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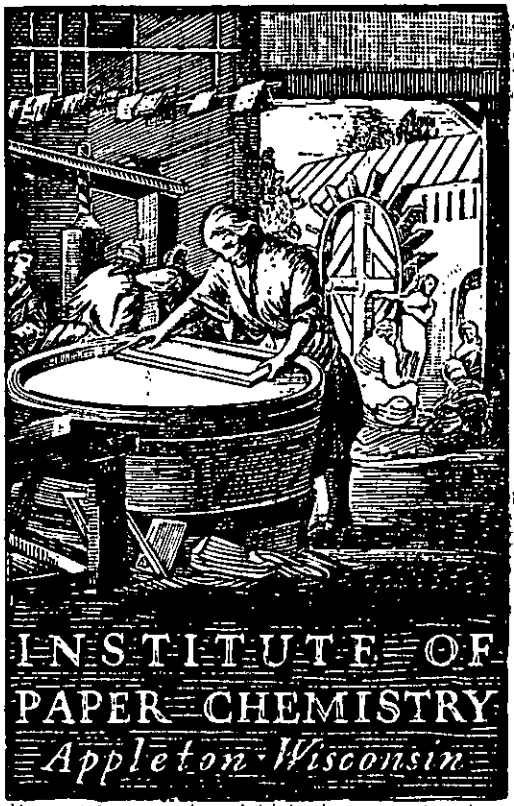

SELECTIVE DELIGNIFICATION OF WOOD AND OTHER FIBROUS MATERIALS

INITIAL STUDIES ON FIBERIZATION OF LOBLOLLY PINE

Research Grant

Project 2500

Report Ten

A Progress Report

to

THE GRANTORS

December 3, 1969

THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

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SUMMARY

Loblolly pine chips have been fiberized by the procedure used previously for aspen. The product was empirically characterized by Bauer-McNett classification which also showed the presence of a significant portion of quite coarse material expected to be a source of potential difficulty in achieving optimum results in chlorine dioxide-alkali pulping.

Delignification of the unfractionated fiberized product was achieved with chlorite-alkali to prepare pulps for obtaining information on changes incurred by fibers as reflected by handsheet properties. Using a chlorite-alkali pulp from "pin" chips as a reference material, it was found that fiberization resulted in a 15-25% lowering of most handsheet strength properties with indications that the fibers had lost part of their inherent strength and probably had been shortened. However, on an equal fiber basis there was apparently better overall realization of inherent fiber strength than found in a kraft reference pulp.

It has been shown through fiber length distribution data for the chlorite-alkali pulps that this fiberization of loblolly chips was accompanied by a reduction in fiber length. In addition, microscopic studies revealed appreciable fibrillation of the fiber walls and other morphological changes including some unwinding of fibers.

Certain aspects of fiberization relevant to what was learned about the fiberized loblolly have been reviewed. The study has been interpreted to mean that the loblolly material was not in a desirable viscoelastic state at the time of fiberization, and it was concluded that to achieve improved results it would be better to use more suitable equipment.

BACKGROUND

In extending the work on the selective delignification of aspen to include softwoods, loblolly pine can be regarded as a species that qualifies especially in view of its commercial importance. Assuming selective delignification of loblolly is approached by a process involving fiberization followed by at least a two-step delignification with chlorine dioxide-alkali, it would be preferable to reduce chips in size without undesirable changes in the papermaking properties of the fibers.

Since the conditions previously described for the fiberization of aspen have appeared promising this seems to be about the best starting basis for loblolly pine.

The initial objective of the work described here was to determine what kind of product could be obtained by applying the previously established fiberizing conditions for aspen to loblolly pine.

DISCUSSION AND RESULTS

LOBLOLLY FIBERIZATION AND GROSS PRODUCT PROPERTIES

Loblolly pine chips were fiberized in a Bauer pulper by a procedure described in the experimental part which is in accord with the above objective. The resultant product was characterized by Bauer-McNett classification data as given in Table I.

TABLE I

BAUER-McNETT CLASSIFICATIONS OF THE FIBERIZED LOBLOLLY CHIPS[a]

Screen	Drum 1, %	Drum 2, %	Drum 3[b], %
On 6 mesh	12.2	10.2	16.0
On 12 mesh	21.2	20.7	23.0
On 35 mesh	40.9	45.4	37.2
On 65 mesh	9.0	8.2	7.4
On sintered-glass filter[c]	13.2	(15.5)[d]	13.4
Water soluble (by difference)	3.5		3.0
Water temperature, °C.	3.5	3.5	3.5

[a] Chips: moisture content 114.4% o.d. basis; chip density 30.0 lb. o.d./ft.^{3} green; bulk density 10.4 lb. o.d./ft.^{3}.
Fiberized chips: moisture content 50% o.d. basis.

[b] The infeed throat of the Bauer refiner was full of chips during most of the time this drum was collecting the fiberized material. A higher feed rate was experienced with the ammeter indicating a 500-amp. load. Consequently, a slightly higher proportion of coarser material was to be expected. Normally, the chips are fed manually onto the spiked feed roll.

[c] The outflow from the 65-mesh screen was collected in a drum and was subsequently filtered on a 3-liter sintered-glass funnel.

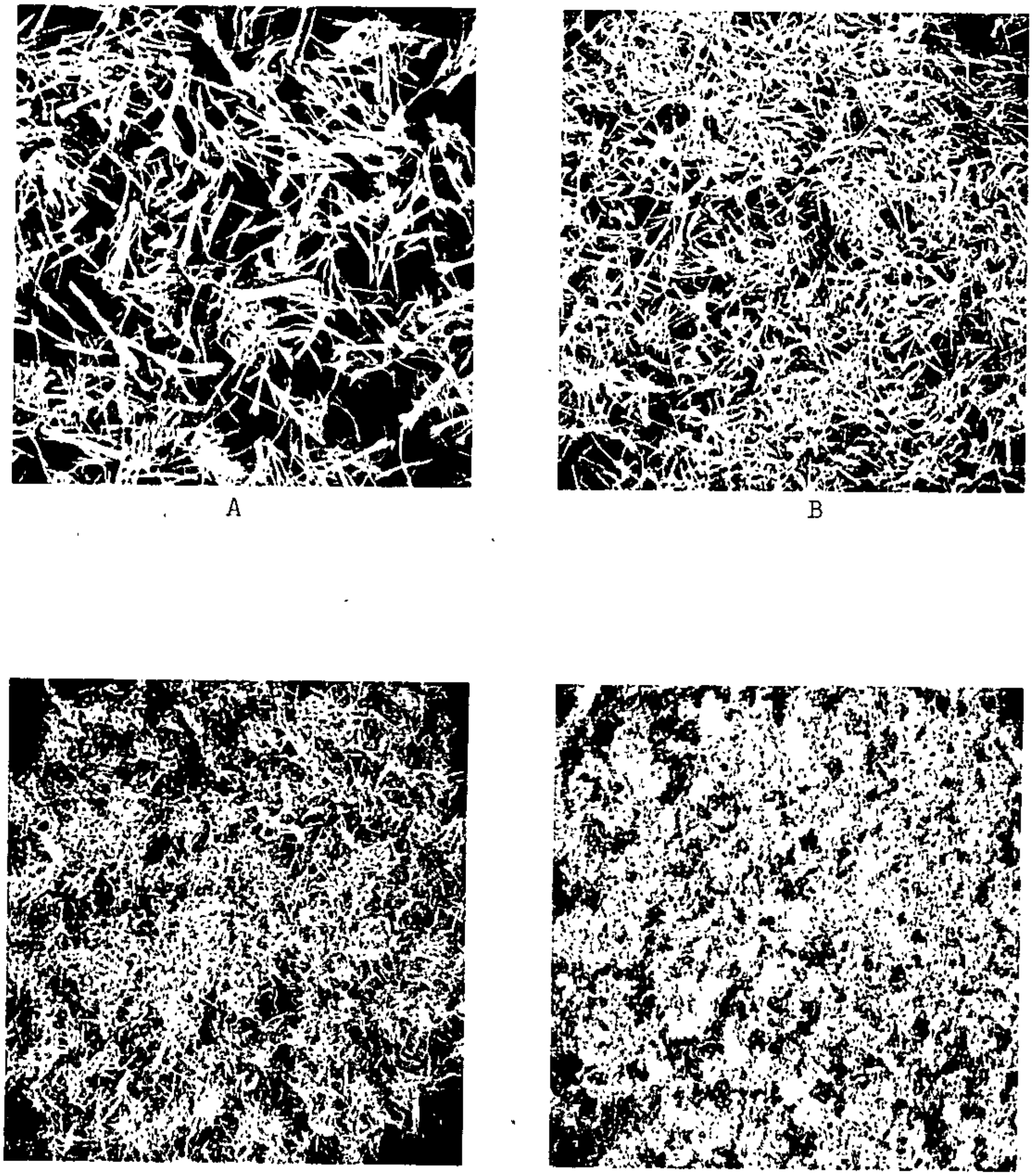
[d] Through 65-mesh screen by difference.

