



Institute of Paper Science and Technology

THE USE OF A CHLORITE HOLOPULPING PROCEDURE TO EVALUATE CHANGES IN FIBER STRENGTH IN THERMOMECHANICAL PULPING

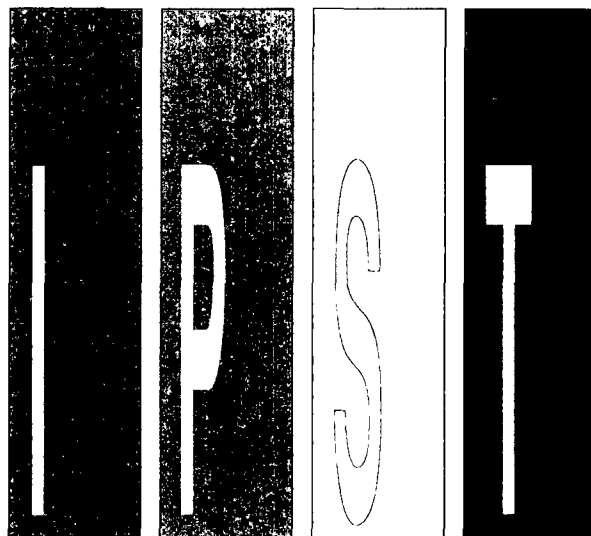
Project 3729

Final Report

to the

MEMBER COMPANIES OF THE INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

March 1995



Atlanta, Georgia

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CHANGES IN FIBER STRENGTH IN THERMOMECHANICAL PULPING

Project 3729

Final Report

A Progress Report

to the

MEMBER COMPANIES OF THE INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

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Summary	3
Introduction	3
Chlorite Holopulping	4
Evaluation of Fiber Damage in Plug Screw Feeders	6
Influence of Holopulping Parameters on Handsheet Strength	7
Results of The Beater Run Testing	8
Seasonal Strength at Bowater Calhoun	10
Results and Discussion	10
Wood Chip Analysis	13
Conclusions	15
Experimental Procedures	16
References	17
Appendix	19

Summary

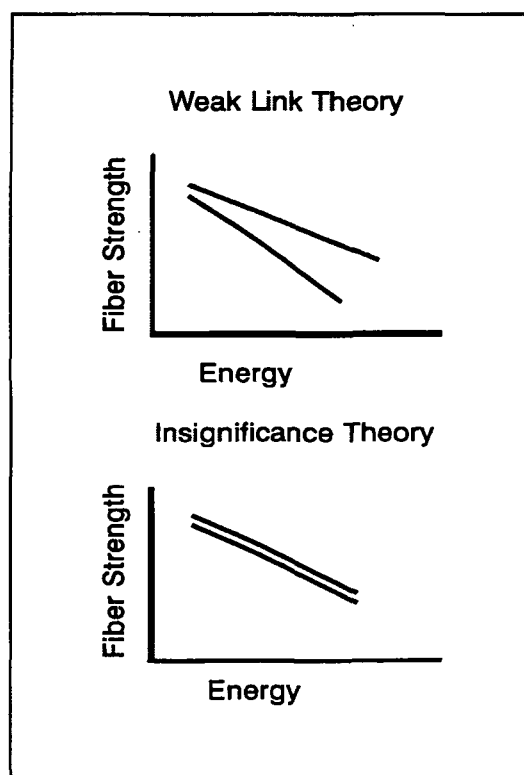
The chlorite holopulping process has been used to delignify thermomechanical pulps from various stages of the process ranging from wood chips to latency chest fiber. The strength testing of this fiber has shown surprisingly little loss in native fiber strength as measured by a zero-span tensile index, but some loss in fiber tear strength related primarily to fiber shortening. The method has been used to confirm strength losses induced by an impregnator type plug screw dewatering device operating with inadequate presteaming. The method has also been used to evaluate seasonal strength changes in TMP and has shown that these are related to seasonal changes in wood supply and native fiber strength.

Introduction

Papers made from mechanical pulp are generally a fiber bonding limited structure.¹ In an ideal bond limited paper, fibers pull out of the sheet at failure and relatively few fibers break. Tensile and tear strength depend only on surface area and bond shear strength. However, for sulfite chemithermomechanical pulp² (CTMP), alkaline peroxide chemimechanical pulps³ (APMP), and low freeness thermomechanical pulps (TMP),⁴ tear index goes through a maximum with increasing specific energy and begins to decline with additional refining. These pulps reach the point where bond strength exceeds fiber strength and the paper will benefit from improved processing to preserve the native strength of the wood fibers.

To avoid fiber strength losses in high yield pulping, it is important to consider fiber damage that occurs early in the process. Fiber damage in chipping, chip handling, chip compression in the plug screw feeders, and in the initial size reduction in the first stage refiner, may increase the susceptibility of the fiber to cleavage later in the process. The result would be a magnification of damage throughout the process. If this occurs, the gap between the fiber strength resulting from good processing and poor processing will expand with increasing energy. This situation is depicted in figure 1 labeled as the weak link theory.

It is also possible that early fiber damage becomes lost in the damage induced in the later stages of mechanical pulping. This possibility is shown (Fig. 1) as two parallel lines for fiber strength with the additional strength loss from poor processing before refining making just a small contribution to lower fiber strength in the final pulp. It is referred to as the insignificant loss theory.



The conventional method of evaluating damage that occurs early in the refining process is to complete the refining under controlled conditions and pass judgement on the merit of the chip treatments and early stage refining by the effects observed on final pulp quality. Overall, this is not a bad concept, final pulp quality is the issue of real commercial interest and both the weak link theory and insignificant loss theory suggest that under controlled conditions, the damage from early stage processing would be observable in the final pulp. Unfortunately, the concept of control in refining is as much a goal as a reality and the remaining uncontrolled process instability limits the sensitivity for detecting change. In addition, without a detailed analysis, it is impossible to determine from a refining study whether the process change has preserved the native fiber strength or increased the interfiber bonding strength. This knowledge is useful for fully exploiting a new process and understanding its applications and limits.

