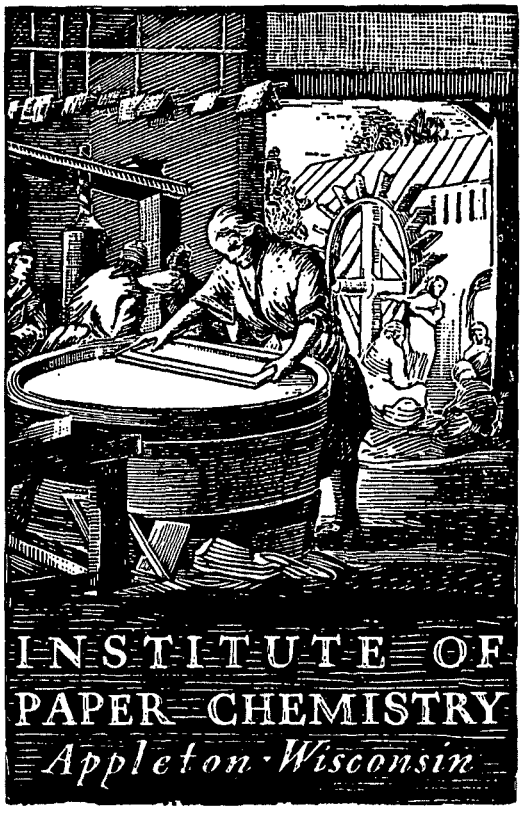

OXIDATIVE DELIGNIFICATION WITH OXYGEN/ALKALI TO HIGH-YIELD PULPS

PULPING OF SOFTWOODS—LOBLOLLY PINE

Project 3264

Report Two
A Progress Report
to
MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY

January 19, 1978

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

Loblolly pine was pulped with oxygen and sodium bicarbonate and/or carbonate to give pulps of higher yields than corresponding kraft pulps. Two different pulping systems were used and compared. These were high consistency (ca. 25%) with oxygen, and low consistency with circulation of liquor, using air at pressures up to 2550 psi. The high-consistency work was done entirely with fiberized wood, whereas wood in a number of forms was used in low-consistency pulping.

Mechanical damage during fiberization was examined by electron microscopy and swelling studies. Damage was considered to be primarily compression, rather than cutting. This was partially responsible for reduced strength properties.

Strength properties were determined for a number of pulps and compared with those from corresponding kraft reference pulps. In general, the strength properties were lower, particularly tear and folding strengths. The low consistency pulps were better than the high consistency pulps.

The pulps were found to bleach reasonably well with a CEDED sequence. Brightness reversion was not serious, and chemical consumption was only a little higher than typical kraft bleaching.

OBJECTIVE

As stated previously (1), the main objective of this project is to study the effect of pulping parameters on oxygen/alkali delignification for preparing high-yield papermaking pulps from softwoods and hardwoods. The goal is to provide papermaking pulps with unbleached yields greater than 65% and bleached pulps of 55% or greater yield coupled with TAPPI brightnesses ranging up to at least 85.

INTRODUCTION

Oxygen/alkali (OA) delignification offers several attractive advantages over present technology. For pulping, the elimination of sulfur is a big step toward reducing air pollution problems related to odor. High yields provide more pulp per ton of wood. On-site generation of oxygen can solve chemical availability and transportation problems. Oxygen can also cut down on the amount of chlorine compounds required in bleaching. This can reduce corrosion problems and minimize the production of toxic, chlorine-containing reaction products. This project is concerned only with the pulping aspect of OA delignification.

Previous work on this project (1) involved red maple as a typical hardwood. Most of the pulping was done at high consistency, using pure oxygen as the oxidizing agent and sodium carbonate as the alkali. A limited amount of work was also included at low consistency using air at ca. 2600 psi and a mixture of sodium carbonate and bicarbonate. OA pulps of 65% yield had properties almost as good as 50% yield kraft pulps. Low consistency offered the advantages of being useful on hardwood chips, giving higher brightness, higher viscosity, and faster delignification, compared to high-consistency pulping.

The present report contains the results for a representative softwood, loblolly pine. In general, softwoods are more difficult to pulp than hardwoods, and this was found to be the case with loblolly pine. Since softwoods are used for strength, maintaining strength was a primary consideration. This is the second report on this project and represents a stopping point while several other projects on the fundamental aspects of OA pulping continue, as well as a detailed economic evaluation of various process configurations. No further experimental work will be done on this project unless new information becomes available that changes either the relative merit or chance of success of this process.

CONCLUSIONS

Oxygen/alkali (OA) pulps can be prepared from loblolly pine at yields higher than kraft pulps of the same kappa number. Low consistency pulping with air gives pulps of higher brightness, higher viscosity, and greater strength than high consistency pulping with oxygen. Fiberization of the wood chips before pulping gives rapid delignification, but better pulp properties were obtained using 1.1-mm thick flakes or compression rolled (Hosmer) chips. These latter materials could be delignified with the low-consistency pulping, although cooking times were long.

The fiberization process did not shorten the fibers, but did appear to give some compression damage. Pulp viscosities were considerably below those of kraft pulps. Most of this drop in viscosity occurred in the first 10 minutes of pulping. After that, a good linear correlation between yield and viscosity was obtained.

Strengths of the oxygen pulps were lower than those of corresponding kraft reference pulps. Although this was compensated for by higher yields, the overall strength advantage still appeared to be with the kraft process. Therefore, oxygen/alkali pulping appears to have its greatest advantage where strength is not a primary consideration (e.g., hardwoods) and where pollution and chemical recovery problems dictate a sulfur-free process.

The oxygen pulps could be readily bleached to 85 brightness and still maintain a yield advantage over bleached kraft pulps. Chemical consumption, although somewhat high, was not excessive. The pulps had reasonable brightness stability.

Hardwoods are probably the best candidates for oxygen pulping. Low consistency with circulating liquor offers some advantages, and the use of air instead of oxygen is feasible. Economic and engineering considerations will dictate what system might be used commercially.

RESULTS AND DISCUSSION

CHIP FIBERIZATION

In the previous report on red maple (1), all of the work at high consistency was done on fiberized chips. At low consistency, some success was achieved with chips as well. In the present work on loblolly pine, all of the high-consistency cooks were done with fiberized chips, whereas a variety of starting materials was used for low-consistency pulping.

Fiberization Conditions and Simulated Yields

Loblolly pine chips were fiberized under four different sets of conditions. The results are shown in Table I. Fiberization was done on two occasions at Bauer Brothers (a subsidiary of Combustion Engineering, Springfield, Ohio). On the first occasion, conditions nominally the same as A-2 were used, and this material was used for the high-consistency work. On the second occasion, all four conditions were followed in an attempt to minimize mechanical damage to the fibers. This was done on a different lot of wood as well.

The fiberization involved an impregnation step with alkali, preheating to the fiberization temperature, and fiberization with a wide plate gap. The high temperatures were chosen to be above the softening point of lignin. This should give fiber separation with minimal fiber fragmentation. The simulation was done on a laboratory scale to obtain yield estimates. An Asplund mill was operated under steam pressure and then discharged directly into an atmospheric Sprout-Waldron disk refiner. The simulated yields fit the expected trend and were used for calculating overall yields. However, the absolute values may be off by 2-3%. Therefore, comparison of overall yields from the same starting

material should be meaningful. However, comparison of yields from different starting materials should take this uncertainty into account.

TABLE I

CONDITIONS AND SIMULATED YIELDS FOR CHIP IMPREGNATION AND FIBERIZATIONS

	A-1	A-2	A-3	A-4
Impregnation				
NaOH, % on wood	41.6		0.0	
Na_2CO_3, % on wood	0.0		8.8	
$NaHCO_3$, % on wood	0.0		6.7	
Temperature, °C	90		90	
Time, min	30		30	
Air pressure applied, psi	100		100	
Washed yield, %	90.9		94.6	
Fiberization				
O.d. solids in, %	36.4		37.3	
Steam pressure, psig	100	80	80	100
Temperature, °C	170	160	160	170
Plate setting, inch	0.030	0.025	0.025	0.030
Feed rate, ADT/day	8.2	7.9	9.9	8.8
Live load, HPD/ADT	0.9	2.9	1.9	1.0
Simulated yield	77.1	81.0	91.6	90.8

Bauer-McNett classifications were done on the material as a measure of the coarseness. For comparison, the material from the first fiberization (LP76), which was used for high consistency pulping, is included (see Table II).

It should be noted that the "Thru 200 mesh" values are determined by difference, and that all of the error accumulates in these values. The first two samples appear to be quite similar. LP76 appears to be slightly finer than its more recent counterpart (A-2), but quite similar to A-3, which was emphasized in the low-consistency work (see below).

TABLE II

BAUER-McNETT CLASSIFICATION DATA FOR FIBERIZED CHIPS

	A-1	A-2	A-3	A-4	LP76
On 14 mesh, %	57.3	60.0	46.8	64.0	44.7
On 28 mesh, %	23.5	22.0	18.8	16.5	24.9
Subtotal	80.8	82.0	65.6	80.5	69.6
On 48 mesh, %	10.6	9.6	7.3	6.6	8.2
On 100 mesh, %	3.4	2.4	1.8	1.8	2.1
On 200 mesh, %	1.1	1.2	0.7	0.8	0.8
Thru 200 mesh, %	4.1	4.8	24.6	10.3	19.3

The material produced in these fiberizations was quite coarse compared to a thermomechanical pulp. It consisted of fiber bundles rather than individual fibers. Hence, the Bauer-McNett results do not reflect fiber length distributions, but rather, a measure of the coarseness of the fiber bundles. A finer distribution should give a more rapid and uniform reaction with oxygen. On the other hand, minimal fiber damage suggests a coarser distribution.

