Pulping Qualities of Refractory vs. Permeable Douglas-fir Heartwood

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Heartwood samples of Pacific Coastal Douglas-fir (Pseudotsuga menziesii var. menziesii) and Rocky Mountain Douglas-fir (Pseudotsuga menziesii var. glauca) were rated for relative liquid permeability, and evaluated for kraft pulping characteristics. Highly impermeable wood of the Rocky Mountain variety was termed "refractory," while the pervious wood of the Pacific Coastal variety was termed "permeable."

Under constant pulping conditions, refractory Douglas-fir underwent less delignification than permeable Douglas-fir, giving higher screened rejects and lower screened yields. Pulp made from refractory Douglas-fir required less refining to achieve a given level of Canadian Standard Freeness.

Handsheet properties were direct functions of fiber characteristic variations. Paper made from refractory Douglas-fir heartwood had greater sheet density, tensile strength, zero-span tensile

strength, stretch, and fold endurance. Paper made from permeable Douglas-fir had greater internal tear resistance and a higher sheet brightness. No significant difference existed between the Mullen strengths of paper made from the two varieties.

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PULPING QUALITIES OF REFRACTORY VS. PERMEABLE DOUGLAS-FIR HEARTWOOD

INTRODUCTION

Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco) has been recognized as existing naturally on the west coast in two varieites (Fowells, 1965). The Rocky Mountain form (Pseudotsuga menziesii, var. glauca) and the Pacific Coast form (Pseudotsuga menziesii var. menziesii) are classified according to morphological and environmental differences. Anatomical differences between the two forms cannot normally be distinguished. The two forms differ somewhat in the size and color of their foliage, but the principal botanical difference is in the structure of their cones.

The summit of the Cascade Mountains is considered the boundary between the two forms. Thus, Douglas-fir found on the east side of the Cascades is considered to be of the Rocky Mountain form, while that on the west side is of the Pacific Coast form.

Researchers have noted a significant difference in the heartwood permeability of the two varieties. The heartwood of the coastal variety can be easily penetrated by liquids and gases, and adequate retention of preservative can be obtained. Heartwood of the mountain form, however, is virtually impermeable to the passage of either liquids or gases. Because of this known difference in permeability, the term "refractory" has been coined to describe the impermeable heartwood of the Rocky Mountain form, while the term "permeable" describes the pervious heartwood of the Pacific Coast form.

Although wood permeability has been long recognized as a significant variable in the pulping process, no detailed research has been performed to compare directly the pulping properties of the two varieties of Douglas-fir. Research has been performed on the two varieties separately, but the results were not comparable due to different experimental procedures.

The objective of this study was to compare the kraft pulping properties of refractory and permeable Douglas-fir heartwood under comparable conditions. Two basic areas of interest were evaluated: (1) the effect of wood variety upon the pulping operation, and (2) the relative properties of paper made from the two varieties, especially as they relate to wood and fiber characteristics.

LITERATURE REVIEW

Kraft Pulping

Since its discovery in 1884, the kraft or sulfate pulping process has been evaluated for many wood species. The process is widely used to pulp Douglas-fir since the presence of the extractive taxifolin limits the use of the calcium based sulfite pulping method. Although much research has been performed to evaluate effects of liquor concentrations, cooking cycles, and various wood growth parameters, no direct comparison has been made to evaluate fully the pulping responses of permeable and refractory Douglas-fir.

Pulping experiments have been performed on Rocky Mountain Douglas-fir (Wilson, Worster and O'Meara, 1960; Worster and Sugiyama, 1963) and Pacific Coastal Douglas-fir (Hammond and Billington, 1949; Holzer and Booth, 1950; Legg and Hart, 1960). But the results do not lend themselves to comparison because different liquor concentrations, cooking cycles, and freeness bases for hand-sheet values were used.

Permeability

The importance of proper chip impregnation in pulping has been accepted for many years. Numerous investigations have been conducted on the nature of penetration and diffusion of liquids and gases into wood, as well as upon the relative impregnability of various wood species. Air or liquid permeability may not be directly related to the process of penetrating pulpwood with cooking liquor (Noe, 1960), but it may be indicative of the relative success of the pulping process.

Although many investigators have found large differences in permeability between the Rocky Mountain and Pacific Coastal forms of Douglas-fir, two problems exist in correlating their results to the relative permeability of pulping liquor: (1) The rate of liquid flow is dependent not only upon wood permeability, but on pressure, temperature, viscosity, and the chemical reactivity of the fluid (Stone, 1956); (2) Steady state, liquid flow values are substantially lower than initial permeability, due to self-contamination from debris plugging (Kreir, 1951; Crawford, 1958; Estep, 1961). The results of previous studies, therefore, must be considered only in terms of the relative rankings cf wood permeability.

Craig (1963), and Miller (1961) reported significantly greater air permeability in coastal Douglas-fir than in the mountain form. Miller also noted that air permeability was not a reliable index of creosote treatability. Bramhall (1967) found air permeability to be greater in the latewood portion of Douglas-fir heartwood.

Liquid permeability was also shown to be substantially greater in coastal Douglas-fir by the studies of Estep (1961) and Erickson and Estep (1962). Liquid permeability studies by Balatinecz (1963) confirmed earlier evidence that heartwood of Douglas-fir was more impermeable than sapwood.

Studies of treatability also indicated a wide difference in permeability between the two varieties. Miller (1961) reported permeable Douglas-fir retentions to average several times those of refractory material. Work by Bramhall (1967) gave similar results. Fleischer's (1950) study of Wolman salt impregnation indicated that permeable Douglas-fir developed three times the retention and penetration of refractory samples. Miller and Graham (1963) mapped the relative permeabilities of Douglas-fir for five western states according to a visual estimation of penetration developed by Miller in 1961.

Graham (1964) developed a sink-float test for permeability in Douglas-fir. Plugs 1/2" in diameter and 1/2" in length were cut from sample material and oven dried. The plugs were submerged in water and subjected to a vacuum of 500 mm mercury for six minutes, the vacuum was released, and samples which sank within one minute were termed permeable. The vacuum was applied a second time for a period of 20 minutes. Those samples still floating after the vacuum was broken were termed refractory, and those which sank termed intermediate in permeability.

Structural Factors in Permeability Differences

Several structural characteristics of Douglas-fir have been correlated with the known difference in permeability. The factors considered include the bordered pit structure, resin canals, specific gravity, and the relative permeability of earlywood and latewood contents.

Bordered Pit Structure

The bordered pit structures found in Douglas-fir have been widely investigated in studies concerning the pathways of air and liquid translocation. The physical nature and relative abundance of these structures were considered to be major determinants of relative wood permeability.

Early investigations (Tiemann, 1909; Weiss, 1912) attributed the permeability of wood to slits or checks which developed on cell walls during seasoning. However, Gerry (1913) studied preservative flow in Larch, and could find no evidence of extensive flow through these structures.

Bailey (1913) studied the flow of carbon particles through wood. His results were the first to show conclusively the translocation of liquids through the bordered pit structure.

The position of the tori has been studied by many investigators. Gerry (1916) reported that the majority of the tori in refractory samples of Douglas-fir were aspirated, while tori in permeable samples were normally found in the central or un-aspirated position. Stamm (1929) reported that 14.6% of the permeable Douglas-fir bordered pits were aspirated, while 40% were aspirated in the refractory material studied. Stone (1939) confirmed earlier work by reporting that the frequency of aspirated pits was greater in refractory Douglas-fir than in permeable samples.

However, Stone (1939) also concluded that the interface of the torus and pit border in aspirated pits was too rough to seal completely the structure to liquid flow. Later photomicrographs by West (1941) showed an irregular tori surface, suggesting that a complete seal by aspiration would be improbable. Krahmer (1962) reported that, on the basis of electromicrographs, minute capillaries could readily exist if the torus was rough, or if the adjacent pit wall possessed a predominant warty layer, as in hemlock.

Many investigators (Bailey, 1913; Gerry, 1916; Stamm, 1929; Crawford, 1958; Koran, 1964; Bramhall, 1967) have concluded that pit aspiration alone is the predominant causative factor in the reduction of wood permeability. Other authors, however, have felt that pit aspiration is of prime importance only when accompanied by a substantial degree of encrustation.

Early work by Sutherland (1932) indicated that in some cases, the tori had been sealed in the aspirated position by resin. Stone (1939) concluded that differences in permeability may be due to the deposition of extractives in such a manner so as to close off or seal the aspirated pits. In later work, Stamm (1953) confirmed Sutherland's conclusions with examples of tori sealed in an aspirated position by resin deposits. More recent work by Krahmer (1962) found both the pit membrane and pit border to be occluded by heartwood extractives in some aspirated pits. Krahmer (1962) also found an insoluble "ligno-complex" encrustation on aspirated pits.

A study by Krahmer (1961) indicated that the size of the bordered pits in permeable and refractory Douglas-fir were nearly identical, but that the permeable samples had a higher frequency of fibers containing multiple rows of pits on the radial face than did the refractory samples.

Resin Canals

In Douglas-fir, air and water flow in the longitudinal direction may be enhanced through resin duct passageways. Erickson, Schmitz and Gortner (1938) reported that longitudinal flow in Douglas-fir heartwood was not affected by the presence of resin canals. Crawford (1958) and Erickson and Crawford (1959) concluded that resin canals have a negligible effect on longitudinal water flow through the sapwood of Douglas-fir. Koran (1964) studied permeability within refractory Douglas-fir, and determined that neither creosote retention nor air permeability were affected by the presence of longitudinal resin canals. Stamm (1946) stated that occlusion of resin ducts causes limited penetration, and thereby eliminates them as an effective pathway for air and liquid movement.

However, conflicting evidence is available. Procter and Wagg (1947) concluded that permeability was related to the number and size of longitudinal resin ducts. They found that, for both number per unit area and number per growth ring, permeable Douglas-fir contained seven times as many resin ducts as equivalent refractory samples. Permeable samples had longitudinal resin canals roughly twice as large as those found in refractory material. In addition, tylosoids were abundant in the ducts of refractory samples, whereas they were seldom found in permeable Douglas-fir.

Resin duct assistance has been credited for Douglas-fir's greater radial permeability in comparison to the tangential direction (Erickson et al., 1938; Sargent, 1959). Erickson (1938) showed that, under pressure, fine streams of water were shot through radial resin canals of refractory Douglas-fir, while only limited movement was noted through the tracheids.

More recent work indicated that resin canals were important as channels of translocation only in refractory material. Balatinecz (1963) studied liquid flow through permeable Douglas-fir, and concluded that the bulk of the longitudinal flow was through the tracheids, with only minor contributions from the resin canals. Craig (1963) confirmed Balatinecz's work on permeable samples, and concluded that due to pit aspiration in refractory Douglas-fir, the flow through the tracheids was insignificant, and that the majority of the limited flow was through the longitudinal resin ducts.

Permeability of Earlywood and Latewood

In many instances, latewood has been found to be more permeable than earlywood from adjacent growth rings. Panshin, DeZeeuw and Brown (1964) postulated that the torus of latewood is often absent, which could cause an increased permeability. Raphael and Graham (1951) felt that the localization of resin ducts in the latewood and the thicker cell walls accounted for the permeability differential. Krahmer (1961) proposed the hypothesis of capillary action due to the smaller lumen in latewood tracheids.

Stone (1939) found latewood to be more permeable to oil than earlywood, but no differences were noted in water permeability comparisons. Stone concluded that the difference was related to the relative moisture contents of earlywood and latewood.

Erickson and Estep (1961) noted a correlation between a higher percentage of latewood and a higher radial permeability. Bramhall (1967) indicated that gas permeability in Douglas-fir was greater in latewood than in earlywood.

However, Crawford (1958) did not distinguish a difference in longitudinal stain penetration of unseasoned earlywood and latewood of Douglas-fir. Miller (1961) also reported that no clear relation existed between penetration and latewood content.

Koran (1961) reported that a linear relationship appeared between percent latewood and creosote retention, but that further statistical analysis showed this relationship to be unjustified.

Specific Gravity

Most researchers have reported that no significant correlation exists between wood specific gravity and permeability (Raphael and Graham, 1951; Miller, 1961; Koran, 1961; Estep, 1961; Koran, 1964). Bramhall (1967), however, found a significant correlation between longitudinal gas permeability and specific gravity in Douglas-fir.

Summary

The cause of refractory behavior in Rocky Mountain Douglas-fir is as yet unknown. Some investigators have found definite correlations to several structural characteristics, but their results are not indisputable, nor are they conclusive.

Extractives

Extractives are the low molecular weight components, extractable from wood by water or organic solvents, excluding components which by definition belong to the holocellulose or lignin fractions (Rydholm, 1965).

Investigations of gas or liquid permeability have indicated a significant relationship between relative wood permeability and extractive content. Koran (1964) found an inverse relationship between both alcohol-benzene and acetone soluble extractive contents and creosote penetration. Other investigators (Miller, 1961; Krahmer, 1961; Craig, 1963) noted that extraction with various organic solvents, or water, greatly increased the relative water permeability of Douglas-fir.

Although kraft liquor impregnation may be somewhat controlled by permeability, the influence of extractives is probably minimal, as virtually all of these components are dissolved or dispersed by the active chemical solution. The major influences of extractives are their reactions with the pulping chemicals, which cause a dilution of liquor strength (Rydholm, 1965).

Various authors have reported the extractive content of Douglas-fir as shown in Table 1 (Lewis, 1950; Legg and Hart, 1960; Miller, 1961; Koran, 1964; Hancock and Swan, 1965; Campbell, Swan and Wilson, 1966). Hancock and Swan (1965) reported that no significant difference existed between the ether soluble extractives of permeable and refractory Douglas-fir. Miller (1961) found that permeable and refractory Douglas-fir lost the same percentage of weight in alcohol-benzene extraction, but differed in their relative rates of extraction. Both varieties, studied by Miller, indicated a 5% weight loss after eight hours of extraction, but the rate of loss for refractory heartwood was about twice as rapid as permeable samples during the first two hours.

Table 1. The extractive content of Douglas-fir heartwood.

Solvent	% Extractive removed by each solvent (Original O. D. weight basis)	
	Refractory samples	Permeable samples
Ether	0.31 to 6.14	0.39 to 1.30
Alcohol	no value	3.81
Alcohol-benzene	4.50 to 7.01	2.20 to 5.00
Water	no value	1.04 to 3.60

Koran (1961) reported that the effect of alcohol-benzene and acetone soluble extractives on wood permeability was not proven statistically significant. He did note, however, that higher extractive contents gave higher creosote retentions.

Fiber Characteristics

Due to the extreme variability of the wood properties of Douglas-fir, investigation of the fiber dimensions was required for full analysis of the pulping characteristics of permeable and refractory wood.