Ideally, to understand the process, it is vital to separate paper strength into its component parts, fiber strength, surface area, relative bonded area and specific bond strength and to be able to evaluate the progress in developing each of these component throughout the chip treatment and refining process. This project has evaluated a chemical delignification method for measuring fiber strength throughout the refining process. The technique was then used successfully to evaluate pulp strength problems at the MacMillan Bloedel TMP mills in British Columbia, and the Bowater TMP mill in Calhoun Tennessee.

Chlorite holopulping and handsheet testing were selected for evaluation in this project. Chlorite holopulping provides a means to selectively delignify wood at any point in the wood handling and pulping process.^{5,6} Handsheet testing has the advantage of using a fully representative sample of the wood or pulp. Although fiber bond strength, fiber strength and the presence of handsheet defects all influence handsheet strength, these problem can be minimized by relying on zero span⁷ tensile testing and both the tensile and tear indices^{8,9} as a measure of fiber strength. Handsheet defects such as shives and variations in basis weight will increase the standard deviation in the handsheet testing. These can be controlled by proper preparation of the pulp for sheet forming.

Chlorite Holopulping

A room temperature acetate buffered chlorite technique and a 60° C buffered chlorite technique were evaluated for preparing the pulp. The principle advantage offered by the high temperature procedure is in faster sample handling. At 60° C each chlorite treatment was complete in about 24 hours. This allowed the samples to be treated with five chlorite treatments (and 4 alkaline extractions) in about 15 days. The room temperature procedure required five days for each chlorite treatment and 8 weeks to completely delignify wood chips. In both cases, pulps were evaluated for Kajaani fiber length and handsheet testing of zero-span tensile index, tensile index and tear index. The hot chlorite techniques showed lower fiber length, tear strength and scattering coefficient than the pulps prepared with the room temperature procedure (Table 1). The room temperature method was used in all subsequent experiments.

Several changes were required in the standard literature holopulping procedures to make them suitable for this project. It was not acceptable to grind or slice wood chips prior to delignification since this introduces unwanted chip and fiber damage. This makes it difficult if

not impossible to solvent extract the wood or pulp prior to the chlorite treatment. The solvent usual extraction step was eliminated¹⁰ but to improve the removal of soluble lignin fragments and saponify and remove extractives, the chlorite delignification was separated into several steps and the partially delignified wood extracted with dilute sodium hydroxide between the chlorite treatments. Long treatment times and additional treatment stages were required to fully delignify the wood chips. Vacuum impregnation of the chlorite solution was used on the first two treatment stages to assist penetration of the chemicals into the wood chips¹⁰ and wood chips were screened on a Williams Classifier and the retained 1 inch fraction was replaced with an equal weight of pass 1" retained 3/4" chips. After the wood is broken up in the British Disintegrator, a gel like precipitate forms during the alkali extraction steps. This plugs filter papers and makes fiber recovery more difficult. To avoid this problem, filtration was carried out on a 150 mesh wire screen. It was assumed this would increase the loss of fines and hemicellulose but these were judged to make a minimal contribution to the testing and the use of the wire screen improved handling substantially.

In the initial evaluation (Table 1), two chip sources were evaluated with replicate samples treated by the room temperature procedure to provide additional information on test reproducibility. Chip source 1 had been destructured by compression at room temperature to 28% of its initial volume. Chip source 2 had also been to 28% of its initial volume, but after atmospheric presteaming for 30 minutes.

Table 1: Replicate chip crushing and holopulping experiments.

Run #	Tensile Index N·m/g	Tear Index mN·m ² /g	Z-Span Tensile N·m/g	Fiber Length mm	Scattering Coefficient m ² /kg	Chlorite Yield %
1a	60.8	12.1	142.7	3.64	20.0	53
1b	65.0	13.5	120.2	3.44	20.4	50
1 _{60°}	59.9	9.1	147.6	3.36	18.1	55
2a	55.1	11.7	139.6	3.40	18.9	71
2b	73.1	13.9	144.5	3.40	21.4	67
2c	69.6	14.0	123.3	3.56	21.5	72
2 _{60°}	78.7	10.2	130.6	3.29	18.1	46

Run 1_{60°} is the same chip source as runs 1a and 1b but with the chlorite delignification conducted at 60° C. Run 2_{60°} is the same chip source as runs 2a, b and c but with the chlorite delignification carried out at 60° C.

A high level of variability was observed in this initial evaluation. This made it impossible to observe differences in fiber quality caused by the two wood chip compression methods. The room temperature compression procedure does appear to have a significant influence on the yield after chlorite treatment. Presumably, this is due to a larger percentage of fines generated in the room temperature compression procedure and liberated by the holopulping.

Evaluation of Fiber Damage in Plug Screw Feeders

Although the precision of the chlorite procedure was still in doubt, the project needed information on the magnitude of fiber damage from commercial processes to know what level of precision was needed and whether the chlorite delignification procedure could be useful. For an initial test, samples were collected from the feed and discharge of two Impressafiners® in the MacMillan Bloedel British Columbia TMP mills. The impregnation stage in mill A was thought to be operating well, but the impregnation stage in mill B was thought to suffer from steam channelling problems. The four samples were holopulped in duplicate. After completing the holopulping, the samples were screened on an 0.008" slot Valley Flat Screen before making the hand sheets for testing. This removed the remaining shives that were not completely delignified and it was hoped would improve the reproducibility between duplicate samples. The averages are reported in table 2 with the variation between duplicates indicated in brackets.

For these samples, the average values show a strength loss in the chips exiting both Impressafiners®. In mill A the strength losses range from 1% in Tear Index to 9% in Tensile Index. Mill B shows a loss ranging from 7% in Tear Index to 19% in Number Average Fiber Length. The average loss for all tests is 4.5% in mill A and 12% in mill B.

The agreement between duplicates is quite good and the results clearly indicate that there is measurably greater fiber damage occurring in the Impressafiner® in mill B.

Table 2: Holopulping results from the feed and discharge of two impressafiners®.

