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Characterization of Fiber Fines by Drainage Resistance Analysis

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CHARACTERIZATION OF FIBER FINES BY DRAINAGE RESISTANCE ANALYSIS

by

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ABSTRACT

This thesis begins by developing a literature background for a variety of pulp characterization methods and relating them to present-day techniques.

Two methods are evaluated and compared with the Canadian Standard Freeness (C.S.F.) test. They are the Centrifugal Water Retention (C.W.R.) method and the constant rate filtration resistance method using the Pulmac Permeability instrument. A standard pulp was used and the fines content was varied from 0% to 25% at 5% intervals. Handsheets were made and the pulp characterization values were related to sheet strength values.

In general, the data indicated that the specific surface area, from the constant filtration test, the C.S.F., and the C.W.R. values, tended to follow the same trends. These values also tended to follow handsheet strength tests. However, the specific volume value calculated from the filtration resistance test data was more precise in its correlation with the handsheet strength tests.

ACKNOWLEDGEMENTS

The author would like to express deep appreciation for the advice and counseling of Dr. Raymond Janes, Head of the Department of Paper Technology at Western Michigan University who was the advisor for this thesis. With his guidance much was accomplished in preparing literature and performing laboratory work as well as analyzing and explaining subsequent results.

Appreciation is also extended to Richard Houchard, a mathematics major at Western Michigan University, for his assistance in writing a computer program which greatly reduced the time required for calculation of data.

LITERATURE REVIEW

INTRODUCTION

While a considerable amount of literature was reviewed in preparation for this paper, only the most significant and relevant work will be covered extensively in this literature review.

The topics reviewed were, in most cases, found to be directed at specific areas related to the subject at hand. Many articles have been published pertaining to pulp evaluation methods using the techniques of centrifugal water retention and drainage resistance (1-17). No work was found relating both of these tests with the fines content of pulp or their relationship to fiber bonding and sheet strength.

PAST WORK

Through the years, much work has been done to devise a simple, fast and accurate test to replace and improve the Canadian Standard Freeness (C.S.F.) method of evaluating pulp. Several investigations using various techniques have been carried out, three of the most significant of which will be discussed here.

Centrifugal methods for estimation or determination of the hydration or water-holding capacity of pulps go back to at least 1942. Among those who have pursued this path most actively is Jayme, who, with his co-workers, has published a number of articles on the subject (1-3). In 1944, Jayme suggested a centrifuge water retention method that was adopted for use in some European mills as a control method. In 1948 the method was standardized, and in 1958, after reinvestigation, a revised method was introduced (2).

Among the important things Jayme concluded were:

(1) "Measuring the amount of water held by the dry substance of a pulp after centrifuging it under standardized conditions represents a very simple and accurate method for determination of affinity of pulps to water. It is termed 'water retention value' (W.R.V.)" (2).

(2) "During dispersion of dried pulp the disintegrator may exert an influence on W.R.V." (2).

(3) "A straight line relationship between W.R.V. and breaking length developed during beating of a pulp" (2).

At about the time Jayme, Ghaneim and Kruger (3) were publishing a paper giving results of comparisons of five different centrifuge cup assemblies for evaluation of beaten pulp, claiming a great amount of success with a thimble-shaped nickel fourdrinier wire, Thode, Bergomi, and Unson were evaluating a technique and device they had developed (4). The Thode, Bergomi, and Unson investigation used the conventional flat septum, and to avoid confusion, called their centrifugal water retention value C.W.R. instead of Jayme's W.R.V.

Thode found many results parallel to those of Jayme (4). He concluded that swollen specific volume of both unbeaten and highly beaten wood pulp fibers could be rapidly estimated with C.W.R. However, because of effects of incomplete removal of interfiber water and possible removal of some intrafiber water by fiber deformation, slightly different values were obtained from this method than from the filtration resistance method for estimating swollen specific volume. He also noted that the C.W.R. correlated well with tensile strength of handsheets.

In 1965, Yiannos (5) researched the swellability of pulps determined by centrifugal isopropanol retention. With this method it was thought that the centrifugal force

removed all solvent from the large pores of the fiber and from the interstices between the fibers, but it does not remove all of the solvent from the pores which were created by water swelling because they were quite small.

The results of Yiannos showed that the test was quite reproducible and able to detect differences among pulps not easily detected by freeness and fiber length values. The swellability test could detect differences among pulps dried in different sequence and from different liquids. The swellability test correlated very well with beating and strength properties, particularly tensile.

Another major pulp evaluation method which involved water removal from pulp slurries was the filtration resistance method. The filtration technique was approached through two separate mechanisms: the constant-pressure and the constant-rate method.

The foundation upon which the filtration resistance methods were based is the Kozeny-Carman equation. The equation related the rate of flow of an incompressible fluid through a fixed porous bed to the specific surface, the effective specific volume, and the compressibility characteristics of the bed. Ingmanson (6) showed that the equation could be applied in differential form to the filter mat of a compressible material such as pulp fibers, and then integrated over proper boundary conditions. By

this means, the filtration resistance could be expressed in terms of it components.

Ingmanson (6) worked closely with both constant-rate and constant-pressure methods. While both methods used the Kozeny-Carman equation, they were each based on different forms of the equation. It was concluded that the use of the varied forms of the Kozeny-Carman equation to calculate specific surface of specific volume from either constant-rate or constant-pressure filtrations showed them to be reasonably similar and in good agreement.

A more recent research in the characterization of pulp was carried on by Gertjejsen (7) of the United States Forest Product Laboratory. A simple constant-pressure filtration method was developed to obtain the average specific filtration resistances of pulps. The method utilized a constant-pressure tube, an electronic load cell, and a recorder system. Filtrate was collected in a container resting on the load cell, and a curve of filtrate versus time was obtained automatically. The validity of this method was determined by comparing specific volumes and surface areas using filtration and compressibility data from the study in the formula established by Ingmanson (6) as well as data obtained by the water permeability method and formulas developed by

Robertson and Mason (8). The comparison showed good agreement between the two methods. The consistency range covered the normal operating consistencies of the laboratory experimental paper machine. The results showed that consistencies within that range did not affect the average specific filtration resistance.

In an effort to put pulp characterization on an ideal basis, the Institure of Paper Chemistry, working with the filtration method, designed an automated, highly controlled, sensitive instrument for evaluating pulp. However, the method was rather involved and has not proved suitable for practical, routine control use. (9)

Another more recent drainage resistance analyzer called the Pulmac Permeability Tester, built by Pulmac Instruments Ltd., was designed for rapid and meaningful evaluation of pulp. The instrument measures the variables which can be calculated by means of the Kozeny-Carman equation to yield the specific surface of the pulp as well as the specific volume.

Because of the recent introduction of the instrument, little is known about its effectiveness. However, a recent trial at Kruger Paper Co. (10) indicated that the Pulmac tester was very effective for evaluating groundwood pulps. It was shown to be a simple, rapid, and easily adapted control measure. The most difficult problem involved with

the instrument was the calculation of results. A computer or sophisticated desk calculator was a necessity to obtain the specific surface promptly.

PRINCIPAL APPLICATION AND THEORY OF CENTRIFUGAL METHODS

The C.W.R. method is a measure of the water retained after a fiber mat has been subjected to a centrifugal force. Centrifugal methods date back over thirty years and, while much information has been acquired, it has been difficult to differentiate between water retained by pores due to swelling, and by pores such as the lumen and the interstices between fibers.

