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Pulp Viscosity vs. Zero Span Tensile for Determining Pulp Strength

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PULP VISCOSITY VS. ZERO SPAN TENSILE FOR DETERMINING PULP STRENGTH

BY

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ABSTRACT

Pulp viscosity and zero span tensile tests can be used to predict the ultimate fiber strength of pulp fibers. A project to compare the traditional test with the newer zero span tensile test was completed. In order to compare both tests for precision, bleached softwood kraft pulp was disintegrated and made into handsheets. The handsheets were exposed to 10N HCL vapor from a few minutes to a few hours to get a range in fiber strength since the HCl degrades the cellulose. Pulp viscosity and zero span tensile tests were completed on the handsheet paper samples. It was observed that both test methods showed a correlation with predicted decreased fiber strength and time in HCl vapor. In order to determine which test method is superior, variables such as precision, ease of use, quickness, maintenance, and cost were considered. The pulp viscosity test was observed to be more precise than the zero span tensile test when giving an indication of the fiber strength of a handsheet.

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INTRODUCTION

For years industry has used pulp viscosity as the standard for pulp strength measurement. Unfortunately, pulps with the same viscosity may produce different handsheet properties depending on their pulping and bleaching conditions.(1)

It has been shown that the zero-span tensile test can also be used to measure the intrinsic strength of pulp fibers.(1)

Both the standard pulp viscosity and zero span tensile methods will be compared to see which test should be recommended for use in the pulp and paper industry.

BACKGROUND DISCUSSION

Pulp Viscosity Method

The measurment of pulp viscosity, or more correctly, the viscosity of a solution with cellulose as the solute and cupriethylenediamine (CED) as the solvent is of prime interest in today's pulp testing laboratory. The viscosity of these solutions have been observed to be directly proportional to the DP or *degree* of *polymerization* of cellulose molecules. The test relates to the degree of shortening of the chain when subjected to chemical attack such as in cooking and/or bleaching. Thus, the viscosity test is generally recognized as one of the important indicators of pulp strength.(2)

Historically, the original idea for measurement of pulp solution viscosity dated back to the 1800's. In the 1850's, Mercer and Schweizer(3) observed that cupraammonium acted as a solvent for cellulose. However, nothing major happened until 1928 when Clibben and Geake(3) showed that the measurement of the rate of flow of a solution of cupraammonium

offered a useful method for detecting the loss of strength caused by chemical attack of cellulose in the bleaching of cotton textiles. This opened the door to much of the present day knowledge of cellulose systems. (2)

Work by F.L. Straus and R.M. Levey(2) using cupriethylenediamine (CED) as a solvent in the 1940's laid the foundation for many of the current methods used today. As was mentioned earlier, the viscosity test gives a measure of the average degree of polymerization of the cellulose, which is related to the degradation (decrease in molecular weight) resulting from the pulping and/or bleaching process.(2)

Experimentally, the measurement of the viscosity of a cellulose solution is one of the simplest of all physical methods. Hence, it is used as a gage of the amount of degradation of cellulose.(2)

Research has shown that the number of glucose units forming a cellulose molecule is usually from 600 to 1000 for wood pulp. The weighted average DP is used for control tests. The intrinsic tensile strength of fibers, and particularily the stretching ability depends on their DP.

In TAPPI T-230, the official viscosity test method, it is noted that "caution should be exercised in drawing conclusions about pulp strength properties strictly from viscosity results unless previous investigation has identified the relationship."(3) This is because there are five principal pulp properties (as mentioned by Clark)(1) that might make the pulp unsuitable for papermaking. These properties include intrinsic fiber strength, fiber length, coarseness, wet compactability, and cohesiveness.

The viscosity test essentially consists of weighing out a pulp sample of 0.250 g oven dry fiber(hence moisture must be determined), then adding the CED solvent and shaking for a few minutes. The solution is then mixed

and 10 ml is added to a glass viscometer. The glass viscometer is in a 25°C water bath. The solution is brought up above a line on the viscometer by suction and the vacuum is released. The time it takes for the solution to cross between both of the lines is a function of its viscosity which is related to cellulose D.P. This procedure is described in Tappi T-230.

Since the CED Viscosity test is somewhat time consuming many shortcuts are taken in industry with some error involved.

Early Zero Span Tensile Tests

In 1923, as was noted in an article in Svensk Pappers-Tidning(1), there was evidence that the intrinsic strength of fibers was connected with a special tensile test of paper if the test span could be reduced to one that was suitably small. It was observed that the breaking load was much higher for the closed span test than of the traditional tensile test.

Later it was observed that the zero-span breaking lengths correlated well with the strenth qualities of eight different pulps which were tested in a number of ways.(1)

Years later, improvements in the apparatus and slight modifications in the procedure followed. In 1950, the Institute of Paper Chemistry (IPC) designed a more elaborate instrument.(1)

In the early days there were problems associated with the gripping pressure and jaw wear of the apparatus. These were corrected and today's zero-span apparatus was perfected.

The zero-span tensile apparatus is believed to be a function of the intrinsic strength of the fibers. Intrinsic fiber strength is fiber strength not only of the body of the fibers but also of their fibrils and thus has an important influence on the strength of paper made from the pulp. The tester uses an opposing pair of jaws to grip a 15 mm wide paper strip in

close contact so that all or most of the fibers lying across the line of contact between the jaws are ruptured.(1)

A compressed air operated instrument for rapidly measuring the short span tensile strength was designed by Cowan and is shown in Figure1.(1) This instrument is described in Figures 1 and 2.