Sample	Zero Span Tensile N•m/g	Tensile Index N•m/g	Tear Index mN•m ² /g	Fiber Length (Number Average) mm
Mill A				
Feed (chips)	148(4)	103(1)	8.4(.3)	1.76(0)
Discharge	145(5)	94(9)	8.3(.2)	1.65(.06)
% Retention	98	91	99	94
Mill B				
Feed (chips)	148(2)	104(3)	8.2(.2)	1.81(.05)
Discharge	135(6)	90(3)	7.6(.1)	1.47(.08)
% Retention	91	86	93	81

Each value is the average of duplicate experiments, the number in brackets is range ÷ 2

Influence of Holopulping Parameters on Handsheet Strength

The initial project demonstrated that chlorite holopulping and handsheet testing could be used to detect relatively small changes in fiber strength in wood chip handling and treatment prior to refining. For a second trial, it was decided to test the process throughout a refiner line where large changes in fiber strength, fiber surface area, and fiber length could be expected. In this task, the project encountered several more issues with the chlorite holopulping technique that had not been faced previously and needed to be addressed.

1. The different wood particle sizes delignify at vastly different rates. Typically, the refiner discharge samples appear to be fully delignified and fiberized while the wood chips still have hard centers.
2. The time spans between sample periods requires a comparison of results from one series of chlorite delignification experiments to another. This introduces more opportunities for differences in sample treatment that could reduce the precision of the testing.
3. After completing a series of samples, it was discovered that there was considerable variation in holopulping yield and final pulp brightness between samples (table 3).

The first problem was handled by treating all the samples with 5 chlorite stages. Thompson indicated the chlorite treatment was mild enough that the differences in rate of delignification would not seriously influence the results.¹¹ The second question was evaluated by repeating the chlorite treatment and pulp testing of a chip sample after several months had elapsed. The yield variation was thought to be due to the more rapid delignification of TMP fiber than wood chips, and loss of fines when the pulps were filtered and washed. The fines loss was not considered to be a problem since the intent was to measure the fiber strength. The influence of yield on final strength needed to be addressed. Because of the concern over yield, it was deemed necessary to improve the reliability of the final pulp weight measurement. This was a difficult measurement since the high hemicellulose content of the pulps and the need to make quantitative transfers when handling the samples. This usually resulted in samples with about a 6% consistency, where it is difficult to collect a representative subsample. It was decided to air dry samples before determining final mass and preparing handsheets for testing.

As a check on the effect of air drying pulps on sheet strength, and of the test precision over time, the two samples of wood chips from the initial series of tests were holopulped and tested a second time. This data is listed in table 3 for one set of wood chips and is graphed as a percent of maximum bar chart in figure 2 for the other. In figure 2, the first two bars in each group are the duplicate samples for the initial test made from slush pulps. The third and fourth bars are the same chips pulped about three months later and tested as once dried pulp. The zero span tensile index, tear index and fiber length show a reproducibility between periods of 90% or better, in spite of the air drying procedure. However, the tensile index is 25% lower for the chips treated with the spring samples. This low tensile index is observed in all the samples that were air dried before making the handsheets.

Table 3: Yield, Brightness, Zero Span Tensile Index and Tensile Index .

Sample	Yield	Brightness	Z-Span	Tensile Index
Chips	60.23	77.95	134.41	58.15
	58.33	76.42	136.30	59.52
Chips (AD)	61.40	81.85	141.10	43.80
	62.20	82.52	138.00	42.80
Primary	53.70	81.29	138.96	75.22
	53.10	81.98	135.84	75.30
Secondary	47.40	80.67	142.11	80.51
	49.90	81.57	151.82	80.33
Latency	48.41	77.10	139.03	71.17
	48.10	76.89	135.36	77.15

To answer the remaining questions, an extended series of tests was carried out on a sample of wood chips supplied by the Bowater mill. Twenty two 30 OD gram samples were delignified with sodium chlorite as indicated in the experimental section. Half were treated to three cycles of acid chlorite followed by alkaline extraction, and a final fourth chlorite stage. The other half were treated to an additional extraction and chlorite stage. The four stage treatment gave a final pulp yield of 64% with a brightness of 80. The additional stages reduced the yield to 63.2% and increased the brightness to 84%.

Pulps were beaten in a PFI mill to 0, 2300, 4000, 6500, and 9000 revolutions giving freeness levels ranging from 760 to 355. Half the sample at each freeness level was air dried. TAPPI handsheets were then made from each sample giving sets for two yield levels, 5 freeness levels and air dried vs. never dried. Handsheets were tested for zero-span tensile, tensile, tear and burst and optical properties. Pulps were tested for Canadian Standard Freeness (before drying) and Kajaani fiber length.

Results of The Beater Run Testing:

The number of chlorite treatment stages and the air drying process proved to have a significant effect (95% confidence level) on all the handsheet tests. The pulp freeness had a significant influence on all variables except the zero-span tensile index. The number of treatment cycles was significant in all cases except for Burst Index. Fiber length was reduced about 3% in the air drying process and decreased by about 7% in beating.

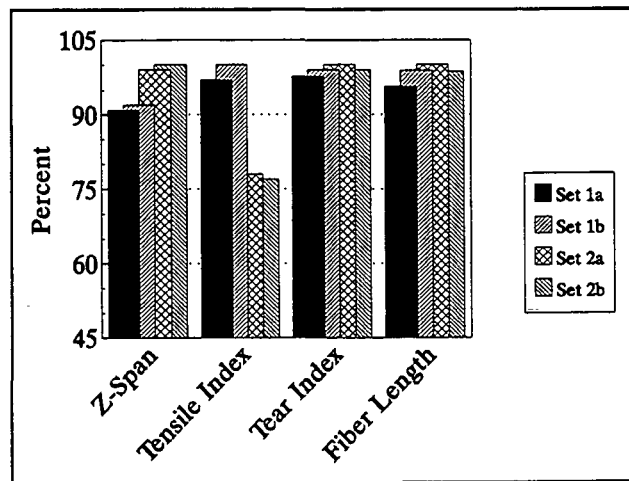


Figure 2. Normal mill production wood chip samples. Set 1 a & b are winter chip data. Set 2 a & b are winter chips processed with the spring samples.