Although Table I provides data that can be useful empirical classifications of the fiberized materials, such data leave much to be desired in knowing what kind of potential a whole product has for making a selectively delignified papermaking pulp. A little better idea may be obtained by looking at the fractions, some samples of which are shown in actual size in Fig. 1. From this it will be obvious that the on-65-mesh sample is considerably finer than the on-6-mesh sample. Furthermore, the latter has a significant portion of quite coarse material. For minimizing chlorine dioxide consumption while achieving uniform lignin modification and minimum screen rejects it appears a fiberized material should be as uniform as possible. On the basis of previous experience where fiberized chips have been reacted with chlorine dioxide, the coarsest material in Fig. 1 would be expected to be a potential source of screen rejects.

Thus, Fig. 1 provides an indication that the coarseness of the on-6-mesh part of the fiberized loblolly chips under consideration could be a source of potential difficulty in achieving good results in chlorine dioxide-alkali pulping.

Now it could be argued that since this is a reflection of mechanical factors it would be possible to eliminate these by excluding the on-6-mesh fraction. This was considered for a time and subsequently abandoned. Apart from any other reasons, such an approach would leave some doubt over the extent to which the proportion of springwood and summerwood fibers may have been changed. Since these two types of softwood fibers have marked differences in properties that are reflected in paper handsheet properties, doubt over interpretation of the latter should obviously accompany uncertainty about the former.

To obtain a better indication of the papermaking potential of this fiberized loblolly than is possible from Table I and Fig. 1, the unfractionated material was considered as described on page 7.



A

B

C

D

Figure 1. Bauer-McNett Fractions of Fiberized Loblolly, Actual Size. A, B, C, and D Represent On-6, On-12, On-35, and On-65-Mesh Fractions, Respectively

CHLORITE DELIGNIFICATION AND PULP EVALUATION

In previous work on aspen, covered in Report Six, some indication of the absence of fiber damage was obtained by comparing data from handsheets of chlorite-alkali delignified pulps prepared from both fiberized chips and "pin" chips. The data for aspen indicated that chip fiberization apparently was achieved without detriment to paper properties such as breaking length, burst factor, and Elmendorf tear, although zero-span tensile strength showed a decrease of about 5%.

In further consideration of the fiberized loblolly obtained as in this report, a similar approach has been included. For this purpose, chlorite-alkali pulps were made using the procedure described in the experimental part. The coded identity of the pulps, their yields and data on their preparation are included in Table II. In accordance with previous experience, the oxidation time required was longer for the "pin" chips which are appreciably larger than the fiberized chips. A second pulp, namely SP-3, was made from fiberized chips to achieve a yield after chlorite oxidation more comparable to that for "pin" chips than was obtained at first. It is noted that the yield of the chlorite pulp (viz. SP-1) from the "pin" chips is between the yields obtained for the two pulps from fiberized chips.

TABLE II

CHLORITE-DELIGNIFIED PULPS

<table>
<tr><td>Chips</td><td>"Pin"</td><td colspan="2">Fiberized</td></tr>
<tr><td>Code</td><td>SP-1</td><td>SP-2</td><td>SP-3</td></tr>
<tr><td>Alkali conditioning</td><td></td><td></td><td></td></tr>
<tr><td>NaOH, %[a]</td><td colspan="3">10</td></tr>
<tr><td>Consistency, %</td><td colspan="3">10</td></tr>
<tr><td>Temperature, °C.</td><td colspan="3">50</td></tr>
<tr><td>Time, min.</td><td colspan="3">60</td></tr>
<tr><td>Initial pH</td><td colspan="3">>13</td></tr>
<tr><td>Final pH</td><td colspan="3">12.8</td></tr>
<tr><td>Yield, %</td><td>--</td><td>--</td><td>96.6</td></tr>
<tr><td>Chlorite oxidation</td><td></td><td></td><td></td></tr>
<tr><td>$NaClO_2$, %</td><td colspan="3">100</td></tr>
<tr><td>HAc, %</td><td colspan="3">15</td></tr>
<tr><td>Consistency, %</td><td colspan="3">10</td></tr>
<tr><td>Temperature, °C.</td><td colspan="3">25/40</td></tr>
<tr><td>Time, hr.</td><td>72</td><td>20</td><td>25</td></tr>
<tr><td>Initial pH</td><td colspan="3">4.4</td></tr>
<tr><td>Final pH</td><td>4.1</td><td>4.5</td><td>4.8</td></tr>
<tr><td>Yield, %</td><td>72.3</td><td>81.4</td><td>73.5</td></tr>
<tr><td>Alkali extraction</td><td></td><td></td><td></td></tr>
<tr><td>NaOH, %</td><td colspan="3">5</td></tr>
<tr><td>Consistency, %</td><td colspan="3">10</td></tr>
<tr><td>Temperature, °C.</td><td colspan="3">25</td></tr>
<tr><td>Time, min.</td><td colspan="3">60</td></tr>
<tr><td>Initial pH</td><td colspan="3">12.9</td></tr>
<tr><td>Final pH</td><td colspan="3">12.6</td></tr>
<tr><td>Yield, %</td><td>65.3</td><td>68.7</td><td>63.5</td></tr>
</table>

[a] All percentages based on original o.d. fiberized chips.

The pulps obtained as in Table II plus a kraft reference pulp prepared as described in the experimental part were beaten, then made into handsheets which were tested. Complete data are given in Table III. From this it can be seen how quickly the chlorite pulps beat down to a 250-ml. freeness which had been selected as a guide value to discontinue beating. Subsequent work to be reported later on red maple has indicated this can be an unsatisfactory approach since at least with some freeness data for chlorite pulps it is very difficult to know what meaning, if any, freeness can be anticipated to have.

Unlike what was found in the case of aspen, the handsheet properties in Table III reveal loblolly chip fiberization when carried out according to the procedure described here was accompanied by changes to the fibers that reflect unfavorably on some paper properties. This is illustrated for convenience in Fig. 2-6. After fiberization the handsheet properties of zero-span breaking length, breaking length, tensile energy absorption, tensile stiffness, Elmendorf tear, in-plane tear, and burst factor are about 15-25% below those obtained for pulp from "pin" chips. Stretch and M.I.T. fold data are exceptional in not showing this reduction.