ASSESSMENT OF MECHANICAL DAMAGE DURING FIBERIZATION

No good methods exist for putting mechanical damage into quantitative terms. Furthermore, it is difficult to estimate the effect of mechanical damage on the strength properties of paper eventually produced. In this section, we attempt to evaluate mechanical damage by the following techniques:

1. Appearance under electron microscopy
2. Fiber length distribution
3. Swelling

In a later section, comparison of strength properties of kraft pulps from various starting materials is given.

Electron Microscopy

Fibers were examined by electron microscopy for mechanical damage. In order to look at individual fibers, rather than fiber bundles, the samples were delignified by chlorite and by hydrogen peroxide in acetic acid before examination. The micrographs are shown in Fig. 1 and 2.

At 50X magnification (Fig. 1), a general overview of the sample is obtained. It should be pointed out that this technique looks at a very limited sample, and it is difficult to be sure that it is representative of the entire lot of material. Some fractionation could occur during dewatering and washing. The first thing to be noted is that even after the above-mentioned delignification, a signification amount of fiber bundles still existed. At the ends of some of these bundles, cutting can be noted, particularly in Sample A-1. The samples fiberized in sodium carbonate-bicarbonate (A-3 and A-4) appear somewhat stiffer than those fiberized in sodium hydroxide. The samples fiberized at 170°C (A-1 and A-4) appear to have more debris than their 160° counterparts (A-2 and A-3, respectively). This is particularly dramatic in the case of Sample A-4. It is possible that part of this is due to sampling difficulties, since fines could be filtered into a portion of the sample during dewatering, as mentioned above. This seems likely, since the Bauer-McNett classification on the samples before delignification suggested that A-3 contained the most fines.

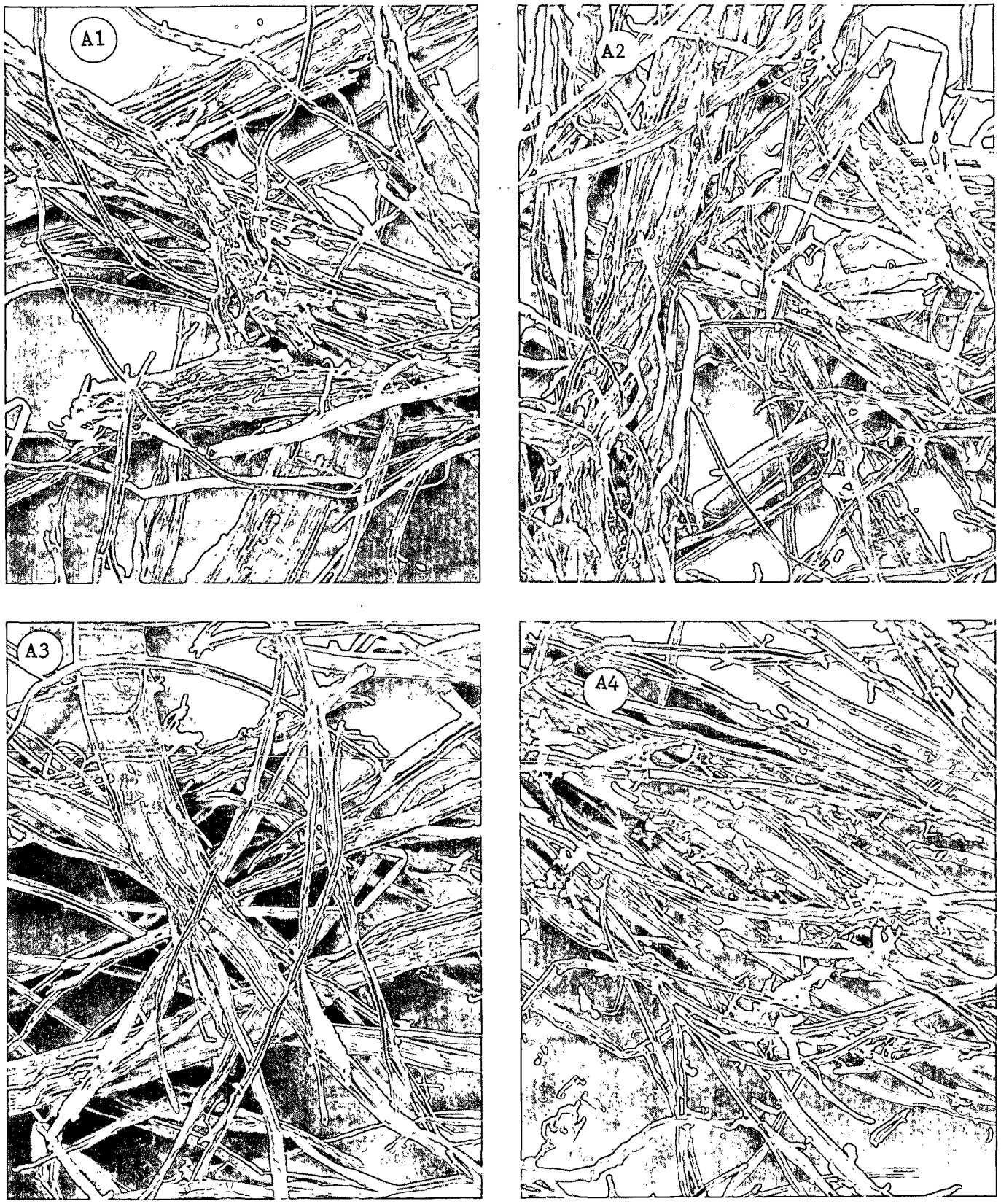

Figure 1. Scanning Electron Micrographs of Fiberized Wood, 50X Magnification

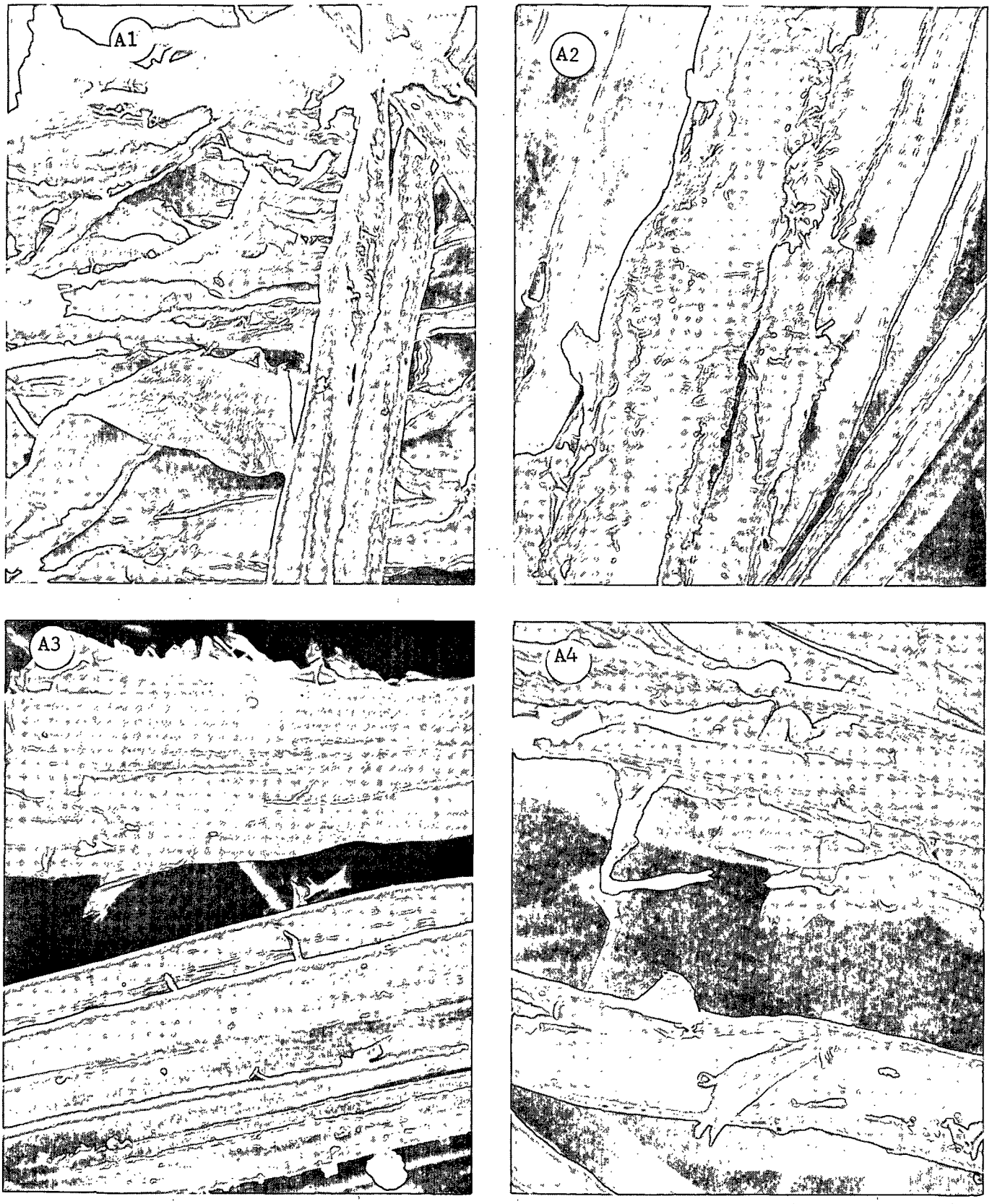

Figure 2. Scanning Electron Micrographs of Fiberized Wood, 500X Magnification

The 500X samples (Fig. 2) were taken not to be representative of the entire sample but, rather, of specific fiber damage in each case. The greater stiffness in the sodium carbonate-bicarbonate samples (A-3 and A-4) is still evident in these micrographs. Some cracking (A-3), delamination (A-4), ragged ends (A-2), and debris are evident. Due to the limited field at this high magnification, it is not possible to say which sample has the most of each type of damage, and it is probable that all samples have some degree of all of these faults. However, at low magnification, Sample A-3 looks the "cleanest," with the fewest flaws and the least amount of debris.