The effect of air drying the pulp on zero span tensile relative to starting freeness is shown in figure 3 for the wood samples treated with four and five stages of sodium chlorite delignification. The four stage data are shown as trend lines since this data gave a good fit to a linear regression. The line for the air dried pulp sample runs nearly parallel to the never dried response but is displaced to lower values of z-span tensile index by about 12 N·m/g (8.6%). The data for the five chlorite stage case are plotted as data points for comparison to the trend lines for the four stage case. Although these data do not adhere to the slopes of the four stage data, nearly all the points for the five stage data fall below the trend lines of the four stage data. The average loss in zero span tensile index is 5.2 N·m/g (3.7%) for the fifth stage of chlorite treatment.

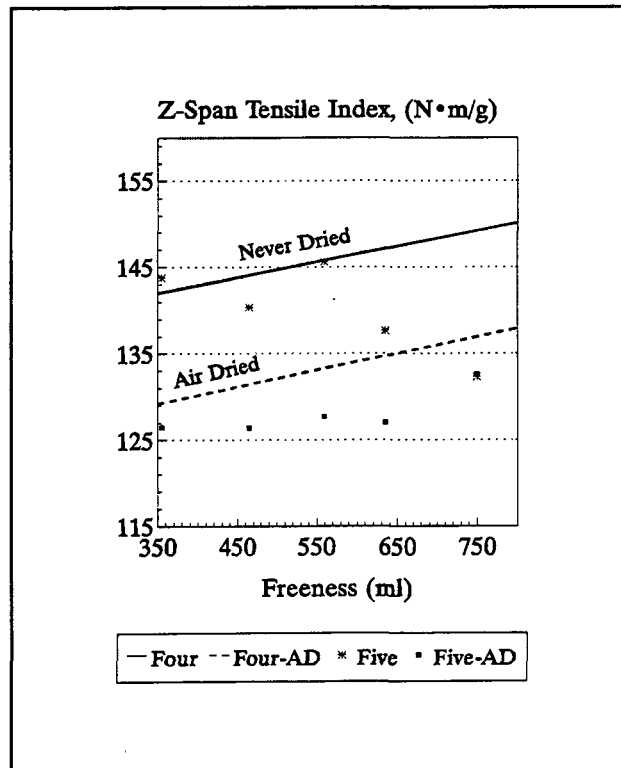


Figure 3. Zero Span Tensile Index for the beater run samples.

With chemical pulps, zero-span tensile strength usually increases with beating, rising to a maximum or broad plateau around 600 ml CSF.¹² In the freeness range of these experiments the zero span tensile should be nearly independent of freeness. In a multiple regression analysis of the zero span data, the air drying process and the number of chlorite stages were significant, but pulp freeness was not significant at the 95% confidence level.

The influence of the three pulp variables on tensile index is shown in figure 4. The air drying process reduces the tensile index by about 20 N·m/g (25%) at all freeness levels. The fifth stage of chlorite treatment has a much smaller effect, reducing the tensile index by just 3.5 N·m/g (4.4%). The graph for tear index relative to never-dried freeness is shown in figure 5. The fifth chlorite stage reduces the tear index by about 0.45 mN·m²/g (4.7%) and the air drying process increases the tear index by about the same amount.

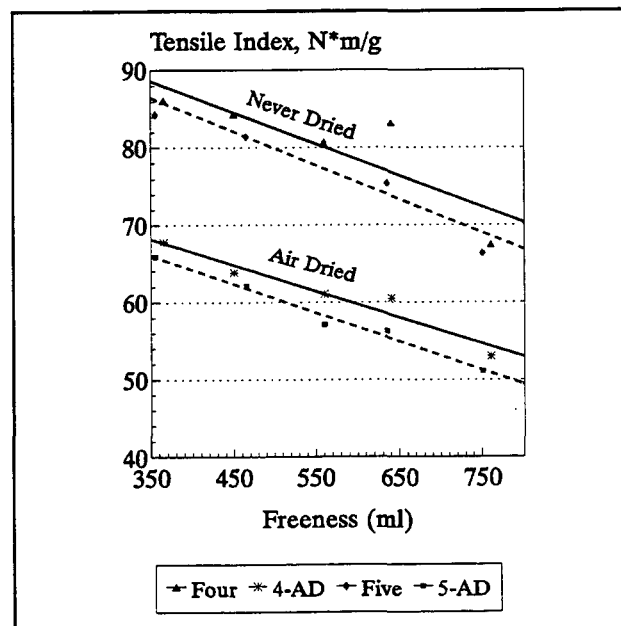


Figure 4 Tensile strength data.

The relationship of tear index to tensile index is generally considered to be a better measure of pulp strength than the individual

measurements. This data is shown in figure 6. Lines are provided for the five stage never-dried and five stage air-dried data. The air drying procedure has clearly reduced the tensile index without a corresponding increase in tear. The fifth stage of chlorite treatment also appears to have reduced the fiber strength, particularly in the case of the air-dried pulp samples.

Seasonal Strength at Bowater Calhoun

In January 93, the project began an evaluation of a seasonal strength loss in the Bowater southern pine TMP mills. The Bowater mills had observed a regular loss in pulp strength during the winter months. The Bowater Mercy mill in Liverpool Nova Scotia recently reported a similar problem and provided evidence that the winter strength loss was related to seasonal changes in the chip size distribution.¹³

Arrangements were made to test samples from the Bowater Calhoun, Tennessee TMP mill during the January, March/April, June/July, and October/November time periods. The final sample was postponed in favor of collecting a second set of samples during the seasonal low strength period. When the mill did not experience a definite loss in strength the second winter, a control sample was sent and used for the PFI beater runs reported above.

For each season, the mill supplied samples of wood chips from the chip washer and fiber from the discharge of the primary refiner, the discharge of the secondary refiner, and the latency chest. Samples were collected twice during the sample period. One set of samples was taken under conditions as closely controlled as possible. The second set of samples was representative of the normal mill operation at the time. Wood chip samples were screened on a Williams Classifier to determine if there was a significant change in chip size distribution between the seasons. Each sample was pulped and analyzed in duplicate. All samples were holopulped and tested as before. A complete table of results is given in the Appendix.