Generally, the average tracheid length of Douglas-fir was found to increase for the first 20 to 50 years of growth, but after a certain point a maximum size was reached and the average length remained constant, although the individual fiber lengths varied greatly (Spurr and Hyvarinen, 1954). Anderson (1951) reported that average tracheid length increased with distance from the pith, independent of height. Latewood tracheids have been found to be longer than earlywood tracheids taken from equivalent tree samples (Spurr and Hyvarinen, 1954). The tracheids of Douglas-fir range from three to six millimeters in length and overlap from one-fourth to one-third of their length (Burr and Stamm, 1947). Tracheid length and lumen area are directly related, indicating that cross sectional fiber area follows the same relationship previously noted for fiber length (Graff and Miller, 1939).

Fleischer (1950) found that tracheids of permeable Douglas-fir were significantly longer than those of refractory woods, and that the lumen cross-sectional size was greater. Krahmer (1961) also found a significant difference in the average fiber length of permeable (5.59 mm) and refractory (3.68 mm) Douglas-fir, and found that lumen diameters of refractory samples were significantly smaller than those of permeable wood. Fibers in the permeable samples appeared to be hexagonal in cross section with a double row of bordered pits on the radial wall, while fibers in refractory samples were square in cross section with only one row of bordered pits on the radial wall.

Effects of Fiber Characteristics on Paper Properties

The effects of fiber characteristics on paper properties have been examined by many authors who have shown significant correlations between certain fiber dimensions and physical properties of paper. No relationship has been noted between pulp yield and fiber characteristics. Barefoot, Hitchings and Ellwood (1964) reported that up to 93% of all paper property variation was explained by fiber characteristics.

Barefoot et al. (1964) reported that beating time, to a given Canadian Standard Freeness, was inversely related to both the average cell wall thickness and to the Runkel ratio. They determined that while cell wall thickness accounted for 78% of the variation in beating time of loblolly pine (Pinus taeda L.), the Runkel ratio would account for 85% of the variation. Dinwoodie (1966) found beating time to be inversely related to the Runkel ratio in Sitka spruce (Picea sitchensis (Bong.) Carr.).

Barefoot et al. (1964) reported that Mullen strength in loblolly pine was inversely related to both latewood cell wall thickness and the Runkel ratio, and that each would explain 75% of the variation when considered separately. Fiber length was directly related to Mullen strength, but at a lower degree of significance. These findings were confirmed by Dinwoodie (1966) for Sitka spruce, and by Einspahr (1964) for slash pine (Pinus elliotii Engelm.).

Tensile strength in loblolly pine was inversely related to latewood cell wall thickness and to the Runkel ratio, which respectively accounted for 82% and 76% of the variation. A lesser degree of significance was reported for a direct relationship to fiber length (Barefoot et al., 1964). These relationships were confirmed in Sitka spruce by Dinwoodie (1966), and in slash pine by Einspahr (1964).

Barefoot et al. (1964) found that latewood cell wall thickness was directly related to tear strength in loblolly pine, and accounted for 74% of the variation. This relationship was confirmed in work by Jayme (1958), Dadswell (1962), and Dinwoodie (1966). No relationship to fiber length was noted by Barefoot et al. (1964), but a direct relationship was noted by Clark (1942), Hentschel (1959), Wangaard (1962), and Dinwoodie (1966). Barefoot et al. (1964) reported a direct relationship to the Runkel ratio in loblolly pine, which accounted for 58% of the variation.

Stretch was reported to be directly related to fiber length in Sitka spruce (Dinwoodie, 1966). A similar relationship was noted by Clark (1958).

Fold endurance was reported as directly related to fiber length by Clark (1942), Hentschel (1959), and Wangaard (1962). Dinwoodie (1966) reported that fold endurance may be inversely related to cell wall thickness in Sitka spruce.

Dinwoodie (1966) related Canadian Standard Freeness directly to fiber length in Sitka spruce. A similar relationship was noted by Clark (1958).

In summary, paper properties were functions of fiber characteristics. Beating time, Mullen, and tensile strength were inversely related to both the latewood cell wall thickness and the Runkel ratio. Freeness, stretch, fold, and tear were directly related to fiber length. The tear factor was also reported as directly related to latewood cell wall thickness and to the Runkel ratio. Since previous investigations (Fleischer, 1950; Krahmer, 1961) had determined that refractory Douglas-fir contained fibers of significantly shorter fiber length with thinner cell walls, it was hypothesized that refractory paper would give higher Mullen and tensile values, lower stretch, fold, and tear values, give a lower initial freeness, and require a longer beating time than permeable papers.

EXPERIMENTAL PROCEDURE

Sample Selection

The heartwood samples of Douglas-fir were collected from sawmills located in areas of known relative wood permeability, as established by the work of Graham and Miller (1963). Samples of Rocky Mountain Douglas-fir were obtained from mills located in Joseph, Elgin, La Grande, Union, and Prairie City, Oregon. Samples of coastal Douglas-fir were obtained from mills in Medford and Grants Pass, Oregon.

At each mill site, material was selected in the form of freshly cut, green, dimension lumber. Each piece selected in a mill sample conformed to a set criterion which allowed acceptance only for boards containing minor amounts of included sapwood, or juvenile wood, and showing no evidence of rot, reaction wood, or abnormal knot formation. The selection method created a stratified random sample of the heartwood lumber cut at each mill at the time of selection.

Sample Preparation

From one end of each board a 13-inch-long piece was taken for permeability tests and fiber analysis. The remainder of the board was cut to eliminate included sapwood, juvenile wood, and knots, then chipped in a twin knife laboratory chipper. Fines and excessively large chips were screened from the samples. The acceptable fractions, from 15 to 25 mm in length and from 2 to 4 mm in thickness, were sealed in polyethylene bags, and stored in a cold room maintained at 36° F.

Permeability Rating

Miller's (1961) copper sulfate impregnation rating test was utilized to establish relative permeability. From the 13-inch long pieces from each board, two end-matched permeability blocks, measuring six inches along the grain and one square inch in cross section, were prepared. One block from each board was measured for growth rate, and kept in the green condition for testing. Prior to testing, the volume of the second block was measured by water displacement. The block was then oven dried at 105° C for specific gravity determination.

Green and oven-dried blocks were treated separately with a 5% aqueous copper sulfate solution for 30 minutes at a temperature of 80° F and an air pressure of 110 psi. After treatment, the blocks were removed from the solution, wiped clean, split radially, exposed to hydrogen sulphide gas to form a black precipitate of copper sulfide, and then visually rated for penetration using standards shown in Figure 1.

Blocks given impregnation ratings from 1.0 to 1.5 were considered refractory, while those rated 5.5 to 6.0 were considered

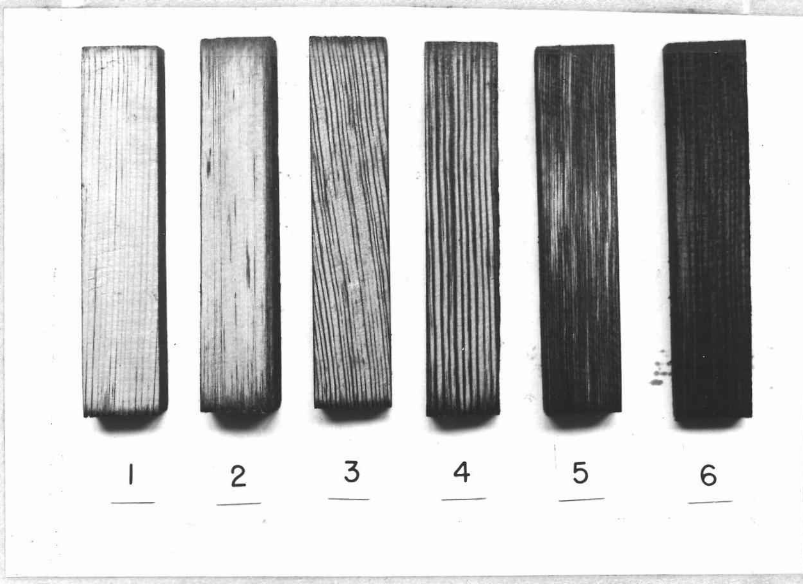

Figure 1. Previously treated and selected specimens used as standards for impregnation rating (Courtesy Oregon Forest Research Laboratory).

permeable. All blocks rated 2.0 to 5.0 were considered intermediate in permeability and the corresponding samples were discarded.

Fiber Analysis

Nine permeable and ten refractory Douglas-fir fiber analysis samples were prepared from the 13-inch test blocks corresponding to wood specimens used in the pulping trials. One-inch cubes were cut from the outermost growth rings of each piece. From each of the cubes, several growth rings were randomly chosen for cross sectional analysis. Single adjacent growth rings were chosen for maceration, and separate samples were prepared from both earlywood and latewood.

Wood sections, 15 μ thick, were made by standard cross sectioning techniques with a sliding knife microtome. Saffarin stained, glycerin mounts were used. Measurements of lumen diameter and cell wall thickness were made for both earlywood and latewood with a binocular microscope set at 400 power, using the following technique: The approximate center of both earlywood and latewood was used for measurement. Five radial rows of cells were chosen randomly. From each row, five adjacent cells were measured for tangential and radial lumen diameters, and for average cell wall thickness. Average Runkel ratios were calculated from measured lumen diameter and cell wall thickness values.

Maceration was performed by treating the wood with an acidified sodium chlorite solution as described by Spearin and Isenberg (1947). Slide preparation was by Echol's (1968) method of Chlorozol Black 'E' stain, and polyvinyl acetate mount on glass slides. Twenty-five fibers from each slide were randomly chosen, and measured for fiber length. Individual fiber lengths were established by direct measurement from the Forest Products Department's ampliscope. The ampliscope was set to project enlarged fiber images onto a calibrated screen for direct measurement of fiber length.

Fiber diameters were measured microscopically on macerated fibers, and compared with the fiber diameters determined by the cross sectioning study to verify that statistically comparable fibers were measured in each analysis.

Extraction

A random sample of chips from each wood specimen was selected, and oven dried at 105° C. Individual chip samples were disintegrated in a Wiley mill until the wood meal particles would pass through a 40 mesh screen. Wood meal was re-dried at 105° C.

Six refractory and six permeable Douglas-fir wood meal samples were exhaustively extracted sequentially with hexane, benzene, ethyl ether, ethyl alcohol, and water. Normal techniques and standard Soxhlet extraction apparatus were used. Extractions with hexane, benzene, ethyl ether, and ethyl alcohol were each performed for 36 hours. An average solvent exchange rate of five per hour was established, yielding an average of 180 solvent exchanges. Water extraction proceeded at a much slower rate of about two exchanges per hour, requiring a total extraction time of 90 hours.

After each series of solvent extractions, the samples were removed, air dried for 12 hours, oven dried for 36 hours, cooled in a dessicator, and weighed to determine weight loss. No qualitative analysis of extractives was attempted. Quantative analysis was calculated as the percent weight loss through each extraction.

Lignin Determination

Klason lignin determinations were performed on three refractory and three permeable Douglas-fir heartwood samples in accordance with TAPPI Standard T 13 m-45.

Sink-Float Test

The sink-float test developed by Graham (1964) was evaluated for its effective use on green chips. No modifications of the treatment cycle were made, but green chips were tested in lieu of oven-dry wood plugs. Three chip samples of refractory, and three of permeable Douglas-fir were separately evaluated. Twenty-five chips were tested in each trial, and two repetitions of each sample were performed.

Pulping Procedures

Two duplicate kraft cooks of each original wood sample were performed in a stainless steel digester of 12 liter capacity. Liquor was circulated and heated by an external steam heat exchanger. The temperature cycle was controlled by a Honeywell Electronic 15 cam controller. Chip samples were measured for percent solids content. Individual chip charges of 908 gms (oven dry basis) were placed in a fine mesh, wire screen container and sealed in the digester for each cook. The cooking schedule is shown in Table 2.

The simulated white liquor was an aqueous solution of sodium hydroxide and sodium sulfide. The high liquor to wood ratio (8.5:1) was necessary to completely cover the chips throughout the cooking

Table 2. Pulping conditions.

Chemical	
Active alkali (as Na_2O)	32 gpl
Sulphidity	22 %
Liquor to wood ratio	8.5:1
Schedule	
Impregnation time	45 min.
Cooking time	120 min.
Blow time	5 min.
Digester conditions	
Final temperature	340° F
Final pressure	105 psig
Wood input	
Total chip weight per cook (O.D.)	908 g

cycle.

Immediately after blowing each cook, unscreened yield determinations were performed by sampling the freshly cooked chips, obtaining their wet weight, disintegrating them in a high speed blender, washing out all extraneous materials on a Buchner funnel, oven-drying the remaining fiber, and obtaining the oven-dry weight. Unscreened yield was determined by the formula:

$$\text{Unscreened yield } (\%) = \frac{\left[\left(\frac{\text{O.D. sample wt.}}{\text{Wet sample wt.}}\right) \times \text{Total, wet chip wt.}\right]}{\text{Original O.D. total chip wt.}} \times 100$$

A black liquor sample was taken when each cook was blown. After cooling to room temperature, samples were analyzed for active Na_2O and total Na_2O by volumetric titration of five cc of black liquor with HCl. Active and total Na_2O contents were determined by the formulae:

Active Na_2O (gpl) = Volume HCl to pH 7.5 x Normality HCl x 6.2

Total Na_2O (gpl) = Volume HCl to pH 4.0 x Normality HCl x 6.2

Pulp Preparation

After pulping, cooked chips were equally divided into two five gallon buckets, and sufficient water added to bring the volume to four gallons in each bucket. Water temperature was maintained at approximately 30° C. Each chip suspension was agitated for 15 minutes with a stirrer manufactured in accordance with TAPPI Standard T 200 ts-66.

Subsequent to disintegration, pulp slurries were screened in a Valley Laboratory Pulp Screen, with .009" wide slots, to remove shives remaining in the suspension. Water flow was approximately 10 gallons per minute. Pulp which passed through the screen was accepted for further work; rejects were removed, oven dried, and weighed for screened yield calculation. The screened yield was estimated by subtracting the weight of oven-dry screenings from the calculated oven-dry unscreened pulp weight. This figure was then divided by the original oven-dry chip weight to give screened yield in percent.

Screened pulp was collected, dewatered, sealed in polyethylene bags containing a few drops of formaldehyde, and stored in a cold room maintained at 36° F.

Pulp Testing

A Kappa number determination was made on a random sample of each pulp in accordance with TAPPI Standard T 236 m-60. Two determinations were made and averaged to give the value reported for each cook.

Two refractory and two permeable cooks were analyzed for fiber classification in a Bauer-McNett Fiber Classifier. Each cook was tested at each of the four beater intervals made during refining. Screen sizes were set at 20, 35, 65, and 150 mesh. This test was performed in accordance with TAPPI Standard T 233 Su-64.

Pulp Refining and Handsheet Preparation

Pulp refining was performed in a Valley beater in accordance with TAPPI Standard T 200 ts-66. Samples for one Canadian Standard Freeness evaluation, six handsheets, and 1000 cc of pulp slurry were taken at 0, 15, 30, and 45 minute intervals. From each of the two duplicate cooks per wood sample, 180 gms (oven dry basis) of pulp were removed and combined for one beater run.

