO_2	2.70		1.80	
		2.70		1.80
B. Utilities				
Electric power for cells	7.05		4.70	
Other power	2.05		2.05	
Steam (assuming no steam generation), say	3.75		3.75	
Chilling	1.50		1.00	
		14.35		11.50
C. Labor				
Assume as in Appendix I	7.50		7.50	
		7.50		7.50
Totals:		24.55		20.80

used (Tables VIII and IX), sodium hydroxide addition remained at 10%. Therefore, reducing previous costs in proportion to the lowered chlorine dioxide requirement leads to an underestimate regarding sodium hydroxide. However, just roughly and conservatively, the inclusion of sodium hypochlorite and the underestimate for sodium hydroxide would probably about balance out for the case of 6% ClO_2.

Hence, costs covered in Table XV for producing pulps such as in Tables VIII and IX would be about \$20-\$25 per ton airdry pulp. If the basis for electrical power costs were to be changed from 5 to 7.5 mils/kw.-hr., this would add about \$3.40-\$4.55. With optimization of the pulping conditions, it seems unlikely that as much as 9% chlorine dioxide would be needed, so that even if electrical power costs were 6.0 mils/kw.-hr., a \$20-\$25 range plus wood and capital costs probably can be taken as a guide to a first approximation of pulp costs per ton.

In Report Eleven (4), capital requirements for 500 tons of pulp per day were estimated to be about \$27.50 x 10^6 or \$55,000 per daily ton. It has been suggested that this estimate is low. For this discussion, it simply will be assumed that capital costs per ton of pulp would be equal to those for a 55% high-yield kraft pulp mill, in which case they can be put to one side.

What appears as the most outstanding feature of the above considerations is that as wood costs increase, the dollar benefit at the yield levels being investigated tends to approach all other operating costs and could even outweigh them if labor costs are excluded. Obviously, in those areas where wood costs are already at or near the high values given in Table XV, the possibility of producing pulp at the higher yield level should be of greatest immediate interest.

Table XVI has been prepared to illustrate this situation. In Table XVI the wood cost ranges in Columns A, B, and C are based on Table XIV. The range of other costs (excluding capital) for the case of an 80%-yield ClO_2-NaOH pulp is from the discussion above where it was noted that this range probably can be used as a guide in obtaining a first approximation of costs. This \$20-\$25 range of other costs (excluding capital) is applicable to both ends of the range for wood costs. In other words, the low range total would be \$40-\$45.40 and the high range total would be \$60.80-\$65.80. The range of totals in Table XVI has been simplified by considering the lowest and highest sums. To balance out the difference in wood costs between Columns A and B it is necessary to add \$10.60-\$6.20. This implies that for a 55%-yield kraft pulp to equal an 80% ClO_2-NaOH pulp in cost, corresponding other costs (excluding capital) for the kraft pulp would need to be \$10.60-\$6.20. The figure of \$11.30 used in Column C exceeds this range and thereby indicates a cost disadvantage for the kraft pulp. This figure of $11.30 is derived on the basis shown in the footnote to Table XVI in which labor was equated to that allowed for in the case of a ClO_2-NaOH pulp.

TABLE XVI

COMPARISON OF ClO_2-NaOH AND KRAFT PULP COSTS PER AIRDRY TON ($)

	80%-Yield ClO_2-NaOH	55%-Yield Kraft	
	A	B	C
Wood cost ranges (Table XIV)	20.40-40.80	29.80-59.60	29.80-59.60
Other costs (see discussion)	20.00-25.00		
Range of totals	40.40-65.80		
To balance Column A range		10.60- 6.20	
Range of totals		40.40-65.80	
Other costs for Column C[a]			11.30 11.30
Range of totals			41.10-70.90

[a] Bases of estimated other costs (excluding capital) for kraft:

Chemical make-up:	Salt cake at $25.00/ton	$ 1.25
Utilities:	Power at $0.012/kw.-hr.	0.44 (6)
	Fuel oil at $0.00956/gal.	2.11 (6)
Labor:	One man-hour	7.50
		$11.30

These approximate cost estimates are interpreted as showing it is realistic to consider a very high-yield ClO_2-NaOH softwood pulp as a possible alternative to a high-yield kraft pulp, and that where wood costs are high the former pulp could be significantly lower in cost.

EXPERIMENTAL

RAW MATERIALS

Loblolly

The loblolly pine (Pinus taeda L.) chips were prepared as described in Report Ten (1). The wood used was the remaining half of the sticks of loblolly kindly supplied by one of the Grantors.

The screened chips had an initial ovendry content of 46.7% and a density of 30.0 lb. o.d./ft.^{3} green.