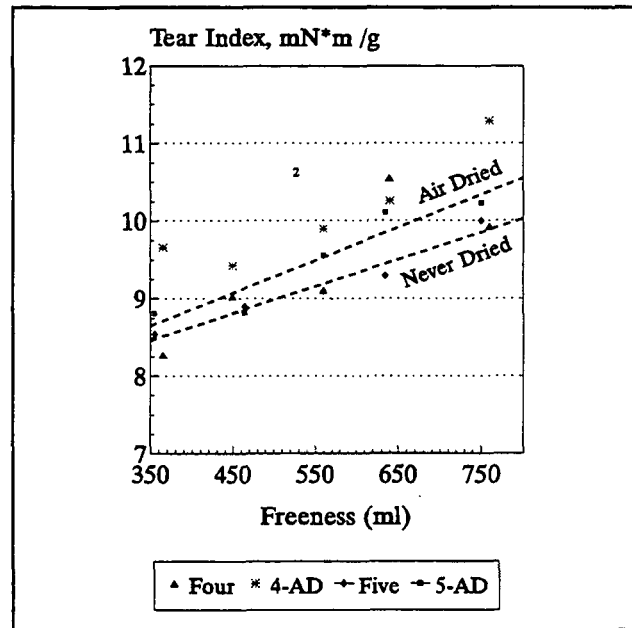


Figure 5. Tear index data.

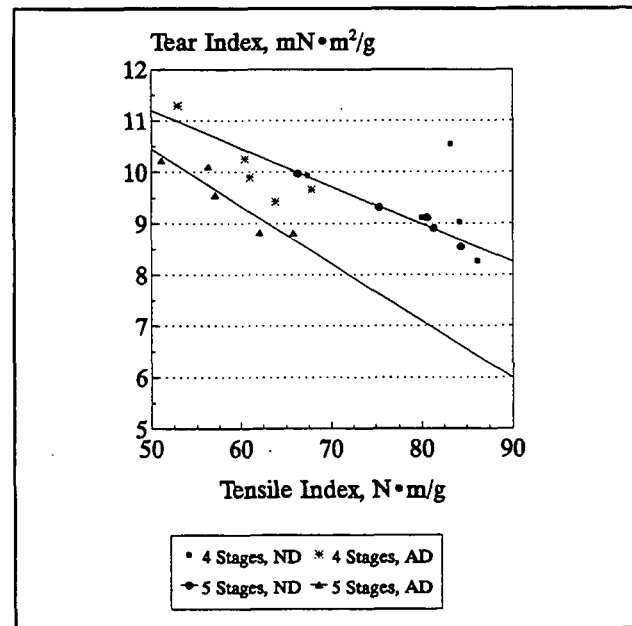


Figure 6 Tear vs Tensile.

Results and Discussion

The mill operating data and secondary refiner pulp test results for the controlled production samples are listed in the appendix.

TMP strength was low for the winter sample period, when the mill was experiencing an extended period of low pulp strength. However, it was also low during the summer sample period which was not expected. Specific energy (estimated from total motor load divided by rpm) was highest in spring and lowest in winter, correlating with tensile strength. Tear Index is plotted against freeness in Figure 7. A linear regression line

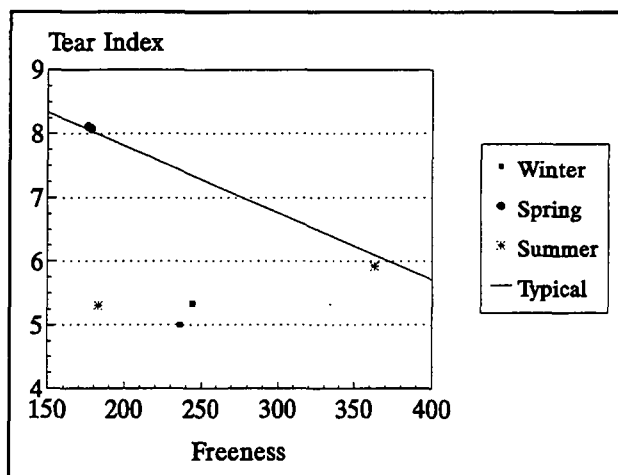


Figure 7. Tear Index plotted against freeness for the latency chest samples (mill data). The response line is generated using Pilot Plant data.

using pilot plant data from the Andritz Sprout-Bauer laboratory in Springfield has been added as a typical response curve. The two samples from the winter collection period confirm the low strengths reported by the mill. The low freeness sample from the summer collection period also shows a low tear strength relative to the expected response line.

Table 4: Student t values for pooled Zero-Span Tensile Index data.

	Winter		Spring		Summer	
	Normal	Control	Normal	Control	Normal	Control
Z-Span N·m ² /g	139	139	150	152	142	144
WinNorm	0	-0.218	3.230	3.668	0.621	1.627
WinCont	-0.218	0	3.693	4.130	0.912	2.059
SprNorm	3.230	3.693	0	0.526	-2.706	-1.918
SprCont	3.668	4.130	0.526	0	-3.169	-2.425
SumNorm	0.621	0.912	-2.716	-3.169	0	0.994
SumCont	1.627	2.059	-1.918	-2.425	0.994	0

Entrees in bold print are significantly different . $t_{crit.} = 2.977$ for a 99% confidence level two tailed test, 2.145 for a 95% confidence level two tailed test and 1.761 for a 95% confidence level one tailed test.

Since freeness was not a significant variable for zero span tensile testing of the chlorite pulps, the data from the three seasonal test periods was pooled and tested for significant differences. The average zero-span data and results of the t tests are summarized in table 4. There are no significant differences between the control and normal production samples from each period, but

the spring zero span tensile strengths are significantly greater than the winter 92/93 and the summer samples. The summer controlled production zero-span is significantly greater than the winter samples at a 90% confidence level (one tailed t-test). Since the Winter samples were not dried before making the handsheets, these zero-span tensile indexes should be about 12 N m/g higher than would have been obtained with air dried samples. This would make both sets of summer samples significantly stronger than the Winter 92/93 samples.

It was expected that holopulps produced from wood chips would give the highest zero span tensile index since these are the strongest fibers available to the process. As seen in figure 8, this is not the case. There appears to be an improvement in zero span tensile index from the refining, probably from increased bonded area. Typically, the range of zero span tensiles for the four samples in each set was less than 8%, confirming the relative insensitivity of the test to beating and largely justifying the pooling of this data.