Canadian Standard Freeness evaluations were made in accordance with TAPPI Standard T 227 m-58. Handsheets were formed in accordance with TAPPI Standard T 205 m-58. The 1000 cc pulp samples were formed into pads on a Buchner funnel, and air dried for Bauer-McNett fiber classification.

Handsheet Testing

After sheet formation, handsheets were conditioned for a minimum of 48 hours, then tested at 23° C and 50% R.H., as set by TAPPI Standard T 402 m-49. Five of the six handsheets were selected for physical tests at each beater interval, with the remaining sheet saved for reference.

Prior to physical testing, average sheet weight, caliper, and density were determined in accordance with TAPPI Standard T 220 m-60. Brightness was determined with an Elrepho Colorimeter, at filter position number eight, by averaging five readings taken from the rough face of each sheet. Operation of the Elrepho followed TAPPI Standard T 425 m-58.

Physical testing of handsheets was performed in accordance with TAPPI Standard T 220 m-60, with the exception of sheet division. Each of the five handsheets per beater interval were prepared for physical testing by cutting to the diagram shown in Figure 2. At each beater interval, ten Mullen, five tensile, five stretch, five fold, and four tear tests were performed.

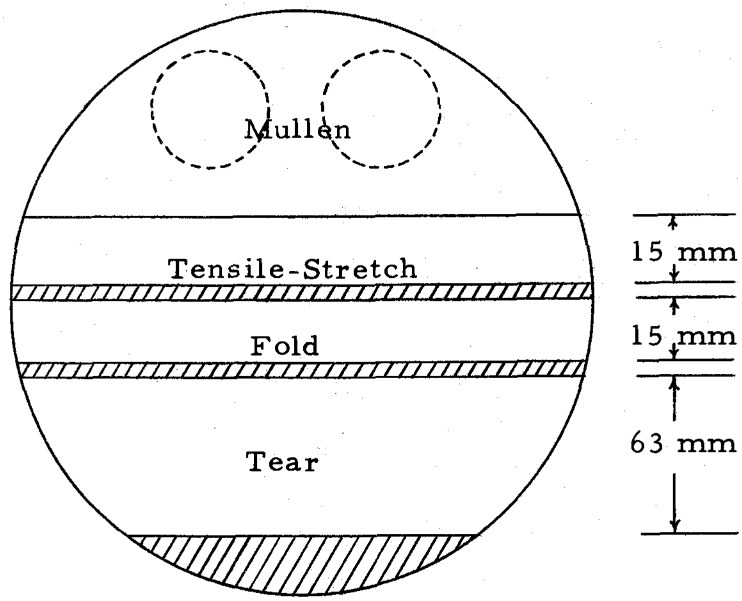

Figure 2. Cutting diagram for handsheet testing.

Tensile strength and stretch tests were performed simultaneously on an Instron TT-BML set to a crosshead speed of one cm per minute, in accordance with TAPPI Standards T 404 ts-66, and T 457 m-46. Mullen tests were performed on a Perkins Model C Mullen Tester in accordance with TAPPI Standard T 403 ts-63. Internal tearing resistance was measured on an Elmendorf Tearing Tester in accordance with TAPPI Standard T 414 ts-65. Folding endurance was measured on an M. I. T. Fold Tester in accordance with TAPPI Standard T 423 m-50.

Three refractory and three permeable paper samples were randomly chosen and tested for zero-span tensile strength on an Instron TT-BML, set to a crosshead speed of one cm per min., in accordance with TAPPI Standard T 231 sm-60. Samples for zero-span analysis were obtained from the sheet remnants of tear tests at the zero minute beating interval.

Statistical Analysis

The Student's "t" Test was used to determine the relative significance of differences found between the two varieties of Douglas-fir. The 0.10-probability level was used as the highest numerical criterion for significance.

A stepwise, multiple linear regression analysis (O. S. U. program OSU-01) was used to determine the correlations between paper strength values and fiber characteristics. The independent variables chosen for this analysis were (1) wood growth rate, (2) wood specific gravity, (3) earlywood and latewood fiber length, (4) earlywood and latewood tangential lumen diameter, (5) earlywood and latewood radial lumen diameter, (6) earlywood and latewood cell wall thickness, (7) earlywood and latewood Runkel ratio, and (8) handsheet density.

Two analyses were made at 'F' levels of zero. The first included all independent variables in the regression. The second included all independent variables with the exception of handsheet density and the Runkel ratios. The regression equations were generated in the form:

$$Y = A + b_1x_1 + b_2x_2 + \ldots b_nx_n.$$

The generated equations, however, included all programmed variables and, after a certain point, r^2 was heavily influenced by the loss of degrees of freedom. This point in each program was determined to be the step at which the standard error of Y began to increase. The "significant variables" were, therefore, defined as those which entered the equation prior to the minimum value of the standard error of Y. All variables entered after the minimum value of the standard error of Y were considered to be relatively unimportant as their presence in an equation did not increase the accuracy of prediction.

For each equation, the "significant variables" were further subdivided into three classifications: (1) the single most important estimator, (2) the "significant variables" of secondary importance, and (3) the remaining "significant variables." The computer's order of variable entrance was determined by the maximum contribution to r^2 when previously entered variables were retained. By this definition, the single most important estimator was the variable entered first in the regression equation. "Significant variables" of secondary importance were considered to be those which were highly correlated to the property under investigation, but at a lower level than the single most important estimator. Variables of secondary importance were defined as those with a simple correlation coefficient greater than 0.5, and whose regression coefficient was significant at the 0.10 level by the Student's 't' test. As a general rule of thumb, the variables of secondary importance were those entered at an equation 'F' level greater than 1.0. The third group included all remaining "significant variables."

The amount of explained variation was represented by r^2. In the discussion to follow, the average r^2 of the single most important estimator was given, while the minimum and maximum r^2's of the six equations were reported to indicate the total range. Also in the discussion, (+) was used to indicate a direct relationship (positive correlation) between an individual variable and a handsheet property, while (-) indicated an inverse relationship (negative correlation).

RESULTS AND DISCUSSION

Wood Parameters

As prerequisites to the meaningful analysis of pulping characteristics, several wood properties were evaluated. Measurements of relative permeability, specific gravity, growth rate, and percent solids were performed to qualify the samples, and are tabulated in Table 1 of the Appendix. Measurements of fiber characteristics, extractive content, and Klason lignin were performed to aid in the evaluation of pulping properties, and are tabulated in Tables 2 and 3 of the Appendix.

Permeability

A total of 41 boards of Rocky Mountain Douglas-fir, and 25 boards of Coastal Douglas-fir, were evaluated for relative wood permeability using Miller's copper sulfate impregnation technique. Of the 41 boards of Rocky Mountain Douglas-fir, only 11, or 26.8%, met the criterion to be classified refractory. From the 25 boards of Coastal Douglas-fir, a total of 11, or 44%, met the criterion to be classified as permeable. The results are summarized in Table 3. All other boards were considered to be relatively intermediate in permeability.

As the prime concern of this study was the extremes of permeability, only the 22 boards rated as either refractory or

Table 3. Number of boards given various impregnation ratings.

Impregnation rating	Coastal Douglas-fir	Rocky Mt. Douglas-fir
1.0 to 1.5	0	11
2.0 to 3.5	1	30
3.5 to 5.0	13	0
5.5 to 6.0	11	0

permeable were selected for further use. All other specimens were discarded. Initial failures in pulping trials reduced the total usable sample to nine permeable and ten refractory Douglas-fir heartwood boards.

Specific Gravity

Measurements for specific gravity (SpGr) indicated a significant difference between the refractory and permeable wood specimens used in this study. The specific gravity of the refractory specimens averaged .4614, while permeable specimens averaged .4341.

Growth Rate

The growth rates of the refractory and permeable specimens were not significantly different. Refractory specimens had an average growth rate of 17.8 rings/inch, while permeable specimens averaged 14.6 rings/inch.

Moisture Content

No significant difference was noted between the moisture contents of refractory and permeable specimens. The moisture contents averaged 31.92% (68.08% solids) in the refractory specimens, and 32.53% (67.47% solids) in the permeable specimens.

Fiber Analysis

A fiber analysis was performed to evaluate the characteristics of fiber length, lumen diameter, and cell wall thickness. The fiber characteristic measurements are tabulated in Table 2 of the Appendix.

The average fiber lengths (FL) of refractory Douglas-fir heartwood specimens were found to be significantly shorter than those of permeable specimens. The FL of refractory Douglas-fir averaged 3.49 mm in earlywood, and 3.74 mm in latewood. The FL of permeable Douglas-fir averaged 5.03 mm in earlywood, and 5.52 mm in latewood.

The average tangential lumen diameters (TLD) of refractory specimens were found to be significantly smaller than those of permeable specimens. Refractory specimens had an average TLD of 33.37 μ in earlywood, and 20.06 μ in latewood. Permeable specimens had an average TLD of 40.82 μ in earlywood and 23.06 μ in latewood.

The earlywood, radial lumen diameters (RLD) of both refractory and permeable specimens were larger than the TLD's. The earlywood RLD's of refractory specimens were found to be significantly smaller than those in permeable specimens. The average RLD in refractory earlywood averaged 34.19 μ, while those of permeable wood averaged 44.73 μ. The RLD's in latewood showed no significant differences.

The average cell wall thickness (CWT) of refractory specimens was found to be significantly thinner than that of permeable specimens. The average CWT in refractory specimens averaged 3.29 μ in early-wood, and 6.62 μ in latewood. In permeable specimens, the CWT averaged 4.11 μ in earlywood and 9.58 μ in latewood.

The Runkel ratio (RR) was calculated for each wood specimen for use as an estimator of paper properties. No significant difference was noted between the values for earlywood of the two varieties, but latewood values were significantly different. The latewood RR of refractory specimens averaged .6692, while that of permeable specimens averaged .8460.

<u>Extractive Content</u>

Significant differences were noted between the quantity of alcohol and water soluble extractives present in the wood samples. No significant differences were noted between the hexane, benzene, and ethyl ether soluble extractives of the two varieties. The significantly lower alcohol and water soluble extractive contents of permeable Douglas-fir were probably natural in occurrence, as no experimental variables existed which would account for the difference. Table 4 summarizes the results of the extractions, while Table 3 of the Appendix tabulates the individual values recorded for each specimen.

Table 4. Average extractive contents of refractory and permeable Douglas-fir specimens.

Solvent	% Extractive Content (O. D. original wood weight basis)	
	Permeable samples	Refractory samples
Hexane	0.939	0.601
Benzene	0.532	0.435
Ether	0.183	0.212
Alcohol	2.756	3.319
Water	11.238	14.261

Lignin Content

As shown in Table 5, the average Klason lignin contents of refractory and permeable specimens were not significantly different. Klason lignin contents were substantially below the 29 to 30% figures reported by Kurth (1948) for Douglas-fir. The cause of the discrepancy was unknown, but the relative rankings of the two varieties were considered reliable.

Table 5. Average Klason lignin contents of permeable and refractory Douglas-fir specimens.

Sample number	% Klason lignin
Permeable samples	
5	26.85
6	27.20
9	26.10
Average	26.72
Refractory samples	
10	25.55
16	26.55
19	26.05
Average	26.05
Student's 't' test data	
't'	1.571
Sig. level	0.192

Sink-Float Test

Three permeable and three refractory chip samples were subjected to Graham's (1964) sink-float test. The results conclusively indicated that the technique was applicable to green chips, and that the refractory and permeable samples may readily be distinguished. An average of 94% of all permeable chip samples sank after six minutes of vacuum application. No permeable chip samples remained floating after the second application of vacuum. An average of 96% of all refractory samples remained floating after the second application of vacuum. No refractory samples sank after the first six minute vacuum application. However, it must be noted that further work is necessary to evaluate the effectiveness of the technique on chip material of intermediate permeability. The results of the test are summarized in Table 4 of the Appendix.

Pulping Trials

Throughout this study, the pulping conditions were maintained constant. The time-temperature relationship was monitored and maintained to a maximum deviation of $\pm 5^{\circ}$ F and an average variation of $\pm 2^{\circ}$ F. If the pulping temperature deviated from these limits, the cook was rejected. Table 5 of the Appendix tabulates the individual values recorded for each pulping variable.

The simulated white liquor was prepared with a goal of 32.0 gpl (as Na_2O) active alkali and 22.0% sulphidity. Active alkali (as Na_2O) averaged 32.05 gpl for "refractory cooks," and 32.03 gpl for "permeable cooks." Sulphidity averaged 21.96% for "refractory cooks," and 21.91% for "permeable cooks." The differences were not significant.

Analysis of the calculated yield figures indicated three important relationships. The unscreened yields of the two varieties were not significantly different. "Refractory samples" pulped to an average unscreened yield of 45.30%, while "permeable material" pulped to an average of 44.89%. When comparing screened yield, however, a significant difference at the .11 level was noted. The average screened yield of refractory samples was 43.27%, while that of permeable material was 44.45%. This small difference was caused by a highly significant difference between screened rejects. The screened rejects of "refractory samples" averaged 2.07%, while "permeable rejects" averaged only 0.54% based on original, oven dry, chip weight.

Analysis of the spent black liquor indicated that cooks of refractory and permeable samples consumed the same amount of active chemical. Active Na_2O content of the spent black liquor averaged 14.59 gpl for "refractory cooks," and 14.89 gpl for "permeable cooks." Total Na_2O content averaged 21.19 gpl for "refractory cooks," and 21.16 gpl for "permeable cooks."

Kappa number determinations indicated a highly significant difference between the lignin contents of "refractory" and "permeable pulps." "Refractory pulp" had an average Kappa number of 27.45, while "permeable pulp" measured 21.59.

The experimental results suggested that refractory and permeable Douglas-fir did not undergo the same degree of delignification. This conclusion was based on the higher Kappa number, higher screened rejects, and slightly lower screened yield of refractory material. Since the relative lignin contents of the two varieties was the same, the difference could possibly be explained by a differential degree of liquor penetration during the impregnation phase of the kraft process. It was believed that, in refractory samples, the lack of full penetration created a "burned" chip core. Rydholm (1965) attributed "burned" chip cores to carbohydrate hydrolysis and lignin condensation above the critical temperature of 120° C. This "burning" effect resulted in the higher screened rejects and lower screened yield of "refractory cooks." The higher Kappa number of "refractory pulps" indicated a higher lignin content, again, an indication of a lower degree of delignification. The equivalent chemical consumption values may be partially accounted for by the higher alcohol and water soluble extractive contents of refractory samples. Many of these compounds are known to undergo lignin-like reactions with kraft pulping liquor under the conditions maintained in the digester (Rydholm, 1965).

Refining

Refractory and permeable Douglas-fir pulps were refined in a Valley beater. The time-freeness data for individual cooks are tabulated in Table 6 of the Appendix, and average beater curves are plotted in Figure 3.