Clark states(1), "In order to obtain the true zero span breaking lengths, it is necessary to make several tests with known, slightly increasing spans to about 1mm, and to extrapolate the resulting line or curve to the true zero span." This is shown in Figure 3.

It was also observed that the zero-span tensile and degree of polymerization were related. Data from Baker(1) showed that a relation exists between a pulps zero span breaking length and its viscosity after treating it with 2N HCl for 1/2, 7.5, 15, and 40 hours at 50^0C. This is shown in Figure 4.

It was observed by Swenson(4) that a linear relationship existed between zero span and weight average DP but not number average DP. As was noted earlier, the viscosity test gives an indication to the weight average DP.

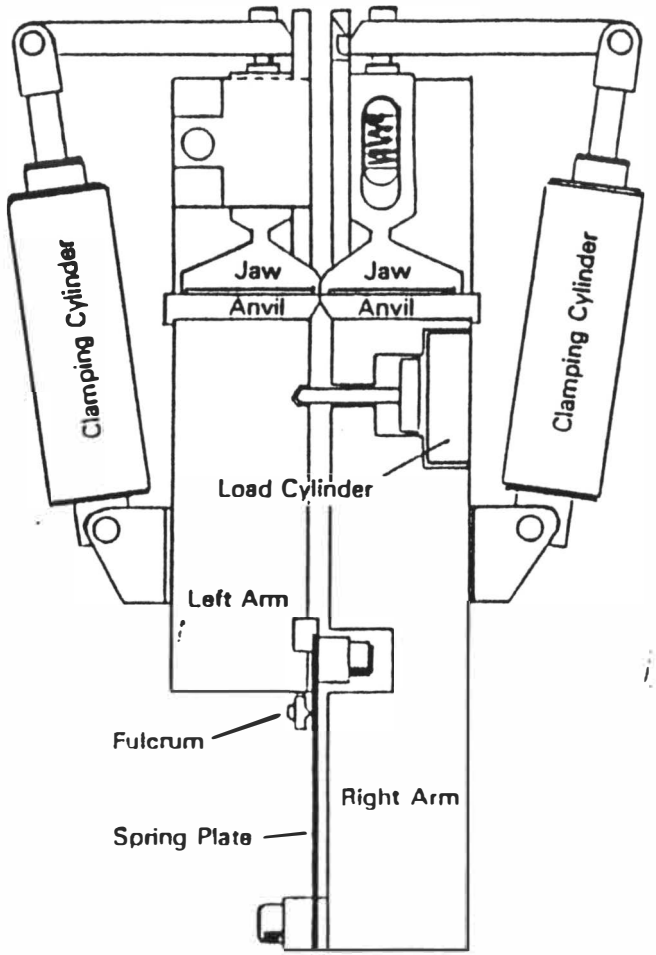
In the past there has been contradictory views of the efficiency of the zero-span tensile test.(5) The gripping pressure of the jaws must be high enough to prevent slippage of the fibers, but high pressures are found to damage the fibers. Basis weight is found to influence the zero-span test. It was observed that a basis weight of 40 grams/m^2 resulted in the highest zero-span tensile. But, the standard 60 g/m^2 is often used since its difference from the 40g/m^2 is only a few percent.(1)

In 1978, Ionides(6) indicated that the tear, tensile, and burst strengths of chemical newsprint can be predicted by short span tensile.

Cowan(7) noted that tests for fiber strength, fiber length, fiber

FIGURE 1. The Zero Span Tester.(11)

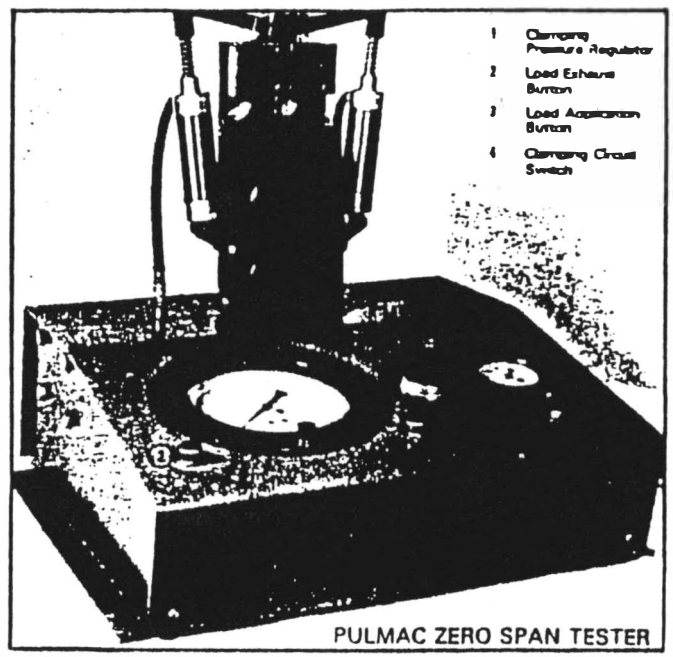
THE PULMAC ZERO SPAN TESTER



Schematic Illustration

The paper strip is inserted horizontally between the jaws and anvils of the Zero Span Tester. A plastic jig facilitates the insertion of wet strips.