The tear/tensile data for the three sample periods and four sample locations are graphed in figure 9 as a bar chart showing percent Tear Index relative to the control five stage PFI data (see the appendix). For this graph, the tear index and tensile index data of the standard production and control samples were averaged, and the average Tear Index compared to the estimated tear index of the appropriate control sample. For the winter samples, the control sample is the five stage never dried PFI beater run data, for the spring and summer samples the control is the five stage air dried beater run data. Each bar represents four holopulping and handsheet testing replicates. At all four positions the spring samples give the highest tear index (at constant tensile). For the wood chips, primary refiner discharge and secondary refiner discharge, the winter samples have a higher tear index than the summer samples. At the latency chest, the relative tear index of the summer samples exceeds the tear index of the winter samples.

The average % Tear Index relative to the controls is shown in the last cluster of bars. The spring samples show the highest overall average test followed by the winter and summer

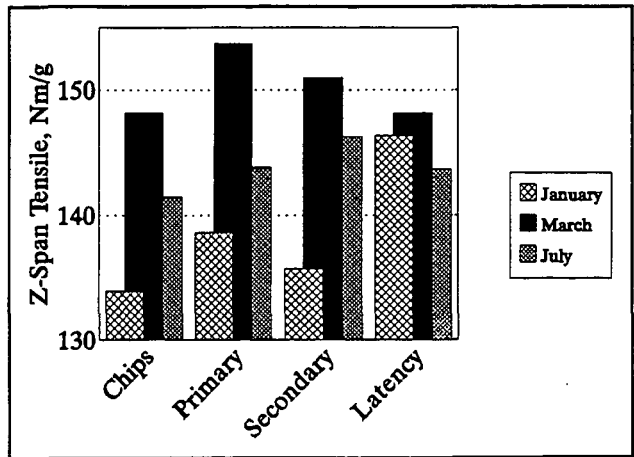


Figure 8. The zero span tensile data for the normal mill pulps from the three sample periods.

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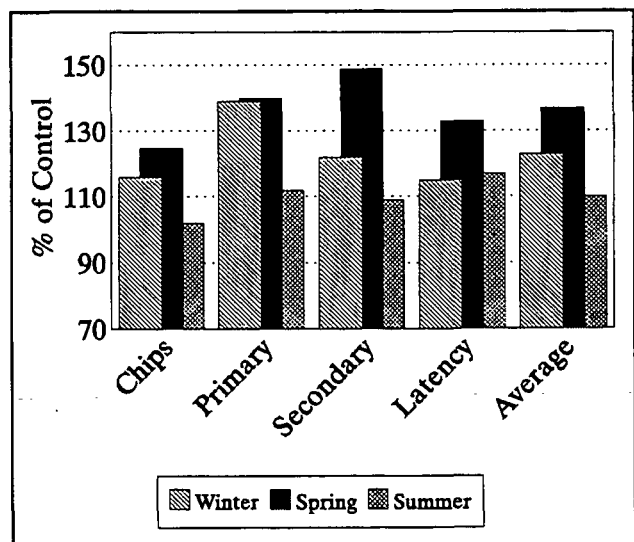


Figure 9. Sample tear index divided by the estimated control tear index at the sample tensile index, expressed as a %.

samples in that order.

The fiber length for the three sample periods is shown in figure 10. Fiber length gives a clear indication of fiber damage in the refiners. The fiber lengths reported in this study are Kajaani weight weighted averages and are carried out on latency relieved holopulped samples. The latency treatment reduces the fiber curl that develops in refining and the extended chlorite holopulping process. For the testing in this study, the winter and spring wood chips have slightly longer average fiber length than the summer samples. The fiber length decreases substantially between the wood chips and

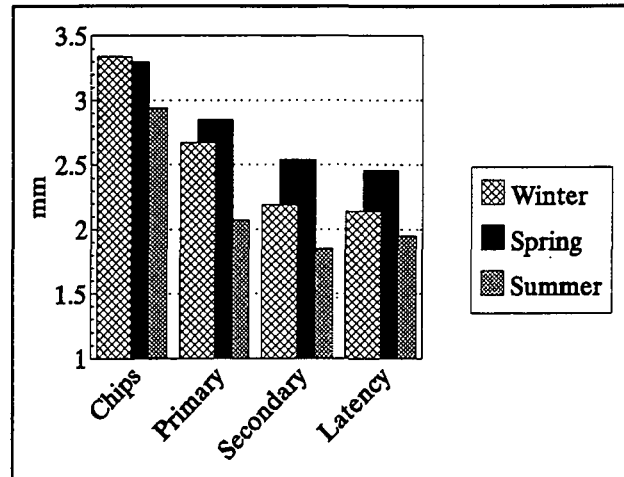


Figure 10. The chlorite holopulp fiber lengths for the controlled refining conditions.

primary refiners in both the winter and summer samples, confirming the fiber shortening expected in high yield refining. Unfortunately, there is little information of what was causing the fiber shortening in these samples. Primary refiner plate gap, motor load (divided by RPM) and plug wiper flow were all in the normal range for both periods. The closing pressure was higher on the primary refiner for both the winter and summer samples and was quite high for the normal mill samples collected in summer. This is probably representative of the higher plate hours on the refiner when these samples were taken. Chip compression damage from chipping frozen wood in winter, and dry wood in summer is a possible source of the problems and needs to be evaluated more fully. The loss in fiber length with refining may simply reflect the native fiber strength of the wood in the spring samples relative to winter and summer. Net fiber length retention from the wood chips to the secondary refiner is 77% in spring, 65% in winter and 63% in summer for the controlled production samples. The standard production samples had similar losses in fiber length in winter and spring, but the summer sample retained only 48% of the native wood fiber length through the refiners.

Wood Chip Analysis

The chip size distribution changed only slightly during the sample period, with the spring period having the largest retained 1/2 and 1/4 inch fractions.(figure 11) There did not appear to be a significant change in the pass 1/4 inch fraction but there were considerably more chips retained on the 1 inch screen for both winter and summer samples. If the Bowater Mersey data is equally applicable to southern pine, this change in chip size distribution is sufficient to cause a 7 to 10% loss in burst strength.¹³ It is, however, unlikely that the increase in large chip content would reduce the native fiber strength of the wood and for this reason, the increase in large chips is not considered a likely source of the strength variation.