Before considering something of the nature of the changes incurred by these fiberized loblolly fibers, certain specific aspects of the handsheet properties are worthy of note. For example, if zero-span breaking length as in Fig. 2 is taken as a guide to the strength of fibers, those in Pulps SP-2 and SP-3 from fiberized loblolly appear significantly weaker than those in the Pulp SP-1 from "pin" chips. Since yield differences which reflect the number of fibers in a given weight of handsheet do not account for the observed difference, then presumably the fibers have lost part of their inherent strength at one or more points

TABLE III

PRIMARY HANDSHEET DATA

Pulp	Chlorite												Kraft						
Starting material	"pin" chips				fiberized chips								screened chips						
Yield, %	65.3				68.7				63.5				45.6						
Code	SP1				SP2				SP3				SPK						
Canadian freeness, ml.[a]	550	410	340	260	660	470	330	270	575	470	340	250	730	690	690	650	570	410	265
Beating time, min.[b]	7	10	12	14	10	20	25	30	10	14	18	23	5	10	15	20	30	40	50
Density, g./cc.	0.551	0.561	0.578	0.576	0.552	0.597	0.596	0.606	0.594	0.606	0.614	0.625	0.512	0.547	0.580	0.590	0.617	0.642	0.655
Spec. scattering coeff. (650 mμ.)	133	132	127	130	167	145	133	147	141	131	130	115	209	-	182	-	162	-	144
Breaking length, km.	8.66	9.15	8.89	9.28	7.23	7.88	7.71	8.96	7.56	8.45	8.61	9.45	6.82	7.92	7.92	9.55	10.0	11.2	11.9
Stretch, %	2.0	2.0	2.0	2.1	2.0	2.1	1.8	2.2	2.3	2.4	2.3	2.3	1.7	2.0	2.0	2.4	2.2	2.9	2.6
Tensile energy absorption,[c] g. cm./cm.^{2}	67.4	71.6	69.6	77.8	57.9	66.6	54.8	79.0	71.7	85.1	82.3	87.7	47.5	64.3	60.5	91.3	87.6	127	119
Tensile stiffness, Et, kg./cm.[c]	600	634	607	614	481	535	559	574	526	574	562	614	517	526	518	593	583	617	643
Burst factor	43.9	54.2	53.6	52.3	36.7	41.8	43.1	47.7	44.3	48.9	51.7	53.9	43.0	52.8	62.7	66.2	71.4	81.8	75.0
Tear factor (Elmendorf)	130	123	119	114	92.1	79.7	76.7	76.4	88.9	83.3	79.2	76.7	230	194	174	172	155	143	139
Tear factor x yield/65	131	124	120	115	97.3	84.2	81.1	80.8	86.9	81.4	77.4	74.9	161	136	122	121	109	100	97.5
In-plane tear, g. cm./cm.[c]	112	115	113	113	78.2	72.8	73.5	68.9	83.4	81.5	79.4	75.4	119	131	128	150	143	159	137
Zero-span breaking length, km.	18.7	18.4	18.2	18.8	14.4	15.7	15.3	15.5	16.0	15.1	16.0	16.6	19.8	20.7	19.9	21.1	21.5	21.4	20.9
Zero-span breaking length x yield/65	18.8	18.5	18.3	18.9	15.2	16.6	16.2	16.4	15.6	14.8	15.6	16.2	13.9	14.5	14.0	14.8	15.1	15.0	14.7
M.I.T. fold[c]	239	248	242	300	154	209	274	291	215	252	329	429	672	868	965	1017	1230	1590	1740

[a] Unbeaten freenesses: SP1 = 800 ml.; SP2 = 790 ml., SP3 = 680 ml., and SPK = 740 ml.

[b] Bedplate load for all chlorite pulps = 4.5 kg.; bedplate load for the kraft pulp = 5.5 kg.

[c] Corrected to 60.0 g./m.^{2} basis weight by simple proportion.