Turning on the clamping switch causes pressurization of the two clamping cylinders which, acting through the linkage arms, cause the clamping jaws to descend and clamp the paper strip. The clamping pressure is indicated on the small panel gauge and can be readily altered to any desired level by adjusting the pressure regulator on the panel.



PULMAC ZERO SPAN TESTER

FIGURE 2. Zero Span Tester. (11)

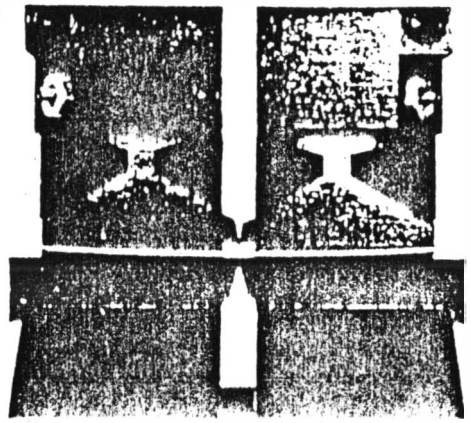
AND HOW IT WORKS

Depressing the load applicator button causes air to flow at a controlled rate into the load cylinder located in the stationary right arm of the tester. This causes a piston to exert a force on the opposite left arm. This arm is held in place by a spring plate and is free to rotate about a fulcrum. Such rotation is resisted by the clamped paper strip and thus a tensile load is built up in the specimen. This tensile load is directly proportional to the pressure in the load cylinder which is indicated on the large panel gauge. At failure, the maximum reading is preserved on this gauge and the tensile failure load in lbs or kg per 15 mm is read from a calibration chart.

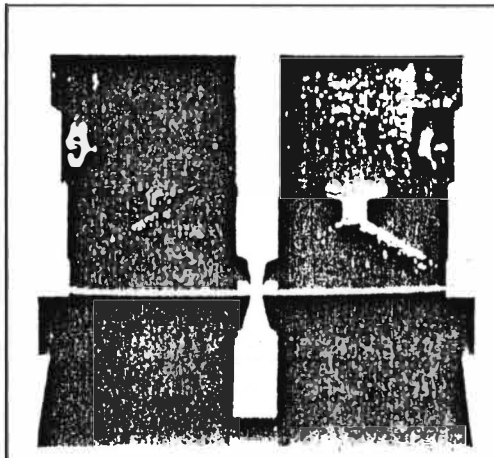
Switching off the clamping circuit exhausts the clamping cylinders and internal springs in the jaws of the tester cause them to lift up. The paper strip is removed. Depressing the load exhaust button causes the left arm to return gently to its rest position and zeros the pressure gauge.

A set of feeler gauges can be mounted on the right arm of the tester using a magnetic clamp. Any particular gauge can then be rotated upward until it is located so that when the left arm of the tester returns to its rest position, it will be separated from the right arm by the thickness of the feeler gauge. In this way, the instrument span can be accurately set at a variety of spans in the range from zero (jaws in contact with each other) to a few millimeters.

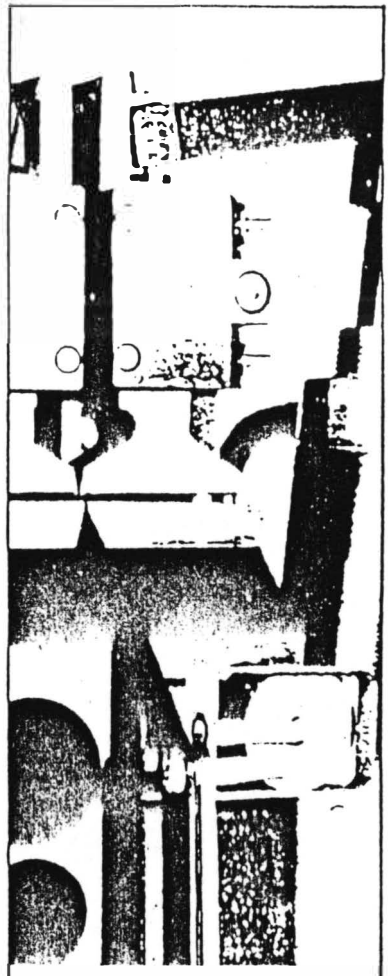
Individual tests (typically 500 in a normal working day) can be run at various spans to provide the short span tensile data required for analysis.