A too strong centrifugal force will distort a fiber and, consequently, a low C.W.R. will result. On the other hand, a force of too low a value will result in a C.W.R higher than that attributed to intrafiber retention of liquid. Thus, the centrifugal force is a prime factor in the test. Because no substantial work has been done to find the optimum force it is arbitrarily taken to be from 2,500 to 3,000 g's. (4, 11)

In an attempt to measure the water retained only within the fiber (swellability), rather than the water

both within and trapped between the fiber (swollen volume), Yiannos (5) replaced the water with a solvent. This lowered the surface tension of the water and added little or no solvating power. This technique allowed centrifuging at lower forces, thus avoiding collapsing of the fibers. The centrifugal force removed all of the solvent from the large pores of the fiber and from the interstices between the fiber, but it did not remove the solvent from the pores created by water-swelling because of their small size. This method was shown to be particularly effective in determining the degree of collapsing of pulp fibers at different moisture contents.

The C.W.R. of a pulp in all cases correlated with sheet strength. However, because of the difficulty in determining exactly where and how the fiber retained the water, a controversy remained as to whether the test was dependent on fiber length (12).

PERMEABILITY THEORY AND TECHNIQUES

All drainage rate methods for pulp characterization are based on the Kozeny-Carman equation:

(1.) $$Q = KAP/L$$

where: Q = volume rate of flow through bed, cm^3/sec

P = pressure drop across the bed, $dynes/cm^2$

A = bed cross-sectional area, cm^2

L = bed depth, cm

η = viscosity of fluid, g/cm/sec

K = Permeability coefficient of the bed, 5.55 (The Kozeny constant is usually taken to be 5.55 for pulp fibers (8). The paper by Labreque (13) contains a good discussion of the Kozeny constant for various situations.)

The resistance to viscous flow of a fluid through a uniform pad of porous material can be expressed, by means of the Kozeny-Carman equation, in terms of the void fraction and surface area of the material comprising the bed. In applying this method to a bed of pulp fibers, it is necessary to know the effective volume of the fibers. Since this is not generally known, the Kozeny-Carman equation has been modified so that the specific surface area and the effective specific volume can be determined simultaneously. The two parameters are defined in equations (2.)-(5.) below.

(2.) $$R_p = \frac{A^2 P}{Q W \eta}$$

(3.) $$C = \frac{W}{A L}$$

$$(4.) \qquad \left(\frac{C}{R_p}\right)^{1/3} = \frac{1}{K\ S_w^2}\ (1-VC)$$

R_p = Filtration resistance at constant pressure, cm/g

A = Bed or mat area, cm^2

P = Pressure drop, dynes/cm^2

Q = Flow rate, cm^3/sec

W = Mat weight, g

η = Viscosity, poises (g/cm/sec)

L = Mat thickness, cm

K = Kozeny constant, for pulp fiber, 5.55

S_w = Specific surface, cm^2/g

V = Specific volume, cm^3/g

C = Consistency, g/cm^3

Plotting from equation (4.), C/R_p versus the value of equation (3.), C, for the data collected, a straight line relationship is obtained using the least squares method. The line is extended to intercept the y and x axis (Fig. 1).

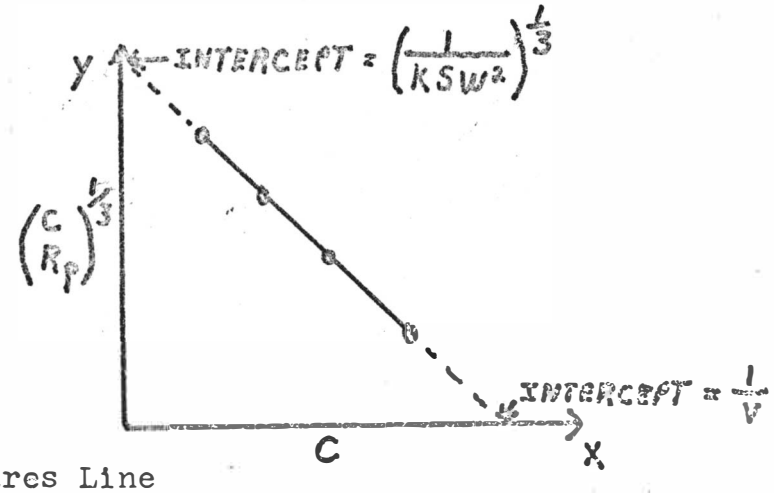

Fig. 1. Least Squares Line

S_W is solved from the y intercept yielding the specific surface area and V is solved from the x intercept giving the specific volume measurement.

In order to speed calculations, a computer program was used (Appendix B). A sample read-out sheet from the computer calculations is shown in Fig. 4, page 21.

There are two basic drainage rate techniques to which the Kozeny-Carman equation can be applied effectively. These are the constant-rate and the constant-pressure methods.

For the case of a constant-pressure filtration involving a noncompressible filter bed, the validity and unity of an average specific filtration resistance was demonstrated (6). Such a concept was also shown to be valid for a compressible filter mat (6). Although in this case both the degree of compaction and the resistance to flow offered by a unit mass of solids varied markedly through the mat from face to septum, the average specific filtration resistance through the mat was not a function of mat thickness or mass, but only of the pressure differential across it.

For the constant-rate case, the pressure differential across the bed increased with time. Since, with a compressible bed, the average specific filtration resistance was a function of the pressure differential, it

too must have decreased with time.

In spite of the difficulties in interpretation of these results, there are many experimental advantages to the constant-rate method. Each experiment is simple and less time-consuming, fewer calculations are involved, and it is possible to obtain the filtration resistance at any desired pressure differential from one experiment.

EXPERIMENTAL PROCEDURE

INTRODUCTION

The approach that was taken in comparing the centrifugal water retention test, the constant rate filtration test, and the Canadian Standard Freeness test, was to use an identical pulp with all three tests and vary a specific controllable characteristic of that pulp. The tests were then evaluated on their response to the change of that parameter.

It is a generally accepted fact that the fines content of a pulp is a definite characteristic and one which influences paper strength (14). Therefore, the variable characteristic that was used for the test evaluation was the fines content. The amount of fines in the pulp was varied from 0% to 25% at 5% intervals with several runs being made on each instrument at each interval. The base pulp used was unbeaten natural kraft softwood. British Sheet Mold handsheets were made from the pulps containing the different amounts of fines, while drainage rates in the sheet mold were also determined.

Although the tests were evaluated from only one pulp characteristic viewpoint, it was anticipated that

the results would yield an insight as to the relationship of the characterization methods.

THE MANUFACTURE OF FINES

The first problem in varying the fines content of a pulp was to define fines and, once defined, to devise a method of procurement. The definition of fines as referred to in this paper is the very fine fiber from a bleached softwood kraft pulp beaten to C.S.F. of 300 and collected from the whitewater of Western Michigan University's pilot paper machine, using a 75/60 mesh wire. A 44-lb. (25x38-500) basis weight paper formed at 90 feet/min was run while collecting the whitewater. Once collected, the whitewater was allowed to settle in a glazed tile chest. The concentrated fines from the bottom of the tank were scooped into buckets and allowed to resettle. The clear effluent was siphoned off and the fines concentrate was poured into another container to concentrate further. After the fourth concentration, approximately twenty liters of concentrate at 0.74% solids was obtained for the experimental work at hand. This volume facilitated refrigeration and reduced the possibility of microbiological degradation.

THE CENTRIFUGAL WATER RETENTION METHOD

The centrifugal water retention procedure that was used in evaluating the pulp was recommended by Czappa (11). Czappa adapted the centrifuge equipment at Western Michigan University to parallel the technique used by Thode (4) at the Institute of Paper Chemistry. The only piece of equipment that was changed was the pad-forming stand into which the centrifuge cup was placed. The cup contained a septum on which a pad was formed from a slurried sample. The original equipment used by Czappa was not in good working order and was replaced by a machined stainless steel and brass stand.

The sample size used for a C.W.R. determination was 1.5 grams in 2 liters of water. The sample was dearated under vacuum for 20 minutes under continuous stirring with a magnetic stirrer. A 200-ml portion of the sample was taken and put into the pad-forming stand (Fig. 2).

