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CHEMICAL PULP BEATING  
RELATED TO FIBER STRUCTURE

by

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BIBLIOTECA  
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## INTRODUCTION

Paper is a commodity used in a wide range of products, which, depending on their end use, have to comply with a variety of specific requirements. The papermaker can influence the characteristics of a paper in a number of ways, from the choice of the fibrous raw material to the different treatments during stock preparation, in the papermachine operation, and in the finishing and converting process. Of these the choice of raw material seems to be the most important and economical way to achieve the desired characteristics.

Apart from the variables of the pulping process the properties of a particular pulp are mostly dependent on the wood species from which it is produced, and from the quality of the wood as influenced by environmental factors during its growth. Fiber morphology and chemical properties are both important from a pulp utilization point of view, although in some cases morphological aspects are considered of greater importance. After a judicious choice of the pulp has been made, this including the pulping process utilized for its manufacture, one of the most important ways to improve its properties is through the beating process. The assessment of the problem of what are the best beating conditions to improve the paper properties in a unified approach is very complicated.

The beating behavior of pulps is generally described as being the ease with which the fibers undergo morphological and chemical modifications that result in improved paper characteristics. Consequently this subject involves the complex field of pulp quality-paper quality relationships. A number of studies have been dealing with these questions since the late 1950's, but there are still many problems waiting for solution.

The present study is an attempt to contribute with additional information to the same topic, most specifically through an analysis of the mechanisms involved in beating, how they are affected by fiber properties, and ultimately how these interactions influence the quality of paper. To avoid the added complexity of the numerous possible ways of affecting the pulp quality during pulping and bleaching, the work was restricted to commercial bleached kraft pulps (southern pine, silver birch, Eucalyptus grandis and Eucalyptus rostrata) primarily intended to be used in the production of fine paper grades. These pulps from different species were selected mostly because of their vastly different characteristics, therefore providing a broader range of variables for this study.

With the increased use of hardwoods in the papermaking field it was decided to concentrate on three pulps, one from the traditionally used birch, and two made from the fast-

grown eucalypts, which are being introduced in the market in increasing quantities. The southern pine pulp was chosen as representative of a commercially important long-fibered pulp, in an attempt to quantify the beating mechanism in a more generalized approach.

The results of the present research are divided into the following sections:

1) Characterization of the raw material. The four pulps were analyzed from the point of view of morphology, including microscopic and ultramicroscopic properties (performed on wood and pulp fibers), and their chemical composition determined.

2) Evaluation of the response of these pulps to beating in the PFI mill. Two different pressures were applied to the pulps: the standard beating load, and a higher one with the purpose of achieving conditions closer to the high pressures encountered in industrial refining. This methodology allowed the observation of differences in the response of the four pulps when a higher load was applied between the beater bars and the house as compared to the standard load. Even under these conditions the PFI mill can be considered an idealized refiner, because the cutting action is minimized, and the beating is performed in a batchwise fashion at high consistency. The main purpose of the study, however, was to provide some qualitative data relating the different beating responses of the four pulps to their fiber structure.

The various changes occurring during beating were assessed by measurements in the beater, such as energy consumption and clearance between roll and house, and by quantitative determinations of these effects on the wet fibers and fines, utilizing water permeability techniques. Based on these results some aspects of the papermaking were analyzed, and these included the dynamic drainage properties of the pulps, wet web strength, and the routine evaluation of the properties of dry handsheets. The analysis of the relationships between these characteristics and fiber structure constitute the bulk of the present dissertation.

## LITERATURE REVIEW

### The Importance of Fiber Morphology in Papermaking

It has been recognized for a long time by the paper industry that the characteristics of the fibrous raw material play a fundamental role not only in the economics of the pulping process but also on final paper properties. Information has appeared since the 1960's on the evaluation of relationships of numerous fiber dimensions, fiber dimension ratios, fiber strength, wood density, and its chemical composition to certain physical properties of paper (46,53,98,101,120,154,193,214,222,230).

Despite the number of studies in the literature trying to relate wood and fiber morphology to paper properties the industry still does not seem to know exactly which characteristics to demand from the trees. One of the reasons for this is the wide range of paper grades, which have different quality demands from consumers. Another is the complex number of variables involved in the assessment of each paper property (there is still a need for an unifying theory of sheet formation and sheet strength). A third reason is the limited knowledge from silviculture and genetic studies about how different practices influence wood and fiber characteristics.

Many correlations between wood fiber and paper properties have been published for different species (17,54, 120,154,218,230), but concerning generalizations for species of industrial importance the results are sometimes contradictory, mostly because of partial interpretation of statistical data (53), and due to the natural variation among trees of the same species.

This difficulty in generalization is also due, in great part, to the intensified interest in the use of hardwoods for papermaking. The traditionally used softwoods have much simpler anatomies than the hardwoods, and this allowed early recognition of the close relationship between tracheid dimensions and wood density. Conifers are very uniform in structure (the main cells being tracheids and parenchyma), with the earlywood generally composed of thinwalled cells and wood density closely related to the amount of latewood present. Due to this relatively simple situation it was recognized in the early models of paper that woods of low specific gravity produced papers of high density and strength, although low in opacity. The increased use of hardwoods has directed the attention towards more basic studies of their characteristics, to see whether the results obtained for softwoods could be applied in this case.

Research in this area has revealed that many of the relationships between fiber and paper properties found for softwoods can be applied to hardwoods (46,85,96,98,100,120, 236), although the more complex anatomy of the deciduous

species should be considered when paper qualities are compared after the same type of processing (5). In this context the presence of a number of additional anatomical elements, and particularly of vessels should be given increased consideration in the study of hardwoods (101).

Among the hardwoods in commercial use the fast-grown eucalypts constitute important sources of fiber being used in increasing quantities. The study of their papermaking properties when compared to other hardwoods more traditionally used (e.g. birch, beech) has received some attention since their introduction in the European market (3,6,163,190), and even more emphasis has been directed lately to research on the properties of the pulps from these species (52,54,74,116,162). However there still seems to be a lack of a generalized approach for the study of these fibers and those of conifers and other hardwoods at a more basic level, e.g. involving fiber structure and the properties of paper.

Nevertheless, it can be said that the results obtained so far for softwoods and hardwoods (including the eucalypts) show some general trends in the desired properties of fibers for optimum papermaking. It is well accepted that fiber strength, fiber length, fiber length to diameter ratio (slenderness), fiber coarseness, and fiber flexibility (related to lateral conformability in the sheet) are the most important characteristics to consider. Of these, fiber flexibility and conformability has been recognized as a key

factor in characterizing the quality of a pulp (5,53,98, 230). In this context it is also important to notice that fiber length, fiber strength and fiber flexibility are not totally independent of each other.

#### Fiber Flexibility and Collapsability

It became evident in earlier studies that thinwalled fibers collapse to flattened ribbons during papermaking with highly effective fiber to fiber contact areas, giving rise to compact and dense sheets of high tensile and burst strength and low opacity. On the other hand, the thick-walled fibers have more resistance to collapse as the sheet is formed, yielding papers with high bulk, high stiffness and opacity, but of low strength, due to the reduced area of contact between such fibers.

With the recognized importance of interfiber bonding for the consolidation and strength of paper, as evident at the Oxford Symposium in 1961, the concept of fiber flexibility and conformability gained increased acceptance as one of the controlling factors of paper strength. However, the lack of a method for a quantitative measurement of fiber flexibility has led the majority of investigators to the estimation of this property through the ease with which the lumen collapses (i.e. fiber collapsability). Many parameters have been proposed to control the collapsability of a fiber, all based on the geometry of its cross section, including the well-known

Runkel ratio, the ratio of lumen diameter to fiber width (83), and the more recently suggested shape factor (142), among others.

Indeed, even the force required to collapse of the lumen has been measured (90,91,142), yielding stress-strain curves for a fiber under lateral compression, and confirming the influence of fiber cross-sectional dimensions on its collapsability. However, these considerations alone could not explain why beaten and unbeaten fibers with the same geometry would collapse differently, the same holding true for comparisons between kraft and sulfite fibers. These differences have been usually explained in terms of the plasticity of the fiber wall as indicated by its shear modulus in the transverse plane, which would also be an important factor controlling the resistance to collapse (91,172).

Nevertheless, it has never been proven beyond any doubt that collapse of the lumen is a necessary condition for the conformability of a fiber to others in the sheet. (The lumen collapse only indicates that the cell wall is able to yield to the forces inducing sheet consolidation, and does not indicate whether the whole fiber is able to bend and conform to the others in the sheet.) As pointed out by Scallan and Borch (195a) lumen collapse for the purpose of converting cylindrical fibers into flat ribbons capable of more inter-fiber contact does not need to be complete. The collapse of the lumen has more to do with a considerable loss of light scattering ability than with conferring strength to the paper

sheet. However, since the geometrical models worked well in explaining the differences between paper properties obtained from earlywood and latewood fibers, or from high density and low density hardwoods it has been generally implied that fiber collapsability is a good estimate of its conformability.

Although differences such as these can be well explained by geometrical considerations of the cross-sections of the fibers, an actual measurement of the fiber flexibility should provide better results when comparing fibers not so different in their relationships between cross-sectional dimensions, (e.g. Runkel ratio, shape factor) such as the many medium-density hardwood species available for pulping.

The measurement of flexibility of a fiber is not an easy task, and has not been tackled by many investigators. Qualitative estimates were provided by the work of Forgacs, Robertson and Mason (68), while direct measurements of fiber stiffness were reported by Samuelsson (191), through the deflection of cantilevered fibers under the frictional force of water flowing past them. This method however is extremely tedious, and would tend to reduce the applicability of the measurement. Other techniques have been reported more recently, but they either involve very sophisticated methodology (216) or still provide only indirect measurements of fiber stiffness (160,199).

As proposed by Leopold and Iyer (138) a direct measurement of the bending stiffness of a fiber could be derived from knowledge of its axial Young's modulus and cross-sectional dimensions. However this approach still involves the measurement of the load-elongation properties of single fibers, which could be extremely difficult to perform with the thin and short hardwood fibers. From our present knowledge of the mechanical behavior of wood fibers under axial stress as strongly dependent of the thickness and microfibrillar orientation of the various cell wall layers (149,150), it could be possible to calculate the axial Young's modulus of pulp fibers using ultrastructural data. These and the calculation of the moment of inertia of the fiber cross-section based on its dimensions would allow a direct estimate of fiber stiffness.

To our knowledge this approach has never been tried, but it is possible that, using only properties based on the fiber structure and cross-sectional geometry, a good indication about its flexibility and conformability could be obtained. This estimate might lead to a more basic understanding of the behavior of the fibers in the unbeaten state and could help to evaluate their response to the beating process.

#### The Beating Process

Beating (or refining) can be defined as the mechanical

treatment through which pulp fibers undergo structural modifications that result in the general improvement of paper properties. It is therefore not surprising that this topic is one of the most studied in papermaking, and involves a large amount of technical literature published every year, which is impossible to survey thoroughly. Fortunately some good reviews of the advances in beating have been made (9, 56, 66, 71, 97), and they provide the starting point for a more concise analysis of the published results in this area.

Existing beating theories are divided into beating action and beating effect. Much of the research done since the early stages of papermaking has been concentrating on the evaluation of the beating effects, through the response of fibers to the mechanical treatment, and pulp evaluation. With the increased use of refiners and the outlining of the Specific Edge Load theory (238) a considerable amount of research has been recently done towards the characterization of the refiner action, with little further contribution directed to the understanding of the fundamental changes imparted to the fibers during this process. In this review the attention will be focused first on the recent findings on beating action. Following this the effects of beating on fibers will be reviewed.

### Beating Action

Refining is a stochastic mechanical treatment that

produces a number of results on the fibers. Although these results have been identified and most of the investigators agree on what the effects on fibers are, there seems to be no generally accepted theory that would indicate to the papermaker how the various controlling variables should be manipulated to achieve a desired output in the form of pulp properties.

The Severity and Number of Impacts.- The concept of the Specific Edge Load has provided a significant contribution in the search for an index representing the intensity of the beating performance describing the beating absorption of a pulp, at least with respect to its nature. In this theory an empirical measure of the relative severity and the relative number of impacts that a fiber receives in the refiner is given (238). This concept has been developed into other beating theories, by Brecht (23), Danforth (49) and Leider and Nissan (134), all involving the character of the beating action as described by the type (or intensity) of refining and the extent of action, which is related to the amount of refining (or work) absorbed by the fibers.

The intensity of treatment is an inverse function of the area of refining, as given by the number and width of bars, and a direct function of the unit pressure exerted between the surfaces. The degree or amount of treatment is directly related to the actual bar-to-bar surface area and

the relative speed of the refining surfaces. According to this concept the heterogeneity of the treatment would be reduced to a minimum by impacting the fibers at a relatively low level of severity a relatively large number of times, and would result in optimum strength development. On the other hand a harsher treatment would be provided by increasing the severity of impacts and minimizing their frequency, resulting in increased fiber cutting at the same energy consumption.

Indeed this approach has helped significantly in the operation and design of refiners, indicating the type of tackle (no. of bars and grooves), refiner peripheral speed, and load to be used, depending on the predominant action desired, whether "hydration" (promoting strength development) or cutting. The industry seems to show increased acceptance of this theory (127,128,164), despite the fact that it does not include other parameters of importance during refining, such as the pulp consistency, the cutting angle and the material of construction used in the refiner plates.

More Recent Theories.- As pointed out by the most recent reviewers (9,56) the basic problem of how the energy is transmitted to the fibers in the refining gap resulting in changes in their structure has not yet been answered. It is not known, for instance, why softwoods respond better to a

higher specific edge load and hardwoods display faster strength development when beaten under a lower specific load (139).

In this context two major contributions have been recently made towards the basic understanding of the mechanisms of refining. The first was proposed by Nissan (167), and is one of the few which relates a fiber property dependent on its structure to the absorption of beating energy for fiber modification. It is postulated in this theory that the energy applied during beating would be utilized for breakage of internal bonds, by straining the fiber in tension to a point very close to its elastic limit. This process would result in internal helicoidal breaks at the  $S_1$ - $S_2$  interface and within the  $S_2$  layer, according to Mark's calculations (149).

Nissan then proposed that the energy absorption by each fiber could be expressed as the product of the fiber's axial Young's modulus by the square power of its elongation during straining. Unfortunately he did not extend the theory to a more basic level, involving the influence of fiber structure on its Young's modulus, which could probably explain better the responses of different fibers. Instead, it was assumed that all fibers considered would have the same axial modulus of elasticity, and their different responses to refining would be given by their probability of being impacted in a single pass through a refiner.

A more recent theory was proposed by Steenberg (208) and is based on the treatment of flocs, rather than single fibers between the refiner bars. As indicated in the literature (12,56,70,84,174,208) and during the 1980 International Refining Symposium held in Appleton there seems to be an increased acceptance of the idea that beating involves primarily the treatment of fiber networks. Experimental evidence for this has been provided by observations during low consistency refining (12,70) and in the PFI mill (158).

This concept brings up immediately the subject of fiber interaction, as influenced by their properties (e.g. fiber length, fiber length-to-width ratio, fiber flexibility). It also shows the need for a basic understanding of the mechanical behavior of flocs under the shear and compression forces imposed by the refiner bars. In his proposal Steenberg did not try to tackle these problems, which still lack a considerable amount of study, but instead he was more concerned with the similarities between beating and grinding, and how the presence of water makes beating even more singular.

The main thrust in this theory is that refining involves irreversible and catastrophic changes in the particles being impacted, and each change is an energy consuming process. This unit process was termed a "Kappa" process, and involves a critical path for the formation of a stress concentration chain until a yield point in the material is

reached and a structural breakdown occurs. A particle which once has taken part in a Kappa process may or may not again undergo further Kappa processes. For another Kappa process to occur the particles must be rearranged. It was postulated that optimization of the milling operation should involve the search for the mode of operation which for each unit of applied work gives the maximum number of Kappa processes.

When considering the presence of water the concept of Kappa process provides an outline for a beating theory. According to Steenberg, as the flocs are compressed in the beating gap there is a threshold concentration below which the formation of a stress concentration chain in the bed of fibers is not possible because the fiber slurry will "ooze", and escape the stress concentration chain. Above the threshold consistency the flocs will consolidate under the action of the refining force and the Kappa process can occur and cause a major primary effect of refining. To increase the probability of new kappa processes the particles must be rearranged into new configurations, which would happen in the grooves of a refiner. It was then proposed that the yet neglected work to create new configurations is the main component in grinding energy consumption.

Although very difficult to comprehend thoroughly, Steenberg's theory points out to the study of the behavior of flocs as fundamental for the advancement of the present

knowledge about beating. These studies, when connected with basic fiber properties, namely fiber structure could be the starting point for more fundamental understanding of beating at both macroscopic and microscopic levels. There appears to be a considerable potential for energy savings to be achieved from a better understanding of what is at present a somewhat empirical operation. In addition, a greater fundamental knowledge of the factors involved in the effects of beating could result in a reduction of the deleterious effects and be applicable to the refining of new types of pulps.

#### Beating Effects

At the time when the operation of the beater was essentially an art the beaterman used to test a pulp by squeezing a sample in his hand. By judging how easily the water drained he sensed the "wetness" of the pulp. Usually pulps which had a high ability to retain water produced strong papers, and this gave rise to the old papermaker's "hydration" term, used to describe how a pulp should be beaten to improve the strength properties of the paper. Yet, although it has been known that hydration is the most desirable beating effect, its significance in terms of how the fibers had been modified were not well understood.

The freeness test was introduced with the intention of

providing the same measurement as the squeezing test, although in a quantitative way. However, as the new method was primarily intended to indicate the drainability of the pulps, its correlation with "wettness" or "hydration" was soon found not to be always applicable. As pulps are beaten and get "wetter" they are usually more difficult to drain, but the use of freeness as a single yardstick to quantify beating cannot explain differences due to wood species, pulping and bleaching processes and beating variables.

As a consequence the bulk of the investigations concerning the beating process have concentrated in evaluating the effects of this treatment on fibers. Although the study of the responses of fibers to beating is as old as papermaking itself it can be said that the symposium on the "Fundamentals of Papermaking Fibers", held in Cambridge in 1957 marked the beginning of intense research in this area, with the works of Giertz (75), Gallay (71), Robertson (180), among others. Almost at the same time Emerton published his book (62), and since then an enormous amount of information on the beating of pulps from different species, prepared using different pulping processes and beaten in various equipments have been released.

As indicated in different reviews of the study of beating and refining (9, 66, 97) there is very little disagreement among the investigators about what are the effects of

the mechanical treatment on fibers. They respond to the complex set of bending, shearing, bruising, twisting and tensioning forces in the beater with a number of structural changes that can be summarized as follows:

- delamination of internal cell wall layers, or internal fibrillation;
- sequential breakage of external layers of the fiber and their separation from the cell wall - external fibrillation and fines formation;
- creation of dislocations in the cell wall structure, leading to longitudinal compression;
- dissolution of the cell wall constituents, mostly hemicelluloses, with consequent formation of a colloidal suspension in interaction with the separated external layers of the fibers and debris - also called molecular fibrillation;
- fiber cutting and splitting.

Due to the inherent stochastic characteristic of the treatment it is highly probable that many of the beating effects occur almost simultaneously. As a matter of fact, since all the effects can be classified as deriving from creation of new surfaces (56), of which creation of new particles and generation of structural damage are particular cases, it becomes difficult to envisage their occurrence in a completely independent fashion. However the extent of

these beating effects may differ considerably, depending on the numerous possible combinations of pulp and treatment characteristics.

Consequently there is still some controversy among the investigators on what are the important beating effects. Perhaps this is due to the fact that most conclusions have come from deductive reasoning, since the quantitative measurements of the beating effects are difficult to make. With the well established knowledge of the importance of inter-fiber bonds on the strength of paper the argument has been concentrated on which of the beating effects makes the fibers more amenable to the consolidation forces during the sheet forming process.

Since the external fibrillation and formation of fines have always been easily noticed with the use of the microscope it was taken for granted for a long time that the "hydration" of the pulp was due to the absorption of water at the surface of the detached fibrils and fines, and that the increased external area produced by beating was the main factor inducing sheet consolidation and interfiber bonding (8,112), as a result of the Campbell's effect. In this context, the fines were believed to play an important role in the development of strength of paper, due to their large specific surface area. Probably their presence in all beaten pulps and the study of their properties (105,113,192,215)

has kept alive until today (33) the idea that external fibrillation is the main beating effect.

However, in spite of the obvious influence of external fibrillation and the fines formed during beating on the sheet consolidation process, these effects could not explain the rapid rise in strength at the early beating stages, when little or no fibrillation is apparent. Emerton's proposal (62) that the important factor during beating was the water uptake within the cell wall started to gain recognition as being the main beating effect when methods developed to indicate swelling of the fiber (114,183,184,195,213,220) showed good correlations with the development of paper strength. Further experimental evidence that this process was accompanied by loosening of the internal fiber structure during beating (155,175) provided increased support to the concept that it is the increased fiber flexibility and conformability (achieved through internal cell wall delamination) which is important for the formation of interfiber bonds and consequently for paper strength (157).

More recent studies have confirmed the patterns of ultrastructural organization in the cell walls of different fibers (120a,187,194,196), which allows delamination to start during pulping and proceed, although more dramatically, at the onset of any mechanical treatment. It is thus well established today that the cell wall can delaminate into

coaxial lamellae during beating, and that this effect can be followed by the swelling degree of the fibers. However, these changes have not been connected in a generalized approach to the responses of different fibers to beating. In other words, although some more recent studies have involved the evaluation of the various responses of fibers to the beating action (121,122,125,226) and related them to fiber outer structure, the conclusions achieved have always derived from qualitative evaluations, restricted to the characteristics of one or two wood species. Perhaps the only reported work in which the ultrastructural properties of the fibers were involved in the evaluation of their responses to beating is that of Alexander, Marton and McGovern (2), but a quantitative study of the responses of fibers to beating based on their ultrastructure is still lacking.

In this context the simultaneous measurement of the external fibrillation, internal fibrillation and formation of fines on a number of species (and their possible relationships with basic fiber properties) could yield very relevant information on the general mechanisms of the beating process. The estimation of internal and external fibrillation using the measurements of specific surface area and specific volume of wet pulps using the technique developed by Robertson and Mason (184) has been used in the past, although with limited success due to experimental

difficulties. With the development of instruments able to perform these measurements with higher accuracy (41) it became possible to use it for control and research purposes. In the following a critical analysis of the method and its shortcomings will be done.

Measurement of Specific Surface Area and Specific Volume of Pulps.- The measurement of specific surface of discrete particles is of interest in a number of scientific and industrial applications, the manufacture of pulp and paper being one of them. Recognized as one of the major factors controlling the drainage of papermaking pulps a number of techniques have been applied for the measurement of the surface area of wood pulps. Among them the fluid flow methods provide the best estimates of specific surface area, as pointed out by Mason (153), Campbell (29) and Han (87) in their reviews.

The flow of water through pulp beds, in particular, has been given special attention, due to its applicability to pulps in their natural state before papermaking. An additional advantage of this method is the simultaneous estimation of the specific volume of pulp fibers, which is an indication of their swelling ability.

The literature covering different aspects of filtration and permeation is vast (29), most of it concerned with

different techniques to measure drainage resistance. The permeability measurement was applied initially by Robertson and Mason (184) and has been used since then, with different refinements of the technique (41,64,67,225,227,237). The method is based on the ease with which water flows through a mat of pulp fibers. The theory underlying it is the same derived for fluid flow through porous media, with special consideration given to the geometry of pulp fibers and to the fact that, unlike most other materials, they swell in water. The following theoretical derivation of the Carman-Kozeny equation is included for a better understanding of the limitations in the applicability of the permeability method to pulp fibers.

Theory - The flow of a Newtonian liquid through a porous bed can be related to the pressure drop in the direction of flow across an uniform bed by Darcy's law:

$$Q = \frac{-K \cdot \Delta p}{\mu \cdot L}$$

where Q = volumetric flow rate per unit cross sectional area of the bed;

K = permeability coefficient, a function of bed porosity ( $\epsilon$ );

$\mu$  = viscosity of the fluid;

L = bed thickness in the direction of fluid flow;

$\Delta p$  = pressure drop across the bed.

Carman developed the best known equation for the relationship between the permeability, the porosity and the internal surface area of a bed, using Kozeny's model for the flow of a fluid through a porous solid (39). The simplest model is for a network of straight parallel channels (or capillaries) of circular cross-section and uniform diameter, arranged along the axis of the bed (this leads to Poiseuille's law). The Kozeny model is more sophisticated, in that the capillaries are not necessarily circular in cross section, although they are still supposed to be identical in length and cross sectional area. They can also be oriented at various angles to the axis of the bed and are interconnected with one another. The generalized Carman-Kozeny equation for low to medium porosities then takes the form:

$$K = \frac{1}{k S_o^2} \times \frac{\epsilon^3}{(1-\epsilon)^2} \quad \text{where } K = \text{permeability coefficient, defined earlier;}$$

$k$  = Kozeny constant, or shape factor;

$S_o$  = surface area per unit volume of the solid material;

$\epsilon$  = bed porosity.

Unlike many materials pulp fibers swell in water, and a considerable amount of liquid is imbibed in the fiber and contained in the fiber lumen. Therefore the void fraction  $\epsilon$  cannot be obtained from mass concentration. In the case of

swollen pulp fibers the following additional relationships are used:

$$\epsilon = 1 - v \cdot C, \text{ and } v \cdot S_0 = S$$

where  $v$  = effective volume of the swollen fibers (specific volume);

$S$  = external surface area per gram of fibrous material (specific surface area);

$C$  = concentration (dry fiber weight per unit volume of the bed).

The Carman-Kozeny equation then takes the form,

$$(KC^2)^{1/3} = \left(\frac{1}{k S^2}\right)^{1/3} (1 - v \cdot C)$$

from which  $v$  and  $S$  can be calculated when permeability measurements are made at several different concentrations of pad material. By plotting  $(KC^2)^{1/3}$  against  $C$  from a series of measured corresponding values of  $K$  and  $C$  a straight line should be obtained.  $S$  is determined from the value of  $(KC^2)^{1/3}$  obtained by extrapolation of the line to  $C = 0$ , and  $v$  is obtained from the slope of the line divided by the intercept at  $C = 0$ .

Ingmanson and co-workers (106,107,109,110,220) have extended the method by introducing a specific filtration resistance term. Equations for constant-rate and constant-pressure filtration were derived, with further applications to the study of drainage resistance on the wet-end of paper-

machines. The permeability method and the method of specific filtration resistance give comparable results if the rates of flow or filtration are kept low enough that the fibers are not deformed by the fluid drag.

The Carman-Kozeny equation is derived from models with well defined geometric and physical characteristics. Although the permeability method appears theoretically straightforward it involves a number of limitations and experimental difficulties. For an adequate analysis and interpretation of the data generated through the technique a good understanding of the limitations is appropriate. The following theoretical aspects in the development of the equation, as related to actual practice should be considered.

(a) VISCOUS FLOW - the pressure applied during the measurements must be such that the fluid flow is not turbulent. Carman (30) defined the Reynolds number for a porous bed by the equation

$$Re = \frac{\rho \cdot Q}{\mu \cdot S}$$

Re = Reynolds number;

$\rho$  = density of the fluid;

Q = volumetric flow rate per unit cross-sectional area of the bed;

$\mu$  = viscosity of permeating fluid;

S = external surface area of porous mat.

Robertson and Mason (184) suggested that for viscous flow the Reynolds number should be numerically less than 2.0,

while according to Ingmanson and Andrews (108) inertial effects become significant at  $Re > 1.0$ . Due to the tortuous nature of the flow through a porous medium there are inevitably inertial as well as viscous effects, but the transition from inertial to turbulent flow is very unlikely to be achieved in the experiment, as true turbulence starts in the region of  $Re = 1000$ , and the flow rates used are very small.

(b) BED UNIFORMITY - local variation in the degree of packing of the bed should be avoided so that the void fraction is constant and the equation can be applied. These variations are usually caused by flocculation during pad formation and variability of fines distribution, the latter being particularly serious when working with pulps containing large amounts of fines.

(c) CREEP EFFECTS - because of the viscoelastic nature of the wet pulp pads creep effects are known to happen to wet pulps under compressive stresses (106). The partial movement of fibers at the boundaries of the pad, thus causing a felting, makes the permeability at a fixed pad concentration to decrease with time (creep effects are neglected in the theory). To avoid non-uniform bed compaction the hydraulic pressures applied across the pad should be only a fraction (approx. 10%) of the mechanical pressure applied by pad.

compression (106,153,179). This introduces an experimental restraint, especially when working with high fines-content pulps. Under these conditions small liquid pressures are obtained only through very low flow rates, sometimes very difficult to measure accurately.

(d) THE KOZENY CONSTANT ( $k$ ) - the capillaries in most porous media form an interconnecting network such that there can be no typical cross-sectional shape. Consequently the Kozeny constant must be evaluated experimentally. For a bed composed of discrete particles the Kozeny constant depends on the shape and orientation of the particles - e.g. different values have been found for cylinders parallel and perpendicular to the direction of flow (29).

The Carman-Kozeny equation may be derived using a circular capillary model, and the behavior of other capillary models with different cross-sectional shape can be compared with it. The Kozeny constant is then adjusted for the non-circular-capillary model, and it is assumed that a valid equation can be obtained for any porous medium, even if the flow takes place in a three dimensional network of channels.

Usually the Kozeny constant is found experimentally using a porous medium with the required type of structure and with known porosity and surface area. When the porous medium is built up of discrete particles and the Kozeny constant is calculated for uniform-size particles, this value

will not apply when the particles are of mixed size.

It has been shown (110) that it is impossible to find exactly the Kozeny constant for wood pulp fibers, due to their irregular shape and swelling ability. The effects of fiber cross-sectional shape on the resistance to the flow of fluids through fiber mats have been investigated by Labrecque (131). The results showed that as the fiber aspect ratio (the ratio of the major to the minor axis of an ellipsoidal cross section of a fiber) was increased, a slight increase was found in the flow resistance, but when going from circular to 4:1 aspect ratio fibers the Kozeny constant could be 70% off. The use of such erroneous value of  $k$  in determining the surface area could easily result in a calculated value which would be approximately 20% lower than the correct value for the surface area. It was concluded, however, that no serious errors would be involved as long as the fiber aspect ratio remains less than 3:1.

The constant also varies widely with the structure of the bed - it can be considered virtually constant for porosities less than 0.7, but increases rapidly for  $\epsilon > 0.75$  (86) - and has no specific value for particles of any shape. As a compromise, the value found (69) for randomly packed plugs of cylindrical synthetic fibers ( $k = 5.55$ ) has been accepted for calculating the surface area and specific volume of wood pulps.

Practical Considerations.- It can be concluded that, as far as the Kozeny constant is concerned, the accuracy of the permeability method for measurement of specific surface area and swollen volume depends very much on the structural similarity between the models and the actual pad. The permeability method gives the surface area for an equivalent bed of smooth cylindrical fibers so that as beating proceeds (and fiber debris and fines are formed) the calculated surface area will be increasingly less accurate in measuring the actual surface area of the pulp. Such a hydrodynamic surface, however, provides the best estimates of these properties of wet pulps, as allowed by present technology, and are of peculiar interest in papermaking, since several important steps in the processing of pulps are essentially hydrodynamic in nature.

The value of the hydrodynamic specific volume as an estimate of fiber swelling is also subjected to restrictions. The physical picture of exactly what surface the permeability method measures is not entirely defined. Theoretical considerations indicate that the calculated area is the one at the hydrodynamic plane of shear, or the swollen fiber-free water interface. The specific volume of the pulp is defined as the volume per gram of dry fiber which is denied to the flow of water. Being enclosed by the hydrodynamic plane of shear the volume per gram of dry fiber is a measure of the

total volume of the swollen fiber, including the lumen, swollen fiber walls and the water externally associated with fibrils detached from the fiber's surface. As a consequence the measurements of the total specific volume yields values which are slightly larger than other estimates of fiber swelling (220).

However when the specific volume of cellulose obtained from picnometric measurements ( $0.625 \text{ cm}^3/\text{g}$ ) is subtracted from the values obtained for the specific volume of the fiber the results are very much comparable to the widely used Jayme's (114) water retention value (21,109). Yet, these two methods for measuring swellability have been criticized as not being able to measure accurately the water associated with the fibers. Nevertheless, comparisons of WRV, obtained by centrifugation, with perhaps the best method to measure the amount of water closely associated with the swollen fibers - the dextran solute exclusion technique (213) - gave remarkably close agreement between the two. Consequently, although a direct comparison between the hydrodynamic specific volume obtained from permeability method and the solute exclusion technique has not yet been made it appears safe to consider the first as a good indication of swellability of pulp fibers.

#### Beating and Wet Web Strength

Although our knowledge about the strength of paper in

the dry state as predominantly dependent on the degree of interfiber bonding and bond strength has increased significantly in the past 30 years, the understanding of the mechanisms involved in the development of strength of the wet web is still scant. In the following a concise survey of the most relevant studies in this area is made.

After being formed on the wire the wet web attains a reasonable degree of structural integrity, but soon after the couch it will have to be able to withstand the stresses imposed upon it in the unsupported draws of the paper machine. Breaks occur in the web very often with open draws at the couch and are responsible for poor machine efficiency. With the development of pick-up at the couch the breaks were minimized at this point, but still the web is subjected to significant forces which are developed to overcome its adhesion to solid surfaces. In modern machines this usually occurs in the open draws somewhere in the press section, due to the web adhesion to press rolls. With today's trends towards lower basis weights, increased use of hardwood pulps and demanded faster machines the sensitivity of the papermaking process to wet web properties has received increased recognition.

From a machine efficiency point of view it is interesting to know the operating parameters influencing the behavior of the web. In this context some studies have indi-

cated the importance of adequate analysis of the tensions involved in the peeling of the web. Mardon (145) developed an equation relating to force required to separate a wet web from a solid surface, involving an inertial term, as a product of the web basis weight and the square power of its speed, and another term related to the adhesion of the web to the surface, with also the inclusion of an exponential relationship with take-off angle. This equation has been used for some time, especially as an aid to operation of the machines, although the equation indicates that information on the load-stretch carrying abilities of the wet webs is essential for the perfect understanding of the variables controlling machine runnability. More recent analyses have shed some light on the changes occurring in the webs under tension (94,229,239), but have also corroborated previous indications that a perfect understanding of the process of wet web transfer depends mostly on the basic mechanisms involved in the load-elongation properties of the wet sheet.

Several investigators have dealt with this subject some more involved with the basic mechanisms of wet web strength development (20,24,25,72,123,124,140,143,168,169,181,182,183,210), while others have tried to apply them to mill operation and correlations with paper machine runnability (14,15,16,145-148,132,188,198,204,211). Although

some agreement has been reached, e.g. about the importance of fiber flexibility for the strength of the wet web, general conclusions are somewhat contradictory, mostly because the experiments have involved different conditions of specimen preparation, testing and reporting of results.

Based on the fundamental studies of Lyne and Gallay (143) who demonstrated the importance of the water content in the web, many works were performed with test strips under constant moisture content. However, with the recognition that the test information should relate as closely as possible to the conditions present in the paper machine it has been increasingly emphasized that the testing of different furnishes should be done after their preparation under the same conditions of e.g. drainage time, vacuum and wet pressing (15,188,198,210). If different dry contents and strengths values are found after forming, then these must be considered as actual differences in the qualities of the furnishes, and they should be dealt with as representative of their conditions before reaching the open draw, as simulated by the test.

Another source of contradictions, especially with relation to machine runnability, is which of the properties measured in a load-elongation test is the limiting factor for the understanding of its behavior in the paper machine.

When discussing about the wet web strength of a given furnish an author might be referring to either its tensile strength, the stretch at break or the wet rupture energy (WRE), as measured by the integration of the load-elongation curve.

Obviously all properties are important and somehow interrelated, which is probably why a recent study (198) has adopted all of them as criteria of wet web quality. Results from mill experience (14,15,16,188) and some other studies (94,169,229,210) have expressed an increasing acceptance of stretch as the important wet web characteristic required to withstand the draws in the machines. Their explanation is based on the observed fact that the ability in terms of stress prior to rupture which is available to withstand localized stress concentrations is normally present at adequate levels in common furnishes, while the ability to redistribute web stresses within the structure by stretching and avoiding crack propagations is not usually afforded solely by a strong furnish. Since these factors appear to interrelate somewhat and, in many cases, the results of different investigators are not comparable it may be concluded that the relative importance of stretch and load at rupture remains to be fully explained.

As a consequence of some of the contradictions found in the literature as discussed above, there still seems to be a

lack of understanding of the mechanical properties of wet webs required for fast machines producing lightweight papers. Particularly the effects of fiber properties and degree of beating have received only limited attention.

Fundamentals of Wet Web Strength Development.- The first studies involved with basic mechanism of web strength development were reported by Lyne and Gallay (143). These authors analyzed the changes occurring in the wet web as water was removed, mostly by air drying with a very little amount of wet pressing. Their results were widely accepted, and only a few investigators pursued in the efforts towards the understanding of the fundamentals of the fiber-water interactions in the wet end, among them Brecht and co-workers (24,25), Nordman and Eravuo (168) and Robertson (181, 182,183), the latter extending significantly the results of Lyne and Gallay. Surprisingly enough other fundamental studies of the same caliber were not found in the literature, which includes mostly additional data on different conditions supporting those previous limited conclusions. A review of the literature on wet web strength including works from 1945 to 1970 has been published (146). The following picture of wet web strength is based on the results and ideas formulated by the authors mentioned above.

After the web is formed on the wire and until it reaches up to 10-12% solids its strength is very low, and due entirely to the frictional resistance among the fibers. This resistance to deformation is provided by the degree of interweaving or entanglement of the fibers, and on fiber flexibility. Therefore longer and thinner fibers, with a low bending modulus are expected to form networks of considerable strength, even at low solids contents. The good correlations found between the strength of flocs under the shear forces imposed by flow conditions and wet web strength (68) seem to agree with this mechanism. Increased surface area of the fibers would also be expected to enhance the degree of association and friction among the fibers. The number of bends and kinks (68) in the fibers, as developed during high consistency bleaching (123,124) also seem to affect the degree of frictional resistance. It should be remembered that this stage of fiber association will play a part in strength at any degree of dryness in the sheet. As Lyne and Gallay have expressed "it may be assumed that the frictional resistance operates superimposed on other factors" (143).

As the web is being formed water removal beyond 12% solids is accomplished by compression of the web. The draining water and/or compression in the press nip promotes the minimization of water-air interface by surface tension forces.

The normal forces between the fibers will be increased as they are brought together, and the whole network is compacted until its resistance to further deformation exceeds the driving force of the draining water, and air intrudes in the structure (201). This point is determined by the compressibility of the web, its water retention properties and the specific surface area, which directly influences the pore size distribution (182), and usually occurs between 20-25% solids content. A state of increased density is then reached, at which the fibers are pulled close together and are connected to neighboring fibers by liquid patches of water at each point of contact or near contact.

This second stage of strength development is then characterized by the increased effect of surface tension forces at the fiber-water-air interfaces. With progressive water removal the air replaces water at increasing depths in the structure with the development of greater water-air interface and a consequent adhesion between fibers, additionally held up by some sort of "surface energy" (143). A further increase in density of the web is then noticed. This stage of removal of large amounts of interfiber water progresses until a dry content of 28-36% is reached, after which only water at fiber crossover regions, within fiber walls and possibly in the lumen remains. According to Robertson (182) this point corresponds to the amount of water

strongly associated with the fibers, and should be predicted by the permeability specific volume. The increase in strength beyond this point is very steep, with flattening of fiber walls and closer interaction among adjacent fibers until actual bonding becomes possible, around 50% solids content.

A larger specific surface area in the pulp, mostly due to the fines should be expected to increase the strength in the 20-36% solids region by providing a larger associated water-air interface, and thus larger surface tension forces (20,68,80,72,123,132,140,161,169). Studies on the wet web strength of mechanical pulps (72,132,161,169) have shown that long fibers, as well as fines have distinct beneficial effects, the fibers probably enhancing the first stage of strength development.

Thus, although it is known that the degree of fiber entanglement during web formation plus the increased surface tension forces due to air water interfaces after further removal of water from the web are very important factors influencing the strength of the wet web, it is not really understood how fibers and fines behave with respect to water removal, which is fundamental for the development of strength when air is present in the structure. As most of the studies were performed at a given dry content, usually in the 20-30% solids range, and after different sets of preparation conditions (e.g. suction, and/or wet pressing,

and/or drying) the considerable amount of data available probably represents the results of different states of association of fiber and water. In other words, under certain preparation conditions the water remaining in the web at a given dry content could have been left in the fiber wall, in the lumen, as an envelope around the fibers fibrils and fines, or different combinations of these possibilities.

Robertson (182,183) was the first to recognize the importance of not only the amount of water held by the fibers, but the manner by which it is associated. He proposed a dye migration test, which should indicate the moisture content at which the migration of water from fiber to fiber under the action of capillary forces become negligible. A lower dye migration point at the same equilibrium water retention value indicated how well the fibers would deform under compressive forces and collapse, resulting in a stronger wet sheet. Unfortunately these studies were not pursued and our knowledge today seems to be limited to the interpretation of numerous published values under the mechanisms proposed initially by Lyne and Gallay, without actually understanding why different pulps have such particular behavior in the wet end.

An interesting work, somewhat related to Robertson's findings, but more applicable to the surface tension forces

developed as a result of air entrainment in the web was reported by Bogomol and Luchinkina (20). They proposed the concept of a mean thickness ( $\delta$ ) of water film at the outer surface of the fibers, which was calculated by the ratio between the volume of the water associated with the fibers and the specific surface of the pulp. Although lacking a fundamental basis, in view of what the permeability specific surface and volume actually represent, this parameter was able to account for differences in web web strength of a groundwood, a sulfite and a kraft pulp. It was proposed that the smaller the  $\delta$  the larger the surface tension forces which pull the fibers together, thereby increasing the wet web strength.

Wet Web Stretch and Tensile Strength.- When referring collectively to the term wet web strength the investigators are most usually alluding to both tensile and stretch. As discussed previously the relative importance of each with respect to papermachine runnability is not certain, but it is important to realize how these different properties behave for various furnishes. The investigators have reported somewhat contradictory effects of fiber characteristics on wet tensile strength and stretch.

For instance there are those who insist that fiber flexibility and length have a significant effect on elongation, due to the increased elasticity of the network, with

only modest increases in tensile (14,169,210). Others have indicated that for chemical pulps the elongation would be a function of twisting <sup>lyratura</sup> and stretching of the fibers, independently of fiber length (171), while just the opposite conclusion was reached when comparing a softwood TMP and birch CTMP (169), as fiber length was shown to have a significant influence on stretch.

With reference to wet tensile strength good correlations have been found between this property and the average fiber length and specific surface of mechanical pulps (132) and an unbeaten sulfite pulp. The influence of the specific surface is also evident by examining the behavior of the wet webs when the fines are removed (140) and the importance of having fibrillated TMP fibers, as well as flexible, for maximum wet tensile and elongation (16). However, the influence of fiber flexibility on wet tensile strength has also been demonstrated when the effects of fiber length were eliminated (182,183), and also as an additional factor to the beneficial influence of the surface area of the pulp.

This confusing panorama of events seems to indicate that tensile and stretch of the wet web are pretty much interrelated. Therefore the different characteristics of the furnishes and their components appear to exert their specific influences at different stages of wet web strength

development, as discussed earlier, and their overall effect becomes evident in both elongation and tensile strength. In view of the increasingly important necessity for a better understanding of the factors controlling paper-machine runnability there seems to be an urgent need for an agreement among the investigators about standardized conditions for wet web testing, followed by a considerable amount of research on the fundamentals of the wet web strength, particularly with respect to fiber-water interactions.

With respect to beating and refining not much has been reported since the initial studies on the strength of the wet web (24,143,182,183). It is commonly accepted that the strength of the wet web will follow a beating curve very similar to the development of dry strength, but considerations on the different possible ways of treating a pulp to achieve the best wet web strength are very limited.

Some results based on mill experience can be cited, such as increased wet web strength at a given energy consumption when refining at higher load (132,211), increased wet tensile when refining at low consistency and a higher stretch when using high consistency refining (211). It has also been reported that during the beating of semi-bleached kraft pulp the largest increases in wet web strength occurred at the early stages (148). During the

preparation of TMP it has been recently shown that the refiner power should be more efficiently used for fiber separation at the  $S_1$ - $S_2$  interface, thus yielding fibrillated fibers that would be chemically softened later for maximum wet web strength, rather than softening the fibers prior to refining, which would yield fibers with little fibrillation and hence a smaller wet web strength (16).

## MATERIALS AND METHODS

### Raw Materials

All pulps used in this study were samples from commercial bales received directly from the manufacturers. They were fully bleached kraft pulps, available as dry laps. These pulps were prepared under conditions normally used for manufacture of low yield (47-52%) kraft pulps and bleached using modern bleaching sequences, aimed at high brightness while preserving strength.

Some characteristics particular to each pulp are given below, together with the respective manufacturer.

Eucalyptus grandis - produced from a fast-grown hybrid of Eucalyptus grandis W. Hill, ex Maiden and Eucalyptus Urophylla S. T. Blake, (referred to as simply E. grandis throughout this study) and supplied by ARACRUZ CELULOSE, S.A. Brazil. One particular aspect of this pulp was its manufacture including all bolebark.

Because of the high extractives content in the bark, pulping conditions were more drastic than usual, including higher than normal alkali charges and high sulfidity (about 35%). This pulp is being introduced in the United States and Europe in increasing quantities.