Jaw position at Zero Span



Jaw position at Finite Span



Feeler Gauge Assembly

THE
SHORT SPAN TENSILE CURVE

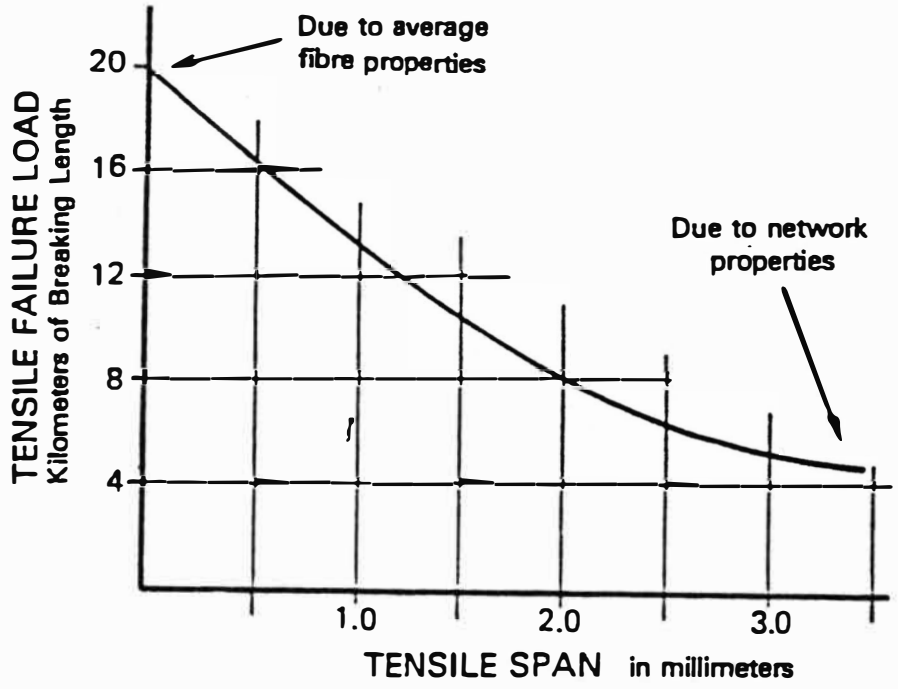


FIGURE 3. Short Span Tensile Curve.(11)

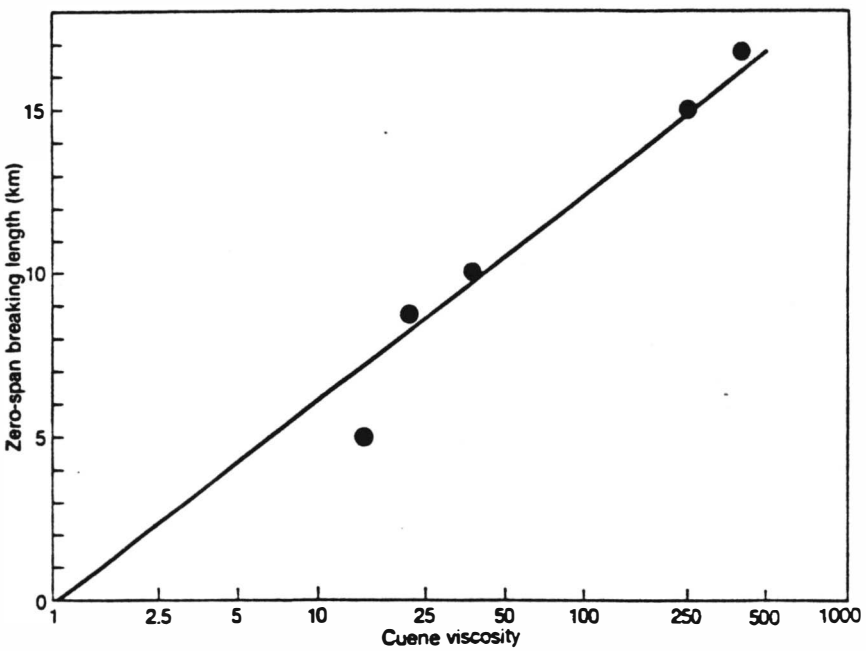


Figure 4 *Viscosity 1969 TAPPI Reference sulfate pulp and zero-span tensile.* (1)

alignment, and fiber bonding can be used to rapidly assess the above parameters of paper using the zero span tensile tester. He said that its limitations are that the same grade must be used and that heavy basis weights should not be used because the mass of the fibers between the jaws defeates the ability to securely clamp all the fibers.(7)

Smook(8) notes that the strength parameters which combine the measurement of fiber strength and bonding strength can and should be monitored daily for the production of pulp.

It is known that paper strength depends on the strength of the fibers, strength of the fiber to fiber bonds, number of bonds, and distribution of bonds as indicated by fiber distribution or sheet formation.(9)

Boucai (10) noted that the only other way to measure fiber strength is by single fiber testing. Unfortunately, this does not indicate the behavior of the fibers as they are found in paper. It should be noted that this is often a tedious and time consuming approach.

Process Control

A number of processes separate the finished product at the end of a paper machine from the trees which provide its primary raw material. Most of the processes are controlled by measuring and maintaining at constant levels the important input variables such as consistency, temperature, etc. Rarely does a property of a pulp affected by a process get measured and result in process adjustments.(11)

It has been recognized that the desired paper properties are due to the constituent fibers themselves and their arrangement in the sheet. Therefore, the goal of process control should be to control the wood room, pulping, bleaching, and stock preparation processes in order to develop appropriate fiber properties.(11)

Testing For Fiber Strength

In order to get an indication of fiber strength by the Cowan method, a 15mm wide test strip is soaked in water allowing the fibers to become unbonded. Therefore, only the strength of the fibers are measured and not the fiber to fiber bonds.

The wet zero span test value is determined by (1) the number of fibers clamped, (2) the orientation of the fibers relative to the direction of loading, and (3) the average wet strength of the individual fibers.