Beater curve evaluations indicated that "refractory pulps" required significantly less refining time to a given degree of Canadian Standard Freeness (CSF), and had a significantly lower initial CSF than did "permeable pulps." A significant refining difference was also noted when comparisons were made at an average sheet density of .7 gms/cc. To achieve a sheet density of .7, "refractory pulps"

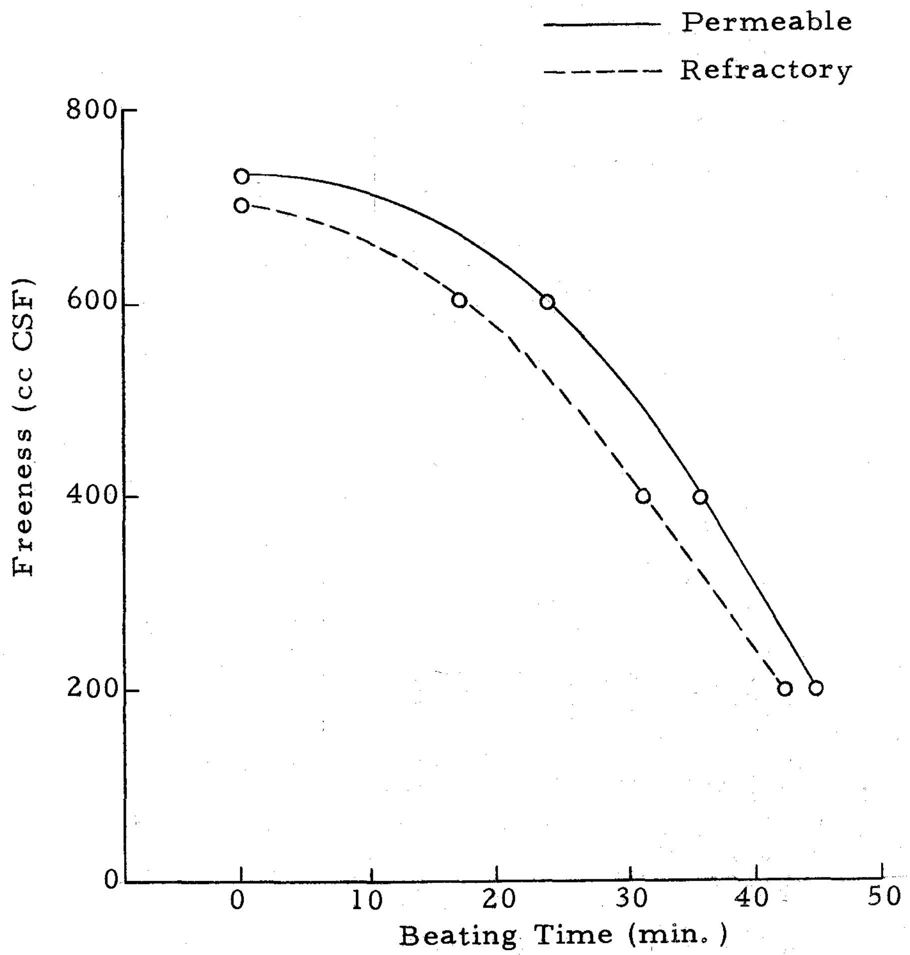

Figure 3. Average freeness.

required an average beating time of 9.9 minutes to an average CSF greater than 700, while "permeable pulps" required 29.7 minutes of beating time to an average CSF of 515.

The regression analysis was used to determine the degree of correlation of fiber characteristics and beating time. Table 14 of the Appendix tabulates the regression equations and allied data used in this analysis.

The FL (+) was the most important single estimator of beating time to the 600 and 400 CSF levels. FL accounted for 62% of the variation in beating time to the 600 CSF level, and 53% of the variation to the 400 CSF level. CWT (+) and SpGr (-) were correlated to beating time at the 600 and 400 CSF levels, but at lower degrees of significance than FL. At the 200 CSF level, TLD (+) was the most important single estimator, accounting for 30% of the variation in beating time. RLD (+), RR (-), FL (+), and CWT (+) were also significantly correlated. From 66 to 80% of all beating time variation was explained by regression equations containing all "significant variables."

Bauer-McNett Fiber Classification

Bauer-McNett fiber classifications served to amplify the known differential in refractory and permeable Douglas-fir fiber lengths. Two "refractory" and two "permeable pulp" samples were classified for fiber length at the four beater intervals. The results were summarized in Table 6.

The values verified the known fiber length differential at the zero beating interval, and showed that refining greatly increased the proportion of short fibers in refractory samples. The increased quantity of short fibers was attributed to a greater degree of cutting action experienced by the "refractory pulps." The effect of refining on the permeable samples was much less pronounced, indicating a lesser degree of actual fiber cutting by the beater roll.

Handsheet Properties

Evaluations of paper strengths were performed on handsheets prepared at 0, 15, 30, and 45 minute beating intervals. Tables 7 through 13 of the Appendix tabulate the average handsheet properties at the initial, 600, 400, and 200 CSF levels. Figures 4 through 10 graphically summarize the average values for each characteristic at the four freeness levels.

The regression analysis was used to determine the degree of correlation between fiber characteristics and the various handsheet properties. Table 14 of the Appendix tabulates the regression equations and allied data used in this analysis. The correlations established between handsheet properties and fiber characteristics were in general agreement with the previous work of Barefoot et al. (1964), Dinwoodie (1966), and Einspahr (1964).

Table 6. Average Bauer-McNett fiber classification results.
Description of columns: (O.D. weight basis)
N. Beater interval (minutes)
1. % of fibers on 20 mesh
2. % of fibers on 35 mesh
3. % of fibers on 65 mesh
4. % of fibers on 150 mesh
5. % of fibers past 150 mesh

$$* \quad \Delta\% = \frac{(\text{value at 0 min. interval} - \text{value at 45 min. interval}) \times 100}{\text{value at 0 min. interval}}$$

N	1	2	3	4	5
		Permeable samples			
0	83.22	6.00	4.12	3.16	3.50
15	81.79	6.29	4.57	3.47	3.88
30	76.10	7.45	7.58	3.10	5.77
45	56.34	14.76	9.49	9.81	9.60
Δ%	(-32)	(+146)	(+130)	(+210)	(+174)
		Refractory samples			
0	56.74	21.39	12.64	7.44	1.78
15	35.04	24.98	19.62	16.76	4.60
30	21.64	22.74	23.15	27.12	5.35
45	21.88	17.68	23.02	26.96	10.42
Δ%	(-61)	(-12)	(+82)	(+262)	(+485)

Sheet Density

As shown in Figure 4, the average sheet density of "refractory paper" was significantly greater than that for "permeable paper" at constant freeness. Paper strength properties would normally have been compared on the basis of sheet density. However, since average sheet density differed greatly between the "permeable" and "refractory papers," only a common comparison interval of .7 gms/cc was available and used.

Analysis of the regression equations indicated that FL (-) was the best single estimator of sheet density. FL alone accounted for about 70% of the sheet density variation. Strong correlations with RLD (-), TLD (-), and CWT (-) were also noted. From 66 to 84% of the total variation could be explained if all "significant variables" were included in the regression equations.

Tensile Strength

Average tensile strength, as expressed in breaking length, was significantly greater for the "refractory paper" samples, as shown in Figure 5. When compared at .7 gms/cc sheet density, the two varieties showed no significant difference in tensile strength. On the .7 gms/cc basis, "refractory papers" had an average of 10,520 m breaking length, and "permeable papers" had 10,839 m.

Up to 53% of the total variation in tensile strength could be

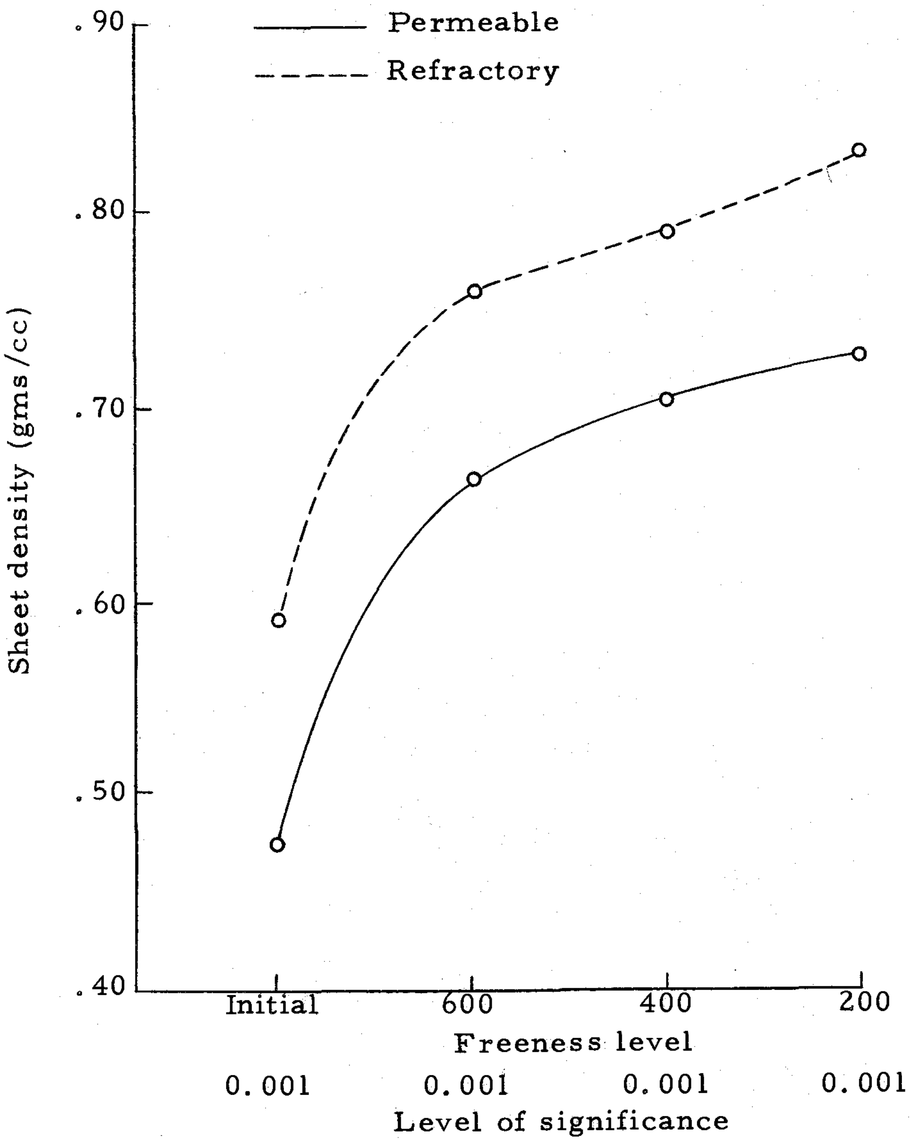

Figure 4. Sheet density development.

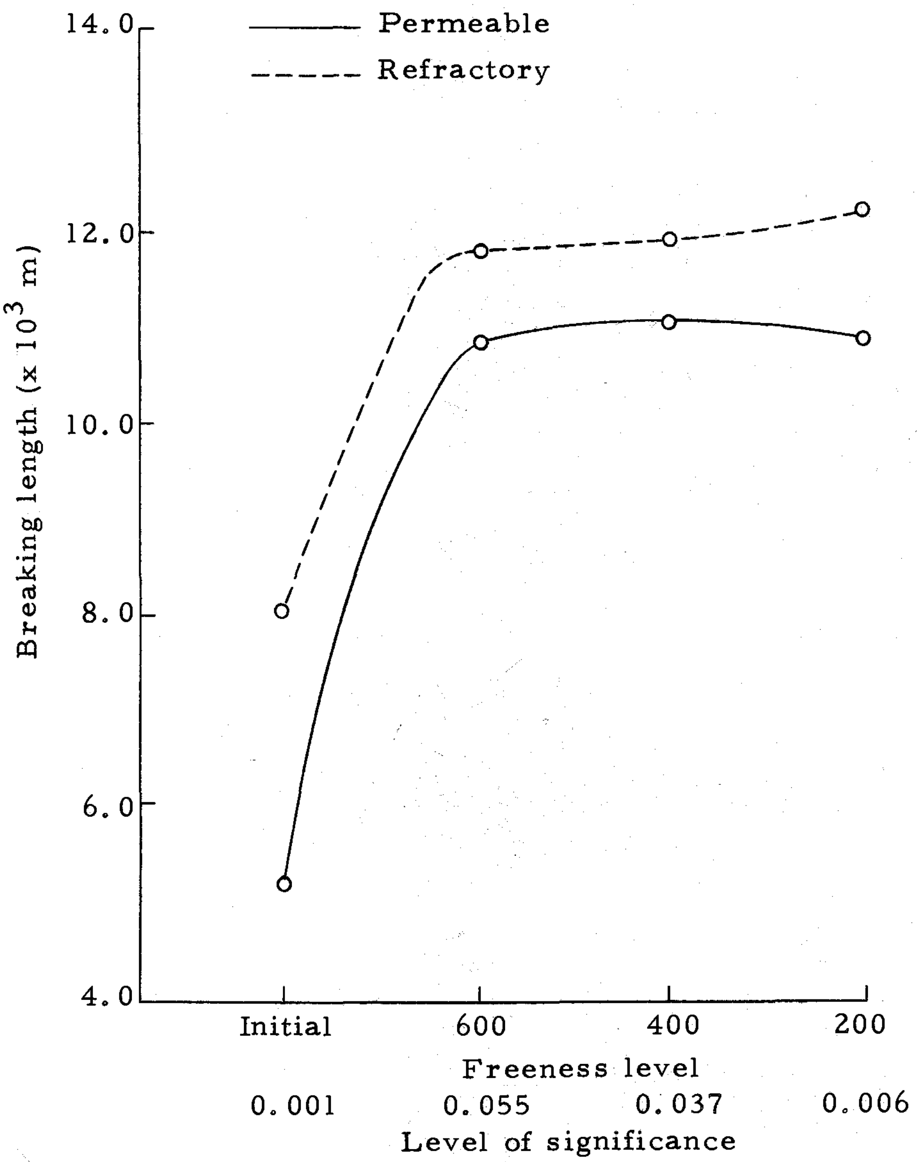

Figure 5. Breaking length development.

accounted for by the interaction of sheet density (+) and the RR (-). Significant correlations were also noted for CWT (-), RLD (-), and SpGr (+). From 58 to 73% of tensile strength variation was explained if all "significant variables" were included in the regression equations.

Zero-Span Tensile Strength

As shown in Table 7, a significant difference was noted between the zero-span tensile strengths of the three "refractory" and three "permeable papers" tested at the zero beater interval.

The ratio of normal tensile strength to zero-span tensile strength may be used as an indication of the relative amount of interfiber bonding (Casey, 1961). The calculated ratios for the samples, as shown in Table 7, indicated a significant difference between the two varieties. "Refractory papers" showed a greater degree of interfiber bonding. Although the values were not subjected to the stepwise regression analysis, it was believed that the greater specific surface of smaller "refractory fibers" caused the increase in interfiber bonding through a greater abundance of potential bonding sites.