Cupriethylenediamine Swelling Studies

Swelling studies were undertaken as another approach for characterizing the type and amount of mechanical damage to the samples. This technique is based on the concept that cupriethylenediamine should penetrate the damaged areas of fibers more rapidly than the intact areas, resulting in localized swelling. The procedure involves placing one drop of 20% cupriethylenediamine solution on a sample of fibers on a microscope slide, and then examining it within a few seconds. Excessive time results in the entire sample swelling.

The results are shown in Fig. 3. It can be seen that the swelling occurs in the middle of fibers and at kinks, both of which probably represent localized compression damage. In addition, blunt fiber tips, which have probably been cut, also swell, whereas tapered tips do not generally show swelling.

Comparison of the four samples suggests that Sample A-3 suffered the least amount of mechanical damage. However, the usefulness of this technique for quantitative comparisons has not been demonstrated, and the very short swelling times make reproduction of the conditions difficult. Nevertheless,

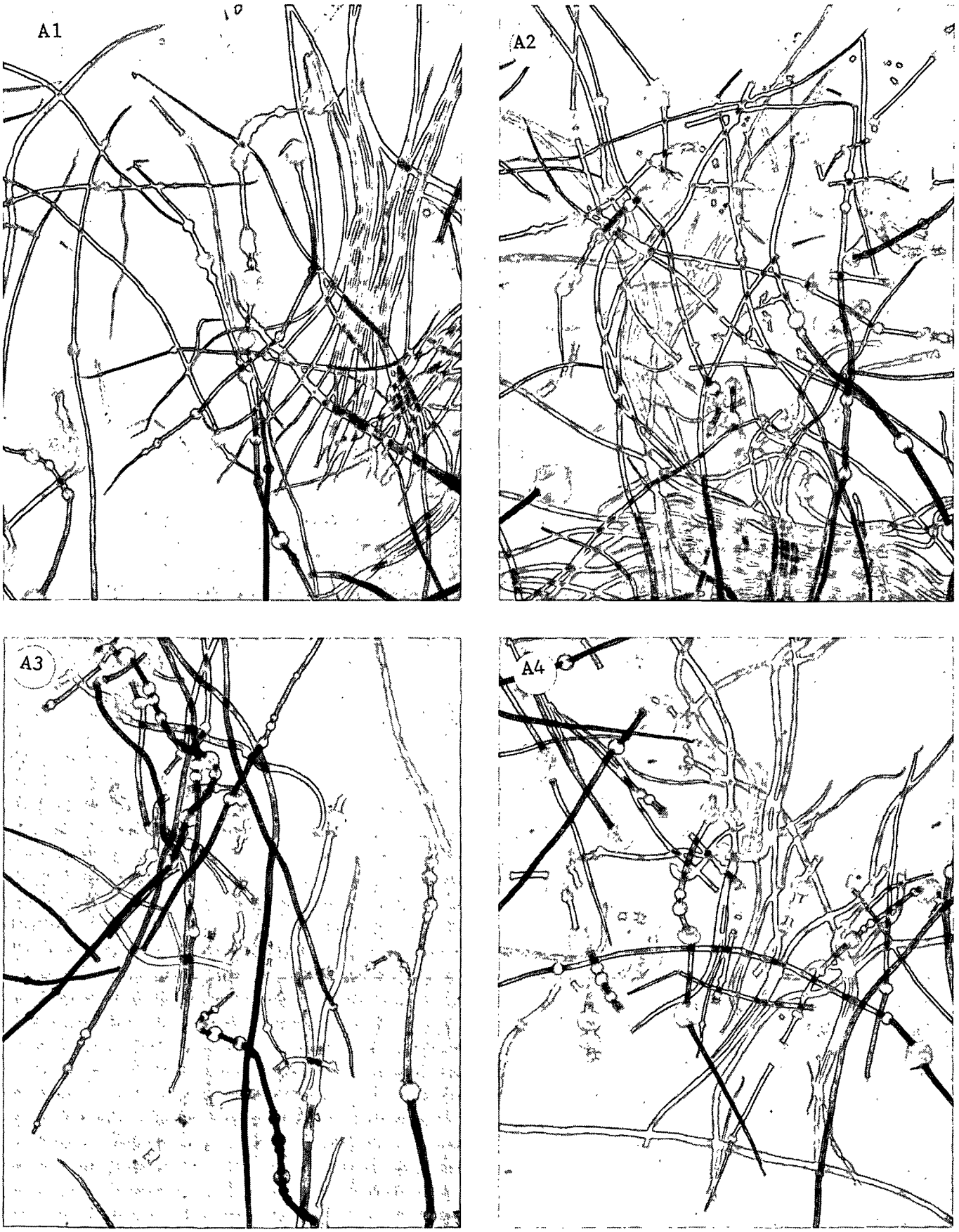

Figure 3. Light Micrographs of CUENE Swollen Fibers, 30X Magnification

this observation is consistent with the electron microscopy work, where A-3 also appeared to be the least damaged.

Fiber Length Distributions

After further delignification with hydrogen peroxide in acetic acid, fiber length distributions were determined by counting on microscope slides. The histograms are shown in Fig. 4 for the four samples, along with a control sample which had been prepared by prolonged treatment of chips with chlorite. The averages are given in Table III. Comparison of the histograms and averages shows only minor differences between the samples. The fiberized samples are not shortened, relative to the control, and, therefore, extensive cutting was not experienced. This is consistent with the high temperature and wide gap used in the fiberization process and, in that respect, the objective was accomplished.

TABLE III

FIBER LENGTH DISTRIBUTIONS[a]

Sample	Arithmetic Average	Standard Deviation	Weighted Average I[b]	Weighted Average II[c]
A-1	2.8	1.3	3.4	3.8
A-2	2.4	1.2	3.0	3.5
A-3	2.3	1.3	3.1	3.6
A-4	2.4	1.3	3.1	3.7
Control	2.2	1.2	2.9	3.3

[a] All values given in mm.

[b] Sum (AB^2)/sum (AB) were B is length and A is frequency.

[c] Sum (AB^3)/sum (AB^2).

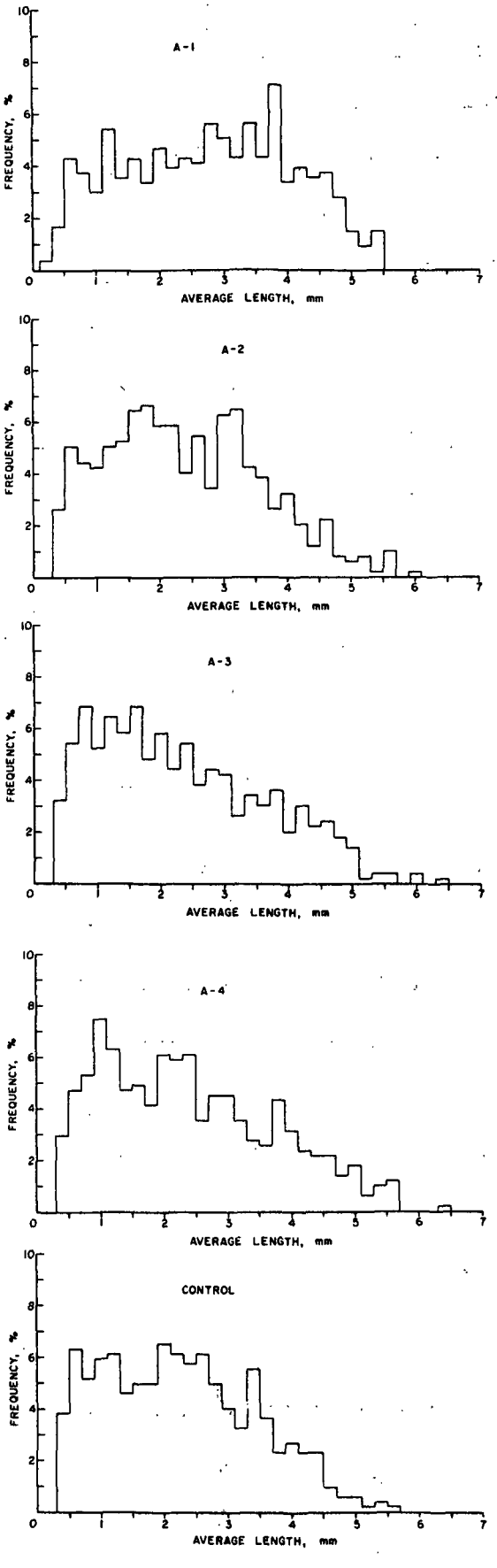

Figure 4. Fiber Length Distributions for Fiberized Chips and Control from Chips

Conclusions Concerning Mechanical Damage

The above results, taken together, suggest that the major portion of the damage was on the surface of the fibers, rather than being due to cutting. The occasional blunt ends, which swelled, may well have been produced primarily in the chipping stage. Both electron microscopy and swelling suggest that Sample A-3 suffered the least amount of damage, which is interesting in view of the fact that Bauer-McNett classification showed that this material was divided into the smallest bundles. This apparent inconsistency is either due to sampling problems or indicates bundle size does not determine fiber length.

Based on the above considerations, together with the high yield obtained in laboratory simulation, Sample A-3 was selected for emphasis in the pulping work. However, some results on other samples are also included.

EFFECT OF COOKING CONDITIONS ON YIELD AND KAPPA NUMBER

Two sets of cooks were done in which the cooking conditions were varied in a systematic fashion. The yield and kappa number data were then treated statistically by multiple linear regression to determine the effect of the variables. This method assumes a linear relationship between the independent variables and the dependent ones (yield and kappa number). With a limited number of data, this linear assumption is about as valid as any other assumed relationship. The two sets were for high and low consistency, and they are considered separately in the following sections.

High Consistency

Twenty-two cooks were made in which alkali applied, temperature, time, and pressure were varied. All possible combinations of the following conditions

were done except 20% alkali, 140°, and 300 psi equivalent, where the exotherm for the reaction was difficult to control. These conditions,

Sodium carbonate:	10 and 20% on wood, as Na_2O
Time:	4 and 15 hours
Temperature:	100, 120, and 140°C
Pressure:	130 and 300 psi, corrected to 100°C

were chosen for the purposes of experimental design, even though it was realized that some combinations would give pulps with unsatisfactory papermaking properties.