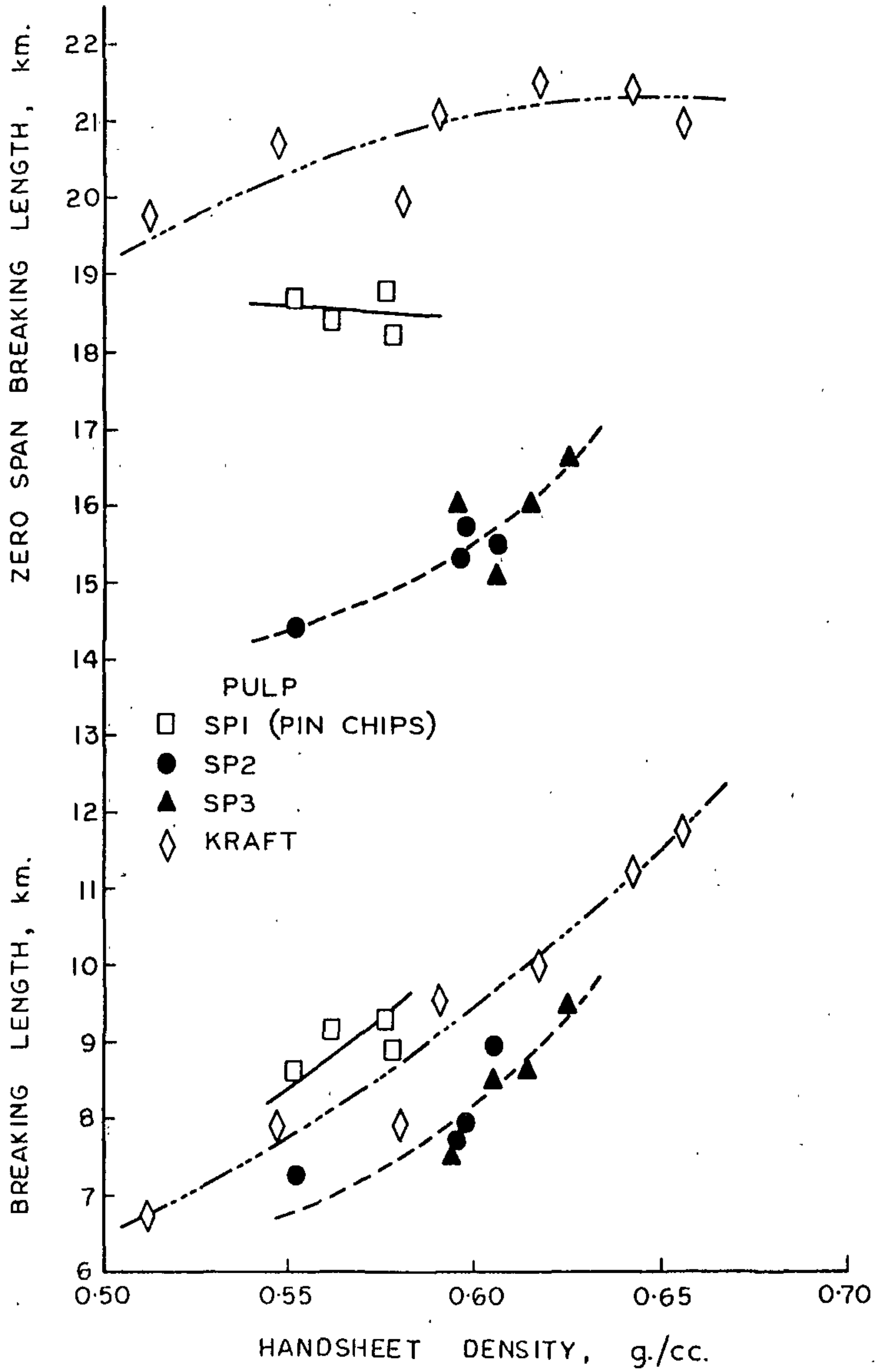


Figure 2. Effect of Handsheet Density on Zero-Span Strength and Breaking Length

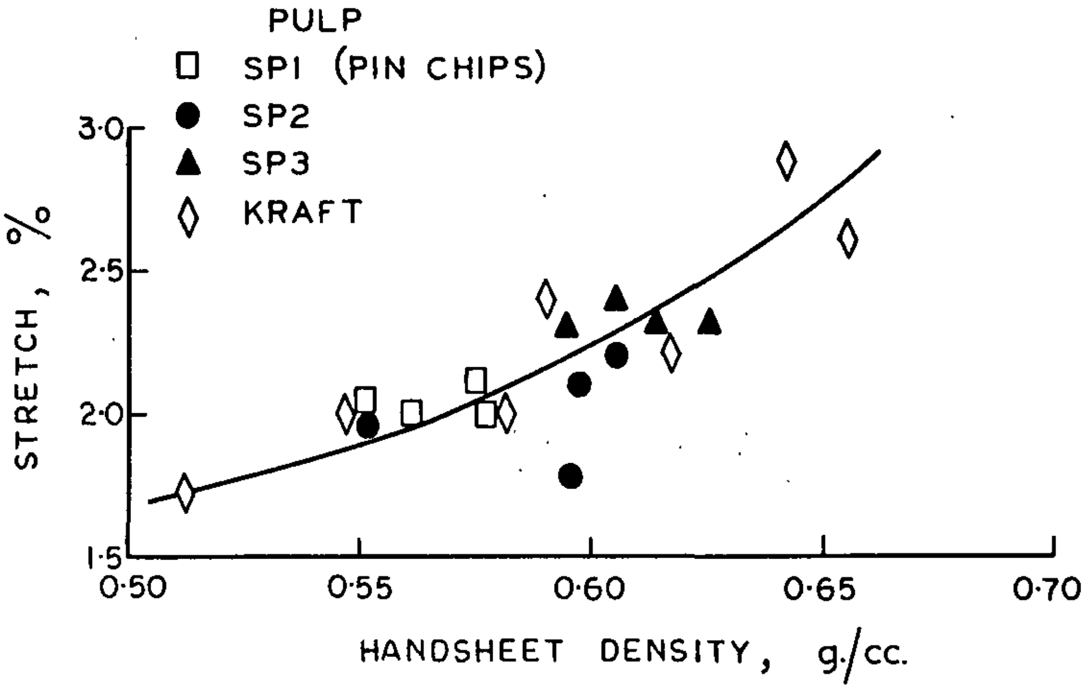


Figure 3. Effect of Handsheet Density on Stretch

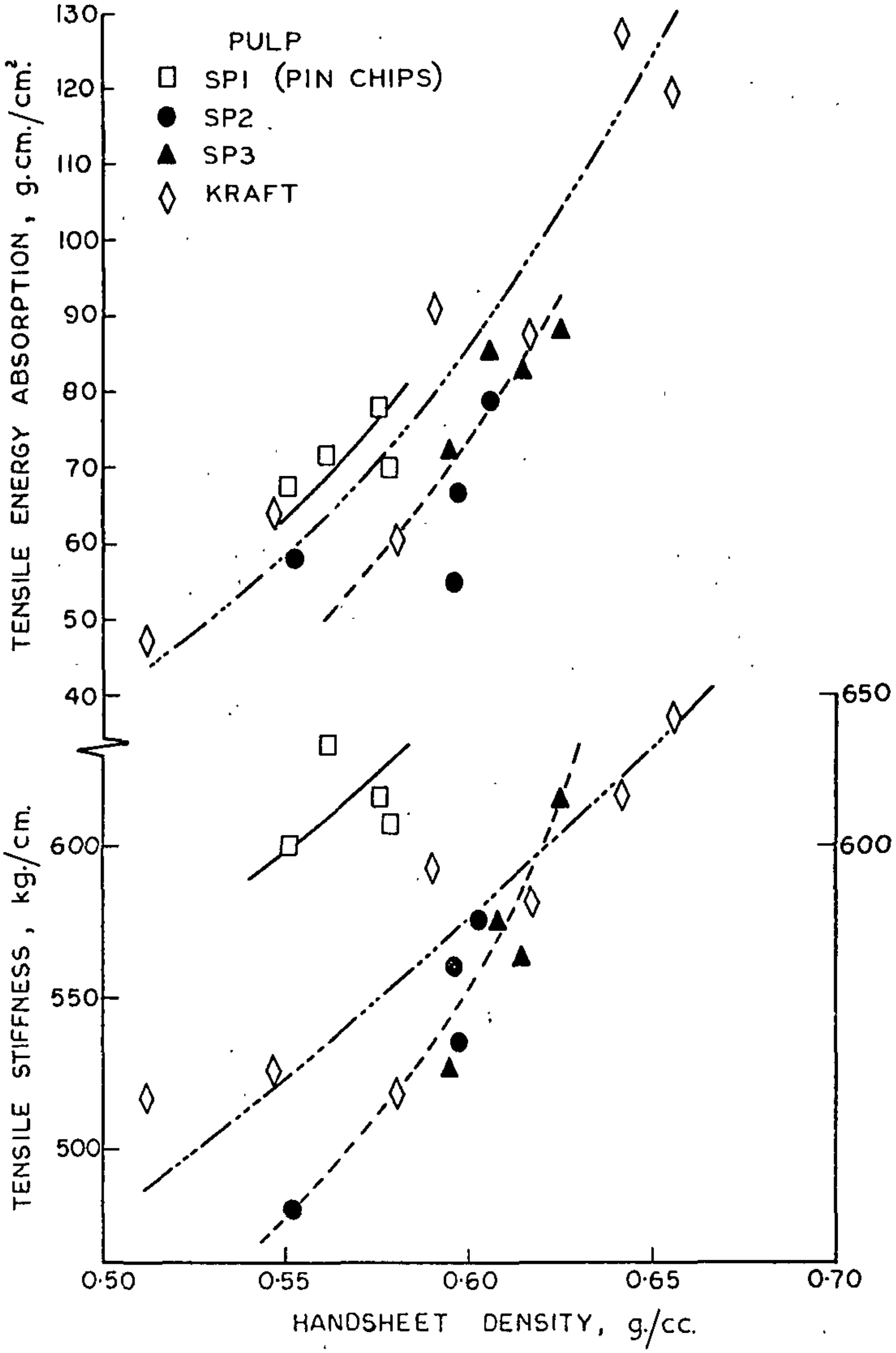


Figure 4. Effect of Handsheet Density on Tensile Energy Absorption and Tensile Stiffness