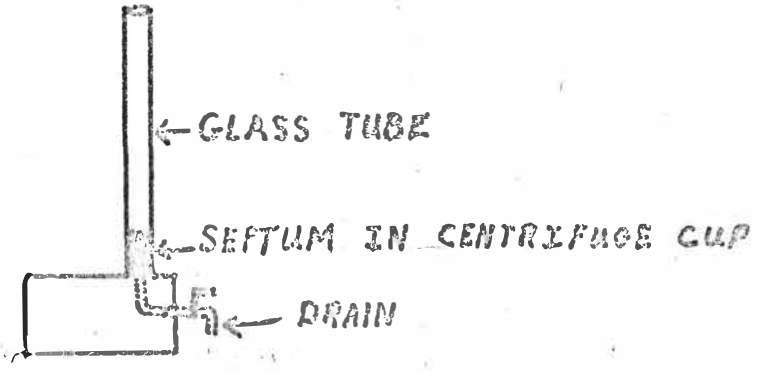

Fig. 2. Pad Forming Stand

A pad was formed on the septum in the centrifuge cup. The cup was then put in the centrifuge and subjected to a force of 2600 gravities for 15 minutes. The sample was then placed in a tared weighing bottle and weighed. It was then dried in an oven overnight at 105°C. The sample was then weighed again. The amount of water per 100 grams of fiber is the C.W.R. measurement. A detailed operating procedure may be found in Appendix A.

THE PULMAC PERMEABILITY TEST METHOD

The operation of the Pulmac Permeability tester was carried out as directed in the instrument manual (15). A summary of the procedures and calculations is given below.

To prepare the Pulmac Permeability tester for operation, it is important that deaerated, deionized water be used to purge the water lines of all air bubbles. The septum is put into place under water so as not to trap any air bubbles under its fine mesh wire.

The sample normally required for an accurate determination of filtration resistance and consistency in the Pulmac Permeability tester is between 4 and 6 grams. This sample is slurried to about 0.15% and deaerated under vacuum

for approximately twenty minutes.

The slurry is then gently poured into the plexiglass cylinder (C), Fig. 3, page 18. The septum (T) is located at the bottom of the cylinder with the drain valve outlet below the septum. The drain valve (D) is opened and a pad is formed on the septum.

The level of slurry is lowered with the drain valve to approximately level (L), the fibers are allowed to settle, and a rod and piston (P) are brought down onto the surface of the pad. The level of the water is then lowered by means of a small weir outlet (W). This assures a constant head of water above the pad. A 500-gram force is placed on the septum by fitting it on the rod at (M). The instrument is furnished with several spacers. The largest spacer (S) that will fit is placed between the 500-gram mass and the frame.

The apparatus is so adjusted that the distance between the mass and the frame is equal to that between the piston and the septum, which is the pad thickness. A 3-kilogram mass is added to the 500-gram mass, thus compressing the pad to a thickness equal to the spacer length.

Through the means of an overhead water supply tank and a rotameter, a laminar flow rate of water is set up through the pad from septum to piston, such that the pressure differential across the pad, as measured by a manometer, is

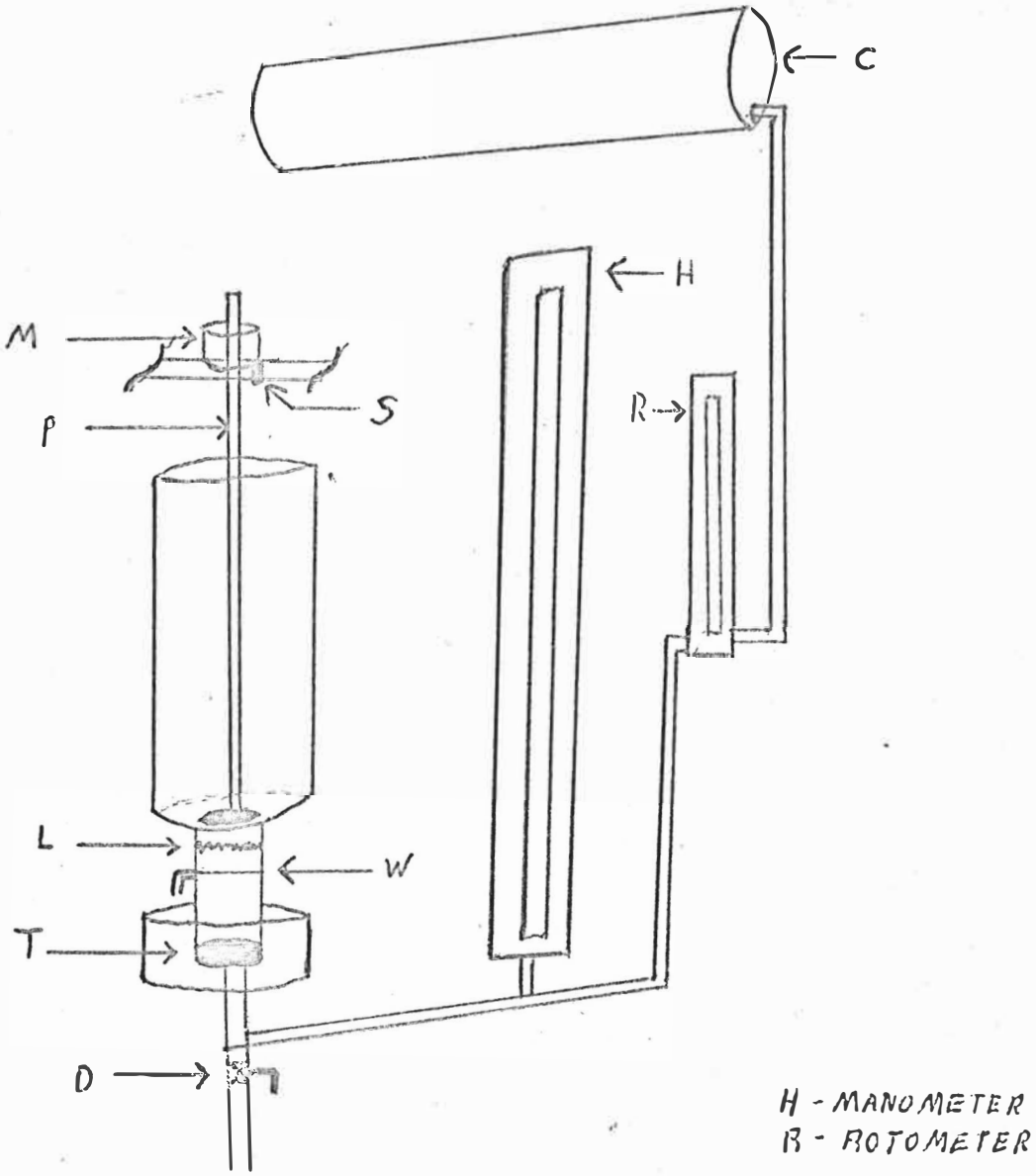

Fig. 3. Pulmac Permeability Tester

about 1.5 cm.

As the water flows upward through the pad, the constant head above is maintained by the weir. The flow rate, pressure differential, and pad depth are recorded. The next largest spacer that will fit between the mass and frame is put in place and the supporting one removed. This allows the 3.5 kilogram mass to compress the pad to a new thickness equal to the new spacer length. When the readings are stabilized, the new flow rate, pressure differential and spacer length are recorded. This procedure is repeated until six to eight sets of data are obtained.

When data collection is complete, the pad is removed, dried to oven dryness at 105°C, and weighed. Three Pulmac Permeability determinations were run on each of the 0%, 5%, 10%, 15%, 20%, and 25% fines contents. Using a modified version of the Kozeny-Carman equation, the specific surface area of the fibers and their specific volume was calculated.

