Effect of Refining on the Brightness of Softwood CTMP and TMP Pulps

Ashok Kumar Mishra

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EFFECT OF REFINING ON THE BRIGHTNESS
OF SOFTWOOD CTMP AND TMP PULPS

by
Ashok Kumar Mishra

A Thesis
Submitted to the
Faculty of The Graduate College
in partial fulfillment of the
requirements for the
Degree of Master of Science
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and Engineering

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EFFECT OF REFINING ON THE BRIGHTNESS
OF SOFTWOOD CTMP AND TMP PULPS

Ashok Kumar Mishra, M.S.
Western Michigan University, 1990

The main objective of this study was to investigate the effect of refining on the brightness of softwood chemithermomechanical (CTMP) and thermomechanical (TMP) pulps. Seven different pulps were refined using a laboratory Valley beater. Brightness, opacity, tensile index, TEA, and fines content of these pulps were determined at four different freeness levels. Scattering (s) and absorption (k) coefficients of these pulps were calculated. One-way analyses of variance (ANOVA) were applied to determine the significance of change in brightness.

Changes in s-values were significantly affected by fiber morphology of different wood species. Refining increased the k-values of the pulps. When the increase in s-value outweighed the increase in k-value, the result was an increase in brightness. Southern pine unbleached TMP showed increases, while bleached CTMP pulps showed decreases in brightness with refining. Other pulps did not exhibit a definite trend. Opacity and tensile strength generally increased with refining.
ACKNOWLEDGEMENTS

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Special recognition goes to my parents; my wife, Suman and; my sons, Rahul and Manish; for their patience, love, support, and sacrifice needed to bring this study to completion.

Ashok Kumar Mishra
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<thead>
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<th>Description</th>
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<tbody>
<tr>
<td>ANOVA</td>
<td>Analysis of Variance</td>
</tr>
<tr>
<td>BCTMP</td>
<td>Bleached Chemithermomechanical Pulp</td>
</tr>
<tr>
<td>Chemi.</td>
<td>Chemical</td>
</tr>
<tr>
<td>CPPA</td>
<td>Canadian Pulp and Paper Association</td>
</tr>
<tr>
<td>C.S.F.</td>
<td>Canadian Standard Freeness</td>
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<tr>
<td>CTMP</td>
<td>Chemithermomechanical Pulp</td>
</tr>
<tr>
<td>cu.</td>
<td>Cubic</td>
</tr>
<tr>
<td>DTPA</td>
<td>Diethyltriaminepentaacetic Acid</td>
</tr>
<tr>
<td>EDTA</td>
<td>Ethylenediaminetetraacetic Acid</td>
</tr>
<tr>
<td>k</td>
<td>Absorption Coefficient</td>
</tr>
<tr>
<td>kPa</td>
<td>Kilopascals</td>
</tr>
<tr>
<td>Lpm</td>
<td>Liters per Minute</td>
</tr>
<tr>
<td>Mech.</td>
<td>Mechanical</td>
</tr>
<tr>
<td>min.</td>
<td>Minute</td>
</tr>
<tr>
<td>PIMA</td>
<td>Paper Industry Management Association</td>
</tr>
<tr>
<td>RMP</td>
<td>Refiner Mechanical Pulp</td>
</tr>
<tr>
<td>s</td>
<td>Scattering Coefficient</td>
</tr>
<tr>
<td>sq.</td>
<td>Square</td>
</tr>
<tr>
<td>TAPPI</td>
<td>Technical Association of Pulp and Paper Industry</td>
</tr>
<tr>
<td>TEA</td>
<td>Tensile Energy Absorption</td>
</tr>
<tr>
<td>TMP</td>
<td>Themomechanical Pulp</td>
</tr>
<tr>
<td>WRV</td>
<td>Water Retention Value</td>
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CHAPTER I

INTRODUCTION

Pulping of wood can be accomplished by either mechanical energy or chemical reaction or a combination of both. Depending upon the end use requirement, a particular pulping process is chosen. Unrefined pulp is given proper mechanical and chemical treatments to make it suitable for paper making.

High-yield pulping processes such as TMP (thermomechanical pulping) and CTMP (chemithermomechanical pulping) have become established pulping processes. Pulps above 90% yield are produced by employing these pulping processes. These pulps are characterized by high bulk and opacity, and good printability. Low brightness and brightness reversion have been the main constraints in the utilization of these pulps. Since the use requirements of paper indicate that brightness and opacity are the two most important optical properties of paper sheet and opacity advantage is well established with these pulps, it is imperative to find out the effect of further treatments on the brightness of these pulps. Bleaching of TMP and CTMP pulps provides significant improvement in brightness but this happens at the loss of some yield and additional load.
on the environment.

A variety of grades of end products are made by refining these high-yield pulps to low levels of freeness. Effects of refining on the optical properties of chemical pulps have been studied extensively. It has been found that refining lowers the brightness and opacity of chemical pulps because it reduces the scattering coefficient of the pulp. CTMP and TMP pulps behave differently than chemical pulps when subjected to mechanical treatment.

An increased understanding of the effect of refining on the brightness of CTMP and TMP pulps is the main objective of this study. The results obtained from refining of these pulps will be considered in terms of scattering and absorption coefficients. The reasons for changes in $s$ and $k$ values will be explained. The effects of refining on the opacity, tensile index and TEA, will also be presented. Differences in the behavior of pine and spruce TMP pulps will be discussed.
CHAPTER II

LITERATURE REVIEW

The literature review covers the main points of TMP and CTMP processes, softwoods used for TMP and CTMP, definition of brightness, factors affecting unbleached brightness of pulp, generation of fines due to refining and their effect on brightness.

High-Yield Pulping Processes

Because of increasing raw material costs and stringent pollution control laws, high-yield pulps are becoming more and more important for the success and survival of the pulp and paper industry. The core idea in these pulping processes has been to preserve lignin to the highest extent possible instead of removing it from the wood matrix as is done in chemical pulping processes. High-yield pulps are not only cheaper than chemical pulps, they also provide characteristic properties to the end product, e.g. opacity, stiffness, bulk and printability.

Thermomechanical pulping (TMP) and chemithermo-mechanical pulping (CTMP) processes are widely used high-yield processes for producing pulps which are used in a variety of end products such as fluff pulp, paperboard,
newsprint, tissue, magazine, coated, and writing and printing grades of paper. In a recent roundtable of PIMA magazine on pulping and bleaching trends it has been predicted that TMP and CTMP will show strong growth in coming years. (1)

Thermomechanical Pulping

Thermomechanical pulping involves steaming of the wood chips prior to refining. Wood chips become soft due to steaming and therefore refining does less damage to the fibers. Thus, the resultant pulp has a high percentage of long fibers and low shives content. The long fibers increase the strength of the pulp.

Thermomechanical pulping of softwoods yields a strong pulp but this increase in strength is obtained only at low freeness. The need to decrease the freeness of TMP to obtain good strength arises from the fact that TMP process yields fibers which are long, stiff, and unfibrillated, and, therefore, do not bond properly to each other. (2)

Chemithermomechanical Pulping

The chemithermomechanical pulping (CTMP) process is an improved version of thermomechanical pulping (TMP) process in which chips are chemically treated, heated for a short period of time and then refined in a disk refiner.
The impregnation of the chips is usually done with sodium sulfite in a pH range of 9 to 10. The pH of sodium sulfite is maintained with sodium hydroxide. This impregnation makes the chips soft. The heating of the chips takes place in a steaming vessel at 130 to 170°C. Refining of the softened chips is performed in a steam pressurized or atmospheric disk refiner.