By maintaining constant basis weight sheets and random fiber orientation in the laboratory, the wet zero span value can only be due to changes in the strength of the fibers.(11)

Poor Performance Papermaking

Paper being produced in which the profitability falls significantly below normal causes much concern for the papermaker. There are four cases when this can happen.(11)

(1) When the paper being produced exhibits poor quality.

(2) When excessive breaks occur.

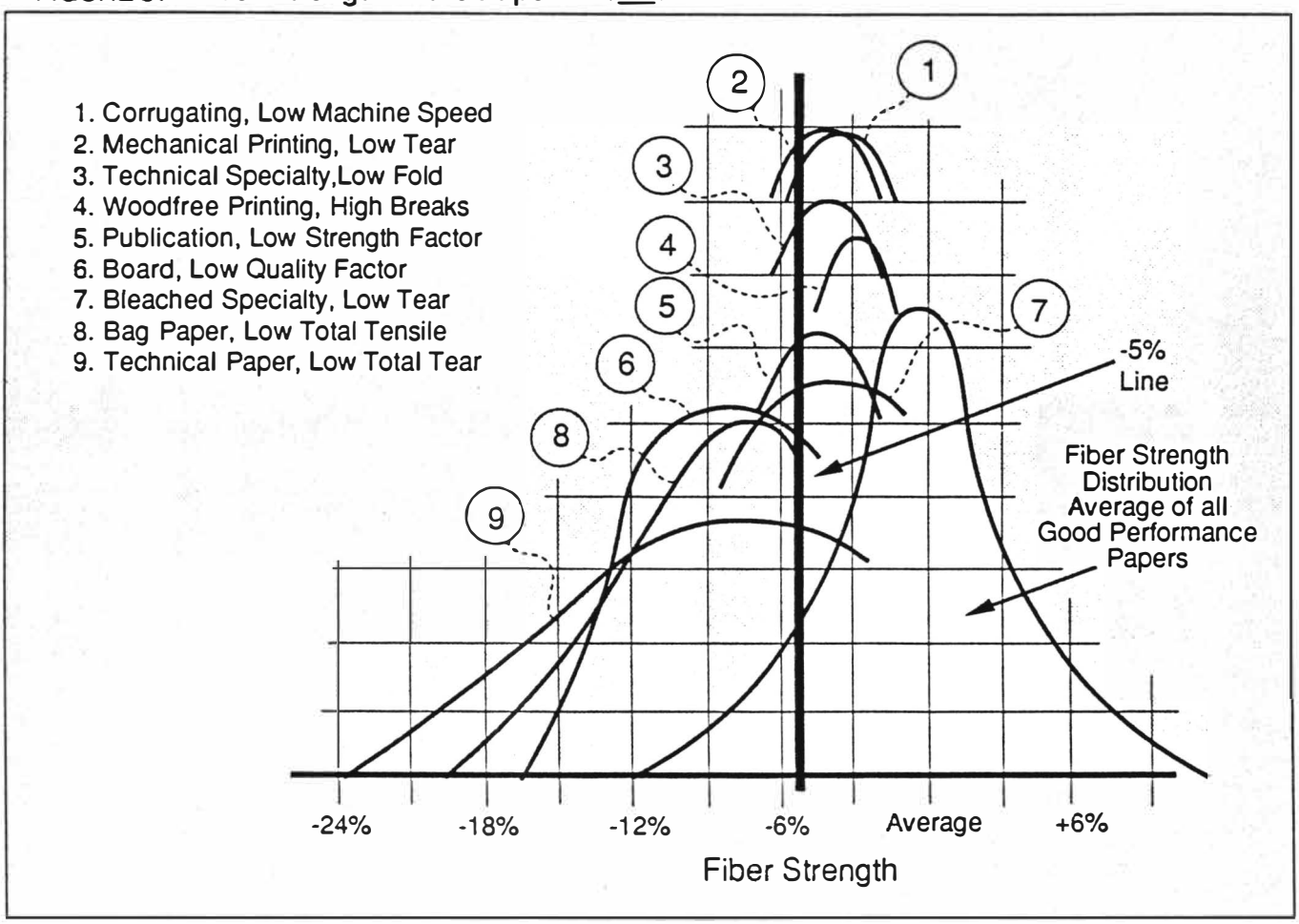
(3) When a mill is forced to reduce machine speed in order to maintain proper quality standards.

(4) When a mill must use more kraft softwood in order to maintain satisfactory runnability.

If any of these cases happen to the papermaker, profitability will be reduced.

Papermill studies conducted by Pulmac have shown that poor performance papermaking is two to eight times more likely to occur if fiber strength falls more than 5 percent below the average value for good performance papermaking. Figure 5 presents data from nine different mill

FIGURE 5: **Fiber Strength in the Papermill(11)**



Loss of fiber strength can only occur in the chemical processes of pulping and bleaching. Once the pulp leaves the pulp mill, fiber strength is fixed and cannot be changed by any of the subsequent stock preparation or papermaking processes. The process control response to low fiber strength is to take fiber quality testing back into the pulp mill and discover why and when the pulp mill is producing lower fiber strength pulp.

studies in which standardized fiber strength distributions for poor performance papers are shown in relation to a comparable average distribution for all good performance papers. It should be noted that loss of fiber strength can only occur in the chemical processes of pulping and bleaching. Once the pulp leaves the mill, fiber strength is fixed and can't be changed by any of the subsequent stock preparation or papermaking processes.(11)

Pulmac states(11) that the fiber strength value (the wet zero span value of a 60 g/m^2 test sheet) of pulp from anywhere in the pulp mill can be readily determined using the Pulmac zero span system. Once frequent fiber strength testing is added to support process improvement in a pulp mill, specific process problems which are likely to cause an exceptional loss in fiber strength can be identified.