The specific filtration resistance (cm/g) is defined by equation (2.). Equation (4.) relates the pad consistency to weight of the pad, the cross-sectional area of the sample cell, and pad thickness. All of these quantities are measured with the permeability tester. The specific filtration resistance can be related to

specific surface area and the specific volume (8,15) of pulp by means of equation (4.) given on page 10.

HAND SHEET FORMATION AND TESTING

Six British Sheet Mold handsheets were made with the base pulp and from each of the percent levels of fines addition. The procedure for forming the handsheets was taken from T.A.P.P.I. T 205 m-58. Physical tests were run according to the following standards: tear, T 414 ts-65; mullen, T 403 ts-63; basis weight, T 410 os-65; fold, T 423 m-50. Tensile was run on the Instron Tensile tester using a strip 10 cm in length by 1.5 cm in width and a constant head drive of 0.5 cm/minute. Tensile and percent elongation were determined from the recording chart while the integrator reading obtained from the machine was used to compute Tensile Absorption Energy according to T.A.P.P.I. T 494 su-64.

Tear, tensile, and mullen were corrected for basis weight by dividing the observed readings by the observed basis weight and multiplying by 60 g/m^2 $\frac{\text{reading}}{\text{B.W.}} \times 60 g/m^2$.

RUN 1 25. PERCENT,
BASED ON WATER VISCOSITY OF .00981000 AND PAD WEIGHT OF 5.7406

LENGTH	PRESSURE	FLOW RATE
2.80	1.31	.75
2.60	1.48	.73
2.40	1.78	.72
2.30	2.00	.71
2.20	2.20	.68
2.10	2.38	.68
2.00	2.64	.66
1.90	2.98	.66
1.80	3.30	.63
1.70	3.81	.62

SPECIFIC VOLUME = 4.01699

SPECIFIC SURFACE = 46115.32200

Y INTERCEPT = .00043920

SLOPE = -.00176430

THE MAXIMUM DEVIATION ALONG Y-AXIS IS .00000296

FILTRATION RESISTANCE CM/GM	CONSISTENCY GM/CC	CR**1/3
1838521000.	.06459402	.00032752
2134014800.	.06956279	.00031944
2602232300.	.07535969	.00030708
2965037800.	.07863620	.0002 821
3405433200.	.08221057	.00028901
3684059500.	.08612536	.00028593
4210353700.	.09043163	.00027797
4752596300.	.09519119	.00027157
5513558600.	.10047959	.00026315
6468325900.	.10639015	.00025431

Fig. 4. Sample Computor Readout Sheet

PRESENTATION AND DISCUSSION OF DATA

Figure 6 shows that with an increase of the percent of fines in a pulp, the centrifugal water retention increased linearly with the fines content. This linear relationship was also true with the specific surface area value as calculated from the information obtained on the Pulmac Permeability tester. Freeness decreased linearly with fines addition. The second value calculated from the Pulmac test, the specific volume, reacted in a non-linear manner and actually went through a maximum at 20% fines addition. (Data used in Fig. 6 may be found in Appendix C.) British sheet mold drainage times (B.S.M.) were taken during handsheet formation. The rate was affected only slightly by the addition of fines. From the graph it can be seen that the sensitivity of the tests to the fines addition can be ranked with specific surface being the most sensitive followed by freeness, C.W.R., and B.S.M. drainage respectively.

The unique behavior of the specific volume parameter is again shown when the various pulp characterization measurements are plotted against Instron tensile (Fig. 7). It can be noted that the tensile strength is at its maximum at 15% fines. Elongation tests followed a nearly identical pattern with tensile.