The results are given in Appendix I. The following equation was obtained. All possible cross-terms (interactions) were tried, but only those with pressure were found to be significant.

$$\text{Yield} = 76.64 - 0.113\ Na_2CO_3 - 0.0302\ \text{temp.} - 0.017\ \text{time} + 0.2384\ \text{pressure} - 0.001988\ \text{temp.} \times \text{pressure} - 0.00219\ Na_2CO_3 \times \text{pressure} - 0.00226\ \text{time} \times \text{pressure}$$

This equation had an R^2 of 0.97, indicating that 97% of the variation in yield can be accounted for by the variation in the four process parameters which were changed. The T-test indicated that all terms, including the interaction terms with pressure, were significant at the 95% confidence level.

The correlation between yield and kappa number was not as good ($R^2 = 0.60$), indicating that kappa number was dependent on what conditions were used to get to a given yield level. The following equation was obtained:

$$\text{Kappa} = 3.15\ \text{yield} - 81.4$$

Examination of the data (see Appendix I) suggests that at a given yield level, the kappa number was lower at the lower sodium carbonate level. The higher oxygen pressure offered a similar advantage.

Low Consistency

The series for low consistency was carried out on fiberization Sample A-3. The alkali concentration was not varied, since it was chosen to represent a typical liquor from a wet-combustion recovery (9.9 g/liter of Na_2CO_3, 7.6 g/liter of $NaHCO_3$). The following conditions were examined:

Temperature:	130° and 140°C
Pressure:	2550 and 1500 psig air
Time:	2 and 4 hr

In addition, a ninth cook was included which was 150°C, 2550 psig, and 1.5 hr. The data are given in Appendix I.

When yield was calculated as a function of the variables, only temperature and time were found to be significant. Pressure and the cross-products were insignificant. The following equation was obtained, which had a value for R^2 of 0.99.

$$\text{Yield} = 231.5 - (1.16 \pm 0.06)\ \text{temp.} - (5.25 \pm 0.39)\ \text{time}$$

In treating kappa number as a function of the variables, the significance of the variables changed, depending on which were included. This was due to the fact that yield was a dependent variable and therefore correlated with the other variables (except pressure). The best equation was obtained when kappa number was considered to be a function of yield and pressure ($R^2 = 0.98$). Both variables were significant, and the following equation was obtained:

$$\text{Kappa} = -119 + (3.66 \pm 0.20)\ \text{yield} - (0.0123 \pm 0.0030)\ \text{pressure}$$

Hence, even though pressure did not have an effect on yield, it did have an effect on kappa number. The equation predicts that at a given yield, a lower kappa number will be obtained at higher pressure. This means that relative carbohydrate retention increases with increasing pressure, while lignin decreases. The 1050 psi pressure increase was responsible for approximately a 13 kappa number improvement.

The effect of pressure becomes more obvious if the data are considered in matrix form. The data from Appendix I are arranged according to the values for the variables in Table IV. The yields are divided into lignin and carbohydrate by converting kappa numbers into lignin contents by multiplying by 0.15. All values are based on 100 g of original o.d. wood.

TABLE IV

HYPOTHETICAL MATERIAL BALANCE FOR LOW-CONSISTENCY OA COOKS

(Basis — 100 g o.d. wood input)

Temp., °C	Time, hr	Pressure	
		1500 psi	2550 psi
130	2	70.4 g pulp	70.3 g pulp
		12.1 g lignin	11.3 g lignin
		58.3 g carbohydrates	59.0 g carbohydrates
130	4	59.5 g pulp	60.1 g pulp
		7.0 g lignin	6.0 g lignin
		52.5 g carbohydrates	54.1 g carbohydrates
140	2	58.8 g pulp	56.6 g pulp
		7.4 g lignin	5.3 g lignin
		51.4 g carbohydrates	51.3 g carbohydrates
140	4	46.6 g pulp	49.2 g pulp
		2.2 g lignin	1.9 g lignin
		44.4 g carbohydrates	47.3 g carbohydrates

With the data in this form, it can readily be seen that the higher pressure did not have a significant effect on the yield of pulp. However, the yield of lignin was lower in all four cases, while the yield of carbohydrates was higher in three cases and essentially the same in the fourth case.

Alternative Starting Materials at Low Consistency

Experiments with red maple demonstrated the feasibility of using coarser starting materials with the low-consistency equipment. Three additional starting materials were evaluated in the loblolly pine work. These included the following:

1. Conventional chips, nominally 5/8"
2. Thin chips (flakes) 1/2" x 1/2" x 0.045" cut with the grain
3. Compression rolled (Hosmer) chips (nominally 5/8" before rolling)

An important factor with these coarser materials is the uniformity of the cook. This is reflected in the amount of rejects, which represent uncooked wood. The results are given in Table V.

TABLE V

LOW-CONSISTENCY PULPING OF ALTERNATIVE STARTING MATERIALS[a]

Cook number	230	259	241	260	245	250
Starting material	Chips	Chips	Flakes	Flakes	Hosmer	Fib'd A-3
Temperature, °C	130	130	130	130	130	130
Time to temp., min	30	30	30	30	10	10
Time at temp., min	300	450	240	390	450	240
Yield (kappa)[b]	76.0(73)	61.0(43)	65.1(70)	58.9(39)	59.5(41)	60.1(67)
Rejects, %	58.5	21.0	16.1	0.5	7.6	0

[a]All cooks at 2550 psi; 9.9 g/liter Na_2CO_3, 7.6 g/liter $NaHCO_3$, 1.5% consistency.
[b]Yields include rejects; kappas on accepts.

The chips gave a large amount of rejects and were not considered promising. On the other hand the flakes gave an acceptable amount of rejects (particularly for the lower kappa number) in a shorter time. The Hosmer chips gave an encouraging level of rejects, but the cooking time was quite long. These last two samples were considered worthy of further evaluation. They were of particular interest since they did not go through the fiberization process with its associated mechanical damage and yield uncertainty. Strength data are given in a later section.

CARBOHYDRATE REMOVAL BY FIBERIZATION AND HIGH-CONSISTENCY PULPING

In order to determine what was removed from the wood during the impregnation and also pulping, carbohydrate analyses were performed on the starting wood chips, the fiberized material, and several oxygen and kraft pulps. The method of analysis (2) involves hydrolysis, sodium borohydride reduction of the monosaccharides to alditols, derivatization to the alditol acetates, and gas chromatographic analysis. This procedure does not pick up uronic, aldonic, and other sugar acids, so the total values tend to be low. Nevertheless, meaningful comparisons can be made on the basis of the neutral sugars.

The data are given in the Appendix II, while Table VI shows the percentages of the various components which were removed, compared with the original chips.

The first thing to be noted from Table VI is that the impregnation/fiberization process removed 20.1% of the carbohydrates originally in the chips. This corresponds to a yield loss of 12.4%, out of the total of 19%. Hence, approximately 65% of the material removed from the wood was carbohydrate. Further

examination of the table reveals that pulping this fiberized material by either the OA or kraft process to approximately 50% yield reduced the carbohydrates only by an additional 8-11%. Thus, the pretreatment removed more carbohydrate in going to 81% yield than the pulping did in going from 81 to 50% overall yield.

TABLE VI

CARBOHYDRATE REMOVAL BY FIBERIZATION AND HIGH-CONSISTENCY PULPING[a]

	Fib'd Chips	OA Pulp 179C	OA Pulp 182C	Kraft Pulp 3186-90A	Kraft Pulp 3186-90B
Yield	81%	59.0	46.5	43.9	47.9
Kappa	156[b]	96	42	32	74
Araban[a]	22.4	61.2	76.7	84.5	76.7
Xylan	11.5	45.1	57.3	55.8	60.3
Mannan	55.5	69.6	76.1	78.6	76.1
Galactan	60.1	86.7	90.2	90.0	86.7
Glucan	8.8	11.6	12.9	15.9	12.0
Total[a]	20.1	29.4	33.5	35.9	33.8

[a] Percentages removed based on carbohydrate content of pulp and wood, both on an o.d. wood basis.

[b] Kappa numbers are not very meaningful on "raw" fibers.

It is not known at this point whether or not the carbohydrates which were lost during the pretreatment might be retained by modifying the procedure. If high yields with low lignin content are to be achieved, it will be necessary to retain at least part of that fraction. It may turn out that what is not removed of that fraction during a modified pretreatment will be lost in the early stages of subsequent pulping and the goal will not be achieved. Previous experience in holopulping with chlorine dioxide showed that this fraction was associated with increased consumption of oxidant (3).

PULP STRENGTH PROPERTIES

Reference Pulps

A number of kraft pulps and a holopulp (chlorine dioxide) were prepared. These provide the basis of comparison for the OA pulps. The kraft pulps represent existing technology and can be considered a target, whereas the holopulp represents a high-yield, oxidative pulp. Kraft pulping also provides a method of evaluating mechanical damage due to fiberization, since the kraft process can be used on all types of starting materials.

Table VII gives strength properties for a number of reference pulps in the high kappa number range. Values are given for constant freeness and for constant sheet density. Table VIII contains similar data for bleachable pulps.

Several things should be noted from these tables. First of all, the kraft pulps prepared from fiberized chips gave lower strength values than the corresponding pulps from chips. This was true for both the LP76 fiberization and the LP77 A2 fiberization, and at both kappa number ranges. This mechanical damage also shows up in the ClO_2 pulp. The kraft pulp from flakes appears to be intermediate in its properties. Also, with the exception of tear strength, the LP77 wood gave stronger pulps than the LP76 wood. This should be taken into account when comparisons are made between high and low consistency, since high consistency used LP76 and low consistency used LP77 wood.