Black Spruce

Fourteen 100-in. long bolts of black spruce (Picea mariana) pulpwood were obtained from Consolidated Papers, Inc., Appleton, Wisconsin. The bolts came from Manitoba and had underbark diameters varying from 4 to 6 in. Part of each log was debarked and put through a 4-knife, 38-in. Carthage chipper at the Institute. The chips were screened on a 24-in. Sweco Dynoscreen with the through-1-inch-and-on-4-mesh fraction being retained and stored in plastic-lined drums at 40°F.

The screened chips had an initial ovendry content of 63.4% and a density of 26.0 lb. o.d./ft.^{3} green.

CHIP FIBERIZING

No. 418 Bauer Pressurized Refiner

Halves of each lot of loblolly and black spruce chips were impregnated with water prior to chip fiberizing with the remainder of the chips being fiberized without any pretreatment. The water impregnation of the chips was carried out batchwise as follows: The equivalent of about 15 lb. o.d. chips was packed into a basket and placed in a 2-cu.-ft. stainless steel digester and steamed at 15-20 p.s.i. for 2 min.; the pressure was relieved to atmospheric followed by steaming again at 15-20 p.s.i. for a further 2 min. The basket of chips was then transferred to a second 2-cu.-ft. stainless stéel digester, covered with distilled water, and an overpressure of 100 p.s.i. supplied from a nitrogen gas cylinder applied for 30 minutes. The water-impregnated chips were allowed to drain for 15 min. before being emptied into a plastic-lined barrel.

Chip fiberizing was carried out using the no. 418 Bauer pressurized refiner at Bauer Bros. Co., Springfield, Ohio. A diagram of this refiner is given in Fig. 5. Both the water-impregnated and the "raw" or untreated chips were made into batches equivalent to about 45 lb. ovendry which was the minimum quantity that could be run through this machine. For each run, a batch of chips was emptied into the storage bin, conveyed along the digester, which could be maintained at a given steam pressure, and then fed directly to the no. 418 refiner and fiberized at the same steam pressure. From the storage bin to the refiner outlet was a continuous operation so that each chip theoretically had the same treatment. The "dwell time" in the digester, i.e., the time the chips were subjected to a preset steam pressure, could be controlled within limits independently of the throughput of the refiner which was set at a nominal 5 tons o.d. per day for all runs. The plate pattern used was no. 36325/36326 with a 0.010-in. taper. The refining conditions and related power figures are given in Table VI. A guide to the plate clearance to use was obtained by running bucket lots of water-impregnated chips through the refiner. After each run, the fiberized material was air cooled by passing it through a Bauer fluffer and then a cyclone. The material was collected in 50-gal. fiberboard drums.

SODIUM CHLORITE-ALKALI DELIGNIFICATION

Details of this procedure are given in Progress Report Twelve (2).

CHLORINE DIOXIDE-ALKALI DELIGNIFICATIONS

All reactions were carried out in plastic bags with the products being mixed at regular intervals. After each reaction, the products were washed four times with deionized water. Dewatering was effected using a laundry-type spin drier with the reacted products being contained in a nylon bag. Up to about 800 g. o.d. fiberized chips were used in preparing each pulp.

Alkali Conditioning

Where included, alkali conditioning was carried out under conditions recorded in Tables VIII and IX.

Lignin Modification

The lignin modifications, using an aqueous solution of chlorine dioxide, were carried out as recorded in the tables.

Alkali Extraction

Alkali extractions were carried out using conditions as recorded in the tables.

PRIMARY REFINING

Primary refining of the high-yield pulps was carried out in a modified model 105-A 12-in. Sprout-Waldron single-disk refiner. This equipment and the procedure used were kindly made available to us by one of the Grantors. For each refining run, the pulp was initially steamed at atmospheric pressure for 5 min. and then fed to the refiner at 3.5% consistency with the dilution water coming in at 80-85°C. All refining was a single pass through the equipment with the extent of fiber bundle reduction and the formation of fines being determined visually on a very dilute suspension. It should be noted that the plate clearances given in Tables XI and XII are only nominal as zero clearance was determined when the refiner was cold. The refined pulps were dewatered in a centrifuge with recycling of the water to prevent loss of fines.

PULP BEATING AND EVALUATION

The pulps that were beaten in a 1-1/2 lb. Valley beater were processed according to TAPPI Standard Method T 200 ts-66 with the exception that a 4.5-kg. weight was used on the end of the bedplate lever and 1600 ml. of stock were withdrawn at each beating interval. One 50-g./m.^{2} and ten 60-g./m.^{2} handsheets were formed at each beating interval.

The conditions for beating pulps in a PFI mill were as follows:

pulp charge, equivalent to 24.0 g. ovendry,

beating load, 3.4 kg./cm.,

beating consistency, 10%, and

beating temperature, 25 ± 5°C.