Eucalyptus rostrata - also produced from fast-grown trees,

which, however, require a completely different set of environmental conditions for optimum growth than E. grandis. Eucalyptus rostrata is the more commonly used name of Eucalyptus camaldulensis Dehnh., which grows particularly well in the Mediterranean countries. The pulp was supplied by Empresa Nacional de Celulosas S.A. Huelva, Spain, one of the main suppliers of eucalypt pulp in Europe.

Silver birch (or European birch) - is probably the most widely used hardwood pulp in Europe. This species (Betula verrucosa Ehrh.) has characteristics very similar to white birch (Betula papyrifera Marsh.), which is grown in North America. The choice of silver birch was dictated by its ready availability as a kraft pulp in pure form, rather than mixed with other hardwoods. It was manufactured by Billerud-Uddeholm AB - Sweden.

Southern pine - one of the most important sources of long fiber in the United States and in the world, this term comprises a number of different species with very close chemical and morphological properties. The pulp used was manufactured from a mixture of loblolly (Pinus taeda L.), short leaf (Pinus echinata Mill.) and long leaf (Pinus palustris Mill.) pines, in unknown proportions, and can be considered as typical of a southern pine bleached kraft pulp. The supplier was the Hammermill-Riverdale plant, of Selma-Alabama.

For precise morphological characterization of the raw

materials, samples of the same wood as used for pulping were supplied by the manufacturers of the three hardwood pulps. These samples consisted of wood discs cut at breast-height from different trees at harvesting age. Samples of wood from the southern pines were not supplied, but this did not create any problem since the pulp was a mixture of three species, and a wealth of information on them is available in the literature.

The measurements of wood fiber cross sectional dimensions were made directly on photomicrographs prepared under different procedures. To ensure representative sampling from the wood discs, blocks were cut at different positions across the grain, the relative amount of wood at each position was computed and weighed average calculations were performed on every measurement for each sample.

#### Preparation of Pulps for Measurements and Beating

The pulps were slushed in distilled water before any measurement or treatment was performed. The slushing operation consisted of soaking the pulps in water overnight without tearing the sheets and then suspending them in 40 liter plastic containers using a Lightning NC 4 mixer at 1725 rpm. The consistency used was 3.0%, with agitation continuing for two hours. This extended time for reslushing was due to the difficult of dispersion of birch and E. rostrata pulps, which

had a tendency to display a large quantity of "fisheyes". The suspensions were then drained over a classification cloth, handpressed to about 17% dry content, and stored in plastic bags for further utilization. The slushing operation was repeated many times throughout the course of this work, the same conditions being applied each time. A total of about 15 kg. of each pulp were used.

### Microscopy

#### Transmission Electron Microcopy

The transmission electron micrographs of wood cross sections were prepared according to the following procedure: small cubes of wood were cut at different positions across the grain. They were boiled in water for softening and then dehydrated sequentially in water-ethyl alcohol mixtures of increasing alcohol content, finishing with absolute ethanol. This procedure ensured good penetration of the embedding medium and avoided moisture, to which the curing of metacrylate is particularly sensitive.

The embedding mixture consisted of 80% butyl-metacrylate and 20% methyl-metacrylate, with 1% w/v benzoperoxide accelerator and 0.1 percent uranyl nitrate, to reduce the possibility of "explosion" artifacts. The dehydrated wood blocks were embedded in the polymerizing medium within gelatin capsules and left in oven at 60°C for 24 hours. The polymerized blocks were then trimmed down to about 1 mm<sup>2</sup> cutting

face, whereupon ultrathin sections (500-600 Å thick) were cut with a Ultramicrotome Porter-Blum MT2B equipped with a DuPont diamond knife. The sections were picked-up on 100 mesh substrate grids, and placed in chloroform to dissolve the metacrylate embedding. The grids were then shadow-casted in a high vacuum evaporator, and the samples were ready for pictures. The electromicrographs were taken in a RCA-EMU 4A transmission electron microscope, using magnifications ranging from 2000 to 3000X.

The negatives were then enlarged to prints with a total magnification of 6240X where the measurements were made. At least 50 fibers from each species were measured at random from representative samples of the wood cross section.

### Scanning Electron Microscopy

Pulp Fibers and Fines. - Suspensions of pulp fibers free of fines and of separated fines were spread out on microscope slides, frozen by immersion in liquid air, and then sublimated in a freeze-drying unit. The samples were then transferred to specimen holders with pieces of double-stick tape, which were grounded to the specimen holder with colloidal carbon. The next step was sputtering with gold-palladium film of approximately 200 Å thickness.

The electron micrographs were taken with an ETEC Auto-scan Scanning electron microscope, at magnifications of 100X to 5000X. Before choosing an area for pictures the

samples were observed in detail at different spots to avoid bias towards fibers showing under-or over-treatments. Choices of the most "typical" areas were made based on agreement between two observers.

Paper. - Handsheet samples were embedded in cyanoacrylate and cured in oven at 60°C for 10 minutes. This procedure was repeated two more times in order to "bulk" the paper so that it could be cut with a fresh razor blade at right angles to the paper surface. The cyanoacrylate was then removed with acetone and the specimens mounted on tape. The rest of the procedure was the same as for pulp fibers. All pictures were taken at 400X magnification.

### Light Microscopy

Wood Cross Sections. - Small cubes of wood were again cut at different positions across the grain. They were boiled in water until they sank and then cross sections 15-20 microns thick were made on a sliding microtome. The sections were double stained with hematoxylin and safranin after pretreatment with ferric-ammonium sulfate, used as a chelating agent for the hematoxylin. The staining was followed by dehydration, mounting in Histoclad synthetic resin and, finally, drying.

Light micrographs were prepared under different conditions, for measurements in the image analyzer, and for general

observation. The pictures for Image Analysis must have a great deal of contrast, and since the cross sections were stained in safranin the best contrast was obtained with a Kodak No. 45 blue filter. The micrographs were taken with a Zeiss Photomicroscope II on Kodak Plus-X film, using 100X and 16X magnifications. The pictures for general observation were taken using the same equipment with 0.12 and 0.50 neutral density filters and a magnification of 40X.

#### Pulp Samples

Fiber Cross Sections - The fines-free fibers from the slushed pulps were first stained with Victoria Blue 4R and then dehydrated sequentially in water-ethanol mixtures of increasing alcohol content, finishing with absolute ethanol. The fibers were then transferred into propylene oxide, followed by sequential immersions in mixtures of propylene oxide and the embedding medium of increasing contents of the latter. The embedding medium used was EM-bed-812 (Electron Microscope Sciences), an epoxy-based resin very similar to EPON 812, which is known to have negligible swelling effects on fiber cross section.

The embedding was done in rubber molds, where the fibers were oriented parallel to each other in the best way possible with dissecting needles. After curing in oven for 72 hours at 50°C the sections (4  $\mu$ m thick) were cut using a sliding microtome. Twelve sections from each pulp were then

mounted on microscope slides, using the same embedding medium described above. In order to achieve maximum flatness in the mountings, the slides were clipped together while curing in the oven.

Light micrographs of randomly selected fiber cross-sections were taken with Zeiss Photomicroscope II on Kodak Plus-X 35 mm film, using 310X magnification. The technique used was phase-contrast.

Microfibrillar Orientations. - Fibers were photographed for measurement of the  $S_1$  and  $S_2$  angles. The technique used was the same as described by Crosby and Mark (44), and consisted of phase-contrast microscopy using near-ultraviolet illumination. The fibers were mounted in distilled water using microscope slides and cover slips, which were sealed with wax to retard drying.

After viewing the cell wall striations under phase-contrast illumination the light source was changed to a high pressure mercury burner lamp to improve the resolution. The resultant illumination was filtered with a BG3 exciter filter No. II and polarizer filter, to pass wavelengths in the 300-475 nm range. The pictures were taken using a Zeiss Universal Photomicroscope I with American Optical external camera on high-resolution metallographic plates. Total magnifications on plates were in the range 1285-1645 X.

## Measurements on Wood and Pulp

### Fibers and Vessels

#### Lumen Diameter of Fibers and Vessels

Pictures from wood cross sections were taken with the light microscope using a blue filter as described earlier. After development, the images from the negatives were fed to an image analyzer via a television scanning system, using transmitted light from a back-up illuminated screen. A Cambridge Scientific Instruments Quantimed 720 Image Analyzer equipped with a 2-D auto detector was used. The image from each negative picture of wood cross sections was converted to a string of digits, each representing a defined gray level at a specific corresponding point on the image. The computer logic counts and obtains precise size information about each lumen diameter in the field of view by detecting a change in the gray level.

For measurement of areas of fiber lumens on 100X negatives the computer was programmed to count and size only the areas ranging from 0.0 to 5.0 mm<sup>2</sup>, in 0.1 mm<sup>2</sup> intervals. The computer provides an output for each image containing a histogram for the percentage of total number of features measured in each area interval, as well as other statistical information, such as the total number of features counted, their average, and variance. For each species the weighed average

fiber lumen diameter was then calculated from the weighed average fiber lumen areas, obtained by association of image analysis results and the respective percentage of each image in the total wood, assuming circular cross sections. A total of 642 fibers in birch, 1322 in E. grandis and 1313 in E. rostrata were measured.

The same procedure was followed for measurement of lumen diameter of vessels on 16X negatives, although the computer program was changed to count and size only the areas ranging from 0.2 to 6.2 mm<sup>2</sup> in birch and 1.0 to 16.0 mm<sup>2</sup> in E. rostrata and E. grandis. A total of 103 vessels in E. grandis, 73 in E. rostrata and 186 in birch were measured. The number of vessels per square milimeter was calculated from the known area of the image and the number of vessels counted on each of the images.

#### Cell Wall Layers in Wood Fibers - Fibers Wall Thickness

These were performed directly on 6240X enlargements of transmission electron micrographs of wood cross sections. The cell walls were measured two at-a-time following a line perpendicular to the wall layers, at points where adjacent cells had their walls more or less flattened. Each fiber was measured in at least three different locations, avoiding cell wall corners. The S<sub>1</sub>, S<sub>2</sub> and S<sub>3</sub> layers were perfectly visible in the electron micrographs, but the primary walls were not distinct from the middle lamella. Cell wall thick-

ness was calculated by adding up the measured dimensions of  $S_1$ ,  $S_2$  and  $S_3$  layers. At least 50 fibers were measured in each species, with special care taken to avoid bias towards preferential measurement of small fibers, due to the limited number of photographs available.

### Pulp Fibers

Cross Sectional Dimensions.- The 35 mm negatives were projected on heavy paper (approx.  $200 \text{ g/m}^2$ ), whereupon the cross-sections of the fibers were drawn, giving a total magnification of 2600X. The projected cross sections were then cut, and weighed to the nearest  $10^{-4} \text{ g}$  under constant humidity and temperature (TAPPI Standard T402 os-70). The projected image of a 1mm scale, with 0.01 mm subdivisions, which was photographed at the same magnification as the fibers, was used to draw a square equivalent to  $10 \text{ } \mu\text{m}$  wide. This square was cut from the same paper as the fibers, and weighed in the same conditions. The cross-sectional areas of the pulp fibers were then determined by dividing the weight of their projections on paper by the weight of the square. The reproducibility and accuracy of this method were excellent, even when compared with the same measurements on enlarged projections of cross-sections of the fibers using a planimeter. The perimeter of the fibers was also measured on the projections using a plan measure. Ten fibers from each of

the four pulps were measured at random, resulting in small 95% confidence intervals for each case.

S<sub>1</sub> and S<sub>2</sub> Microfibrillar Angles.- The negatives on metallographic plates were projected on paper, total magnifications ranging from 3400 to 4300X. The striations and fiber edges were drawn and the angles measured in at least three points (average of 9 measurements per fiber) near the center of the fiber, avoiding edges and tips. Measurements on 20 fibers of each pulp were performed for S<sub>2</sub> angle determination, with very satisfactory variance, which in turn provided the estimation of small 95% confidence intervals. For measurement of the fibril orientation in the S<sub>1</sub> layer the variability was much larger, partly due to the difficulties with resolution of this layer in the light microscope. Even with 25 fibers measured the variance was still very large, although allowing comparisons between the species.

Weighed Average Fiber Length.- The procedure followed was essentially TAPPI Standard T 233 os-75, with pulp classification performed with the Bauer-McNett Classifier using 30, 50, 100 and 200 mesh screens. This procedure was repeated three times. The average length of fibers in each of the fractions was determined by projection of the fibers mounted on microscope slides to a screen at known magnifications, and measurement of the lengths of at least 200 fibers to the nearest 0.01 mm using a plan measure. The

projector used was a Bioscope. The average length of vessel elements was determined using the same procedure.

Fiber Coarseness. - This property is a very important one to consider in defining fiber morphology. It has a direct effect of paper formation and when combined with the weighed average fiber length it allows the calculation of the number of fibers per gram of pulp, another property of great significance. The determination of fiber coarseness in pulp was made using Britt's technique (26). Two sheets of approximately  $1\text{g/m}^2$  basis weight from different weighings were made from each pulp in the British mold. The 200 mesh wires carrying the sheets were removed from the mold and allowed to dry in oven at  $105^\circ\text{C}$  for 10 minutes. On each dry wire two strips of transparent tape were applied, firmly pressed, peeled off and transferred to microscope slides. Fibers were counted with an eyepiece with a pointer in a light microscope, making two complete transverse movements of the slide. This gave eight measurements per pulp, four from each weighing, which greatly reduced the variance of the results. The coarseness, in  $\text{mg}/100\text{m}$ , was calculated according to the formula:

$$C = \frac{2000}{\pi} \times \frac{W \times l}{N}$$

where  $W$  = basis weight of the sheet,  
 $\text{g/m}^2$   
 $l$  = length of transverse slide  
movement, cm  
 $N$  = number of fibers counted  
across slide.

Chemical Analysis of Pulps.- The chemical analysis involved the determinations of ash content, extractables content, lignin content, and percentages of carbohydrates.

Ash Content.- The determinations were made according to TAPPI method T 211 om-80. Two repetitions on each pulp were enough to provide accurate results, since the reproducibility was excellent. The results were reported on an oven-dry basis.

Extractables Content.- Different mixtures of solvents can be used to solubilize resin, fatty acids and their esters, waxes and unsaponifiable substances in pulps. The TAPPI standard T 204 os-76 suggests the use of either alcohol-benzene or dichloromethane for the extractions for wood, but indicates that the mixture ethanol-benzene appears to provide the most complete removal of solvent-extractables in pulp. However, the results indicated that extractions of pulps with dichloromethane were much more reproducible, and they were therefore adopted. Two replications were performed in each pulp, and results were reported on an oven-dry, unextracted sample basis.

Sugar Analysis.- All pulps had very high brightness, and so it was initially intended to perform only an analysis of pentosans. However, since one of the pulps was from a softwood, additional information about the hemicelluloses

was required. A more complete analysis was performed using instrumental methods. The pulps were pre-hydrolyzed with 72% sulfuric acid for one hour at 30°C, followed by a second hydrolysis with 0.5 molar sulfuric acid at 120°C in an autoclave. The separation of sugars was done using liquid chromatography and colorimetry, with two replications. The results are reported on an oven-dry, ash-free and extracted sample basis. It was interesting to notice that the results from pentosan analysis alone using TAPPI T 223 ts-63 method matched the xylan plus arabinan contents obtained using chromatography within  $\pm 0.5\%$ . Lignin content of all pulps was zero.

#### Additional Tests for Pulp Characterization

Viscosity.- The determinations were made using the capillary viscometer method, as described in TAPPI Standard T 230 os-76. The slushed pulp samples at approximately 17% dry content were exchanged with acetone and then dried in a desicator under vacuum. After weighing the pulps were dissolved in 0.5 molar cupriethylenediamine using the closed bottle procedure. Calibration of the viscometer was done with oil "J" - density  $0.8505 \text{ g/cm}^3$  and viscosity 18.42 cp, at 25°C and the measurements of efflux time of cupriethylenediamine solutions replicated three times. The results were reported in centipoises (cP).

Zero-Span Tensile Strength.- A good estimate of the average strength of the fibers in a pulp can be obtained with this test. The method chosen was based on the measurement of the failure load of a wet sheet of paper with a zero nominal clamping length, denominated wet zero span test. The theory underlying the method is based on the assumption that in a wet sheet of paper the strength of fiber bonds is very small when compared to the strength of the fibers (42). Thus, the tensile index measured on a wet sheet is an indication of the failure load of its fibers since they will theoretically be stretched until they break. In practice, however there are always some fibers which are not effectively clamped by both jaws, and the value obtained deviates from the true strength of the fibers. This method was chosen due to its high reproducibility as compared to zero span tests on dry sheets.

Handsheets of  $60 \text{ g/m}^2$  dry basis weight were prepared according to TAPPI standard T 205 os-71, dried and conditioned according to T 402 os-70. After cutting into strips 15 mm wide, they were soaked in water overnight. After pressed between two blotters under 196 KPa for two minutes the strips were tested in the Pulmac Zero Span Tester. Whole pulps beaten in the PFI mill for 500 and 1500 revolutions (1500 and 3000 rev. for southern pine) under standard conditions (T 248 pm-74) were used for testing. Six replications were made for each pulp and the results reported

as tensile index in N m/g, on a dry sheet basis.

**Pulp Brightness.-** Buchner pads were prepared from slushed pulps according to TAPPI Standard T 218 os-75. The reflectance tests were performed with the Zeiss Elrepho light reflectometer at 457 nm, and the results reported as degrees ISO.

**pH of Pulp Suspension.-** The slushed pulps were resuspended in distilled water of pH = 5.5 for 30 minutes and their pH's determined with a Beckman zeromatic SS-3 potentiometer at 23°C.

### Beating of the Pulps

#### Beating in the PFI Mill

This operation was performed following TAPPI T 248 pm-74 method. The pulps were beaten at 10 percent consistency and  $20 \pm 5^\circ\text{C}$  starting temperature, using a 30g o.d. charge for each run. The vernier adjusting screw was set at a completely backed off position so that no fixed minimum clearance was set between the roll and the housing. Particular care was taken concerning the conditioning of the surfaces prior to the actual beating of individual pulps. This was done by beating the same pulp three times for 8000 revolutions, other conditions being the same as for actual beating, and discarding the beaten pulps.

Large quantities of pulps were needed at each beating level because of the large number of tests to be performed. Therefore the beating of each pulp for a given number of revolutions was repeated a number of times. After each run the beater roll and housing were cooled down to approximately 20°C. The beaten pulps at each treatment level were directly transferred to a plastic bag, hand-mixed at 10 percent consistency and stored under cold temperature. Special care was taken to avoid the losses of fines. The bulk of the analysis was performed on pulps prepared in a sequence of days with approximately the same ambient conditions. However, during the course of this work more samples were needed for testing, and more beating had to be done. Whenever this happened the same care was taken concerning the conditioning of the beater surfaces, using the same water and ambient temperatures, and monitoring of the conditions in the beater, such as speeds of roll and housing. At the same time each pulp would be beaten at least three times at each treatment level and then hand-mixed in a plastic bag.

The pulps were beaten at two different loadings of the lever, the standard 3.33 kN/m (of bar length) and 4.90 kN/m, the latter obtained by application of an additional known weight to the lever. At each load the pulps were treated, for four different numbers of revolutions, 500, 1500, 3000 and 5000 for the hardwoods and 1500, 3000, 5000, and 8000

for the southern pine pulp. By measuring the rotor speed with a stop-watch and the housing speed with a tachometer it was possible to calculate the differential speed between roll and housing and to monitor it for the different pulps at the two-load levels. It was found that the differential speed was essentially the same for all pulps beaten at each load, but slightly higher when beating at 4.90 kN/m.

In addition to these measurements there was an interest in following the beating operation with other parameters, such as the changes in clearance between the rotor and house and the net energy consumption. These were determined in a separate batch of experiments, under the same conditions as observed during beating of the pulps used for testing.

Measurement of Beating Clearance - As discussed above all beatings were done with the vernier adjusting screw at a completely backed off position. In this way no fixed minimum clearance was set, and the pulp could receive the full load when impacted. However, once the layer of pulp is distributed evenly over the housing surface a distance is set between the roll and the housing when the weight lever arm is released. This distance (or clearance) can be measured by moving the adjusting screw against the stop peg and making readings on the vernier. The calibration of the vernier is done by setting it in zero when the adjusting screw first

touches the stop peg, as contact is made between roll and house with the lever fully released. This is done under static conditions and without pulp. The calibration can be reproduced without difficulty, and actually it was not changed during the trials.

Actual beating clearances were measured under dynamic conditions, i.e. with movement of roll and house. Each pulp was beaten for 8000 revolutions and the clearances recorded at each desired level. This procedure was repeated ten times at each loading level, until satisfactory confidence intervals were obtained. Another technique, which employs an inductive displacement transducer in conjunction with a direct reading measuring bridge has shown higher accuracy in each measurement of clearance (233,234), therefore needing less replications than used in this work. Nevertheless, their results obtained for pine and eucalypt pulps are of the same order of magnitude as the ones here reported. The negative values obtained for clearances are due to the fact that the relative positions of the roll and house change under static and dynamic conditions. This phenomenon does not create any practical problem since all data is referred to the same zero static clearance.

Power Measurement.- During the same twenty runs for each pulp as performed for measurement of clearance, the PFI mill was connected to a wattmeter designed especially for

measurement of power intake during refining. In this unit the total electrical current from the rotor and house motors is passed through current coils. The voltage is applied through potential transformers, which in turn transfer it to a watt transducer. This information is relayed to chart recorder, which provides the final power intake against elapsed time. Since each recording was done for a total of 8000 revolutions in the mill the power intake at each level of sampling (e.g. 500, 1500 rev., etc.) was calculated using determinations of elapsed times with a stop-watch. The calculations of net energy consumption were done by integration of the power intake vs. elapsed time curve, after subtracting the relayed power intake during running of roll and housing motors with pulp inside the beater, before releasing the weight lever. The results were reported as MJ/kg, on an oven-dried pulp basis.

Absolute values of power measurements were not critical since only the net power intake was calculated from them. In any case the accuracy of the wattmeter is expectedly high, from the extremely small tolerance of its components. These considerations are based on the experience with the performance of the instrument during power measurements of a number of other refiners for many years. No attempt was made to measure the power intake of the house and roll motors separately as others have done (65,235). It is known that under

certain conditions the interaction between the roll and house results in the roll actually driving the house, and power is fed back to the house motor. Since no major changes in differential speed were observed among the pulps under the same conditions, it was considered that if there were any differences between the measurements and the actual power consumptions they were approximately the same for all pulps, which could therefore be compared amongst themselves.

### Evaluation of Beating Results

#### Measurements in Wet Pulps

The procedures included pulp classification, determination of fines content in beaten and unbeaten pulps, Canadian Standard Freeness, dynamic drainage time, wet web strength and caliper, and hydrodynamic specific surface and specific volume.

Pulp Classification. - In order to identify changes occurring in fibers, as well as in whole pulps during beating, part of the samples obtained at each treatment level were fractionated in the Bauer-McNett classifier. For the purposes of this study the fines (both from unbeaten

and beaten pulps) were defined as the fraction passing the 200 mesh screen during classification. After disintegration, the pulps were charged to the classifier in batches of 10 g.o.d., which ran for 20 minutes each, at 10 g/l water flow rate. All fractions retained on screens up to 200 mesh, were then mixed back while draining over a classification cloth, hand-pressed to about 22% dry content, and stored under cold temperature. The fines were discarded. The pulps obtained through this procedure were termed fines-free, or pure fibers. Samples of these pulps were tested for fines content in the Dynamic Drainage Jar, as described below, whereby the efficiency of separation in the classifier was confirmed.

Fines Content of Whole Pulps.- Although the fines-free pulps were prepared using the Bauer-McNett Classifier the determination of fines fraction was done using the Dynamic Drainage Jar (DDJ) (Figure 1), which can be used as essentially a single screen classifier. The use of this device allowed a much higher degree of reproducibility than obtained with the Bauer-McNett, although the results correlate quite well (95) when using a screen with a 76  $\mu$ m round hole - 14.5%

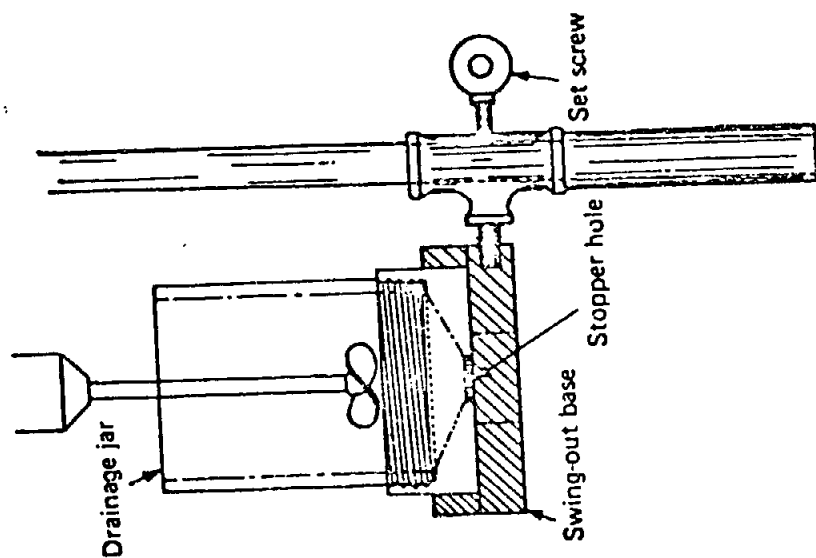


Figure 1 - Dynamic drainage jar.

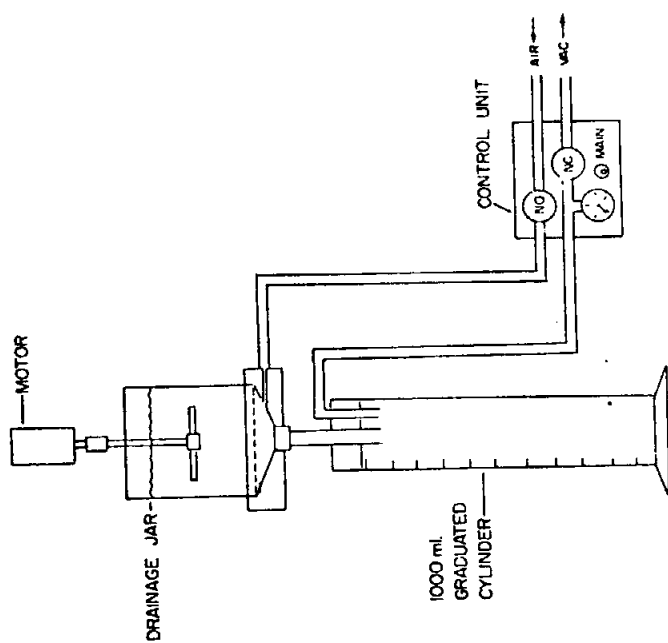


Figure 2 - Apparatus for measurement of dynamic drainage time.

open area (nominally 200 mesh) in the DDJ. The procedure followed is outlined in TAPPI method T 261 pm-79 but some minor modifications were included for higher accuracy. A batch of pulp at approximately 0.5% consistency was prepared and kept under stirring during the test. For accurate determination of consistency about 200 ml of pulp were sampled from the batch each time into four completely dry 250-ml beakers, which were weighed on a fast-weighing balance. In order to avoid eventual losses of fines during filtering of these samples through weighed filter papers (Whatman No. 30) the pulps in each beaker were flocculated using sequential treatment of 0.5% cationic polycrylamide and 0.01% anionic polyacrylamide. It was found out that this procedure increased dramatically the accuracy of consistency determinations. After filtering the flocculated suspensions the filter papers were dried in oven at 105°C, and consistency calculated as usual.

For the fractionation procedure about 150 ml were sampled from the suspension of known consistency, weighed in a fast-weighing balance and diluted to 500 ml with "wash water" (5 ml of a non-foaming dispersant - 2.5% w/w sodium carbonate, 2.5% w/w sodium tri-polyphosphate and 2.5% w/w Tamol 850 - in 4 liters of distilled water). This suspension was transferred to the DDJ, and drained at 750 rpm, with the propeller set at 3.5 mm from the screen. The fines passing

the screen were collected in a beaker, allowed to settle, and used later for microscopic observation. Additional 500 ml portions of wash water were added to the jar and drained as before, until no more fines were observed in the filtrate. Usually six to eight washings had to be done until this was observed, especially with the well-beaten pulps. The fibers remaining on the screen were then transferred to a weighed filter paper, which was dried in oven and again weighed. The amount of fines in the original pulps was calculated by subtracting the final weight of pure fibers from the known dry weight of the pulp sampled for fractionation (from consistency determinations). Although very reproducible this procedure was repeated from five to seven times for each beaten pulp, until a satisfactory 95% confidence interval could be constructed, and the pulps could be compared in relation to this property. The results are reported as percentage in weight of whole pulps, on an oven-dry basis.

Freeness - This test supposedly gives an arbitrary indication about the drainability of a pulp suspension. The Canadian Standard Freeness (CSF) tester was designed specifically to monitor the quality of groundwood pulps, but since it is influenced most by the quantity of fines present, and these are also formed during beating of chemical pulps, the method is widely used for control of the extent of mechanical

treatment of chemical pulps as well. The procedure is very straightforward and performed routinely in many laboratories. The methodology followed was according to TAPPI Standard T 227 m-58, with the pulps being directly sampled from plastic bags, as described earlier, and disintegrated with a Lightning mixer at 1.0% consistency for 3 minutes. All measurements were done using distilled water and are reported in milliliters. The freeness of fines-free pulps was not measured.

Dynamic Drainage Time. -Although it can give an indication of how difficult a stock will be to drain the freeness test cannot be used to predict the performance of a pulp in the wet-end of a papermachine. Reasons for this are many and they are all derived from the lack of similarity between the test and actual conditions on the paper machine. A dynamic drainage time was measured on all beaten whole pulps, by including two operating variables of primary importance during dewatering which are neglected in the freeness test: the suction present in table rolls and foils, and the turbulence of the stock coming from the headbox.

The high vacuum ensures a high pressure drop across the mat which is being formed, therefore producing some compression, which is very important for drainage. The outstanding effect of turbulence on drainage has been noticed only very recently (28), and yet is not well understood.

A Dynamic Drainage Jar was adapted for this type of measurements (Figure 2). The conditions set for the experiment were empirical, although based on industrial experience, as indicated by the authors of the technique (28). There are two principal ways of assessing the drainage properties of a pulp, one can measure the amount of water drained during a given period of time and another measures the time necessary for a stock to drain. The latter approach was adopted.

One liter of pulp at 0.2% consistency was introduced in the DDJ with the stirrer set at 100 rpm, which provides an adequate level of turbulence in the suspension. A modified propeller was utilized to avoid adherence of pulp to it, and set at 35 mm from the screen. The suspension was left under agitation for ten seconds to achieve homogeneity while drainage was prevented by an air seal below the 200 mesh screen. At this point special care was taken to avoid air entrainment in the suspension. After this homogenization period the air seal was removed and, simultaneously a vacuum of 10 inches of mercury was applied in the cylinder receiving the drained water. The elapsed time for drainage of 950 ml of white-water (equivalent to reach 4% consistency) was measured with a stopwatch within  $\pm 0.01$  sec. Only distilled water at constant temperature 20°C was used.

A few remarks should be made about the conditions used. The vacuum in the table rolls and foils depends on the speed

of the papermachine. The average suction pressure, applied in table rolls for a machine running at approximately 500 m/min, is very close to the 10 inches of Hg which were applied during the measurements. The final consistency arbitrarily chosen was based on average reported conditions at the end of the forming zone of papermachines (28,228). In order to achieve a good degree of reproducibility in the measurement it was necessary to use two grams of pulp (oven-dry basis) in the jar, so that the drainage time was increased and could be determined manually with a stopwatch. Only three determinations were necessary since the reproducibility was excellent. The height of the propeller was set at a point where the mat being formed would not be disturbed by its motion, as the agitation was primarily intended to induce turbulence only in the pulp suspension being drained. The results are reported in seconds elapsed during draining of the pulp suspensions from 0.2% to 4% consistency.

Wet Web Properties.- After forming on the wire the wet web must be able to withstand the tensions during peeling from the press rolls and pulling across the draws. Information about the ability of a given pulp to respond to those forces can be provided by testing for its load-elongation properties in the wet state. The tests were performed using instruments designed specifically for this use, as described previously (92).

After disintegration for five minutes in the British Standard Disintegrator each pulp suspension was transferred to a wet web former (Figure 3), where (4x10 cm) wet web strips were formed on a 100x96 mesh wire using a rectangular mold. After forming the mold was removed and the wire was driven, carrying the strips across a vacuum box operating at 25 in. Hg, and under a rubber-press roll loaded to 8.76 kN/m. The strips were then removed from the wire by hand and immediately taken for tests. This procedure simulates quite well how the web would come out of the first press of a paper-machine, and experience has indicated that moisture contents for different pulps range from 30 to 36%. Comparisons of different furnishes at similar conditions of specimen preparation rather than at the same moisture content, seems to be gaining recognition as more representative of actual paper-machine operation (198).

Since most properties of wet web vary considerably with basis weight all measurements were made at three different grammages around a target value of  $52.5 \text{ g/m}^2$ . The test results were then interpolated to  $52.5 \text{ g/m}^2$  using linear regression, thereby increasing the accuracy of the technique (92). A minimum of six strips were tested at each basis weight level.

The wet strips taken from the web former were tested first for their caliper, then for their load-elongation

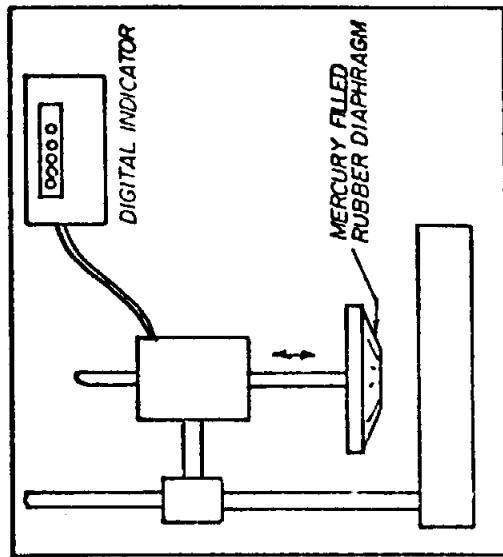


FIGURE 4 WET SHEET CALIPER GAUGE.

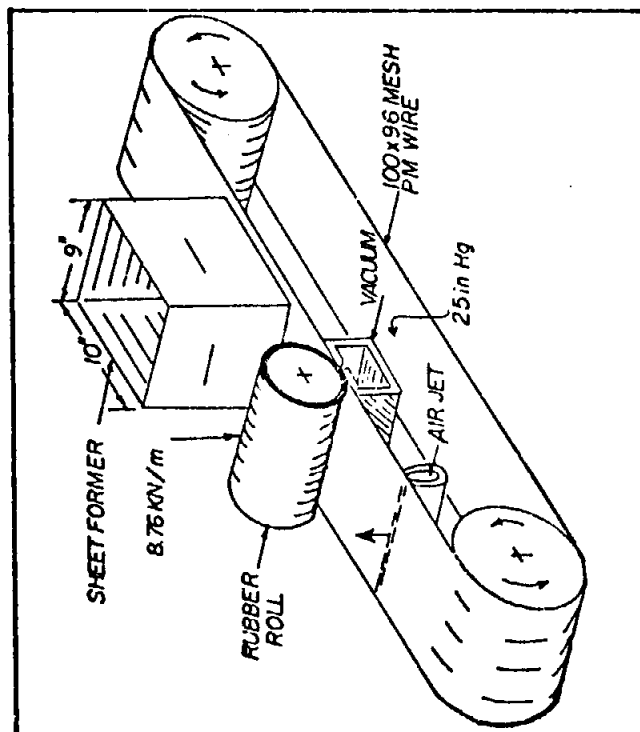


FIGURE 3 WET WEB FORMER.

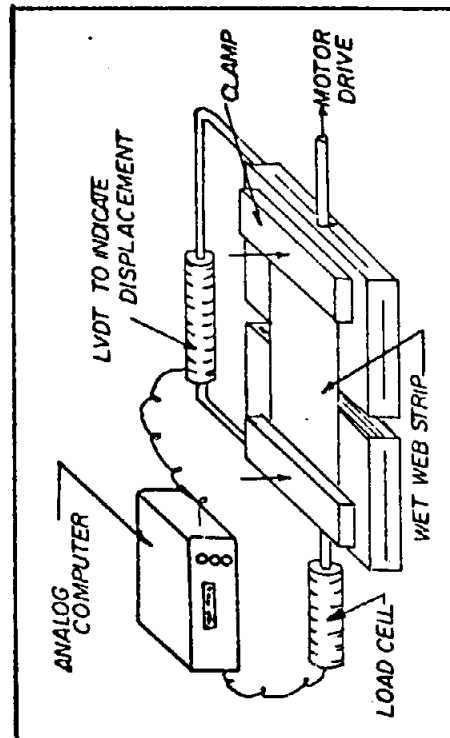


FIGURE 5 TESTER TO MEASURE WET WEB TENSILE AND STRETCH AT MAXIMUM LOAD.

properties, and finally for moisture content. From the web former and through all the tests the wet strips were kept inside closed plastic boxes to prevent changes in moisture content. The caliper of the wet sheet can give an indication of the sheet's resistance to consolidation. This measurement was made using a caliper gauge with a foot made with a mercury-filled rubber diaphragm (Figure 4). A digital indicator provided readings in thousandths of millimeter. Experiments with the instrument have indicated that it actually simulates quite well a "zero-pressure caliper measurement".

Load-elongation properties were measured in a especially designed tester (Figure 5). The wet strips were clamped between jaws and tested horizontally at high loading rate. The jaws were designed to avoid crushing of the sheet when clamped, so that failure should occur only more or less in the middle of the 6.67 cm clamped length. The instrument was calibrated in a way to allow direct readings of breaking load and displacement from the load-elongation curve recorded on chart paper. The wet web tensile strength is expressed in N/m, indicating the maximum load that the web can withstand during the draw of the papermachine per linear width of web. The stretch is reported as a percentage of the initial clamped length.

With the stress-strain curve plus caliper and moisture contents of the sheets interpolated to  $52.5 \text{ g/m}^2$  different

pulps can be compared. There is no comparison on a preset moisture content or thickness, and the results have to be analyzed taking all the factors into consideration.

Specific surface and Specific Volume.- Wet pulp characterization can be greatly improved with the additional information provided by these values. Among the techniques described in the literature the fluid flow methods provide the best estimates of specific surface area. Particularly the water permeability method has been given special attention, due to its applicability to pulps in their natural state, and the simultaneous estimation of their hydrodynamic specific volume.

The Pulmac Permeability Tester was used for water permeability measurements. This instrument is based on the same principle as that developed by Robertson and Mason (107), who modified the Carman-Kozeny equation which relates the permeability, the porosity and the internal surface area of a bed, by taking into account the effective swollen volume of pulp fibers:

$$(KC^2)^{1/3} = \left( \frac{1}{5.55S^2} \right)^{1/3} (1 - vC)$$

where: K = permeability coefficient

C = pad concentration - dry fiber weight per  
unit volume of bed

S = external surface area per gram of fibrous

material

$v$  = effective volume of the swollen  
fibers per gram.

After forming a cylindrical pad of pulp fibers the flow of water through it at approximately constant rate is monitored. Using a permeable piston the pad concentration is varied by compaction, and the hydrostatic head, the flow rate and the dimensions of the pad are recorded, allowing the determination of the permeability coefficient  $K$ . From a series of corresponding values of  $C$  and  $K$  (usually a minimum of seven points) the product  $(KC^2)^{1/3}$  is plotted against  $C$  and a straight line is obtained. The calculation of  $S$  is made from extrapolation of the line to  $C=0$ , and  $v$  is obtained by dividing the slope by the  $C=0$  intercept.

The limitations of the method have been discussed in the literature review and special care was taken regarding experimental restraints such as bed nonuniformity, especially due to flocculation and fines redistribution, and creep effects. To avoid non-uniform bed compaction the initial hydrostatic head was always set at approximately 1.5 cm  $H_2O$ . When working with highly beaten whole pulps very small flow rates had to be used in order to achieve this low initial head, making it mandatory to use a different rotameter (0.3-3.0 ml/min) than normally used with the instrument. Flocculation was avoided by judicious agitation of the pulp suspension as the pad was being formed. This procedure

was followed even when measuring highly beaten pulps, which had extremely long formation times. It was found out that the good degree of reproducibility achieved was due to both the use of very small initial heads and extreme care during pad formation. The rapid and accurate measurement of flow rate, hydrostatic head and pad height provided by the instrument are also believed to have been key factors towards the good results obtained.

The procedure followed was essentially the one suggested in the Pulmac Permeability Tester Manual (199). For each test approximately 6 - 7 grams (dry basis) of moist pulp were taken from the plastic bags where they were stored, as described earlier, resuspended to about 0.2% consistency in distilled water and deaerated for two hours by agitation under vacuum. This slurry was then poured into the filtration tube of the instrument with special care to minimize air entrainment, after which the pad was allowed to form, and tested. An important point in the whole procedure was to avoid air bubbles in all lines of water flow in the instrument. After testing the pulp pad was removed and dried at 105°C for accurate weight determination.

Usually seven points were enough to plot the straight line and calculate the specific volume and specific surface. These calculations were done using a computer program which accounted for the variations in water viscosity and density with the temperature, as recorded during the test. It was

decided to accept only values deriving from straight lines with a minimum correlation coefficient of 0.90. In some cases suspected bad experimental points were tested whether they constituted outliers, according to procedures described later. In case positive these points were rejected and not used in the regression line. The fines-free pulps required only three replications (i.e., data for construction of three straight lines), while the whole pulps, especially the highly beaten ones needed at least five replications for calculation of 95% confidence intervals within 10% of the average values.

Two different rotameters were used in the experiments, the standard in the instrument (1.0-10.0 ml/min.) with fines-free pulps and another one for the whole pulps, allowing measurement of very small flow rates (0.3-3.0 ml/min.). Both rotameters were calibrated under the same conditions, at 25°C and using the same deaerated distilled water as that utilized in the experiments. The specific surface and specific volume are reported in  $\text{cm}^2/\text{g}$  and  $\text{cm}^3/\text{g}$  respectively, on an oven-dry basis.

#### Testing of Standard Handsheets

The usual evaluation of development of paper properties during beating is the final step in the assessment of a pulp's potential for papermaking. When combined with the characteristics of wet pulps the measurements of paper properties provide valuable information about the overall range

of utilization of a given raw material.

For these tests handsheets of  $60 \text{ g/m}^2$  basis weight were prepared according to TAPPI standard T 205-os 71, although 200 mesh screens were used in the sheet mold, replacing the standard 150 mesh screens. The handsheets were conditioned and tested under constant 50% relative humidity and  $23^\circ\text{C}$ , according to TAPPI Standard T 402 os-70. Preparation of samples for physical tests was performed following Standard T 220 os-71. The following tests and respective procedures were performed on all fines-free and whole pulps:

Basis Weight - TAPPI Standards T 220 os-71 and T412 su-69.

Thickness/Apparent density - TAPPI Standards T 220 os-71 and T 411 os-76, with results reported in  $\text{kg/m}^3$ .

Bursting Strength - TAPPI Standard T 403 os-76. Five sheets from each sample were tested, with measurements made on both sides and averaged. The results are reported as burst index, in  $\text{kPa m}^2/\text{g}$ .

Tensile Strength - TAPPI Standard T 494 os-70, using an Instron Universal Testing Instrument at a constant  $1\text{cm/min}$  rate of elongation and clamped length of 10 cm. Ten strips of paper were tested from each sample. The results are reported as tensile index, in  $\text{N m/g}$ .

Internal Tearing Resistance - TAPPI Standard T 414 ts-65, using the Elmendorf tearing strength tester and five plies torn at each time. At least six measurements were made on each sample. The results are reported as tear index, in  $\text{N m}^2/\text{kg}$ .

Light Scattering Coefficient - the measurements were performed using a Zeiss Elrepho Light Reflectometer under green light (557nm). After determination of the reflectance factor  $R_0$  of each of five sheets of paper with a black backing, the intrinsic luminous reflectance factor  $R_\infty$  was measured for each of the same five sheets, after backing the test pieces with others of the same kind to form an opaque pad. Using the average values of  $R_0$  and  $R_\infty$  the scattering power (SW) was calculated using Kubelka-Munk equations, from which the light scattering coefficient was determined after division by the basis weight. The results are reported in  $\text{m}^2/\text{kg}$ .

Air Resistance - the measurements were performed using the Gurley apparatus, following ISO International Standard 3687. The instrument consists of a cylinder closed at one end upmost in another vertical cylinder which is partly filled with oil. A mean air pressure of 1.21 kPa due to the weight of the inner, floating cylinder is applied to a circular area of clamped paper through a central tube, forcing the passage of air through the test piece. The total elapsed time in seconds during the flow of 100 ml of air, as indi-

cated in the graduated cylinder was measured with a stopwatch to the nearest 0.01 second. For the unbeaten and lightly beaten samples the time was recorded for the flow of 200 ml to increase the accuracy of measurement. Five sheets from each sample were tested, with readings at two different points at both sides. The results are reported as Gurley air resistance, in seconds/100 ml air.

### Statistical Analyses

Standard procedures for calculation of averages and standard deviations were used. The confidence intervals for the sample means were calculated using the appropriate degrees of freedom and tabulated percentiles of the Student's *t* distribution (166).