One mill noted that poor fiber strength was found (5% below average or more) when the H factor increased much above a particular normal level or when its sulfidity fell below its normal level.(11)

The distinction between poor and acceptable paper quality can usually be traced to its origin by tracing the poor zero span test results back through the pulping process.(11)

Unfortunately, until fiber quality testing is introduced as a process control tool, no substantial reduction in poor performance papermaking is likely to occur.(11)

In theory, an examination of the short span tensile curves for wet sheets of paper can provide information about the average strength of the constituent fibers, the extent to which they are oriented, their average length and the nature of their length distribution.(11)

In order to find out which test method is superior, both methods will be rated based on their precision, ease of use, quickness, space

requirement, cost, and maintenance.

EXPERIMENTAL PLAN

Canadian bleached softwood kraft pulp was disintegrated and made into pulp handsheets at a basis weight of 60 grams/m^2. The sheets were made using the British Sheetmold, and conditioned in a constant temperature and humidity control room.

The handsheets were subjected to 10N HCl acid vapor by placing them in an desiccator with HCl in a beaker. The vapor went out of the beaker and through the handsheets to the desiccant in the bottom of the desiccator.(12)

This treatment reduced the zero span tensile strength of the sheet by the hydrolysis of cellulose. After exposure, the acid was thoroughly removed from the handsheets by aeration, and no further reduction in the zero span tensile strength of the sheets occurred. This treatment has little effect on sheet properties such as apparent density and scattering coefficient which depend primarily on fiber geometry and sheet structure. It also has no effect on the elastic modulus of the sheet because the attack on the cellulose is localized.(12)

The sheets were exposed to the vapor for times ranging from a few minutes to a few hours. After aeration, the sheets were tested by the pulp viscosity and zero span tensile tests. It was hoped that the results would show definate trends.

Initially the holopulping method(13) was to be used in this experiment. Sodium chlorate and acetic acid were to be reacted togther to produce chlorine dioxide which would be applied to wood chips in order to produce pulp. This method was not used because the HCl vapor procedure provided a quicker laboratory method.

Paper strength tests were completed on the handsheets and the results were correlated with the viscosity and zero-span tensile results in order to see which test is better at predicting fiber strength.

In order to make a comparison, handsheet strength (viscosity and zero-span readings) was plotted at the different D.P. values(time). This is shown below.

RESULTS

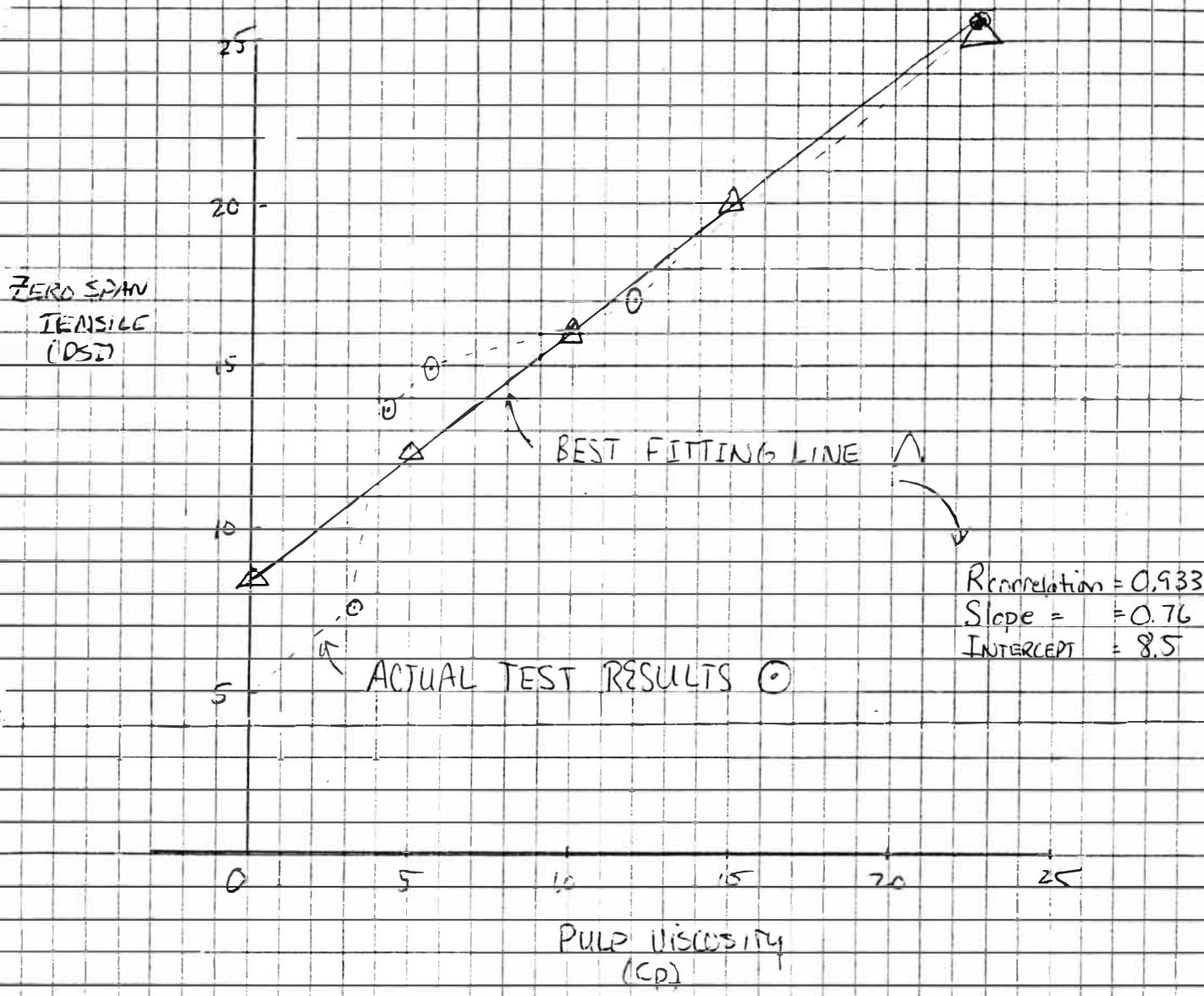
Time in HCL vapor minutes	Pulp Viscosity* cp	Zero Span tensile(psi)	Breaking length(km)	Tear gf
0	22.65	25.7	1.91	80
5	11.95	17.0	---	---
7	5.67	15.0	---	---
15	4.31	13.8	1.80	36
60	3.67	7.75	1.35	28
1day	very low	very low	---	---