The mill TMP pulp test data shows a seasonal loss in both tensile index and tear index for the low strength periods. In contrast, carefully controlled studies of chip size confirm the loss in tensile strength but show relatively little change in tear index when increasing the larger chip content.^{14,15} This inconsistency suggests that chip size may not be the source of the reduced pulp strength and may instead be caused the changes in the wood supply responsible for the low

pulp strength.

The reason for the broader chip size distribution in winter is presumably chipping of frozen wood.¹⁶ The reason for the broader chip size distribution in summer may have been worn chipper knives or dryer wood. Whereas the winter chips were received at a moisture content of 120% (moisture on dry wood basis, 45% consistency) and the spring wood chips at 109% (48% consistency), the summer wood chips were received at just 84% moisture content (54% consistency). Further analysis of these chips has been carried out with the intent of determining if there was a change in wood quality typical of a reliance on more plantation wood in Winter. The density and average growth increment were measured on the retained 1/2 and retained 3/4" size fractions. The average results are given in table 5.

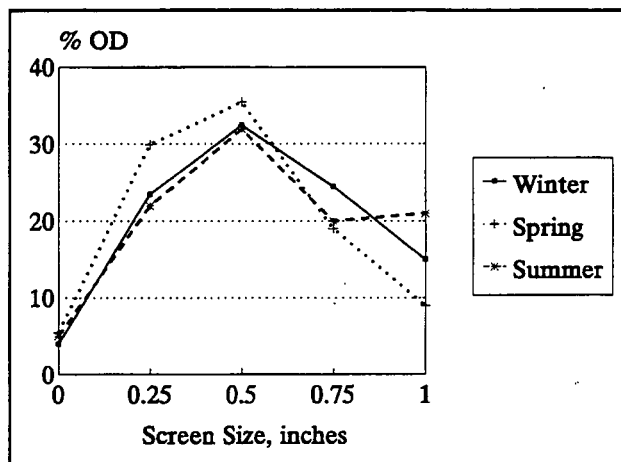


Figure 11. Chip size distribution for the Winter, Spring and Summer samples.

The difference in density between the three periods is small and not as large as would be expected with a significant shift in wood source. The growth ring widths are also quite similar at 3 to 4 mm and are only significantly higher in the 1/2" size category. Typical plantation wood has growth rings of 6 mm or larger. Approximately 10% of the growth rings measured in the spring wood samples are in this size range compared to 12% for both winter and summer and 19% for the second winter sample used as a control.

Table 5: Wood chip density and average growth increment.

	Density g/cc	Growth Increment, mm				
		3/4" Average	RMSD	1/2 "Average	RMSD	% ≥ 6mm
Control	0.433	4.15	1.72	4.09	1.48	19
Winter	0.437	3.77	1.01	3.70	1.48	12
Spring	0.446	3.71	1.50	3.36	1.47	10
Summer	0.443	3.93	1.76	3.17	0.994	12

Although none of these differences appear to be particularly significant, the ranking by percent of large growth rings matches ranking by wood densities, and both density and % of large growth rings match the relative order of native fiber strength for the four periods. When the wood density is plotted against average zero span tensile index, a straight line relationship is obtained with an R² of 0.94 (figure 12). Based on the results of a CHYPS project on the influence of wood growth and density on southern pine TMP (April 91 PAC), a 0.03 g/cc

increase in wood density can increase burst index by 40 to 50% and tear by about 15%. The 0.01 g/cc change in density observed in the Bowater wood chips should change burst by 10 to 15% and Tear by about 5%.¹⁷ These changes are within the range observed in the Bowater samples.

Conclusions:

Using the holopulping process to evaluate fiber strength losses in thermomechanical pulping has succeeded in tracing the seasonal strength loss observed by the Bowater TMP mills to a decrease in wood fiber strength during the winter and summer. This fiber weakness shows up as low wood chip and refiner holopulp zero-span tensile index and low holopulp tear index at constant tensile index. Fiber length is also lower in the winter and summer samples. An analysis of the wood chips for the three seasons evaluated shows a slight increase in oversized chips during the winter and summer periods, and a decrease in the wood density of the winter and summer samples. In addition, there is an increase in the number of large

growth rings observed in winter and summer, indicative of a higher juvenile wood or plantation wood content. The change in wood density observed is sufficient to induce the changes in strength observed in the mill. It needs to be emphasised that the fiber strengths observed in the TMP holopulps greatly exceeds the strength obtained in mill TMP. The mill paper strength is still bond strength limited in these samples, and an appropriate choice of refining conditions may provide the bond strength and paper strength desired.

This project has provided an excellent test of the chlorite holopulping technique and its utility on mill scale projects. The changes observed in pulp strength in this project appear to be at the resolution limits of the technique. However, the technique demonstrates low wood chip fiber strength during the periods of low TMP strength. The changes observed can be traced to changes in wood density and the size of the annual growth ring and are consistent with other observations on the influence of juvenile wood on pulp quality.

Acknowledgement:

We would like to express our thanks to Bob Harley of Bowater Corporation, and the Bowater Southern mill in Calhoun Tennessee for providing an interesting problem and the samples to study it. We thank the TMP mill operators and staff for their effort to reproduce the controlled refining conditions on a periodic basis and Melanie Gray and Shawn Wendell who collected all the samples and mill data to support the project. Thanks also to Jim Turnbull and Peter Joyce

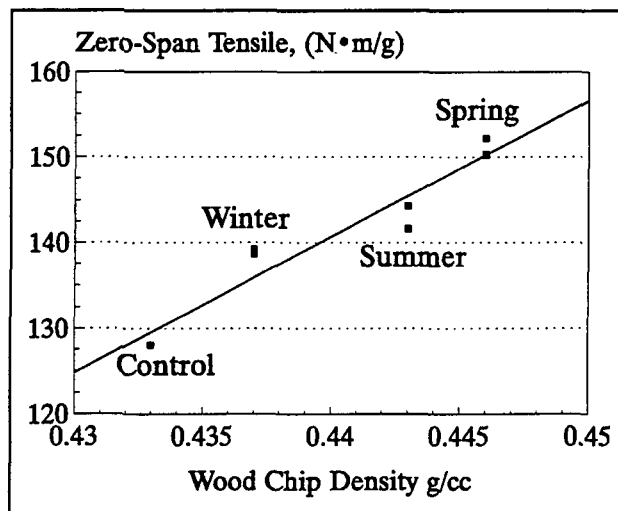


Figure 12. Average wood chip density influences chlorite holopulp fiber strength. $R^2 = 0.94$.

of MacMillan Bloedel Ltd. for providing the impregnator samples and background information. Finally, we thank the members of the Mechanical Pulping Project Advisory Committee for their advice and support of the project and for the member companies of the Institute of Paper Science and Technology who provided the financial support for the project.