Prior to beating, each pulp charge was diluted to 2 liters and disintegrated for 10 min. in a TAPPI disintegrator, and after each beating the charge was cleared for 5 min. using the same conditions. From each charge of beaten pulp five 60-g./m.^{2} handsheets were formed.

Canadian freeness determinations were made as set out in TAPPI Standard Method T 227 m-58 and handsheets were formed and conditioned as in TAPPI Standard Method T 205 m-58.

Details of the handsheet testing carried out on the 60-g./m.^{2} handsheets are as follows:

Density, g. ovendry/cc.; breaking length, km.; stretch, %; burst factor, tear factor, and M.I.T. fold: TAPPI Standard Method T 220 m-60.

Tensile energy absorption, g.cm./cm.^{2}: TAPPI Standard Method T 494 su-64.

Tensile stiffness, E.t., kg./cm.: calculated from the slope of the initial load/elongation curve determined for the tensile energy absorption tests.

In-plane tear, g.cm./cm.: J. A. Van den Akker, W. A. Wink, and R. H. Van Eperen, Tappi 50, no. 9:466-70 (1967).

Bendtsen smoothness, ml./min.: determined on the rough side of handsheets only, according to the Bendtsen Handbook for the Model VI Smoothness and Porosity Tester, issued by Anderson and Sorenson, 1964.

Specific scattering coefficients and specific absorption coefficients (7, 8) were determined using a GE recording spectrophotometer, serial no. 716228, at a wavelength of 650 nm. with the "matte" side of the handsheet test piece facing the incident light beam. The two values measured were reflectance, R_∞, and transmittance, T. Conditioned basis weights (73°F. and 50% R.H.) were used in the calculations. Both coefficients are expressed in cm.^{2}/g., i.e. reciprocal basis weight units.

Modified ring crush, lb./in.: carried out according to TAPPI Standard Method T 472 m-51 but with the following modifications. Each test piece was wrapped around a 0.597-in. diameter mandrel with the overlap being glued with a rubber-based adhesive and the loading edges reinforced by dipping them into molten paraffin wax (Mobil D).

The tensile tests were performed on a table-model Instron tester with a test span of 4.0 in., a test piece width of 1.00 in., and a crosshead speed of 1 in./min.

M.I.T. fold, tensile energy absorption, tensile stiffness, in-plane tear, and modified ring crush results were adjusted to a 60.0-g./m.^{2} basis weight by simple proportion.

Zero-span breaking length tests were carried out on the 50-g./m.^{2} handsheets essentially according to TAPPI Standard Method T 231 sm-60.

CONCLUSIONS

On the basis of experiments described and discussed in this report various related conclusions have been reached in connection with using a process including the steps of chip fiberization with or without alkali pretreatment, and delignification by chlorine dioxide and alkali extraction to produce unbleached semichemical papermaking pulps from softwoods.

By fiberizing loblolly chips at higher temperatures than used in previous work it has been concluded there is significantly less fiber damage and that pulps can be obtained with no apparent reduction in fiber length distribution.

In delignification by chlorine dioxide plus alkali extraction, pretreatment with alkali is concluded to be advantageous since it resulted in greater lignin removal and the same pretreatment would provide for the removal of tall oil components for recovery. Delignification has been concluded to be selective with respect to the lignin and carbohydrate components of the softwoods considered, namely loblolly pine and black spruce.

Semichemical chlorine dioxide-alkali pulps, obtained in 78-90% yield, have significant papermaking properties and develop appreciable strength with beating. A black spruce 85%-yield pulp made using 6% chlorine dioxide compares favorably with a slash pine 59%-yield kraft pulp in terms of sheet density, bonding properties, and ring crush values. The implication from consideration of loblolly chlorine dioxide-alkali pulps is that it should be possible through further research to obtain somewhat comparable results at about 80% yield.

From approximate cost estimates it is concluded that consideration of a very high-yield chlorine dioxide-alkali softwood pulp as a possible alternative to a high-yield kraft pulp is realistic, and that where wood costs are high the former pulp could be significantly lower in cost.

FUTURE WORK

In view of the promising results obtained for chlorine dioxide-alkali pulping of loblolly pine and black spruce, as described in this report, consideration is given here to possible future work.

The objective would be to demonstrate more clearly the future prospects for producing an unbleached semichemical chlorine dioxide-alkali softwood pulp such as may be suitable for linerboard, especially with the possibility of a pilot plant in view.

A tentative plan is envisaged as being based as follows:

Raw Materials. Loblolly pine and at least another softwood would be chosen as the wood species.

Process. Process steps would follow directly from the description in this report which includes alkali conditioning after chip fiberization. This raises the question of whether the alternative of chip impregnation with alkali instead of water could allow simplification of the process with omission of alkali conditioning after chip fiberization. An answer to this question would be sought.