Because of the sodium sulfite impregnation, CTMP pulps are highly flexible and therefore can be refined with less fiber cutting. This impregnation also improves the brightness of the pulp. As a result CTMP pulps are brighter than their corresponding TMP pulps. CTMP pulps produced with 12% sodium sulfite are about 14 points brighter than those produced with 4% sodium sulfite. (3)

CTMP yields are generally 2 to 3% lower than TMP. Temperature has an adverse effect on yield. At 170°C the yield is about 5% less than the yield obtained at 135°C. It is noteworthy that an increase in the amount of sodium sulfite in the range of 2 to 14% actually increases the yield of the pulp. This increase in yield is due to the action of chemical treatment to neutralize the acidity which prevents the dissolution of lignin and hemicellulose. Increasing the amount of sodium sulfite increases the flexibility of the fibers. Therefore, the breaking length of pulp increases. (3)
Softwoods are commonly used for high-yield pulping. Pine (loblolly and lodgepole), spruce (white, black, and jack), Douglas fir and fir (balsam and others) are the most widely used softwoods for TMP and CTMP processes.

Softwoods

Since this research work utilizes softwoods, it would be proper to mention a few important points about softwoods. Softwoods are gymnosperms which are also called conifers or evergreens. Wood density varies between 368 to 657 kg/cu.m. Southern softwoods have higher wood density than northern softwoods. For example, white spruce (northeast region) has a wood density of 417 kg/cu.m. and southern pines have a wood density of 577 to 689 kg/cu.m.

Southern pine fibers are long, coarse, and thick walled. Spruce fibers are long (but shorter than pine fibers), and medium-slender. (4)

Tracheids, ray cells, and epithelial cells are the principal cell types found in the softwoods. Tracheids are the main papermaking cells and their average length is 3 to 5 millimeters. Sometimes these are referred to as fibers. Ray cells may be ray parenchyma or ray tracheids. High-yield pulps contain ray cells. Epithelial cells are present in resin canals and contain pitch. During pulping, these cells are usually disintegrated. (5)
Brightness

Brightness is a misnomer because pulp and paper do not emit light. Basically, brightness is the reflectivity of the paper measured with a light source having the effective wavelength of 457 nanometers (nm) under specified geometrical conditions when the paper sheet is backed by an opaque pad of its own kind of paper sheets.

The ability of pulp to scatter the light is the main reason for reflection, R. Pulp also absorbs a part of the incident light, A, and the remainder of the incident light, T, passes through due to transparency. (6)

This gives the mathematical expression:

\[ R + A + T = 1 \]

According to Macdonald (6), Kubelka and Munk first gave the following equation by introducing the scattering coefficient s and absorption coefficient k for monochromatic light:

\[ k/s = (1-R_{\text{inf}})^2/2R_{\text{inf}} \]

Here \( R_{\text{inf}} \) denotes the brightness. Thus, brightness can be calculated when \( k/s \) ratio is known.

Importance of Brightness

In modern times when every single point of gain in brightness is important for improving the quality of most
paper products, it is essential to understand the role of refining on the brightness of TMP and CTMP pulps. According to Russel-Moreno, corporate buyer for Gannett Supply Corporation (publishing company of USA Today), eye appeal for newspapers and magazines is very important. This is why Gannett's minimum requirement for brightness of newsprint is 59. (7)

Factors Affecting the Brightness of the Pulp

Wood quality, pulping, refining, and chemical treatments are all important factors which contribute to unbleached pulp brightness.

Cellulose and hemicellulose do not contribute to color because they are inherently white. They are also not easily transformed into colored compounds unless they are treated severely with alkali. Severe treatment with alkali makes them yellow. It is suspected that during wood storage and pulping some chromophoric complexes are formed with the lignin molecule which cause light absorption in the visible spectrum.

Presence of resin and extractives is detrimental to unbleached pulp brightness. (8)

Old trees provide low unbleached pulp brightness as compared to young trees because old trees contain higher amounts of heartwood. (9)
Storage method has an influence on the unbleached pulp brightness. Fresh cut wood always provides higher unbleached pulp brightness than stored wood. (10) The more the exposed surface area of the wood during storage the more is the brightness loss.

Presence of decayed wood causes reduction in unbleached pulp brightness. (11) Wood or chips can be given chemical treatment prior to storage or can be stored in water to reduce the brightness loss. (12)

Presence of bark has a negative effect on unbleached pulp brightness. (13) Fresh bark causes less brightness loss than stored bark. (14)

Some metal ions have a detrimental effect on the brightness of pulps. Wood, mill water, and bleaching chemicals are sources of the metal ions such as iron and manganese. (15)

Thermomechanical pulp is difficult to brighten because most of the iron which comes with the wood chips is altered into an extremely insoluble dark colored form of iron oxide which is difficult to remove. (15)

Chelating agents such as DTPA (diethyltriaminepentaacetic acid) and EDTA (ethylenediaminetetraacetic acid) are added at different stages of high-yield pulping and bleaching to sequester the metal ions and thereby prevent the brightness loss.
Chemithermomechanical pulps are brighter than thermomechanical pulps because of the pretreatment of wood chips with sodium sulfite in chemithermomechanical pulping. Chemical pretreatment also increases the long fiber content of pulp due to more selective fiber separation. (16) Chemithermomechanical pulp fibers exhibit better fiber flexibility than thermomechanical pulp fibers. (17) Improved flexibility and cohesiveness of fibers lead to improved paper strength. (18)

According to Palenius (19), equal increments in brightness are possible by using hydrosulfite bleaching at different initial brightness levels. With peroxide bleaching, higher brightness increment is achievable at high initial brightness level and the brightness increment is low if the initial brightness is low.

High temperature and a prolonged pretreatment with sodium sulfite result in pulps of low brightness. Falk and Dillen (20) observed a substantial loss in brightness when the preheating temperature was increased above \(130^\circ C\) in presence of sulfite. The pulp produced in this way also gave low bleaching response. In the presence of sodium sulfite, pH close to neutral yielded maximum unbleached brightness. Sulfonation at a temperature higher than \(125^\circ C\) gave rise to increased formation of chromophores; therefore, the unbleached pulp brightness was low. Another
reason was that high temperature pulping resembled chemical pulping. Therefore, light scattering power of the pulp was reduced with mechanical treatment and the result was less bright unbleached pulp.

High temperature and long retention times affect adversely the chromophores present in wood. This results in further brightness loss. Low presteaming temperature is beneficial from the brightness point of view, while higher presteaming temperature increases the energy efficiency.

Optical Properties as a Function of Fiber Classification

Parsons (22) studied the optical characteristics of pulp as a function of fiber classification. He found that the fines fraction of spruce groundwood had the highest absorption coefficient (k-value) when compared to any other fraction of the same pulp. He assumed that during fractionation dirt was picked up by fines. This increased the k-value of the fines. It is important to note here that he used softened water for the fractionations. He also found that scattering coefficient of all fractions of groundwood, including the fines, was a linear function of the surface area.