Oxygen Pulps

Several oxygen pulps were selected for strength evaluation. The data for high kappa number pulps are given in Table IX. One high-consistency pulp is given; the remaining samples are low consistency cooks of various starting materials. The first thing to note is that the low consistency pulps tend to

TABLE VII

STRENGTH PROPERTIES OF HIGH KAPPA REFERENCE PULPS

Cook number	3186-87A	3186-90B	186	15B	22E	23B	19E
Type	Kraft	Kraft	ClO_2	Kraft	Kraft	Kraft	Kraft
Starting material	LP76 chips	LP76 fib'd	LP76 fib'd	LP77 chips	LP77 flakes	KP77 Hosmer	KP77 A-2
Yield (kappa)	52.2(74)	47.9(74)	58.8(58)	51.7(78)	52.0(69)	51.6(73)	52.7(82)
			(500 Freeness)				
Density, g/cc	0.597	0.568	0.556	0.592	0.610	0.618	0.540
Breaking length, km	9.4	7.1	7.1	11.0	9.9	9.3	7.9
Burst factor	64	51	57	84	74	69	57
Tear factor	170	140	107	126	133	117	124
ZS breaking length, km	20.8	14.8	14.3	19.9	20.4	18.9	15.2
MIT fold	850	470	430	1160	1040	940	830
			(0.600 Sheet density)				
Freeness	470	250	300	445	550	610	170[a]
Breaking length, km	9.4	7.6	8.7	11.0	9.7	9.3	9.2
Burst factor	66	58	71	85	72	66	66
Tear factor	170	117	108	123	138	126	101
ZS breaking length, km	20.5	15.2	16.1	19.8	20.5	19.9	16.6
MIT fold	900	730	800	1280	1180	830	1320

[a]Extrapolated values (greater uncertainty). All others interpolated.

TABLE VIII

STRENGTH PROPERTIES OF LOW KAPPA REFERENCE PULPS

Cook number	3186-87B	3186-90A	14E	20E	25E	23E
Type	Kraft	Kraft	Kraft	Kraft	Kraft	Kraft
Starting material	LP76 chips	LP76 fib'd	LP77 chips	LP77 A-2	LP77 A-3	LP77 Hosmer
Yield (kappa)	48.7(40)	43.9(32)	46.9(33)	47.2(40)	44.8(39)	47.3(29)
			(500 Freeness)			
Density, g/cc	0.602	0.593	0.638	0.610	0.611	0.588
Breaking length, km	9.5	7.3	10.8	8.6	8.2	7.6
Burst factor	66	53	85	66	62	54
Tear factor	188	124	142	108	116	162
ZS breaking length, km	20.4	14.7	20.8	15.7	15.3	18.4
MIT fold	990	520	1450	1050	910	830
			(0.600 Sheet density)			
Freeness	495	460	300	550	530	480
Breaking length, km	9.4	7.4	11.6	8.3	8.1	8.5
Burst factor	66	53	90	63	61	60
Tear factor	189	121	133	113	120	141
ZS breaking length, km	20.4	14.7	22.3	15.6	15.1	19.2
MIT fold	1000	520	1860	940	620	1010

be stronger than the high-consistency pulp. This may be due in part to the difference in wood used (LP76 vs. LP77), since a similar trend was noted in the kraft pulps. Secondly, the sodium carbonate/bicarbonate-fiberized samples gave higher yields than the other fiberized samples. Even with the yield uncertainties mentioned previously, these differences are probably real and represent an advantage of fiberizing with carbonate/bicarbonate rather than sodium hydroxide. Finally, the flakes gave the best overall strength properties.

TABLE IX

STRENGTH PROPERTIES OF HIGH-KAPPA OA PULPS

Cook number	208C	235	242	239	241
Starting material	LP76 fib'd	A-2	A-3	A-4	Flakes
Consistency	High	Low	Low	Low	Low[a]
Yield (kappa)	59.5(74)	58.7(89)	64.1(86)	61.8(80)	65.1(70)[a]
	(500 Freeness)				
Density, g/m	0.550	0.601	0.566	0.573	0.601
Breaking length, km	6.4	8.1	8.3	8.0	9.5
Burst factor	40	58	49	49	60
Tear factor	78	84	73	71	77
Zero span, km	11.6	13.9	13.2	13.1	15.3
MIT fold	95	330	190	170	250
	(0.600 Sheet density)				
Freeness	240	505	270[b]	330	505
Breaking length, km	7.2	8.0	8.5	8.3	9.5
Burst factor	44	58	55	53	60
Tear factor	65	85	65	68	77
Zero span, km	12.1	13.9	12.4	13.5	15.3
MIT fold	130	330	265	210	250

[a] Kappa and strength values after removal of 16.1% rejects.

[b] Extrapolated (greater uncertainty).

A similar set of data for low kappa OA pulps are contained in Table X. The high consistency cook (201) included 10% KI, since the strength properties obtained without KI were very poor (ca. 50% of those obtained on Cook 201). Comparing this pulp with the corresponding low-consistency pulp (258C), it can be seen that strength properties were similar, even though the low consistency cook did not contain KI. Unlike the high kappa number pulps, the two yields were similar under these conditions. The alternative starting materials gave significantly higher yields, although these values included a significant amount of rejects from chips and Hosmer chips. Overall, strength properties were similar for the whole set.

TABLE X

STRENGTH PROPERTIES OF LOW-KAPPA OA PULPS

Cook number	201[a]	258C	245	259	260
Starting material	LP76 fib'd	LP77 A-3	LP77 Hosmer	LP77 chips	LP77 flakes
Consistency	High	Low	Low	Low	Low
Yield (kappa)	53.8(43)	52.6(39)	59.6(41)[b]	61.0(43)[c]	58.9(39)[d]
		(500 Freeness)			
Density	0.590	0.642	0.632	0.582	0.620
Breaking length, km	7.6	8.3	8.3	8.1	8.8
Burst factor	57	55	53	52	50
Tear factor	105	76	76	76	81
Zero span, km	14.8	13.8	15.4	15.1	16.0
MIT fold	450	250	160	150	200
		(0.600 Sheet density)			
Freeness	405	640	650[e]	410	600[e]
Breaking length, km	8.2	7.3	7.8	8.4	7.9
Burst factor	60	50	47	53	46
Tear factor	98	88	86	72	88
Zero span, km	15.1	14.0	15.8	15.6	15.4
MIT fold	520	220	150	170	120

[a] Cook included 10% KI on o.d. wood basis.

[b] Kappa and strength values after removal of 7.6% rejects.

[c] Kappa and strength values after removal of 21% rejects.

[d] Kappa and strength values after removal of 0.5% rejects.

[e] Extrapolated values at this sheet density (greater uncertainty).

Comparison of Strength Properties of OA and Kraft Pulps

Individual comparisons of particular OA pulps with particular reference pulps can be made by referring to Tables VII-X. However, generalities can be drawn by classifying the pulps according to type and then taking mean strength properties. This was done for the pulps in Tables VII-X, and the means are given in Table XI. The ClO_2 pulp was not included, since it did not fit with any of the others.

It can be seen that the fiberization caused a substantial loss of strength in the kraft pulps, as mentioned previously. The OA pulps were weaker than the kraft pulps. Breaking length and zero-span breaking length of the OA pulps were comparable to the corresponding kraft pulps on fiberized starting material. Burst was slightly lower, while tear and fold were considerably lower.

Effect of Cooking Conditions on Strength Properties

The set of nine low-consistency pulps which were discussed earlier in "Effect of Cooking Conditions on Yield and Kappa Number" were evaluated with respect to strength properties. In an attempt to determine which variables influence strength properties, a correlation matrix was generated on the computer. The original strength data are in Appendix III. This matrix (Table XII) shows the degree of correlation between individual pairs of variables. The independent variables were temperature, time, and pressure. The rest of the variables can be considered dependent. In-plane tear strengths were also included in this series.

Yields and kappa numbers varied considerably in this series (see Table XVI, Appendix I). These two variables were very highly correlated ($r = 0.97$), and they correlated reasonably well with the strength values, except tear ($r = 0.48 - 0.55$) and in-plane tear ($r = -0.02 - 0.03$). The negative values for

TABLE XI

MEAN STRENGTH PROPERTIES FOR KRAFT AND OA PULPS

Kappa range	High	High	High	Low	Low	Low	Low
Type	Kraft[a]	Kraft	OA	Kraft[b]	Kraft	OA[a]	OA
Starting material	Altern.	Fib'd	Fib'd	Chips	Fib'd	Altern.	Fib'd
Yield (kappa)	51.9(74)	50.3(78)	61.0(82)	47.6(34)	45.3(37)	59.8(41)	53.2(41)
			(500 Freeness)				
Density, g/cc	0.604	0.554	0.573	0.602	0.605	0.611	0.616
Breaking length, km	9.9	7.5	7.7	9.3	8.0	8.4	8.0
Burst factor	73	54	49	68	60	52	56
Tear factor	137	132	77	164	116	78	91
ZS breaking length, km	20.0	15.0	13.0	19.9	15.2	15.5	14.3
MIT fold	1000	550	200	1090	830	170	350
			(0.600 Sheet density)				
Freeness	410	210	340	425	510	550	520
Breaking length, km	9.9	8.4	8.0	9.8	7.9	8.0	7.8
Burst factor	72	62	53	72	59	49	55
Tear factor	139	109	71	154	118	82	93
SZ breaking length, km	20.2	15.9	13.0	20.6	15.1	15.6	14.6
MIT fold	1050	1030	230	1290	690	150	370

[a] Includes chips, flakes, and Hosmer chips.

[b] Includes chips and Hosmer chips.