Stretch

The average stretch values developed similarly to tensile strength. As shown in Figure 6, "refractory papers" developed significantly greater stretch than did "permeable papers." Virtually no difference

Table 7. Zero-span tensile strength.

Sample number	Average breaking length (m)	Normal tensile/ zero-span ratio
	Permeable samples	
3	12,683	.4270
4	13,834	.4537
5	12,739	.3941
Average	13,085	.4249
	Refractory samples	
13	13,584	.6545
14	14,578	.6002
16	13,949	.6936
Average	14,037	.6494
	Student's 't' test data	
't'	2.009	6.991
Sig. level	0.102	0.001

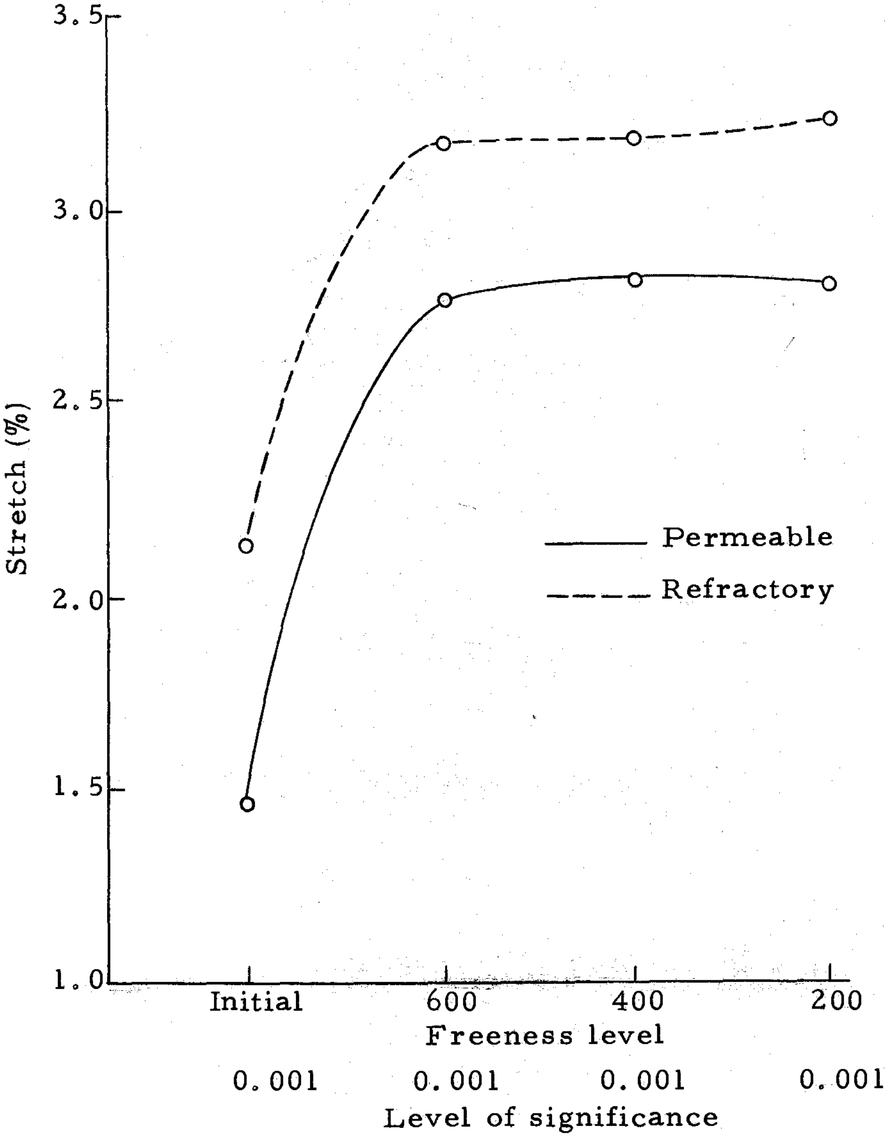

Figure 6. Stretch development.

existed between stretch values compared at the .7 gms/cc sheet density level. At the .7 level, "refractory papers" gave an average stretch of 2.84%, and "permeable", 2.80%.

The FL (-) was the most important single estimator of stretch. It accounted for approximately 60% of the total variation. The RLD (-) was also correlated, but at a lower degree of significance. From 66 to 87% of the total variation was explained if all "significant variables" were included in the regression equations.

Bursting Strength

With the exception of the initial values, no significant difference was noted between the Mullen factors of "refractory" and "permeable papers" at constant freeness. However, as shown in Figure 7, the general shape of the curves approximated the shape of the previously shown curves for tensile and stretch values. On the basis of sheet density at .7 gms/cc, a significant difference between Mullen strengths was noted, where "refractory papers" averaged 61.55 m^2, and "permeable papers" averaged 68.17 m^2.

The RR (-) was the most important single estimator of Mullen strength. However, used alone it would account for only 30% of the total variation. RLD (-), CWT (-), and FL (-) were also correlated, but at a lower degree of significance. Approximately 60 to 76% of the variation was explained if all "significant variables" were included in

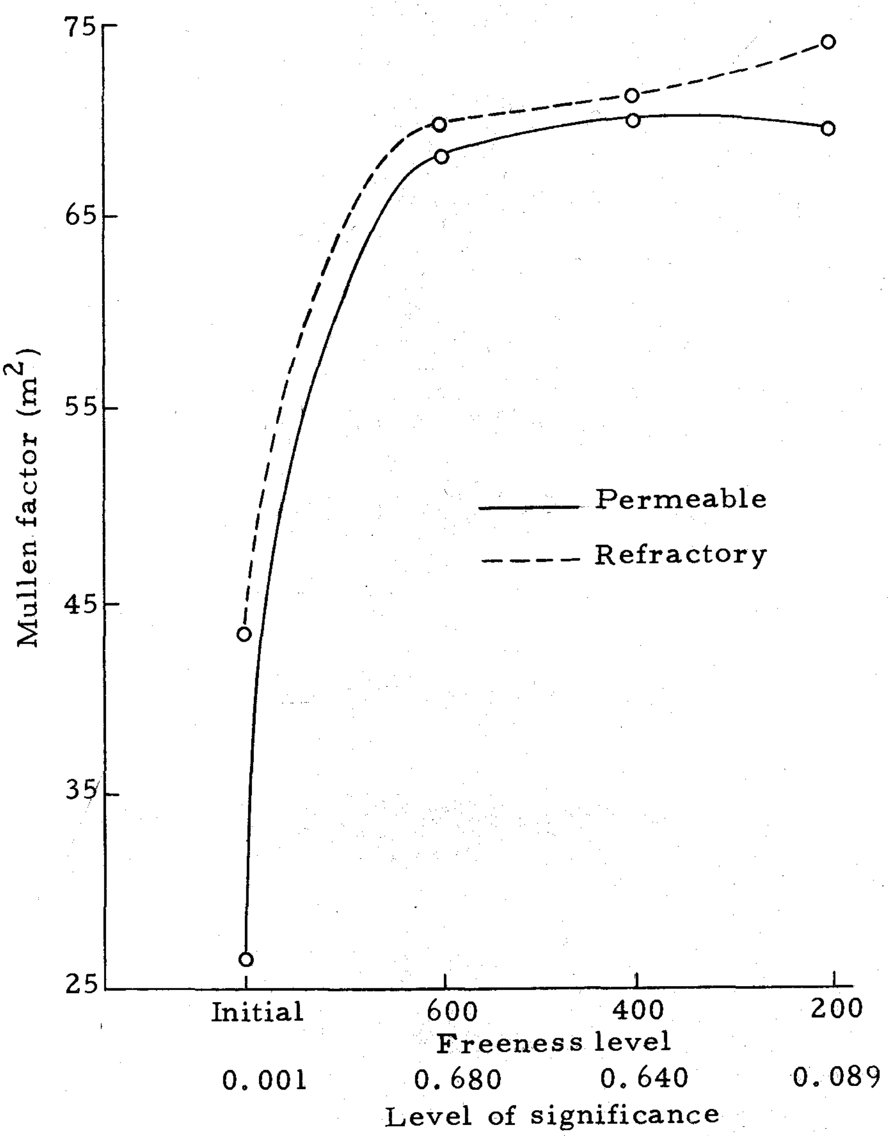

Figure 7. Mullen factor development.

the regression equations.

Internal Tearing Resistance

The average tearing strengths of "refractory papers" were significantly lower than "permeable" values at the freeness intervals reported. The curves, shown in Figure 8, sloped downward at the decreasing rate expected for Douglas-fir. Average tearing strengths were also significantly different when compared at the .7 gms/cc level. At this level, "refractory papers" had an average tear factor of 164.3 dm^2, while "permeable papers" averaged 205.0 dm^2.

FL (+) was the most important single estimator of internal tear resistance. FL alone accounted for 70% of the variation. The CWT (+) was also highly correlated, while SpGr (-), and RLD (+) were correlated at a much lower level of significance. Handsheet density (+) was also a significant estimator of tear strength. When used alone, handsheet density accounted for 73% of all variation. Utilization of all "significant variables" in the regressions would account for 90 to 96% of all variation in tear strength.

Folding Endurance

The average folding endurance was the most variable of the strength properties analyzed. Extreme variations in the recorded values were noted for all handsheet trials, but relatively significant

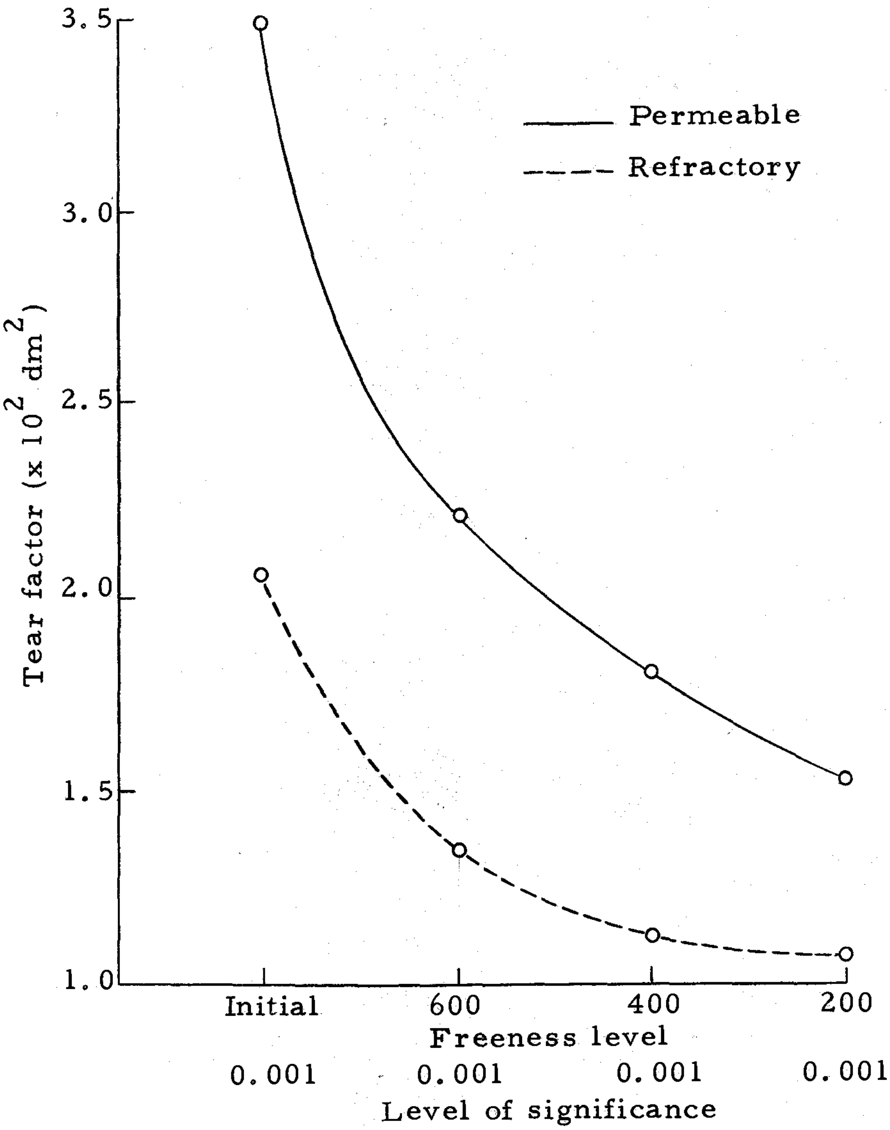

Figure 8. Tear factor development.

differences were noted at all but the 600 CSF level. As shown in Figure 9, the rate of fold endurance development appeared to be equal through the initial stages of refining. However, after approximately the 600 CSF level, the permeable samples gained little strength, while the refractory samples continued to increase in fold endurance. Comparison at the .7 gms/cc sheet density level indicated no significant difference between the fold endurance of "refractory" and "permeable papers." At this level, "refractory papers" averaged 1014 double folds, while "permeable papers" averaged 1129 double folds.

Due to the extreme variations in fold endurance, no definite correlations were established. Values within the regression equations followed no particular pattern, but low level correlations existed with the RR (-), FL (-), and CWT (-) values.

Brightness

Analysis of handsheet brightness indicated a significant difference between "refractory" and "permeable papers." Figure 10 shows the average readings as measured at the four beater intervals. Average brightness dropped significantly, during the first few minutes of refining, for both "refractory" and "permeable papers." As refining continued, "permeable papers" leveled off to a relatively constant brightness, while "refractory papers" continued to decline, but at a lower rate.