During a study, Shriver (23) visited Bowater Carolina Inc., Catawba, SC. Technical personnel of this company...
reported to him that by decreasing the freeness of southern pine TMP by refining, brightness gain was realized. Shriver hypothesized that refining increased the scattering coefficient \((s)\) of the pulp while absorption coefficient \((k)\) remained constant. Thus, the decrease in the ratio \(k/s\) could be responsible for this brightness gain. It is important to mention here that no laboratory work was performed in this study to find out the \(k\)-value and \(s\)-value of the pulp before and after refining.

Role of Fines

Fiber fines form that fraction of a pulp which passes through the fourdrinier wire and consists of a complex mixture of fragments of primary and secondary fiber walls, fibrils, fibril bundles, colloidal matter, short fiber pieces, ray cells, parenchyma cells, and vessel segments and fragments (in case of hardwoods). These are also referred to as crill, debris, or slime. (24)

According to Giertz (25), fines play an important role in determining the optical as well as strength properties of both mechanical and chemical pulps. As a general rule, fines constitute that fraction of a pulp which is smaller than about 0.2 mm in size. In other words, the pulp fraction passing through a 200 mesh screen is termed as fines content of that pulp.
Softwood thermomechanical pulps have many long and stiff fibers that do not bond extensively to each other. Thus, increase in bonding is attained by refining these pulps to low drainage levels. As a result of refining these stiff fibers, a large number of fines are generated. These fines increase the scattering coefficient of the pulp. Chemical pretreatment increases bonding potential of fibers and, therefore, the higher the amount of chemicals added, the less is the increase in the scattering coefficient due to refining. At some particular pretreatment level it is possible that refining does not affect the scattering coefficient. Figure 1 (26) illustrates a similar situation where the bottom curve indicates no change in s-value with an increase in the breaking length. This happened when 13% sodium sulfite was used at 170°C.

The Characteristics of Fines

Doshi and Hawes (27), in a comparison of TMP, kraft, and recycled paper fines found that kraft and recycled paper fines were quite effective, while TMP fines were ineffective in increasing breaking length, burst index, and handsheet density. TMP fines were very effective in scattering light. This difference was attributed to the nature of fines. Kraft and recycled paper fines were identified as "slime stuff" because these fines were
cellulose-rich and had good bonding potential. TMP fines were described as "flour stuff" as these fines were lignin-rich and had poor bonding potential. Doshi and Hawes concluded from their experiments that compressibility of fines was a good indicator of their effectiveness for bonding. Compressibility of fines was indirectly measured from the variation of filtration resistance with pressure using following equation:

\[ R = a \Delta P^b \]

Scattering Coefficient (cm²/g)

- **0** CTMP 13% Na₂SO₃  T = 170°C
- **Δ** CTMP 6% Na₂SO₃  T = 135°C
- ***** TMP  T = 135°C

**Figure 1.** Breaking Length vs. s-value of CTMP and TMP. (26)
Here, \( R \) = Specific filtration resistance of fines (cm/g),
\( a \) = Specific filtration resistance of uncompressed fines pad (cm/g),
\( \Delta P \) = Pressure drop across the pad (g/cm.s^2), and
\( b \) = Compressibility constant (this is dimensionless).

For incompressible fines pad \((b = 0)\), pressure change will have no effect on the specific filtration resistance. A high value of \( b \) indicated high compressibility of fines. Higher compressibility meant better bonding potential. In their experiments, they found that the compressibility constant of TMP fines was only 0.274, whereas the values for kraft and secondary paper fines were 0.739 and 0.649, respectively.

Iwamida and Sumi (28) studied the properties of fines in high-yield pulps. They determined water retention values (WRV) of fines for predicting their bonding ability. They found that high-yield sulfite pulp fines had much higher WRV than stone ground wood fines. They also found that decreasing the yield of the pulp increased WRV of the fines. Increase in the WRV decreased the \( s \)-value of the pulps. Figures 2 and 3 illustrate the relationships of % yield versus \( s \)-value and WRV versus \( s \)-value, respectively. It is important to note that \( s \)-value of fines decreased dramatically with decrease of cooked yield as shown in Figure 2. Decrease in \( s \)-value of fiber fraction was smaller than that of the corresponding fines. \( s \)-value of
Legend.  △ = Whole pulp, X = Fiber fraction, O = Fines

Figure 2. Pulp Yield vs. s-value of Fines and Fiber. (28)

Figure 3. WRV vs. s-value of Fines of Refined Pulps. (28)
fines was remarkably higher than that of the fiber fraction at the yield of more than 90% and it became almost the same as that of the fiber fraction at the yield of about 85%. At the yield of approximately 75%, s-value of fines approached to zero.

Effect of Refining on Optical Properties

Most of the literature relating to the effect of refining on pulp and paper properties deals with chemical pulps. Primary consideration has been given to the development of strength properties.

It is well documented that refining enhances bonding due to the Campbell effect. According to W. B. Campbell (29), refining exposes the surfaces of fibers which already exist in cellulose saturated with water. This process is known as fibrillation. Campbell proposed that all cellulose surfaces in water are covered with layers of cellulose in partial solution. These surfaces when brought into contact, are believed to unite by "crystallization" of cellulose on the evaporation of the water. This action improves the strength properties of the paper and is termed the Campbell effect.

Swanson and Steber (30) observed that an increase in the tensile strength due to refining was accompanied by a decrease in the scattering coefficient (s). If the absorption coefficient (k) remained unchanged during the
process of refining, the ratio \( k/s \) increased. This increase in \( k/s \) ratio meant a decrease in the brightness according to the following equation:

\[
R_{(\text{INF})} = 1 + \frac{k}{s} - \left[ \frac{k^2}{s^2} + 2\frac{k}{s} \right]^{1/2}
\]

Here,

\( R_{(\text{INF})} \) = Reflectivity (or brightness) of paper backed by an opaque pad of like sheets,
\( k \) = Absorption coefficient (m\(^2\)/Kg) and
\( s \) = Scattering coefficient (m\(^2\)/Kg).

The above mentioned formula is a different form of Kubelka-Munk equation given on page number 7. (31)

Effect of Refining on the Scattering Coefficient

Since the development of high-yield pulping, most of the efforts have been directed to the study of the development of strength properties as a function of refining. (32)

Ryberg, Falk, and Lowgren (33) performed a study on newsprint production from bagasse and hardwood. They hypothesized that refining of high-yield pulp would increase its scattering coefficient. Figure 4 illustrates their view about the effect of refining on scattering coefficient (s) and tensile index of pulps of different yields. Arrows on the curve indicate the development in s-value by refining starting from the freenesses mentioned on the arrows.
Figure 4. Tensile Index vs. S-value of Various Pulps. (33)

Corson (34) found that decrease in the freeness of New Zealand grown radiata pine TMP and RMP by refining resulted in an increase in s-value of the pulps. He also showed that s-values of these pulps were positively related to tensile index and density. Figures 5, 6, and 7 show these relationships.
Figure 5. Freeness vs. s-value of TMP. (34)
Figure 6. Tensile Index vs. s-value of TMP. (34)

Figure 7. Sheet Density vs. s-value of TMP. (34)
Rapson (35) noted the effect of refining on the optical properties of several bleached pulps. The wood species of these pulps were not mentioned. In general, a decrease in s-value and increase in k-value was observed. Thus, results indicated a decrease in brightness due to refining. He emphasized that decrease in scattering coefficient due to increased bonding was mainly responsible for the decrease in brightness. Any explanation for the increase in k-value with refining was not given. Figure 8 illustrates the relationship of breaking length and scattering coefficient for different kinds of pulps. He explained the different behavior of cotton fiber by postulating that initial refining did not fibrillate the cotton fibers. Instead it increased the exposed surface area. This meant that cotton fibers took more energy than other fibers for fibrillation needed for proper bonding.