Pulp and Estimated Costs. Pulp evaluation and sharpened estimated costs would be of focal concern in determining whether an acceptable unbleached linerboard pulp could be made at favorable cost. Evaluation would include determination of hydrodynamic properties and pulp cost estimates would be for a completely specified process chosen on the basis of the results obtained.

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APPENDIX I

ASPLUND FIBERIZATION OF BLACK SPRUCE

In the main body of this report investigations on loblolly pine are presented and discussed regarding the prospect of obtaining better results on softwoods when higher fiberization temperatures were used. Investigations on black spruce in a companion study were put to one side in order to simplify presentation and because it is believed the results do not significantly alter the picture. These results are presented here as part of the record.

Fiberization and delignification data are set out in Table XVII with the conditions for cooking a kraft pulp for reference given in Table XVIII. Primary handsheet data are shown in Tables XIX, XX, and XXI.

One aspect of the results which is considered worthy of note is illustrated in Fig. 6. From this it can be seen that there is a very significant difference in the slope of the handsheet drainage time vs. beating time curve for Pulp AB-7C compared with either Pulp AB-15C, AB-17C, or AB-18C. This difference is viewed as being of potential importance since the curve for Pulp AB-7C tends to reflect the kind of behavior generally expected for holocellulose pulps insofar as beating for a relatively short time can result in such a marked increase in drainage time that this would be a practical disadvantage. The fact that it is possibly to considerably alter the beating behavior of pulps to give an improvement as illustrated in Fig. 6 demonstrates there can be important exceptions to the general expectation concerning holocellulose-type pulps.

It follows from the process conditions given in Table XVII for preparing the black spruce pulps under consideration in Fig. 6 that the observed difference in pulp behavior is caused by differences in the chip fiberization conditions.

TABLE XVII

BLACK SPRUCE FIBERIZATION AND CHLORITE-ALKALI DELIGNIFICATION DATA

Chip impregnation	([a])		Two 2-min. steam, 14 p.s.i.; cold H_2O impreg. 30 min., 78-86 p.s.i.							
Initial m.c., % wet basis	38		55		38					
Final m.c., % wet basis	62				66			66		
Preheating and fiberization										
Preheater steam pressure, p.s.i./°C.	28/133		71/158		71/158			86/165		
Time to pressure, sec.	--		45		45			55		
Time in preheater, min.	7.0		3.0		1.5			1.5		
Defibrator steam pressure, p.s.i./°C.	28/133	71/158	28/133		71/158			86/165		
Feed period, min.	0-3.0	3.0-6.0	0-4.0	4.0-8.0	0-1.5	1.5-2.8	2.8-4.1	0-1.5	1.5-2.8	
Disk gap, 0.001 in.	8		12	16	16			16		
Fiberized chip code	AB-1	AB-2[b]	AB-6	AB-7	AB-15	AB-16	AB-17	AB-18	AB-19	
Hot-water solubility, % o.d.f.c[c]	--		--	--	4.3	--	4.7	4.9	--	
Bauer-McNett classification, % o.d.f.c.[d]										
On 6 mesh	9.7		21.9	25.3	27.4	--	19.5	28.0	28.3	
On 12 mesh	29.0		28.1	25.9	26.0	--	27.7	26.1	25.6	
On 35 mesh	37.0		30.9	29.2	28.7	--	33.9	29.1	32.5	
On 65 mesh	12.3		8.1	7.0	6.1	--	6.9	6.2	4.6	
Through 65 mesh (by difference)	12.0		11.0	12.6	11.8	--	12.0	10.6	9.0	
Comments on photographs (35X magnification) of the on- and through-65-mesh Bauer-McNett fractions	most fibrillated		no difference, slight evidence of fibrillation						--	
Chlorite-alkali delignification										Pin chips[e]
Alkali conditioning[f], yield %	--		--	91.2	91.4	--	91.5	93.2	92.2	--
Chlorite oxidation[g], yield %	--		--	70.4	73.2	--	72.2	70.8	73.5	75.8
Alkali extraction[h], yield %	--		--	64.1	64.7	--	65.3	63.8	66.5	65.2
Brightness, GE (ethanol)	--		--	64.0	55.7	--	60.5	64.3	59.7	64.3
Chlorite-alkali pulp code	--		--	AB-7C	AB-15C	--	AB-17C	AB-18C	AB-19C	BS/P1

[a] *Cold water at 14 p.s.i. for 60 hr.*

[b] Sample discarded.

[c] TAPPI Standard Method T 207 m-54.

[d] Water temp. 19.5°C. except for AB-19 where it was 8°C.

[e] 3/32 x 3/32 x 3/4-in. pin chips.

[f] *8-10% consistency; 10% NaOH on o.d.f.c. for 60 min. at 50°C.* (45 min. from ambient).