For comparisons of sample means the one-way analysis of variance was used, with significance level  $\alpha \leq 0.05$ . The Statistics Package of Hewlett-Packard 41-C pocket calculator was used for this analysis. The Bartlett's test of homogeneity of variances (205) was used prior to each analysis, and in no case heterogeneity was found.

Linear models were employed for all regression equations, most usually for linear regression, using the Statistics Package described above, but also multiple linear regressions were performed when necessary, using the SPSS - Statistics Package for the Social Sciences - and an IBM/370

computer. Special attention was given to outliers during analysis of permeability data. As a suspected bad experimental point was found a new regression line was fitted through the remaining data points. The point not used in the new fitting was then tested for the probability that in the total number of observations its deviation from the fitted line could be obtained by chance. When this probability was sufficiently small the outlier was discarded.

## RESULTS AND DISCUSSION

### Wood and Pulp Fiber Characteristics

The morphological properties evaluated were wood fiber transverse dimensions, including the thicknesses of cell wall layers ( $P + S_1$ ,  $S_2$  and  $S_3$ ), and in pulp the microfibrillar orientations in  $S_1$  and  $S_2$  layers, weighed average fiber length, fiber coarseness, fiber cross sectional area, perimeter and wall thickness. For additional information about other pulp components the dimensions of the vessels in the three hardwoods were also measured. Chemical composition of the pulps was then determined, followed by viscosity, wet zero span strength, pH of pulp suspensions and brightness measurements.

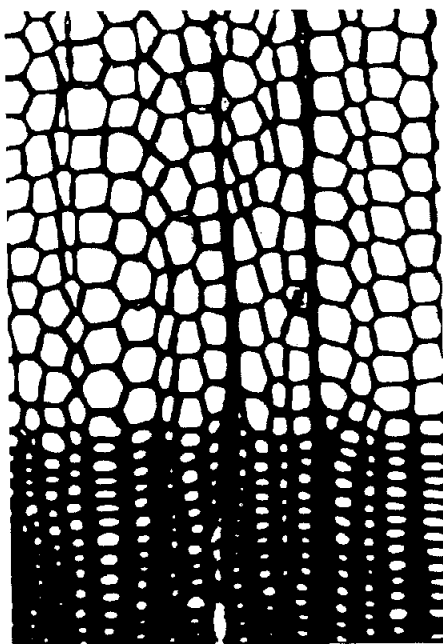
### Wood Fiber Transverse Dimensions

Lumen and Fiber Diameter - The results reported in Table I, and seen in figures 6 and 7 demonstrate the variability within each species studied, as well as the differences among them. The use of the image analyzer to measure the lumen cross-sectional areas of over one thousand fibers at representative positions across the grain was found to be very effective and accurate, allowing immediate statistical treatment and representative results. The lumen diameters were calculated assuming a circular cross section of the fibers, therefore avoiding the difficulty of measuring different diameters in each fiber, as they have no

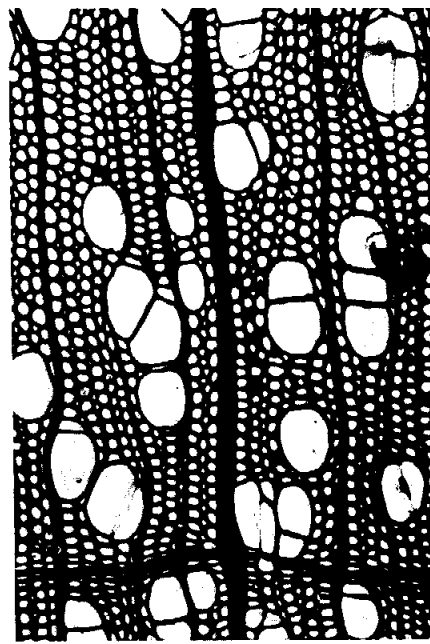
TABLE I. CHARACTERISTICS OF WOOD FIBERS CROSS SECTIONS

	Lumen Diameter, $\mu\text{m}$	Thickness of cell wall $S_1 + P$	$S_2$	$S_3$	Wall Thickness, $\mu\text{m}$	Fiber Diameter, $\mu\text{m}$	Luce's Shape Factor (142)
Southern pine	early wood	$38.0 \pm 3.4^a$	-	-	$3.50 \pm 0.4^a$	$45.0 \pm 3.5^a$	0.17
	late wood	$18.3 \pm 3.3^a$	-	-	$8.1 \pm 0.8^a$	$34.5 \pm 3.4^a$	0.56
Silver birch		$12.5 \pm 1.7$	$0.34 \pm 0.03$	$2.61 \pm 0.16$	$0.08 \pm 0.01$	$18.56 \pm 1.60$	0.38
<u>E. grandis</u>		$9.1 \pm 1.4$	$0.24 \pm 0.01$	$2.17 \pm 0.13$	$0.09 \pm 0.01$	$14.1 \pm 1.4$	0.41
<u>E. rostrata</u>		$8.2 \pm 1.3$	$0.27 \pm 0.02$	$1.80 \pm 0.05$	$0.10 \pm 0.02$	$12.54 \pm 1.3$	0.40

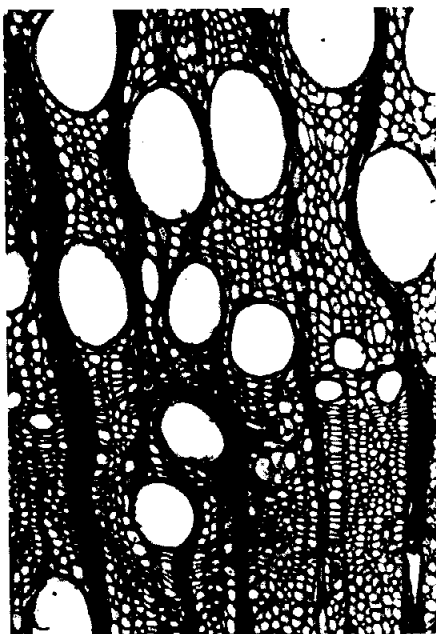
a - based on published results (129)



a. Slash pine



b. Silver birch

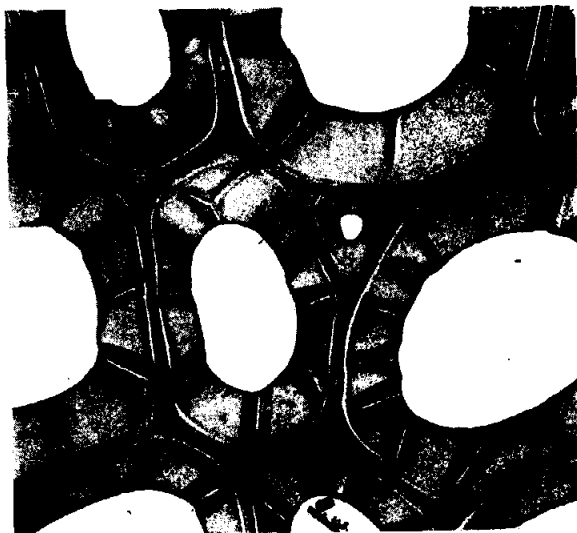


c. Eucalyptus rostrata



d. Eucalyptus grandis

Figure 6 - Light micrographs of wood cross sections at the same magnification.



a. Silver birch



b. Eucalyptus rostrata



c. Eucalyptus grandis

Figure 7 - Transmission Electron Micrographs of wood cross sections at the same magnification [ $3.0 \mu\text{m}$ ].

defined shape.

The measurements of the thickness of the different cell wall layers ( $P + S_1, S_2, S_3$ ) were carried out directly on transmission electron micrographs, of the same type as the ones shown in figure 7, although for measurements enlargements to 6240X were used. These pictures were taken at representative positions on wood cross sections. They provided accurate values, which coupled with the calculated lumen diameters gave an overall picture of the average cross-sectional dimensions of the fibers. In general the data obtained were of lower magnitude than corresponding values reported in the literature (3,11,13,17,22,45,47,48,52,54,74,83,88,93,102,116,162,163,218,219). One reason for this discrepancy might be found in the natural variation among sampled trees and the influence of different environmental factors on the properties of the woods of the same species. Another possibility is the higher accuracy of the methodology employed in this study.

In the usual methods for fiber width and wall thickness measurements a light microscope with attached eyepiece and micrometer is used for direct measurement on wood cross-sections or on holocellulose fibers. Considering the variability of shape and size of transverse dimensions across the grain, the experimenter may be biased towards the measurement of particular diameters, perhaps the largest ones. The additional limited resolution of the light

microscope might make it even more difficult to arrive at representative results for a tree. Thus, in this study the measurement of the lumen cross-sectional area was considered more accurate than measuring lumen diameter. It is interesting to notice that results on the transverse anatomical characteristics of South African E. grandis published recently were evaluated by measurement of lumen areas (54). The values from this source are much closer to ours than other measurements on the same species using a light microscope with attached eyepiece and micrometer (218, 219).

Similar precautions may not be necessary when comparing fibers with very different dimensions, such as fibers from earlywood and latewood in conifers, or when comparing hardwood and softwood fibers. However, when comparing different species, especially among hardwoods, the use of techniques with limited accuracy might sometimes lead to conclusions not quite justified.

Some published studies on the anatomical characteristics of three eucalypt species being used commercially (E. grandis, E. rostrata and E. globulus) have concluded that they all had the same wall thickness as silver birch, i.e. 3.0 microns (52, 74, 116). Therefore, considered as having the same wall thickness as birch, but with smaller width, the eucalypt fibers have been collectively treated as thick walled, collapsing less

easily and yielding bulky papers of high opacity and low strength. These conclusions are based on the parameters commonly used to assess fiber flexibility and collapsability, such as the Runkel ratio, Mulsteph ratio, lumen diameter-to-fiber diameter ratio, ratio of the area of cell wall to total cross sectional area, or the more recently proposed shape factor (142). The latter is defined as an estimate of the force required to collapse a fiber, and is also dependent only on its geometry:

$$F = \frac{(FD)^2 - (LD)^2}{(FD)^2 + (LD)^2} \quad \text{where FD = Fiber Diameter} \\ \text{LD = Lumen Diameter.}$$

The results in Table I indicate that E. grandis, E. rostrata and Silver birch have very different weighed average cell wall thickness (2.50, 2.17 and 3.03  $\mu\text{m}$  respectively) and that the calculated shape factors are actually smaller for birch fibers, but not to an extent that alone could explain their much higher conformability, as observed in the higher apparent density of handsheets. Thus, unlike previous generalized opinions, the differences between the flexibility and conformability of birch and eucalypt fibers might have other reasons in addition to geometrical factors. Other factors, mostly related to ultrastructural organization of the cell wall, may come into play, and they are discussed later.

The values for transverse dimensions of Southern

pine wood fibers were taken from one of the best surveys of published anatomical data for these species (129). Since the commercial southern pine pulp contained unknown proportions of loblolly, short leaf and long leaf pines an average was taken of the surveyed values for the transverse dimensions of tracheids of these three species. Since they are of much larger magnitude than those of the hardwood fibers under study a direct comparison can be made even if the methods of measurement are not the same. As seen in Tables I and III, and figures 6, 18 and 19 the pine fibers are much larger than the fibers of the three hardwoods. The shape factors were also calculated and are the first indication of the well-known large differences between earlywood and latewood.

Cell Wall Layers. As observed in Table I the  $S_1$  and  $S_2$  layers of birch fibers are thicker than those of both eucalypt fibers, the  $S_3$  layers being of about the same magnitude. The proportions of the different layers to the total cell wall area are also different, as seen in Table II. The fibers of E. grandis showed a smaller proportion of  $S_1$  layer than E. rostrata and birch, while the proportions of  $S_2$  layer in birch and E. grandis were pretty much the same, and significantly larger than in E. rostrata. It can also be noticed that the eucalypts

studied show a tendency to have proportionally thicker  $S_3$  layers. With similar measurements not being found in the pertinent literature a confirmation of these results was not possible. Absolute values of the thickness of cell wall layers for the three pine species composing the pulp were not found for comparison. Their relative proportions to the wall area are also shown in Table II, and are based on published values (82, 115, 150).

TABLE II. PERCENTAGE OF AREAS OF CELL WALL LAYERS

		$S_1$	$S_2$	$S_3$
Southern pine	Earlywood <sup>a</sup>	21	74	5
	Latewood <sup>a</sup>	12	85	3
	Composite <sup>b</sup>	16	80	4
Silver birch	-	13	85	2
<u>E. grandis</u>		11	86	3
<u>E. rostrata</u>		14	82	4

a - based on published results, (82,115,150).

b - calculated, considering 60% latewood, (117).

The percentage of  $S_2$  layer in latewood is substantially larger than in earlywood while the opposite

is observed for the  $S_1$  layer. It is interesting to notice that the proportions of the cell wall layers in the thickwalled pine latewood fibers are very close to the ones observed for the three hardwood fibers.

The relative proportions of the cell wall layers of a hypothetically representative pine "composite" fiber were calculated, assuming to have 60% latewood fibers in the pulp, as indicated in the literature (117). This procedure was necessary for later calculations of Young's modulus of an average pine pulp fiber. Similar calculations of values representative of the overall commercial pulp were also based on characteristics determined in earlywood and latewood fibers, and considering 60% of the latter by weight.

#### Characteristics of the Commercial Pulps

Weighed Average Fiber Length and Pulp Coarseness. The results in Table III indicate that all three hardwoods show fiber lengths close to 1 millimeter, both eucalypts being shorter than birch, and E. rostrata significantly shorter than E. grandis. These values appear to be characteristic for the three species (45,116,162,190). When these figures are combined with the coarseness of pulp fibers one of the most interesting characteristics of eucalypt pulps becomes evident: the short and thin fibers also have a small weight per unit length, leading to

a large number of fibers per gram, calculated according to the formula:

$$\text{Number of fibers per gram} = \frac{10^8}{\text{weighed av. fiber length (mm)} \times \text{pulp coarseness (mg/100 m)}}$$

These calculations show that E. rostrata has over twice the number of fibers per gram as birch, and E. grandis about 62 percent more than birch. Besides having a direct influence on paper formation (26), this characteristic is believed to have a significant importance on the pulp's response to the beating treatment, as will be discussed later.

TABLE III. AVERAGE FIBER LENGTH, FIBER COARSENESS AND NUMBER OF FIBERS PER GRAM OF THE COMMERCIAL PULPS.

Pulp	Weighed average fiber length, mm	Fiber Coarseness, mg/100 m	Number of fibers per gram of pulp
Southern pine	2.91	29.2	$1.2 \times 10^6$
Silver birch	1.10	10.0	$9.1 \times 10^6$
<u>E. grandis</u>	0.97	7.0	$14.7 \times 10^6$
<u>E. rostrata</u>	0.87	6.2	$18.5 \times 10^6$

As also seen in Table III the pine pulp fibers are much coarser and longer than the hardwood fibers. As a consequence the calculated number of fibers per gram is

one order of magnitude lower than for the hardwoods. These values for Southern pine were found to be in general agreement with those reported elsewhere (18, 26, 104, 116, 241).

#### Orientation of Microfibrils in Cell Wall Layers.

The technique of phase-contrast microscopy with near-ultraviolet illumination (40) allowed direct observation of the cell wall striations on fibers of the commercial pulps, with a high precision of measurement of  $S_2$  angles. This method also revealed interesting features of the fibers examined, such as right-handed helices (Z form) in  $S_2$  layers of fibers from all three species grown in the Northern Hemisphere - E. rostrata (Spain), silver birch (Sweden), Southern pine (United States), and left-handed helices (S form) on fibers of E. grandis, grown in Brazil. Differences in the sense of the helices for different species growing at different sites are not much reported, and it is therefore not possible at this point to determine whether these observations are due to simple coincidence or they follow a more general pattern, under the possible influence of coriolis forces. More conclusive evidence would probably be provided by a systematic study of a number of species grown in the two hemispheres, especially by comparison of the orientations for the same species, when grown both South

and North of the equator.

The measurements of the fibrillar angle of  $S_2$  layer were carried out on enlarged projections of the negatives of the photomicrographs, some of which are shown in figure 8. The microfibrillar striations can be easily seen with this technique, and at least 5 measurements on each of 20 fibers from the four commercial pulps gave very reproducible results, as shown in Table IV. Particularly interesting, as claimed by its authors, is the fact that the technique overcomes many difficulties encountered in other methods, and one of its main applicabilities is the measurement on thickwalled cells with small fibrillar angles. With direct observation it is also possible to avoid the interference of  $S_1$  or  $S_3$  layers, by simple adjustment of the focus in the photomicroscope.

The results of these determinations revealed characteristics previously unexplored in some of these fibers, i.e. the very small fibrillar angles in  $S_2$  layers of both eucalypts. The microfibrillar orientations in E. grandis and E. rostrata were also found to be much closer to the fiber axis than that of birch (Table IV). The observed differences between either of the two eucalypts and birch were found statistically significant in the 99% confidence level, the same being observed between each of the hardwoods and the Southern pine. The difference between



4.0  $\mu$ m

a. Southern pine earlywood  
 $S_2$  fibrillar angle -  $22^\circ$



3.0  $\mu$ m

b. Silver birch,  
 $S_2$  fibrillar angle -  $16^\circ$



3.0  $\mu$ m

c. Eucalyptus rostrata,  
 $S_2$  fibrillar angle -  $11^\circ$



3.0  $\mu$ m

d. Eucalyptus grandis,  
 $S_2$  fibrillar angle -  $12^\circ$

Figure 8 - Near-ultraviolet photomicrographs of pulp fibers showing microfibrillar striations in  $S_2$ -layer

TABLE IV. AVERAGE MICROFIBRILLAR ANGLES IN  $S_1$  and  $S_2$  LAYERS OF PULP FIBERS

Pulp	$S_1$ -Angle			$S_2$ -Angle		
	No. of fibers	Range, degrees	95% confidence interval, degrees	No. of fibers	Range, degrees	95% confidence interval, degrees
Southern pine	Early wood	20	69-90			
			73 $\pm$ 11	15	20.5-49.3	25.5 $\pm$ 4.4
	Late wood	20	32-62	15	4.7-13.4	8.3 $\pm$ 2.3
	Composite (a)	40	-	30	-	15.2 $\pm$ 3.1
Silver birch	25	55-80	61 $\pm$ 9	20	14.8-25.2	18.5 $\pm$ 3.5
<u>Eucalyptus</u> <u>grandis</u>	25	40-71	52 $\pm$ 8	20	5.5-15.7	11.5 $\pm$ 2.4
<u>Eucalyptus</u> <u>rostrata</u>	25	40-68	50 $\pm$ 9	20	6.0-18.1	10.3 $\pm$ 3.6

(a) Calculated considering 60% late wood (117).

E. grandis and E. rostrata, however, was found to be not significant.

The similarity in fibrillar angles of  $S_2$  layer of these two eucalypts, which are different in most other properties, and grown at different rates in opposite hemispheres might be due to more than simple coincidence. Some common trend may probably also be detected in other species of this genus, and this could be related to some physiological behavior during growth. Similar measurements for silver birch, E. rostrata and E. grandis pulp fibers were not found in the available literature. The results for southern pine earlywood tracheids were of the same magnitude as previously reported by other methods, while the comparison of latewood values were rather difficult due to the considerable scatter found in a survey of published measurements (129). Average angles for a representative fiber of the pine pulp were also calculated, using the same procedure described earlier.

The angles formed by microfibrillar strands in  $S_1$  layer were more difficult to determine than  $S_2$ -angles, using the same method, partly due to the difficulties with resolution of this layer in the light microscope and partly because of the inherent larger variability of orientation within  $S_1$ -layer (55,89,178). As a consequence more measurements were needed, and these included further examination of scanning electron micrographs of

freeze-dried surfaces of very lightly beaten fibers. In this way good agreement was reached with the values obtained from near ultraviolet light micrographs.

A statistical analysis again indicated a significant difference between microfibrillar orientations in  $S_1$  among the four types of pulp, except between the two species of eucalypts under study. It should be stressed that these measurements are average values for the complex arrangement of different lamellae in  $S_1$ , in some cases observed in gradual stepwise transition to the microfibrillar orientation in  $S_2$ -layer (55,89,178,232). In the present study some of these individual lamellae could be observed only in birch and pine fibers, at different positions of the focus in the light microscope. For the fibers of both eucalypts only the two crossed lamellae were observed, at fairly consistent angles to the fiber axis. An external  $S_1$  layer with microfibrils perpendicular to the fiber axis as proposed earlier (55) was observed only on pine fibers.

The average angles for  $S_1$  layer are within the range of values reported by others (63,89,178). Accordingly, the angles for hardwood fibers are consistently smaller than those of pine earlywood, in agreement with earlier suggestions (63). It is also interesting to notice that smaller average  $S_1$ -angles apparently occur in fibers with small

$S_2$ -angles, which might be considered as a supportive evidence for the hypothesis that the  $S_1$ -layer is a transitional zone between the primary wall and the  $S_2$ -layer (63). The presence of transition layers between  $S_1$  and  $S_2$  in some cases (55,89) also seems to agree with the observed trend of a close relationship between the orientations in the two layers.

Chemical Composition of the Pulps. Although of primary importance when comparing pulps of different species, morphological aspects cannot provide all the information necessary for the understanding of a pulp's behavior during beating. The chemical composition of pulp fibers may be considered a priori as important as morphology, and combination of the two should be complementary.

Chemical analysis were carried out on the four commercial bleached kraft pulps, revealing only the presence of carbohydrates, with zero lignin content for all samples. The results of sugar analysis, ash content and dichloromethane extractables are shown in Table V.

Particularly interesting is the high content of xylan in silver birch. This and other species of this genus (e.g. Betula papyrifera Marsh. Betula pubescens Ehrh. (221)) are known to have abnormally high contents of this polysaccharide (6,190,221), varying from 34 to 37

TABLE V. CHEMICAL COMPOSITION OF THE COMMERCIAL PULPS

	Sugar Analysis, % weight <sup>a</sup>					DCM Extractables, %	Ash, %
	Mannan	Arabinan	Galactan	Xylan	Glucan		
Southern pine	7.6	0.5	0.4	8.5	83.0	0.00	0.16
Silver birch	0.2	0.0	0.0	25.7	74.1	0.33	0.49
<u>E. grandis</u>	0.0	0.0	0.0	15.4	84.6	0.08	0.05
<u>E. rostrata</u>	0.0	0.0	0.0	19.5	80.5	0.24	0.18

a - Lignin content was zero for all samples.

percent on wood. Therefore it is not unusual to produce a fully bleached kraft pulp from birches with 24 to 27 percent xylan content (6,190).

Eucalypts are collectively poorer in hemicelluloses and richer in cellulose, lignin and extractives (48,102,162) than birch (221). As a result the xylan contents in fully bleached eucalypt kraft pulps usually do not exceed 20 percent, and are commonly about 16-18 percent (6,85,190). The results on Table V therefore indicate that E. rostrata is in the upper limit among eucalypt pulps, while E. grandis shows a very low xylan content. The reasons for these differences are more likely to be due to different pulping conditions than the original compositions of the corresponding woods, which are very similar in xylan content. As described earlier E. grandis was pulped with bark, and due to the high extractives content in it sometimes higher than usual alkali charges are employed. The resultant low content of dichloromethane (DCM) extractables is an indication that this has probably happened. For E. rostrata, however, no bark was involved in the pulping, but the usually high extractives content in the wood of this species and the still high percentage of dichloromethane extractables in the commercial pulps seem to indicate that the pulping conditions were milder, preserving more of the pentosans.

For the southern pines much evidence seems to support the general conclusion that they all have essentially the same chemical composition, with only small variations in the amounts of polysaccharides, lignin and resin (129). The pulp composition in Table V can be considered typical, and is close to values reported for other pine species (190). The xylan content is very low when compared with the hardwoods, as should be expected since the predominant hemicelluloses in softwoods are the galactoglucomannans (129,221). These polymers are believed to be located deep in the fiber structure. As much indirect evidence seems to suggest they are in close association with the microfibrils (82,129,165). This aspect is also supported by the reported higher contents of both glucose and mannan in latewood holocellulose (136) and kraft pulp fibers (82) than in earlywood, which, according to earlier discussions, has a lower degree of structural organization, leading to less accessibility of pulping liquors. On the other hand, for most wood fibers xylan polymers seem to be dispersed through the cell wall, more concentrated in the primary wall,  $S_1$  and outer  $S_2$  layers, as indicated for the wood fibers of Scots pine, Norway spruce and silver birch (159). Such a distribution appears to hold for kraft pulp fibers, with even higher concentrations of xylan in the outer layers of the cell wall (103,126,141). This would be expected from the suggested

preferential readsorption of xylans during alkaline pulping (34,242).

Being more hydrophilic than glucomannan and more readily accessible at the fiber surface and throughout the cell wall, the xylan-based hemicelluloses are generally accepted as being responsible for controlling swelling and plasticization (71,75,76) and the mechanical properties of fibers differing in hemicellulose content (137,206). In this context it can be expected from the results in Table V, that the pine pulp will be more difficult to respond to the plasticization effect of hemicelluloses due to its low xylan content. For a more complete understanding of the effects of hemicelluloses in the beating behavior of different pulps the detailed distribution of chemical components in the fiber's internal structure should be known. This type of analysis would constitute another elaborate work in itself, and is therefore beyond the scope of this study.

The analysis of ash content was performed to see if any abnormally high level could be related with crystals within pulp components. An spectroscopic determination later revealed that the ashes in all pulps were mostly composed of calcium salts, probably built up in the respective mills water systems, and that could be easily washed out.

Cross Sectional Dimensions of Pulp Fibers. After measuring the cross-sectional area of pulp fibers and their perimeters the respective fiber diameters and cell wall thickness were calculated, assuming a circular cross section for the fibers. When compared with the measurements for wood fibers in Table I, the results listed in Table VI indicate that indeed some shrinkage has occurred in the fibers as a result of pulping and bleaching, but not to the extent reported recently by Scallan and Green (196). One of the reasons for this maybe the fact that for the measurements of cross sections of pulp fibers in this study the fibers were immersed in the embedding medium after the pulps had been reslushed. As a consequence some swelling might have occurred during the slushing operation, and the results in Table VI should be representative of the pulp fibers in the wet state.

Cell Wall Density. By combining the values of fiber coarseness and measured cross-sectional area of the cell wall, the density of the fiber wall substance (or "packing density") can be calculated, according to the formula:

$$\text{Cell wall density (g/cm}^3\text{)} = \frac{10 \times \text{coarseness (mg/100m)}}{\text{cross sectional area } (\mu\text{m)}^2}$$

The results in Table VI show that each pulp has its

TABLE VI. WEIGHED AVERAGE CROSS-SECTIONAL DIMENSIONS, CELL WALL DENSITY, MOMENT OF INERTIA,  
AXIAL YOUNG'S MODULUS AND BENDING STIFFNESS OF PULP FIBERS

	Cell wall cross- sectional area, $\mu\text{m}^2$	Fiber perimeter, $\mu\text{m}$	Fiber diameter, $\mu\text{m}$	Fiber wall thickness, $\mu\text{m}$	Luce's shape factor	Cell wall density, $\text{g}/\text{cm}^3$	Moment of inertia, $(\mu\text{m})^4$	Axial Young's modulus, $\text{GPa}$	Fiber bending stiffness, $\text{N m}^2$
Southern pine	355.3	103.4	32.9	3.90	0.26	0.822	1753	20.7	$36.3 \times 10^{-12}$
Silver birch :	136.7	56.2	17.9	2.90	0.37	0.732	363	9.0	$3.3 \times 10^{-12}$
<u>E. grandis</u>	79.9	39.9	12.7	2.45	0.45	0.887	147	36.3	$5.4 \times 10^{-12}$
<u>E. rostrata</u>	66.7	38.3	12.2	2.10	0.40	0.930	92	47.2	$4.4 \times 10^{-12}$

own characteristic value, which could be very much related to the microfibrillar orientation in the cell wall of the respective fibers. It can be noticed that birch fibers with  $S_2$ -angles of  $18.5^\circ$  has the lowest packing density ( $0.732 \text{ g/cm}^3$ ), followed by the average southern pine fibers with  $0.822 \text{ g/cm}^3$  in the cell wall and  $15.2^\circ$  in the  $S_2$  layer, and E. grandis and E. rostrata, respectively with  $0.887 \text{ g/cm}^3 - 11.5^\circ$  and  $0.930 \text{ g/cm}^3 - 10.3^\circ$ .

It is thus possible that as the fiber walls are formed with steeper microfibrillar helices a tighter arrangement between the cellulose chains could occur, resulting also in smaller crystallite widths than in the case of fibers displaying larger fibrillar angles. Therefore, fibers with apparently very different morphological characteristics might have ultrastructural similarities not previously recognized, and these could very well reflect in their papermaking properties.

Although the subject of cell wall density has been debated for some time due to the different approaches for its measurement (119) it was felt that the results of this study could be treated with confidence. An important reason for this was the fact that the measurements of cross sectional area and perimeter on wet pulp fibers allowed the calculation of fiber diameter and cell wall thickness which were very close to the ones determined in wood cross-sections, the latter through the use of more

sophisticated techniques, such as transmission electron microscopy and image analysis.

Fiber Stiffness. As also used for the comparison among wood fibers, the shape factor proposed by Luce (142) is included in Table VI. It can be noticed that the values are very close to the ones calculated for the wood fibers, and again it was evident that the different flexibility and conformability of birch and eucalypt fibers could not be explained solely by geometrical factors related to the cross sectional dimensions of the fibers.

When searching for a method to give a quantitative indication of the flexibility of the pulp fibers it was decided to calculate the fiber stiffness following the ideas of Leopold and Iyer (138). According to this report the flexibility of a pulp fiber could be inferred from the inverse of its bending stiffness, calculated by assuming that when the wet sheet of paper is formed the fibers assume an approximately rectangular cross-sectional shape. This assumption has also been recently shown to be more valid for pulp fibers in the wet sheet than the possible alternative consideration of a circular cross-section (19). When trying to conform to other fibers in the sheet it can be assumed that each fiber would be loaded in the direction parallel to its smallest cross sectional dimension, as indicated in figure A-1 (Appendix A). The resistance to conformability would then be expressed by

its bending stiffness, according to the following equation:

$$S = EI \quad , \quad \text{where } S = \text{bending stiffness}$$

$$E = \text{Young's modulus in the axial direction of the fiber}^1$$

$$I = \text{moment of inertia}$$

$$(\text{axis of moment through center of the rectangle}).$$

The moment of inertia for a typical fiber from each pulp was calculated from the cross-sectional dimensions, as described in Appendix A, but E values were not available from direct measurements on the fibers. It was thought that the ultrastructural data obtained from wood fibers such as proportions of cell wall layers, and the microfibrillar angles and chemical properties of pulp fibers would provide an excellent opportunity for calculation of the Young's modulus, utilizing a two-dimensional mechanical analysis of the pulp fiber cell wall, under axial load without shear restraint,<sup>1</sup> as used by Mark and Gillis (150). The calculations were performed using a computer program, with the data generated in this study and some additional assumptions, as described in

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<sup>1</sup>For the purpose of estimating the resistance to conformability of pulp fibers the E used should be obtained from fiber bending analysis. In the present work it was assumed that this value is equal to the calculated Young's modulus using a mechanical analysis of the fiber under tensile load.

The results in Table VI indicate that the birch fibers have a much larger moment of inertia than both eucalypts, as expected by comparison of their cross sectional dimensions. On the other hand the axial Young's moduli of the two eucalypts are at least four times as big as that of birch, as a result of mostly the larger microfibrillar angles in the latter, with a smaller influence of the hemicellulose content (150). By combining E and I, the resultant fiber stiffness values show that birch should have more flexible fibers than the two eucalypts, while E. grandis is expected to have stiffer fibers than the other two hardwoods.

The larger stiffness obtained for southern pine fibers when compared to the hardwoods is very interesting to analyze. Due to the much larger transverse dimensions of these fibers when compared to the hardwoods, the moment of inertia of pine is almost five times as big as the one calculated for birch, and at least ten times larger than the estimated values for the two eucalypts. As a consequence, although for pine the Young's modulus (which is a material property) is akin to values for the eucalypts because of their similar ultrastructural organization, the calculated stiffness of pine fibers ( $36.3 \times 10^{-12} \text{ N. m}^2$ ) relates to the values for the hardwoods ( $3.3 \times 10^{-12}$ ,  $5.4 \times 10^{-12}$ ,  $4.4 \times 10^{-12} \text{ N. m}^2$  for birch, E. grandis and E. rostrata respectively) by at least a factor of seven. It should

then be expected that the pine fibers would be, on the average, much more difficult to conform to each other when the paper sheet is formed than any of the other three hardwoods, which, in turn, also show significant differences among themselves.

The calculation of stiffness values totally from morphological and ultrastructural data has not been reported so far in the literature reviewed, and neither has a comparison between largely different fibers been made on these grounds. It is believed that the methodology and results reported here may help to better comprehend the behavior of pulp fibers in the various stages of papermaking and in the final product. Although some assumptions were made during the calculations described above, it is highly probable that the estimated values of the axial Young's modulus are very close to the actual ones, since similar analyses of the mechanical behavior of the cell wall have allowed good agreement between calculated and experimentally determined fiber strength (58).

Characteristics of Vessels. Vessel segments are also important components of hardwoods, in many cases contributing significantly to the properties of the pulps. Some of the characteristics of vessel segments from silver birch, E. grandis and E. rostrata are listed in Table VII, to illustrate their most common features.

TABLE VII. CHARACTERISTICS OF VESSEL ELEMENTS

Vessel Diameter, $\mu\text{m}$					
Range	95% Confidence interval for the mean	No. vessels/ $\text{mm}^2$	Vessel wall thickness, $\mu\text{m}$	Vessel length, of vessels by volume	Percentage of vessels by weight
Silver birch					
31-110	$72 \pm 11$	$28 \pm 4$	$2.6 \pm 0.3$	$0.80 \pm 0.15$	$\begin{matrix} (6) & (6) \\ 25 & 9 \end{matrix}$
Eucalyptus <u>grandis</u>					
70-230	$153 \pm 34$	$7 \pm 2$	$3.5 \pm 0.5$	$0.41 \pm 0.04$	$\begin{matrix} (218) \\ 15 \end{matrix}$
Eucalyptus <u>rostrata</u>					
71-154	$101 \pm 21$	$16 \pm 3$	$3.4 \pm 0.3$	$0.40 \pm 0.05$	$\begin{matrix} (47) \\ 17 \end{matrix}$

Vessel diameters were calculated from measurements of areas of lumen cross sections using an image analyzer, and assuming circular cross sections. As can be noticed in Table VII and Figure 6 there is a large variability of sizes across the grain for each species. The variability is even greater when comparing vessels from the three species, with the eucalypts showing much larger diameters than birch, although occurring in smaller numbers than in the latter. Particularly interesting are the extremely large diameters of E. grandis vessels, especially those occurring in the sapwood. They are much wider than vessels from E. rostrata, not only due to their inherent genetic differences, but probably also to the extremely higher rate of growth of E. grandis in Brazil.

The thicknesses of vessel elements were determined directly on enlargements of the micrographs shown in Figure 6 and additional ones taken across the grain. It is apparent that the two eucalypts have vessels with basically the same wall thickness, while the walls of birch vessels are significantly thinner. When compared to their overall diameter it is easy to realize that in all cases the walls occupy only a very small fraction of the total corresponding cross-sectional areas, and thus their relative importance can be considered secondary.

The average lengths of vessel elements are also important features, especially when compared to the corresponding diameters. Birch vessels are about twice as long as those of both eucalypts, and their length-to-diameter ratios are approximately 11.1 for birch, 4.0 for E. rostrata and 2.7 for E. grandis. Thus, birch vessels segments can be considered as having shapes close to fibers, while both eucalypts have barrel-shaped vessel segments, which are more difficult to form bonds with fibers, and can therefore lead to the well-known high picking tendency of eucalypt-based papers (35,163). This is an important aspect of the utilization of pulps for printing paper grades, and still needs much study, especially when hardwoods are being used at increasingly higher rates. Unfortunately the picking tendency of the papers prepared in the present work were not assessed, and only some aspects of the behavior of vessels during beating will be discussed. It should be remembered, though, that barrel-shaped vessels such as those of both eucalypts may be broken down into pieces very easily during beating, as already demonstrated (1).

The values in Table VII are in very close agreement with published data (3,45,93,102,163). Some information on the proportion of vessels in wood is also included, with figures taken from the literature (6,47,218).

Additional Pulp Characteristics. For further characterization of the pulps their brightness, viscosities, determined by dissolution in 0.5 molar cupriethylenediamine, pH of aqueous suspension, and wet zero-span breaking length, determined with the Pulmac Zero Span Tester are listed in Table VIII. The pulps' different viscosities

TABLE VIII. ADDITIONAL PULP CHARACTERISTICS

Pulp	Pulp brightness, °ISO	Pulp viscosity, cP	Wet Zero- span tensile index, N m/g	pH of suspension
Southern pine	86.8	14±0.1	104±5	6.3
Silver birch	88.3	18.5±0.2	136±8	6.1
<u>E. grandis</u>	90.4	18.0±0.1	127±5	5.6
<u>E. rostrata</u>	85.2	12.9±0.1	107±7	5.8

may be considered as consequences of different original degrees of crystallinity in each species. Most probably,

however, they are due to different pulping and bleaching conditions. This property is not expected to influence the strength characteristics of pulps to a great extent, as recently reported (130), which do correlate well with estimated fiber strength, as indicated by wet zero span measurements.

A relationship between the zero span breaking length measured on a wet sheet and the fiber strength is based on the idea that in a randomly oriented wet handsheet the strength of fiber bonds is negligible compared to the strength of the fibers (42). Thus in a zero-span test of such a sheet all fibers will be theoretically stretched until they break, and the reading in the instrument is only dependent on the wet fiber strength. The results obtained with this technique were very reproducible, and were therefore adopted as estimates of fiber strength.

The pH of pulp suspensions in distilled water were determined to evaluate whether an adjustment to a common value was necessary when preparing the pulps for beating. Since they were very close the pulps were treated as received.

#### Discussion of Observed Patterns in Fiber Morphology

Even with the observed large differences in dimensions among the fibers of the four pulps some common features can be noticed in their ultrastructure, and they are worthy of mention. It has been observed that both pine latewood

and eucalypt fibers have small fibrillar angles in  $S_2$  layer. At the same time both eucalypts have thin fibers, which are also comparatively thickwalled, the same happening with pine latewood, as compared to earlywood tracheids. On the other hand the opposite characteristics are noticed in birch and pine earlywood fibers; they are relatively thinwalled, with larger diameters and larger microfibrillar orientations.

Although environmental and genetic aspects should not be ruled out it seems that these features could have a common origin in the formation and aging of the cell wall. Some patterns on the influences of physiological conditions associated with growing and aging of the tree can be noticed, as isolated cases, when comparing earlywood and latewood fibers in conifers. The consistently thicker cell walls of southern pine tracheids accompanying smaller fibrillar angles is a well-known feature of latewood (57,129,152,178). Higher crystallinity and crystallite length (115,118,152,200), smaller crystallite widths (151,152,200) and increased cell wall density (202,212,241) are also usually observed in latewood fibers. A higher degree of order in the molecular organization thus seems to prevail in these fibers, as a consequence of different growth conditions.

There is still a lot of controversy about the mechanisms of plant cell wall growth (37,177,178), but as a

number of studies seem to indicate (151,152,177,178,200) the biogenesis and formation of wood cell walls could follow a more general pattern than earlier recognized. Particularly the rate of cell maturation could have an important bearing on fiber ultrastructure. In this context it has been revealed that the angles in  $S_1$ ,  $S_2$  and  $S_3$  layers of red pine had a tendency to decrease with the progress of maturation (43).

When comparing the fibers in this study it is important to notice some differences in their growth conditions. The southern pines are known to grow comparatively fast among conifers, and one of their main characteristics is the sharp transition from earlywood to latewood, and the large morphological differences between the two. Hardwoods generally grow faster than softwoods, and apart from anatomical differences probably related to evolution, hardwoods usually have more fibers per unit weight of wood, also with smaller diameter, and thickerwalled cells. They also present smaller differences between earlywood and latewood, and the latter is usually in lower proportions than in conifers. Following this hypothetical trend it will be noticed that the eucalypts, with extremely high growth rates have many thin fibers per unit weight of wood, and have a tendency to be relatively more thickwalled than slow-grown hardwoods, such as birch.

Therefore, the general ideas proposed in earlier studies (151,152,177,178,200) and the association of ultrastructural properties found for the fibers in this work with some of their growth characteristics may lead to the formulation of a hypothesis about the trends observed. It could be possible that, in general, a more active cambium, during periods of high rates of growth, would yield more fibers with smaller diameter, which would also mature faster, producing thicker cell walls and steeper helices of microfibrils than their counterparts from a less active cambium. Other features would also be common in this pattern, such as an increased crystallinity index, bearing a direct relationship with crystallite length (152). An increased packing density of the cell wall would follow (2), as a result of the combination of low fibrillar angles and small crystallite widths (152).

The observed similarities between birch and pine earlywood, as having characteristics deriving from a less active cambium, and between the fibers of both eucalypts and pine latewood belonging to another group from a more active cambium, seem to support the hypothesis of a general pattern occurring during cell growth and maturation, and reflecting in the fiber ultrastructure. Although no data on the fibers' crystallinity and crystallite width is presented in this study, their different packing densities,

as shown in Table VI are in agreement with the proposed ideas. Thus there seem to be more characteristics to be taken into account when considering the behavior of so-called thickwalled fibers, then those indicated by solely geometrical considerations of their transverse dimensions.

The hypothesis here formulated appears to be sound and in agreement with other observations, although the conclusions taken from the patterns observed in only four types of fibers may seem somewhat speculative. A more systematic approach, involving fibers from many species, could provide a deeper insight into this area, of obvious importance for geneticists and forest engineers. Nevertheless, further similarities in the response of the fibers studied to mechanical action in the beater, as related to their groupings according to ultrastructure, seem to confirm the proposed ideas.

To summarize the findings on the characteristics of the fibers it can be stated that the most important differences among the four pulps used in this study

could be attributed to morphological and chemical origins. The morphological characteristics seem to play the major role in differentiating the pulps, microscopically from the long and coarse fiber in southern pine to the short, thin and numerous eucalypt fibers. The ultrastructural organizations observed, as indicated by the microfibrillar orientations in  $S_1$  and  $S_2$  layers and the density of the cell wall, are probably the most descriptive of the differences between birch and the eucalypts, although they revealed unexpected similarities between these and the pine. The ultramicroscopic characteristics seem to follow a general pattern influenced by growth conditions, which is also proposed to be reflected in the fibers' transverse dimensions. The chemical differences among the pulps are due mostly to their hemicelluloses, particularly xylan contents, which are expected to enhance the differences in beating responses, as influenced mostly by morphological factors.

### Beating and Its Effects

It is well accepted that paper strength is directly influenced by the degree of interfiber bonding. Unbeaten pulps are very weak and do not meet the requirement of runnability of wet or dry sheets or the demands of the paper end-uses. By beating the fibers in any refining device the bonding potential can be enhanced, thus improving some strength properties, while unfortunately in most cases impairing other characteristics, such as bulk, opacity and tear strength. A careful monitoring of the effects of beating on fibers and paper properties can help to understand the process, and consequently narrow down the gap between the advantages and disadvantages of beating.

Indeed, this subject is dealt with in numerous publications, as discussed in the review of the literature, but it was felt that the application of some newer, sophisticated tools and methods might yield interesting, additional information. The four pulps described in the preceeding section were used not only due to the increased interest in their utilization, but most importantly because of their vastly different morphologies, with the hope that their behavior might shed more light on the origins of the various responses to beating of pulps beginning to appear in the papermaking field.

The study of the beating process was divided into three parts. The first is related to characteristics observed in the beater, such as the energy consumption, the beating clearance and the load applied. The second part is concerned with the effects of beating on fibers, and was assessed by measurements of their properties in the wet state, such as the developments of specific surface area, specific volume, and fines formation. Based on these results the characteristics of the beating effects on pulps were analyzed, with emphasis on their dynamic drainage properties, wet web strength and the routine evaluation of the properties of dry handsheets at various beating levels.

On the basis of the above results the beating process is discussed on macroscopic, microscopic and ultra-microscopic levels.

#### Beating in the PFI Mill

All treatments were performed using only this beater. The main reasons for this choice were the reproducibility of the beating operation and the easy manipulation and control of the PFI mill. It was also intended to use an equipment that would minimize the cutting of fibers and where the different influences of the complex flow behavior of the stock usually experienced during low consistency refining could be avoided. The results collected for this aspect

of the study are listed for the four species in Table IX. The net beating energy was determined at several beating levels at two beating loads (3.33 kN/m and 4.90 kN/m). All the hardwoods were beaten for the same number of revolutions, while the pine required additional beating to reach comparable changes in properties, (as discussed in later sections). The effects of the beating loads were compared in order to introduce a different beating condition and to assess its influence on the fibers.

It can be observed from Table IX that within the limits of a customary beating curve significantly different amounts of beating energy are absorbed by the pine and the hardwoods for the development of their papermaking potentials. Thus, a slightly treated pine pulp took up 1.37 MJ/kg and a fully beaten one 6.94 MJ/kg at 3.33 kN/m load, and about 8% more at 4.90 kN/m, while the three hardwoods consumed roughly half of these amounts for development of similar levels of strength, as discussed later. This does not mean that the differences among the hardwoods are minor. It should be kept in mind that, in terms of the work effected on the fibers for a given energy expenditure the results are very different, as will be seen later in the created surface area and fiber delamination.