![Scattering Coefficient (Sq.cm./g)](image)

**Figure 8.** Breaking Length vs. s-value of Various Pulps. (35)

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Effect of Impregnation on the Absorption Coefficient

Norrstrom (36) studied the effect of impregnation of chips with bisulfite on absorption coefficient (k) of pulps. He noticed a 35% reduction in k-value due to impregnation of chips with bisulfite before refining in the production of CTMP. He also found that heating the chips to 150° C reduced the k-value by 50%; however, it reduced the pulp yield by 1.5%. He estimated that bisulfite treatment could increase the brightness by almost 10 points due to reduction in the k-value.

Hartler (37) stated that by increasing chip moisture content in the range of 10 to 65% during primary refining, increases in brightness and strength properties were realized.

Increasing refiner pressures in the range of 0 to 50 psig reduced the s-value and, hence, caused losses in the unbleached brightness. (38) (39)

Brightness Reversion

Since brightness-reversion is an important topic with CTMP and TMP, it would be proper to discuss some important points about this subject. Brightness of mechanical pulps decreases when these pulps are exposed to heat and/or light. This process is termed brightness-reversion.

Gellerstedt, Pettersson, and Sundin (41) studied the factors influencing the brightness stability of high-yield
pulps. They found that yellowing of mechanical pulps (heat or light initiated) was due to the formation of quinone structures as a result of reactions between oxygen and phenoxy radicals created in the lignin. Factors affecting these reactions were: UV light, heat, pH, moisture content, and the presence of transition metal ions.

Bleached mechanical pulps are more prone to brightness reversion than unbleached pulps because of the formation of new, easily oxidizable lignin structures during the bleaching process (hydrogen peroxide or hydrosulfite bleaching). The study (41) established that heat-induced yellowing of mechanical pulps could be reduced to a large extent by the use of sodium bisulfite/sulfite along with DTPA (diethylenetriaminopentaacetic acid). This addition had no effect on light-induced yellowing.

Vasudevan, Panchapkesan, Gratzl, and Holmbom (42) studied the effect of ozone on strength development and brightness-reversion characteristics of high-yield pulps. They suggested that the use of UV light reflectance spectroscopy was a viable technique for measuring the changes in the chromophoric structures. They mentioned that scanning over a wavelength of 240-700 nm revealed much more information regarding the destruction and regeneration of chromophoric structures (as compared to the conventional practice of measuring brightness at a single wavelength of 457 nm) as a result of various bleaching processes.

In a recent study (43), Johnson stated that light-
reverted papers with a thin, darkened layer at the exposed surface do not satisfy the Kubelka-Munk assumption of sample homogeneity because UV light does not penetrate past the top few fiber layers. He suggested that the brightness stability of CTMP-containing papers could be improved in a cost effective manner if thin coatings containing titanium dioxide and clay were applied on the paper.

**Positive Factors of TMP and CTMP**

The problems of brightness reversion and low strength have negatively affected the ideal use of thermomechanical and chemithermomechanical pulps. However, the elimination of dioxins, widening raw material base, end use in fast growing coated and uncoated printing papers of these pulps, and reduction in the need of long-lasting information papers are the important positive factors which outweigh the above mentioned deficiencies of these pulps in many end uses. (40)

In general softwood CTMP is considered comparable to hardwood kraft pulp in strength properties.

**Analysis of Literature**

The preceding literature review clearly indicates that relatively little research has been conducted with the optical properties of high-yield pulps. The studies have mainly concentrated on the relationship of the scattering coefficient and strength properties. The explanation of the
observation of brightness increase with refining by Shriver (23) was not available in the literature. Effect of refining on the absorption coefficient of TMP and CTMP pulps has also not been established.
CHAPTER III

PROBLEM STATEMENT

The primary objective of this investigation is to determine and explain the effect of refining on the brightness of TMP and CTMP. The changes in the scattering and absorption coefficients will be the main criteria for explaining the effect on the brightness. The changes in the scattering and absorption coefficients will be attributed to fines content and nature.

Changes in the brightness, arising from the nature and morphology of wood fibers, will be considered. Other pulp properties such as opacity, tensile index, and TEA also will be studied to get an overall understanding of the refining process.

Refining is as old as the history of paper making and has undergone significant changes during recent decades. Each step of technological development in pulping processes requires simultaneous understanding of the effect of refining on the new type of pulp. This may lead to the development of new refining methods. CTMP and TMP processes are the recent trend in the pulp and paper industry because of several advantages associated with these processes. Therefore, it is important to understand the effect of refining on the brightness of TMP and CTMP pulps.
CHAPTER IV

EXPERIMENTAL DESIGN

Approach

The experimental work in this investigation was performed in two phases.

In the first phase, chemithermomechanical pulp (CTMP) was produced by using the Sunds Defibrator located in the pilot plant of the Department of Paper and Printing Science and Engineering at Western Michigan University, Kalamazoo. Hand-sorted spruce chips were used for pulping.

Effect of refining on this pulp was studied by making handsheets of pulps at four different freeness levels and determining their optical and physical properties.

Because of low unbleached pulp brightness, a part of the unrefined pulp was bleached in the laboratory and the effect of refining on the brightness of this bleached pulp was also determined.

Second phase experimentation involved the procurement of five high-freeness high-yield commercial pulps from three different sources. These pulps were: unbleached Northern spruce CTMP from Tembec Inc., Canada; unbleached Loblolly pine TMP from Bowater Inc., SC; bleached CTMP, unbleached CTMP, and unbleached TMP from Quensel River Pulp
Company, Canada. The pulps of Quensel River Pulp Company were produced from 55% White spruce, 35% lodgepole pine, and 10% balsam fir.

The effect of refining on the optical and physical properties of these pulps also was studied by making handsheets of pulps at four different freeness levels.

Optical properties determined in the first and second phases included brightness and opacity. The Scattering (s) and absorption (k) coefficients were calculated. Physical properties included tensile strength and tensile energy absorption (TEA). Tensile index was calculated from tensile strength.

The Kajaani Fiber Analyzer FS 100 was used for determining the fines content of the different pulps at different freeness levels. Kappa numbers of a few pulps were also determined to relate the changes in the absorption coefficients of these pulps as a result of refining.

Significance of change in brightness was determined by applying one way analysis of variance. explanations for the effect of refining on brightness were presented in terms of changes in s- and k-values. Changes in fines contents and kappa numbers were considered to explain the changes in s- and k-values, respectively. Determination of strength properties indicated the development of bonding properties (affecting the scattering coefficient) of fibers and fines with refining.
Phase I Experimentation

Production of CTMP with Sunds Defibrator

Spruce chips from northern Michigan were used for making the pulp. These chips were procured from American Fibrit Inc., Battle Creek, MI. The chips were stored in closed barrels to prevent decoloration and moisture variation. Moisture content of the chips was determined and the barrels of chips were weighed to determine the oven dry weight of chips used for pulping. Before feeding the chips into Sunds Defibrator, the chips were hand-sorted to remove oversized and decayed chips.