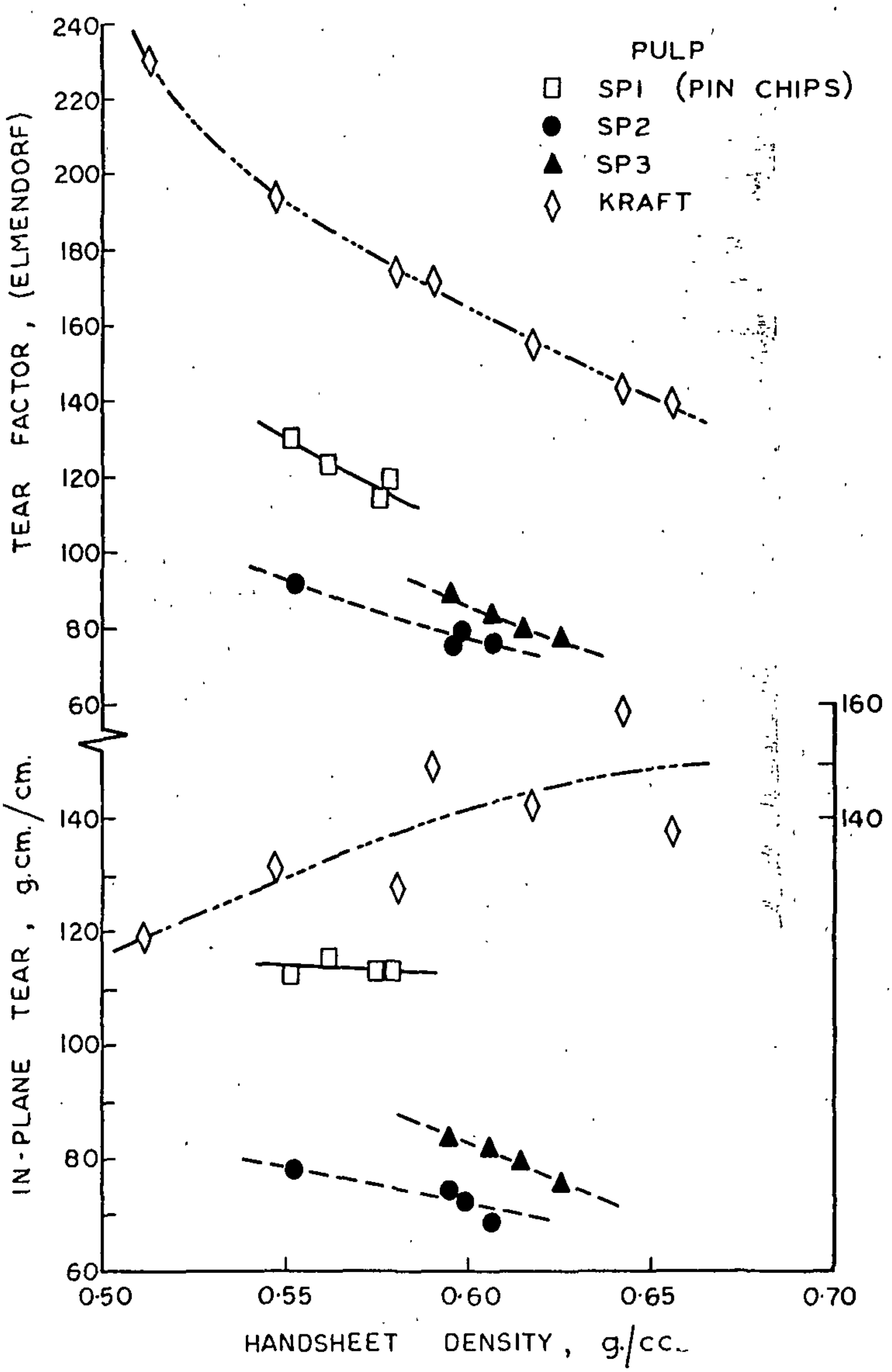


Figure 5. Effect of Handsheet Density on Tear Factor and In-Plane Tear

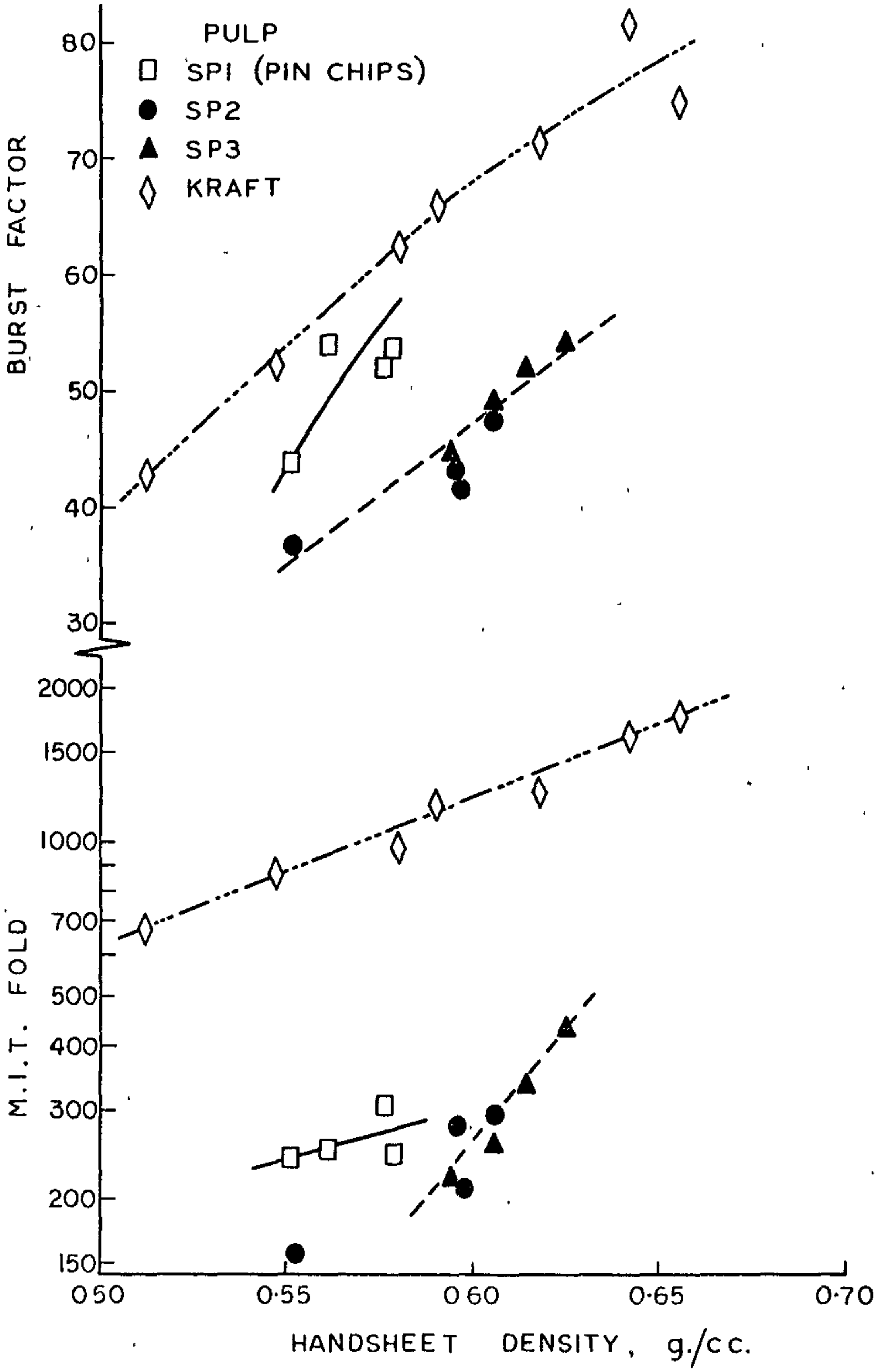


Figure 6. Effect of Handsheet Density on Burst Factor and M.I.T. Fold

in their preparation. This is supported by other handsheet properties such as breaking length data which are also represented in Fig. 2.

Included in Fig. 5 are the relationships of handsheet density versus Elmendorf tear factor, which for a long-fibered pulp from the same wood can reflect changes in fiber length. The curves related to Pulps SP-2 and SP-3 compared with SP-1 indicate the possibility of fiber shortening in the two pulps prepared from loblolly pine fiberized as described above.