The recorded clearances between the rotor and house as set up by the pulp during beating are listed in the fourth column of Table IX. They are related to zero clearance

TABLE IX. PFI - MILL BEATING DATA

Pulp	Beating load kN/m	PFI Revolu- tions	Net beating energy, MJ/kg	Beating clearance, $\mu\text{m}$	CSF ml
Southern pine	3.33	0	0	650 $\pm$ 30	730
		1500	1.37 $\pm$ 0.02	288 $\pm$ 18	715
		3000	2.76 $\pm$ 0.03	200 $\pm$ 13	648
		5000	4.47 $\pm$ 0.03	105 $\pm$ 10	535
		8000	6.94 $\pm$ 0.05	43 $\pm$ 6	300
	4.90	0	0	550 $\pm$ 21	730
		1500	1.52 $\pm$ 0.03	157 $\pm$ 15	705
		3000	3.08 $\pm$ 0.03	58 $\pm$ 7	584
		5000	4.92 $\pm$ 0.04	-13 $\pm$ 4	378
		8000	7.49 $\pm$ 0.06	-95 $\pm$ 2	115
Silver birch	3.33	0	0	0 $\pm$ 5	560
		500	0.41 $\pm$ 0.01	-65 $\pm$ 4	545
		1500	1.26 $\pm$ 0.02	-100 $\pm$ 3	508
		3000	2.46 $\pm$ 0.02	-130 $\pm$ 3	405
		5000	3.93 $\pm$ 0.03	-145 $\pm$ 2	237
	4.90	0	0	-25 $\pm$ 4	560
		500	0.45 $\pm$ 0.01	-115 $\pm$ 3	540
		1500	1.33 $\pm$ 0.01	-148 $\pm$ 3	494
		3000	2.56 $\pm$ 0.03	-168 $\pm$ 2	364
		5000	4.06 $\pm$ 0.03	-183 $\pm$ 2	163
<u>Eucalyptus grandis</u>	3.33	0	0	25 $\pm$ 5	555
		500	0.41 $\pm$ 0.01	-62 $\pm$ 5	520
		1500	1.26 $\pm$ 0.01	-87 $\pm$ 4	500
		3000	2.55 $\pm$ 0.03	-110 $\pm$ 3	420
		5000	3.95 $\pm$ 0.04	-125 $\pm$ 2	306
	4.90	0	0	0 $\pm$ 5	555
		500	0.47 $\pm$ 0.01	-100 $\pm$ 4	520
		1500	1.38 $\pm$ 0.02	-136 $\pm$ 3	488
		3000	2.68 $\pm$ 0.02	-161 $\pm$ 3	400
		5000	4.32 $\pm$ 0.04	-175 $\pm$ 2	235
<u>Eucalyptus rostrata</u>	3.33	0	0	25 $\pm$ 5	480
		500	0.39 $\pm$ 0.01	-56 $\pm$ 5	435
		1500	1.21 $\pm$ 0.01	-93 $\pm$ 3	400
		3000	2.33 $\pm$ 0.03	-114 $\pm$ 3	325
		5000	3.73 $\pm$ 0.04	-140 $\pm$ 2	215
	4.90	0	0	0 $\pm$ 5	480
		500	0.42 $\pm$ 0.01	-100 $\pm$ 4	425
		1500	1.29 $\pm$ 0.03	-145 $\pm$ 4	377
		3000	2.52 $\pm$ 0.03	-171 $\pm$ 3	278
		5000	4.07 $\pm$ 0.04	-189 $\pm$ 2	156

settings obtained under static conditions, without any pulp. The negative values observed mostly with the hardwood pulps are due to the changes in the relative positions of beater roll and housing under static and dynamic conditions, and under different applied loads (233,234,235). However, it should be kept in mind that to assess the progress of the treatment it is rather the change in the clearance and not the actual gap value that should be considered for each pulp .

Many important changes in the fiber structure occur in the beating gap during refining. It is believed that the changes in the clearance in the PFI mill during beating are measures of the amount of energy absorbed by the pulp (234). Indeed, when values recorded during this study were plotted versus the logarithm of the net energy input (Figure 9) a straight line relationship was found, similar to the relationship usually reported between paper properties changed during mechanical treatment (e.g. tensile index, apparent density, etc.) and the logarithm of the energy input. This fact can be interpreted as an indication of a possible close relationship between changes in clearance and the beating mechanism.

The real picture of the beating events is clearer, however, in a direct plot of beating gap vs. net energy, as shown in Figure 10. In this figure three distinct clusters

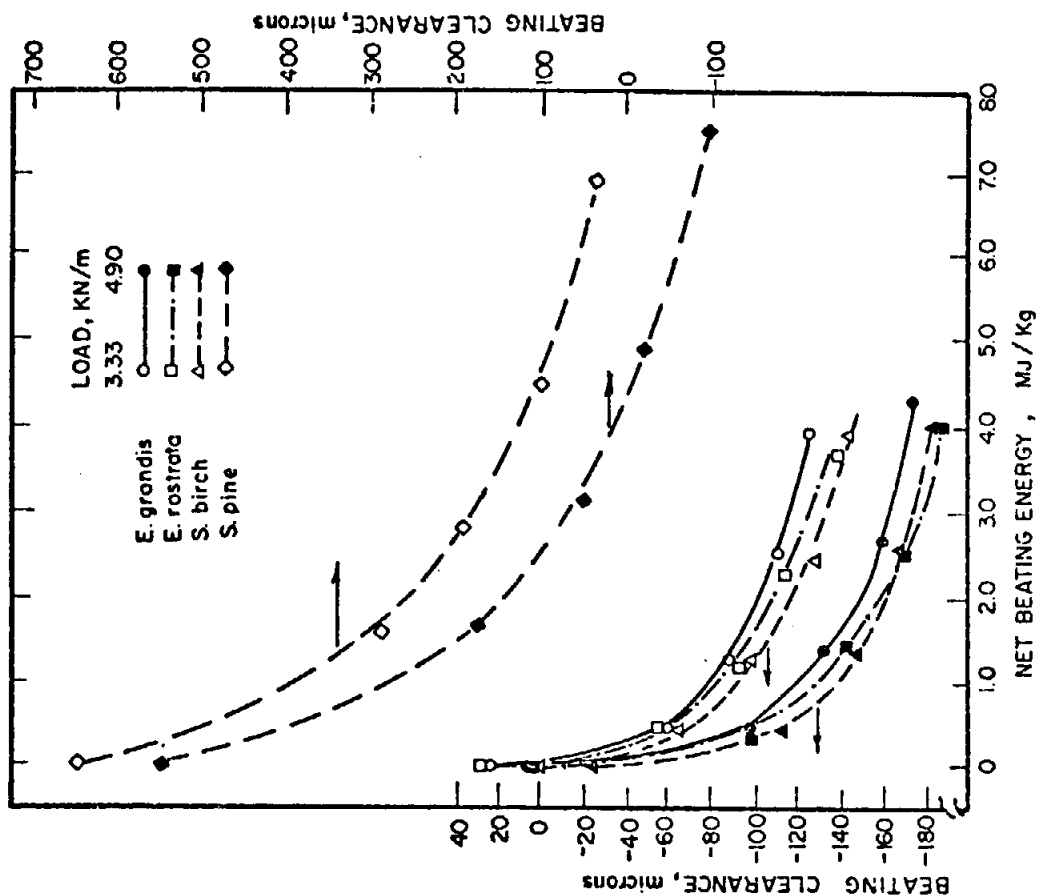


Figure 10 - Relationship between beating clearance and the beating energy.

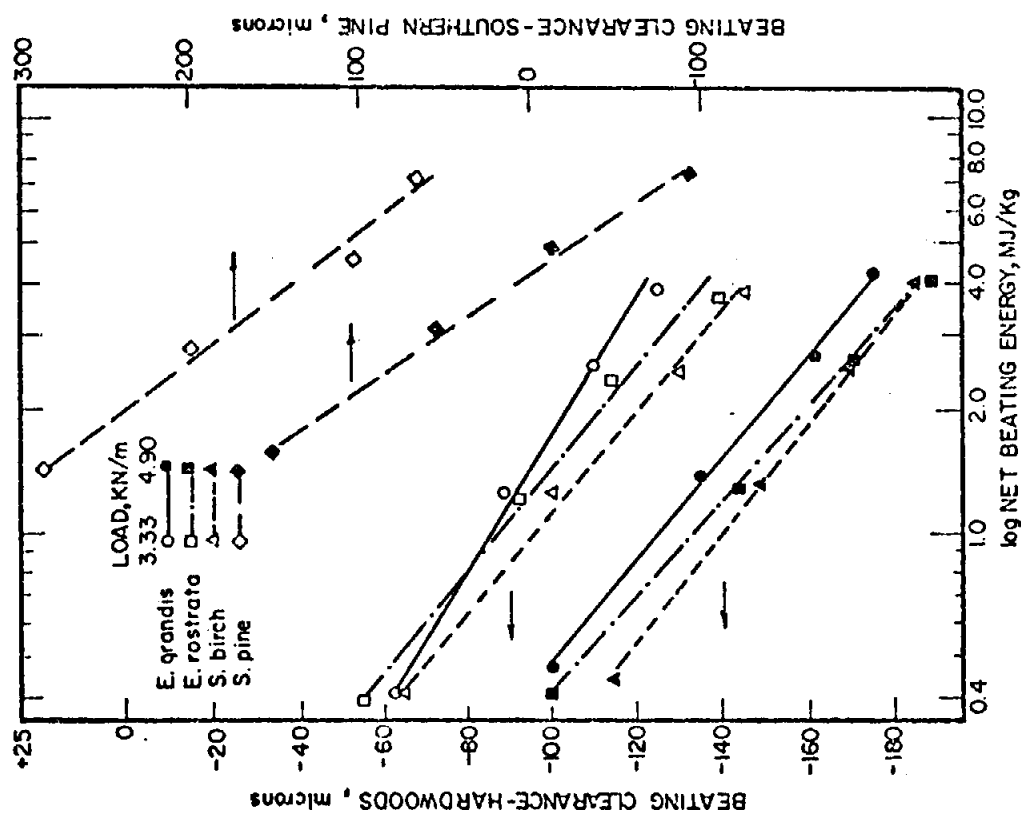


Figure 9 - Relationship between beating clearance and the logarithm of beating energy.

of curves can be noticed. The curves for pine indicate that this pulp in the unbeaten state creates, among all four pulps, the largest gap between the rotor and the house. This clearance width is rapidly reduced by progressive beating, from a value of 650  $\mu\text{m}$  to 43  $\mu\text{m}$ , at 3.33 kN/m load. At the higher load these values are proportionally smaller. The remaining three pulps under similar conditions display much smaller gaps than the pine pulp. The eucalypts practically do not differ in the unbeaten state, while the birch shows an even smaller gap. These differences widen somewhat as beating progresses.

At this point the question arises as to what is the role of the mill clearance in the beating of pulp. From the observation and data collected it appears, that the variation in the gap is a direct indicator of the advancement of the process. Changes in clearance at different loads may be attributed to different levels of pressure forcing the pulp into the grooves, but the changes imparted to pulp properties can also influence the beating gap. Thus, if the clearance is not reduced during beating the properties of the pulp won't be changed much. Interesting related phenomena are the well known difficulty of beating of a high-alpha cellulose pulp, and the small changes in clearance observed during beating of a cold alkali-extracted pulp (234).

The explanation of this behavior may be found in the state of the pulps in the refining zone. The ability to withstand the action of the load applied and/or conform to this force can be considered a function of the configuration that the fibers assume between the bars. There has been increasing acceptance of the idea that beating involves primarily the treatment of fiber aggregates, or flocs, and not of single fibers as independent particles (12,56,70,84,174,208). This has been observed in refiners operating at low consistencies (12,70) and in the PFI mill (158). This concept seems to be very natural to accept, not only due to the known ability of fibers to form networks even at low concentrations, but also because it is difficult to realize how single fibers would withstand the high pressures existing between refiner bars, without being completely crushed. If the space is filled with flocs, built up of many fibers, the stresses can be distributed, thereby preventing crushing of the aggregates, which indirectly control the clearance in the refiner.

Obviously the size and the strength of the flocs depend also on the individual fiber properties. It is well-known that flocculation is promoted by increased fiber length, fiber length-to-width ratio (209) and fiber flexibility (185), all these factors increasing the possibilities of fiber entanglement. However these findings were restricted to the properties of pulp suspensions under flow

conditions, and do not involve the appreciable compressive stresses occurring inside the beater. Therefore even after the suggestion that the straining of the fiber networks in both shear (during flow) and compression may involve the same network deformation phenomena (10) the perfect understanding of the behavior of flocs in the beating zone still lacks a considerable amount of study.

In this context not only the properties of flocs in general, but also the influences of fiber characteristics on the way in which they interact and form networks, which respond differently to mechanical stresses, seem to be of great importance for the understanding of the beating process. Although it is not the purpose of this study to analyze these characteristics in depth, some considerations are presented after the general discussion of the beating effects. At this point it is already indicated from the results in Table IX that the flocs of southern pine are much bigger than those produced from the three hardwoods, and thus are responsible for the large gap observed. These flocs also seem to be very compressible, as indicated by the rapid reduction in clearance. Within the three hardwoods studied, the flocs from birch fibers were noticed to have larger dimensions than the flocs of both eucalypts, but their behavior in the beating gap seems to be mostly related to other properties, as seems to be indicated by

the smaller initial clearance during beating of birch when compared to E. grandis and E. rostrata.

Of course, the changes occurring in the flocs under compressive stresses become increasingly important as beating proceeds (208) and should be dealt with in order to explain the differences in behavior of different pulps during this mechanical treatment. Accordingly the approach taken in the present study was to analyze the formation of flocs, as influenced by the original fiber properties and by the forces exerted by the beater bars. On the other hand, after the fibers have been modified they will, in turn, change the behavior of the flocs. These interactions are believed to play a major part in the beating process, and will be discussed later in great detail. The absence of such considerations in the literature indicates that our interpretation of the beating mechanism is novel.

#### Effects of Beating on Fibers

One of the most common ways to follow the beating of a pulp is by measurement of its changes in freeness. These values are listed in the fifth column of Table IX. It can easily be seen that not much information is provided by the freeness alone since the pulps differ greatly in their initial values. Comparisons at constant freeness would not tell much about the pulps characteristics or how they have absorbed the energy. Other measurements, described in the

next two sections, are believed to provide the information sought about the beating behavior of the pulps. However, the freeness values are still listed here rather as a reference to the earlier ways of analyzing the beating process. The CSF measurements will be dealt with in more detail when analyzing the drainage characteristics of the pulps.

Properties of Pulps in the Wet State. The properties of fibers in the wet state were determined by measurements of their specific surface area, specific volume, and fines content at the successive stages of beating. Data were collected on whole pulps and on those from which the original fines plus the ones created by beating were removed (pure fibers).

All these determinations were carried out in an attempt to quantify the beating effects. As described in the literature review most investigators agree on the definition of the primary beating effects (9,56,66,97). However the extent of development of each of them is still the subject of some controversy. Perhaps this is due to the fact that most conclusions have come from deductive reasoning, since the quantitative measurements of the beating effects are difficult to obtain.

Undoubtedly the changes in water-fiber relationship developed in beating are crucial for the performance of the

pulp during paper manufacture. Evaluations of external fibrillation, swelling, drainage rate, wet web strength, etc. have been done in the past, but unfortunately the techniques did not produce results which relate very well with the behavior of the pulp in the wet-end of the paper machine. The improved methods used here afforded the possibility of acquiring more precise information in this area.

Hydrodynamic Specific Surface Area - The water permeability method was chosen because it provides the best estimate of the external surface area of pulp fibers and fines involved in their interaction with water during paper-making (41,67,106,107,109,153,183,184,220,225,227). As described in the review of the literature the theory underlying this technique is the same as the one derived for fluid flow through porous media (29,87,184). As the method consists of measurement of the ease with which water flows through a pulp mat at different consistencies, the surface estimated is the one at the swollen fiber-free water interface.

Many shortcomings of the technique of permeability testing were eliminated in the laboratory by careful adjustments of the method of handling the Pulmac Permeability Tester, which also allowed a substantial improvement of the reproducibility of the results. Thus the data obtained for

the four pulps studied and presented in Tables X and XI can be dealt with high confidence.

Considering the very significant role of the fines in the hydrodynamic properties of the pulps and the importance they have in the papermaking process, the hydrodynamic properties of pulps liberated from fines were also determined. The properties for the fines were then calculated by difference, using the quantitatively determined fines contents (Table XII) and assuming as valid the additivity rule for specific surface of fibers and fines (67). The corresponding results are plotted in the forthcoming graphs and tables.

As can be seen in figures 11 and 12 all the pulps here studied exhibit similar trends as beating energy is applied. They start at very low specific surfaces, which increase slowly in the beginning of beating and tend to shift upwards as beating proceeds. For pure fibers the surface area increases almost linearly with energy applied (Figures 13 and 14). The differences between the whole pulps and pure fibers (fines-free pulps) are due not only to the fines content, but also to the specific surface of the fines (Table XIII), which visibly differ considerably from that of fibers.

As can be noticed in Table XII and Figure 15 the fines contents are shown to increase almost linearly with

TABLE X - HYDRODYNAMIC SPECIFIC SURFACE OF PULPS,  $\text{cm}^2/\text{g}$  (a)

PFI Revolu- tions	Beating load KN/m	Southern pine		Silver birch		<i>Eucalyptus grandis</i>		<i>Eucalyptus rostrata</i>	
		Whole pulp	Fines Free	Whole Pulp	Fines Free	Whole Pulp	Fines Free	Whole Pulp	Fines Free
0	-	14,500	7,172	24,250	11,261	25,297	12,235	29,800	13,033
500	3.33	-	-	26,000	12,044	26,100	12,547	31,815	13,854
	4.90	-	-	26,670	12,248	26,827	13,027	33,304	14,348
1500	3.33	19,930	9,250	29,948	13,513	29,939	14,332	37,134	15,239
	4.90	21,500	10,872	31,546	14,268	31,933	15,284	39,500	16,275
	3.33	24,100	10,636	39,105	16,109	34,781	16,169	44,748	16,985
3000	4.90	36,430	12,777	45,702	17,335	40,053	17,946	51,800	18,413
	3.33	39,036	12,243	56,090	18,606	43,006	17,908	59,280	18,549
5000	4.90	62,209	16,050	79,076	21,012	53,547	20,657	78,204	21,099
8000	3.33	68,000	14,640	-	-	-	-	-	-
	4.90	100,687	20,038	-	-	-	-	-	-

(a) The calculated 95% confidence intervals for the means were observed to be within 5-8% of the values reported.

TABLE XI - HYDRODYNAMIC SPECIFIC VOLUME OF PULPS, cm<sup>3</sup>/g

PFI Revolu- tions	Beating Load kN/m	Southern pine		Silver birch		Eucalyptus grandis		Eucalyptus rostrata	
		Whole Pulp	Fines Free	Whole Pulp	Fines Free	Whole Pulp	Fines Free	Whole Pulp	Fines Free
0	-	2.14±.03	1.89±.02	2.78±.04	1.84±.03	2.24±.03	1.53±.02	2.37±.02	1.49±.02
	3.33	-	-	3.27±.05	2.02±.02	2.69±.03	1.49±.02	2.80±.03	1.69±.03
	500	-	-	3.40±.07	2.06±.03	2.86±.03	1.60±.02	3.00±.06	1.77±.05
1500	3.33	3.06±.05	2.35±.02	3.75±.04	2.34±.03	3.05±.04	1.70±.03	3.25±.05	1.90±0.4
	4.90	3.30±.08	2.44±.03	3.82±.07	2.38±.03	3.15±.03	1.77±.02	3.35±.05	1.99±.05
	3.33	3.48±.07	2.61±.04	4.15±.09	2.66±.05	3.44±.06	1.95±.05	3.74±.06	2.15±.02
3000	4.90	3.90±.07	2.75±.04	4.28±.08	2.74±.04	3.60±.04	2.01±.04	3.97±.05	2.21±.04
	3.33	3.84±.07	2.70±.04	4.52±.13	2.84±.04	3.70±.08	2.10±.03	4.15±.06	2.40±.03
	5000	4.10±.06	2.86±.03	4.55±.11	2.90±.03	3.90±.09	2.19±.06	4.25±.06	2.45±.03
8000	3.33	4.08±.12	2.78±.04	-	-	-	-	-	-
	4.90	4.40±.19	2.99±.05	-	-	-	-	-	-

TABLE XII. FINES FRACTION (-200 MESH) OF PULPS, % W/W

PFI Revolu- tions	Beating load kN/m	Southern pine	Silver birch	<u>Eucalyptus grandis</u>	<u>Eucalyptus rostrata</u>
0	-	4.0±0.3	6.2±0.4	6.6±0.3	11.8±0.5
500	3.33	-	6.6±0.4	6.8±0.3	12.1±0.4
	4.90	-	6.8±0.5	7.0±0.4	12.6±0.3
1500	3.33	5.1±0.4	7.8±0.4	7.9±0.3	13.8±0.3
	4.90	5.7±0.4	8.2±0.4	8.5±0.3	14.6±0.2
3000	3.33	6.9±0.5	9.8±0.3	9.5±0.5	15.0±0.3
	4.90	8.7±0.4	10.6±0.4	10.3±0.3	16.6±0.4
5000	3.33	9.0±0.4	11.5±0.5	11.1±0.3	16.8±0.2
	4.90	12.3±0.3	13.0±0.3	12.2±0.3	18.8±0.3
8000	3.33	12.3±0.3	-	-	-
	4.90	16.9±0.4	-	-	-

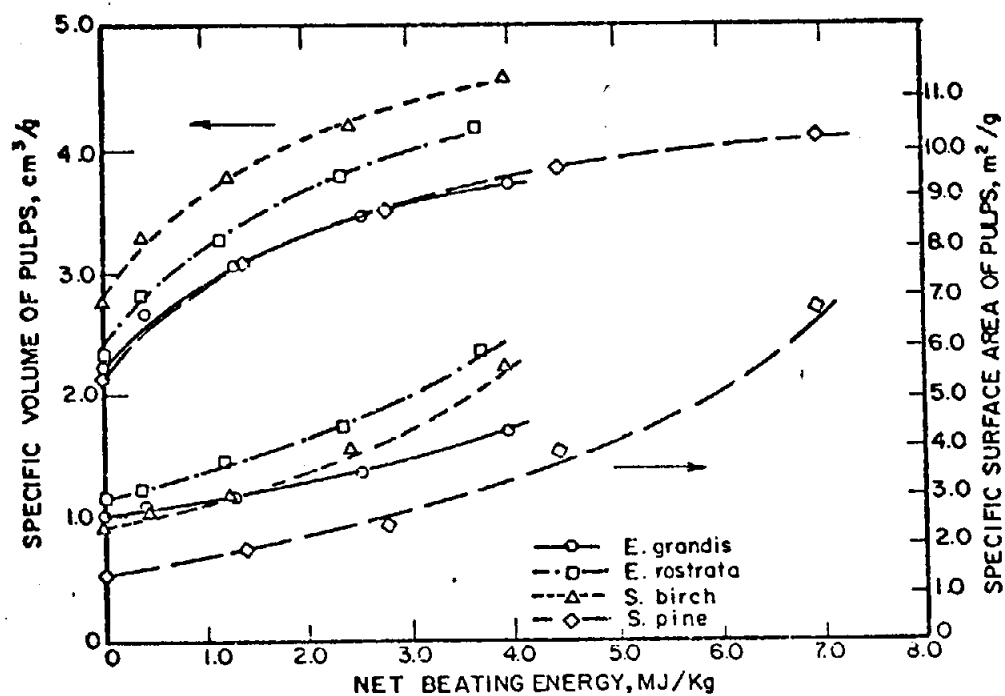


Figure 11 - Development of specific surface and specific volume of whole pulps with beating energy - 3.33 kN/m load.

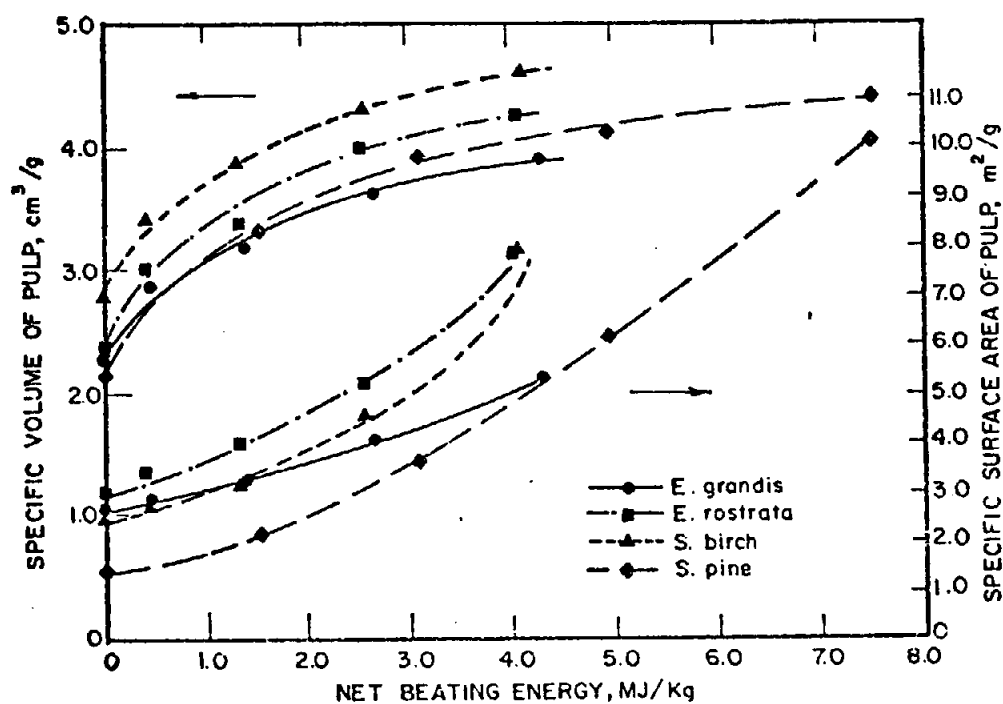


Figure 12 - Development of specific surface and specific volume of whole pulps with beating energy - 4.90 kN/m load.

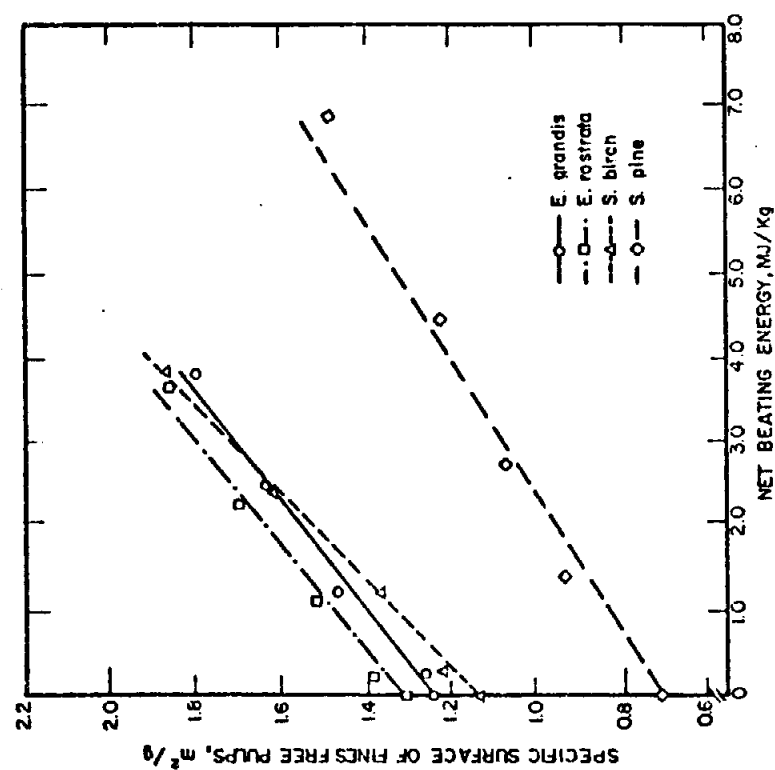


Figure 13 - Development of specific surface of fines-free pulps with beating energy - 3.33 kN/m load.

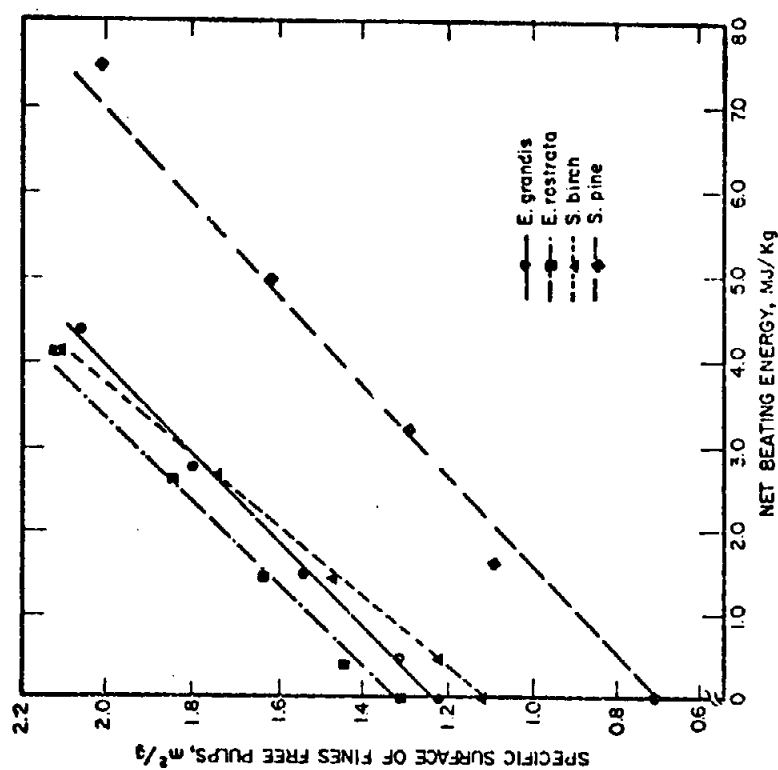


Figure 14 - Development of specific surface of fines-free pulps with beating energy - 4.90 kN/m load.

TABLE XIII - CALCULATED SPECIFIC SURFACE OF FINES,  $\text{cm}^2/\text{g}$ 

PFI Revolu- tions	Beating load kN/m	Southern pine	Silver birch	<u>Eucalyptus grandis</u>	<u>Eucalyptus rostrata</u>
0	-	190,372	220,761	210,141	155,126
	3.33	-	223,498	211,855	162,292
500	4.90	-	224,336	210,169	156,062
	3.33	203,480	224,218	211,888	173,898
1500	4.90	197,328	224,975	211,154	175,380
	3.33	205,766	250,762	212,085	202,071
3000	4.90	284,650	284,948	232,577	219,539
	3.33	309,943	344,553	244,016	260,995
5000	4.90	391,326	467,658	289,509	324,849
	3.33	448,461	-	-	-
8000	4.90	497,251	-	-	-

beating, which is very similar to the preceeding fashion observed for the specific surface of the fibers themselves. This seems to indicate that the amount of fines produced is proportional to the newly created surface on the fibers.

However, as the fines are formed their characteristics seem to change continuously with the progression of treatment (Table XIII). The calculated specific surface of fines is seen to increase almost exponentially, the higher values approaching the reported figure of  $500,000 \text{ cm}^2/\text{g}$  for the specific surface of pure "crill" (secondary fines only) (192). The specific surfaces of the fines in the unbeaten pulps consisting mostly of ray cells and debris of fibers are low compared to the surfaces of fines developed through beating. When beating starts new "chunks" are formed, from vessels in the hardwoods, some cut fiber pieces and primary wall debris. With continuation of the treatment the action upon the "coarse" fines and on fiber surfaces is extended, with the progressive peeling of the outer layers and liberation of fibrils, of very high specific surface. The formation of fines with increasingly high surfaces is responsible for the increase of the specific surface of whole pulps in a similar manner. These phenomena are believed to affect significantly the pulp properties which depend mostly on interactions with water, such as drainage resistance and the consolidation and frictional forces in

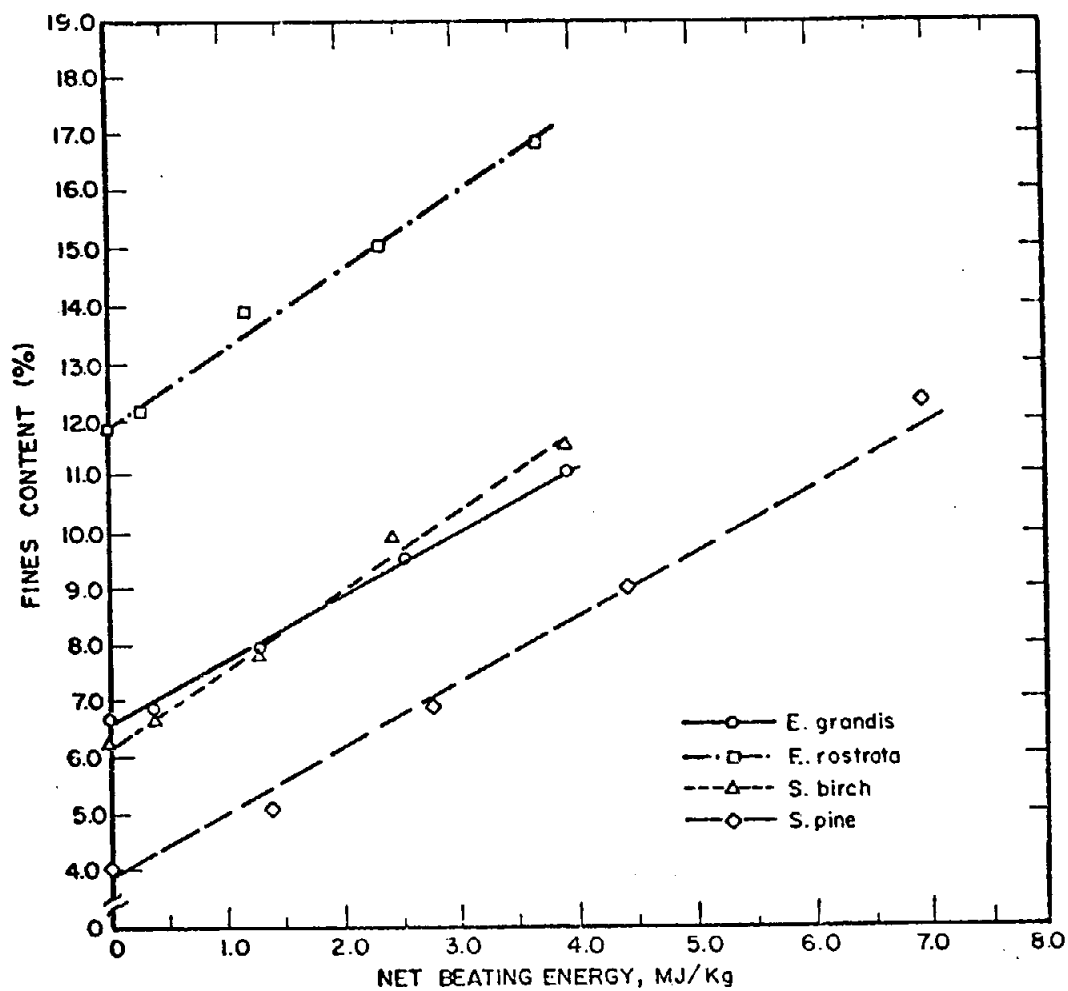
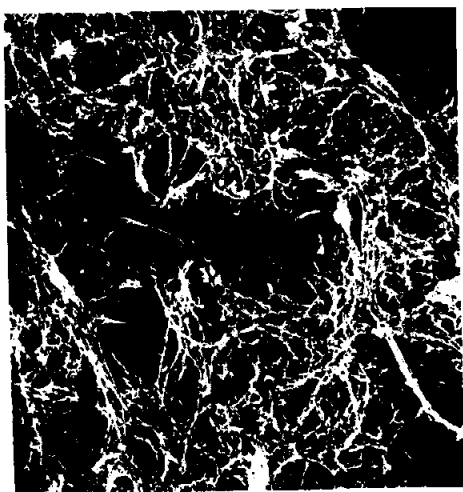


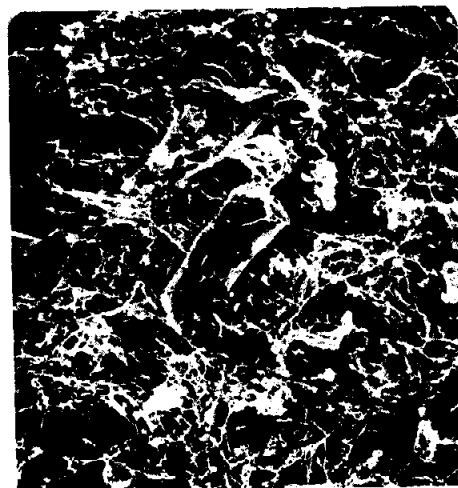
Figure 15 - Relationship between fines content of pulps and beating energy - 3.33 kN/m load.

the wet web. The scanning electron micrographs of the fines from the very strongly beaten pulps are shown in Figure 16. Their appearance are in agreement with the values in Table XII, e.g., the large amounts of fibrillar material in southern pine have the highest specific surface value, followed by birch, which already shows some debris and a somewhat lower specific surface; E. rostrata displays many unbroken ray cells but, yet lots of fibrillar material, and finally E. grandis shows mostly pieces of lamella and fiber debris, with the lowest value of specific surface among the fines of all four pulps.

The reported values from measurements of specific surface in the literature have shown different patterns of development. Some authors have reported increasing rates of specific surface development with extent of treatment (38,184,237), while others have indicated linear relationships for pulps (107,215,220,227) and fibers (107,220). Since these studies have involved different beating equipments and often the results discussed refer to only one type of pulp, it appears difficult to draw a definite conclusion. The important aspect however, seems to be the understanding of how this beating effect relates to concomitant changes in the fibers, as indicated by other measured properties. Most importantly, the differences in trends of development between specific surface and specific volume



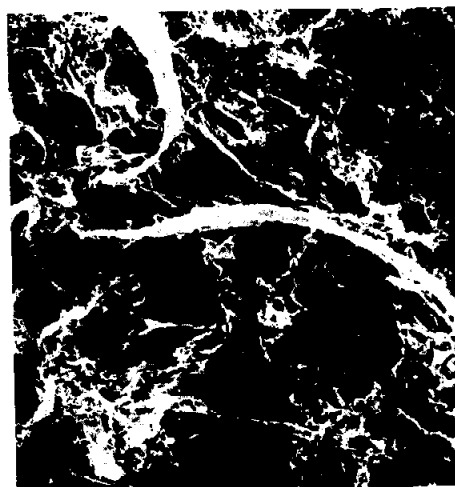
a. Fines from Southern pine pulp, beaten with 7.49 MJ/kg.



b. Fines from Silver birch pulp beaten with 4.06 MJ/kg.



c. Fines from *E. rostrata* pulp, beaten with 4.07 MJ/kg.



d. Fines from *E. grandis* pulp, beaten with 4.32 MJ/kg.

Figure 16 - Scanning Electron Micrographs of fines fraction from beaten pulps.

should be taken into account and related back to the beating process.

Comparing the four pulps in Figures 11-14 it can be seen that as far as surface area is concerned the hardwoods seem to form a group by themselves, while the pine shows much lower values. This may be due not only to the lower content of primary fines in the latter, but also to its much smaller number of fibers per gram. Comparing the three hardwoods it can be seen that the eucalypt pulps have very high specific surfaces in their unbeaten state, which is expected from their large number of small and thin fibers per gram. Comparisons of specific surface of other pulps with E. rostrata in its unbeaten state have to account for the larger amounts of primary fines in it, which are due to the large content of ray cells in this species. Although exhibiting a smaller specific surface than the eucalypts in the unbeaten state birch pulp develops new surfaces at a much higher rate than E. grandis, and at a little higher rate than E. rostrata. When comparing the pure fibers, however, the eucalypts show approximately the same behavior, with birch still displaying higher rates in the development of external fibrillation, at both applied loads. Thus, as far as the development of specific surface is concerned, the differences between the two eucalypt pulps seem to be due to the different rates of development of fines. Hence,

it becomes evident how important it is to assess the characteristics not only of whole pulps, but also of those of fines-free ones.

It is also interesting to notice that the rates of surface development of pine and E. grandis whole pulps are very similar at a low beating load. When the pure fibers are compared, the pine shows the lowest rate of surface area development among all pure fibers at low load, and about the same as eucalypts at the higher load.

This brings up another important observation resulting from this work: the effects of different loads on the responses to beating. It seems that the main difference between the two beating conditions is reflected in the specific surface of the fibers. As a higher load is applied in a given period of time a faster development of specific surface can be observed in all whole pulps and pure fibers, with larger differences between the two treatments showing up in southern pine. By comparison of Figures 11 and 12 it can be seen in the latter that birch and E. rostrata still display faster developments of specific surface than the other two pulps with an even higher rate being detected in birch. At high load E. grandis also shows a lower rate in surfaces creation than the other two hardwoods, and it can be noticed that the pine pulp specific surface approaches that of E. grandis, at a rate that resembles that of the

other two hardwoods. By comparison of pure fibers (Figures 13 and 14) it can be noticed that the relative positions of hardwoods are apparently maintained at both loads, while pine fibers display a much higher rate of surface development under the higher loading, approaching the behavior of the two eucalypts.

Yet a better view of these effects is obtained in Figure 17, where the values for specific volume have also been included. In addition to the observations discussed above it can be noticed that very little changes occur in the hardwoods by application of a higher load. For the pine, however, at a given energy expenditure a load of 4.90 kN/m vs. 3.33 kN/m is shown to produce both a larger specific surface and volume. The data in Tables XII and XIII also indicate that as the load is increased more fines are produced and relatively more for the pine than in the case of the other pulps. However, the specific surface of fines does not change much in the early stages of beating, but increases a little more at high than at low load as beating proceeds. These observations are in close agreement with the observed higher rates of surface development in pure fibers at higher load, and both effects help to explain the much faster development of specific surface at 4.90 kN/m in whole pulps.

In order to confirm the quantitative indications of

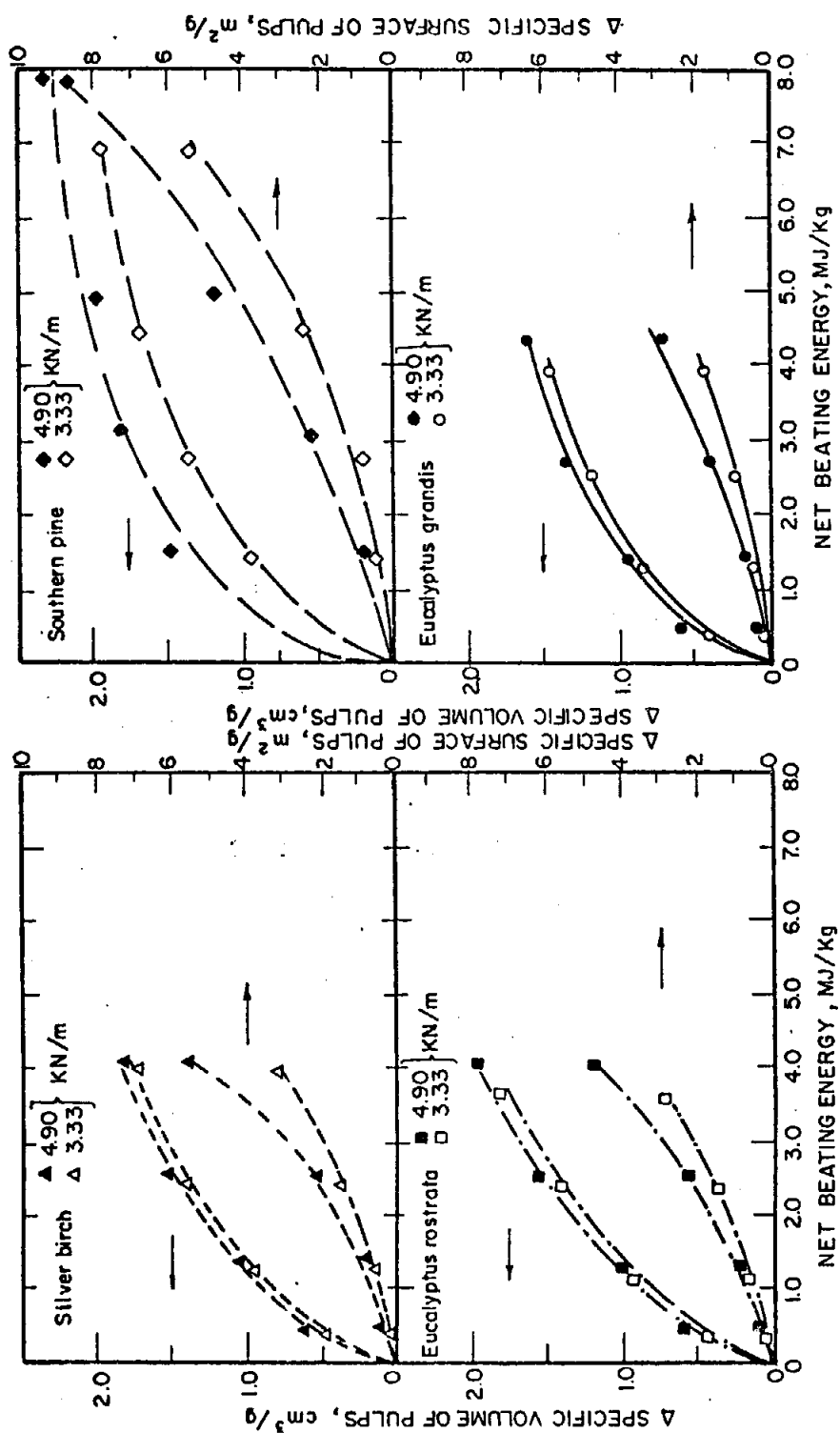


Figure 17 - Variation in specific surface and specific volume of whole pulps with beating energy at two different beating loads.

external fibrillation obtained from specific surface measurements, scanning electron micrographs of freeze dried suspensions of pure fibers were prepared at the same magnification. These are shown in Figures 18 and 19, for comparisons between the fibers in their unbeaten state and after application of the largest amount of beating energy. (i.e., 4.32 MJ/kg for E. grandis, 4.07 MJ/kg for E. rostrata, 4.06 MJ/kg for silver birch and 7.94 MJ/kg for southern pine.) (Figures 18b, 18d, 19d, 19b respectively.) It can be noticed that birch fibers display a larger extent of external fibrillation than both eucalypts, especially E. grandis. even by application of roughly 6% more energy to the latter. It should be remembered that the specific surface measurements indicate total area per gram of dry material. Therefore after reaching approximately the same total values of specific area as the hardwoods (around  $2.0 \text{ m}^2/\text{g}$ ) by application of a larger amount of energy each southern pine fiber should display a relatively higher degree of damage, and is indicated in Figure 19b.