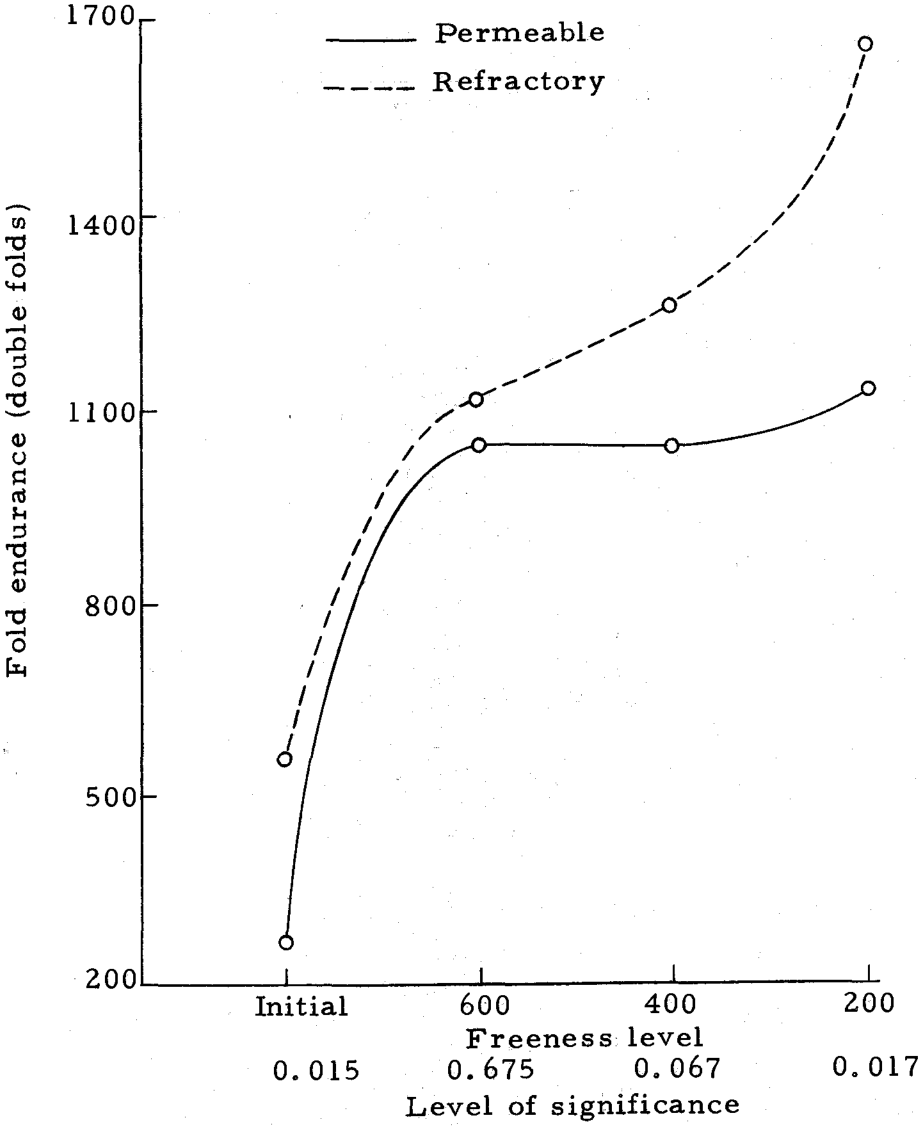

Figure 9. Fold endurance development.

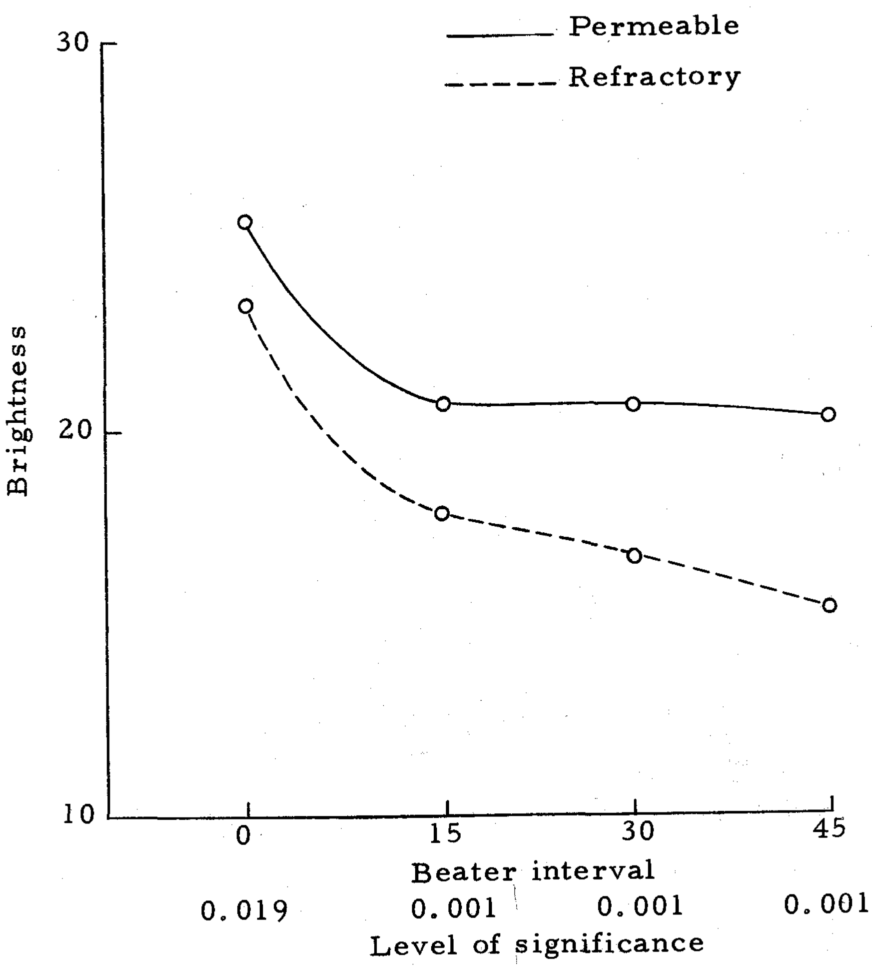

Figure 10. Brightness development.

SUMMARY

Significant differences were noted between the pulping characteristics of refractory and permeable Douglas-fir.

The significant differences in fiber dimensions and wood specific gravity confirmed the results of previous investigators, but the associative variation could not be used to explain refractory behavior. However, the significant difference in alcohol and water extractive contents offered a potential explanation, namely that the amount and mode of extractive deposition in refractory wood may decrease permeability by occlusion of bordered pits. An investigation of this factor was beyond the scope of this study, and neither a qualitative nor a quantitative analysis of individual extractives was performed.

Application of Graham's (1964) sink-float test offered an easy method of differentiating green wood chips of definite refractory or permeable behavior. To be used in full application, further evaluation of the technique would be required to determine its accuracy on samples of intermediate permeability.

Kraft pulping trials indicated that the yields of the two varieties were similar, but that significant differences existed between the percentages of screened rejects. The "burned" appearance of refractory screened rejects and the equal lignin content of the wood specimens led to the assumption that the higher percentage of screened rejects was caused by carbohydrate hydrolysis and lignin condensation

due to inadequate penetration of the pulping liquor. The cause of decreased impregnation was not conclusively established. But, it was believed that the small difference was caused by structural impediments remaining after extractive removal by the hot alkaline medium. At the high temperatures and chemical concentrations used, the extractives should have been readily dissolved and dispersed in the alkaline medium. It was possible that less drastic conditions or other pulping chemicals would have shown greater differences in the rate of chip impregnation.

The lower initial freeness of "refractory pulp" was attributed to the shorter fiber lengths, and not to wood permeability differences. The greater increase in the short fiber fraction through refining indicated that "refractory pulps" experienced a greater cutting action, but the cause was not determined. The higher lignin content, indicated by the Kappa number, of "refractory pulp" did not interfere with refining, as shown by the parallel freeness development of the two varieties. This indicated that adequate delignification had occurred, even though it was not as great as in permeable samples. The real significance of the higher Kappa number was the indication of higher bleach demand for refractory pulps due to a higher lignin content.

The significant differences in paper strength properties were attributed to the variations in fiber dimensions, and not to permeability differences. The correlations between paper properties and wood permeability were statistically significant, but were considered associative correlations which could not offer physical explanations of paper property variation. The factors which might have caused permeability differences in the wood, such as extractive content, extractive deposition, or fiber packing, were considered to be eliminated by the pulping process. The higher lignin content, as indicated by the Kappa number, of refractory pulp did not seem to interfere with fiber bonding, as shown by the satisfactory development of refractory paper strength properties. This fact was considered reliable evidence that adequate delignification of refractory pulp had occurred even though it was less than that of permeable pulp.

CONCLUSIONS

1. Statistically significant differences exist between the kraft pulping characteristics of permeable and refractory Douglas-fir heartwood.

2. No significant difference exists between the Klason lignin contents of the two varieties.

3. The alcohol and water soluble extractive contents of refractory Douglas-fir heartwood are significantly greater than those for permeable Douglas-fir heartwood.

4. The average fiber length, lumen diameter, and cell wall thickness are significantly greater in permeable Douglas-fir heartwood.

5. Graham's (1964) sink-float test may be successfully utilized to differentiate green wood chips of pronounced permeable or refractory behavior.

6. With the cooking conditions used, refractory Douglas-fir heartwood undergoes less delignification than permeable Douglas-fir, which results in higher screened rejects, a lower screened yield, and a higher pulp Kappa number for refractory material.

7. Paper made from refractory Douglas-fir heartwood requires significantly less refining time to achieve a given level of Canadian Standard Freeness.

8. Paper made from refractory Douglas-fir heartwood has greater sheet density, tensile strength, zero-span tensile strength,

stretch, fold endurance, and initial freeness; and also requires less beating time to a given CSF. Paper made from permeable Douglas-fir heartwood has greater internal tear resistance and a higher sheet brightness. No significant difference exists between the Mullen strengths of paper made from the two varieties.

9. The handsheet properties are functions of fiber characteristic variations. Beating time and internal tearing resistance are directly correlated to fiber length, while sheet density and stretch are inversely correlated to fiber length. Tensile strength and Mullen strength appear to be inversely correlated to the Runkel ratio. No significant relationships were developed for zero-span tensile strength or fold endurance.

RECOMMENDATIONS

Although the goals of this study were achieved, several questions arose that should be answered for full understanding of the pulping characteristics of refractory and permeable Douglas-fir heartwood.

1. Further analysis of the pulping responses should be made using different cooking chemicals, chemical concentrations, cooking cycles, and woods of varying permeability.

2. A full analysis of the extractives of the two woods should be made to evaluate both the effect on permeability and the effect on the pulping characteristics.

3. Further evaluation of Graham's (1964) sink-float test should be performed to determine its effectiveness on samples of intermediate permeability.

4. A study should be performed to determine the causal factors influencing the beating responses of the pulps from the two varieties.

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DEFINITIONS AND ABBREVIATIONS

Definitions

1. Moisture Content: Used in this paper as the weight of water contained in wood, divided by the wet weight of that wood, times 100.
2. Percent Solids: An expression of solid wood content defined as the weight of oven dry wood divided by the wet weight of that wood, times 100.
3. Permeable Douglas-fir: Heartwood of Pacific Coastal Douglas-fir that has been proven to be pervious to liquid translocation. (Also: Specimen, material, or sample)
4. Permeable Cook: A kraft cook performed on samples of permeable Douglas-fir chips.
5. Permeable Pulp: Pulp produced by the kraft pulping process from permeable Douglas-fir.
6. Permeable Paper: Handsheets prepared from permeable pulp.
7. Refractory Douglas-fir: Heartwood of Rocky Mountain Douglas-fir that has been proven to be impervious to liquid translocation. (Also: Specimen, material, or sample)
8. Refractory Cook: A kraft cook performed on samples of refractory Douglas-fir.
9. Refractory Pulp: Pulp produced by the kraft pulping process from refractory Douglas-fir.
10. Refractory Paper: Handsheets prepared from refractory pulp.
11. Runkel Ratio: A calculated value defined as twice the radial cell wall thickness divided by the lumen diameter of the fibers.
12. Significant Variables: Independent variables entered into the multiple linear regression analysis prior to the minimum value of the standard error of Y.

Abbreviations

SpGr	Wood Specific Gravity
RC	Ring Count (growth rate)
FL	Fiber Length
TLD	Tangential Lumen Diameter
RLD	Radial Lumen Diameter
CWT	Cell Wall Thickness
RR	Runkel Ratio
EFL	Earlywood Fiber Length
ETLD	Earlywood Tangential Lumen Diameter
ERLD	Earlywood Radial Lumen Diameter
ECWT	Earlywood Cell Wall Thickness
ERR	Earlywood Runkel Ratio
LFL	Latewood Fiber Length
LTLD	Latewood Tangential Lumen Diameter
LRLD	Latewood Radial Lumen Diameter
LCWT	Latewood Cell Wall Thickness
LRR	Latewood Runkel Ratio

Appendix Table 1. General characteristics of wood specimens.
Description of columns:
N. Sample number
1. Collection site in Oregon
2. Average impregnation rating
3. Average growth rate (rings/inch)
4. Average wood specific gravity
5. Average percent wood solids

N	1	2	3	4	5
		Permeable samples			
1	Grants Pass	6	17	.4404	71.35
2	Grants Pass	6	15	.4404	75.68
3	Grants Pass	6	9	.4529	70.24
4	Grants Pass	5.5	12	.4365	68.96
5	Grants Pass	6	27	.4204	72.22
6	Grants Pass	6	10	.4635	43.38
7	Grants Pass	6	21	.4296	72.08
8	Grants Pass	6	10	.3949	67.32
9	Grants Pass	5.5	10	.4286	65.99
Average			14.6	.4341	67.47
Std. dev.			6.1	.0195	9.48
		Refractory samples			
10	Joseph	1	8	.4861	70.38
11	Elgin	1.5	21	.5016	71.84
12	Elgin	1.5	23	.4366	74.01
13	Joseph	1.5	24	.4091	65.59
14	Joseph	1.5	14	.4288	65.72
15	Joseph	1	16	.4284	63.01
16	Prairie City	1	15	.4583	64.70
17	Prairie City	1	10	.4984	71.79
18	Prairie City	1.5	21	.4652	61.47
19	Prairie City	1	26	.5018	72.27
Average			17.8	.4614	68.08
Std. dev.			6.1	.0109	4.53
		Student's 't' test data			
't'			1.153	2.086	0.183
Sig. level			0.280	0.055	0.875

Appendix Table 2. Fiber characteristics of refractory and permeable Douglas-fir wood specimens.
Description of columns:
N. Sample number
1. Average earlywood fiber length (EFL) (mm)
2. Average latewood fiber length (LFL) (mm)
3. Average earlywood tangential lumen diameter (ETLD) (μ)
4. Average latewood tangential lumen diameter (LTLD) (μ)
5. Average earlywood radial lumen diameter (ERLD) (μ)
6. Average latewood radial lumen diameter (LRLD) (μ)
7. Average earlywood cell wall thickness (ECWT) (μ)
8. Average latewood cell wall thickness (LCWT) (μ)
9. Average earlywood Runkel ratio (ERR)
10. Average latewood Runkel ratio (LRR)

N	1	2	3	4	5	6	7	8	9	10
					Permeable samples					
1	4.84	5.23	38.09	23.65	42.70	6.86	3.48	9.83	.1827	.8313
2	4.99	5.35	32.05	17.82	37.90	6.14	4.86	8.91	.3033	1.0000
3	4.66	5.50	40.76	19.25	49.15	4.20	4.20	11.57	.2061	1.2021
4	5.17	5.56	37.68	25.80	41.47	5.43	4.30	9.73	.2283	.7543
5	5.72	6.20	43.83	24.37	46.49	6.14	3.69	8.09	.1684	.6639
6	4.43	5.10	36.45	19.66	40.76	3.99	4.66	9.11	.2557	.9268
7	5.46	5.80	44.54	20.99	46.28	5.43	3.94	10.24	.1769	.9757
8	5.30	5.54	43.42	31.54	45.88	6.45	3.84	8.09	.1789	.5130
9	5.02	5.36	50.59	24.47	51.92	3.58	4.04	10.65	.1597	.8705
Average	5.03	5.52	40.82	23.06	44.73	5.36	4.11	9.58	.2067	.8597
Std. dev.	0.39	0.33	5.46	4.25	4.41	1.18	0.45	1.16	.047	.202
					Refractory samples					
10	3.32	3.44	32.26	19.15	24.88	6.04	3.69	6.35	.2288	.6632
11	3.65	4.07	28.98	18.82	33.08	6.55	3.94	7.58	.2719	.8055
12	3.46	3.65	33.59	19.15	40.35	5.94	3.38	6.87	.2013	.7164
13	3.78	4.20	34.41	23.65	32.97	3.69	3.74	6.04	.2174	.5108
14	3.44	3.80	28.98	18.74	25.80	6.45	3.07	6.04	.2119	.6446
15	3.54	3.77	33.28	21.91	40.76	5.63	2.97	6.76	.1785	.6171
16	3.62	3.83	38.09	21.50	35.74	5.12	3.02	6.55	.1586	.6093
17	3.29	3.50	33.79	18.02	38.91	5.32	3.28	7.37	.1941	.8180
18	3.36	3.49	37.27	23.35	33.89	5.53	2.87	6.45	.1540	.5525
19	3.39	3.64	33.08	16.28	35.53	5.43	2.92	6.24	.1765	.7543
Average	3.48	3.74	33.37	20.06	34.19	5.57	3.29	6.62	.1993	.6692
Std. dev.	0.16	0.25	2.96	2.42	5.45	0.81	0.38	0.54	.035	.103
					Student's 't' test data					
't'	10.37	13.39	3.76	1.94	.60	5.36	4.33	7.29	0.39	2.63
Sig. level	0.001	0.001	0.002	0.071	0.001	0.650	0.001	0.001	0.710	0.017

Appendix Table 3. Extractive content data for refractory and permeable Douglas-fir specimens through successive extractions.
Description of columns:
N. Sample number
1. % Hexane soluble extractives*
2. % Benzene soluble extractives*
3. % Ether soluble extractives*
4. % Alcohol soluble extractives*
5. % Water soluble extractives*
* All percentages on basis of original O.D. wood weight.