The standard deviation for the base sheet (0 min) was 1.18 cp for the pulp viscosity test and 2.74 psi for the zero span tensile test. This corresponds to an percent error of 5.2 percent for the pulp viscosity test and 10.7 percent for the zero span test. Overall, the pulp viscosity test had a percent error around 5 percent at each of the different times.(as shown in the appendix) The zero span tensile test had errors up to 20 percent.(shown in appendix)

*Usually, acceptable pulp has a viscosity of 15 cp or higher.

Fig. 6

PULP VISCOSITY VS. ZERO SPAN TENSILE



DISCUSSION

The results showed that the HCl vapor did indeed make it possible to predict the decrease in expected fiber strength with increased times in the HCl. It was observed that both the zero span tensile and pulp viscosity readings decreased as the time in the HCl vapor increased. This showed that both the zero span and pulp viscosity tests can be used to give an indication of pulp fiber strength.

It was observed that after about 5 minutes of HCl exposure the pulp viscosity and zero span tensile readings of the sheets go way down. It was also interesting to note that the pulp viscosity results dropped at a faster rate than the zero span results. This could be because the pulp viscosity test might be more sensitive to pulp degredation than the zero span test.

A regression (R) correlation was completed on the pulp viscosity and zero span results. The R correlation observed for the results was 0.933.(shown in appendix) This shows a very good positive correlation between both sets of results. The curve of pulp viscosity vs. zero span tensile is shown in Figure 6. The best fit for the curve is also shown and it has an intercept 8.5 and a slope of 0.76.

The handsheets were tested for tear and tensile and the results indicated that their strength properties decreased with decreasing pulp viscosity and zero span readings. The R correlation between pulp viscosity and tear was 0.99 and the R correlation between zero span tensile and tear was 0.98.(shown in appendix) This shows that both tests have a good positive correlation between predicted fiber strength and the tear of the handsheets. An R correlation of 0.67 was observed between pulp viscosity and tensile breaking length and a correlation of 0.87 was observed between zero span tensile and tensile breaking length. Even though the R

values were lower for the tensile results they still show good positive correlation.

PROBLEMS

Unfortunately, some of the handsheet samples were not aerated long enough to allow all of the HCL vapor to escape from them. This made the sheets turn black when placed in the laboratory oven causing the sheets to become brittle.

Final Comparison

Both tests were compared on a number of variables. The variables are listed below and the test which was the best suited was given a "+" while the test not as good was given a "-".

PRECISION

The results showed that the viscosity test was repeatable to a standard deviation of 1.18 cp which is within 5.2 percent of the mean for the basesheet. The Zero span tensile reading showed a standard deviation of 2.74 which is within 10.7 percent of the mean for the basesheet. Overall, the pulp viscosity test showed percent errors less than 6.5 percent(shown in appendix) and the zero span tensile test had percent errors of up to 19 percent.(shown in appendix)

Score: Scale + or -. (+ being good and - poor)

Since the pulp viscosity test had the best precision, it received the best score.

Pulp Viscosity: +

Zero Span Tensile: -

QUICKNESS/EASE OF USE

Both tests can be done in 30 minutes. The pulp viscosity test doesn't require a handsheet. A sample of pulp can be disintegrated using a blender and made into a Buchner pad with relative ease. But, a 60 g/m^2 handsheet must be made for the zero span tensile test. This makes the test a little more time consuming. The pulp viscosity test requires the use of chemicals and mixing which has to be done perfectly in order to get an accurate reading. Production workers can easily understand both tests in a short period of time. Since both tests are about the same in simplicity and quickness they both receive the same score.