Hydrodynamic Specific Volume - The specific volume of a pulp as determined by water permeability is defined as the volume per gram of dry fiber which is not participating in the flow of water. Being enclosed by the hydrodynamic plane of shear, the volume per gram of dry fiber is a measure of the total volume of the swollen fibers, including the lumen,



10.0 μm

a. SEM - E. grandis, unbeaten.



10.0 μm

b. SEM - E. grandis, beaten  
with 4.72 MJ/Kg.



10.0 μm

c. SEM - E. rostrata, unbeaten



10.0 μm

d. SEM - E. rostrata, beaten  
with 4.07 MJ/Kg.

Figure 18 - Scanning Electron Micrographs of unbeaten  
and beaten fibers of E. grandis and E. rostrata.



10.0  $\mu$  m

a. SEM - Southern pine, unbeaten.



10.0  $\mu$  m

b. SEM - Southern pine, beaten with 7.49 MJ/kg.



10.0  $\mu$  m

c. SEM - Silver birch, unbeaten.



10.0  $\mu$  m

d. SEM - Silver birch, beaten with 4.06 MJ/kg.

Figure 19 - Scanning Electron Micrographs of unbeaten and beaten fibers of southern pine and silver birch.

the fiber walls, and the water externally associated with fibrils detached from the fiber surfaces (184,220). As a consequence, the measurement of the total specific volume yields values which are larger than other estimates of fiber swelling (107,114). However, by comparing closely all available methods of determination of water associated with fibers (114,195,213,220) it is found that they really differ very little in the final results.

In view of those findings the permeability method was accepted and used as a true indication of water associated with the fibers under equilibrium at zero force of water removal. The water retention properties of papermaking fibers are believed to be affected by both macro and microstructure and to depend on lumen size collapsability, porosity and external fibrillation (183).

The results obtained with the four pulps used in the present study are listed in Table XI for whole pulps and fines-free pulps. Some results are also presented graphically in Figures 11 and 12. In all cases it is clear that, contrary to what was noticed for the surface area, the specific volume increases very fast in the early beating stages, even with a minimum energy applied, and then tends to level-off as beating proceeds. This can be seen in both whole pulps and pure fibers, (Table XI and Figures 20 and 21), which is an indication that the main effects are occurring at the fiber level, being affected

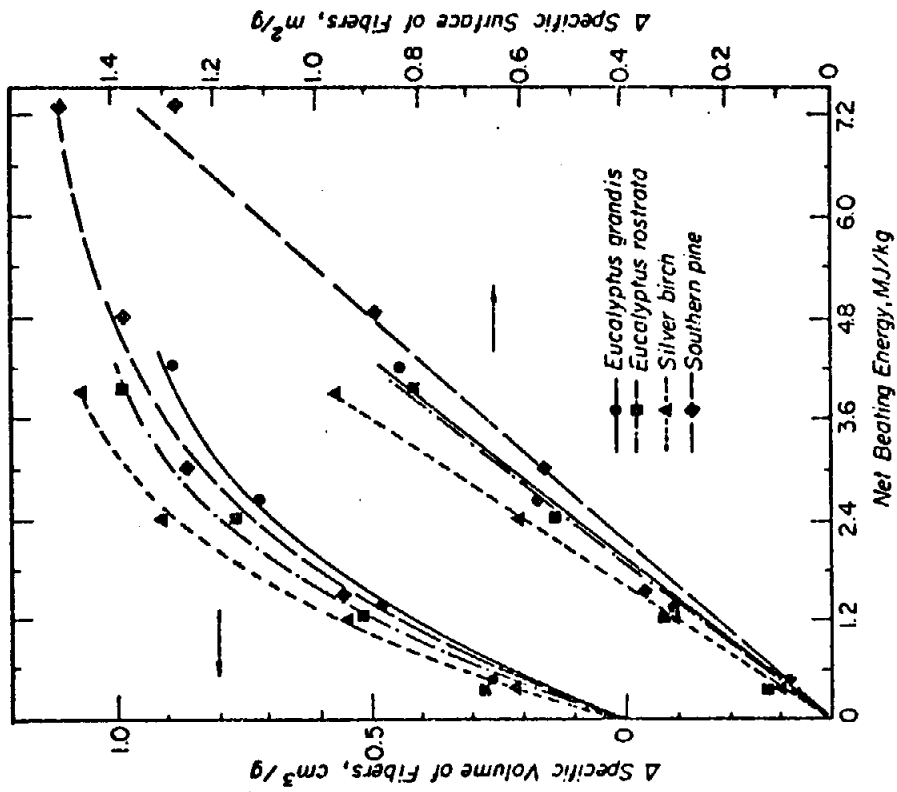


Figure 21 - Variation in specific surface and specific volume of pure fibers with beating energy - 4.90 kN/m.

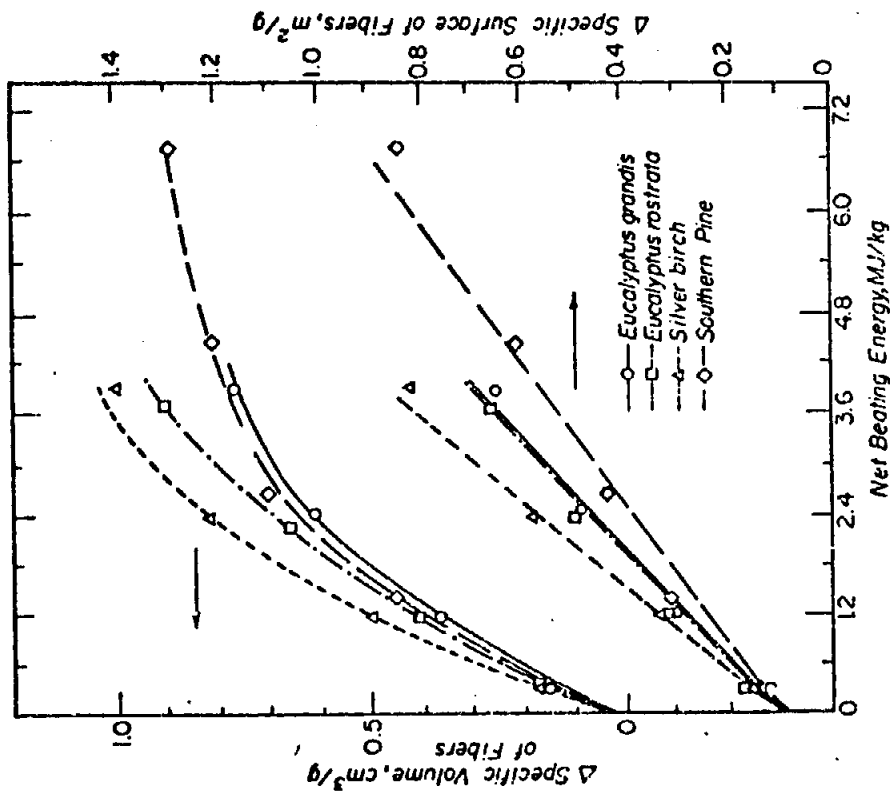


Figure 20 - Variation in specific surface and specific volume of pure fibers with beating energy - 3.33 kN/m.

by the fines only quantitatively, and not qualitatively as observed for specific surface. Similar measurements on other types of fibers have always shown a rapid increase in the amount of water associated with the fibers in the very early beating stages (21,38,114,184,185,220). Furthermore, it was indicated that a strong correlation existed between pulp swelling and the development of strength in the dry sheet of paper, while no relationship whatsoever was found between fiber swelling and freeness.

The differences in swelling ability among the four pulps achieved through beating seem to proceed in a way very similar to the differences observed when comparing their surface areas. The birch pulp is already much more swollen in the unbeaten state than all the other pulps, and also develops this property faster, although at a rate very close to E. rostrata. Again a very similar behavior of E. grandis and pine pulps can be noticed, as far as their rates of increase in specific volume at low load is concerned.

The variation in specific volume and specific surface of the pure fibers with applied energy can be better visualized in Figures 20 and 21. In these figures it can be observed that although the rates of development of specific surface of fibers of both eucalypts are practically the same, as discussed earlier, they differ significantly in the

way the specific volume is changed. Therefore, besides the differences in the rates of fines formation and the specific surface of these fines, which accounted for the main differences of the surface areas of E. grandis and E. rostrata pulps, a difference in their swelling behavior is also noticed at the fiber level. Reasons for this can be sought in the fiber's morphological and chemical characteristics, as discussed below.

Incidentally a higher load does not seem to change the relative differences among the hardwoods (Figure 21). However, as observed in Figure 17 and by comparison of Figures 20 and 21 the pine fibers seem to benefit largely from a higher load towards the development of specific volume. Associating this fact to what was observed for the changes in specific surface with load it can be concluded at this point that a higher beating load seems to help significantly in the treatment of pine fibers, with better utilization of energy for changes in the internal and external structures. For the three hardwoods, however, apart from a faster development in external fibrillation and the production of fines, a higher load does not prove beneficial.

To form a better picture of the changes occurring in the pulps it is also interesting to notice the behavior of the fines. Using the additivity rule described earlier

the values of the specific volume of fines were also calculated and are listed in Table XIV. First of all it is found that even the primary fines show already much larger values than the pure fibers. As beating proceeds there is a rapid initial rise until the first beating point is reached, followed by a gradual and slow decrease in the swelling abilities of these fractions, although not to the level of the primary fines. This behavior is in agreement with the picture of development of fines discussed earlier and the reported values for the distribution of hemicelluloses across the fiber wall (126,141,159), following the accepted mechanism of xylan redeposition in the later stages of kraft cooking (34,242). Since the hemicelluloses have a high tendency to swell in water, as soon as beating starts and the fibers are slightly brushed, secondary fines rich in hemicelluloses are created, therefore increasing dramatically the water holding capacity of the total fines fraction. With the progress of the beating the detached fibrils have decreasing hemicellulose contents as more internal layers of  $S_1$  are peeled off, with relatively smaller specific volume values for the fines fraction. It is also interesting to notice that when beating starts, at higher loads the swollen volume of the fines is slightly higher than that at low load but the former tends to reach

TABLE XIV. CALCULATED SPECIFIC VOLUME OF FINES,  $\text{cm}^3/\text{g}$

PFI Revolu- tions	Beating load $\text{kN/m}$	Southern pine	Silver birch	<u>Eucalyptus grandis</u>	<u>Eucalyptus rostrata</u>
0	-	8.14	17.00	15.12	8.95
500	3.33	-	20.96	19.14	10.86
	4.90	-	21.77	19.60	11.53
1500	3.33	16.27	20.42	18.79	11.68
	4.90	17.53	19.94	18.01	11.31
3000	3.33	15.22	17.86	17.63	12.75
	4.90	15.97	17.27	17.45	12.81
5000	3.33	15.37	17.45	16.51	12.82
	4.90	12.94	15.59	16.21	12.02
8000	3.33	13.35	-	-	-
	4.90	11.33	-	-	-

lower values at later beating stages, in a way confirming the conclusions reached previously, that the main effect of a higher load was to act upon the surface of the fibers at a higher rate.

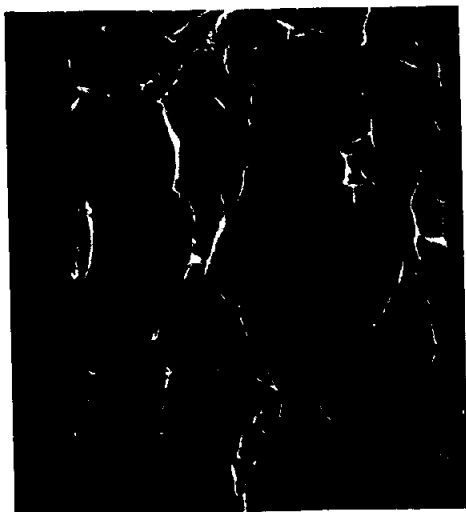
The calculation of the swollen volume of fines by difference has been criticized on the grounds that in the whole pulp a part of the immobilized water is probably shared between the fines and the fibrils of the fibrillated fibers (226). However, since according to this postulate the calculated values are much smaller than the actual ones, the data here reported are considered valid for the purpose of showing the differences between the fractions as beating proceeds. Even if those values are considered conservative the outstanding water holding capacity of fines as compared to fibers is noticeable. Though the changes in specific volume with beating energy proceed very similarly for whole pulps and pure fibers, the fines seem to play a major role in the pulp's interactions with water, because of both their extremely large specific surfaces, and their large volumes of associated water.

The characteristics noticed in the four pulps' pure fibers and fines fractions at various beating stages are fundamental for the understanding of the beating process. General trends can be related to the basic mechanisms underlying the refining, while the individual pulp's behavior is

believed to be caused by each pulp's morphological and chemical characteristics.

As observed with all the four pulps the main utilization of the energy applied in the beater to the fiber networks seems to be directed towards changes in their water retention abilities. This mechanism appears to proceed independently of how the surface of the fibers is modified. Comparisons of electron micrographs of surfaces of unbeaten and lightly beaten (500 revolutions PFI) fibers showed only minor changes (Figures 22a, 22b, 23a, 23b), which confirmed the results obtained from the surface area measurements. The primary wall had been peeled-off in most fibers and some fibrils detached from the outer  $S_1$  layer. These were very short however, creating rather a "fuzz", and not an actual fibrillation. Major changes in the fiber surfaces could be noticed only with heavy beating (Figures 22c and 23c), with a high degree of detachment of  $S_1$  layer in most fibers and, in birch an almost complete peeling of this layer.

Thus, the rapid increase in specific volume in the early stages of beating cannot be explained by the externally held water, since at this level of treatment the external fibrillation is very incipient. Changes in the internal structure of the fibers are more likely to be the main factors responsible for the increase in specific volume, as energy is applied to the fibers.



1.0 μm

a. SEM - Silver birch, unbeaten.



1.0 μm

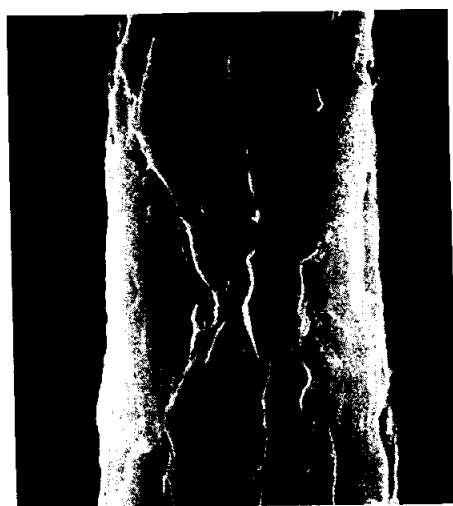
b. SEM - Silver birch, beaten with 0.45 MJ/kg.



1.0 μm

c. SEM - Silver birch, beaten with 4.06 MJ/kg.

Figure 22 - Scanning Electron Micrographs of the surface of silver birch fibers at three beating levels.



a. SEM - E. rostrata, unbeaten.



b. SEM - E. rostrata, beaten  
with 0.42 MJ/kg.



c. SEM - E. rostrata, beaten  
with 4.07 MJ/kg.

Figure 23 - Scanning Electron Micrographs of the surface of E. rostrata fibers at three beating levels.

Following the hypothesis of treatment of flocs, rather than single fibers between the beater bars, (12,56,70,84,174,208) it is noticeable that the swelling behavior of pulps proceeded in a way very similar to the reduction in the beating gap with energy applied, i.e. there seemed to be a dependence of both phenomena on the logarithm of beating energy. A relationship between the status of the pulp observed in the beater and changes occurring in the pulp during refining was thus thought as very likely, and could be a part of a more general picture of the beating process.

As indicated in Figure 24 there seems to be a straight line relationship between the changes in beating clearance and the variations in specific volume of the pulps. These correlations were found to be different for each pulp, but independent of the load applied. A possible explanation for these behaviors can be based upon the ideas formulated recently by Steenberg (208). As the flocs are compacted under the huge compressive stresses between the beater bars they will escape from the action until a threshold consistency is reached and the flocs consolidate. When this is accomplished the forces imposed by the beater bars will be utilized efficiently in promoting changes in the fibers through mechanical deformation.

The proposed escaping or "oozing" of the flocs is very similar to the beaterman's squeezing test. In a floc

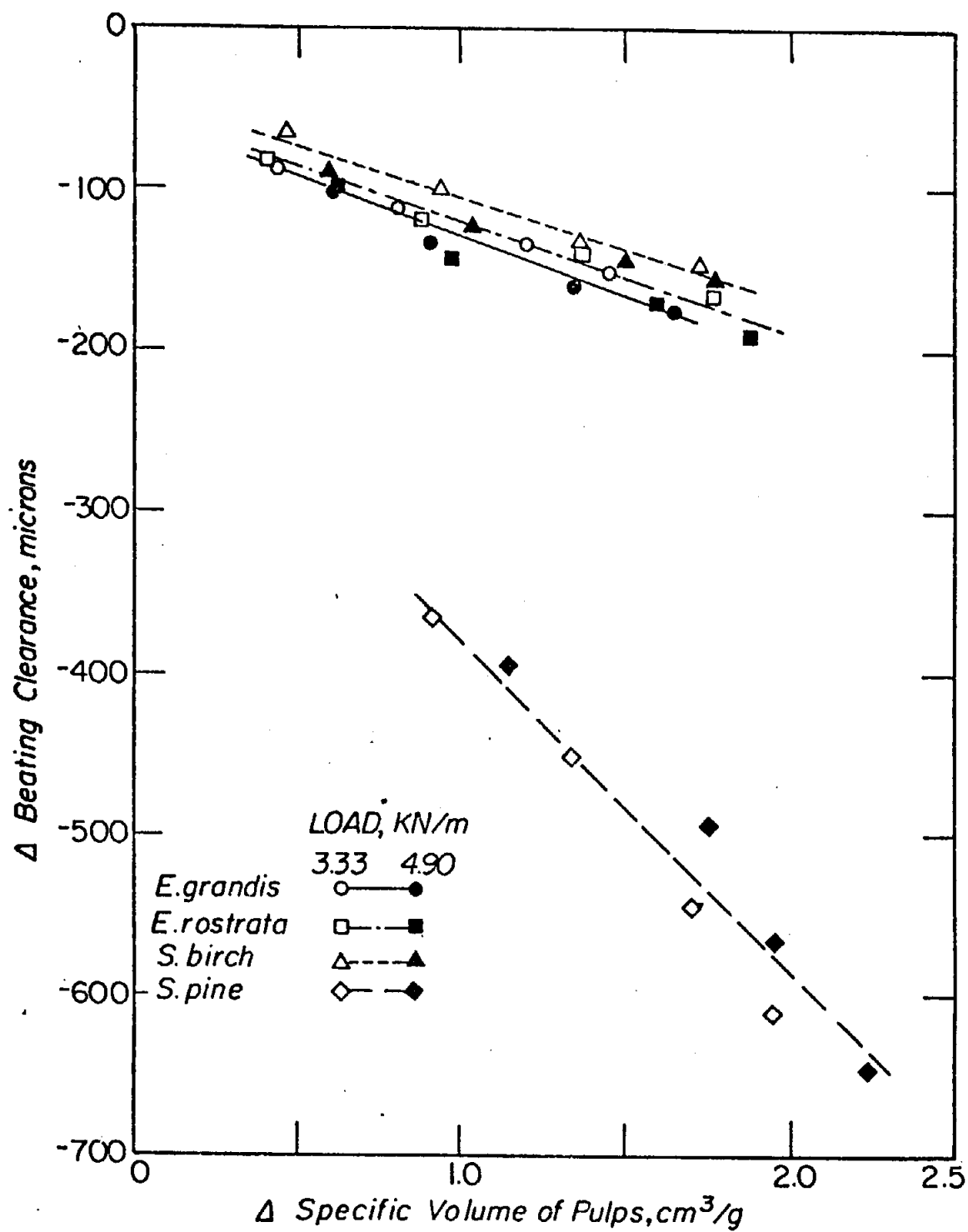


Figure 24. Relationship between variation in beating clearance and variation in specific volume of whole pulps.

made up of unbeaten fibers most of the water is not yet closely associated with the pulp. Therefore, as the floc is compressed, water can flow easily from the beating gap, providing fast consolidation while most of the fibers in the original floc are still in the gap. At this stage it is mostly water that escapes and the following consolidation and stressing of the floc will bring about the reduction of the gap and exert work on the fibers. The bigger and more coherent are the flocs the larger will be the gap reduction before consolidation. As beating advances and the fibers get worked up and swell by increasing their water holding capacity, the expression of water from the floc by compression becomes increasingly more difficult. As a result more fibers will flow from the beating area as consolidation proceeds, resulting in less fibers between bars and a smaller gap, also due to the higher floc compressibility at this stage.

The main effect of beating therefore seems to occur at an early beating stage, when the flocs consolidate faster due to the rapid expression of water, and the applied stresses can be distributed through most of the fibers. It can thus be postulated that the changes in beating gap occur as a consequence of the water holding or hydration capacity of the pulp, as indicated by its specific volume. The treatment of fibers as flocs and the changes occurring in the gap are believed to be important steps in the beating process

at a macroscopic level. The phenomena discussed above are believed to occur generally for all four pulps studied, while a particular behavior will depend on each fiber's response to treatment. Of course the properties of flocs are expected to be more complex than the ones depicted here. They must be included in the study of the beating process as macroscopic entities, with simultaneous inclusion of shear and compressive forces in the analysis of their mechanical behavior. A better understanding of these phenomena, as provided by new research in this area, would contribute significantly towards a more advanced beating theory.

#### The Influence of Fiber Properties

Macroscopic Level. The discussion on the behavior of flocs as function of pulp swelling and its influences on the beating clearance was applicable to all four pulps in general. Further analysis on additional properties of flocs is beyond the scope of this study, although some considerations about their particular behavior as influenced by fiber characteristics are afforded by the results presented here.

One of the first aspects to consider is the size of the fiber aggregates. As expected from the average fiber length in each of the studied species the size of the flocs varied (as observed visually) in the following order:

E. rostrata < E. grandis < silver birch < southern pine, at the consistency used in the PFI mill. This fact might have important consequences, such as the well-known tendency of hardwood fibers to escape from the beating action because the small flocs are able to concentrate in the grooves between the refiner bars. Mill practice has proved advantageous to refine these pulps, particularly eucalypts, using tackles equipped with many small bars and shallow grooves (135), which, among other things, diminishes the probability of flocs to hide away in the spaces between bars. Even under the conditions in the PFI mill, where stock flow is very restricted, in the case of the three hardwoods some pulp was always found in the spaces between the bars at the end of the beating, while this was never observed with the pine pulp.

Floc formation and stability are also thought of as important factors controlling the absorption of energy by the fibers. As the floc is stressed it must be able to resist deformation so that the applied energy can be absorbed by the fibers. If the floc yields too easily there will be less opportunity for straining the fibers. With very short and stiff fibers it seems very unlikely that the eucalypts would form stable and cohesive networks, therefore diminishing the possibility of being activated. With these characteristics, a higher probability of the fibers being impacted would possibly be warranted by promoting the formation

of stable flocs from eucalypts between the refiner bars through increased consistencies, plus a restricted flow of stock from the refining zone, and use of convenient tackles, as industrial experience has reported (51). The flexible and somewhat longer birch fibers are expected to entangle more readily than eucalypts, therefore providing stable structures to be impacted by refiner bars. For southern pine fibers the stiffness of the latewood fibers associated with the higher likelihood of entanglement provided by the large fiber length (and length-to-width ratio) and flexibility of the earlywood fibers should set up conditions for the formation of networks of considerable cohesion and strength. Being also very large these flocs would require more energy for deformation and consequent straining of the fibers. This could be a possible explanation of the better beating results for pine pulps obtained at a higher load.

In addition to floc size, ease of formation and stability the number of fibers per gram should also be considered. If it is assumed that between the beater bars the same amount of pulp is being treated at each impact, a larger number of fibers per gram of pulp will provide a large area for stress distribution when the flocs are stressed. In this respect the eucalypts show a distinct disadvantage, as E. grandis and E. rostrata have respectively 63 and 103% more fibers per gram than birch and approximately 12.3 and 15.4 times that of southern pine,

and consequently smaller probabilities of activation at the fiber level. The pine pulp seems to be especially favored from this point of view, but it is expected that more energy will be consumed during compaction and straining of its large and cohesive networks, thus offsetting this apparent advantage of small number of fibers.

More study on the flocs is necessary before any definite conclusion can be reached as to the role of their behavior in beating. Yet the macroscopic considerations made earlier allow the understanding of some of the theoretical backgrounds for optimum operating conditions during beating of pulps of the same type as studied here. This can be noticed in the observed advantageous use of higher specific edge loads for softwoods and lower loads for hardwoods (139). For hardwoods disc patterns with numerous bars and grooves are usually recommended, and especially for eucalypts better results are obtained when refining is carried out at higher than normal consistencies and under pressure.

Microscopic Level - Fiber Structure - Obviously the changes occurring in the fibers at a microscopic and ultra microscopic levels should be related to the response of flocs, in the beating gap. This, in turn, affects the way further treatment of the fibers proceeds, as limited by the oozing and consolidation processes. As discussed previously the main effect of beating at an early stage of

energy absorption seems to be the increase in the water holding capacity of the pulps, both at the fiber level and through the production of highly swollen fines. These changes followed general trends, as observed for all four pulps, although each of them differed in the extent of variation. This aspect is better understood by comparisons of the different fiber characteristics.

The study of beating has always been limited by the difficulties in analyzing its effects on fibers. While it was always relatively easy to examine the fibers under the microscope and detect the external fibrillation, the changes occurring inside the fibers were assumed without being backed up by experimental evidence. Equally, the well-known increase in hydration (or wetness) of the pulp achieved in beating and which was explained by the retention of water by the fibrils raised from the fiber surface was missing factual support. Moreover, this concept could not explain the rapid rise in sheet strength occurring in the early beating stages, because at the beginning of beating little external fibrillation was evident.

The concept that the most important factor for water uptake was a consequence of cell wall delamination followed by water imbibition and swelling (62) has gained increased recognition since the development of methods to indicate these changes in the fiber (114,184,195,213) and experimental evidence for the proposed splitting of the cell wall

into a series of co-axial lamellae (155,175). More recent studies have confirmed the patterns of ultrastructural organization in the cell walls of different fibers (120a,187, 194,196) which allows delamination to start during pulping and proceed, although more dramatically, at the onset of any mechanical action.

This theory seems to apply to the results obtained in this study, although it should be kept in mind that all pulps used were originally in dry form. Hence, the loosened up and separated microfibrils in the freshly cooked pulps were drawn back together upon drying, probably this time into a more compacted state, since all the interfibrillar lignin and some hemicellulose were removed. The remaining hemicellulose should prevent aggregation of the microfibrils to a certain extent, and its occurrence in larger quantities might reduce the possibility of strong cellulose-cellulose bonding during microfibrillar association.

These mechanisms might have direct impact on the behavior of the cell wall when the fiber is loaded. As the flocs are consolidated they are subjected to a complex set of huge stresses. These, in turn are transmitted from fiber to fiber until the whole structure cannot bear the stress concentration any more. Ultimately the stresses are relieved, as work is absorbed by the fibers, and thus changes are produced in their structure. In agreement with Steenberg's suggestions (208) the flocs should then reach a new configuration, by

rearrangement in the disc grooves, as a preparation for a new loading process.

Work on fibers might be used for relieving internal stresses too. If bonds exist among components of the fiber's internal structure the stress will be concentrated on these links which prevent the fiber from deforming in a completely plastic fashion. These bonds are mostly hydrogen bonds between the microfibrils, and their cleavage will depend on the degree of microfibrillar association and bond density.

The concepts just dealt with apply closely to observed changes during beating and the corresponding fiber characteristics found in this study. Thus, the birch fibers were shown to have a larger value of specific volume than all the other pulps in the unbeaten state, and this value was further increased at rates much higher than in the case of the two eucalypts and the southern pine. This behavior can be expected from birch's high hemicellulose content and large fibrillar angles, with accompanying large crystallite widths and a less packed microfibrillar arrangement. As the fibers are loaded a small number of microfibrils can be separated but more efficiently as stresses are concentrated over a smaller area, and this is also aided by large amounts of hemicelluloses in the intermicrofibrillar spaces as shown by Rånby (179a).

As cellulose-cellulose and cellulose-hemicellulose-cellulose hydrogen bonds are being broken water enters the structure, preferentially into the more accessible (101)-tangential planes between the microfibrils, forming new hydrogen bonds and swelling the hemicellulose matrix. According to a swelling theory (32), as the hemicellulose becomes a gel a considerable osmotic pressure due to swelling also comes into play, with consequent action on cellulose-cellulose bonds which restrict further swelling. So it can be assumed that the more hemicellulose is present in the fiber wall and the more water is associated with it, the easier will become the separation of fibrils. With all these mechanisms coming into play in birch's fibers with high hemicellulose content and low density cell walls it is possible to understand the fast increase in the swelling ability observed for this pulp.

The different behavior of E. grandis and E. rostrata in beating is also very interesting. Of particular importance is the fact that although the geometry of their fibers are different, their ultrastructural organizations are nevertheless very similar. Both types of fibers exhibited steep helices in their respective  $S_2$  layers, slightly thicker cell walls when compared to birch, and very close packing densities. Following the trends discussed previously these fibers are accordingly expected to have smaller crystallite widths and a higher degree of packing of microfibrils than

birch, as already indicated by their differences in cell wall densities. After pulping, bleaching and drying the fibers of these species might then end up with a higher degree of structural integrity than birch fibers. Therefore as loads are imposed on eucalypt fibers the smaller and more numerous microfibrils will offer considerably greater surfaces for stress distribution than in the case of birch fibers. In addition, the lower content of hemicelluloses would be expected to increase the possibility of cross-linking among the microfibrils, which require larger energy expenditures for cleavage, and decrease the plasticization ability of the fibers at a given energy expenditure.

These effects are particularly important in the case of E. grandis pulp, which has a hemicellulose content even lower than E. rostrata. The higher hemicellulose content of the latter appears to be mostly responsible for the higher rates of development of specific volume of E. rostrata fibers, in agreement with the picture of the influence of hemicellulose on the ease with which internal bond breakage occurs. Therefore although the differences between the changes in swelling occurring in birch and the two eucalypts can be attributed to both ultrastructural and chemical factors, when comparing E. grandis and E. rostrata the difference in hemicellulose contents seems to be the main responsible one. This finding brings new evidence to the influence of hemicellulose in the

beating process, since the results here reported are independent of other factors which impair the comparison of pulps with different hemicellulose contents, such as large differences in morphology or structural changes inevitably occurring in studies involving hemicellulose removal (31).

The interpretation of results obtained for southern pine is made more complicated due to the presence of both earlywood and latewood fibers, the latter not present in the eucalypts, and only in very small quantities in birch. As discussed previously the latewood fibers have ultrastructural characteristics resembling those of the two eucalypts, while earlywood fibers are structurally more similar to those of birch. However, it is also typical to southern pines to have a much higher content of latewood when compared to other conifers. As a consequence, when considering the weighed contributions of earlywood and latewood fibers to a calculated average value representative of the pulp it is seen that the southern pine fibers could have an overall behavior ultrastructurally close to eucalypts (Tables IV and VI). Indeed, the results obtained for southern pine were similar to E. grandis, with rates of increase in specific volume of the two pulps very close to each other.

It is possible that the observed similarities between the pine and E. grandis are also highly influenced by their comparable hemicellulose contents. Even though the pine is

poorer in xylan, which is believed to be the hemicellulose mostly responsible for swelling, its glucomannan content should make up for the difference and keep the microfibrils in a state of aggregation very similar to that occurring in E. grandis fibers. So, it is very likely that the fibers from these vastly different species could end up with very similar ultrastructural characteristics, which are reflected in their similar delamination and swelling abilities, as indicated by the changes in specific volume. On the other hand, it should be remembered that the flocs of these two pulps have very different characteristics, and could be responsible for the small differences still observed between the pulps in question, and for the special behavior of the pine at a higher load.

As for the development of specific surface area, it is tempting, in view of the previous discussion, to analyze it as one of the consequences of a progressive loosening of the fiber structure. As a matter of fact it has already been proposed that the external fibrillation could occur after the fibrils are separated in the internal structure of the fiber, making their detachment from the surface easier (75). Due to the inherent stochastic characteristic of this mechanical treatment it is highly probable that many of the beating effects occur almost simultaneously. Actually since all these effects can be classified as deriving from the creation of new surfaces (56), of which creation of new particles

and generation of structural damage are particular cases, it becomes difficult to envisage the occurrence of the various beating effects in a completely independent fashion.

The results have shown that for all pulps the developments of internal fibrillation and swelling and of external fibrillation proceed in different ways, but they do occur at the same time. Especially, when observing pure fibers it is seen that at an early beating stage their specific volume has increased significantly (e.g. 20% for southern pine, 28% for birch, 26% for E. grandis and 27% for E. rostrata for 1.2 MJ/kg applied), while the specific surface areas show somewhat smaller relative increases at the same energy input (18% for southern pine, 21% for birch and 15% for both E. grandis and E. rostrata). With continuation of the beating the rate of development of specific volume tends to diminish gradually, indicating that a limit will be reached when the fibers have been worked up so much that they are very swollen, and when the flocs under impact ooze easily from the beating zone no more useful work can be absorbed by the fibers. On the other hand the specific surface of fibers increases continuously with beating, and as the fibrils are detached, fines of increasingly higher surface areas are formed.

As a consequence, although it is possible that as more delaminated and swollen fibers are formed the outer fibrils

may be loosened up more easily, this process seems to proceed almost independently of what is happening internally to the fibers. This is better understood by microscopic observation of the fibers at different beating stages, whereby indication was obtained that the external fibrillation proceeded through peeling of only the  $S_1$  layer, at least at the levels of beating used in this study. Since most of the changes in internal structure have been observed in the  $S_2$  layer (120a,155,175,194,196,187) (which comprises the bulk of the cell wall), it seems very unlikely that the delamination in  $S_2$  would have a direct influence on the way the  $S_1$  layer is being removed.

The mechanism of development of external fibrillation could be understood as being mostly a consequence of the friction occurring between the fibers as the flocs are sheared and compressed. The resultant from these interactions would depend on characteristics of the fibers'  $S_1$  layers, among them their physical integrity (as influenced by their hemicellulose content, and the state of aggregation of the microfibrils), and their orientation relative to the fiber axis. In addition, the development of specific surface could be dependent on the way the flocs behave under stresses, i.e. how the relative contributions of shear and compression forces influence the extent of interactions among the fibers.

The morphological properties of the fibers seem to

corroborate the considerations above. Considering for instance, birch fibers, it is seen that external fibrillation increases at rates higher than in all other pulps here studied. This behavior can be explained by the less aggregated fibrils in the outer  $S_1$ -layer, because of the higher hemicellulose content of these fibers. The angle in  $S_1$  helices might also play an important role, since the force necessary for peeling each filament would increase very significantly as this angle becomes steeper, as indicated by similar previous considerations (223). The large  $S_1$  fibrillar angles observed in birch fibers would thus make the whole process of fibril detachment much easier than in the other fibers studied.

The fibers of the eucalypts were shown to fibrillate externally in a very similar fashion, although their internal structures were reacting differently. These observations seem to confirm the earlier conclusion about the possible relationships between the increases in specific volume and in specific surface on beating. As also discussed previously the fibers of E. grandis and E. rostrata have very similar ultrastructural characteristics which could explain their almost equal rates of increase in specific surface at the fibers level. The higher degree of order in fibril aggregation associated with steeper fibrillar angles in  $S_1$  layer might have been the main reason for the observed limited degree of fibrillation of these fibers when compared

to birch, especially when considering that both eucalypts had thinner  $S_1$  layers than birch, and yet they were not completely peeled-off as noticed with the latter. An additional reason for this similar response of the eucalypts to the beating may be found in the behavior of their flocs in the beater and the large number of fibers per gram, which could interact to a smaller extent than the more flexible birch fibers.

Another fact differentiating the two eucalypt species is the larger amounts of fines formed when beating E. rostrata, as compared to E. grandis, particularly when it is remembered that both types of fiber developed external fibrillation very similarly. Since it was observed for all pulps and concluded previously that the fines formed were derived from the fibrils raised from fiber's surfaces, it is possible that somehow the fibrils detached from the surface of E. rostrata's fibers were easier to be broken up and separate than those of E. grandis. An additional explanation may be the fragmentation of the more numerous vessel elements from E. rostrata as compared to those of E. grandis (17 and 6 vessels per  $\text{mm}^2$  of wood cross section respectively), as also indicated by the smaller values of the specific surfaces and volumes of fines of the former at early beating stages.

As to the results obtained with southern pine, account should be made for the presence of earlywood and latewood fibers. Microscopical observation indicated that earlywood

fibers were very easily disrupted externally while latewood tracheids were more difficult to develop external fibrillation. When considering a weighed average value of early and latewood characteristics as representative of the pine pulp, it can be observed that its fibers develop specific surface at rates even smaller than the eucalypts. Thus, the ultrastructural and chemical similarities with E. grandis cannot solely account for the beating results obtained here. The larger and more compressible flocs of southern pine might need a great deal of impact for consolidation and consequent absorption of energy by the fibers. As a consequence most of the work applied may be absorbed towards deformation of the floc and the more readily accomplished modification in the fibers' internal structure. In this way higher loads would be able to provide the necessary energy for floc consolidation and fiber straining at increased rates, thereby increasing the degree of shear imposed on the fibers and their subsequent more efficient interaction. In fact, this has, indeed, occurred with southern pine, for which the application of a higher load was proven to be very beneficial. Under these conditions it was observed that the rate of development of external fibrillation in pine fibers was very close to the ones of eucalypts fibers (Figure 21).

It may thus be concluded that the beating process can be monitored quantitatively by the changes in the pure fibers' specific surfaces, specific volume and the

corresponding increases in whole pulps. The amounts and characteristics of the fines fraction are of primary importance for the understanding of the mechanisms involved in the process. Therefore, the measurement of any pulp property providing an actual indication of changes occurring during beating or refining should be performed on both the whole pulp and the fibers separated from fines.

Changes occurring in fibers can be related to their morphological and chemical characteristics, but the extent to which the fibers interact and form networks should not be neglected, because the so-called flocs are indeed the structures which are impacted by the beater bars. Their properties not only influence the action in the beating gap, but also seem to control the adequate absorption of energy by the fibers. It seems possible that the proposed beating theories involving the relative severity and number of impacts are actually suggesting ways of a more efficient treatment of flocs of different characteristics without knowing actually why.

As flocs and the fibers composing them are stressed, the applied energy seems to be utilized in different ways, although as absorbed by the pulp the effects seem to be all evident in the creation of new surfaces. The most important of them seems to be the delamination of the internal fiber structure with consequent water imbibition and swelling. This increase in water holding capacity is believed to

change the behavior of the flocs, which in turn affect the way the beating action proceeds. The rate of load application seems to affect the way fiber networks consolidate and how the fibers interact, particularly affecting the rates of increase in external fibrillation and fines formation. The bigger flocs of southern pine showed distinctly better absorption of energy for fiber modification at a higher load than the smaller flocs from the three hardwoods.

Concerning the question of energy transfer to the fibers, it seems that major accomplishments could be reached after a thorough analysis of fiber networks under stresses. At the fiber level the results indicate that the logarithmic dependence of development of internal fibrillation with beating energy may be related to failure mechanisms by fatigue. In this respect the concepts developed by those who promote the analysis of beating through intensity and number of impacts (23,49,134) are very applicable, but they should definitely include in their considerations the variables affecting the behavior of the flocs, as also influenced by fiber properties.

It should now be of considerable interest to see how the results based on permeability measurements and other fiber properties would be applicable to some of the processes involved in papermaking.

## Effects of Beating on the Behavior of Pulps

Drainage Properties. The removal of water in the forming zone of papermachines (i.e. from the headbox slice to vacuum boxes) is of critical importance to the speed that the machines can reach, therefore influencing directly the efficiency of the whole process. As mentioned previously the freeness test was designed to monitor the changes occurring during beating, more specifically to give an indication about the drainability of groundwoods. Nevertheless this measurement has been applied indistinctively to both chemical and mechanical pulps, not only as a means of testing the drainability of the pulps in the wet-end but also as an estimate of the degree of beating imparted to a pulp.

As experience has indicated the applicability of the freeness test is very limited in the case of chemical pulps (33). Particularly when assessing the beatability of different pulps comparisons at the same freeness can be very misleading (144). It is well known that different types of pulps show different initial freeness values and also the freeness does not illustrate the rate with which the pulp is beaten. The latter is rather a measure of how easily the cell wall delaminates internally and swells under the repeated application of stresses, which the freeness tester was not designed to account for.

When considering only the drainage properties of the

accurately the dewatering behavior of pulps (27), mostly because the method lacks the conditions normally present on the wire of the papermachine, in particular the elevated suction pulses in table rolls and foils (73,228). Improvements over this limitation have led to the design of various drainage tests (92,116,217), which have attained only limited acceptability. In addition to the presence of high vacuum, the recognition of the turbulence of the stock while it drains over the wire as an important variable affecting drainage and its introduction in a drainage tester (28) may prove very advantageous in predicting the behavior of the stock in the forming zone.

This newly developed tester was utilized in the present research on all whole pulps and the results obtained are listed in Table XV. A rapid look at the data gives already an indication that the resistance of the pulps to drain is not developed gradually with the advancement of beating, but rather remains stagnant at the early stages and then increases dramatically as beating proceeds. The influence of a higher load is also noticeable, particularly at very high beating levels. A plot of the logarithm of the drainage time versus CSF (Figure 25) shows that freeness alone cannot explain the drainage behavior of the different pulps. Each of them presented a specific drainage rate at a given freeness, and, in addition these relationships were different at each applied beating load.

TABLE XV. DYNAMIC DRAINAGE TIME OF WHOLE PULPS - Seconds

PFI Revolu- tions	Beating load kN/m	Southern pine		Silver birch		<u>Eucalyptus grandis</u>		<u>Eucalyptus rostrata</u>	
		Whole Pulp		Whole Pulp		Whole Pulp		Whole Pulp	
0	-	7.6±0.5		10.8±0.7		11.2±0.8		13.4±0.8	
	3.33	-		11.0±0.7		11.4±0.8		14.0±1.1	
500	4.90	-		11.5±0.8		11.5±0.8		16.0±1.0	
	3.33	7.9±0.4		11.2±0.7		11.7±0.7		19.0±1.3	
1500	4.90	9.7±0.5		12.6±1.0		13.1±0.8		22.1±1.7	
	3.33	8.5±0.5		17.0±1.3		14.6±0.9		26.1±2.1	
3000	4.90	13.1±0.6		23.2±1.8		18.1±1.0		32.3±4.3	
	3.33	12.9±1.1		28.9±2.1		20.0±0.8		41.1±3.2	
5000	4.90	79.0±3.2		227.0±8.4		36.5±1.3		387.0±15.6	
	3.33	123.0±5.3		-		-		-	
8000	4.90	1176.0±25.5		-		-		-	

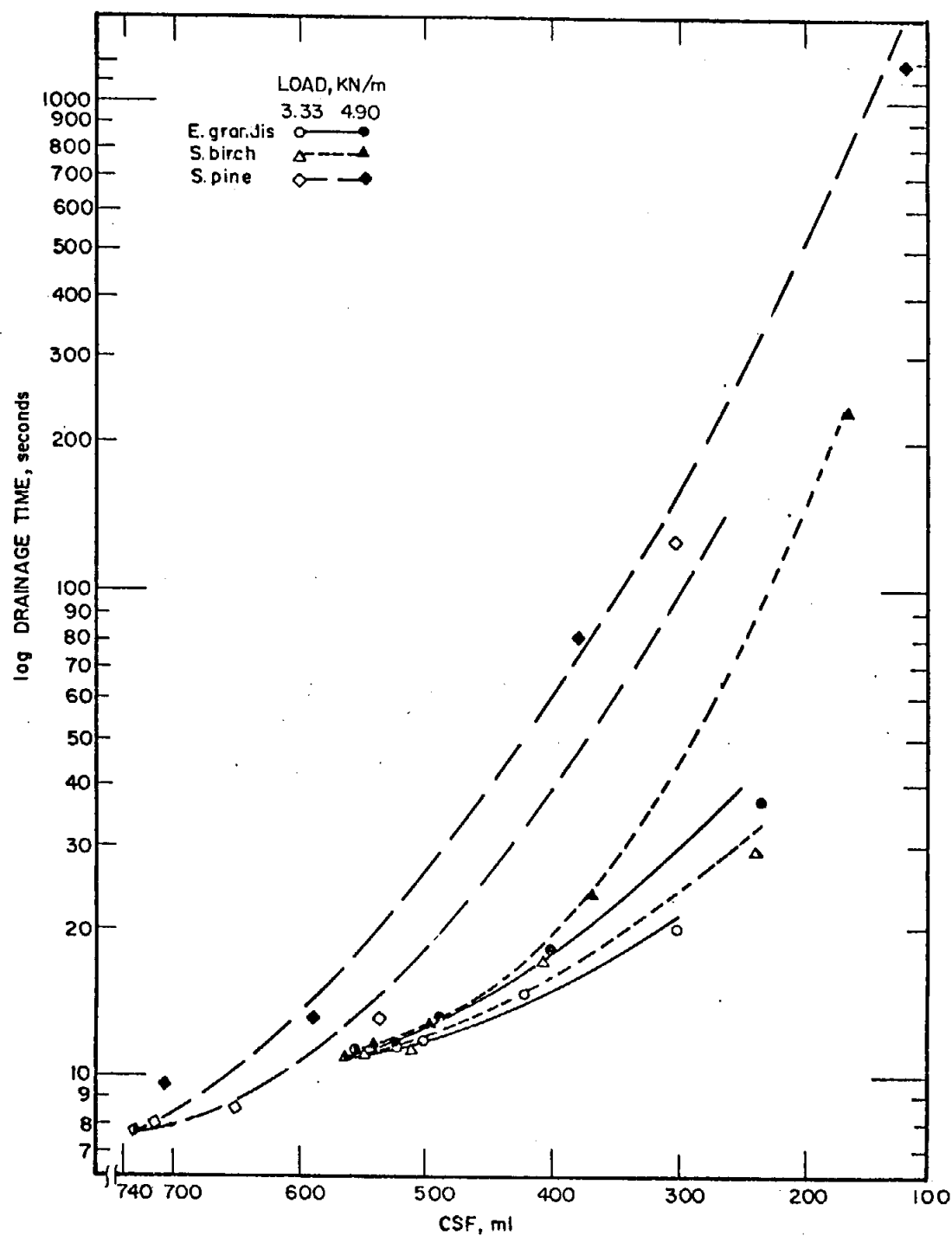


Figure 25. Relationship between the logarithm of dynamic drainage time and Canadian Standard Freeness of whole pulps.