Aside from the above comments on Fig. 2 and 6, it will be noted from Table III that, when compared on an equal fiber basis, handsheets from all three chlorite pulps have higher zero-span breaking lengths than the kraft pulp handsheets, indicating better realization of inherent fiber strength. It is also interesting to see from Table III that, when compared on an equal fiber basis, tear factors related to Pulp SP-1 from "pin" chips approximate those for the kraft pulp handsheets. In addition, for the cases of breaking length, tensile energy absorption, and tensile stiffness versus handsheet density as in Fig. 2 and 3, the curves related to the chlorite-alkali Pulp SP-1 from pin chips fell above those for the kraft pulp handsheets.

FURTHER EXAMINATION OF PULPS

Since there is some reason to believe from the foregoing that the procedure used to fiberize loblolly leaves room for improvement, this raises the question of how the procedure should be modified to obtain better results. To consider this question more ably, Pulps SP-1 and SP-3 were examined further.

Bauer-McNett classifications along with the average fiber lengths data were obtained as recorded in Table IV. From the table it is apparent this shot at fiberization of loblolly chips was accompanied by a reduction in fiber length, as confirmed by obtaining fiber length distribution data for whole pulp samples as presented in Fig. 7.

TABLE IV

BAUER-McNETT CLASSIFICATIONS AND FIBER LENGTH ANALYSIS OF THE CHLORITE PULPS

Pulp	SP-1		SP-3	
Starting material	pin chips		fiberized chips	
Yield, %	65.3		63.5	
	Retained, %	Av. Fiber Length, mm.	Retained, %	Av. Fiber Length, mm.
On 12 mesh	43.0	3.06	34.2	2.82
On 20 mesh	19.0	2.59	17.2	2.22
On 65 mesh	33.6	1.78	36.2	1.40
On 100 mesh	1.6	0.79	2.6	0.62
Through 100 mesh by difference	2.8	--	9.8	--
Water temperature, °C.	3.5		3.5	

Average fiber length determined by conventional projection/individual measurement technique.

To determine what other changes could be detected, samples of the fractions from the Bauer-McNett classifications were looked at more closely through microscopic examination. The two on-12-mesh fractions for Pulps SP-1 and SP-3, respectively, are illustrated in Fig. 8 and 9. If these are compared, the most obvious difference is the fibrillation occurring on the fibers Ex-Pulp SP-3 from

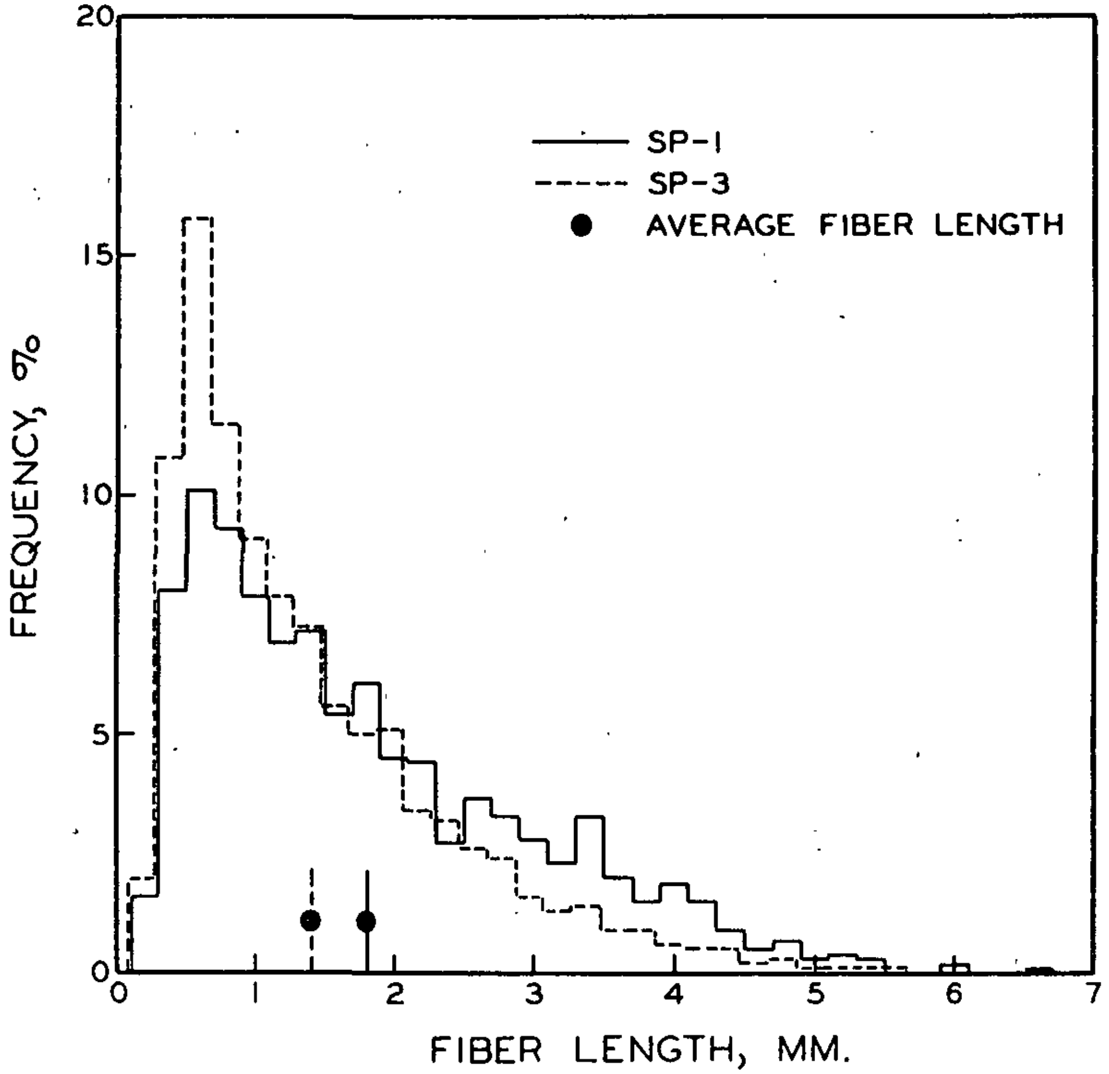


Figure 7. Fiber Length Distribution of Loblolly Pulps SP-1 and SP-3 from "Pin" Chips and Fiberized Material, Respectively

Figure 8. Material from the On-12-Mesh Fraction of Pulp SP-1.
Dry Mount, Unstained Sample, Magnification 35X

Figure 9. Material from the On-12-Mesh Fraction of Pulp SP-3 Showing Some Evidence of Broken Fibers and Fibrillation. Dry Mount, Unstained Sample, Magnification 35X

fiberized loblolly. A corresponding difference could be observed in each analogous pair of pulp fractions from the Bauer-McNett classifications covered in Table IV.

More careful consideration of the photomicrographs revealed additional differences that are illustrated to some degree by comparison of Fig. 10 and 11. In addition to showing fibrillation on the fibers Ex-Pulp SP-3, Fig. 11 includes a fine example of fiber end-brooming which might be expected to occur from the macroscopic appearance on the on-6-mesh fraction in Fig. 1.

In Fig. 11, to the right center about halfway below the end-broomed fiber, there is a fiber that appears to have been unwound. This may seem to be hardly worthy of comment. However, in the material from the on-100-mesh fraction of Pulps SP-1 and SP-3 depicted in Fig. 12 and 13, respectively, a greater number of unwound fiber fragments can be discerned in the latter. Furthermore, these appear to have been derived from relatively thin-walled fibers.

The morphological outcome of the procedure described here to fiberize loblolly thus has been found to include a shift in fiber length distribution resulting from shortening of fibers, appreciable fibrillation of the fiber walls, some end-brooming, and noticeable unwinding of fibers.