Sodium sulfite solution was prepared in a chemical tank near the Sunds Defibrator by dissolving anhydrous sodium sulfite in water. The strength of sodium sulfite solution was determined according to the calculations given in Appendix A. The strength of sodium sulfite solution was 3.7%. DTPA was added to this solution before pumping to the defibrator. The consumptions of sodium sulfite and DTPA were 2.87% and 0.4% (based on dry wood), respectively. The description of Sunds Defibrator is given in Appendix B.

Description of Flow in Sunds Defibrator

Hand-sorted spruce chips were added to the infeed hopper where presteaming was done. The presteamed chips traveled from the hopper to the preheater via a plug screw where compression took place. Sodium sulfite liquor was
added to the chips in the impregnator before releasing the chips into the pressurized preheater for best impregnation of the chips with the liquor. Impregnated chips traveled through the discharge screw feeder to the refiner where defibration took place. Pulp flowed through the blow line to the cyclone where atmospheric discharge of the pulp took place.

The pulp sample was collected from the cyclone discharge after the pulp flow was stabilized and all the desired operating parameters were met. The operating parameters of the Sunds Defibrator are shown in Table 1. Calculations for the determination of the pulp yield are shown in Appendix C.

Table 1

Operating Parameters of Sunds Defibrator

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infeed hopper</td>
<td>: presteam for 10 min.</td>
</tr>
<tr>
<td>Hopper plug screw feed</td>
<td>: crush chips at 172 kPa</td>
</tr>
<tr>
<td>Impregnator</td>
<td>: with water at 1860 kPa</td>
</tr>
<tr>
<td></td>
<td>(10 % to the pulp flow)</td>
</tr>
<tr>
<td>Preheater</td>
<td>: 10 min. at 241 kPa</td>
</tr>
<tr>
<td>Refiner load</td>
<td>: 57 HPD / ODT</td>
</tr>
<tr>
<td>Plate clearance</td>
<td>: disc .. 0.5 mm</td>
</tr>
<tr>
<td></td>
<td>: cone .. 0.4 mm</td>
</tr>
<tr>
<td>Through-put</td>
<td>: 2.455 kg / min.</td>
</tr>
<tr>
<td>Defibrator dilution water flow</td>
<td></td>
</tr>
<tr>
<td>Primary infeed dilution flow</td>
<td>: 0.95 Lpm</td>
</tr>
<tr>
<td>CD dilution</td>
<td>: 0.91 Lpm</td>
</tr>
<tr>
<td>Chips moisture</td>
<td>: 37 %</td>
</tr>
<tr>
<td>Blow pulp consistency</td>
<td>: 16 %</td>
</tr>
<tr>
<td>Sodium sulfite (based on dry wood)</td>
<td>: 2.87 %</td>
</tr>
</tbody>
</table>
Pulp Handling and Handsheet Making

In high consistency refining, twisting action on the fibers introduces kinks and curves into the fibers. These are then frozen into the fiber as they are cooled. This condition is known as latency. It is important to remove the latency of the pulp before screening because the screen is unable to differentiate between fiber bundles, and the kinks and curves caused by the latency.

Latency of the pulp sample collected from the digester run was removed according to Tappi Method: Preparation of Mechanical Pulps (T 262 pm-81). The pulp was screened on a laboratory vibrating 6-cut flat screen (0.015 cm opening). A few drops of formaldehyde were mixed by shaking the pulp containing bags thoroughly to prevent pulp deterioration. Whenever necessary the pulp was stored in cold and dark room to avoid brightness reversion.

Pulp was refined in the Valley beater as per Tappi Method: Laboratory Processing of Pulp (T 200 os-70). Thoroughly mixed unrefined pulp was collected for handsheet making. At three different time intervals of refining, the refined pulps were collected for handsheet making. Freenesses of all the pulps were determined. A Noble and Wood handsheet machine was used for making 60 gram/sq.m handsheets. Distilled water was used in the experiments. During refining, 0.4% EDTA was added to the pulps. Handsheets were made after adjusting the pH of the pulp to
5 with HCl and/or NaOH.

When not in use (i.e., not being tested), the handsheets were kept in closed envelopes and stored in a cold and dark place to avoid brightness reversion.

**Handsheet Testing**

The following tests were performed on the handsheets: (a) brightness ($R_{INF}$), (b) reflectance with black backing ($R_0$), (c) Tappi opacity, (d) tensile strength to get tensile index, and (e) TEA (tensile energy absorption).

Opacity values of the handsheets were corrected to 60 gram/sq.m. basis weight according to the equations given in Appendix D.

The scattering (s) and absorption (k) coefficients were calculated according to the formulae listed in Appendix E. The handsheet average properties are presented in Appendix F.

All the work done was duplicated at separate times. The results of the duplications are also presented in Appendix F. Concise results of this and all other pulps are presented in the following chapter along with the discussion.

**Circulation of the Pulp in Beater Without Load**

This work was done to determine whether the changes in the pulp properties were only due to refining or circulation itself affected pulp properties significantly.
To see the effect of the circulation of the pulp in the Valley beater on pulp properties, the unrefined pulps were circulated in the beater (for a time equal to that of the original beater run) without any load. Handsheets were made from the pulps collected before and after circulation. The properties of these handsheets are shown in Appendix F.

The standard test methods used in this investigation are listed in Table 2.

Bleaching of Pilot Plant CTMP in Laboratory

Because of the low brightness of the unbleached CTMP, it was decided to bleach the unrefined CTMP. The bleaching was carried out by using V-Brite® (sodium dithionate) received from Hoechst Celanese Company, Virginia. Single stage low consistency bleaching was performed in the laboratory. The amount of bleach used was 16 kgs per ton. Bleaching temperature, consistency, and pH were 63°C, 3.9%, and 5.6, respectively. EDTA was used at a rate of 1% of the pulp. The reaction time for bleaching was one hour. Gentle mixing was done during the bleaching reaction. The pulp was washed with distilled water after bleaching. About 8 points of brightness gain was realized with this procedure.

Bleached CTMP was also refined in the Valley beater. Handsheets were made and the properties (listed already) were determined. The average properties and the standard deviation values are listed in Appendix F. Results of
duplication and circulation without load are also presented in this Appendix.

Table 2
Standard Test Methods

<table>
<thead>
<tr>
<th>Test</th>
<th>TAPPI Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laboratory processing of pulp</td>
<td>T 200 os-70</td>
</tr>
<tr>
<td>Freeness of pulp</td>
<td>T 227 om-58</td>
</tr>
<tr>
<td>Kappa number of pulp</td>
<td>T 236 cm-85</td>
</tr>
<tr>
<td>Consistency of pulp</td>
<td>T 240 om-81</td>
</tr>
<tr>
<td>Moisture content of wood chips</td>
<td>T 258 os-76</td>
</tr>
<tr>
<td>Preparation of mechanical pulps</td>
<td>T 262 pm-81</td>
</tr>
<tr>
<td>Standard conditioning of handsheets</td>
<td>T 402 om-83</td>
</tr>
<tr>
<td>Opacity of handsheets</td>
<td>T 425 om-86</td>
</tr>
<tr>
<td>Brightness of handsheets</td>
<td>T 452 om-83</td>
</tr>
<tr>
<td>Tensile index and TEA of handsheets</td>
<td>T 494 om-81</td>
</tr>
<tr>
<td>Preparation and standardization of</td>
<td>T 610 n-60</td>
</tr>
<tr>
<td>volumetric solutions</td>
<td></td>
</tr>
</tbody>
</table>
Phase II Experimentation

Commercial TMP and CTMP from three different pulp sources were used in this phase. These were high freeness pulps. The details of all the five pulps used in this phase are presented in Table 3.