This fact could be noticed especially at medium to very advanced beating points, although for the hardwoods the points were somewhat clustered at the early beating stages. Thus if a comparison of these pulps was desired at a certain freeness level (e.g. 400 CSF) they would have to be beaten to different extents and a completely different behavior would be obtained for each of them in a number of desired properties, starting at their different characteristic of water removal in the forming zone. Hence it is seen that E. grandis drains at the highest rate, followed by birch and E. rostrata (omitted from the figure), and then southern pine, with much longer drainage times at a certain freeness.

The mechanisms of water removal from a stock, as the mat is being formed on the wire, are better understood by analyzing the process as being predominantly that of filtration (176). Consequently the drainage time measurements could be related to the filtration resistance as derived by Ingmanson & Andrews (107), with basis on Darcy's law. This relationship has demonstrated that the filtration resistance of a pulp mat is mostly dependent on the square power of the average specific surface area of pulps beaten under different conditions (18,87), although the derived equations included also a number of other pulp characteristics. In addition it was observed that this dependence was essentially linear. Although the influence of original fiber form on the drainage resistance may be also considered an important

factor, it has been recently reported that for beaten pulps the fine material formed has a more decisive influence on the flow resistance of the fiber mat, with reduced effect of fiber shape (7).

By considering the freeness measurement as essentially that of a filtration process it has been indicated in a very recent study (61) that CSF is also a very complex function of pulp variables of which the specific surface would be the most important, while relatively insensitive to changes in mat density and in specific volume. The results in Figure 26 have shown such linear relationships between CSF and the specific surface of the corresponding pulps, although different curves were obtained for each of them when loads of 3.33 and 4.90 kN/m were applied, and not single curves for specific surface vs. freeness for each value of specific volume as indicated by El-Hosseiny and Yan (61).

As seen also in Figure 26, at each level of freeness the pulps show very different values of specific surface, the lowest values being observed for E. grandis (as well as E. rostrata, which was excluded from this figure for the sake of clarity), followed closely by birch and finally by the southern pine. It was also noticed that at a given CSF level the pulps beaten under a higher load unexpectedly showed lower specific surface values. Different curves of drainage time vs. freeness and freeness vs. specific surface (Figures 25 and 26) for each pulp when beaten in the same

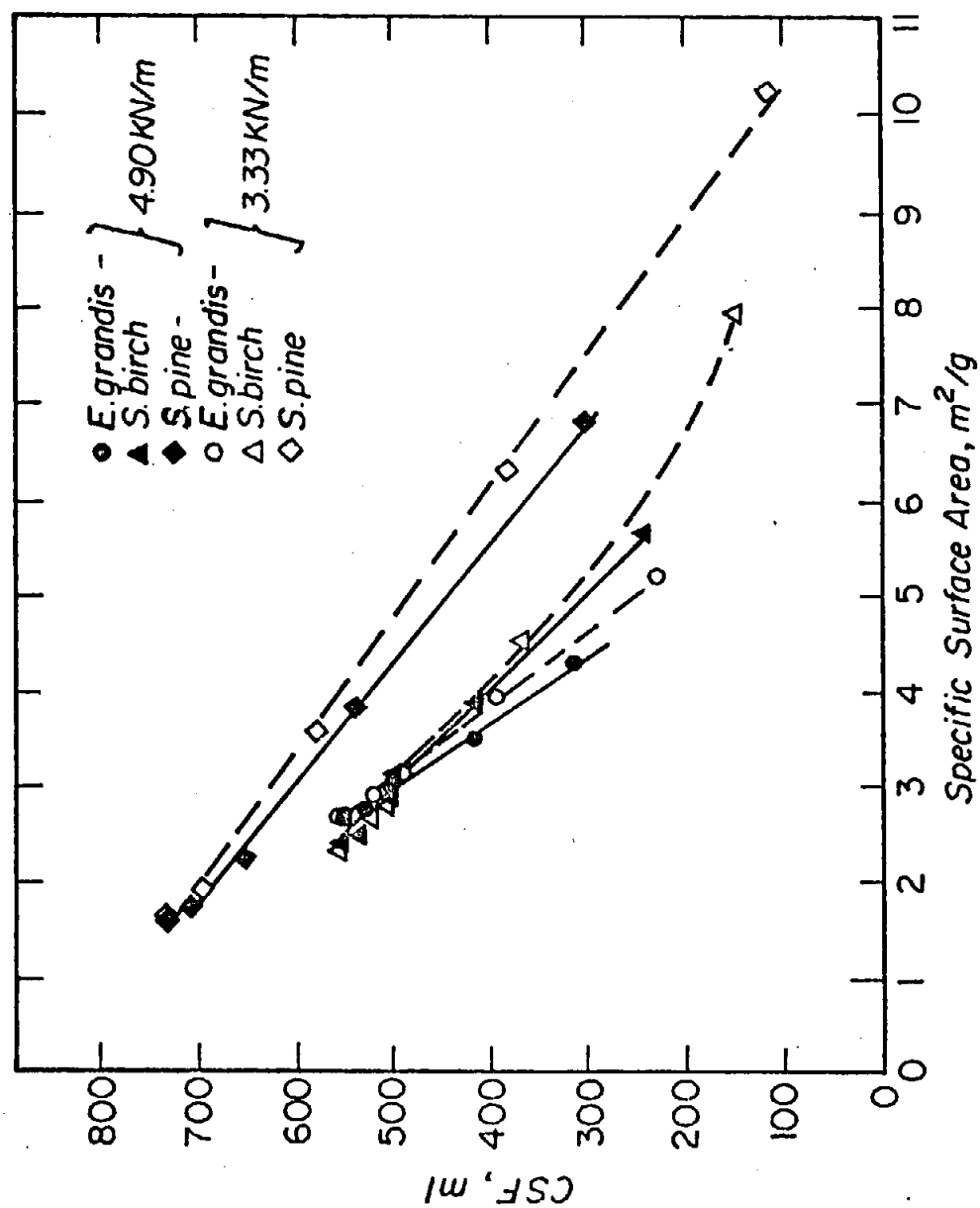


Figure 26. Relationship between Canadian Standard Freeness and Specific Surface of whole pulps.

equipment, under different conditions, may be seen as indications that CSF involves a number of variables probably interrelated in a very complex way, and not explained explicitly by the simple result of the measurement.

The previous findings of a linear relationship between the filtration resistance and the square power of the specific surface were initially checked for the results of this study. An excellent correlation between dynamic drainage time and  $S^2$  was obtained for all the pulps, even for different beating loads, although the relationship was not linear, but rather the curve shifted upward. Observations of the newly reported effects of turbulence on drainage (28) indicated that this variable somehow produced drainage times increasingly longer. Under constant pulp characteristics it was observed that as turbulence was enhanced the drainage time increased in an almost exponential fashion. It was suggested then, that the retarding effect of turbulence on drainage resulted from a fines-rich deposit on the screen.

Therefore the observed upward shift in the drainage time vs.  $S^2$  curve for the results reported here could be mostly due to the effects of turbulence, a variable not included in previous experiments related to filtration resistance. By plotting the logarithm of drainage time of all pulps against the square power of the respective specific surfaces (Figure 27) a straight line was obtained, with correlation coefficient very close to 1.0. Consequently

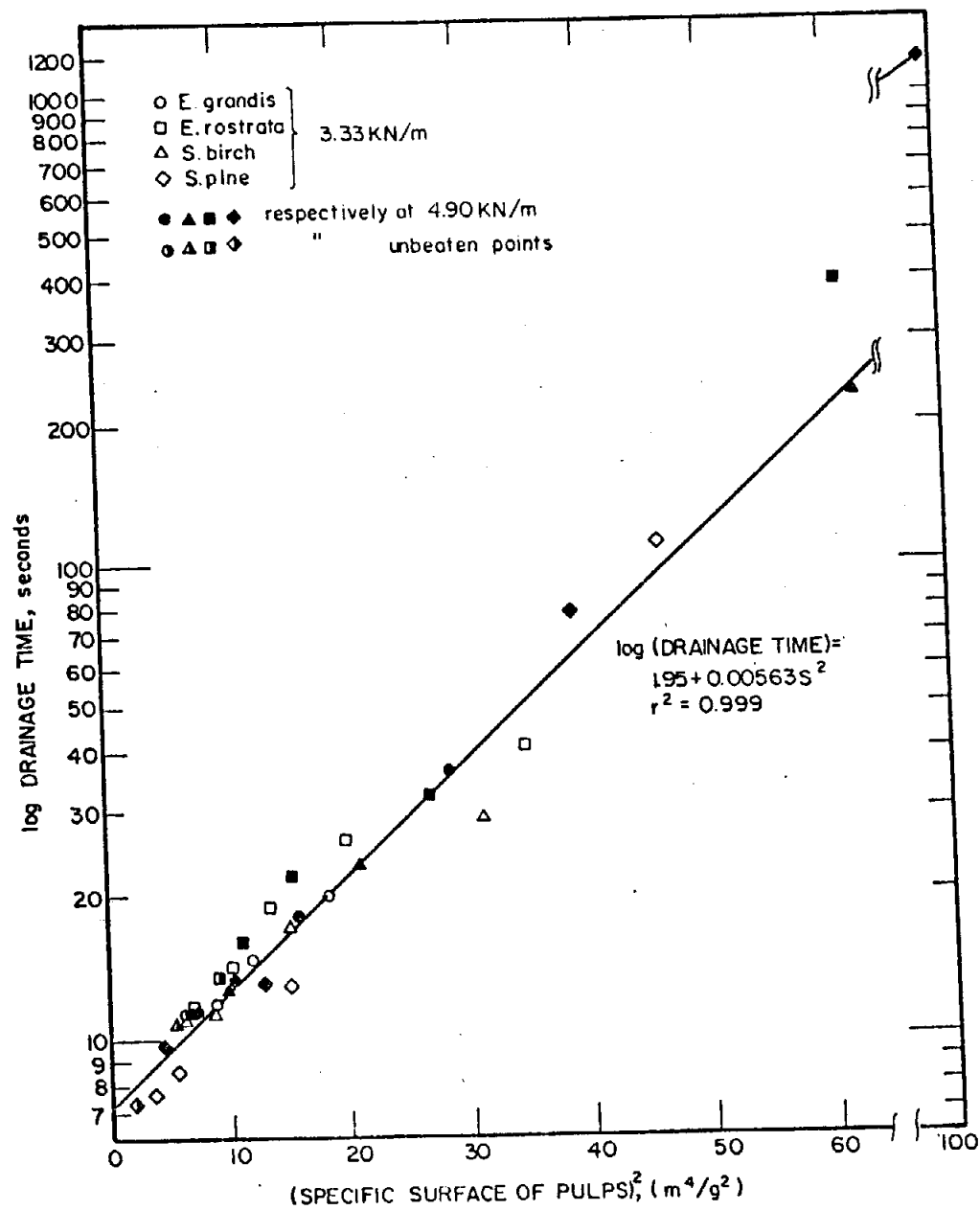


Figure 27 - Correlation between the logarithm of dynamic drainage time and the square power of the specific surface of whole pulps.

one single pulp characteristic, its specific surface area, could be related to a simulated rate of water removal in the forming zone for a variety of pulps, with different morphological and chemical characteristics, and beaten to different extents under different conditions. It should not be forgotten that, as shown previously, the average specific surface area of a pulp depends on its content and surface area of the fines, as well as the characteristics of the fibers, including those in the unbeaten state, such as fiber geometry and coarseness, plus the external fibrillation promoted by the beating process.

So, as a first consequence of the changes imparted to a pulp during beating it can be seen that the rate of water removal in the forming zone of a paper machine is directly influenced by the newly formed surfaces in the form of fines and on the exterior of the fibers. The freeness test, as it has been used, cannot account for these changes as a generalized measurement, although for each pulp beaten at defined conditions the freeness determination still provides some indication about its particular behavior. Being a measurement very easy and rapid to perform the CSF is still used extensively in laboratories and industry, despite the increasing awareness of its inadequacy, especially when applied to chemical pulps. However, the use of a drainage test involving conditions closer to actual paper machine operation,

and equally simple to perform as the freeness, may prove very advantageous for control in the mill as well as for a more adequate follow-up of beating.

Wet Web Strength. - An additional factor to the efficiency of the papermaking process is the ability of the wet web to withstand the tension forces in the open draws of the papermachine. A fast draining furnish may speed up water removal on the wire, but if the web after the couch and through the presses is not strong enough breaks will occur, slowing down the entire process. Usually a compromise must be reached for optimum operation. As discussed in the review of the literature the increasing demands for operation at high speeds with low basis weight papers more than ever show the need for a better understanding of the load-elongation properties of the wet web. This subject has been included in this study, with particular attention to the beating behavior of fibers with different morphological and chemical characteristics.

The tests were performed after forming the webs under constant conditions, as described in the experimental section. This procedure ensured a high degree of reproducibility and provided additional information about the behavior of the various whole pulps and pure fibers in the forming process. All pulps ended up with dry contents ranging from 30 to 36%, and as reported earlier (92) this should have only a small influence on wet tensile strength,

and affects stretch negligibly. The results are summarized in Tables B-1 through B-IV in Appendix B. These tables contain the dry content, tensile strength, elongation and caliper of the wet webs of the four pulps, at three levels of beating, each of them under 3.33 and 4.90 kN/m load. The following figures illustrate the different aspects of the observed values.

The development of wet web strength with beating showed an unexpected straight line relationship with the applied energy, at both beating loads. This can be observed with tensile strength (Figures 28 and 29) and wet stretch (Figures 30 and 31), for all pulps studied. The differences between the pulps are smaller when considering tensile strength than when comparing their stretch values, and it can be seen that the effect of beating load is considerable.

A closer look at Figures 28 and 29 indicate that at a load of 3.33 kN/m three of the curves are somewhat clustered, with experimental points for southern pine and E. grandis lying almost in the same line, followed by slightly larger values for E. rostrata, and then birch, with a much faster development of tensile strength with beating energy. At a higher load all pulps develop wet tensile strength at faster rates, but in this case it is seen that the eucalypts have much closer properties, the pine has improved its development of strength relatively to the eucalypts, and finally birch shows a characteristic behavior, with improvements relative to the lower load very

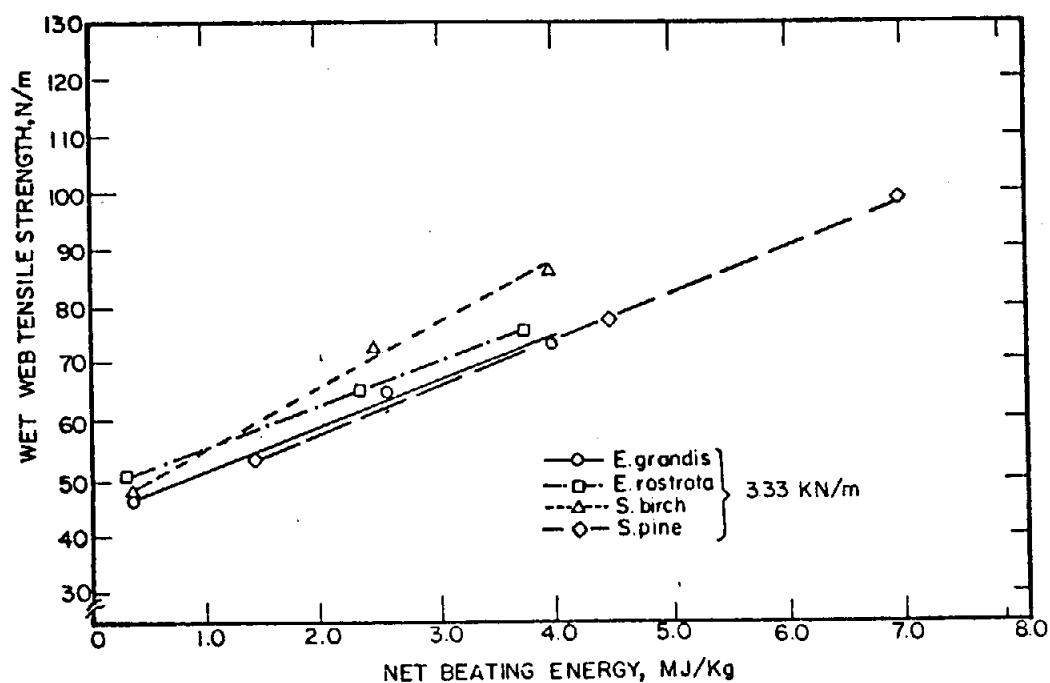


Figure 28 - Development of wet web tensile strength of whole pulps with beating energy - 3.33 kN/m load.

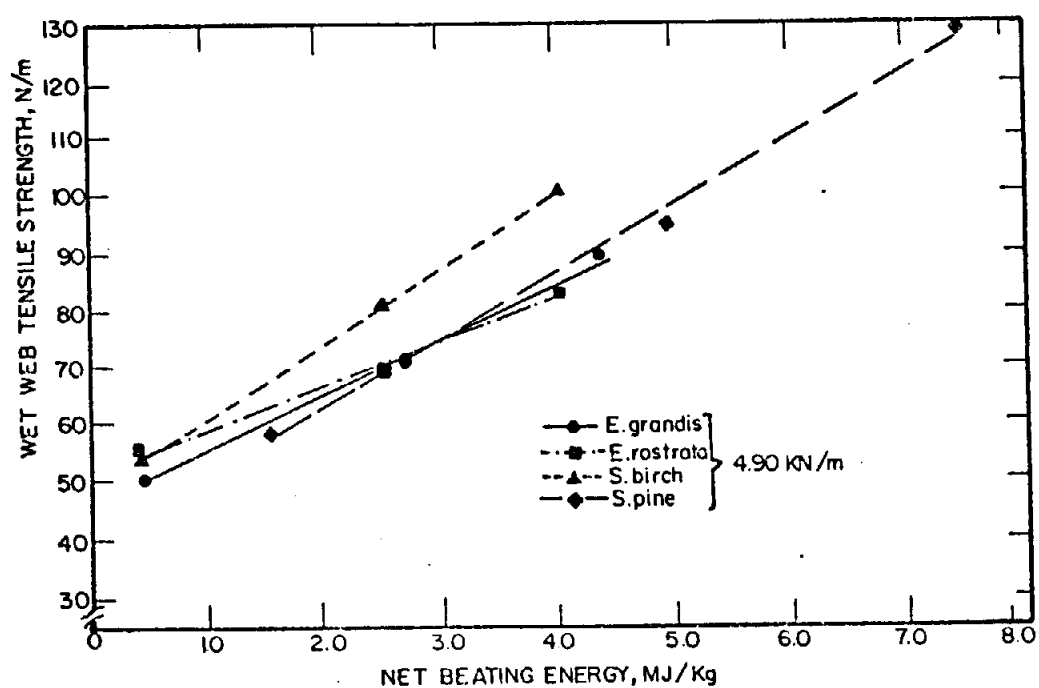


Figure 29 - Development of wet web tensile strength of whole pulps with beating energy - 4.90 kN/m load.

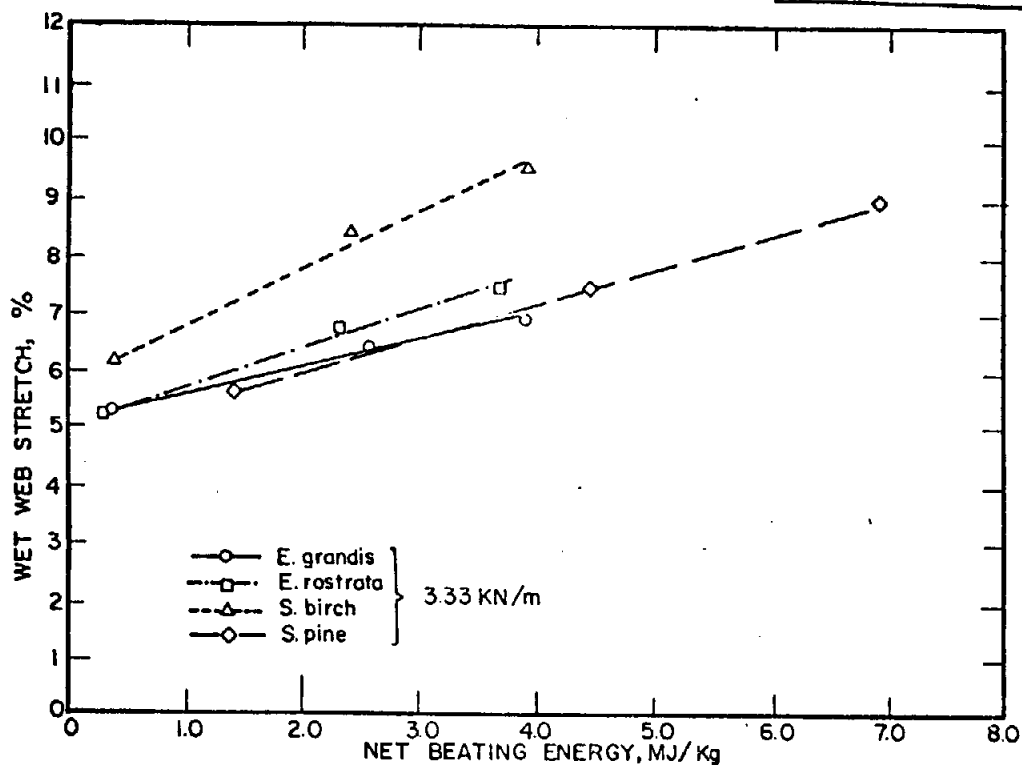


Figure 30 - Development of wet web stretch of whole pulps with beating energy - 3.33 kN/m load.

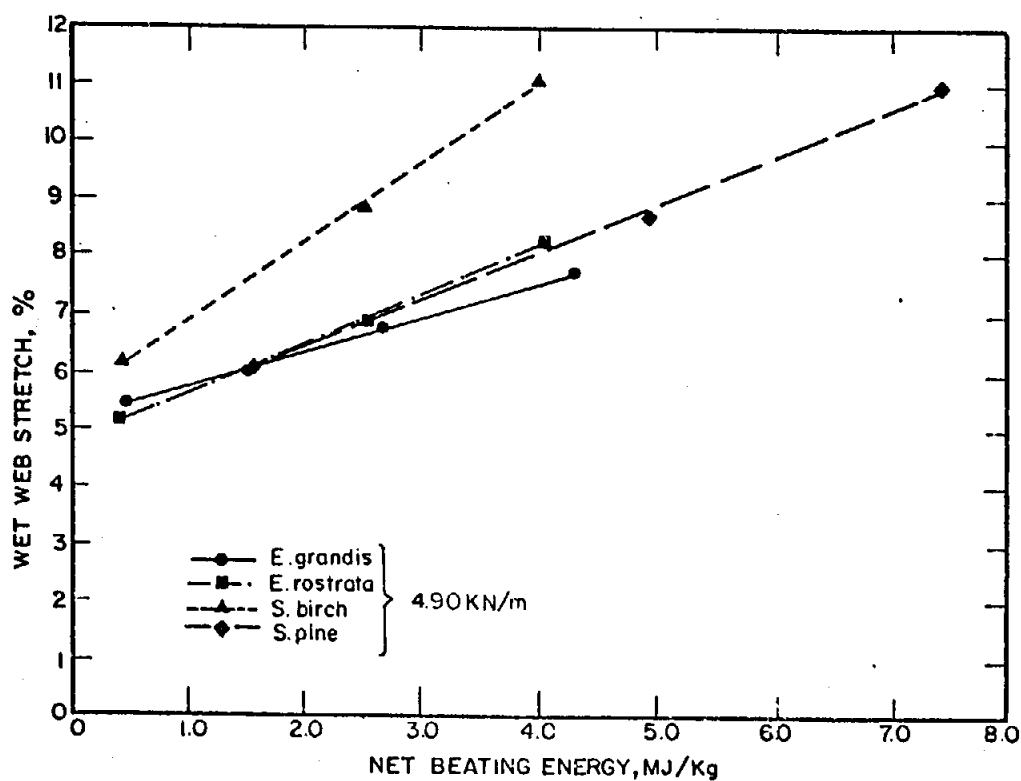


Figure 31 - Development of wet web stretch of whole pulps with beating energy - 4.90 kN/m load.

similar to the southern pine. When comparing the stretch values (Figures 30 and 31) approximately the same conclusions may be reached as in the case of tensile, although silver birch now displays values of much larger magnitude than all the other three pulps.

Beating Effects and Wet Web Strength - The data on fiber properties and their changes with beating discussed previously should be helpful towards the understanding of the wet web strength. It should be mentioned that multiple regression analysis was used when trying to correlate the different pulp and fiber characteristics with the development of wet tensile and stretch, but the results were of limited applicability. The reason for this was found to be the great extent of interactions among the properties selected as independent variables, e.g. specific surface area, specific volume and weighed average fiber length of pure fibers and whole pulps.

In view of today's understanding of the mechanisms of wet web strength development it was concluded in the Literature Review that various pulp characteristics affect differently the sequential steps in web formation and consolidation, and the overall effect in tensile strength and stretch seems to be a result of the combination of fiber flexibility, external fibrillation, fiber twisting and kinking, and the formation and quality of fines. This may be one of the reasons for the contradictory results found in the

literature. As a consequence it becomes very difficult to separate distinctively their effects on the behavior of the wet web unless each property could be developed alone at each time, and its consequences evaluated.

Since all the pulps were formed under the same conditions which resulted in solids contents over 30% it may be assumed that the corresponding wet webs had reached a state of consolidation in which most, if not all of the free interfiber water had been removed (it should be remembered that the webs were formed under the application of suction and pressure). Therefore the effects of fiber entanglement and surface tension forces imparting coherence of the webs may be assumed to have been fully developed by the time the tests were performed, with the particular influences of fiber flexibility, specific surface and weighed average fiber length acting in an additive fashion.

An indication that a simple property could not account for the differences observed could be seen when plots of wet web tensile and stretch against specific surfaces (not reported here), showed a considerable scatter that could not be well understood. Thus a simple explanation of the web resistance to deformation as being a function of frictional forces mostly influenced by the external fibrillation and production of fines (97) could not be applied to the data.

Turning to the first step during strength development

of the wet web as possibly the fundamental mechanism upon which all others are superimposed showed to be a better choice. Indeed, as indicated by the observed changes in the fibers with beating energy the increase in delamination and swelling, as measured by the specific volume, with consequent increase in fiber flexibility may be considered as the main beating effect. This increased flexibility of the fibers should result in more entanglement and consequent increased network strength. Previous results with the beating of semi-bleached kraft showing the largest increases in wet web strength in the early beating stages (148) seem to be in agreement with this idea.

A first indication of these expected trends can be observed in Figures 32 and 33. For fines-free pulps a single relationship appears to exist between their specific volume and the corresponding wet web tensile and stretch for all the four pulps, beaten at different conditions. A single line for whole pulps is also observed for stretch versus specific volume, but when plotting wet tensile strength against specific volume of whole pulps three distinct curves are observed: a single one for pine and E. grandis, showing larger tensile values at a given specific volume, an intermediate curve for E. rostrata, and thirdly the lowest tensile values at constant specific volume occurring for birch.

When considering that for the fines-free pulps there is only one line relating the wet web tensile strength to

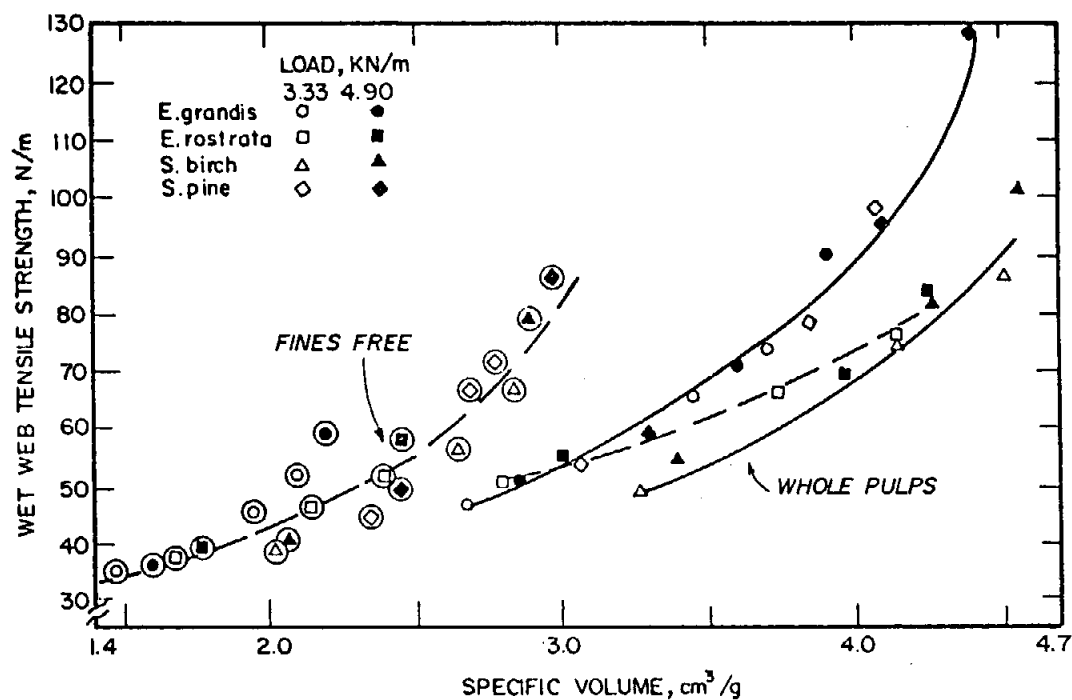


Figure 32 - Relationship between wet web tensile strength and specific volume of whole pulps.

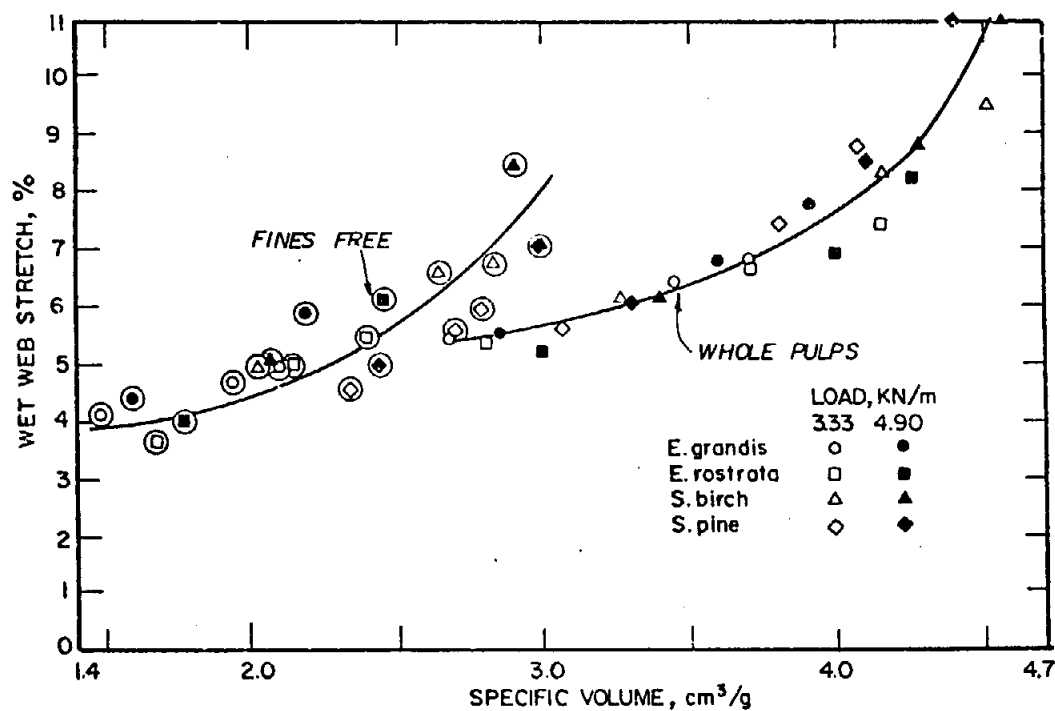


Figure 33 - Relationship between wet web stretch and specific volume of whole pulps.

the specific volume of the fibers from the four species, the different curves for the whole pulps in Figure 32 could be explained in terms of the relative contributions of increased fiber flexibility and the surface area in the wet web available for the development of increased surface tension forces. Thus, a higher specific surface at a given level of swelling (such as occurring in the more open structures of webs from southern pine and E. grandis) would tend to enhance the frictional forces between the fibers when air is present in the web, after suction and pressing.

A slightly different mechanism might be present in the amount of stretch that the webs can support before rupture. Similarly to wet tensile the web elongation would be a strong function of fiber entanglement and density of the network, but with no further influence of specific surface area, as indicated by the single line for whole pulps in Figure 33.

The Role of Web Consolidation. - By bringing up the subject of fiber flexibility considerations must be made about the initial fiber morphology as well as chemical properties. It has long been recognized that the strength of paper in general is increased with the water retention (or hydration) properties of the pulp. Although for some time it was believed that this increase in wetness was due to external fibrillation (as some still claim today) which would be measured by the freeness test, it is today more or

less accepted, as has also been confirmed in this work, that swelling is related mostly to changes in the ultra-structural organization of the cell wall associated with its hemicellulose content, thus increasing the fiber flexibility. However swelling by itself is not an absolute measurement of fiber flexibility after beating. The dimensions and geometry of the fiber's cross section and the rigidity of the fiber wall are also important factors which, although influencing the degree of swelling that the fiber is able to achieve during beating, could play their particular roles in the flexibility of the fiber, as indicated by the calculated stiffness of the fibers in the unbeaten state.

The most apparent consequence of fiber flexibility is the degree of consolidation of the network, which, as advocated by some (4,33) is the main characteristic to be followed during beating. Indeed, the more flexible birch fibers produced wet sheets with smaller caliper at a given beating degree than E. rostrata, followed by the stiffer fibers of E. grandis and southern pine (Tables in Appendix B). In order to observe the effects of sheet consolidation on strength properties both wet tensile and stretch were plotted against sheet caliper, and are seen in Figures 34 and 35.

First of all it is very interesting to observe in these figures that the points for whole pulps and pure fibers fall into the same line for each pulp, indicating that, with or without the fines, their respective properties influenced

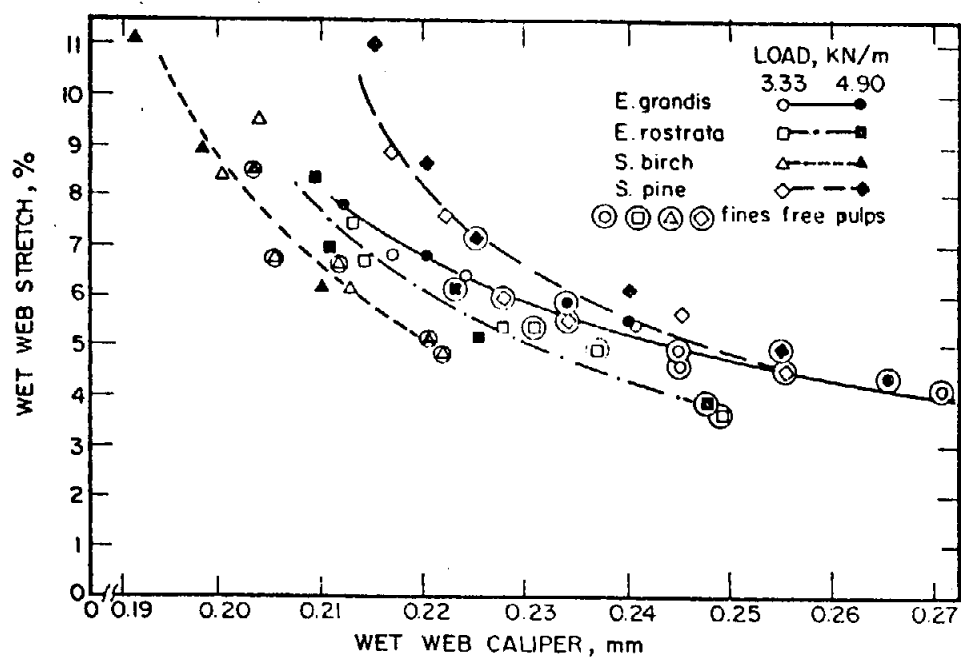


Figure 34 - Relationship between wet web stretch and wet web caliper.

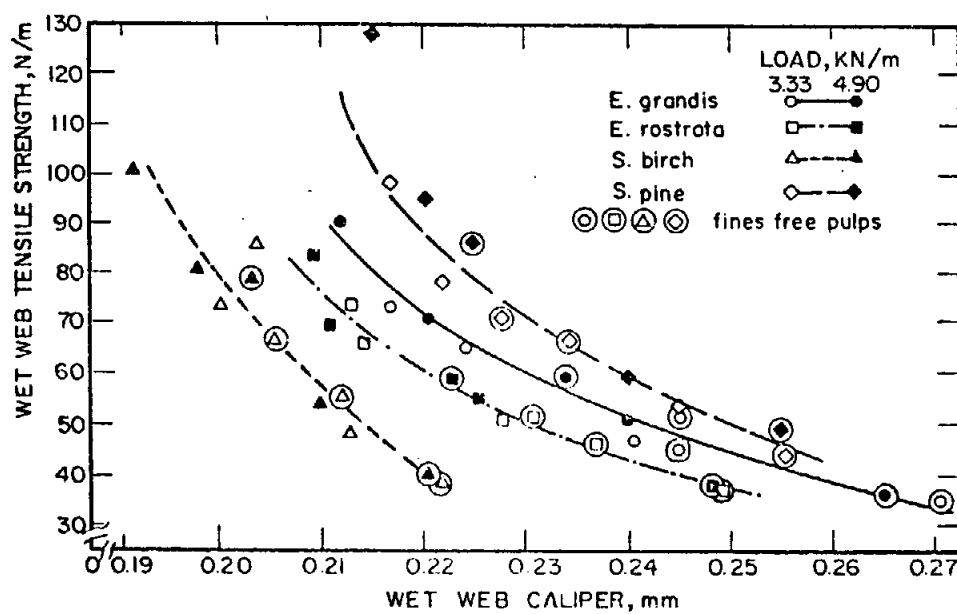


Figure 35 - Relationship between wet web tensile strength and wet web caliper.

somehow the consolidation process in an additive fashion, i.e. first the fibers would interact and form networks at low solids content (below 10%) and then by application of suction and pressure the webs would consolidate, this process being enhanced by their compressibility and the action of surface tension forces, as influenced by their specific surface area. Hence the main consequence would be the density of the network, which can thus be interpreted as a fundamental property of the wet web. Nevertheless, each pulp is shown to have its particular strength-density relationship. For example at 0.212 mm sheet thickness silver birch shows approximately 50 N/m tensile strength, E. rostrata 70 N/m, E. grandis 83 N/m and southern pine 105 N/m.

These differences could not be explained simply by the respective fiber lengths, specific surfaces or number of fiber contacts per unit sheet weight. A possible explanation could be based on initial fiber morphology, mostly the bending stiffness and on the way the pulps hold water after suction and wet pressing.

Studies on wet mat compressibility have indicated that the flexural rigidity of the fibers is one of the main controlling factors (19). In addition to that it has been suggested (203) that a more compressible sheet, as indicated by its wet caliper, might be able to have more water removed under vacuum and pressure in the web former. It is thus possible that fibers with lower rigidity would be more able to

lose water in the forming machine during the test, but since all pulps ended up with dry contents very close to one another the manner of holding of the remaining water becomes also of great importance.

Obviously the free interfiber water is removed easily, and when the web is compressed the water from the lumen is more easily expressed out the more delaminated and rich in capillaries the fiber wall is (this process being enhanced by the presence of pits and broken fiber ends). Of course after the pressure is removed the network will tend to expand, this happening at higher extent the less conformable the fibers are. Thus, for sheets of different pulps at a given caliber it could be postulated that the more flexible and swollen fibers, especially those with high hemicellulose content, (such as from birch and E. rostrata pulps) will tend to hold the remaining water within the capillaries of the cell wall and at fiber crossover regions after web formation, with little space left for air water interfaces, due to the more compacted structure of the web.

The stiffer fibers, although made more conformable through beating, might be able to "spring back" after compression, leaving a more open space, over which the water can spread, creating a larger air-water interface. Surface tension forces would then promote the adhesion among fibers and between fibers and fines, thereby increasing the wet web strength. At the same time the stiffer fibers of

southern pines and E-grandis could impart a higher degree of rigidity to the web, therefore increasing the strength.

This proposed mechanism is similar to Robertson's mobile water concept (183), which indicated that, at a given swelling degree, a lower moisture content at which water migration ceased within the web would yield a stronger network. However this suggestion differs from the concept presented here, since in our case the web was not compacted until the surfaces of the fibers became dry (the end of water migration), but rather the wet web was compacted once, and the remaining water could be redistributed over a larger area, in a more open and porous structure.

Thus, a certain level of caliper (or density) does not necessarily mean a given state of compactness in the sheet, and probably this is the main reason why more open structures at a given caliper in the following order: silver birch < E. rostrata < E. grandis < southern pine (as indicated later by porosity values) show increasingly larger wet tensile strengths at approximately the same moisture contents and the same web thickness.

This hypothesis seems to agree, to a certain extent, with mill experience (133), which has indicated that furnishes with lower water retention value at a given wet web strength ran with fewer breaks on papermachines.

The differences among the pulps for the wet stretch

versus web caliper curves (Figure 34) are smaller than in Figures 35, indicating again, as discussed when comparing Figures 32 and 33, that surface tension effects are less important for the development of wet stretch. As the fibers slide past each other when the web is stressed the elongation of the network before rupture, at a given density, would appear to be influenced by the ability of the fibers to conform to each other and redistribute the stresses. Fiber flexibility and curvature (24), such as kinks developed at high consistency bleaching (123,124) and latency in TMP (15) are considered to be important factors in this process. Fibers with larger amounts of hemicellulose on their surfaces, as deriving from their higher hemicellulose contents and extensive detachment of xylan-rich fibrils, such as birch and E. rostrata, would have a tendency to be more "slippery" than E. grandis and southern pine, therefore diminishing the coefficient of fiber-fiber friction, and consequently also lowering the stretch.

Wet Web Strength - Dynamic Drainage Relationship. - Obviously as beating proceeds and the strength of the wet web is increased the changes occurring in the pulps will affect other stages in the papermaking process. With respect to drainability of the pulps on the forming zone there is no point in producing a stronger web that will be very difficult to drain, slowing down the paper machine anyway. This aspect is illustrated in Figure 36, where the logarithm of

dynamic drainage time was plotted against wet web tensile strength and web stretch.

It can be noticed at first that with respect to both wet tensile and stretch E. rostrata shows a definite disadvantage in relation to the other pulps, with much longer drainage times for a desired level of wet web strength. If wet tensile is the desired property it is seen that birch and E. grandis display a very similar behavior, with southern pine giving better tensile at a given drainage time than any of the hardwoods. When wet stretch is considered, however, an outstanding performance is observed for birch pulp, with intermediate behaviors of E. grandis and pine.

From the discussion and results above it is inferred that the strength of the wet web depends strongly on the ability of the fibers to form networks that consolidate well, not only due to the flexibility of the fiber which assists interweaving and conformability, but also enhanced by the specific surface area of the pulps.