TABLE XII

CORRELATION MATRIX FOR OA PULPS

	Temp.	Time	Press	Yield	Kappa	Breaking Length	Burst	Tear	Zero Span	Fold	IP Tear
Temp.	1.00	-0.32	0.22	-0.75	-0.72	0.35	0.60	-0.22	0.66	0.50	0.03
Time		1.00	-0.14	-0.39	-0.39	0.54	0.29	-0.39	0.14	0.46	-0.06
Press			1.00	-0.09	-0.30	0.36	0.39	-0.22	0.39	0.12	0.09
Yield				1.00	0.97	-0.72	-0.81	0.48	-0.76	-0.82	-0.02
Kappa					1.00	-0.75	-0.82	0.55	-0.79	-0.82	0.03
Breaking length						1.00	0.92	-0.49	0.85	0.86	0.24
Burst							1.00	-0.37	0.94	0.87	0.36
Tear								1.00	-0.47	-0.45	0.68
Zero span									1.00	0.86	0.25
Fold										1.00	0.22
IP tear											1.00

correlation coefficients indicate a trend toward greater strength at lower yields and kappa numbers. The positive coefficients for tear suggest a trend toward lower tear as pulping is continued to lower yields and kappa numbers. However, this correlation is quite weak with this set of data.

The correlations within the pairs of strength tests were also interesting. Breaking length, burst, zero-span breaking length, and fold were strongly correlated with positive coefficients. On the other hand, tear and in-plane tear did not correlate well with the other strength tests. There was a modest ($r = 0.68$) correlation between the two types of tear tests.

Strength-Yield Factors

The OA pulps gave higher yields but lower strengths than the reference kraft pulps. One way to assess the relative merits is to calculate strength-yield factors. If strength is multiplied by yield (in decimal form), a factor is obtained which is proportional to the amount of "strength" obtained from a ton of wood. Another way of looking at it is that, if strength is proportional to basis weight, then these factors are a measure of the amount of paper one could produce of a given strength from a ton of wood. Tensile strength, zero-span tensile strength, and burst are generally considered to be proportional to basis weight.

Tear strength and fold have a generally more complex relationship with basis weight (4). Therefore, the usefulness of such factors might be questioned. Nevertheless, they are included in Table XIII.

Examination of the factors in Table XIII leads to several conclusions. First of all, even taking credit for the higher yields does not make the OA pulps as strong as the kraft pulps. If comparable starting materials are compared (e.g., OA on alternate vs. kraft on chips), breaking length, burst, and zero-span do not

TABLE XIII

STRENGTH-YIELD FACTORS FROM TABLE XI

Kappa range	High	High	High	Low	Low	Low	Low
Type	Kraft	Kraft	OA	Kraft	Kraft	OA	OA
Starting material	Altern.	Fib'd	Fib'd	Chips	Fib'd	Altern.	Fib'd
Yield	51.9	50.3	61.0	47.6	45.3	59.8	53.2
			(500 Freeness)				
Breaking length factor	5.1	3.8	4.7	4.4	3.6	5.0	4.3
Burst factor factor	38	27	30	32	27	31	30
Tear factor factor	71	66	47	78	53	47	48
ZS breaking length factor	10.4	7.5	7.9	9.5	6.9	9.3	7.6
MIT fold factor	520	280	120	520	380	102	190
			(0.600 Sheet density)				
Breaking length factor	5.1	4.2	4.9	4.7	3.6	4.8	4.1
Burst factor factor	37	31	32	34	27	29	29
Tear factor factor	72(37)	55(28)	43(26)	73(35)	54(24)	49(29)	50(27)
ZS breaking length factor	10.5	8.0	7.9	9.8	6.8	9.3	7.8
MIT fold factor	550	520	140	610	310	90	200

look bad, but tear and fold still come up short. All pulps prepared from fiberized material come out weaker than those prepared from chips and the other alternative starting materials.

In addition, when compared on this basis, the low kappa number pulps of a given type look slightly weaker than the high kappa number pulps. Thus, the only reason to continue pulping is to attain bleachability. Otherwise, slightly more strength per ton of wood is obtained at the high kappa number level.

Miscellaneous Strength Properties

Since the oxygen pulps did not offer any strength advantage over conventional kraft pulps, it was decided to look briefly at specialized properties which might suggest their suitability for board. Modified ring compression tests were run on two oxygen/alkali pulps and compared with a kraft pulp. The OA pulps were 64-65% yield, while the kraft pulp was 51.7% yield. This was done on available standard Tappi handsheets, rather than the heavier sheets normally used. This leads to greater uncertainty in the comparison. Nevertheless, all three pulps gave comparable ring compression values. The higher yields with the OA pulps may make them more attractive, but these results are too fragmentary to arrive at that conclusion without further testing. Results for a higher yield kraft pulp would aid the comparison.

A 70% yield OA pulp (107 kappa) was also tested for taber stiffness. This was again done on standard weight handsheets. No comparable reference pulp was available, but the value obtained fell into the range of "soft" pulps, rather than "stiff" pulps. This might suggest additional testing for specialized uses, but the conclusion should be considered tentative, in view of the limited data.

These experiments are not considered definitive. Normally, they would not be included in a report such as this, but they are given as qualitative observations to suggest future areas of research for suitable applications of OA pulps.

PULP VISCOSITY

Viscosity is considered by many to be a reasonable measure of the inherent strength potential of a pulp. Since it is a chemical property, it is not influenced by stock preparation, etc. In general, it reflects the molecular weight of the carbohydrate fraction of the pulp (lignin is removed by sodium chlorite for high-kappa pulps). Typically, higher molecular weight means greater strength.

With high-yield pulps, such as OA pulps, the appreciable hemicellulose content tends to lower the viscosity. Chemical degradation also contributes to lower viscosity. Hence, oxygen pulps tend to have viscosities which are much lower than typical kraft pulps. This was indeed the case in the present work.

Viscosities were found to drop very quickly in OA pulping. At high consistency, the viscosity dropped from 36.3 to 7.6 in 10 minutes at 120°C. During this time the yield dropped only from 81.0 to 73.1. After that, a trend was noted toward lower viscosities at lower yields. The data are plotted in Fig. 5 and given in Appendix IV. High-consistency results are kept separate from low consistency. Each set gave a straight line, and the following equations were obtained from linear regression analysis:

High consistency: Viscosity = -3.8 + 0.155 yield (r^2 = 0.90)

Low consistency: Viscosity = -5.1 + 0.224 yield (r^2 = 0.98)

Within the parameter range investigated, these two equations predict higher viscosities at low consistency. Also shown in Fig. 5 are viscosities for two high-consistency cooks which contained potassium iodide. It can be seen that these viscosities are much higher than the other high-consistency viscosities. The low-consistency pulps are intermediate in viscosity.

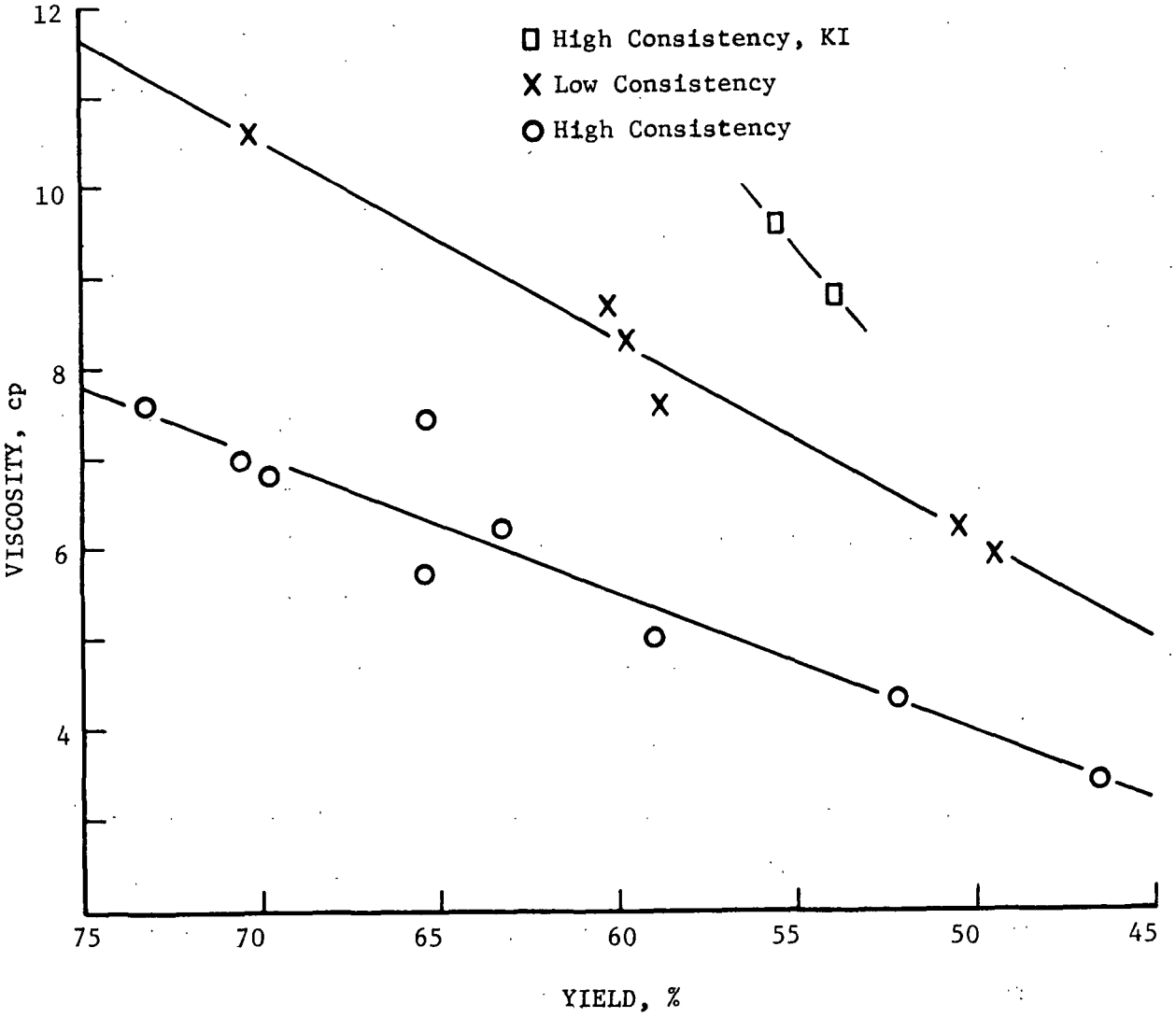

Figure 5. Viscosity vs. Yield for High and Low Consistency OA Pulps

These viscosity results provide a partial explanation for the observation that strength properties did not improve appreciably as lignin was removed in going from high to low kappa number pulps. During this time, viscosity was also decreasing, which counteracted the effect of lignin removal.