Table 3
Details of Commercial Pulps

<table>
<thead>
<tr>
<th>Pulp</th>
<th>Pulping Yield %</th>
<th>Process</th>
<th>Unbleached</th>
<th>Raw Material</th>
<th>Freeness (CSF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tembec</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Canada</td>
<td>CTMP</td>
<td>93</td>
<td>Unbleached</td>
<td>Spruce</td>
<td>680</td>
</tr>
<tr>
<td>Quensel</td>
<td></td>
<td></td>
<td></td>
<td>White spruce/</td>
<td></td>
</tr>
<tr>
<td>Canada</td>
<td>CTMP</td>
<td>93</td>
<td>Unbleached</td>
<td>Lodgepole pine/</td>
<td>740</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Lodgepole pine/</td>
<td>Balsam fir(55/35/10)</td>
</tr>
<tr>
<td>Quensel</td>
<td></td>
<td></td>
<td></td>
<td>Same as above</td>
<td>450</td>
</tr>
<tr>
<td>Bowater</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carolina</td>
<td>TMP</td>
<td>95</td>
<td>Unbleached</td>
<td>Same as above</td>
<td>680</td>
</tr>
<tr>
<td>Carolina</td>
<td>TMP</td>
<td>95</td>
<td>Unbleached</td>
<td>Loblolly pine</td>
<td>700</td>
</tr>
</tbody>
</table>
Experiments similar to that of Phase I were performed to study the effect of refining on the properties of the pulps listed in Table 3. All the data are presented in Appendix F. In the following chapter selected results are presented along with the discussion.

Fines contents and kappa numbers of pulps were determined to explain the changes in the scattering (s) and absorption (k) coefficients of the pulps (of this and the previous phase) due to refining.

Since it was considered that fines play the dominant role in determining the scattering coefficient of pulps, measurement of fines content of all the pulps was done by employing the Kajaani Fiber Analyzer FS 100. The Kajaani Fiber Analyzer gives the number counts of fibers in different ranges of fiber length. The fraction of the pulp which was smaller than 210 micrometers (0.21 mm), was regarded as the fines fraction. This cut off point was based on the definition of fines given in Chapter II. A brief description of Kajaani Fiber Analyzer FS 100 is given in Appendix H.

The main problem encountered with the Kajaani Fiber Analyzer was that the fines content of the unrefined pulp was found higher than the corresponding refined pulp in many cases. After discussing with R. Piirainen, Kajaani Automation Incorporated, Georgia, it was concluded that refining generated many small fines (smaller than 20 micrometers) which were beyond the measurement sensitivity.
of the instrument. The measurement sensitivity of the Kajaani Fiber Analyzer FS 100 is 20 micrometers. Thus, the fines content measured by this instrument consisted of fines 20 to 210 micrometers in size. The fines smaller than 20 micrometers (0.02 mm) could not be detected by this instrument.

Since an increase in the absorption coefficient was observed with refining and kappa number predicts the bleachability of pulps, some unrefined and correspondingly highly refined pulps were tested for kappa numbers. These tests were performed to see if there existed some relationship between k-value and kappa number. The pulps tested for kappa number were: (a) unbleached TMP from Bowater Carolina, SC.; (b) unbleached TMP from Quensel River Pulp Company, Canada; and (c) unbleached CTMP from Quensel River Pulp Company, Canada.

Tappi method: Kappa Number of Pulp (T 236 cm-85) was used to determine the kappa numbers. The results are given along with the discussion in the following chapter.
CHAPTER V

RESULTS AND DISCUSSION

The results obtained for each pulp are presented with discussion. Detailed results are given in Appendix F.

Graphs were plotted to show the relationships of refining and handsheet properties. The vertical lines on the graphs represent the standard error. One way analyses of variance (ANOVA) were applied to determine the significance of change in brightness of pulps at 95% confidence level due to refining. The $P$-values for refining time and brightness for all pulps are presented in Table 4. During the discussion of results in the following pages, the $P$-values for all pulps have been obtained from this Table. A $P$-value of equal to or less than 0.05 indicates that the change in the dependent variable due to the change in the independent variable is significant at 95% confidence level. The ANOVA Table for all pulps is given in Appendix G.
Table 4

P-values for Refining Time and Brightness of Pulps

<table>
<thead>
<tr>
<th>Name of the Pulp</th>
<th>Independent Variable</th>
<th>Dependent Variable</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pilot Plant Unbleached CTMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.0097</td>
</tr>
<tr>
<td>Laboratory Bleached Pilot Plant CTMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.1064</td>
</tr>
<tr>
<td>Quensel Canada Bleached CTMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.0502</td>
</tr>
<tr>
<td>Bowater Carolina Unbleached TMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.0017</td>
</tr>
<tr>
<td>Tembec Canada Unbleached CTMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.4258</td>
</tr>
<tr>
<td>Quensel Canada Unbleached TMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.0194</td>
</tr>
<tr>
<td>Quensel Canada Unbleached CTMP</td>
<td>Refining Time</td>
<td>Brightness</td>
<td>0.0073</td>
</tr>
</tbody>
</table>

Phase I

Effect of Refining on Pilot Plant Unbleached CTMP

Handsheet results and fines contents obtained for this pulp are presented in Table 5. Figure 9 shows the effect of refining on the brightness. P-value (from Table 4) for refining time and brightness was 0.0097. Thus, it could be concluded that the change in the brightness due to refining was significant. This change was significant for the last refining interval. Figure 9 shows that refining decreased the brightness significantly for the last refining interval.
interval. Decrease in brightness can be explained in terms of s- and k-values. Figures 10 and 11 show the effect of refining on s- and k-values, respectively. As refining progressed, increases in s- and k-values were observed, but in the last stage, at the lowest freeness (54 C.S.F.) decrease in scattering coefficient was significant which was probably due to increased bonding of fibers. This is evident from the increase in tensile and TEA values.

Table 5
Properties of Pilot Plant Unbleached Pulp

<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>20</td>
<td>30</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Mean Std.</td>
<td>Mean Std.</td>
<td>Mean Std.</td>
<td>Mean Std.</td>
<td>Mean Std.</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>530 0.00</td>
<td>290 2.50</td>
<td>186 9.00</td>
<td>54 4.50</td>
<td></td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>30.4 0.10</td>
<td>30.7 0.25</td>
<td>30.3 0.50</td>
<td>27.6 0.40</td>
<td></td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>21.85 1.59</td>
<td>26.42 4.40</td>
<td>21.49 2.63</td>
<td>19.23 1.21</td>
<td></td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>17.42 1.38</td>
<td>20.78 3.77</td>
<td>17.30 2.64</td>
<td>18.24 0.69</td>
<td></td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>96.0 0.35</td>
<td>95.8 0.10</td>
<td>95.9 0.20</td>
<td>96.4 0.00</td>
<td></td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>18.04 2.58</td>
<td>33.28 4.40</td>
<td>36.39 0.92</td>
<td>42.14 4.22</td>
<td></td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td>0.80 0.17</td>
<td>1.43 0.31</td>
<td>1.54 0.07</td>
<td>1.80 0.34</td>
<td></td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>61.48 0.95</td>
<td>55.81 0.55</td>
<td>67.26 0.78</td>
<td>72.53 0.16</td>
<td></td>
</tr>
</tbody>
</table>
Figure 9. Effect of Refining on Brightness of Pilot Plant Unbleached CTMP.