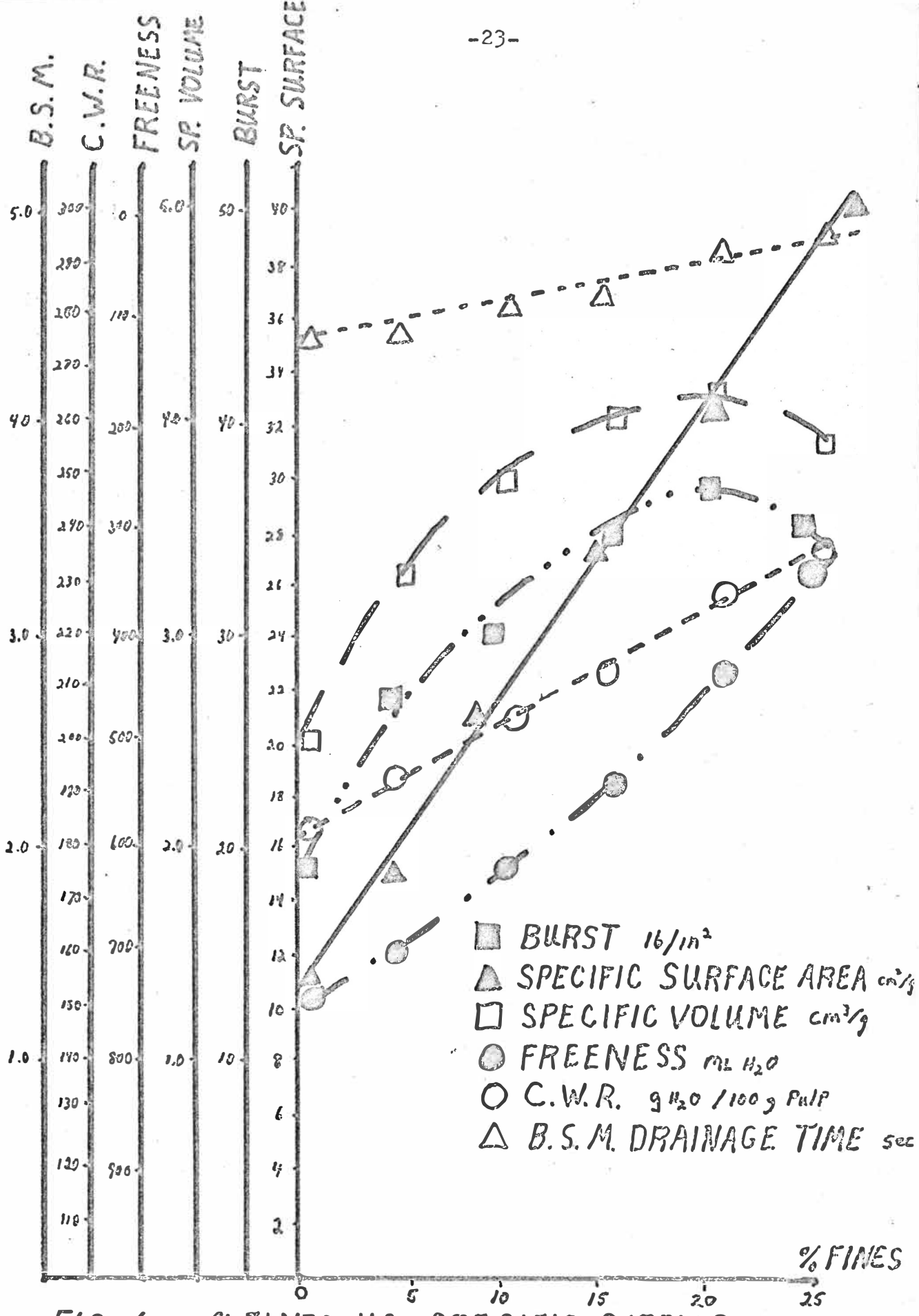

FIG. 6 % FINES V.S. SPECIFIC SURFACE, SPECIFIC VOLUME, FREENESS, C.W.R., B.S.M DRAINAGE TIME AND BURST

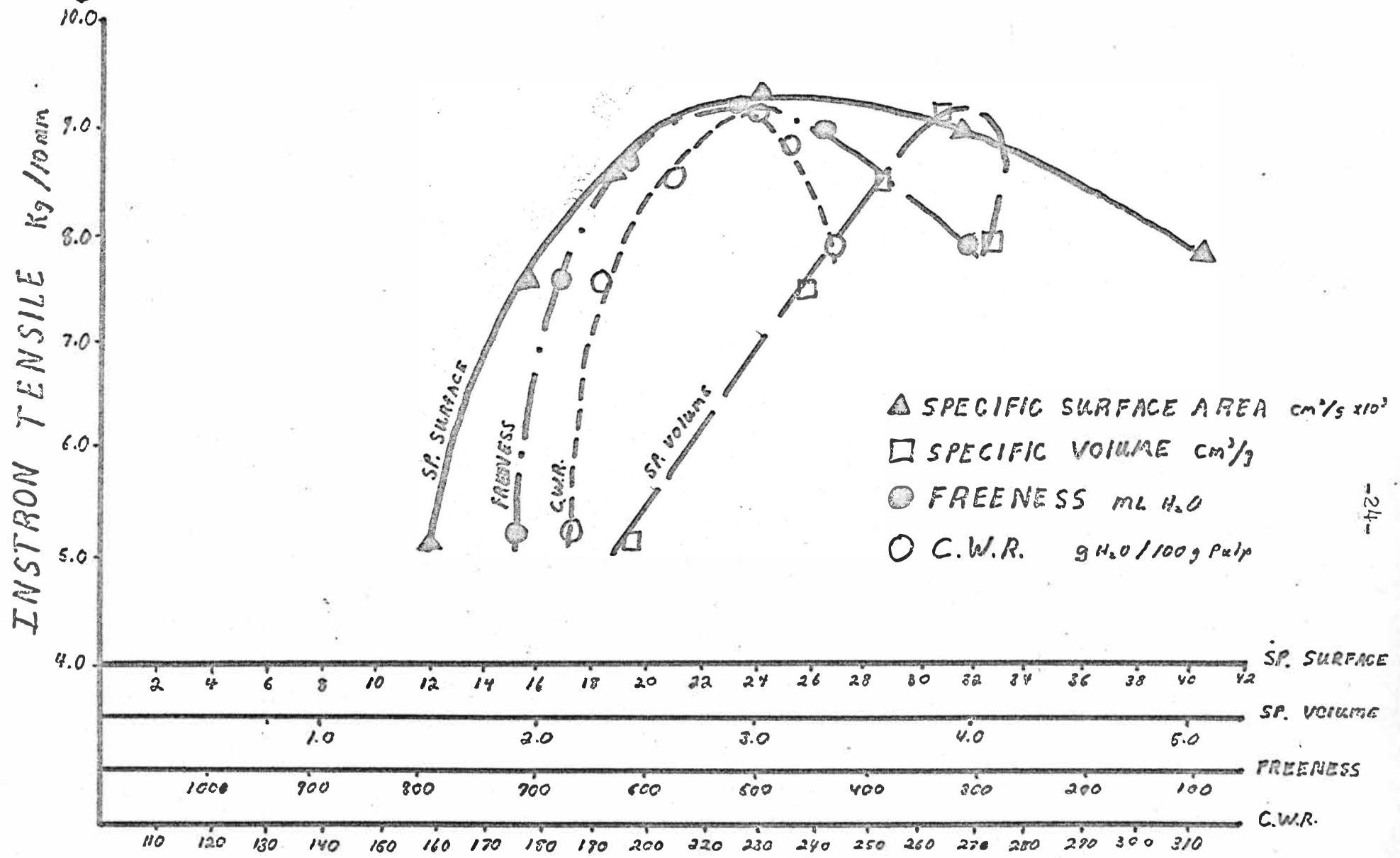

FIG. 7 TENSILE V.S. SPECIFIC SURFACE, SPECIFIC VOLUME, % FINES, FREENESS AND C.W.R.

It is also important to note that the specific surface values tend to follow the percent fines in the sheet, as is the general trend of freeness and C.W.R. (Fig. 6). The value for specific volume has a near-linear relationship with tensile strength and actually increases and decreases with the tensile.

This relationship is even more distinguishable when the same pulp characterization measurements are plotted versus the burst test (Fig. 8). The burst test reached a maximum at 20% fines content and again the specific surface, freenes and C.W.R. followed the same general trend. The specific volume value increased and decreased linearly with the burst test. Fig. 6 also very plainly shows the direct relationship between the specific volume and burst. These results concur with the work done by Robertson and Mason (8). In their investigation, they found no substantial correlation between specific area and strength tests. However, they did find a direct relationship between mullen and specific volume.

Figures 9 and 10 show the various characterization methods plotted versus tear strength and tensile energy absorption respectively. The same pattern is developed as in Figures 7 and 8. Again, specific volume follows the strength tests to a greater degree than the other pulp characterization methods.

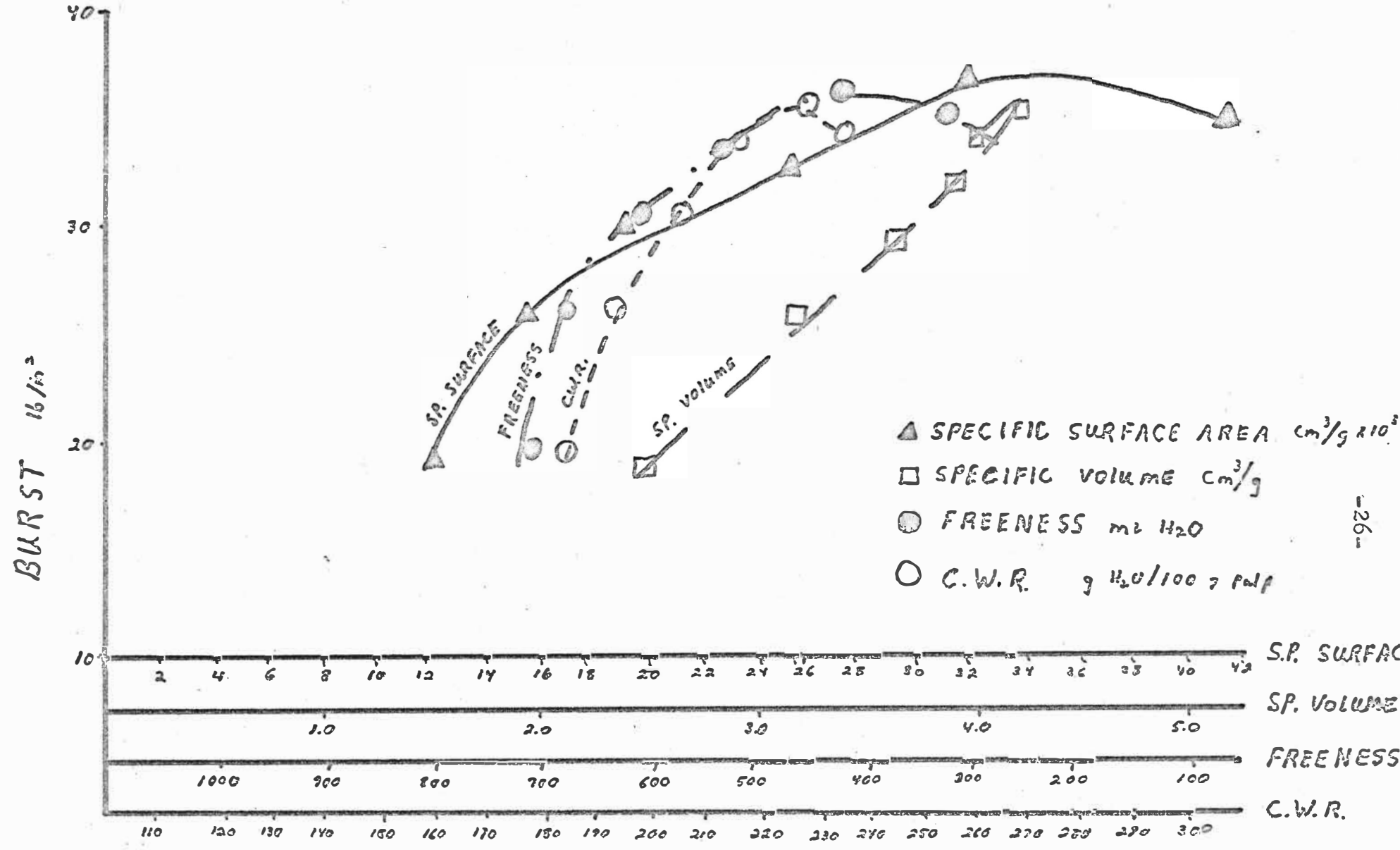

FIG. 8 BURST VS SPECIFIC SURFACE, SPECIFIC VOLUME, % FINES, FREENESS AND C.W.R.