PULP BLEACHING

Although the emphasis in this work was on pulp strength, a limited amount of work was done on bleaching. This was primarily to demonstrate that the OA pulps were bleachable, and no attempt was made to optimize a sequence. Two high-consistency pulps and one low consistency pulp were bleached. The results are given below (Table XIV). Bleaching conditions are given in the Experimental section.

The first pulp (kappa number 95) would not normally be considered a bleachable grade. The idea was to see the effect of stopping the oxygen pulping before degradation was too severe. One might consider the first stage of bleaching to be continued pulping with chlorine. In any event, the strength of the bleached pulp does appear to be better than the other high-consistency bleached pulp.

In order to complete the table, a number of extrapolations were necessary, particularly with the bleached pulps. These are indicated with footnotes. Some of these extrapolations were extreme enough to produce absurd results, such as a negative fold and a freeness above 1000. This makes direct comparisons difficult. Nevertheless, strength values and sheet densities generally went up upon bleaching. This is what would be expected from removing lignin, since bonding should be improved and there would be more fibers per handsheet due to the lower yield.

TABLE XIV

BLEACHED PULP YIELDS AND STRENGTH PROPERTIES

Sample	179C		182C		258C	
Pulping consistency	High		High		Low	
Unbleached yield (kappa)	63.3(95)		49.5(46)		52.6(39)	
Bleached yield	48.3		44.3		46.9	
Brightness	87.2		83.4		83.3	
Aged brightness	84.8		81.1		79.5	
	Unbl.	Bl'd.	Unbl.	Bl'd.	Unbl.	Bl'd.
	(500 Freeness)					
Sheet density	0.507	0.666	0.499[a]	0.602[a]	0.642	0.692
Breaking length, km	5.0	6.4	3.9	4.9	8.3	9.4
Burst factor	27	42	17	24	55	61
Tear factor	67	72	53	51	76	67
ZS breaking length, km	9.6	12.1	8.1	9.9	13.8	15.2
MIT fold	16	141	-1	3	254	626
	(0.600 Sheet density)					
Freeness	105	925[a]	240	505[a]	640	1115[a]
Breaking length, km	6.0	4.9	4.8	4.9	7.3	0.84
Burst factor	33	38	24	24	50	93
Tear factor	50	102	48	51	88	127
ZS breaking length, km	10.8	10.7	9.0	9.9	14.0	10.2
MIT fold	43	22	10	2	224	87

[a]Values represent extrapolated data (greater uncertainty), rather than interpolated data.

There was no particular difficulty reaching brightnesses of *ca.* 85. Although chemical consumptions were a bit high, they were not excessive. The pulps prepared at high consistency have considerably lower strengths than those prepared at low consistency. This can be attributed to the fact that potassium iodide was not used, and it is required to obtain good strengths at high consistency, particularly at low kappa numbers.

EXPERIMENTAL SECTION

Most of the methods and techniques used were described previously in the report on OA pulping of red maple (1). These will not be repeated here.

CHIP IMPREGNATION AND FIBERIZATION

This work was done on a cooperative basis at The Bauer Brothers, Springfield, Ohio. Debarked loblolly pine chips (142.6 lb, 52.6% solids, 75.0 lb o.d.) were impregnated with 30.5 lb of sodium hydroxide and 79.4 gallons of additional water at 93°C for 30 minutes under 100 psi air pressure. Excess liquor (73.4 gal.) was drained from the chips and the material (192 lb, 39.7% solids) was then heated with 80 psig steam (162°C) for 3.8 minutes before being fiberized. Fiberization was carried out at the same pressure and temperature and 0.030 inch clearance over an 8.0 minute period. The material was washed and stored in a cold room at 4°C until used.

On another occasion, four fiberizations were carried out similarly, using the conditions given in Table I.

Yields were simulated, using the equipment shown schematically in Fig. 6. Impregnated chips were brought up to temperature in the preheater, held in the Asplund mill under pressure for the same length of time as at Bauer Brothers, and then discharged through an atmospheric Sprout-Waldron refiner. The results are given in Table I.

FIBER MICROSCOPY

Delignification

Samples were bulk delignified by a variation of the method given for the removal of lignin from unbleached pulps prior to viscosity determination (5).

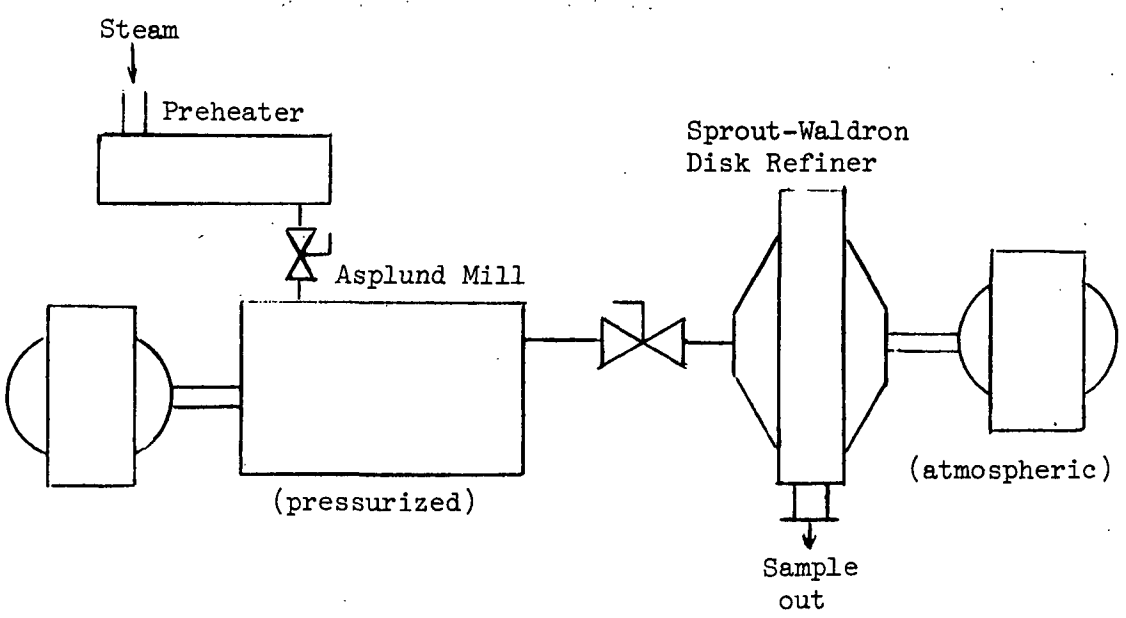

Figure 6. Equipment Used for Laboratory Simulation of Chip Fiberization

A sodium chlorite solution was prepared by combining 5.0 g of $NaClO_2$, 99 g of distilled water, and 1 ml of glacial acetic acid. The equivalent of 2 g of moisture-free fibers was placed in a large test tube and 20 ml of the $NaClO_2$ solution was added and mixed well. The test tube was stoppered and stored in the dark at room temperature with occasional mixing. For fiberized samples, the treatment was for 24 hours. For chips, the treatment was for ca. 7 days, with occasional replenishing of the $NaClO_2$ solution. The samples were then washed with water.

Maceration

The chlorite method left some fiber bundles. Therefore, Franklin's method was used to separate the sample into individual fibers. The samples were put into 1 part glacial acetic acid and 1 part 30% hydrogen peroxide for 4 hours at 65°C. They were then washed several times by dilution with water and decantation. For scanning electron microscopy, they were metallized with gold-palladium,

whereas for light microscopy, they were stained with 0.05% toluidine blue O for 10 minutes and washed.

Swelling

A small amount of the stained fibers was placed on a microscope slide and the excess water was drawn away with bibulous paper. One drop of 20% cuene was placed on the sample, followed by a coverslip. Excess cuene was drawn from the edge of the coverslip with the bibulous paper and pictures were taken immediately of representative areas.

COMPRESSION ROLLED (HOSMER) CHIPS

The equipment used in this work was made available and assistance was given by The Mead Corporation. Loblolly pine chips were soaked in a 5% solution of sodium carbonate at 10% consistency at room temperature for 1 hour and then steamed for 15 minutes at 15 psig. They were then placed in a hopper and fed to the rollers by means of a rapidly moving conveyor whose purpose was to feed one chip at a time (which wasn't always realized). The steel rollers were 24 inches in diameter and 6 inches wide and were set for a clearance of 0.030 inch (ca. 0.8 mm). The rollers rotated in opposite directions, so that both were feeding into the nip. The yield was 96.2%.

FLAKES

Loblolly pine logs were peeled with a drawknife to remove bark, and then cut into 1/2 inch lengths (ca. 1.3 cm). These were then pushed into a rotating cutter blade which cut them 1/2-inch wide and 0.045 inch (ca. 1.1 mm) thick, with the grain. This produced a very uniform product, with a small amount of fines which was screened out.

OXYGEN/ALKALI DELIGNIFICATION

The procedures and equipment for high and low consistency cooks were given in the previous report (1), and will not be repeated here. Table V and Appendix I give the conditions, yields, and kappa numbers of most of the cooks. The following table (Table XV) gives the same information for cooks not described elsewhere, from which data were given in this report.