N	1	2	3	4	5
		Permeable samples			
2	0.873	0.693	0.170	3.271	10.965
4	0.327	0.416	0.214	2.266	11.752
5	0.456	0.298	0.146	3.347	10.619
7	0.800	0.560	0.136	2.796	11.683
8	1.565	0.486	0.271	3.011	12.013
9	1.613	0.742	0.161	1.848	10.395
Average	0.939	0.532	0.183	2.756	11.238
Std. dev.	0.544	0.168	0.050	0.591	0.668
		Refractory samples			
12	1.015	0.410	0.210	3.151	14.321
14	0.710	0.426	0.213	3.680	16.738
15	0.294	0.401	0.198	2.884	14.261
16	0.454	0.625	0.301	4.012	12.971
17	0.383	0.305	0.170	2.982	15.570
19	0.752	0.444	0.177	3.206	13.035
Average	0.601	0.435	0.212	3.319	14.483
Std. dev.	0.272	0.104	0.047	0.437	1.465
		Student's 't' test data			
't'	1.361	1.205	1.008	1.876	4.938
Sig. level	0.204	0.259	0.340	0.092	0.001

Appendix Table 4. Sink-float test data.
Description of columns:
N. Sample number
1. % of chips which sank after six minutes of vacuum
2. % of chips which sank after second vacuum application
3. % of chips which remained floating after second vacuum application

N	1	2	3
	Permeable samples		
3	92	8	0
	96	4	0
5	88	12	0
	96	4	0
8	100	0	0
	92	8	0
Average	94	6	0
	Refractory samples		
11	0	0	100
	0	8	92
14	0	4	96
	0	8	92
19	0	0	100
	0	4	96
Average	0	4	96

Appendix Table 5. Pulping data for refractory and permeable Douglas-fir.
Description of columns:
N. Sample number
1. Active alkali of white liquor (gpl as Na_2O)
2. Percent sulphidity of white liquor (%)
3. Active Na_2O of black liquor (gpl)
4. Total Na_2O of black liquor (gpl)
5. Unscreened yield (% of original chip weight)
6. Screened yield (% of original chip weight)
7. Screenings (% of original chip weight)
8. Kappa number

N	1	2	3	4	5	6	7	8
				Permeable samples				
1	31.98	21.98	13.37	19.47	46.05	45.07	0.98	23.0
	32.08	22.10	13.09	19.40	49.57	48.76	0.81	24.0
2	32.34	21.89	14.32	18.80	44.41	43.70	0.71	22.3
	31.91	21.99	16.47	22.34	43.80	42.90	0.90	21.7
3	31.98	21.95	16.37	22.85	44.57	43.88	0.69	21.6
	31.82	22.09	15.17	21.21	44.00	43.24	0.76	22.8
4	31.95	21.78	14.53	22.06	46.57	45.99	0.58	21.9
	32.05	21.46	14.32	20.29	44.94	44.30	0.64	23.6
5	31.92	21.80	14.59	20.56	46.17	45.82	0.35	22.0
	32.22	22.00	15.17	21.28	45.45	45.26	0.19	19.8
6	31.98	21.95	14.32	21.14	44.76	43.96	0.80	21.6
	31.98	21.76	14.66	21.38	44.02	42.99	0.77	22.4
7	32.15	22.05	13.98	20.39	44.05	43.63	0.42	22.0
	32.04	21.91	14.70	21.01	46.13	46.08	0.05	20.7
8	31.45	21.97	17.25	23.70	43.29	43.02	0.27	17.7
	32.11	21.86	16.85	23.39	42.24	42.06	0.18	18.0
9	32.08	21.88	15.00	21.31	45.40	45.02	0.38	19.4
	32.04	21.91	13.88	20.22	43.70	43.47	0.23	24.1
Average	32.03	21.91	14.89	21.16	44.89	44.45	0.54	21.59
Std. dev.	0.18	0.14	1.16	1.34	1.62	1.60	0.28	1.85

Appendix Table 5. (Continued)

N	1	2	3	4	5	6	7	8
				Refractory samples				
10	32.08	22.10	13.30	20.46	46.85	42.81	4.04	25.4
	32.02	22.12	13.13	19.37	47.18	45.88	1.30	24.9
11	32.08	22.10	13.20	19.37	48.25	43.96	4.29	29.0
	32.04	22.12	12.00	17.02	47.54	43.70	3.75	25.7
12	31.91	21.99	16.20	22.10	47.31	45.11	2.20	29.7
	32.15	21.84	16.44	22.88	43.91	42.26	1.65	29.7
13	31.95	21.97	15.65	21.52	39.90	38.93	0.97	26.3
	32.18	22.03	13.30	19.98	41.81	39.79	2.02	23.0
14	32.16	21.64	12.48	18.96	46.15	44.02	2.13	29.6
	32.02	21.73	15.14	20.97	44.50	43.06	1.44	25.9
15	32.09	21.55	14.08	21.31	41.87	40.80	1.07	22.6
	32.12	21.84	14.36	20.90	47.54	45.89	1.65	27.5
16	31.95	21.78	16.03	22.50	46.32	44.24	2.08	37.6
	32.12	22.07	16.37	25.85	46.62	45.02	1.60	28.0
17	31.98	21.95	15.41	21.65	42.47	40.81	1.66	32.4
	32.01	21.93	17.46	24.28	41.34	39.50	1.84	28.0
18	32.08	22.10	13.64	20.90	49.84	48.33	1.51	29.9
	32.05	22.12	13.64	21.14	48.24	46.53	1.71	27.3
19	32.05	22.12	14.66	20.97	43.75	42.08	1.67	18.0
	31.98	22.07	15.38	21.62	44.59	42.65	1.94	28.5
Average	32.05	21.96	14.59	21.19	45.30	43.27	2.07	27.45
Std. dev.	0.08	0.17	1.50	1.90	2.75	2.50	0.92	3.98
				Student's 't' test data				
't'	0.432	0.988	0.677	0.059	0.541	1.664	6.544	5.701
Sig. level	0.675	0.330	0.510	0.900	0.590	0.110	0.001	0.001

Appendix Table 6. Handsheet test values - beater curves.
Description of columns:
N. Sample number
1. Initial freeness (CSF)
2. Time to 600 CSF (min.)
3. Time to 400 CSF (min.)
4. Time to 200 CSF (min.)
5. Freeness at sheet density .7 gms/cc (CSF)
6. Time to sheet density .7 gms/cc (min.)

N	1	2	3	4	5	6
			Permeable samples			
1	710	21.0	31.0	41.0	300	36
2	745	23.5	34.5	43.3	385	35
3	735	19.5	31.0	41.0	420	30
4	725	22.5	34.5	44.0	580	23
5	750	27.0	39.0	47.5	420	36
6	750	26.9	38.3	45.5	650	21
7	753	25.0	36.5	45.0	340	39
8	726	26.5	38.5	46.8	590	21
9	750	21.8	35.0	48.0	540	26
Average	738	23.7	35.4	44.7	469	29.7
Std. dev.	15	2.8	3.0	2.6	123	7.1
			Refractory samples			
10	701	15.0	29.5	46.0	660	6
11	754	20.0	32.0	43.5	700	14
12	720	17.5	30.0	44.0	670	9
13	725	17.0	29.3	41.5	685	7
14	723	16.0	28.7	42.0	675	8
15	709	14.5	26.5	40.5	675	5
16	706	16.5	30.0	42.8	655	10
17	739	18.5	28.5	40.5	660	14
18	732	22.5	36.5	47.5	685	11
19	745	19.0	30.0	41.2	680	15
Average	725	17.8	30.1	43.0	674	9.9
Std. dev.	17	2.3	2.6	2.3	14	3.5
			Student's 't' test data			
't'	1.712	5.169	4.056	1.526	5.235	7.851
Sig. level	0.105	0.001	0.001	0.155	0.001	0.001

Appendix Table 7. Handsheet test values - sheet density.
Description of columns:
N. Sample number
1. Average initial value (gms/cc)
2. Average value at 600 CSF (gms/cc)
3. Average value at 400 CSF (gms/cc)
4. Average value at 200 CSF (gms/cc)

N	1	2	3	4
		Permeable samples		
1	.4659	.651	.686	.697
2	.4603	.659	.705	.725
3	.4839	.675	.712	.750
4	.4998	.691	.735	.766
5	.4654	.671	.711	.728
6	.4512	.726	.750	.723
7	.4800	.620	.662	.717
8	.4942	.635	.691	.721
9	.4781	.690	.725	.743
Average	.4781	.668	.709	.730
Std. dev.	.0141	.030	.024	.017
		Refractory samples		
10	.6461	.787	.840	.880
11	.5270	.740	.780	.822
12	.6054	.774	.817	.860
13	.6340	.785	.820	.869
14	.6153	.780	.820	.874
15	.6525	.795	.728	.776
16	.6117	.750	.788	.829
17	.5288	.721	.769	.811
18	.5715	.765	.810	.850
19	.5295	.720	.775	.799
Average	.5921	.762	.793	.937
Std. dev.	.0489	.026	.032	.035
		Student's 't' test data		
't'	6.511	6.780	5.849	8.015
Sig. level	0.001	0.001	0.001	0.001

Appendix Table 8. Handsheet test values - breaking length.
Description of columns:
N. Sample number
1. Average initial value (m)
2. Average value at 600 CSF (m)
3. Average value at 400 CSF (m)
4. Average value at 200 CSF (m)
5. Average value at sheet density .7 gms/cc (m)

N	1	2	3	4	5
		Permeable samples			
1	4892	9760	9950	9750	9770
2	4921	10260	10520	10820	10540
3	5416	10180	10470	10140	10500
4	6276	11650	11770	11800	11630
5	5020	11150	11230	10940	11160
6	5074	10670	11400	11850	10250
7	5991	11660	11840	11560	11650
8	5349	11450	11350	10400	11400
9	6104	10640	10740	10500	10650
Average	5449	10824	11030	10862	10839
Std. dev.	540	689	643	746	655
		Refractory samples			
10	9150	12790	12870	13600	10550
11	5791	10150	10620	11350	9570
12	8882	11610	12170	12450	10400
13	8891	12350	12120	12390	10550
14	8749	12790	12940	12440	10750
15	9542	12280	12580	12870	10500
16	9676	13500	13530	13110	12200
17	5543	9600	10180	10470	9150
18	8458	12160	12450	12350	10750
19	5837	10770	10840	11050	10780
Average	8052	11800	11930	12030	10520
Std. dev.	1563	1255	1111	969	802
		Student's 't' test data			
't'	4.516	2.065	2.301	3.362	0.942
Sig. level	0.001	0.005	0.037	0.006	0.360

Appendix Table 9. Handsheet test values - stretch.
Description of columns:
N. Sample number
1. Average initial value (%)
2. Average value at 600 CSF (%)
3. Average value at 400 CSF (%)
4. Average value at 200 CSF (%)
5. Average value at sheet density .7 gms/cc (%)

N	1	2	3	4	5
		Permeable samples			
1	1.30	2.66	2.75	2.68	2.71
2	1.40	3.05	3.10	3.03	3.07
3	1.60	2.81	2.90	3.02	2.90
4	1.48	2.78	2.77	2.83	2.80
5	1.40	2.90	2.87	2.58	2.80
6	1.40	2.63	2.78	2.78	2.55
7	1.32	2.60	2.72	2.72	2.70
8	1.60	2.85	2.74	2.70	2.85
9	1.62	2.77	2.77	2.90	2.78
Average	1.46	2.78	2.82	2.80	2.80
Std. dev.	0.12	0.14	0.11	0.15	0.14
		Refractory samples			
10	2.32	3.20	3.24	3.43	2.68
11	1.75	3.27	3.44	3.50	2.75
12	2.62	3.08	3.26	3.42	2.90
13	2.48	3.07	2.87	3.09	2.80
14	2.06	3.12	2.88	2.86	2.65
15	2.42	3.33	3.24	3.47	2.70
16	2.34	2.96	3.00	2.96	2.75
17	1.78	3.33	3.54	3.39	3.10
18	2.16	3.34	3.23	3.12	3.10
19	1.50	3.06	3.16	3.17	2.95
Average	2.14	3.17	3.19	3.24	2.84
Std. dev.	0.36	0.13	0.22	0.23	0.16
		Student's 't' test data			
't'	5.361	6.140	4.415	4.795	0.591
Sig. level	0.001	0.001	0.001	0.001	0.570

Appendix Table 10. Handsheet test values - Mullen factor.
Description of columns:
N. Sample number
1. Average initial value (m^2)
2. Average value at 600 CSF (m^2)
3. Average value at 400 CSF (m^2)
4. Average value at 200 CSF (m^2)
5. Average value at sheet density .7 gms/cc (m^2)