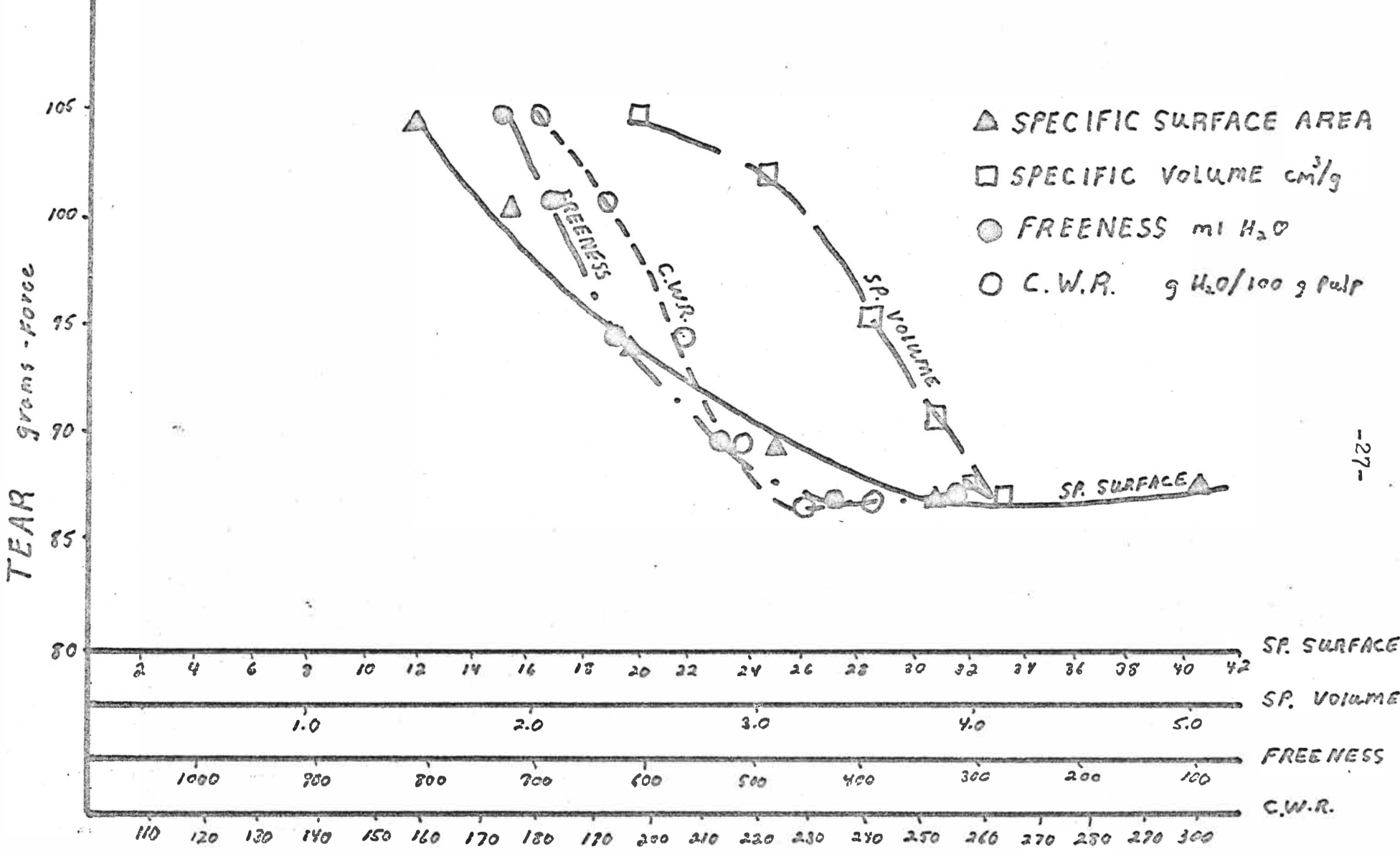

FIG. 9 TEAR V.S. SPECIFIC SURFACE, SPECIFIC VOLUME, % FINES, FREENESS AND C.W.R.

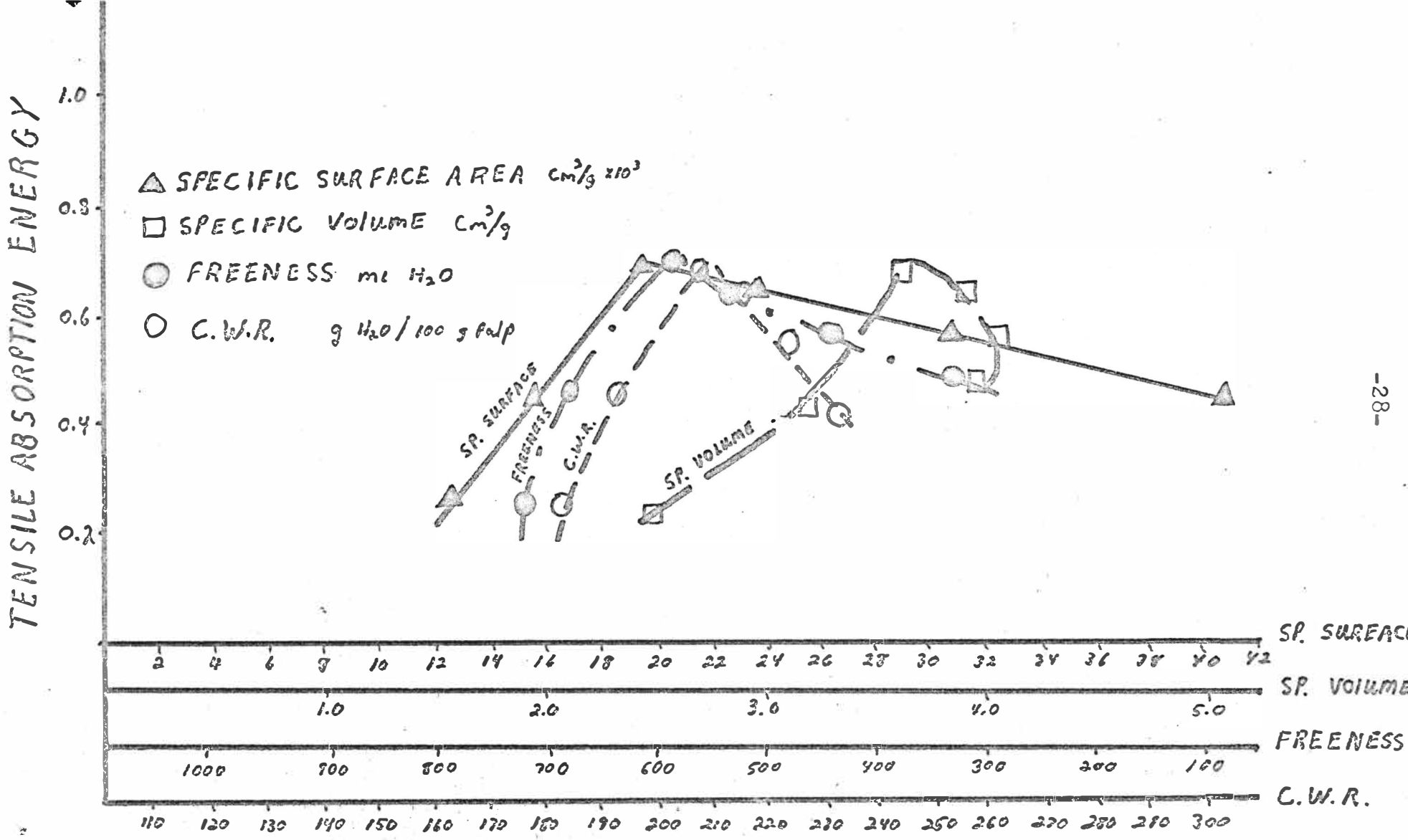

FIG. 10 TENSILE ENERGY ABSORPTION V.S. SPECIFIC SURFACE, SPECIFIC VOLUME, % FINES AND C.W.R.