SCORE:

Pulp Viscosity: +

Zero Span Tensile: +

SPACE REQUIREMENT

Both instruments take up about the same amount of space, so they receive the same score.

Score:

Pulp Viscosity: +

Zero Span Tensile: +

COST

The cost of the lowest model zero span tensile tester is about $18,000.00 and $50,000.00 for the top of the line model from Pulmac Instruments. Zero span tensile attachments can be purchased for the standard tensile tester with significant savings. Maintenance costs run

very low per year because of the air driven nature of the instrument.

The pulp viscosity test requires a glass water bath, glass viscometers, CED, cleaners, a water heater, pipets, stirers, shakers, stop watches, thermometers, and an oven. All of this together might cost a few thousand dollars, which is still much cheaper than the Pulmac zero span tester. The upkeep consists of new CED, plus the replacement of broken glass viscometers, which occur due to mishandling.

Due to the low cost of the pulp viscosity equipment it is possible to have one in each lab of the mill. But, due to the higher cost of the zero span tensile tester, it is likely a mill can only afford to have one and it would probably be located in a central lab. This problem can be solved by purchasing the zero span attachment.

With the attachment to the tensile tester, both instruments are comparable in costs.

SCORE:

Pulp Viscosity: +

Zero Span Tensile: +

Total Score:

Pulp Viscosity: 5+

Zero Span Tensile: 2+

After totaling up the points in this unofficial test it was observed that the pulp viscosity test received the highest score. The preferred method should not only be precise but cost effective, easy to use, and easy to maintain.

PROBLEMS

Unfortunately, a lack of readings for the different testing methods occured due to the slow nature of receiving the proper lab equipment.

In order to get more precise readings more viscosity and tensile tests should have been done. But, the data that was available showed good correlation with what was expected.

CONCLUSIONS

After looking at the results, it was observed that either the pulp viscosity or the zero span tensile tests can be used to give a prediction of pulp fiber strength. The final decision on which procedure to use should be left up to the mill. But, after looking at the precision of the results, it should be noted that the pulp viscosity readings precisely predicted the decrease in expected fiber strength. Its major plus is that it's precision is within 6.5 percent. One advantage of the Pulmac zero-span tensile tester is that it can predict fiber strength, fiber bonding and fiber length at the same time. Even though the pulp viscosity test has been around for a long time it is still the least expensive and most precise at predicting pulp strength.

RECOMMENDATIONS

A mill currently using the pulp viscosity test for predicting fiber strength should continue to do so, but if it would like to look at other variables such as fiber bonding or fiber length, the zero span tensile test should be considered.

References:

1. Clark, J. d'A., *Pulp Technology and Treatment for Paper*, 450-464, 1982.
2. Winslow, P., *Southern Pulp and Paper*, July 10, 1969. 80-90.
3. Tappi T-230, "Pulp Viscosity Test Method", Atlanta, Ga.
4. Swenson, H.A., *Svensk Papperstiding*, (3): 103, (1975).
5. Britt, K.W., and Yiannos, P.N., *Tappi* 47 (7) 427-431, (1964).
6. Ionides, G.N., *Paperi Puu* 60, (4a): 233-234, 1978. (Abs)
7. Cowan, W.F., BPBIF Conf. 119th: 14, 1980. (Abs)
8. Smook, G.A, CPPA Ann. Mtg. Preprints 70b: 249-254, (1984). (Abs)
9. Ibrahim, A.A., *Southern Pulp and Paper*, 47 (10):23-25,(1984) (Abs)
10. Cowan, W.F., *Pulp and Paper*, March 1972.
11. Cowan, W.F., "Pulmac Fiber Quality Testing", Pulmac Instruments Int., Box 50, Montpelier, Vt.
12. Seth, R.S., and Page, D.H., *Tappi*, 71 (2): 103-107. (1988)
13. Thompson, N.S., *Tappi*, 51 (3): 123-126, (1968).

APPENDIX I

STANDARD DEVIATION AND PERCENT ERROR OF TEST METHOD

PULP VISCOSITY

TIME	READING (CP)	n #	STANDARD (cp) DEVIATION	PERCENT ERROR
BASE (0min)	22.65	10	1.18	5.2
5 min	11.95	5	0.72	6.0
7 min	5.67	5	0.37	6.5
15 min	4.31	5	0.22	5.0
60 min	3.67	5	0.22	6.0

ZERO SPAN TENSILE

TIME	READING (PSI)	n #	STANDARD (PSI) DEVIATION	PERCENT ERROR
BASE (0min)	25.71	10	1.91	7.4
5 min	17.0	5	1.71	4.2
7 min	15.0	5	1.58	10.5
15 min	13.8	4	0.50	3.6
60 min	7.8	4	1.50	19.0

CORRELATION			Rcorr
PULP VISCOSITY	VS.	ZERO SPAN	0.93
PULP VISCOSITY	VS.	TEAR	0.99
ZERO SPAN	VS.	TEAR	0.98
PULP VISCOSITY	VS.	BREAKING LENGTH	0.62
ZERO SPAN	VS.	BREAKING LENGTH	0.87