N	1	2	3	4	5
			Permeable samples		
1	22.66	59.5	56.1	55.0	56.0
2	24.58	71.5	75.0	74.0	74.0
3	25.35	57.0	57.8	62.0	58.0
4	31.52	73.0	74.0	69.1	72.0
5	27.41	79.5	79.0	75.0	79.0
6	25.05	70.0	72.3	73.5	64.0
7	24.24	69.0	73.5	75.0	73.5
8	28.97	70.5	73.8	73.4	71.0
9	30.22	65.5	69.0	69.7	66.0
Average	26.67	68.39	70.06	69.63	68.17
Std. dev.	3.02	6.88	7.88	6.87	7.71
			Refractory samples		
10	49.40	72.8	80.0	84.0	58.0
11	26.66	60.5	66.0	73.0	56.0
12	47.43	68.9	70.0	73.5	60.5
13	50.79	72.0	72.0	77.0	61.0
14	42.78	75.4	77.0	85.4	60.0
15	53.68	71.8	70.5	74.5	60.0
16	56.86	80.5	76.9	74.1	73.0
17	26.61	58.5	63.0	66.5	56.0
18	47.34	72.5	74.0	73.2	65.0
19	32.36	66.0	65.9	68.7	66.0
Average	43.39	69.89	71.53	74.99	61.55
Std. dev.	11.02	6.66	5.52	5.92	5.20
			Student's 't' test data		
't'	4.394	0.422	0.476	1.824	2.214
Sig. level	0.001	0.680	0.640	0.089	0.042

Appendix Table 11. Handsheet test values - tear factor.
Description of columns:
N. Sample number
1. Average initial value ($dm^2 \times 10^2$)
2. Average value at 600 CSF ($dm^2 \times 10^2$)
3. Average value at 400 CSF ($dm^2 \times 10^2$)
4. Average value at 200 CSF ($dm^2 \times 10^2$)
5. Average value at sheet density .7 gms/cc ($dm^2 \times 10^2$)

N	1	2	3	4	5
		Permeable samples			
1	3.185	2.44	1.98	1.76	1.90
2	3.791	2.25	1.85	1.65	1.90
3	2.764	2.07	1.72	1.45	1.85
4	3.616	2.14	1.68	1.34	2.10
5	4.229	2.54	2.03	1.67	2.18
6	4.136	2.00	1.61	1.34	2.39
7	3.526	2.34	2.00	1.73	1.93
8	3.279	2.08	1.73	1.46	2.10
9	3.537	2.13	1.63	1.51	2.16
Average	3.563	2.22	1.80	1.55	2.05
Std. dev.	0.46	0.18	0.17	0.16	0.18
		Refractory samples			
10	3.275	1.86	1.18	1.14	2.73
11	2.622	1.46	1.29	1.22	1.80
12	1.583	1.39	1.00	0.89	1.38
13	1.467	0.98	0.90	0.82	1.28
14	1.676	1.12	1.03	0.86	1.40
15	1.346	0.97	0.91	0.79	1.22
16	1.717	1.21	1.05	0.95	1.41
17	2.405	1.60	1.33	1.18	1.72
18	2.190	1.35	1.14	0.98	1.64
19	2.358	1.61	1.36	1.24	1.85
Average	2.064	1.36	1.12	1.06	1.01
Std. dev.	0.61	0.29	0.17	0.17	0.44
		Student's 't' test data			
't'	5.977	7.664	8.930	6.996	2.570
Sig. level	0.001	0.001	0.001	0.001	0.015

Appendix Table 12. Handsheet test values - fold endurance.
Description of columns:
N. Sample number
1. Average initial values (double folds)
2. Average value at 600 CSF (double folds)
3. Average value at 400 CSF (double folds)
4. Average value at 200 CSF (double folds)
5. Average value at sheet density .7 gms/cc (double folds)

N	1	2	3	4	5
		Permeable samples			
1	164	860	700	700	700
2	164	1150	1100	1100	1100
3	296	940	950	1030	940
4	481	1090	1070	1130	1100
5	293	1300	1410	1730	1300
6	262	1010	1050	1080	1000
7	498	1210	1150	1100	1140
8	360	1520	1510	1340	1520
9	451	1380	1370	1600	1360
Average	330	1162	1146	1201	1129
Std. dev.	127	214	252	312	243
		Refractory samples			
10	702	1490	1700	2270	1000
11	187	820	970	1270	770
12	666	1300	1690	1950	960
13	777	980	1320	1670	830
14	562	1080	1380	1700	810
15	628	1800	1540	1540	1020
16	1263	1320	1535	2020	1300
17	78	860	1180	1395	640
18	1404	1775	2015	2390	1860
19	421	990	1087	1240	950
Average	669	1242	1442	1744	1014
Std. dev.	416	357	318	403	346
		Student's 't' test data			
't'	2.737	0.437	2.011	2.625	0.827
Sig. level	0.015	0.675	0.067	0.017	0.410

Appendix Table 13. Handsheet test values - Elrepho brightness.
Description of columns:
N. Sample number
1. Average value at 0 minute beater interval
2. Average value at 15 minute beater interval
3. Average value at 30 minute beater interval
4. Average value at 45 minute beater interval

N	1	2	3	4
		Permeable samples		
1	24.32	20.44	20.64	19.42
2	25.00	20.62	20.74	20.50
3	24.26	19.12	19.02	17.68
4	24.76	20.04	19.18	19.36
5	23.52	19.12	18.82	19.14
6	23.28	21.12	19.68	19.76
7	24.68	20.66	20.88	20.96
8	30.20	23.40	24.16	24.28
9	27.66	22.66	22.32	22.06
Average	25.30	20.80	20.60	20.35
Std. dev.	2.23	1.45	1.74	1.92
		Refractory samples		
10	22.80	18.18	16.46	14.82
11	22.34	17.40	16.86	15.78
12	24.76	19.40	18.30	16.50
13	24.20	18.54	17.28	15.06
14	24.20	18.24	16.70	14.60
15	24.70	18.24	16.94	15.40
16	21.70	17.16	15.98	14.30
17	22.22	17.32	16.80	16.02
18	21.48	16.22	14.78	13.30
19	23.58	18.67	18.06	16.90
Average	23.20	17.94	16.82	15.27
Std. dev.	1.24	0.91	1.00	1.08
		Student's 't' test data		
't'	2.515	5.215	5.898	7.205
Sig. level	0.019	0.001	0.001	0.001

Appendix Table 14. Regression equation data.

Description of columns:

1. Variables entered in 600 CSF level equation
2. Coefficients of 600 CSF level variables
3. Variables entered in 400 CSF level equation
4. Coefficients of 400 CSF level variables
5. Variables entered in 200 CSF level equation
6. Coefficients of 200 CSF level variables

(+) or (-) following variable indicates relationship

* indicates coefficients significant at 0.05 level or greater from 't' test analysis

1	2	3	4	5	6
		Beating time Run 1			
SpGr (-)	0.445	SpGr (-)	0.530	EFL (+)	-1.955
EFL (+)	5.638*	EFL (+)	6.125*	ETLD (+)	1.155*
LCWT (+)	-0.725	LRR (+)	-63.09	ERLD (+)	-0.131
		Den (-)	256.1	LRLD (-)	1.335
				LCWT (+)	-0.941
				ERR (-)	7.219*
r^2 = .6976		r^2 = .6623		r^2 = .7628	
A = 17.42		A = 31.59		A = .1510	
		Beating time Run 2			
SpGr (-)	0.445	SpGr (-)	0.421	RC (+)	0.087
EFL (+)	5.638*	EFL (+)	3.682	EFL (+)	-3.241
LCWT (+)	-0.725	ETLD (+)	0.368	ETLD (+)	0.939
		ECWT (+)	2.787	ERLD (+)	-0.161
		LCWT (+)	-1.615	LRLD (-)	1.859*
				ECWT (+)	5.620*
				LCWT (+)	-0.855
r^2 = .6976		r^2 = .7108		r^2 = .8013	
A = 17.41		A = -12.79		A = 240.6	
		Sheet density Run 1			
EFL (-)	-0.013*	EFL (-)	-0.002*	LFL (-)	-.0037*
SpGr (+)	.0004	ERLD (-)	-.0004*	ERLD (-)	-.0003*
ECWT (-)	0.002	LRLD (-)	-.0014*		
LRR (-)	-0.100	LRR (-)	-.0556		
LFL (-)	0.006				
r^2 = .8747		r^2 = .8308		r^2 = .7692	
A = .1173		A = .1120		A = .1077	
		Sheet density Run 2			
EFL (-)	-.0077*	EFL (-)	-.0031*	LFL (-)	-.0040*
SpGr (+)	-.0007*	ERLD (-)	-.0004*	ERLD (-)	-.0004*
ECWT (-)	.0017	LRLD (-)	-.0014*	SpGr (+)	-.0004
		LTLD (-)	.0003	LRLD (+)	-.0011
r^2 = .8355		r^2 = .8259		r^2 = .8276	
A = .1290		A = .1066		A = .1371	

Appendix Table 14. (Continued)

1	2	3	4	5	6
		Tensile strength Run 1			
LRR (-)	-278.3	LRR (-)	571.9	Den (+)	4691.
Den (+)	9055.	Den (+)	6783.	LRR (-)	-1542.
SpGr (-)	-15.03*	SpGr (-)	-16.65*	ERLD (-)	-5.929
ERLD (-)	-1.365*	ERLD (-)	-8.603	LRLD (-)	-23.63
ETLD (-)	1.382*	ERR (-)	-983.5	r^2 = .5846	
r^2 = .7333		r^2 = .6219		A = 1227.	
A = 1204		A = 1880			
		Tensile strength Run 2			
LCWT (-)	-9.941*	LCWT (-)	-5.485	ERLD (-)	-9.095*
SpGr (-)	-19.61*	SpGr (-)	-17.12*	LRLD (-)	-25.10
ERLD (-)	-14.07*	ERLD (-)	-11.44*	SpGr (+)	-12.65
ETLD (-)	1.448*	ETLD (-)	11.33	LFL (-)	-41.07
EFL (-)	-4.757	EFL (-)	-57.25	r^2 = .5893	
r^2 = .7036		r^2 = .6255		A = 1267	
A = 2311.		A = 2240.			
		Stretch Run 1			
LFL (-)	-0.198*	LFL (-)	-0.219*	EFL (-)	0.319
LRLD (+)	0.045	SpGr (+)	0.033*	ECWT (-)	-0.149
r^2 = .6669		LRLD (+)	0.066*	ERLD (-)	0.041*
A = 3.656		ERLD (-)	0.030*	LRLD (-)	0.011
		ERR (+)	2.629*	LFL (-)	-0.547
		LCWT (-)	-0.062	Den (+)	23.97
		r^2 = .8597		ERR (+)	5.875
		A = .9618		SpGr (+)	0.017
				r^2 = .8769	
				A = .7508	
		Stretch Run 2			
LFL (-)	-0.199*	LFL (-)	-0.278*	EFL (-)	-0.104*
LRLD (+)	0.045	SpGr (+)	0.034*	ECWT (-)	0.370*
r^2 = .6669		LRLD (+)	0.096*	ERLD (-)	0.026*
A = 3.656		ERLD (-)	0.027*	LRLD (+)	0.078
		ECWT (-)	0.231*	LFL (-)	-0.438
		LCWT (-)	-0.069	RC (+)	0.007
		r^2 = .8483		r^2 = .8355	
		A = .8813		A = 2.569	

Appendix Table 14. (Continued)

1	2	3	4	5	6
		Mullen Run 1			
LRR (-)	-526.1*	LRR (-)	-475.6*	ERLD (-)	-0.177*
EFL (+)	14.19*	ECWT (+)	9.622*	SpGr (-)	-1.236*
LTLD (+)	-2.247*	LTLD (+)	-2.038*	ETLD (-)	-0.700
Den (+)	872.8	ETLD (-)	1.301*	LTLD (-)	-1.077
RC (+)	-0.340	LRLD (+)	1.917	r^2 = 6068	
ERR (-)	-28.53	ERLD (-)	-0.510	A = 158.6	
		SpGr (-)	-0.656		
r^2 = .7723		r^2 = .7537			
A = 46.02		A = 106.2			
		Mullen Run 2			
LCWT (-)	-4.271*	LCWT (-)	-4.268*	ERLD (-)	-0.729*
EFL (+)	8.844*	EFL (+)	24.11*	SpGr (-)	-1.009*
LRLD (+)	-2.010	RC (+)	-0.319	ETLD (-)	0.938*
ERLD (-)	-0.312	ERLD (-)	-0.455	LTLD (-)	-0.811
r^2 = .6304		LRLD (+)	-2.666	LCWT (-)	-3.203*
A = 89.20		LFL (-)	-12.41	ECWT (-)	5.965*
		LTLD (+)	-0.462	r^2 = .7477	
		r^2 = .7302		A = 132.8	
		A = 107.4			
		Tear Run 1			
Den (-)	-1.122	EFL (+)	0.458*	Den (-)	-17.49
ECWT (+)	1.728	SpGr (-)	0.043*	EFL (+)	0.225*
EFL (+)	0.261	LRLD (+)	0.068*	SpGr (-)	0.030*
SpGr (-)	0.049*	LRR (+)	4.119*	LRLD (+)	0.074*
LRLD (+)	0.265*	ERR (+)	-0.938	LRR (+)	4.776*
ERR (+)	-23.43*	r^2 = .9564		ETLD (+)	0.022*
LTLD (+)	-0.032	A = 2.924		ERLD (+)	-0.015
ETLD (+)	-0.055			r^2 = .9484	
ERLD (+)	-0.012			A = -.6604	
r^2 = .9483					
A = 1.329					
		Tear Run 2			
EFL (+)	0.649*	EFL (+)	0.449*	EFL (+)	0.360*
SpGr (-)	0.077*	SpGr (-)	0.049*	SpGr (-)	0.047*
LRLD (+)	0.065	LRLD (+)	0.060*	LRLD (+)	0.054
RC (-)	-0.008	LCWT (+)	0.034	LCWT (+)	0.033
r^2 = .8912		r^2 = .9442		r^2 = .9096	
A = 4.644		A = 3.262		A = -2.946	

Appendix Table 14. (Continued)

1	2	3	4	5	6
		Fold Run 1			
LRR (-)	-164.4	EFL (-)	6.125*	Den (+)	53893.*
LTLD (+)	51.11*	Den (+)	256.1	ETLD (-)	92.04*
Den (+)	45663.*	SpGr (+)	0.530	LCWT (-)	-170.6*
LRLD (+)	155.2	LRR (-)	-63.09	LRLD (-)	159.2
				ECWT (-)	336.4
r^2 = .5325		r^2 = .6623		EFL (-)	-232.5
A = -4683.		A = 31.60		LTLD (+)	-12.32
				r^2 = .8483	
				A = -5637.	
		Fold Run 2			
LTLD (+)	6.993*	LCWT (-)	-136.3	LCWT (-)	-161.7
LFL (-)	-792.4*	ETLD (+)	45.35*	ETLD (-)	80.98*
EFL (-)	671.1	RC (+)	-3.294	ERLD (-)	-28.49
ETLD (+)	18.96	LFL (-)	-263.2	LFL (-)	-286.9
SpGr (-)	-32.88	SpGr (-)	-31.29	ECWT (-)	-161.7
		ECWT (-)	164.8		
r^2 = .5106				r^2 = .7327	
A = 2617.		r^2 = .6947		A = 1496.	
		A = 2777.			