The results presented indicate that the specific surface area, freeness, and C.W.R. all measure the same pulp characteristic, which is basically its water-holding ability.

The specific volume, however, measures a different pulp parameter. Its behavior may be explained by considering its relationship to the fines content. Initially, as fines are added, the volume increases. After a point, the volume will begin to decrease because the fines no longer create more bulk, but rather begin to pack into a smaller volume. In Figure 9, the tensile energy absorption reached a maximum value at about 10% fines content. It is felt that above this percentage of fines, the bonding increase credited to the fines is less influential, resulting in a decrease of absorption energy. This same reasoning is applied in explaining the cause for the mullen reaching its maximum value at a higher percentage of fines.

FUTURE WORK

Because the experimentation carried out for this paper was done varying only the fines content of a pulp, the results obtained cannot be applied to the variance of other pulp characteristics. It would be interesting to investigate the possibility of the specific surface values following the fines content of a pulp with other varying parameters. It would also be interesting to discover if the specific volume value would remain in a linear relationship with strength tests, particularly mullen.

It is the author's opinion that the specific surface area and C.W.R. measurements will generally follow the freeness results. The specific volume value, while hard to predict from work done thus far, could turn out to be a beneficial measurement of pulp character.

The evaluation of the measurements available from the Pulmac Permeability tester will become more meaningful when more work is done with the instrument. It is relatively new in the industry and yet to be proven.

While working with the instrument, the author became very familiar with all aspects of operation and calculation. While the device is a pleasure to use

because of the precision of the results obtained, these results cannot be rapidly calculated without the use of a computor or sophisticated desk calculator.

The mechanical operation could be greatly improved with the use of higher quality construction materials and a few added provisions. This refers specifically to the connectors used on the plastic piping which invariably leaked. Some difficulty was also encountered in purging the air from the lines when initially setting up the machine for operation. The water supply tank had no provision for drainage except through the rotameter. This created a problem when trying to clean or prepare the machine for inoperative periods.

Some provision could also be made for connecting the waste lines directly to a sewer drain, for when running several consecutive determinations, it became tedious to empty the waste tank after each run.

With a well-designed unit, it is possible that the constant rate filtration resistance method could be accepted for routine control use. However, presently it is not generally feasible to use the Pulmac Permeability tester for any other use than research.

CONCLUSIONS

The conclusions reached from the data obtained are drawn specifically from the reactions of the Canadian Standard Freeness, Centrifugal Water Retention, specific surface, and specific volume measurements to the previously specified pulp.

(1) While the C.W.R., C.S.F., and specific surface area values tended to follow the same general trends in characterizing a pulp, the specific surface area measurements followed the fines content to a closer degree than the other values.

(2) The fines content of a pulp had an important effect on the strength properties of the paper it made.

(3) The specific volume measurement had the unique property of being directly proportional to the strength properties in handsheets made from the pulp tested.

(4) The Pulmac Permeability tester yielded precision results which are useful in pulp characterization.

(5) While useful as a research instrument, it is doubtful that the Pulmac Permeability tester could be readily accepted by industry as a routine control instrument.

(6) More work must be done evaluating various pulp characteristics with the Pulmac Permeability tester

before any definite conclusions can be drawn as to its usefulness in characterizing a variety of pulp parameters.

APPENDIX A (11)

CENTRIFUGAL WATER RETENTION TEST

Pad Formation

1. The equivalent of 1.5 grams of oven dry fiber in the form of slush pulp is weighed out and dispersed in 1000 ml of distilled water.
2. The pulp is dispersed in a British Disintegrator for 300 counts. Refined samples in slush form do not require this step.
3. Sufficient distilled water is added to bring the volume of the pulp to 2 liters in a 2-liter suction flask.
4. Deaerate under vacuum with continuous stirring for twenty minutes.
5. Place the metal cup in the pad-forming stand and connect the glass tube. (Fig. 2, page 15.)
6. Fill the sample cup with distilled water to a level that just appears visible in the glass tube.
7. Gently mix deaerated sample.
8. Pour 200 ml of deaerated sample in a 250 ml graduated cylinder.
9. If gravity drainage or filtration does not proceed at the desired rate, a vacuum may be applied.

10. The vacuum should be broken before air penetrates the pad.
11. Place the metal sample tube and pad in a centrifuge shield.

Centrifuging

1. Brass spacer rings are used in the bottom of the metal shield tubes, instead of the conventional rubber pads. This allows a small sponge to be placed inside the rings to absorb the water thrown out by the centrifugal force.
2. The sample tube containing the moist pads is placed in the shield. The insertion of the sample tube must be done slowly, tilting the shield in order to allow the displaced air to escape. If the sample tube is wet and is allowed to fall into the shield, the displaced air will "blow" the pad toward the top of the sample tube and thus destroy the pad.
3. The shield-tube assemblies should be placed in directly opposite positions in the centrifuge head.
4. Close and lock the centrifuge cover.
5. To start centrifuge, set the timer indicator, turn rheostat knob back to zero until a click is heard, and then depress the switch with the small white dot down.

6. Lower the tachometer and adjust the speed corresponding to the desired centrifugal force.
7. The timer will cut the power at the required time, and the centrifuge will coast to a stop without need of the brake.
8. Remove the sample tube and drive off the drilled plate using the adapter ring.
9. Remove the compacted fiber pad with tweezers and placed in a tared weighing bottle. Examine the bottom of the metal sample tube; if fibers remain, wipe them off using the pad and tweezers.
10. Weigh bottle and moist pad.
11. Dry in oven at 105°C overnight.
12. Weigh bottle and dry pad.
13. $C.W.R. = \frac{\text{Wet weight - dry weight}}{\text{Dry weight}} \times 100$

C.W.R. = grams water / 100 grams oven dry pulp

Note: The centrifugal force is calculated as follows:

$F = 4\pi^2 N M R$

N = Revolutions per second

R = Rotameter radius

M = Mass of body

Equipment

1. An International Centrifuge Model V, size 2 was used in the laboratory work.
2. The special centrifuge head obtained for this laboratory work was a solid disk of magnesium alloy, 13 inches in diameter and 1¼ inches thick with a collar for attachment to the drive shaft and machine slots to accomodate four shields suspended from trunnion rings.
3. For a complete equipment description, see Thode (4).