[g] 8-10% consistency; 100% $NaClO_2$ and 15% HAc on o.d.f.c. for 12 hr.; temp. increased to 40-50°C. after 4 hr. from ambient, then decreased; initial pH's = 4.3, final pH's = 4.0-4.5; reaction time for the pin chips was 25.5 hr.

[h] 8-10% consistency; 5% NaOH on o.d.f.c. for 60 min. at 25°C.

TABLE XVIII

KRAFT PULPING OF BLACK SPRUCE

Digester chip charge, g. (oven dry)	5044
Active alkali, Na_2O on o.d. wood, %	15.5
Sulfidity, Na_2O basis, %	28
Liquor-to-wood ratio	4:1
Maximum temperature, °C.	180
Time to max. temp. (linear from 80°C. with gas relief at 105°C.), min.	90
Time at max. temp., min.	60
Blowdown time to 80 p.s.i., min.	15
Screened yield on o.d. wood, %	47.1
Screen rejects on o.d. wood, %	1.2
Kappa number	26.4
Code	BS/K1

TABLE XIX

BLACK SPRUCE - PRIMARY HANDSHEET DATA FOR CHLORITE-ALKALI PULPS AB-7C AND BS/P1 AND UNBLEACHED KRAFT PULP BS/K1

Pulp code	AB-7C					BS/P1[a]					BS/K1					
Pulp yield, %	64.1					65.2					47.1					
Beating weight[b], kg.	4.5					4.5					5.5					
Beating time, min.	1	3	8	12	16	2	5	9	13	20	1	6	12	21	36	45
Canadian freeness, ml.	550	505	340	205	105	650	650	580	530	270	685	670	640	580	385	250
Handsheet drainage time, sec.	4.8	4.5	4.8	7.3	13.8	4.2	4.2	5.1	8.0	210	4.5	4.3	4.6	4.5	6.8	11.3
Handsheet density, g./cc.	0.644	0.663	0.689	0.719	0.736	0.640	0.675	0.700	0.706	0.711	0.613	0.657	0.698	0.721	0.762	0.769
Opacity, %	66.2	64.3	60.6	58.5	56.4	67.9	64.0	61.0	59.8	51.9	--	--	--	--	--	--
Spec. scattering coeff.[c]	183	175	157	142	130	193	173	150	141	107	283	254	218	195	166	154
Spec. absorption coeff.[c]	8.83	8.44	10.1	11.0	12.4	7.18	7.60	8.30	8.61	11.2	68.7	65.5	62.5	60.7	59.6	58.6
Breaking length, km.	11.5	12.3	12.9	12.8	13.5	15.9	15.8	16.3	16.8	15.3	11.0	13.2	14.1	14.7	16.0	16.8
Stretch, %	2.6	2.6	2.5	2.5	2.3	2.3	2.3	2.3	2.2	2.0	1.9	2.2	2.6	2.7	3.0	2.6
Tensile energy absorp., g.cm./cm.^{2}	116	121	127	128	121	130	133	138	134	111	82.5	113	146	154	185	166
Tensile stiffness, kg./cm.	615	636	660	687	725	739	741	766	785	790	690	738	704	710	748	781
Burst factor	77.7	79.7	86.6	90.8	90.1	100	112	107	110	101	60.5	81.2	96.0	107	113	116
Tear factor	73.9	72.8	69.2	66.3	59.1	81.4	79.3	76.9	75.5	71.3	142	122	109	103	94.3	87.3
Tear factor x yield/65	72.9	71.8	68.2	65.4	58.3	81.7	79.5	77.1	75.7	71.5	103	88.4	79.0	74.6	68.3	63.3
In-plane tear, g.cm./cm.	86.6	82.1	75.7	73.6	70.3	79.3	74.9	74.9	77.6	69.8	105	113	115	114	111	109
Zero-span breaking length, km.	19.1	19.3	19.7	20.1	19.8	25.5	25.9	24.5	26.1	23.2	25.3	26.7	25.9	26.4	26.1	25.7
Zero-span b.l. x yield/65, km.	18.8	19.0	19.4	19.8	19.5	25.6	26.0	24.6	26.2	23.3	18.3	19.3	18.8	19.1	18.9	18.6
M.I.T. fold	833	907	1120	1120	1260	721	800	910	829	802	1330	1600	2310	2680	3540	3900

[a] Pin chip chlorite-alkali pulp.

[b] Valley beater-weight on end of bedplate lever arm.

[c] At 650 mm.