INTERPRETATION OF STUDY

To understand the possible meaning of the observations made in this study, certain aspects of fiberization warrant consideration.

It is envisaged that in chip fiberization the initial phases of mechanical breakdown essentially follow those described by Atack and May (1). First, the chips are broken along the wood grain into matchstick fragments that are progressively reduced in size to fiber bundles, then to unrefined fibers, but further

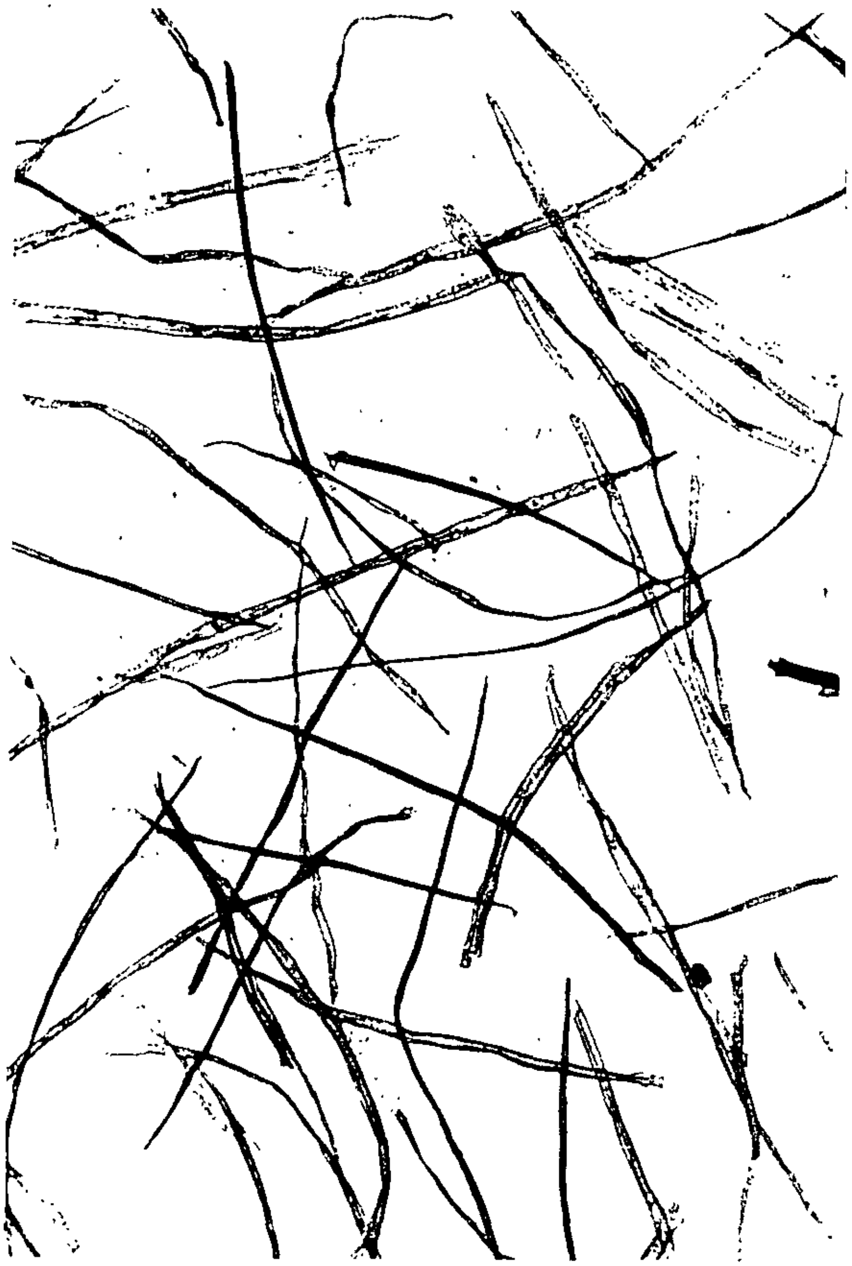


Figure 10. Material from the On-65-Mesh Fraction of Pulp SP-1.
Dry Mount, Unstained Sample, Magnification 35X

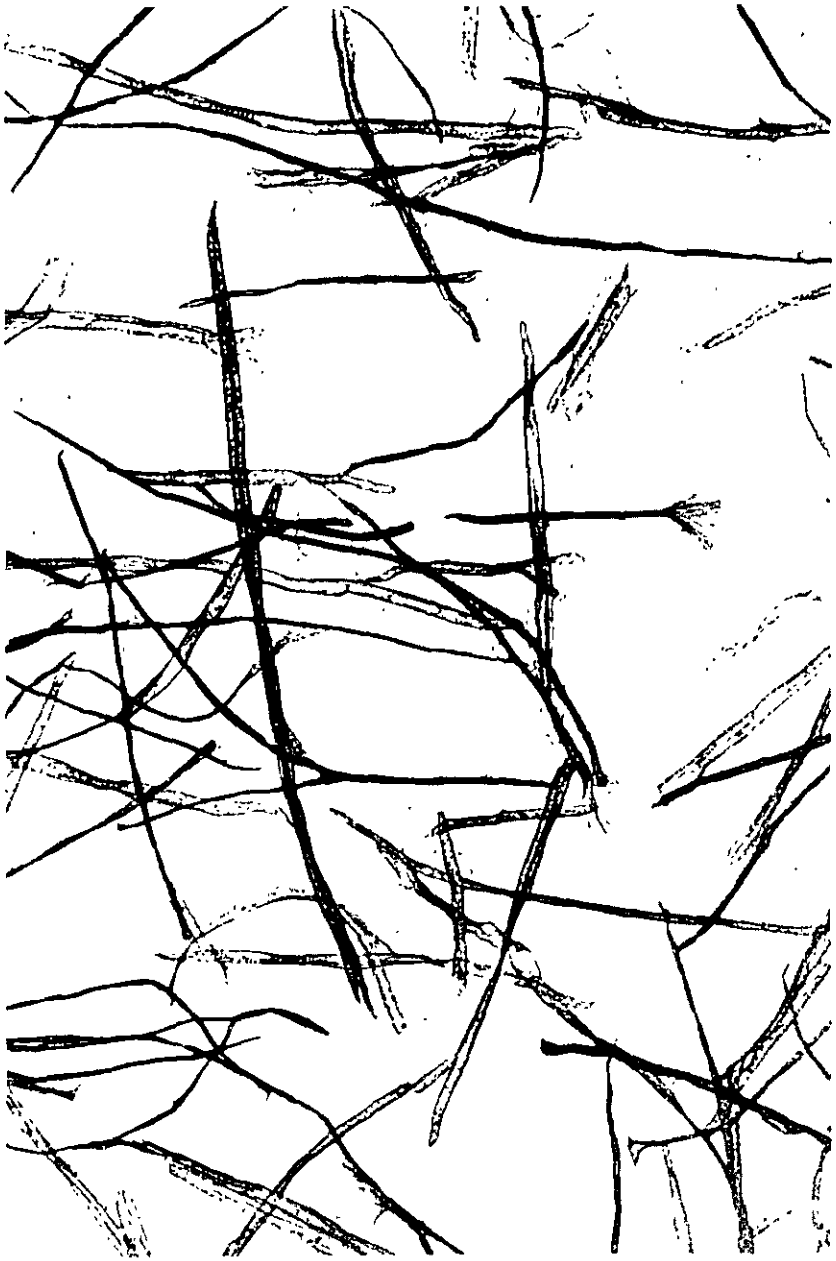


Figure 11. Material from the On-65-Mesh Fraction of Pulp SP-3 Showing Evidence of Broken Fibers, Broomed Fiber Ends, and Fibrillation.
Dry Mount, Unstained Sample, Magnification 35x

Figure 12. Material from the On-100-Mesh Fraction of Pulp SP-1 Showing Little or No Fibrillation of Fiber Walls and Significantly Less Unwinding of Fibers than in Fig. 13. Dry Mount, Unstained Sample, Magnification 35x

Figure 13. Material from the On-100-Mesh Fraction of Pulp SP-3 Showing Evidence of Broken Fibers, Fibrillation of Fiber Walls, and Unwinding. Dry Mount, Unstained Sample, Magnification 35X

refining into a papermaking pulp would defeat the objective of retaining inherent fiber strength and is not wanted. This is a significant distinction between the concept of fiberization and double-disk breakdown to a refiner mechanical pulp for papermaking as an alternative to stone-ground mechanical pulp for papermaking.