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Scattering Coefficient (Sq.m/Kg)

Figure 10. Effect of Refining on Scattering Coefficient of Pilot Plant Unbleached CTMP.
Figure 11. Effect of Refining on Absorption Coefficient of Pilot Plant Unbleached CTMP.
This indicated that two possible competing mechanisms affected the s-value of the pulp. These mechanisms were increase in surface area and increase in bonding. The resultant change in s-value affected brightness. Some increase in absorption coefficient was also observed. The graphs showing the relationships of refining with tensile index and TEA of all the pulps used in this study are presented in Appendix I.

Figure 12 indicates that opacity remained essentially unaffected by refining. Possibly the increase in k-value compensated for decrease in s-value, thus, opacity remained unchanged.

Fines content of pulp increased with refining but as explained in earlier chapter, unrefined pulp showed high fines content because of the lack of required measurement sensitivity of the Kajaani Fiber Analyzer. Possibly the increased bonding ability of fines and fibers due to chemical pretreatment decreased the s-value of the pulp at low freeness levels.

Results obtained by circulating this pulp (and all other pulps used in this study) in the Valley beater without applying any load are given in Appendix F. No significant difference was observed in the handsheet properties made from the pulps collected before and after circulation. Thus, it may be assumed that exposure of the pulps during refining did not cause any darkening (and any significant change in k- and s-values) of the pulp.
Figure 12. Effect of Refining on Opacity of Pilot Plant Unbleached CTMP.
Therefore, the changes in the pulp properties in the actual runs (with load) were entirely due to refining.

The low brightness of the pulp under discussion can be attributed to long storage time of wood chips. Because of low unbleached brightness of this pulp, it was bleached in the laboratory by single stage sodium dithionate bleaching. The bleached pulp was refined, handsheets were made, and the properties were studied.

**Effect of Refining on Laboratory Bleached Pilot Plant CTMP**

Results obtained by refining this pulp are presented in Table 6. Figure 13 illustrates the effect of refining on the brightness of this BCTMP (bleached chemithermo-mechanical pulp).

P-value for refining time and brightness was 0.1064. This meant that the change in the brightness due to refining was not significant at the 95% confidence level. However, the pattern of change in the brightness due to refining showed a decreasing trend.

Figures 14, 15, and 16 show the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. Increase in the absorption coefficient was significant. This was the probable reason for the decreases in brightness and increases in opacity.

Strength properties and fines content of the pulp increased with refining.
Table 6

Properties of Laboratory Bleached Pilot Plant CTMP

<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>15</td>
<td>30</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>500</td>
<td>45.0</td>
<td>330</td>
<td>7.50</td>
<td>178</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>38.4</td>
<td>0.55</td>
<td>38.5</td>
<td>0.75</td>
<td>37.5</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>29.37</td>
<td>0.86</td>
<td>28.73</td>
<td>1.12</td>
<td>29.98</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>14.55</td>
<td>0.05</td>
<td>14.19</td>
<td>1.17</td>
<td>15.63</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>92.5</td>
<td>0.75</td>
<td>93.4</td>
<td>0.25</td>
<td>94.0</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>17.14</td>
<td>2.32</td>
<td>26.88</td>
<td>6.19</td>
<td>33.46</td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td>0.51</td>
<td>0.19</td>
<td>0.97</td>
<td>0.62</td>
<td>1.17</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>45.77</td>
<td>1.12</td>
<td>39.18</td>
<td>0.12</td>
<td>41.27</td>
</tr>
</tbody>
</table>

Figure 17 shows a comparison of unbleached and bleached pilot plant CTMP brightnesses affected by refining. It was seen that brightnesses of both pulps decreased with refining because of decrease in s-value and increase in k-value. Decrease in scattering coefficient was possibly due to increased bonding. A detailed explanation of the effects of refining on k-value is presented later in this chapter.
Figure 13. Effect of Refining on Brightness of Laboratory Bleached Pilot Plant CTMP.
Scattering Coefficient (Sq.m/Kg)

Figure 14. Effect of Refining on Scattering Coefficient of Laboratory Bleached Pilot Plant CTMP.
Figure 15. Effect of Refining on Absorption Coefficient of Laboratory Bleached Pilot Plant CTMP.
Figure 16. Effect of Refining on Opacity of Laboratory Bleached Pilot Plant CTMP.
Figure 17. Comparison of Relationships of Refining and Brightness of Unbleached and Bleached Pilot Plant CTMP.

Legend. 0 = Unbleached, * = Bleached
Phase II

Bleached chemithermomechanical pulp (BCTMP), unbleached CTMP, and unbleached thermomechanical pulp (TMP) from Quensel River Pulp Company, Canada; unbleached TMP from Bowater Carolina, SC; and unbleached CTMP from Tembec Incorporated, Canada were studied in this phase. The discussion of the results obtained by refining these pulps is presented in the following pages.

Effect of Refining on Quensel BCTMP

Results obtained with this pulp are presented in Table 7. Figure 18 shows the effect of refining on the brightness of this pulp.

$P$-value for refining and brightness was 0.0502. This indicated that the change in the brightness due to refining could be considered significant. Trend of change in brightness indicated a declining trend in brightness as refining progressed. Figures 19, 20, and 21 show the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. A decreasing trend was noticed in the scattering coefficient and opacity while the absorption coefficient increased with refining. Decrease in $s$-value and increase in $k$-value were responsible for the decrease in brightness. A considerable decrease in $s$-value was the possible reason for decrease in opacity.
Strength properties and fines content of the pulp increased with refining.

Table 7
Properties of Quensel Bleached CTMP

<table>
<thead>
<tr>
<th>Properties</th>
<th>1</th>
<th>15</th>
<th>35</th>
<th>45</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>460</td>
<td>47.5</td>
<td>335</td>
<td>15.0</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>74.5</td>
<td>0.05</td>
<td>73.1</td>
<td>0.95</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>37.10</td>
<td>0.70</td>
<td>34.72</td>
<td>0.25</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>1.63</td>
<td>0.03</td>
<td>1.73</td>
<td>0.16</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>78.2</td>
<td>0.60</td>
<td>77.3</td>
<td>1.45</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>35.81</td>
<td>5.81</td>
<td>47.24</td>
<td>0.94</td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td>1.89</td>
<td>0.21</td>
<td>2.71</td>
<td>0.16</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>35.77</td>
<td>0.42</td>
<td>31.91</td>
<td>0.95</td>
</tr>
</tbody>
</table>

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Figure 18. Effect of Refining on Brightness of Quensel Bleached CTMP.
Figure 19. Effect of Refining on Scattering Coefficient of Quensel Bleached CTMP.
Figure 20. Effect of Refining on Absorption Coefficient of Quensel Bleached CTMP.
Figure 21. Effect of Refining on Opacity of Quensel Bleached CTMP.

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Effect of Refining on Bowater Unbleached TMP

Results obtained by refining this pulp are presented in Table 8. Figure 22 shows the effect of refining on brightness of Bowater unbleached TMP.

P-value for refining and brightness was 0.0017. This meant that increase in brightness due to refining was highly significant. Shriver (23) found similar results during his mill visit.