TABLE XX

BLACK SPRUCE - PRIMARY HANDSHEET DATA FOR CHLORITE-ALKALI PULPS AB-15C, AB-17C, AB-18C, AND AB-19C

Pulp code	AB-15C					
Pulp yield, %	64.7					
pH unbeaten stock	-					
Beating weight, kg.[a]	4.5					
Beating time, min.	1	4	8	15	27	40
Canadian freeness, ml.	720	690	665	625	500	290
Handsheet drainage time, sec.	4.5	4.4	4.5	4.6	4.9	7.7
Handsheet density, g./cc.	0.568	0.595	0.629	0.658	0.699	0.732
Opacity, %	70.2	70.1	68.5	67.2	62.8	57.4
Spec. scattering coeff.[c]	202	201	188	177	158	133
Spec. absorption coeff.[c]	17.5	17.7	16.6	17.6	16.0	16.6
Breaking length, km.	7.84	9.79	10.9	11.8	12.0	13.0
Stretch, %	2.3	2.5	2.9	2.9	2.6	2.5
Tens. energy abs., g.cm./cm.^{2}	81.4	101	128	139	134	135
Tensile stiffness, kg./cm.	548	567	583	616	637	746
Burst factor	55.9	71.4	80.4	84.4	88.0	91.5
Tear factor	90.6	81.9	75.8	74.5	63.9	59.1
Tear factor x yield/65	90.2	81.5	75.5	74.2	63.6	58.8
In-plane tear, g.cm./cm.	94.4	91.7	86.5	83.1	74.4	66.6
Zero-span breaking length, km.	16.6	17.6	17.9	17.7	18.5	18.5
Zero-span b.l. x yield/65, km.	16.5	17.5	17.8	17.6	18.4	18.4
M.I.T. fold	595	643	788	1040	1310	1570

Pulp code	AB-17C					
Pulp yield, %	65.3					
pH unbeaten stock	-					
Beating weight, kg.[a]	4.5					
Beating time, min.	1	3	8	16	28	38
Canadian freeness, ml.	690	680	665	600	485	335
Handsheet drainage time, sec.	4.5	4.5	4.5	4.5	5.0	7.0
Handsheet density, g./cc.	0.600	0.608	0.641	0.668	0.693	0.729
Opacity, %	69.4	68.7	66.8	64.1	61.4	57.0
Spec. scattering coeff.[c]	206	203	186	171	153	143
Spec. absorption coeff.[c]	10.7	10.2	8.54	9.04	8.76	10.1
Breaking length, km.	10.1	10.8	11.5	12.4	12.9	13.3
Stretch, %	2.9	3.0	2.6	2.6	2.4	2.5
Tens. energy abs., g.cm./cm.^{2}	120	129	117	129	126	131
Tensile stiffness, kg./cm.	552	583	632	672	730	749
Burst factor	75.6	82.8	84.4	92.2	94.7	92.4
Tear factor	88.0	84.1	78.4	73.8	71.5	65.3
Tear factor x yield/65	88.4	84.5	78.8	74.1	71.8	65.6
In-plane tear, g.cm./cm.	95.2	91.5	82.9	77.3	65.8	65.3
Zero-span breaking length, km.	17.2	17.4	18.6	17.7	18.4	18.5
Zero-span b.l. x yield/65, km.	17.3	17.5	18.7	17.8	18.5	18.6
M.I.T. fold	683	804	904	979	1240	1440

Pulp code	AB-18C					
Pulp yield, %	63.8					
pH unbeaten stock	-					
Beating weight, kg.[a]	4.5					
Beating time, min.	1	3	8	16	25	37
Canadian freeness, ml.	690	670	650	580	470	320
Handsheet drainage time, sec.	4.5	4.3	4.3	4.5	5.0	8.0
Handsheet density, g./cc.	0.621	0.638	0.652	0.688	0.713	0.751
Opacity, %	69.3	67.3	65.6	61.5	58.4	54.8
Spec. scattering coeff.[c]	196	200	186	167	151	132
Spec. absorption coeff.[c]	6.46	7.00	6.63	6.62	6.40	8.53
Breaking length, km.	10.4	11.0	11.5	12.5	12.5	13.1
Stretch, %	3.1	3.0	3.1	2.8	2.7	2.4
Tens. energy abs., g.cm./cm.^{2}	128	130	141	137	131	130
Tensile stiffness, kg./cm.	572	605	613	677	685	732
Burst factor	80.9	80.3	88.6	90.7	95.3	95.3
Tear factor	87.0	77.3	77.2	69.5	67.1	61.9
Tear factor x yield/65	85.4	75.9	75.8	68.2	65.9	60.8
In-plane tear, g.cm./cm.	95.0	94.0	87.2	81.6	73.6	63.6
Zero-span breaking length, km.	17.1	17.3	17.4	18.0	18.7	18.2
Zero-span b.l. x yield/65, km.	16.8	17.0	17.1	17.7	18.4	17.9
M.I.T. fold	771	866	922	1060	1510	1590