During the breakdown of chips into fiber bundles and unrefined fibers during fiberization, the material is conceived as axially aligned cylinders moving in a radial direction. By some extension of the thoughts of McMillin on tracheids (2), these cylinders can be regarded as stressed in torsion. The torsional stress about the axial direction can be resolved into shearing, diagonal tensile, and compressive stresses. With this in mind it is possible to consider not only shear stress *vs.* diameter relationships for a given torsional moment, but also other effects on fibers that can be related to the state of the material when stressed in torsion.

Thus, for example, if fiber bundles are in a viscoelastic rather than inelastic state, when stressed in torsion, middle lamella shear might be expected to occur with a minimum of wall fibrillation or end-brooming of fibers. When middle lamella shear does not occur with relative ease, there can occur within-fiber failures such as buckling caused by compressive stresses or interfibril cracking with unwinding of long fibers as caused by shear stresses.

The observations made from studying the fiberized loblolly as described here are interpreted to mean that the material was not in a desirable viscoelastic state at the time of fiberization. It is concluded that to achieve improved results it would be preferable to use equipment mechanically better suited for the control of chip temperature and moisture content which are known to be positive factors in lignin plasticization. Further work that has now begun has involved the use of such alternative equipment.

EXPERIMENTAL

RAW MATERIALS

The wood sample kindly supplied by one of the Grantors consisted of twelve sticks of loblolly pine (Pinus taeda L.) with underbark diameters at the large end 6.8-7.0 in.

A supply of chips was obtained by taking half of each stick, trimming off the end, removing the bark by hand, and then chipping in the Institute's 4-knife, 38-inch Carthage chipper. The chips were then screened through a 24-inch Sweco Dynoscreen with the through-1-inch and on-4-mesh fraction being retained. After sampling for moisture content, chip density, and bulk density, the screened chips were stored outside in two plastic-lined drums. The remaining half sticks had their fresh-cut ends waxed and were then stored outside.

A quantity of "pin" chips, nominally 3/32 x 3/32 inch in cross section, was prepared by manually splitting screened chips in the longitudinal direction.

FIBERIZATION AND CLASSIFICATION

About 34 lb. (ovendry basis) of the screened loblolly chips were fiberized in a no. 185 Bauer double-disk refiner fitted with B 957 plates using the same conditions as for aspen chips. These conditions were as follows: The chips were steamed twice at 15 p.s.i. for a total time of 4 min. and then soaked in cold water for 30 min. under 100 p.s.i. of nitrogen pressure; moisture content 173% (o.d. basis). They were then steamed at 80 p.s.i. for 3 min. and fed manually at atmospheric pressure (moisture content 108%) into the throat of the Bauer refiner. The plate clearance was 0.025 in.; the number one setting on the Reeves drive was used. The

Bauer refiner was preheated with steam prior to use and steam was also introduced between the plates during fiberization.

The fiberized material was classified using a Bauer-McNett classifier and the percentage of cold water solubles determined (see Table I).

CHLORITE DELIGNIFICATIONS AND PULP CLASSIFICATIONS

The chlorite delignifications on the fiberized chips and the "pin" chips were carried out in three stages as follows:

Alkali conditioning was carried out in each case in plastic bags with the pin chips (equivalent to 800 g. ovendry) covered with 8 liters of 0.25N NaOH; then a vacuum was applied for 15 min. at room temperature before heating to 50°C. in 45 min. for a further 60 min., after which the extracted product was collected on a sintered-glass filter, washed with water several times, soaked in distilled water over a weekend, filtered, covered with 8 liters of 4.5% acetic acid for 2 hours, filtered and washed. For the fiberized chips, the vacuum treatment was omitted.

Chlorite reactions were conducted by adding a volume of solution containing 10% sodium chlorite and 1.5% acetic acid to provide 100% $NaClO_2$ on an original fiberized chip basis. Each shaken reaction mixture was left at room temperature for the times recorded in Table II. The temperature of the reaction mixture rose to 40-45°C. during the first hours of oxidation. The products were collected on a sintered-glass filter and washed with several changes of water. Yield determinations were made at this point.

Alkali extractions were carried out at 25°C. for 60 min. using a volume of 0.125N NaOH providing 5% NaOH on an original fiberized chip basis. The products were collected on a sintered-glass filter, washed, covered with 5% acetic [illegible] ([illegible] hours for the "pin" chips and 30 min. for the fiberized material), filtered, and washed.

Bauer-McNett classifications and fiber length measurements were carried out to give data as in Table IV, and photomicrographs of selected Bauer-McNett fractions are presented in Fig. 8-13.

PREPARATION OF KRAFT REFERENCE PULP

One charge of screened loblolly pine chips was cooked in a stainless steel 2-cu. ft. digester fitted with external liquor circulation and a steam heat exchanger. The conditions used are given in Table V. The chips were in a [illegible] basket, and at the end of the cook the black liquor was released through the [illegible] valve and the contents of the digester cooled down with water. The contents [illegible] were given two washes in a 60-gal. tank fitted with a paddle stirrer, later [illegible] batchwise with a Williams disintegrator, and then screened using a 12-cut ([illegible] slots) pulsating flatbed screen. The pulp was dewatered in a centrifuge [illegible] up in a wet pulp shredder, sampled for moisture content and Kappa number, and stored at 5°C.

TABLE V

KRAFT PULPING OF LOBLOLLY PINE

Digester chip charge, g. (ovendry)	57[illegible]
Active alkali, Na_2O basis, %	[illegible]
Sulfidity, Na_2O basis, %	[illegible]
Liquor-to-wood ratio	[illegible]
80-180°C., linear with relief at 105°C., min.	[illegible]
At 180°C. (max. temp.), min.	[illegible]
180°C. to black liquor blow at 100 p.s.i., min.	[illegible]
Screened yield on o.d. wood, %	45.[illegible]
Screen rejects on o.d. wood, %	0.[illegible]
Kappa number	30.[illegible]

PULP EVALUATION

The pulps were beaten in a 1-1/2 lb. Valley beater according to TAPPI Standard T 200 ts-66 with the exception that 1600-ml. samples were withdrawn at each beating interval in order to form ten 60-g./m.^{2} and one 50-g./m.^{2} handsheets, and that for the three chlorite pulps the bedplate weight was 4.5 kg. instead of the standard 5.5-kg. weight. Freeness determinations, handsheet forming, and handsheet testing were all carried out according to the appropriate TAPPI Standard Methods. Tensile tests were performed on a table-model Instron with a test span of 4.0 in., a test-piece width of 1.0 in., and a cross-head speed of 1 in./min. Tensile stiffness was calculated for the initial slope of the load *vs.* elongation curve of the tensile energy absorption tests. The in-plane tear tests were performed as described by Van den Akker, *et al.*(*3*). The 50-g./m.^{2} handsheets were used for the zero-span tests. Data are given in Table III.

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