Beating promotes internal fibrillation and swelling of the fibers, thereby increasing the degree of entanglement when the web is formed, which helps in the development of strength even before the web consolidation is terminated. In addition, the beating process causes the detachment of fibrils from the surfaces of the fibers, with consequent formation of fines of increasingly larger specific surface

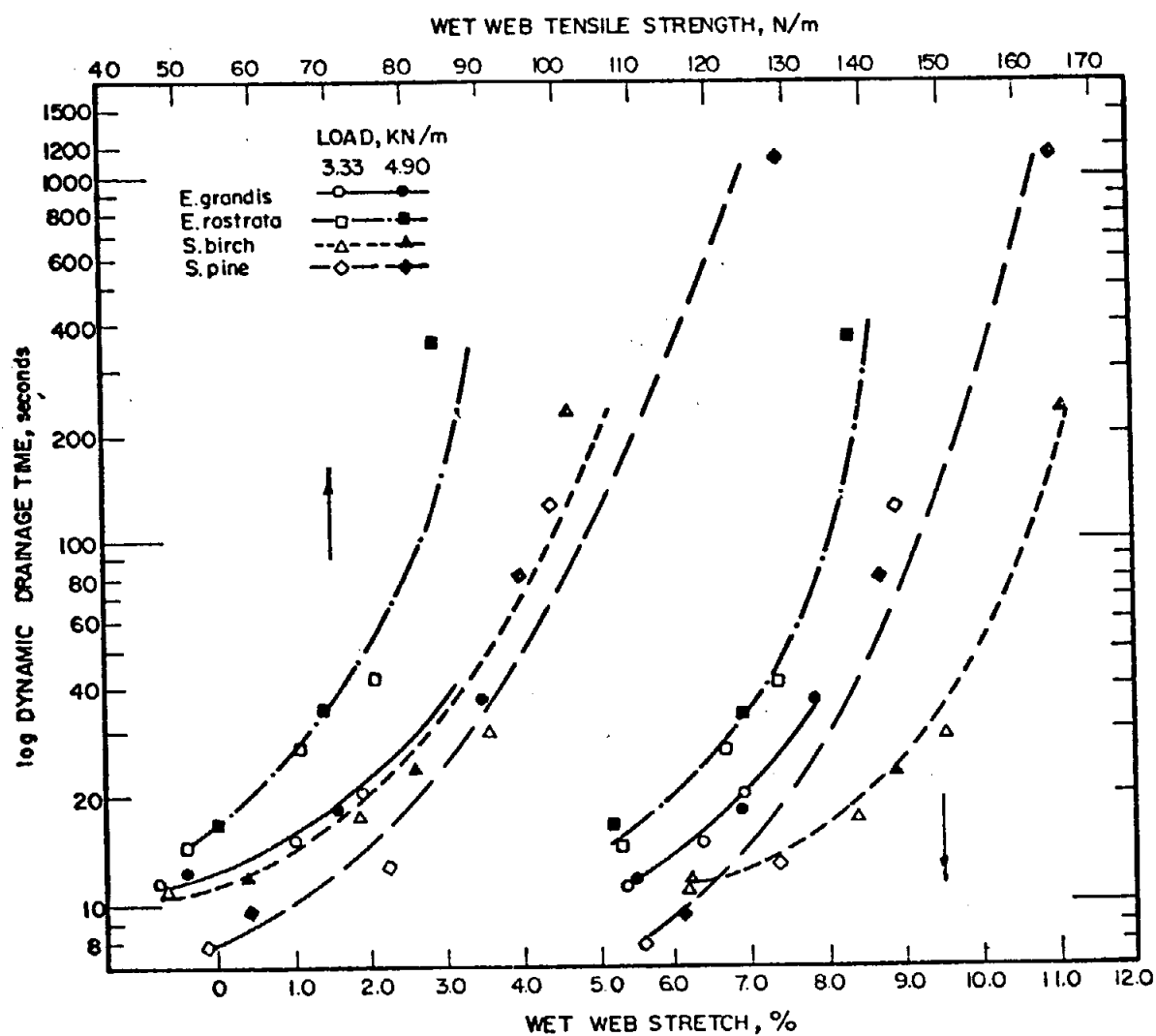


Figure 36 - Relationship between the logarithm of dynamic drainage time of pulps and their wet web tensile strength and wet web stretch.

areas, which will not only aid in the process of consolidation of the web, but also promote the surface tension forces as air enters the structure. The extent of these effects will depend on the initial characteristic of each pulp, in particular their hemicellulose content and flexural rigidity of their fibers, and their responses to beating.

Certainly this study has not provided explanations for all the phenomena observed, but it is believed that in terms of the morphology and chemical properties of the fibers a better understanding of the development of wet web strength with beating was achieved. For stronger webs, pulps with flexible fibers and high hemicellulose content will give better results, especially if they are beaten under conditions which promote web consolidation without increasing the surface area to a point which impairs their drainage times.

The aspect of water retention within the web structure after compaction, which is pretty much related to the earlier suggested needed work on behavior of flocs under compressive and shear stresses, and the complex fiber-fines-water interaction during suction and wet pressing should be given increased consideration in further research, which might make possible great improvements in the understanding and better efficiency of the papermaking process. With respect to beating conditions it was confirmed that a higher load at a given beating power intake provides a more efficient use of

energy towards wet web strength development, although this occurs at the expense of poorer drainage characteristics.

Paper Properties. The manufacture of paper involves a careful balance of pulp properties related to machine efficiency and the characteristics of the final product. Since the choice of the fibrous raw materials, their blends and the transformations achieved through the beating operation are the most important variables to be controlled by the papermaker routine analysis are commonly performed in standard handsheets to assess the development of paper properties. The data obtained is then correlated with the properties of the same pulps when wet, in order to compose a picture of their overall qualities and relate them to the operation of the paper machine. The same sequence of steps was followed also in the course of the present study of the beating of four pulps, of which the wet properties were described in the previous section.

The results from tests performed on dry handsheets formed and conditioned according to TAPPI standards are listed in Tables C-I through C-IV in Appendix C. The properties reported are apparent density, tensile index, tear index, burst index, light scattering coefficient and Gurley-air resistance.

Freeness is the property most commonly used to follow the beating of pulps. Despite increasing recognition of the

limitations of the method, the majority of published results are still compared at constant freeness, which impairs seriously the usefulness of the data. There is no doubt about the simplicity and historical value of the test, but comparisons of properties at a constant freeness particularly for chemical pulps can be very misleading. As already discussed the CSF shows a strong relationship with the specific surface area of a pulp, being influenced by the fiber morphology, external fibrillation and the amount and quality of fines. The results in this study have shown that the specific surface area, which depends on these characteristics, changes to different extents for the four pulps during beating. Furthermore, the development of external fibrillation and the formation of fines were observed to proceed almost independently of the main beating effect (the changes in the specific volume of whole pulps and pure fibers), which bears very little influence on freeness. Additional tests indicated also that the freeness value is not an absolute indication of a pulp's ability to dewater over the wire. Consequently it was decided for the present study to base all comparisons among the four pulps on a strength property of the paper, the increase of which is, after all the main purpose of beating. The tensile index was chosen as the basis for comparisons, as has also been increasingly suggested (78,144,189,197).

Comparison of the Beatability of the Pulps. - The response of the four pulps to beating with respect to paper properties is illustrated in Figure 37, where the development of Tensile Index is plotted against the net beating energy, using 3.33 kN/m load in the PFI mill. It is clearly noticed that the tensile strength of the four pulps increases with beating in a very similar fashion. There is a rapid initial rise in strength at the early stages of the treatment, followed by a tendency to level-off as beating proceeds. The rates of increase in tensile index with energy consumption are about the same for the eucalypts and the southern pine, but since they all display different levels of strength in the unbeaten state, the strength values achieved during beating are roughly proportional to their respective original levels. A higher rate of tensile strength development is noticed for silver birch, which, as well as E. rostrata, has also the largest tensile index when still in its unbeaten state. Therefore, as a consequence of not only the initial characteristics of the pulps, but also due to their different responses to beating, a desired level of strength, for instance 50 N m/g, is reached after the application of different amounts of energy for the four pulps, i.e. approximately 0.75 MJ/kg for birch, 1.00 MJ/kg for E. rostrata, 1.27 MJ/kg for E. grandis and 1.87 MJ/kg for southern pine.

When trying to explain the different behavior of these

pulps in terms of changes imparted to them during beating, it is noticed that the tensile index correlates very well in a linear fashion with the hydrodynamic specific volume of the pulps (Fig. 38). Similar relationships were observed for burst index and apparent density of the handsheets. These are additional indications that the changes in paper properties which are mostly dependent on the degree of web consolidation and interfiber bonding are influenced directly by the extent of variation in the water retention ability of the pulps. The latter, in turn, depends not only on the pulp's hemicellulose content, but also on the initial fiber properties, namely their flexibility, as also indicated by their different levels of strength in the unbeaten state.

The flexibility of fibers has for a long time been recognized to occupy a central position in papermaking, but so far the measurement of this property has been very limited. There seems to be a common misconception as to what fiber flexibility and fiber collapsability really mean. It has been usually accepted that the collapsability of a fiber, (i.e. the ease with which its lumen collapses when the paper sheet is formed) is a strong function of the fiber cross-sectional geometry, more specifically the relationships between fiber width and fiber wall thickness (90,91,142,172,186,240). The shear modulus in the transverse plane of the fiber wall has also been indicated to influence directly the collapsability of the lumen (91,172). Indeed there is no doubt about the

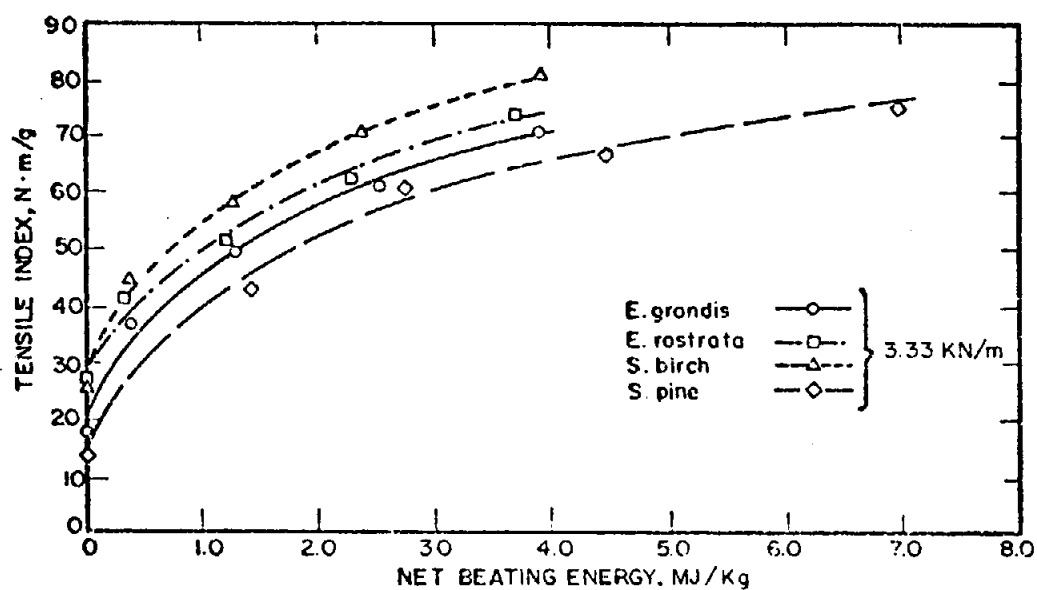


Figure 37 - Development of tensile index of whole pulps with beating energy - 3.33 kN/m load.

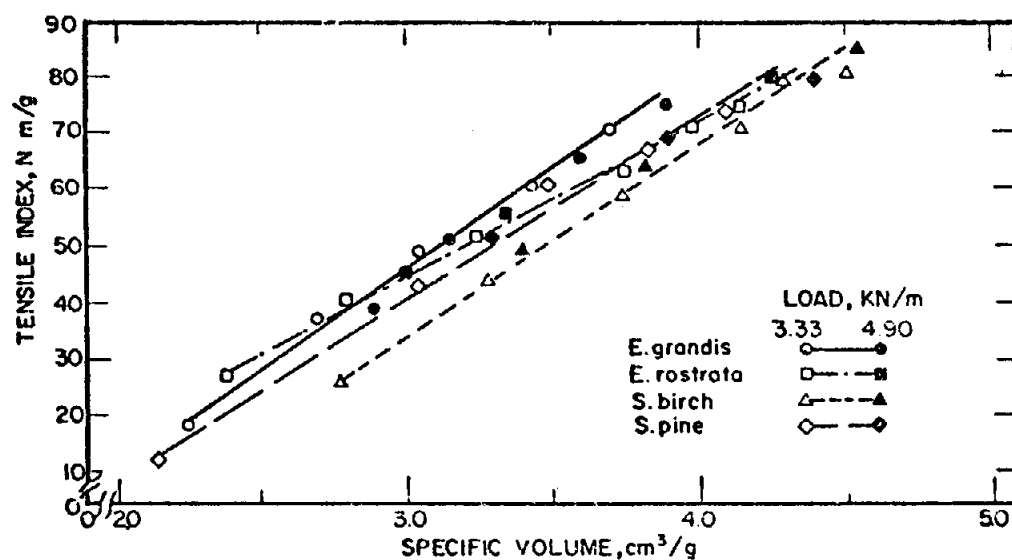


Figure 38 - Relationship between tensile index of whole pulps and their specific volume.

importance of these factors on lumen collapse (9), but it has been taken for granted that a more collapsable fiber would also be more flexible, on the basis that the collapse of the lumen is necessary for interfiber bonding.

Although experimental evidence for this concept has been provided by the well known facts that thick walled fibers yield bulkier sheets of lower strength than thinner walled ones, this being the reason for the poorer beatability of the former, it has never been proved that the collapse of the lumen is a necessary condition for the fibers to conform to each other in the sheet of paper and form bonds. This was recognized by Scallan and Borch (195a), who indicated that for the purpose of transforming unbeaten cylindrical fibers into flat ribbons capable of more interfiber contact through beating the collapse of the lumen does not need to be complete. The lumen collapse itself has more to do with a loss of light scattering ability than with conferring strength to the paper sheet.

A much better and closer indication of the actual flexibility of the fiber can be provided by the reciprocal of its bending stiffness, expressed by the product of its axial modulus of elasticity ( $E$ ) and the moment of inertia ( $I$ ).

As discussed previously in connection with the calculation of pulp fiber stiffness, the moment of inertia is a mere consequence of the fiber's cross-sectional dimensions, with larger values obtained for wide fibers. On the

other hand, the Young's modulus in the axial direction of the fiber is a strong function of its ultrastructural organization, namely the microfibrillar orientations in the various layers of the cell wall (150), being also influenced to some extent by the chemical composition of the fiber. With small moments of inertia, due to the small cross sectional dimensions it would not be expected that the narrow eucalypt fibers would show a greater bending stiffness than the wider birch fibers. However, the calculated elastic moduli for both eucalypts were much larger, as a consequence of their steeper  $S_2$  microfibrillar orientations in the cell wall. As a result of the combination of these factors, larger flexural rigidity values were obtained for E. grandis and E. rostrata ( $5.4 \times 10^{-12} \text{ N m}^2$  and  $4.4 \times 10^{-12} \text{ N m}^2$  respectively) than for birch ( $3.3 \times 10^{-12} \text{ N m}^2$ ).

The stiffness value calculated for a representative southern pine fiber was somewhat surprisingly large when compared to the values obtained for the hardwoods. Although the Young's modulus for the pine is very similar to the calculated values for the eucalypts, the much larger cross sectional dimensions of the former are responsible for a moment of inertia at least ten times greater than for the latter. Consequently the estimated fiber stiffness of an average pine fiber is one order of magnitude larger than the values of the three hardwoods.

Actually, from pure geometrical considerations a larger stiffness should be expected from wider beams. But when comparing beams of very similar material properties, such as the eucalypts and the pine (as indicated by their ultrastructural organization, packing density, etc.), it could be inferred that the differences in geometries among them could also be reflected in their coarseness values. In other words, the larger weight per unit lengths of pine fibers when compared to the eucalypts could be mostly due to the larger cross sectional areas of the former. Therefore when trying to understand how fibers (or beams) of largely different dimensions would conform in a sheet of paper of a given basisweight, the association of their stiffness values with the number of fibers per gram (derived from the coarseness and fiber length) might be able to provide a generalized factor to describe the behavior of pulps of very different characteristics. Following this approach a conformability factor was defined:

$$CF = S \times N, \quad \text{where } CF = \text{conformability factor, } \frac{N \text{ m}^2 \times \text{fibers}}{\text{gram}}$$

$$S = \text{fiber stiffness, } N \text{ m}^2$$

$$N = \text{Number of fibers per gram of pulp.}$$

As shown in Figure 39, a plot of the density of hand-sheets prepared from unbeaten fines-free pulps against the

conformability factor yields a straight line, confirming the previous expectations. As observed, this factor allows the prediction of the density of a sheet of paper solely from considerations of ultrastructural and geometric properties of the fibers. As Clark has been suggesting for a long time, the measurement of the density of standard handsheets is probably the best indication of the conformability of the pulp fibers (33), but to our knowledge the results presented here should provide, for the first time, a fundamental basis for the understanding of this phenomenon in a generalized approach, i.e. independently of species, whether hardwoods or softwoods.

This concept would then be able to explain why the handsheets from the four pulps have different characteristics in the unbeaten state, to start with. When analyzing the beatability of a pulp the degree of structural rigidity in the fiber wall and the number of fibers per gram would be the first factors to consider, indicating to the papermaker how much development should be needed by a certain pulp. The ease with which internal hydrogen bonds are broken during mechanical treatment, thereby decreasing the axial fiber modulus, and hence increasing its flexibility, is the additional factor composing the picture which indicates how difficult it is for a certain pulp to reach a desired characteristic through beating.

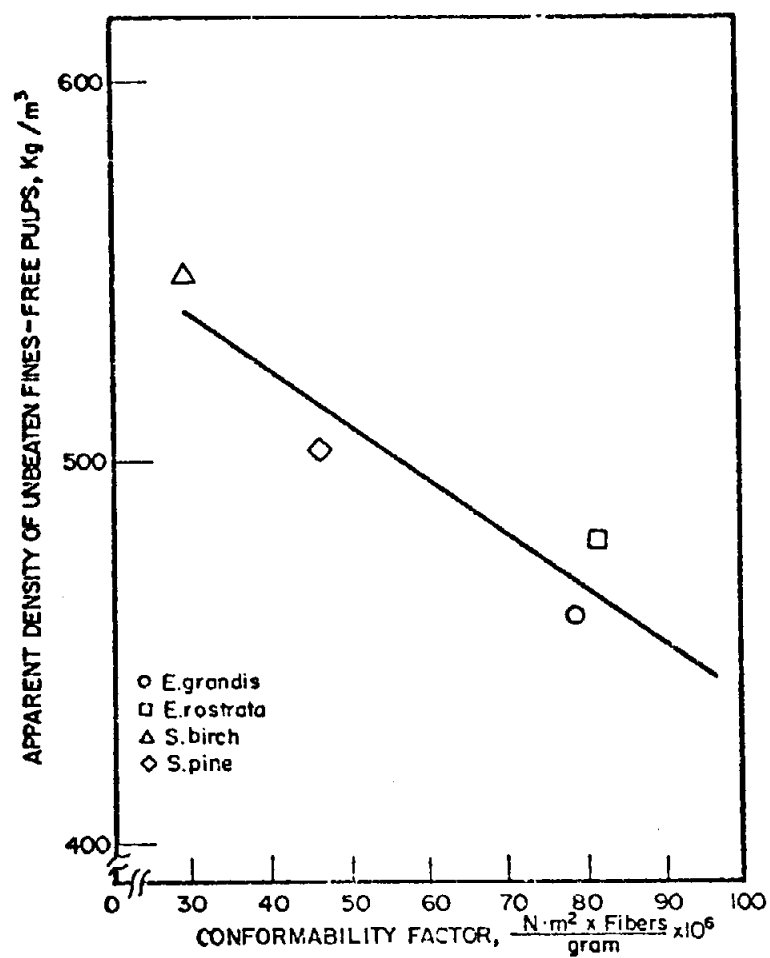


Figure 39 - Correlation between the apparent density of unbeaten fines-free pulps and the conformability factor.

The facility for cleavage of internal bonds would be greater for fibers with a less packed arrangement of microfibrils, as provided by their occurrence in smaller number, larger width, and possibly happening in fibers with less steep microfibrillar orientations. Large amounts of hemicellulose among the microfibrils would make this separation process even easier, and indications of the extent of these modifications in structure could be monitored by the changes in the fiber's specific volume.

It must be stressed that the ideas proposed above do not contradict explicitly the common knowledge that it is because of its thicker walled fibers that a pulp is hard to beat. It just seems to be a coincidence that fibers which have thicker cell walls also appear to be of smaller diameters, have steeper microfibrillar helices and higher cell wall densities. Ultrastructural considerations, such as the ones upon which the theory here presented is based, not only help in the understanding the basic mechanisms involved in the absorption of beating energy by the fibers, but have indicated the actual reasons for the behavior of fibers with thick walls during papermaking. Hence, although the evaluation of the collapsability of a fiber based on its cross sectional geometry is not directly measuring the fiber conformability, indirectly it points to the fiber's ultrastructural organization as a control of the fiber's flexibility and its response to beating.

This interpretation of the beating process seems to be somewhat connected with one of the few theories actually dealing with the mechanism of energy transfer to the fibers, as formulated by Nissan (167). In this publication it was postulated that the energy applied during beating would be utilized for breakage of internal bonds, by straining the fiber in tension up to a point very close to its elastic limit. Since the energy term was expressed as the product of the fiber's axial elastic modulus by the square power of its elongation during straining, it could be expected that fibers with small fibrillar angles, and thus large Young's modulus would absorb more energy during the beating process than those with large  $S_2$ -angles.

Of course, for a better understanding of the factors controlling the different beatability of pulp fibers, the study of beating at a macroscopic level, including the analysis of the behavior of flocs under stress should be given increased importance. It should be remembered that the interpretation of the data revealed in this study is applicable to commercial bleached kraft pulps, manufactured under very similar conditions. Of course a definite type and amount of beating should produce results which are dependent on the previous history of the fiber.

Relationships between Paper Properties. - The application in beating of a higher load was shown to increase

the specific volume of the three hardwood pulps only slightly, while the southern pine appeared to respond to it to a much greater extent. The main effect of a higher load was the creation of more fines and provision for a faster development of external fibrillation in all pulps. Although these effects were accompanied by the pulps' poorer dewatering ability, in general, better wet web strengths were achieved. The changes in tensile index with applied energy were minor for silver birch, E. grandis and E. rostrata under the two loads (Figure 40). For southern pine, however, a higher load was proven beneficial to achieve a maximum tensile strength with energy, especially at the early stages of the beating, although this resulted in lower tear index, which was also observed for the three hardwoods. With respect to the tensile strength these results are in agreement with the previous discussions of the beating effects on fibers, mostly the changes in specific volume of the pulps, which, as indicated in Figure 38 appear to bear a strong relationship with the tensile index. The reduction in tear strength with increased load has also been reported previously (139), but mostly as a result of decreased fiber length due to cutting. However the individual relationships between apparent handsheet density and tensile index for each pulp at both loads, as seen in Figure 41, seem to indicate that the decreased tear at high load was in general mostly due to a higher density in the sheet.

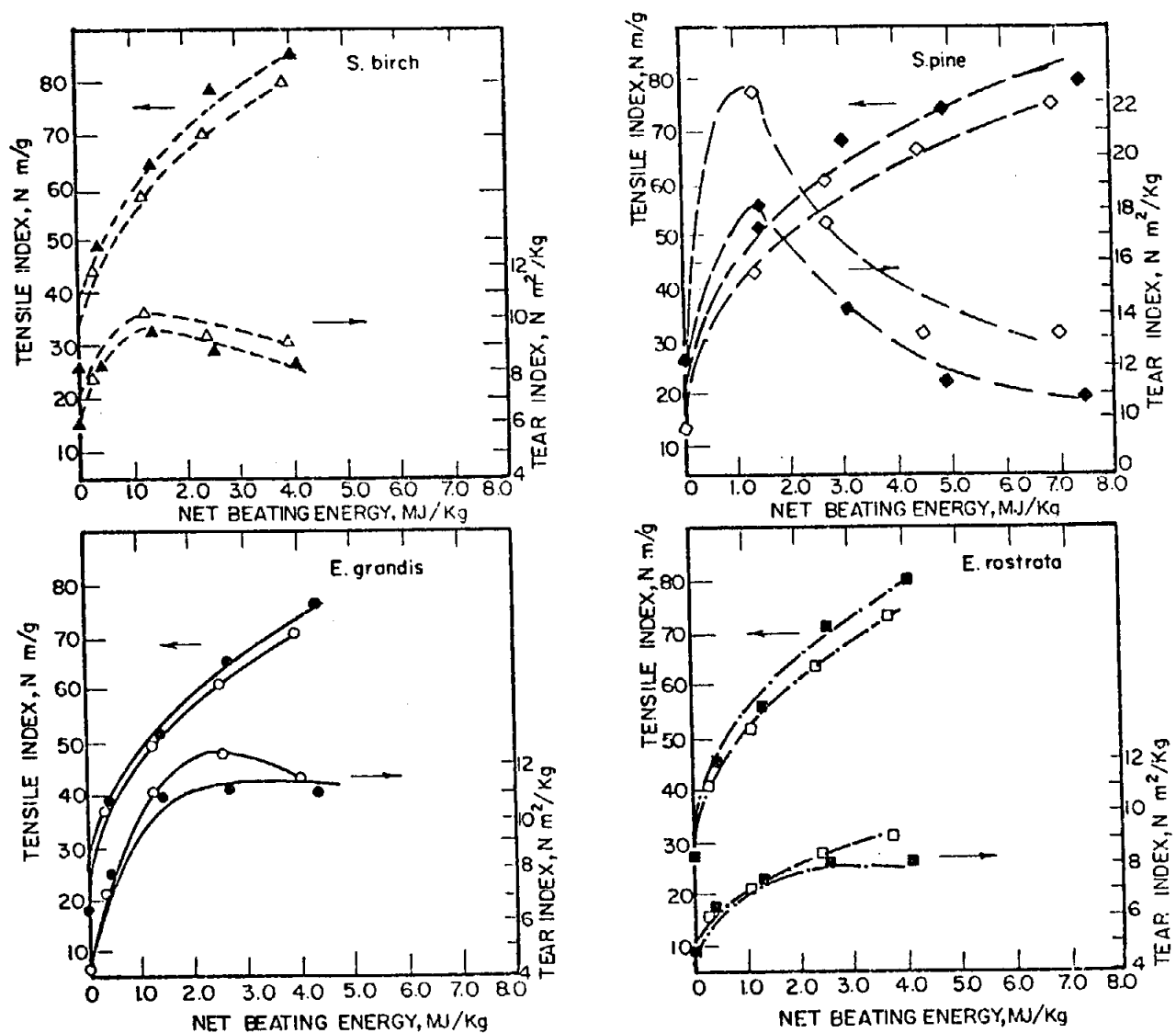


Figure 40 - Developments of tensile index and tear index with beating energy at 3.33 and 4.90 kN/m load.

The results in Figure 42 also show that for southern pine and birch the tear-tensile plot was not changed as a result of the application of different beating loads. However, it can be noticed that both E. grandis and E. rostrata showed a tendency to have lower tear index values after extensive beating at 4.90 kN/m, probably due to some degree of fiber cutting. Single line relationships after beating at the two loads are also observed in Figures 43 and 44, where the changes in light scattering coefficient and air resistance with tensile index are shown.

Much information can be extracted from the results in Figures 41 through 44 about the papermaking potential of these four pulps. It can be noticed for instance, that at a given tensile strength the density of handsheets from birch is much larger than that of the other three pulps (Figure 41). It would be expected, from our present knowledge of the tensile behavior of paper (59,173,224) that at a given relative bonded area (RBA) - or density - only minor variations in strength would be noticed among the pulps, and these would be due to different levels of strength and length of the fibers composing them. However it was observed in previous discussions that the density of the sheet can be indicated by the flexibility of the fibers and their quantity per gram of pulp. Therefore the differences between birch and southern pine could be explained in terms of their vastly different fiber lengths and the higher rigidity of pine fibers,

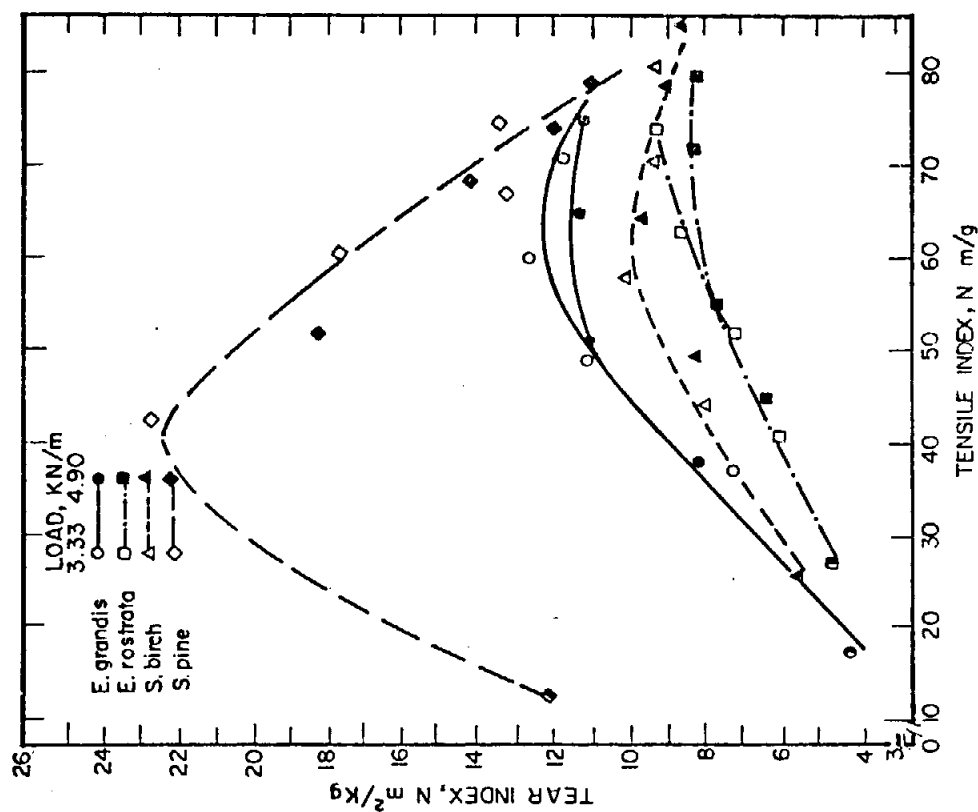


Figure 41 - Relationship between apparent density and tensile index of handsheets from whole pulps.

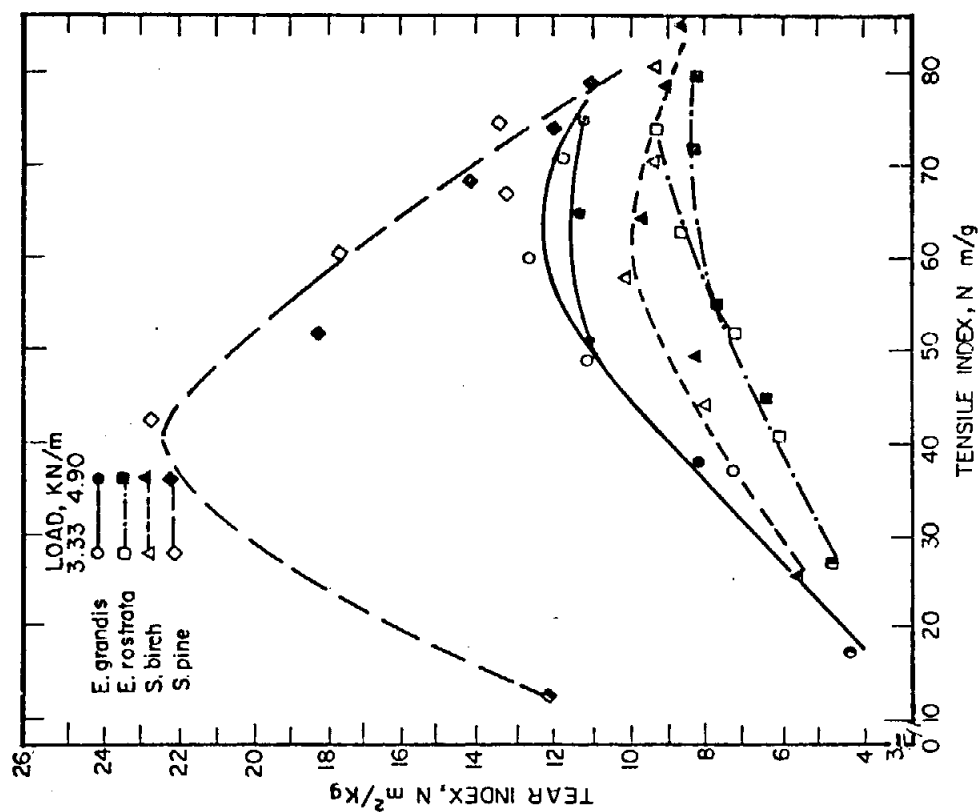


Figure 42 - Relationship between tear index and tensile index of handsheets from whole pulps.

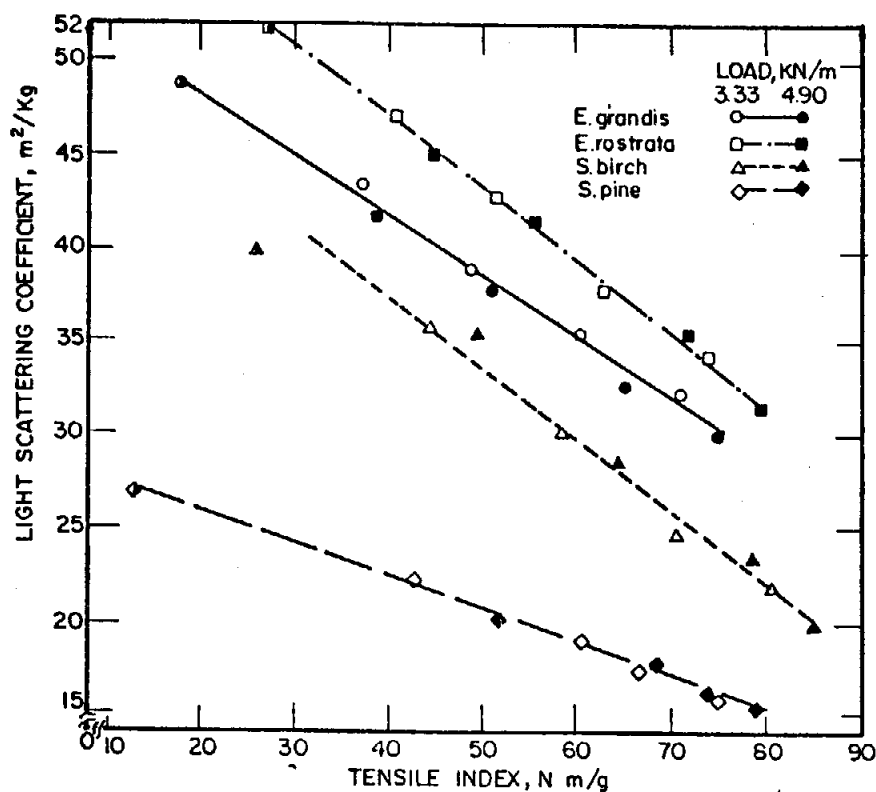


Figure 43 - Relationship between light scattering coefficient and tensile index of handsheets from whole pulps.

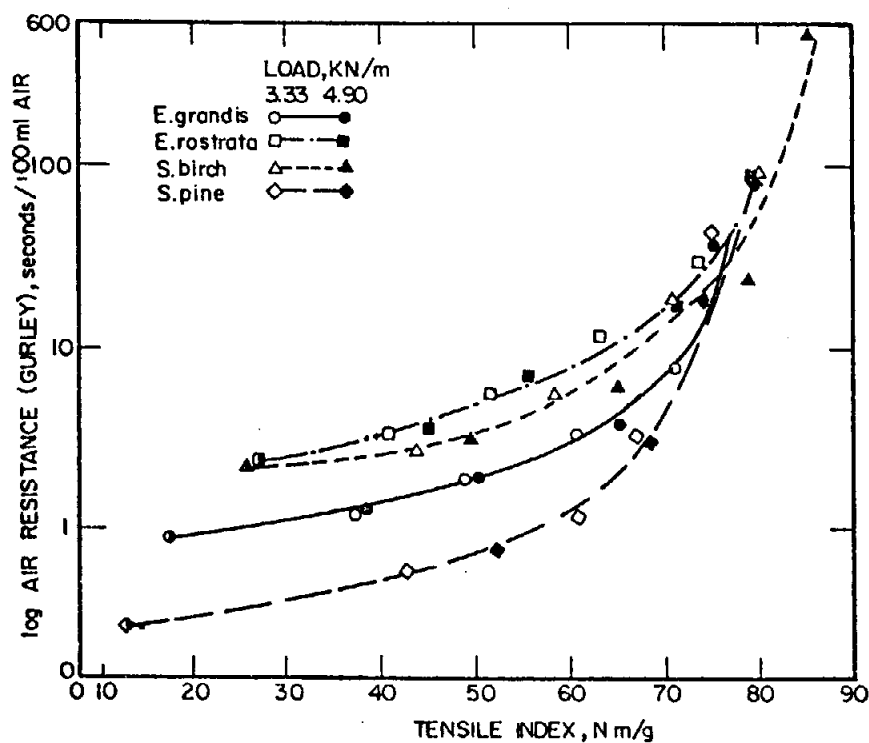


Figure 44 - Relationship between Gurley air resistance and tensile index of handsheets from whole pulps.

with no influence of fiber strength, which is lower for the pine pulp (Table VIII).

In the case of the eucalypts a large number of stiff fibers may be responsible for the density of the sheet, at the same time providing a great rigidity to the structure and a large number of interfiber contacts, the latter probably decreasing the level of stress on each of them when the paper is loaded in tensile. As to the explanation of the differences between E. rostrata and E. grandis, it could be found in the large amount of fines with smaller surface area in the former, which might be able to increase the density of the sheet without appreciably increasing the interfiber bonding. Moreover the smaller and weaker fibers of E. rostrata might not be able to withstand the stresses to the same degree as those of E. grandis.

The properties discussed above help also to understand the tear-tensile relationships plotted in Figure 42. The tear strength of paper is maybe one of its most important properties, and yet, besides the qualitative theory formulated years ago (111) not much has been published about the fundamental aspects of this subject, perhaps because the tear strength seems to be influenced by a complex interaction of variables. As is evident from Figure 42 in the early stages of beating the tear strength depends on the degree of interfiber bonding. Until the development of tear

reaches its maximum most of the work spent during the tearing resistance test is used to break interfiber bonds, the degree of which depends on fiber flexibility and the number of fibers in the sheet. Eventually the excessive bonding causes the tear to decrease, because more and more fibers are broken and less are pulled out of the network through bond breakage.

These mechanisms have been used to explain the strong influence on the tear strength of fiber length, fiber strength (231), and of the number of fibers per gram in the sheet (77). In these terms it is clear that the much longer southern pine fibers yield much larger tear values at a given tensile strength than the short-fibered hardwoods in question. The larger number of fibers per gram of E. grandis, when compared to birch would be the main reason for the higher tear values of the former, especially when considering that their fiber strengths are about the same, and that birch has even longer fibers. In this context the short and relatively weak fibers of E. rostrata are expected to yield lower tear strength values than the other two hardwoods, and this is indeed observed, in spite of the larger number of fibers per gram in the former.

The influence of the number of fibers per gram of paper on the degree of light scattering of the sheets has also been already recognized (77), and is clearly seen in the case of our own pulps in Figure 43. As beating proceeds and

the density of the paper is increased through higher web compactability, the number of fiber-air interfaces decreases, with consequent loss of the opacifying power of the network (60,79). For each type of pulp this loss in opacity is accompanied by an increase in tensile strength in a linear fashion. However, if a large number of light scattering surfaces is still available the opacity may be retained at acceptable levels while strength increases. This can be observed in Figure 43 with the two eucalypts, which show higher values of light scattering at a given tensile index. The larger number of fibers per gram associated with the presence of a big amount of ray cells are probably responsible for the higher opacity of E. rostrata when compared to E. grandis, especially when the higher bulk at a given strength is also observed for the latter. This increased light scattering power does not seem to be mostly a consequence of light diffraction at fiber wall-lumen interfaces of uncollapsed fibers, as recently suggested (116,195a), but rather it appears to be due to the presence of a large number of air spaces among the fibers, since some degree of lumen collapse was always observed, even in the unbeaten pulps (Figures 45 and 46).

With more flexible fibers, the birch pulp consolidates much better than the eucalypts, yielding sheets of high density and little air volume. Yet it is observed that for birch handsheets the number of fiber-air interfaces is

still much greater than in the case of southern pine, which with its lower density has a larger volume of air in its sheet structure than birch. The pine fibers, however, are not as numerous and do not provide the same level of the fiber surface area for light scattering. As a consequence although the birch shows lower light scattering power than the eucalypts at a given strength, the curves for the three hardwoods seem to be clustered together when compared to the southern pine, which has very low light scattering coefficients.

The observations obtained here for the four pulps studied confirm an earlier suggestion (175a) that fibers which have the highest surface area in their unbeaten state (as influenced by their dimensions and number of fibers per gram) are able to form paper of the highest opacity at a given tensile strength.

The question of the volume of air left in the dry sheet after the paper is formed leads us to the analysis of paper porosity. In the case of porosity it is the total fractional volume of air which is of importance, rather than the number of fiber-air interfaces as in opacity. When plotting the logarithm of air resistance against the tensile index (Figure 44) it is seen that the porosity increases somewhat slowly as strength is developed, with large differences among the pulps. However, as very high tensile strength levels are reached the air resistance changes dramatically,

and the curves of air resistance vs tensile for the four pulps tend to converge to a common point.

This phenomenon bears a resemblance to the beating effects on specific surface area previously observed. The tensile index was shown to be closely related to the specific volume of pulps, which rises steeply in the early beating stages and then tends to level-off with continued beating. On the other hand the specific surface area of the pulps, which changed slightly in the beginning of beating, showed a tendency to reach increasingly higher values as beating proceeded, partly due to the increased external fibrillation of the fibers, but mostly because of the continued production of fines of astonishingly higher specific surface area.

Since the porosity of paper is measured as the resistance offered by the structure to the flow of air, the increased density of the sheets achieved during beating will cause the reduction of the total fractional volume of air within the network of fibers. However, this effect appears to be negligible as compared to the filling of the empty spaces in the fibrous structure by fines of increasingly larger surface areas, (particularly at medium to high beating levels), leading to the dramatic decrease in porosity as observed in Figure 44. Thus, it can be concluded that among the properties measured in the dry paper, the porosity seems to be the only one which is largely influenced by the specific

surface area of wet pulps developed during beating.

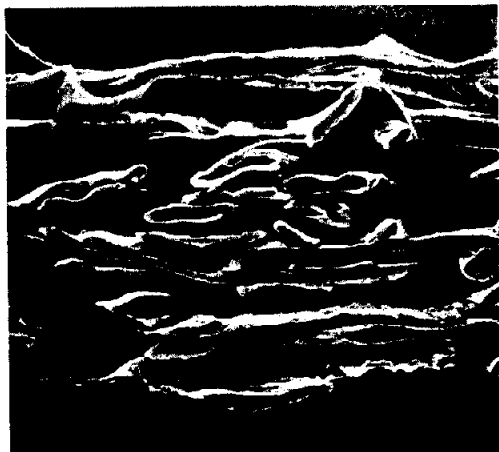
Thus the very low air resistance values of southern pine at a given strength can be explained mostly by the low levels of surface area observed for this pulp. Next in line is E. grandis, which also develops specific surface very slowly during beating, but has a much larger number of fibers per gram, leading to a somewhat lower porosity in the dry sheet. The differences in porosity between the two eucalypts at a given strength can also be accounted for mostly by the different surface areas of the respective pulps, but comparisons with birch have to include the effect of the increased density of handsheets from this pulp, resulting from the higher conformability of its fibers and consequently the reduction of air spaces in the structure. Therefore, even though the specific surface of birch is very similar to that of E. grandis (up to medium beating stages), and lower than that of E. rostrata, birch handsheets exhibit lower porosity values than E. grandis, and an air resistance - tensile strength relationship very similar to E. rostrata.

The photomicrographs of cross sections of handsheets seen in Figures 45 and 46 illustrate quite well the dry network configurations of the four species and help in the visualization of the properties discussed above. All paper sheets in the photomicrographs, have approximately the same

basis weight ( $60 \text{ g/m}^2$ ). The high bulk of E. grandis with its numerous fibers is visible in Figure 46a, with a very open structure, even after beating, and a lot of free fiber surfaces (Figure 46b). The less numerous fibers of southern pine also form a bulky structure, as seen in Figure 45a, but a somewhat more consolidated sheet is observed after beating with an energy of  $2.8 \text{ MJ/kg}$  (Figure 45b), although a lot of air spaces is still noticeable in the paper.

In the case of E. rostrata the sheet looks more consolidated in the unbeaten state (Fig. 46c) than the two previous species. The porosity of the sheet appears to be very low, but there are many fiber air interfaces visible, which should be responsible for the high opacity of this paper. The large number of fibers when compared to birch (Figure 45c) and southern pine (Figure 45a) can also be noticed, but the difference relatively to E. grandis (Figure 46a) is small, though significant. After beating ( $1.70 \text{ MJ/kg}$ ), the sheet from E. rostrata pulp appears more consolidated (Figure 46d), with less air spaces than in the case of E. grandis (Figure 46b), but still with lots of free fiber surfaces.