Experimental Procedures:

Chlorite Holopulping:

A stock buffered chlorite solution is prepared as follows: To a 1 liter volumetric flask, add approximately 500 ml of distilled water, 60 g (0.66 moles) of reagent grade sodium chlorite and 60 g (0.72 moles) of reagent grade sodium acetate. Dilute to the mark with distilled water. This reagent is photosensitive but is otherwise stable and can be stored for several months if kept in the dark.

To delignify wood chips, place 50 g (OD basis) wood chips in a 500 ml Erlenmeyer flask. Add 300 ml of buffered sodium chlorite solution and 20 ml of reagent grade glacial acetic acid. Attach the flask to a vacuum source and evacuate for five minutes (make sure the vacuum has a trap to prevent water vapor and ClO_2 from contaminating the pump oil). Turn off the vacuum source and slowly allow air into the flask. Place the flask under vacuum for another five minutes and release the vacuum to air again. Stopper the flask and place in a dark cabinet underneath a fume hood. After 30 minute to 1 hour, a distinct yellow green color should be observed indicating that chlorine dioxide is forming in the flask. At room temperature the wood chips will take 4 to 5 days to consume the majority of the chlorite. The sodium acetate buffer will maintain the pH around 4 for the duration of the reaction. Once the majority of the sodium chlorite/chlorine dioxide is consumed, filter the wood chips and wash once with distilled water. Transfer the chips back into the Erlenmeyer flask and add sufficient distilled water to cover the chips. Let the wood soak in the distilled water over night and filter again or decant the wash water. Add 200 ml of 0.15 N NaOH, cover the Erlenmeyer flask and let the wood soak for 24 hours. Filter or decant the alkaline solution and check the pH. If below 9.0 repeat the caustic extraction by adding another 200 ml of 0.15 N NaOH and letting the chips soak another 12 to 24 hours. Filter and wash the chips thoroughly. Place them back into the Erlenmeyer flask and repeat the chlorite procedure as described above with the exception of using 200 ml of chlorite stock solution and 20 ml of acetic acid.

The second chlorite procedure is followed by a second wash and caustic extraction. At this point the chips are broken up in the British Disintegrator (3000 revolutions). A third chlorite treatment is performed using 200 ml of stock chlorite solution and 20 ml of acetic acid. Since the wood/fiber is saturated, the vacuum step is ineffective and can be skipped. Follow the third chlorite treatment with another caustic extraction. Wash and disintegrate for 3000 revolutions in the British Disintegrator. This will render most of the remaining chips to shives and fiber. Once the chips are broken into fibers, the filtrates tend to plug filter paper and a 150 mesh wire screen is substituted for filter paper in the Büchner funnel. A fourth chlorite stage, extraction and a final chlorite treatment are required to complete the pulping process. The pulp is then washed thoroughly and disintegrated another 3000 revolutions.

During this project period, a hot chlorite delignification procedure exploded in the Research Services Division and it was decided to perform an additional safety check on this procedure. Blank chlorite samples were prepared substituting dilution water for the mass of the wood chips. The samples were otherwise handled in the same manner as the normal chlorite delignification procedure and were checked daily for the chlorine dioxide concentration. Under these conditions, the chlorine dioxide concentration never exceeded 0.6 gpl which is considered well below the limit at which ClO_2 can form an explosive atmosphere at room temperature.¹⁸ It was concluded that the procedure was safe and we have

continued using it with only slight modification.

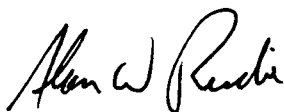
Fiber length was determine using the Kajaani FS-100 optical fiber length analyzer. Samples were screened on a 0.008" Valley slot screen prior to making handsheets. Handsheets were prepared (TAPPI T-205) and tested for tensile index, tear index, scattering coefficient and absorption coefficient according to TAPPI 220. Zero span tensile index was carried out according to TAPPI T-231.

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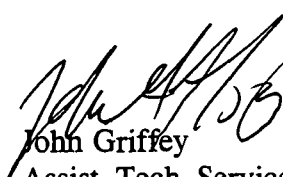
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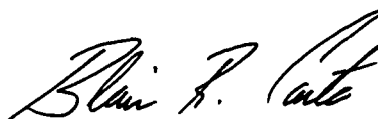
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Appendix

Mill Operating Conditions and IPST Pulp Test Results

Primary	Standard			Controlled		
	Jan.	April	July	Jan.	April	July
Days High ° C	12	28	37	12	28	37
Days Low ° C	-2	15	22	-2	15	22
Plate Hours	275	21	445	279	22	447
Production (RPM)	51	41	45	51	42	51
Load (MW)	7.1	6.6	6.1	7.2	6.8	7.5
Gap (mm)	6.68	5.51	7.92	6.73	5.56	7.32
Closing Pressure (kPa)	4300	3600	6200	4300	4000	5000
Freeness (ml)	707	636	742	665	621	750
Secondary						
Load (MW)	5.4	5.7	5.8	5.7	5.9	6.5
Gap (mm)	5.05	5.10	7.29	5.16	5.18	7.26
Closing Pressure (kPa)	2300	2100	3800	2900	2100	7400
Freeness (ml)	236	178	183	244	176	363
Tensile Index N·m/g	15.1	22.2	17.5	14.9	21.0	16.7
Tear Index mN·m ² /g	4.47	7.16	4.71	4.97	6.74	5.12
Burst Index kPa·m ² /g	0.71	1.05	0.75	0.75	0.95	0.66
Brightness	56.0	58.9	55.8	54.7	59.3	55.3

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