APPENDIX B (15)

COMPUTOR PROGRAM FOR PULMAC PERMEABILITY TESTER

```
ZZFORX              ED GRYS     DR R. JAMES      SENIOR THESIS
      DIMENSION DELTAL(20),DELTAP(20),Q(20),RP(20),C(2,20)
      A=31.74
      TOL=.000007
3     READ 1,N,TEMP,W,NRUN,PERCNT
1     FORMAT (I2,F10.10,F6.4,I1,F2.0)
      STOPRN=0
      PUNCH 4,NRUN,PERCNT,TEMP,W
4     FORMAT (3HRUN,I2,2X,F4.0,2X,7HPERCENT,2H, /
     127HBASED ON WATER VISCOSITY OF,F12.10,3X,17HAND PAD WEIGHT OF,
     2F8.4)
      DO 5 I=1,3
5     PUNCH 99
99    FORMAT (1H )
      PUNCH 6
6     FORMAT (6HLENGTH,6X,8HPRESSURE,6X,9HFLOW RATE)
      DO 7 I=1,N
7     READ 2,DELTAL(I),DELTAP(I),Q(I)
2     FORMAT (F4.2,F5.2,F5.2)
      PUNCH 99
      DO 16 I=1,N
16    PUNCH 17,DELTAL(I),DELTAP(I),Q(I)
17    FORMAT(F6.2,6X,F7.2,7X,F7.2)
      DO 18 I=1,3
18    PUNCH 99
35    DO 8 I=1,N
      DELTAP(I)=DELTAP(I) *.3937*25.40*98.0665
      Q(I)=Q(I)/60.
8     RP(I)=(A**2*DELTAP(I))/(Q(I)*W*TEMP)
34    DO 9 I=1,N
9     C(1,I)=W/(A*DELTAL(I))
      DO 10 I=1,N
10    C(2,I)=(C(1,I)/RP(I))**.33333333
      SUMXY=0
      SUMX=0
      SUMY=0
      SUMXSQ=0
      DO 11 I=1,N
      SUMXY=SUMXY+C(1,I)*C(2,I)
      SUMX=SUMX+C(1,I)
      SUMY=SUMY+C(2,I)
11    SUMXSQ=SUMXSQ+C(1,I)**2
      Z=N
      D=(Z*SUMXY-SUMX*SUMY)/(Z*SUMXSQ-SUMX**2)
      E=(SUMY-D*SUMX)/Z
      IF (SENSE SWITCH 1)28,50
28    DO 26 I=1,N
      IF (C(2,I)-D*C(1,I)-E-TOL)27,31,31
27    IF (C(2,I)-D*C(1,I)-E+TOL)31,31,26
26    CONTINUE
      GO TO 50
31    PUNCH 32,DELTAL(I)
32    FORMAT(37HTHE SET OF DATA WITH LENGTH EQUAL TO ,F6.2/
```

APPENDIX B (Cont.)

```
     162HWAS DELEATED BECAUSE IT FAILED TO MEET TOLERANCE REQUIREMENTS.)
      PUNCH 99
      N=N-1
      DO 33 J=I,N
      L=J+1
      DELTAL(J)=DELTAL(L)
      DELTAP(J)=DELTAP(L)
33    Q(J)=Q(L)
      STOPRN=STOPRN+1.
      IF (STOPRN-3.)34,37,37
37    PUNCH 99
      PUNCH 38
38    FORMAT (44HTHIS SET DISCONTINUED MORE THAN 3 DELEATIONS)
      GO TO 3
50       Z=N
      BIGDEV=0
      DO 41 I=1,N
      DEV=C(2,I)-D*C(1,I)-E
      IF (DEV-BIGDEV)41,41,42
42    BIGDEV=DEV
41    CONTINUE
      PUNCH 43,BIGDEV
43    FORMAT (37HTHE MAXIMUM DEVIATION ALONG Y-AXIS IS,F10.8)
      PUNCH99
      YINTER=E
      XINTER=-E/D
      V=1./XINTER
      SW=SQRT(1./(5.55*YINTER**3))
      PUNCH 19
19    FORMAT (27HFILTRATION RESISTANCE CM/GM,7X,17HCONSISTENCY GM/CC,
     117X,7HCR**1/3)
      DO 14 I=1,N
14    PUNCH 13,RP(I),(C(J,I),J=1,2)
13    FORMAT(F14.0,20X,F10.8,24X,F11.9)
      DO 20 I=1,3
20    PUNCH 99
      PUNCH 21,E
21    FORMAT (10X,13HY INTERCEPT =,F12.10)
      PUNCH 99
      PUNCH 22,D
22    FORMAT (10X,7HSLOPE =,F12.10)
      DO 23 I=1,3
23    PUNCH 99
      PUNCH 24,V
24    FORMAT (40X,17HSPECIFIC VOLUME =,F10.5)
      PUNCH 99
      PUNCH 25,SW
25    FORMAT (40X,18HSPECIFIC SURFACE =,F14.5)
      GO TO 3
      END
```

APPENDIX C

CENTRIFUGAL WATER RETENTION

0% Fines

Run	C.W.R.
1	184.24
2	180.66
3	180.37
4	190.17
Average:	183.86

5% Fines

Run	C.W.R.
1	193.67
2	173.73
3	204.07
4	200.57
Average:	193.01

10% Fines

Run	C.W.R.
1	212.62
2	212.38
3	203.31
4	203.31
Average:	208.32

15% Fines

Run	C.W.R.
1	214.04
2	219.04
3	212.64
4	214.09
Average:	216.20

20% Fines

Run	C.W.R.
1	229.03
2	224.01
3	218.79
4	223.37
Average:	223.80

25% Fines

Run	C.W.R.
1	236.37
2	233.08
3	231.80
4	233.15
Average:	233.60

APPENDIX C (Continued)

CANADIAN STANDARD FREENESS
(Average of three determinations)

0%	720
5%	685
10%	614
15%	532
20%	430
25%	305

BRITISH SHEET MOLD DRAINAGE TIME
(Average of three determinations)

0%	4.4 sec.
5%	4.4 sec.
10%	4.5 sec.
15%	4.5 sec.
20%	4.8 sec.
25%	5.0 sec.

APPENDIX C (Cont.)

TEAR (Corrected)
(Average of five determinations based on 16 sheets)

0%	104.6 grams force
5%	101
10%	97.2
15%	95.9
20%	87.3
25%	87.6

TENSILE ENERGY ABSORPTION AND STRETCH
(Avg. of 5 determinations)

	Absorption Energy	Stretch
0%	0.236 kg-cm	2.30%
5%	0.442	2.40
10%	0.697	2.60
15%	0.618	2.55
20%	0.513	2.40
25%	0.443	2.37

TENSILE (Corrected)
(Avg. of 5 determinations)

0%	505
5%	777
10%	882
15%	916
20%	893
25%	789

MULLEN (Corrected)
(Avg. of 3 determinations)

0%	18.7
5%	26.6
10%	29.1
15%	30.5
20%	36.9
25%	34.5

APPENDIX C (Cont.)

Pulmac Test Results

0%

Run	Pad Weight	Specific Surface	Specific Volume
1	5.9385	12,650	2.14
2	5.8353	11,840	2.47
3	5.9074	12,680	2.31
4	5.7975	10,000	2.74
Average:		12,044	2.42

5%

Run	Pad Weight	Specific Surface	Specific Volume
1	6.2298	15,400	3.31
2	5.9256	15,860	3.55
3	5.9850	16,950	3.03
Average:		16,070	3.30

10%

Run	Pad Weight	Specific Surface	Specific Volume
1	5.8074	20,490	3.55
2	5.7691	20,650	3.55
3	5.7697	18,280	4.22
Average:		19,800	3.77

APPENDIX C (Cont.)

15%

Run	Pad Weight	Specific Surface	Specific Volume
1	5.8200	26,680	4.23
2	5.8881	26,760	3.82
3	5.8064	20,320	3.78
Average:		24,590	3.94

20%

Run	Pad Weight	Specific Surface	Specific Volume
1	5.8922	31,760	4.56
2	5.9044	31,160	4.02
3	5.9461	32,860	3.89
Average:		31,930	3.94

25%

Run	Pad Weight	Specific Surface	Specific Volume
1	5.7406	46,120	4.02
2	5	37,750	4.24
3	5.7533	40,640	3.95
Average:		41,500	4.07

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