TABLE XV

COOKING CONDITIONS FOR MISCELLANEOUS OA COOKS

Cook	Starting Material	Consis-tency	Temp., °C	Pressure, psig	Time, hr	Yield, %	Kappa
235[a]	A-2	Low	130	2550	2.0	58.7	89
239	A-4	Low	130	2550	3.3	61.8	80
242	A-3	Low	130	2550	3.3	64.1	86
258C	A-3	Low	130	2550	6.0	52.6	39
179C	LP76	High	120	300	6.5	59.0	96
182C	LP76	High	140	300	4.0	46.5	42
208[b]	LP76	High	120	300	15.0	59.5	74
201[c]	LP76	High	140	300	4.0	53.8	43

[a]Low consistency cooks 9.9 g/liter Na_2CO_3, 7.6 g/liter $NaHCO_3$, 1.5% consistency.
[b]6% Na_2CO_3 on wood, as Na_2O. All other high consistency cooks 10%.
[c]Contained 10% KI on wood.

KRAFT COOKING CONDITIONS

All kraft cooks were carried out in multiple digesters on a rotating rack in an oil bath. Generally, three digesters were used for each sample. Different liquor:wood ratios were used on chips and fiberized wood. In order to keep the amount of chemical applied the same, more dilute liquor was used with the fiberized samples. The conditions were as follows:

Liquor:wood ratio	4 for chips 8 for fiberized wood
Active alkali	18% on wood, as Na_2O
Sufidity	28%
Temperature	174°C
Time to temperature	90 min

Pulp Number	Cooking Time at Temp., min
3186-87A	43
3186-87B	64
3186-90A	56
3186-90B	23
14E	75
15B	34
20E	120
22E	37
25E	160

BLEACHING OF OXYGEN/ALKALI PULPS

A 5-stage CEDED sequence was employed, as shown below:

Pulp 179C

I	14% Cl_2, 3% consistency, 50 min, 25°C
II	6% NaOH, 10% consistency, 60 min, 70°C
III	2% ClO_2, 10% consistency, 150 min, 70°C
IV	1.0% NaOH, 10% consistency, 60 min, 70°C
V	0.8% ClO_2, 10% consistency, 240 min, 70°C

Pulp 182C

I	6% Cl_2, 3% consistency, 60 min, 25°C
II	2.5% NaOH, 10% consistency, 60 min, 70°C
III	1% ClO_2, 10% consistency, 100 min, 70°C
IV	0.75% NaOH, 10% consistency, 60 min, 70°C
V	0.5% ClO_2, 10% consistency, 180 min, 70°C

Pulp 258C

I	6% Cl_2, 3% consistency, 60 min, 25°C
II	2.5% NaOH, 10% consistency, 60 min, 70°C
III	1% ClO_2, 10% consistency, 75 min, 70°C
IV	0.75% NaOH, 10% consistency, 60 min, 70°C
V	0.6% ClO_2, 10% consistency, 180 min, 70°C

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APPENDIX I

DATA USED FOR MULTIPLE LINEAR REGRESSION ANALYSIS

The data used for statistically determining the effects of the cooking parameters on yield and kappa number are given in the following tables (Tables XVI and XVII).

TABLE XVI

YIELDS AND KAPPA NUMBERS FOR HIGH-CONSISTENCY COOKS

Cook	Alkali[a]	Temperature	Time, hr	Pressure	Yield	Kappa
149	10	100	4	130	72.3	137
141	10	100	15	130	70.8	135
148	10	120	4	151	69.8	130
151	10	120	15	151	64.1	120
154	10	140	4	182	61.1	119
155	10	140	15	182	58.4	114
156	20	100	4	130	71.4	140
163	20	100	15	130	65.6	124
160	20	120	4	151	62.4	131
161	20	120	15	151	58.7	135
158	20	140	4	182	57.7	150
159	20	140	15	182	56.8	144
152	10	100	4	300	73.5	130
153	10	100	15	300	68.6	120
147	10	120	4	330	65.5	114
157	10	120	15	330	52.4	52
150	10	140	4	370	50.2	44
170	10	140	15	370	42.0	24
162	20	100	4	300	65.3	120
167	20	100	15	300	62.0	121
164	20	120	4	330	55.3	102
165	20	120	15	330	46.5	60

TABLE XVII

YIELDS AND KAPPA NUMBERS FOR LOW-CONSISTENCY COOKS

Run	Temp., °C	Pressure, psig	Time, hr	Yield, %	Kappa Number
250	130	2550	4	60.1	66.8
237	130	2550	2	70.3	107
253	130	1500	4	59.5	78.6
247	130	1500	2	70.4	115
246	140	2550	4	49.2	25.3
249	140	2550	2	56.6	62.2
252	140	1500	4	46.6	30.8
248	140	1500	2	58.8	84.4
251	150	2550	1.5	50.4	32.4

APPENDIX II

CARBOHYDRATE ANALYSES

The percentages given in Table VI were calculated from the analytical values, given below in Table XVIII.

TABLE XVIII

CARBOHYDRATE ANALYSES OF SEVERAL SAMPLES

	Pine Wood	Fib'd Chips	Pulp 179C	Pulp 182C	Kraft Pulp 90A	Kraft Pulp 90B
Yield	100	81.0	59.0	46.5	43.9	47.9
Kappa		156[a]	96	42	32	74
Rhamnan	0.05[a] (0.05)	--	--	--	--	--
Araban	1.2 (1.2)	1.2 (0.9)	0.8 (0.5)	0.6 (0.3)	0.4 (0.2)	0.5 (0.3)
Xylan	6.1 (6.1)	6.7 (5.4)	5.7 (3.4)	5.7 (2.6)	6.2 (2.7)	5.1 (2.4)
Mannan	11.3 (11.3)	6.2 (5.0)	5.9 (3.4)	5.9 (2.7)	5.6 (2.4)	5.6 (2.7)
Galactan	2.8 (2.8)	1.4 (1.1)	0.6 (0.4)	0.6 (0.3)	0.6 (0.3)	0.8 (0.4)
Glucan	40.3 (40.3)	45.4 (36.8)	60.4 (35.7)	75.6 (35.1)	77.2 (33.9)	74.1 (35.5)
Total	61.8 (61.8)	60.9 (49.3)	73.4 (43.3)	88.4 (41.1)	90.0 (39.6)	86.1 (41.2)

[a] Values in ( ) are on o.d. wood basis. Others are pulp basis.

APPENDIX III

STRENGTH PROPERTIES OF LOW-CONSISTENCY OA PULPS

The strength properties used to study the effect of cooking variables on pulp strength are given in Table XIX below. These data were used to generate the correlation matrix given in Table XII.

TABLE XIX

STRENGTH PROPERTIES OF LOW CONSISTENCY OA PULPS

Sample	250	237	253	247	246	249	252	248	251
Temp., °C	130	130	130	130	140	140	140	140	150
Time, hr	4	2	4	2	4	2	4	2	1.5
Pressure, psi	2550	2550	1500	1500	2550	2550	1500	1500	2550
Yield	60.1	70.3	59.5	70.4	49.2	56.6	46.6	58.8	50.4
Kappa	67	107	79	115	25	62	31	84	32
				(500 Freeness)					
Density	0.604	0.583	0.617	0.491	0.668	0.612	0.672	0.547	0.664
Breaking length, km	8.17	7.1	8.2	5.0	8.05	7.6	8.2	6.9	8.1
Burst factor	52	43	50	29	51	56	56	46	57
Tear factor	80	69.5	71	79	65	76	66	84	71
Zero span, km	13.7	13.3	14	12.4	13.8	14.3	14.2	13.2	14.8
MIT fold	222	98	210	35.8	173	165	300	120	270
In-plane tear, g cm/5 cm	402	328	362	334	316	389	340	380	360
				(0.600 Sheet density)					
Freeness	530	395	604	23[a]	594[a]	576	846[a]	137[a]	690[a]
Breaking length, km	7.9	7.4	7.5	8.5	7.5	7.5	6.2	8.2	5.8
Burst factor	51	45	48	45	50	51	46	50	45
Tear factor	81.5	65	76	55	78	80	81	66	95
Zero span, km	13.7	13.3	13.3	11.8	13.3	13.9	12.4	13.8	14.1
MIT fold	200	112	180	92	126	154	101	150	181
In-plane tear	403	318	386	283	362	397	500	311	440

[a]Values for this sheet density was extrapolated. All others were interpolated.
Pulps are from Table IV of Interim Report Four.

APPENDIX IV

PULP VISCOSITIES

The viscosities used in Fig. 5 are given in Table XX below, along with the cooking conditions. Viscosities were measured after sodium chlorite delignification.

TABLE XX

PULP VISCOSITIES

Sample	Cooking Conditions	Yield	(Kappa)	Viscosity, cp
Fiberized chips LP76	None	81.0	(156)[a]	36.3
High consistency				
143	120°C, 10 min, 130 psi	73.1	(149)	7.6
148	120°C, 4 hr, 130 psi	69.8		6.8
141	100°C, 15 hr, 130 psi	70.8	(135)	7.0
142	120°C, 7 hr, 130 psi	65.5	(127)	5.7
147	120°C, 4 hr, 300 psi	65.5	(114)	7.4
146	120°C, 4 hr, 300 psi	63.2	(116)	6.2
179C	120°C, 6.5 hr, 300 psi	59.0	(96)	5.0
177C	120°C, 15 hr, 300 psi	52.1	(54)	4.3
182C	140°C, 4 hr, 300 psi	46.5	(42)	3.4
High consistency, KI				
201	140°C, 4 hr, 300 psi	53.8	(43)	8.8
203	120°C, 15 hr, 300 psi	55.5	(54)	9.6
Low consistency				
237	130°C, 2 hr, 2550 psi	70.3	(107)	10.6
250	130°C, 4 hr, 2550 psi	60.1	(67)	8.7
253	130°C, 4 hr, 1500 psi	59.5	(79)	8.3
248	140°C, 2 hr, 1500 psi	58.8	(84)	7.6
251	150°C, 1.5 hr, 2550 psi	50.4	(32)	6.2
246	140°C, 4 hr, 2550 psi	49.2	(25)	5.9

[a] Kappa numbers on raw cooks are not very reliable.