Pulp code	AB-19C		
Pulp yield, %	66.5		
pH unbeaten stock	5.2		
Beating weight, kg.[a]	4.5		
Beating time, min.	4[b]	16[b]	20
Canadian freeness, ml.	670	345	180
Handsheet drainage time, sec.	4.5	6.0	11.8
Handsheet density, g./cc.	0.625	0.710	0.733
Opacity, %	--	---	--
Spec. scattering coeff.[c]	188	137	127
Spec. absorption coeff.[c]	8.49	9.33	10.4
Breaking length, km.	11.0	11.7	12.4
Stretch, %	3.1	2.8	2.8
Tens. energy abs., g.cm./cm.^{2}	131	129	134
Tensile stiffness, kg./cm.	445	481	502
Burst factor	83.9	86.7	90.3
Tear factor	76.0	62.9	55.0
Tear factor x yield/65	77.8	64.4	56.3
In-plane tear, g.cm./cm.	84.8	68.0	62.0
Zero-span breaking length, km.	16.4	17.8	17.2
Zero-span b.l. x yield/65, km.	16.8	18.2	17.6
M.I.T. fold	937	1410	1460

[a] Valley beater - weight on end of bedplate lever.

[b] These beating times are not comparable to the other beating times in that only 152-g. pulp was available.

[c] At 650 nm.

TABLE XXI

BLACK SPRUCE – HANDSHEET SMOOTHNESS RESULTS

Pulp	Beating Time, min.	Bendtsen Smoothness, ml./min. (back side of sheet)
AB-7C	1	1080
	3	1000
	8	1080
	12	1440 (?)
	16	1230
AB-15C	1	1590
	4	1260
	8	1190
	15	1030
	27	1060
AB-17C	1	1090
	3	1100
	8	1070
	16	1020
	28	1130
AB-18C	1	1030
	3	989
	8	1040
	16	1030
	25	1060

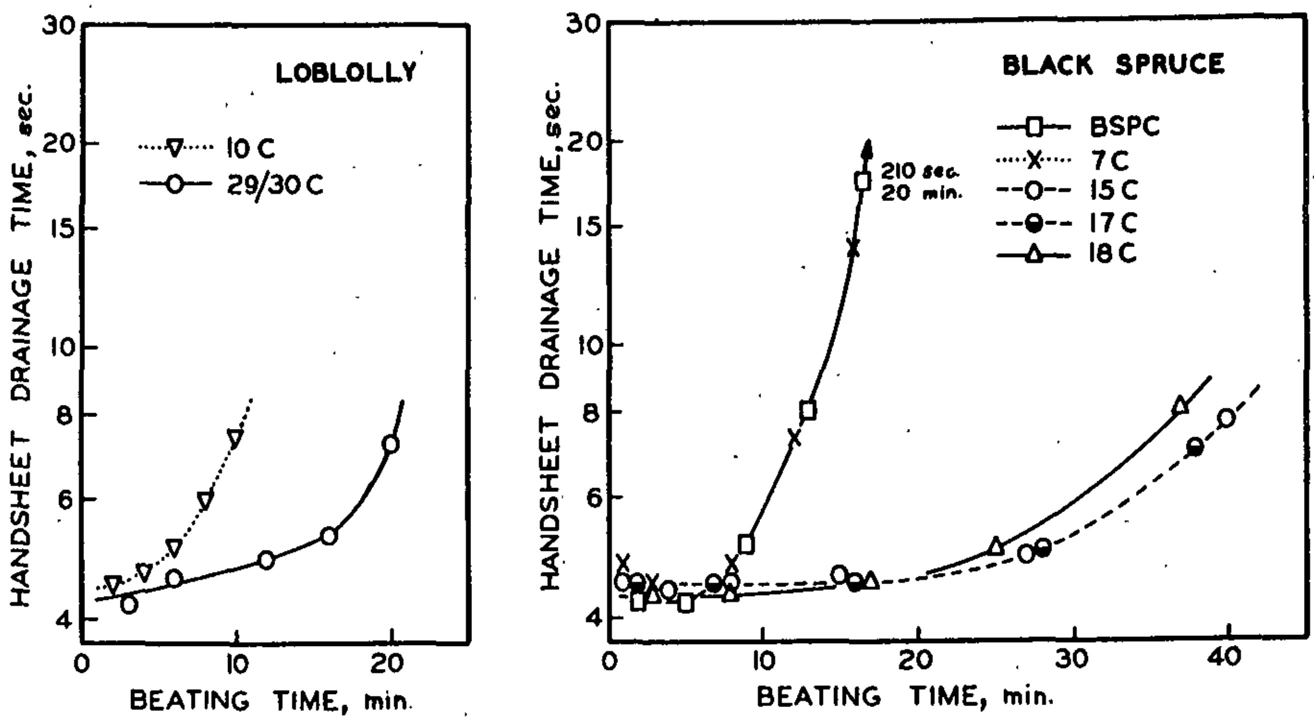

Figure 6. Handsheet Drainage Time vs. Beating Time for Loblolly Pine (A) and Black Spruce Chlorite-Alkali Pulps

The differences relate to steam pressures and/or heating times. When the results for loblolly pine are taken into account, a common factor is fiberization at different steam pressures. More specifically, fiberization at 158°C. instead of 133°C. appears to be a causative factor in giving the improvement discussed above. This conclusion, which applies to softwood chlorite-alkali pulps, ties in with the conclusions reached from investigations on red maple and aspen chlorine dioxide-alkali pulps (2). The question relating to whether the observations made on softwood chlorite-alkali pulps would apply to chlorine dioxide-alkali pulps has not been resolved.