The handsheets from birch are undoubtedly the more consolidated among the four species (Figures 45c and 45d). Even before any beating the paper structure (Figure 45c) looks very similar to that of E. grandis beaten with  $1.9 \text{ MJ/kg}$  (Figure 46b). After beating with  $1.70 \text{ MJ/kg}$  the birch pulp



10.0 μm  
a. SEM - Southern pine handsheet,  
unbeaten pulp.



10.0 μm  
b. SEM - Southern pine handsheet,  
pulp beaten with 2.80 MJ/kg.

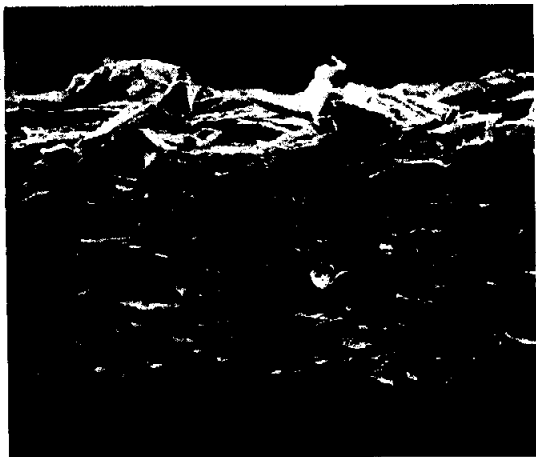


10.0 μm  
c. SEM - Silver birch handsheet,  
unbeaten pulp.

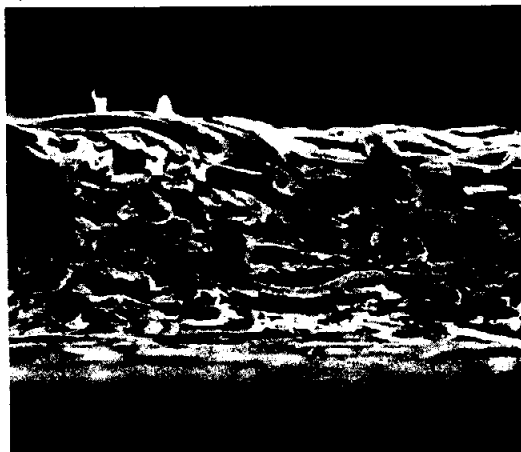


10.0 μm  
d. SEM - Silver birch handsheet,  
pulp beaten with 1.72 MJ/kg.

Figure 45 - Scanning Electron Micrographs of cross sections of  
handsheets from southern pine and silver birch.



a. SEM - E. grandis handsheet,  
unbeaten pulp.



b. SEM - E. grandis handsheet,  
pulp beaten with 1.90 MJ/kg.



c. SEM - E. rostrata handsheet,  
unbeaten pulp.



d. SEM - E. rostrata handsheet,  
pulp beaten with 1.70 MJ/kg.

Figure 46 - Scanning Electron Micrographs of cross sections of  
handsheets from E. grandis and E. rostrata.

forms a very dense sheet (Figure 45d), with many inter-fiber contacts and few spaces left for air in the structure. The fibers appear more conformable than the ones from the other three species.

In addition to the large effects on the porosity of paper, it was expected that the densifying power of the fines during sheet consolidation would have a great influence on interfiber bonding and consequently on strength and opacity of the paper. Indeed, a difference in strength, usually with a higher tear index, and lower burst and tensile was observed for handsheets prepared from fines-free pulps. However, no significant difference was detected between the light scattering power of papers with or without the fines, in agreement with previous observations (78).

The densification effect of the fines can be better visualized in Figure 47, in plot of the apparent density of handsheets prepared with whole pulps against that of handsheets from fines-free pulps. It can be noticed that the influence of the fines in density is more pronounced for southern pine and E. grandis, which, incidentally, showed a moderate response to the beating action (as indicated by the specific volume of their whole pulps and pure fibers). Silver birch appears to be the least affected by the presence of fines, while the density of handsheets from E. rostrata is strongly influenced by the large fines fractions of this pulp.

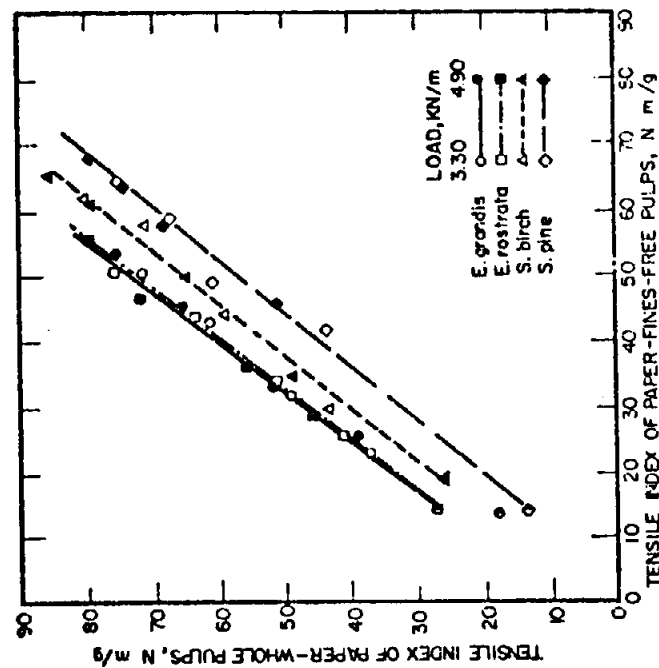


Figure 47 - Relationship between apparent density of handsheets from whole pulps and fines-free pulps.

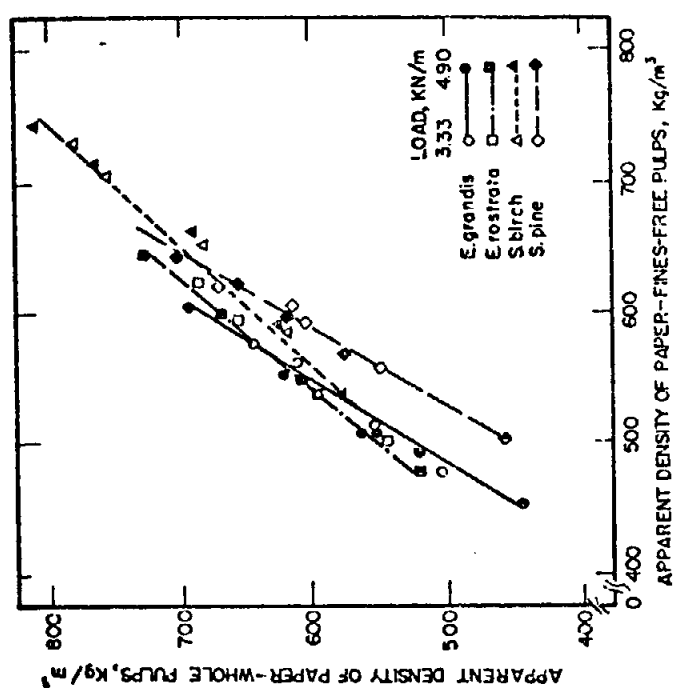


Figure 48 - Relationship between tensile index of handsheets from whole pulps and fines-free pulps.

With respect to tensile index development it is shown in Figure 48 that higher values are always obtained with fines in the sheet. The two eucalypts seem to be the most affected by the presence of fines showing a single line for the relationship between tensile strengths of whole pulps and those of pure fibers. The tensile strength of birch pulps is not much influenced by the fines, and the southern pine seems to be even less affected. It is also interesting to notice in Figure 48 that the lines are straight and appear to be parallel, i.e. the changes in tensile index of whole pulps with beating are proportional to the changes for fines-free pulps, and this proportionality factor is apparently the same for the four species studied. The statements in regard to the strength of paper are in agreement with earlier conclusions in this work that the most important changes in beating occur at the fiber level. In other words, the modifications in the internal structure of the fibers seem to be the overriding factor controlling the behavior of the wet web, as well as the strength of dry paper. External fibrillation and the subsequently formed fines have been shown to affect significantly the drainage properties and the porosity of paper, but their role in the consolidation of the sheet and formation of interfiber bonds can be considered secondary to fiber wall delamination and swelling.

Dry Strength-Wet Web Strength Relationship. - Although the strength of the dry paper is very important in its end-

use, for the papermaker it is also important to know how the same furnish behaves on the papermachine. In this context a relationship between the strength of the wet web and that of the dry paper is of considerable interest. This correlation is illustrated in Figures 49 and 50, where the tensile index of paper is plotted versus wet web tensile strength and wet web stretch, respectively.

It can be noticed from these figures that each pulp displays its own dry strength-wet strength relationship. At a given tensile index of the dry paper E. rostrata and birch show very similar wet web strengths, lower than that of E. grandis, while the southern pine exhibits the highest wet tensile. However, when comparing the wet web stretch at constant tensile index the pulps show different trends. In this case the curves of the two eucalypts are very close to each other, both with lower values of stretch than southern pine and the birch at the same tensile index. Most surprising is the behavior of birch pulp, which has stretch values larger than those of southern pine, but this situation is reversed at high beating levels.

The mechanisms involved in the development of wet web strength might help in the understanding of the phenomena observed in Figures 49 and 50. Accordingly, for a higher wet tensile strength the more flexible and swollen fibers are probably more able to consolidate into dense sheets with

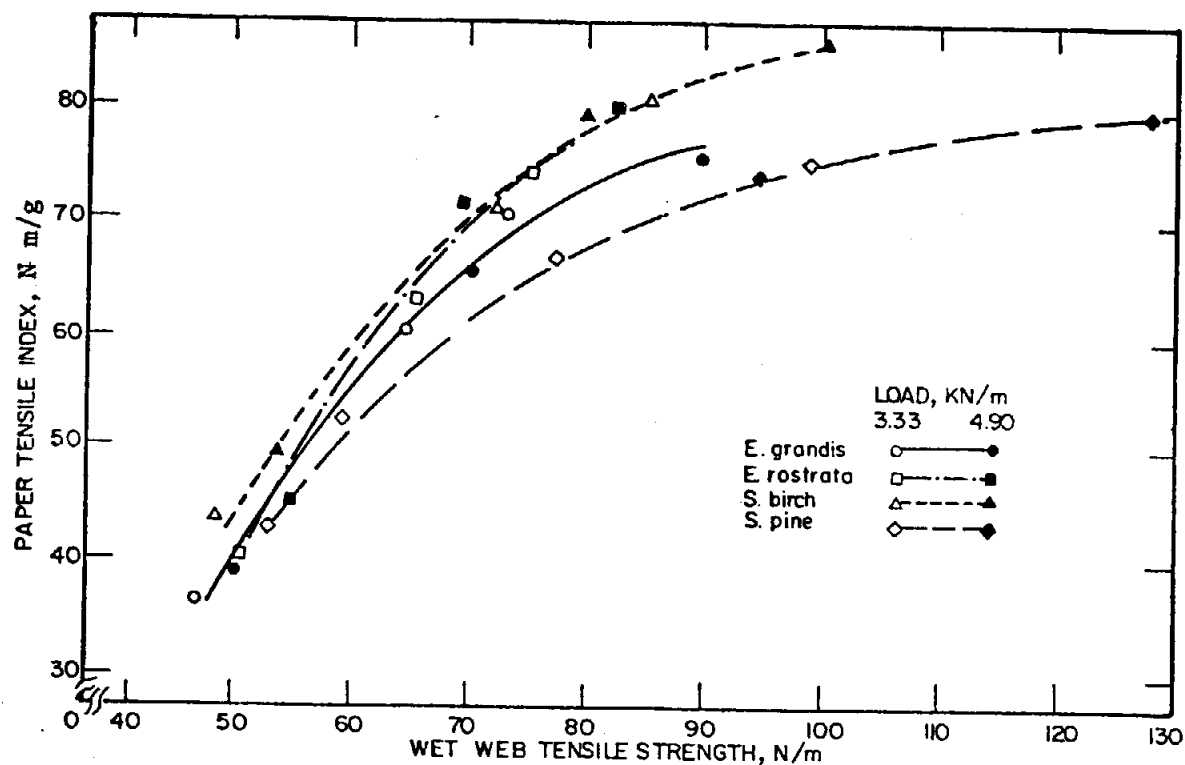


Figure 49 - Relationship between tensile index of handsheets and wet web tensile strength.

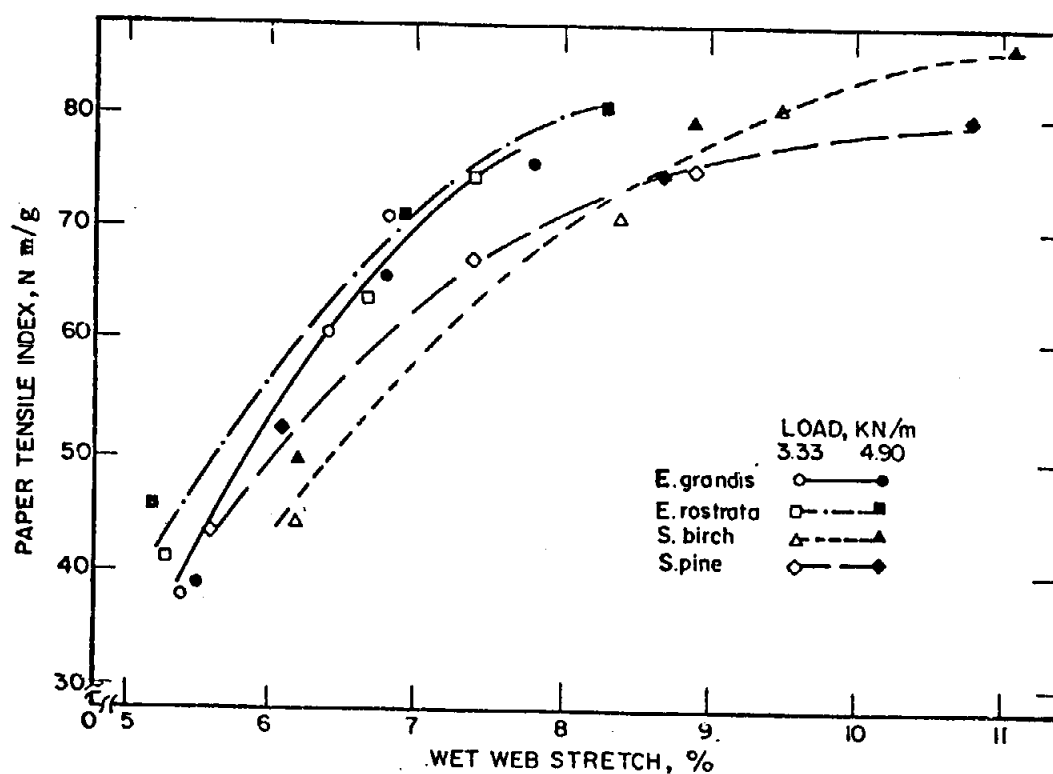


Figure 50 - Relationship between tensile index of handsheets and wet web stretch.

limited space for air-water interfaces. These well-consolidated wet webs such as observed with birch and E. rostrata would thus be influenced to a lesser extent by the surface tension forces aiding in the development of wet tensile, at solid contents over 30% than the more open structures of E. grandis and southern pine.

So, the flexibility and conformability of the fibers would ensure the formation of interfiber bonds leading to the dry paper strength, as well as it would provide better fiber interweaving in the web, leading to higher wet tensile. However, the way the water remaining in the wet web is left in association with the fibers after the consolidation of the network would influence greatly the wet strength through the surface tension forces at air-water interfaces, with practically no influence on the dry strength. As a consequence the more open and less swollen structures, such as those of E. grandis and southern pine webs are able to display larger wet web tensile strength at the same tensile index when compared to birch and E. rostrata, due to influence of surface tension forces, which probably happen to a larger extent for the former two pulps.

When considering the wet stretch it seems that fiber flexibility and the density of the network are the overriding factors. In this context the very flexible fibers of birch pulp should be able to provide a high degree of interweaving,

resulting in better conformability among the fibers when the web is loaded and a higher degree of stress redistribution without rupture of the structure. This mechanism could explain why the eucalypts show lower stretch values than the birch of a given dry tensile index, but the wet elongation data of southern pine are more difficult to understand, especially when considering that the average stiffness of its fibers is the largest among the four pulps.

It seems possible that in this case the larger length of southern pine fibers might be playing an important role in the development of wet web stretch at a given tensile index.

## SUMMARY AND CONCLUSIONS

The beating process was studied using bleached kraft pulps from four species of increasing importance in the papermaking field: southern pine, silver birch, Eucalyptus rostrata, and Eucalyptus grandis. Particular emphasis was directed towards the influence of fiber structure on the response of the pulps to beating.

The structure of fibers was evaluated in the four pulps and respective original wood samples from which the pulps were manufactured. The characteristics analyzed included morphology, using microscopic and ultramicroscopic techniques, and chemical composition of the pulps. Special attention was directed to the measurements of average fiber cross sectional dimensions, and to the thickness and microfibrillar angles in the various cell wall layers.

It was found that the four pulps differed greatly in their geometry, morphology and chemical composition. Thus, southern pine has the longest and coarser fibers, the eucalypts the shortest, thinnest and more numerous fibers, while birch fibers are intermediate between them. The ultrastructural organization of the fibers was described by the microfibrillar orientations in  $S_1$  and  $S_2$  layers and the density of the cell wall, which were very helpful in the identification of the differences between birch and the

eucalypts, and, on the other hand revealed unexpected similarities between these hardwoods and the pine. The ultramicroscopic characteristics appear to be closely related to the transverse dimensions of the fibers, independently of species, and were postulated to follow a general pattern influenced by growth conditions. The chemical differences among the studied pulps were found to be mostly in their hemicellulose contents.

The evaluation of the hydrodynamic properties of the pulps was proven very adequate in the analysis of beating. The advancement of the beating action was followed at various beating levels under two different beating loads, by measurements of the specific surface area and specific volume of the resulting pulps. The same measurements were also performed on pulps from which the fines fractions, (determined quantitatively), were separated. This approach led to an adequate quantitative monitoring of the beating process.

Although deriving from very different species, the pulps responded to the beating treatment following somewhat similar trends. Accordingly, the changes in the internal structure of all four types of fibers occur very rapidly in the early stages of energy absorption, and tend to gradually diminish as the beating proceeds. The same changes, although in a lesser extent, are observed for the fines-free pulps.

A different behavior was observed with respect to external fibrillation and the formation of fines. The detachment of fibrils from the surface of the fibers and the subsequent formation of fines depend linearly on the energy applied to the pulps. These fines show very peculiar characteristics; namely their high swelling potential and, most importantly, a very large specific surface area. The latter increases almost exponentially as beating proceeds. Evidently the rate of creation of the specific surface of fines influences that of the whole pulps, which then display a slow increase in specific surface at the early beating stages, followed by a more rapid surface area development in the more advanced beating. Internal and external fibrillation appear to occur at the same time, but there was no evidence of interdependence between them.

As to the effect of the beating load, the main difference in the response of the four pulps is reflected in the specific surface area. As a higher load is applied in a given period of time a faster development of specific surface can be observed in all whole pulps and pure fibers than with lower pressures in the beating gap. The largest differences are observed in the case of southern pine, which shows a better utilization of energy for changes in the internal and external structures of the fibers. For the three hardwoods, however, apart from a faster development in

external fibrillation and larger amounts of produced fines than the lower load, a higher load does not prove more beneficial at a given energy expenditure.

In the instance of the four studied pulps the general patterns observed during beating can be related to the basic mechanisms underlying the beating action, while each individual pulp's behavior is believed to be caused by the ultra-structural and chemical characteristics of its fibers. In this context the extent to which the fibers interact when in suspension should not be neglected, because the so-called flocs are indeed the structures which are impacted in the beating gap.

Since fiber aggregates, rather than single fibers are present in the beating zone, the study of the behavior of the flocs helps to understand the beating process itself at a macroscopic level. The properties of the flocs are affected by the initial fiber properties, and in turn they affect how the energy is transmitted to the individual fibers. The microscopic aspect of beating involves the changes occurring in the fibers' internal and external structures, and how these modifications can alter the behavior of the flocs with continuation of the mechanical treatment. Although the emphasis in this study was directed towards the analysis of the development of fiber properties, some indication as to the changes occurring at a macroscopic level was provided by

a follow-up of the beating clearance in the process.

The close relationship between the changes in the beating gap during the treatment and the more readily accomplished variations in the swelling properties of pulps with energy consumption led to the conclusion that internal wall delamination and swelling, with consequent increase in fiber flexibility not only is the main beating effect but it also influences the behavior of the flocs in the beating gap. The changes imparted to the properties of the flocs, in turn, seem to control the way the whole process proceeds.

Furthermore it was found that the increase in strength of the wet web and of paper are also significantly dependent on the changes in the swelling behavior of the pulps during beating. These facts were conclusive in pointing out to flexibility of the fibers as the key factor to be considered in papermaking.

Ultrastructural, geometrical and chemical data allowed the estimation of fiber flexibility utilizing a two-dimensional mechanical analysis of the behavior of the cell wall. The results of such analysis obtained for the fibers of the four species could explain their conformability in the sheet, as well as give indications about the macroscopic behavior of the flocs in the beating zone and provide a better understanding for the different beatabilities of the corresponding pulps.

The ease with which a pulp is beaten to achieve a desired characteristic in the wet web or in the dry paper depends on the initial degree of structural rigidity in the fiber wall and on the facility with which internal hydrogen bonds are broken during the mechanical treatment.

Fibers such as birch, with high hemicellulose content, large microfibrillar angle, and low packing density, not only are more flexible in the unbeaten state, but as the beating energy is applied the less packed microfibrillar arrangement can be separated more easily, due to the small number of microfibrils and to the large amounts of hemicellulose among them. As a consequence of the observed steeper helices in their  $S_2$  layers and higher cell wall densities than birch, it could be inferred that fibers from both eucalypt species have smaller crystallite widths and a higher degree of packing of microfibrils. Therefore, the eucalypt fibers are not only stiffer than birch in the unbeaten state, but also as loads are imposed on them the smaller and more numerous microfibrils will offer considerable greater surfaces for stress distribution than birch fibers.

In addition, the lower content of hemicelluloses in the eucalypts would be expected to increase the possibility of cross-linking among the microfibrils, which require larger energy expenditures for cleavage, and decrease the plasticization ability of the fibers at a given energy expenditure.

These effects are particularly important in the case of Eucalyptus grandis pulp, which has a hemicellulose content even lower than that of E. rostrata, making the process of internal bond breakage and fibril separation even more difficult for the former. This seems to be the most important influence of hemicellulose in the beating of pulps, and explains why the fibers from these two eucalypt species display different beatabilities, in spite of being structurally so similar.

Due to the high content of latewood the southern pine fibers have average ultrastructural characteristics very similar to the eucalypts. Small angles in the  $S_2$  layer and the high cell wall density should be also responsible for the large stiffness of southern pine fibers and their poor response to the beating action. As indicated by the measurements of the beating effects, the pine and E. grandis show very similar rates of changes in their internal structure, which could be due both to their comparable ultrastructure and similar hemicellulose contents.

Although it is possible that as more delamination and swelling is induced into the fibers the outer fibrils may be loosened up more easily, this process seems to proceed almost independently of what happens externally to the fibers. The development of external fibrillation can be understood as being mostly a consequence of the friction

occurring between the fibers as the flocs are sheared and compressed. The result from these interactions would depend on characteristics of the fiber's  $S_1$  layers, among them their physical integrity (as influenced by their hemicellulose content and the state of aggregation of the microfibrils) and their orientation relatively to the fiber axis. Fibers with more hemicellulose and large angles in  $S_1$  helices such as those of birch are more likely to fibrillate externally and produce fines than the fibers of eucalypts and southern pine, which have steeper  $S_1$  helices and lower hemicellulose contents.

As a first consequence of the changes imparted to a pulp during beating the rate of water removal in the forming zone of a paper machine is directly influenced by the newly formed surfaces of the fines and those on the exterior of the fibers. In this connection the logarithm of a simulated dynamic drainage rate was shown to have a single linear relationship with the square power of the specific surface area of the four pulps studied. No relationship was found between the drainage rate of the pulps and their respective freeness values, although for each pulp the development of specific surface area was accompanied by a proportional decrease in freeness, with different relationships for the two beating loads applied.

The wet web strength (both tensile strength and elongation) showed a straight line relationship with beating

energy, at both applied loads. A higher load at a given beating power intake provides a more efficient use of energy towards wet web strength development, although this occurs at the expense of poorer drainage characteristics. The strength of the wet web depends strongly on the ability of the fibers to form networks that consolidate well, not only due to the flexibility of the fibers, (which assists in interweaving and conformability), but also enhanced by the increase of the specific surface area of the pulps.

For both wet tensile and stretch the overriding factor controlling the behavior of different pulps is the degree of flexibility of the fibers. The wet tensile strength seems to be additionally influenced by the fines and the degree of external fibrillation of the fibers, which promote the surface tension forces as air enters the structure, thereby increasing the friction between the fibers when the web is stressed. The wet web elongation, however, seems to be mostly influenced by the degree of consolidation of the network, in which the specific surface area plays an important role, although secondary to the effect of increased fiber flexibility. A small influence of fiber length was also observed on the ability of the webs to stretch prior to rupture.

For the dry paper, with the exception of the porosity, which seems to be strongly influenced by the specific surface area of the pulps, the strength properties and the

opacity show a strong dependence on initial pulp characteristics (such as the degree of fiber flexibility and the number of fibers per gram), and on the modifications in the internal structure of the fibers during beating. The role of external fibrillation and the subsequently formed fines in the consolidation of the sheet and formation of inter-fiber bonds can be considered secondary to fiber wall delamination and swelling.

Hence, independently of species, whether hardwoods or softwoods, it can be concluded that at the fiber level the degree of association of microfibrils, their width and number (as indicated by the packing density of the cell wall), and the fibrillar angles of the  $S_1$  and  $S_2$  layers are the controlling factors of the response of a pulp to beating, rather than simply the thickness of the fiber wall as related to fiber width. At the macroscopic level much research is still needed for a better understanding of the role of fiber flocs in the beating process. The study of flocs, including the relationships to fiber properties and their mechanical behavior when under shear and compression forces would contribute significantly towards a more advanced beating theory.

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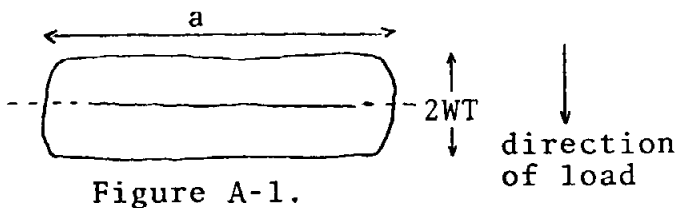
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## APPENDIX A

CALCULATION OF MOMENT OF INERTIA AND  
AXIAL YOUNG'S MODULUS OF PULP FIBERS

APPENDIX A. CALCULATION OF MOMENT OF INERTIA AND  
AXIAL YOUNG'S MODULUS OF PULP FIBERS

Moment of Inertia



The moment of inertia of a rectangle, with the axis of the moment through its center (dashed line) is given by

$$I = \frac{a \times (2WT)^3}{12}$$

where  $a = \frac{\text{Fiber Perimeter} - 4WT}{2}$

and WT = fiber wall thickness.

The moments of inertia for the fibers of each pulp were calculated from the measured fiber perimeter and wall thickness on pulp fibers, as described in the text, and listed in Table VI.

Young's Modulus - The calculations were performed using a computer program, and were based on a two-dimensional analysis of the mechanical behavior of the cell wall under axial tension without shear restraint (150). The proportions of cell wall layers (Table II- text), their respective microfibrillar orientations (Table IV - text) and chemical composition of the fibers (Table V - text) were used in the calculations. The proportion of primary wall

was assumed to be equal to one percent for all types of fibers. The microfibrillar angles in the primary wall and  $S_3$  layer were assumed equal to 90 and 80 degrees respectively, as these values were required by the computer program.

The elastic constants used for cellulose were the same as "Case 2" calculated by Gillis (81), and are listed in Table A-I. The elastic constants for "wet" hemicellulose were taken as ten percent of the values derived by Mark (149), which are in agreement with more recent measurements (40). These values are listed in Table A-II. The respective calculated axial Young's modulus of pulp fibers are reported in Table VI - text.

TABLE A-I. ELASTIC CONSTANTS OF CELLULOSE

Axial Young's Modulus	246.45 GPa
Transverse Young's modulus	16.45 GPa
Shear Modulus of Rigidity	0.17 GPa
Poisson Ratios (axial/transverse, transverse/axial)	0.041, 0.0038

TABLE A-II. ELASTIC CONSTANTS OF WET HEMICELLULOSE

Young's Modulus	0.20 GPa
Shear Modulus of Rigidity	0.08 GPa
Poisson Ratio	0.30

APPENDIX B  
WET WEB STRENGTH DATA

TABLE B-I. WET WEB PROPERTIES \_ SOUTHERN PINE

PFI Revolu- tions	Beating Load, kN/m	Dry Content, %	Wet Web Tensile, N/m	Stretch, %	Web Caliper, mm
1500	3.33	Whole Pulp 34.7±0.2	53.0±1.0	5.6±0.2	0.245±0.003
		Fibers 35.6±0.3	44.0±1.0	4.5±0.2	0.256±0.002
	4.90	Whole Pulp 33.5±0.4	59.0±2.0	6.1±0.3	0.240±0.002
		Fibers 34.3±0.5	49.0±1.0	4.9±0.2	0.255±0.003
5000		Whole Pulp 32.6±0.2	78.0±2.0	7.4±0.5	0.224±0.003
	3.33	Fibers 33.8±0.3	66.0±2.0	5.5±0.2	0.234±0.003
	4.90	Whole Pulp 31.6±0.3	95.0±2.0	8.7±0.3	0.221±0.001
		Fibers -	-	-	-
8000	3.33	Whole Pulp 32.3±0.3	98.0±2.0	8.9±0.3	0.217±0.002
		Fibers 33.8±0.2	72.0±1.0	5.9±0.3	0.228±0.003
	4.90	Whole Pulp 30.6±0.3	129.0±3.0	11.0±0.4	0.215±0.001
		Fibers 33.6±0.3	86.0±2.0	7.2±0.3	0.225±0.003

TABLE B-II. WET WEB PROPERTIES - SILVER BIRCH

PFI Revolu- tions	Beating load, kN/m	Dry Content, %	Wet Web Tensile, N/m	Stretch, %	Web Caliper, mm
500	3.33	Whole Pulp	35.1±0.5	48.0±1.0	6.2±0.2
		Fibers	36.6±0.4	38.0±1.0	4.8±0.2
	4.90	Whole Pulp	35.1±0.6	54.0±1.0	6.2±0.2
		Fibers	35.7±0.3	40.0±1.0	5.1±0.2
3000	3.33	Whole Pulp	35.5±0.3	73.0±2.0	8.4±0.2
		Fibers	34.4±0.4	56.0±2.0	6.6±0.2
	4.90	Whole Pulp	32.8±0.4	81.0±2.0	8.9±0.3
		Fibers	-	-	-
5000	3.33	Whole Pulp	32.6±0.4	86.0±2.0	9.5±0.4
		Fibers	34.1±0.4	67.0±2.0	6.7±0.3
	4.90	Whole Pulp	31.5±0.3	102.0±2.0	11.1±0.4
		Fibers	33.5±0.5	79.0±2.0	8.5±0.2

TABLE B-III. WET WEB PROPERTIES - *Eucalyptus grandis*

PFI Revolu- tions	Beating load kN/m		Dry Content, %	Wet Web Tensile, N/m	Stretch, %	Web Caliper, mm
500	3.33	Whole Pulp	34.2±0.5	47.0±1.0	5.4±0.2	0.241±0.003
		Fibers	35.8±0.6	35.0±0.1	4.2±0.1	0.271±0.001
	4.90	Whole Pulp	34.0±0.4	51.0±1.0	5.5±0.2	0.240±0.003
		Fibers	35.4±0.3	36.0±0.1	4.4±0.1	0.265±0.002
3000	3.33	Whole Pulp	33.6±0.4	65.0±1.0	6.4±0.2	0.224±0.001
		Fibers	34.6±0.2	45.0±1.0	4.6±0.4	0.245±0.002
	4.90	Whole Pulp	33.2±0.4	71.0±1.0	6.8±0.3	0.221±0.001
		Fibers	-	-	-	-
5000	3.33	Whole Pulp	32.9±0.6	73.0±2.0	6.8±0.3	0.217±0.003
		Fibers	33.8±0.4	52.0±1.0	4.9±0.2	0.245±0.002
	4.90	Whole Pulp	32.5±0.4	90.0±2.0	7.8±0.2	0.212±0.001
		Fibers	33.6±0.5	59.0±1.0	5.9±0.2	0.234±0.001

TABLE B-IV. WET WEB PROPERTIES - Eucalyptus rostrata

PFI Revolu- tions	Beating Load kN/m		Dry Content, %	Wet Web Tensile, N/m	Stretch, %	Web Caliper, mm
500	3.33	Whole Pulp	34.6±0.4	51.0±1.0	5.3±0.2	0.228±0.001
		Fibers	35.9±0.5	37.0±0.1	3.7±0.2	0.249±0.002
	4.90	Whole Pulp	34.3±0.4	55.0±1.0	5.2±0.3	0.226±0.002
		Fibers	34.9±0.6	38.0±1.0	3.9±0.2	0.248±0.003
3000	3.33	Whole Pulp	33.5±0.4	66.0±1.0	6.7±0.2	0.214±0.002
		Fibers	34.5±0.5	46.0±1.0	4.9±0.2	0.237±0.002
	4.90	Whole Pulp	32.9±0.3	69.0±1.0	6.9±0.2	0.211±0.001
		Fibers	-	-	-	-
5000	3.33	Whole Pulp	32.5±0.4	76.0±2.0	7.4±0.3	0.213±0.002
		Fibers	33.9±0.4	51.0±1.0	5.4±0.3	0.231±0.001
	4.90	Whole Pulp	32.3±0.3	83.0±1.0	8.3±0.3	0.209±0.002
		Fibers	34.1±0.4	58.0±1.0	6.2±0.3	0.223±0.003

## APPENDIX C

DATA FROM TESTING OF STANDARD HANDSHEETS

FI revolutions	Beating load, kN/m	Net beating energy, MJ/kg		Tensile index, N m/g	Burst index, KPa m <sup>2</sup> /g	Tear Index, N m <sup>2</sup> /kg	Apparent density, kg/m <sup>3</sup>	Light scattering coefficient, m <sup>2</sup> /kg	Gurley Air Resistance, sec/100 ml air
0		0	whole pulp	13.2±0.4	0.67±0.13	12.2±1.4	455±8	26.8±1.9	0.3±0.1
			fibers	14.8±0.6	0.60±0.04	13.2±0.9	502±9	23.4±3.1	-
500	3.33	1.37	whole pulp	42.7±1.3	3.10±0.23	22.6±0.6	551±5	22.1±0.5	0.6±0.2
			fibers	41.9±1.7	2.50±0.11	25.2±1.8	565±10	21.1±0.6	-
	4.90	1.52	whole pulp	52.4±1.2	3.80±0.13	18.2±0.2	576±4	20.0±0.6	0.8±0.2
			fibers	45.8±1.4	3.10±0.26	20.8±1.3	572±9	20.2±0.8	-
000	3.33	2.76	whole pulp	60.7±2.9	4.55±0.28	17.6±0.3	607±7	18.9±0.5	1.2±0.3
			fibers	48.4±2.1	3.88±0.36	21.9±1.0	591±6	19.5±0.6	-
	4.90	3.08	whole pulp	68.5±2.0	5.3±0.39	14.0±0.7	622±8	17.8±0.4	3.0±0.3
			fibers	56.9±1.1	4.2±0.28	16.7±1.5	594±4	18.5±0.4	-
000	3.33	4.47	whole pulp	67.0±2.0	5.59±0.28	13.2±0.5	620±11	17.3±0.5	3.3±0.3
			fibers	59.2±1.4	4.40±0.40	16.5±1.4	615±4	17.9±0.5	-
	4.90	4.92	whole pulp	74.1±2.2	5.98±0.28	11.3±0.6	658±6	16.1±0.4	17.9±0.9
			fibers	63.0±1.7	5.02±0.29	15.1±0.7	625±4	17.6±0.4	-
000	3.33	6.94	whole pulp	74.9±2.0	6.22±0.46	13.3±0.4	671±5	15.9±0.4	41.8±3.6
			fibers	62.9±2.4	4.60±0.21	16.9±0.9	624±7	17.6±0.5	-
000	4.90	7.49	whole pulp	79.5±2.6	6.72±0.36	10.8±0.7	703±6	15.4±0.5	78.0±5.2
			fibers	68.0±2.2	5.25±0.14	15.0±1.2	640±5	17.0±0.3	-

TABLE CII. PAPER PROPERTIES OF STANDARD HANDSHEETS - SILVER BIRCH

PFI revolutions	Beating load kN/m	Net beating energy, MJ/kg	Tensile index, N m/g	Burst index, kPa m <sup>2</sup> /g	Tear index, N m <sup>2</sup> /kg	Apparent density, kg/m <sup>3</sup>	Light scattering coefficient, m <sup>2</sup> /kg	Gurley air resistance, sec/100 ml air
0		0	whole pulp	26.1±1.7	1.36±0.11	6.0±0.6	583±7	59.8±1.6
			fibers	18.1±0.7	0.74±0.11	4.8±0.5	546±4	40.8±0.2
500			whole pulp	44.3±0.9	2.70±0.17	7.9±0.6	621±9	35.8±0.6
	3.33	0.41	fibers	30.1±1.3	1.51±0.08	6.4±0.8	591±5	36.6±0.7
		0.45	whole pulp	49.8±1.8	3.00±0.12	8.2±0.5	622±9	35.4±0.4
	4.90		fibers	35.3±1.3	1.51±0.17	7.5±0.5	595±10	35.1±0.5
1500			whole pulp	58.3±1.0	4.07±0.15	10.0±0.6	681±8	30.0±0.4
	3.33	1.26	fibers	44.4±1.3	2.85±0.15	11.8±1.3	651±6	30.5±0.7
		1.33	whole pulp	64.6±1.6	4.42±0.20	9.5±0.6	693±5	28.5±0.5
	4.90		fibers	50.0±1.3	2.93±0.17	10.8±1.0	660±7	31.0±0.7
3000			whole pulp	70.7±2.0	5.44±0.13	9.3±0.3	752±4	24.6±0.2
	3.33	2.46	fibers	57.8±1.8	3.81±0.11	9.7±0.8	709±6	26.7±0.5
		2.56	whole pulp	79.1±1.9	5.60±0.28	8.9±0.3	760±4	23.4±0.4
	4.90		fibers	60.5±1.4	5.02±0.29	10.2±0.7	714±3	25.4±0.3
5000			whole pulp	80.2±2.0	6.41±0.21	9.1±0.2	776±5	21.7±0.3
	3.33	3.93	fibers	62.0±1.8	4.13±0.10	10.6±0.4	738±5	23.9±0.3
			whole pulp	85.5±2.6	6.45±0.10	8.4±0.3	818±3	19.8±0.3
	4.90	4.07	fibers	65.2±1.4	4.57±0.14	10.8±0.6	748±3	22.8±0.3

TABLE C-III. PAPER PROPERTIES OF STANDARD HANDSHEETS - EUCALYPTUS GRANDIS

revolu- ns	Beating load kN/m	Net beating energy, MJ/kg	Tensile index, N m/g	Burst index, kPa m <sup>2</sup> /g	Tear index, N m <sup>2</sup> /kg	Apparent density, kg/m <sup>3</sup>	Light scattering coefficient, sec/100 ml air	Gurley air resistance sec/100 ml air	
0		0	whole pulp fibers	17.9±0.9 13.9±0.6	0.97±0.14 0.41±0.06	4.2±0.5 3.3±0.5	442±7 455±11	48.6±0.7 44.2±0.4	0.9±0.3 -
	3.33	0.41	whole pulp fibers	37.4±1.2 22.8±1.4	2.14±0.14 0.83±0.06	7.2±0.8 4.0±0.6	501±9 483±9	43.4±0.9 41.7±0.5	1.2±0.2 -
	4.90	0.47	whole pulp fibers	38.5±2.4 26.4±1.4	2.30±0.16 0.93±0.08	8.0±0.6 4.8±0.3	518±11 499±6	41.6±0.9 40.4±0.6	1.3±0.3 -
	3.33	1.26	whole pulp fibers	49.0±2.1 32.1±1.5	3.20±0.16 1.62±0.09	11.1±1.1 6.7±0.8	553±6 516±6	38.8±2.3 38.9±0.6	1.8±0.3 -
10	4.90	1.38	whole pulp fibers	50.7±0.8 34.0±0.9	3.36±0.18 1.90±0.09	10.9±1.6 7.9±0.6	564±7 511±3	37.8±0.9 38.3±0.6	1.9±0.3 -
	3.33	2.55	whole pulp fibers	60.5±1.1 43.2±1.1	4.56±0.14 2.54±0.06	12.6±1.0 7.6±0.6	604±7 569±8	35.4±0.7 34.5±0.6	3.4±0.4 -
	4.90	2.68	whole pulp fibers	65.2±1.2 45.2±1.9	4.90±0.29 2.73±0.12	11.2±1.3 9.9±1.3	623±9 554±13	32.6±0.2 34.0±0.6	3.8±0.5 -
	3.33	3.95	whole pulp fibers	70.8±1.6 51.0±1.5	5.59±0.24 3.07±0.14	11.7±0.5 9.5±1.1	642±6 578±8	32.4±0.4 33.3±0.7	7.9±0.6 -
10	4.90	4.32	whole pulp fibers	75.2±2.5 53.4±1.2	6.0±0.19 3.11±0.13	11.2±0.4 11.4±1.6	687±3 608±7	30.0±0.9 31.0±0.3	35.7±3.8 -

TABLE C-IV. PAPER PROPERTIES OF STANDARD HANDSHEETS - EUCALYPTUS ROSTRATA

PFI revolutions	Beating Load kN/m	Net beating energy, MJ/kg	Tensile index, N m/g	Burst index, kPa m <sup>2</sup> /g	Tear index, N m <sup>2</sup> /kg	Apparent density, kg/m <sup>3</sup>	Light scattering coefficient, m <sup>2</sup> /kg	Gurley air resistance, sec/100 ml air
0		0	whole pulp 27.2±0.9	1.09±0.06	4.6±0.4	521±4	52.0±0.4	2.4±0.2
			fibers 16.3±0.8	0.42±0.10	3.0±0.2	481±8	50.5±0.6	-
	3.33	0.39	whole pulp 40.7±1.1	2.10±0.18	6.0±0.5	543±5	47.0±0.3	3.3±0.1
			fibers 25.8±1.1	1.04±0.11	4.2±0.6	509±7	45.2±0.9	-
500	4.90	0.42	whole pulp 45.1±1.1	2.20±0.18	6.3±0.7	550±7	44.9±0.8	3.5±0.2
			fibers 27.8±0.8	0.95±0.09	4.2±0.5	515±8	44.7±0.5	-
	3.33	1.21	whole pulp 51.8±3.0	3.30±0.12	7.2±0.9	593±5	42.7±0.3	5.6±0.3
			fibers 34.9±0.9	1.80±0.09	5.8±0.3	548±5	38.9±0.2	-
1500	4.90	1.29	whole pulp 55.5±0.9	3.65±0.21	7.5±0.4	603±5	41.4±0.5	6.8±0.5
			fibers 36.0±1.5	1.80±0.14	7.0±0.6	551±7	40.7±0.7	-
	3.33	2.33	whole pulp 62.7±1.9	4.83±0.24	8.5±0.4	655±5	37.9±0.2	11.4±0.9
			fibers 44.2±1.4	2.59±0.14	7.9±0.6	590±10	37.8±0.3	-
3000			whole pulp 71.8±1.8	5.11±0.15	8.1±0.6	670±6	35.3±0.3	17.0±1.5
	4.90	2.52	fibers 46.8±1.1	2.85±0.15	8.6±1.2	600±8	35.7±0.2	-
	3.33	3.73	whole pulp 73.9±2.3	5.24±0.50	9.2±0.7	682±12	34.2±0.4	30.0±4.3
			fibers 52.1±1.4	3.20±0.16	9.0±0.7	626±6	34.3±0.2	-
5000	4.90	4.07	whole pulp 79.7±1.8	5.45±0.31	8.2±0.4	727±5	31.5±0.8	87.2±8.9
			fibers 56.4±2.1	3.70±0.15	9.4±0.4	647±7	32.5±0.7	-

## VITA

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High School	Colegio de Aplicação da Faculdade de Educação da UFRJ		
University	School of Chemistry of the Federal University of Rio de Janeiro (UFRJ) Rio de Janeiro-RJ-Brazil	1971- 1975	BS Chemical Engineering
	Coordination of Programs for Graduate Studies in Engineering (COPPE/UFRJ) Rio de Janeiro-RJ-Brazil	1975- 1976	MS Chemical Engineering
	Technical University of Norway Trondheim-Norway	1977- 1978	Continuing Education- Pulp and Paper Technology

## Employment Experience:

Employer	Dates	Position
COPPE/UFRJ- Dept. Metallurgical Engineering	1972- 1973	Technical Assistant
COPPE/UFRJ Dept. Chemical Engineering	1973- 1974	Computer Programmer
UNICAMP-State University of Campinas - SP - Brazil	1975- 1977	Instructor/Research Assistant
ARACRUZ CELLULOSE, S.A. Aracruz-ES-Brazil	1977- present	Research Engineer