While it is of consequence to know what process factor(s) cause the difference considered in Fig. 6, this falls short of providing an explanation of why the pulps behave differently. Plotting handsheet strength properties vs.

handsheet density revealed no outstanding difference between Pulp AB-7C and Pulps AB-15C, AB-17C, and AB-18C such as might be expected to show up if chemical and/or mechanical degradation were to be the basis for the observation in Fig. 6. There is an apparent difference in initial handsheet density, particularly for Pulp AB-7C compared with Pulp AB-15C. This is maintained during the period of beating which suggests the possible involvement of some phenomenon other than the occurrence of fiber bundles such as indicated for Pulp AB-15C by initial smoothness results (Table XXI). While the nature of the phenomenon being encountered is not clear, it is conceivably related to the degree of plasticization at the time of chip fiberization.

APPENDIX II

ADDENDUM ON ESTIMATED COSTS

TABLE XXII

WOOD COSTS (4)

(Based on a requirement of 3000 lb. o.d. chips for one o.d. ton of pulp.)

(Calculations assume a 15% loss on barking and chipping.)

Aspen Costs

Assume \$18.90 per rough cord delivered.

Assume 85 ft.^{3} solid wood per cord.

Basic density = 0.35.

Need: $\frac{3000 \text{ lb.}}{0.85} \times \frac{1 \text{ cord}}{0.35 \times 62.4 \times 85 \text{ lb.}} = 1.9$ cords/ton o.d. pulp

Cost: 1.9 x \$18.90 = \$36. Aspen = \$36 per ton pulp.

TABLE XXIII

OTHER COSTS (EXCLUDING CAPITAL) (4)

(All values per ton o.d. pulp)

A. Make-up chemicals, etc.

Sodium chloride	\$0.70	
Barium carbonate (sulfate purge)	0.25	
Sodium dichromate	0.40	
Graphite (chlorate cells)	1.50	
Graphite (chlor-alkali cells)	0.72	
Asbestos	0.03	
		\$ 3.60

TABLE XXIII (CONTD.)

OTHER COSTS (EXCLUDING CAPITAL) (4)

(All values per ton o.d. pulp)

B. Utilities (5 mils/kw.-hr. for electrical power
50¢/1000 lb. for steam
$1.50/10⁶ B.t.u. for chilling water)

1. Electrical power:			
Chlorate cells, kw.-hr.	1300		
Chlor-alkali cells, kw.-hr.	580		
Fiberization, kw.-hr.	140		
Miscellaneous, kw.-hr.	400		
Total, kw.-hr.:	2420	=	$12.10
2. Steam:			
Spent liquor evaporation, lb.	5200		
Caustic evaporation, lb.	500		
Fiberization, lb.	500		
Oxidation step, lb.	900		
Extraction step, lb.	400		
Total, lb.:	7500		
Total steam required, lb.	7500		
Less steam generated, lb.	2500		
Steam deficiency, lb.	5000	=	$ 2.50
3. Chilling requirement:			
Recycled liquor, B.t.u.	1,100,000		
Make-up water, B.t.u.	250,000		
Total, B.t.u.:	1,350,000	=	$ 2.00

C. Estimated labor costs (500 tons/day)

Chlor-alkali cell room maintenance	4 men
Chlorate cell and ClO_2 maintenance	4 men
Combined ClO_2 and chlor-alkali operation (4 men/shift)	12 men
Evaporation and combustion operation (3 men/shift)	9 men
Evaporation and combustion maint.	3 men

TABLE XVIII (CONTD.)

OTHER COSTS (EXCLUDING CAPITAL) (4)

(All values per ton o.d. pulp)

Pulping operation (3 men/shift)	9 men
Pulping maintenance	5 men
Total:	46 men

Total man-hr./day = 46 x 8 = 368 man-hr.

Man-hr./ton = 368/500 = 0.74 man-hr./ton

(Hence labor requirement of 1 man-hr./ton is assumed to be reasonable.)

∴ Labor cost of 1 man-hr./ton x $7.50/man-hr. $ 7.50

Total: $27.70

APPENDIX III

GLOSSARY OF TABLES