Figures 23, 24, and 25 show the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. Although an increase in k-value was observed due to refining, the change in s-value had a dominating effect on the brightness. The s-value of the highly refined pulp (55 C.S.F.) was 61.5% higher than the s-value of unrefined pulp. Morphology of pine fibers had probably played the vital role in increasing the s-value. Stiff and thick-walled pine fibers were perhaps resistant to swelling and fibrillation, therefore, the fines generated during refining of this pulp mostly contributed to the increase in s-value.

Opacity of Bowater TMP increased continuously as refining progressed. Approximately 12 points of increase in opacity was observed with this pulp. Increases in scattering coefficient and absorption coefficient had positive influences on opacity.
<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>30</td>
<td>75</td>
<td>105</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>705</td>
<td>15.0</td>
<td>325</td>
<td>0.00</td>
<td>140</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>44.3</td>
<td>0.50</td>
<td>47.2</td>
<td>0.65</td>
<td>50.2</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>24.30</td>
<td>1.14</td>
<td>30.19</td>
<td>1.23</td>
<td>35.27</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>8.50</td>
<td>0.15</td>
<td>8.94</td>
<td>0.03</td>
<td>8.71</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>79.5</td>
<td>0.25</td>
<td>83.7</td>
<td>0.40</td>
<td>88.3</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>4.55</td>
<td>0.45</td>
<td>14.99</td>
<td>0.76</td>
<td>20.08</td>
</tr>
<tr>
<td>T.E.A. (J/Sq.m)</td>
<td>0.18</td>
<td>0.01</td>
<td>0.54</td>
<td>0.04</td>
<td>0.69</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>60.29</td>
<td>0.66</td>
<td>38.72</td>
<td>0.47</td>
<td>42.57</td>
</tr>
</tbody>
</table>

Table 8
Properties of Bowater Unbleached TMP
Figure 22. Effect of Refining on Brightness of Bowater Unbleached TMP.

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Figure 23. Effect of Refining on Scattering Coefficient of Bowater Unbleached TMP.

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Figure 24. Effect of Refining on Absorption Coefficient of Bowater Unbleached TMP.
Figure 25. Effect of Refining on Opacity of Bowater Unbleached TMP.
Effect of Refining on Tembec Unbleached CTMP

Results obtained with Tembec unbleached CTMP are listed in Table 9. Figure 26 illustrates the effect of refining on the brightness of this pulp.

The P-value for refining and brightness was 0.4258. This indicated that the change in brightness was not significant at the 95% confidence level. Simultaneous generation of fines (which contributed to the scattering coefficient) and fibrillation of fibers was the probable reason that a definite trend was not evident.

Figures 27, 28, and 29 illustrate the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. The scattering coefficient initially increased but later decreased. The decrease in s-value was probably because of improved bonding.

The increase in the absorption coefficient had probably the dominant effect on opacity. That was why opacity continuously increased with refining.

Tensile index, TEA, and fines content of the subject pulp increased with refining.
Table 9
Properties of Tembec Unbleached CTMP

<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td></td>
<td>685</td>
<td>5.00</td>
<td>260</td>
<td>53.0</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td></td>
<td>57.8</td>
<td>0.35</td>
<td>60.2</td>
<td>0.40</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td></td>
<td>33.90</td>
<td>0.35</td>
<td>36.62</td>
<td>0.60</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td></td>
<td>5.24</td>
<td>0.07</td>
<td>4.82</td>
<td>0.21</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td></td>
<td>81.2</td>
<td>1.10</td>
<td>81.4</td>
<td>0.10</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td></td>
<td>33.90</td>
<td>0.35</td>
<td>36.62</td>
<td>0.60</td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td></td>
<td>0.68</td>
<td>0.03</td>
<td>1.68</td>
<td>0.06</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td></td>
<td>34.81</td>
<td>0.43</td>
<td>26.97</td>
<td>1.18</td>
</tr>
</tbody>
</table>

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Figure 26. Effect of Refining on Brightness of Tembec Unbleached CTMP.
Scattering Coefficient (Sq.m/Kg)

Figure 27. Effect of Refining on Scattering Coefficient of Tembec Unbleached CTMP.
Figure 28. Effect of Refining on Absorption Coefficient of Tembec Unbleached CTMP.
Figure 29. Effect of Refining on Opacity of Tembec Unbleached CTMP.
Effect of Refining on Quensel Unbleached TMP

Results obtained by refining Quensel unbleached TMP are presented in Table 10. Figure 30 shows the effect of refining on the brightness of this pulp.

The $P$-value for refining and brightness was 0.0194. This indicated that the change in the brightness due to refining was significant at the 95% confidence level. The trend indicated that in the early stages of refining some increase in brightness was observed, but as refining progressed, the brightness decreased.

Figures 31, 32, and 33 show the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. Refining increased all these properties significantly. Initial increase in the brightness was possibly because of the dominant effect of increase in $s$-value over $k$-value. But in the later stage of refining, the effect of increase in $k$-value had more influence on brightness than the increase in $s$-value. This resulted in the decrease in the brightness. Since the increase in $s$-value as well as $k$-value had a positive effect on opacity, refining increased the opacity continuously.

Tensile index, TEA, and fines content increased with refining.
<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<tr>
<td></td>
<td>Mean</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>675</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>54.8</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>36.13</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>6.75</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>84.1</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>9.08</td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td>0.57</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>40.67</td>
</tr>
</tbody>
</table>
Figure 30. Effect of Refining on Brightness of Quensel Unbleached TMP.
Scattering Coefficient (Sq.m/Kg)

Figure 31. Effect of Refining on Scattering Coefficient of Quensel Unbleached TMP.
Absorption Coefficient (Sq.m/Kg)

Figure 32. Effect of Refining on Absorption Coefficient of Quensel Unbleached TMP.
Figure 33. Effect of Refining on Opacity of Quensel Unbleached TMP.
Effect of Refining on Quensel Unbleached CTMP

Results obtained by refining this chemithermo-mechanical pulp (the raw material of this pulp was the same as that of the preceding thermomechanical pulp, i.e., 55% white spruce, 35% lodgepole pine, and 10% balsam fir) are given in Table 11. Figure 34 shows the effect of refining on the brightness of this pulp.

The F-value for refining and brightness was 0.0073. Therefore, the change in the brightness due to refining was significant at the 95% confidence level. The trend in brightness indicated that, in the initial stage of refining brightness increased significantly, but in the following stages it leveled off.

Figures 35, 36, and 37 show the effect of refining on the scattering coefficient, the absorption coefficient, and opacity, respectively. The scattering coefficient increased considerably with refining. Increase in the absorption coefficient was less pronounced than the increase in the scattering coefficient. Therefore, an overall increase in brightness was observed. Opacity increased with refining because of increases in s- and k-values.

Strength properties and fines content of the subject pulp increased with refining.
Table 11
Properties of Quensel Unbleached CTMP

<table>
<thead>
<tr>
<th>Properties</th>
<th>Refining Time (minutes)</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>45</td>
<td>65</td>
<td>80</td>
<td></td>
<td></td>
</tr>
<tr>
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<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
<td>Mean</td>
<td>Std.</td>
</tr>
<tr>
<td>Freeness (C.S.F.)</td>
<td>740.00</td>
<td>5.00</td>
<td>335.00</td>
<td>2.50</td>
<td>170.00</td>
<td>6.00</td>
</tr>
<tr>
<td>Brightness (%)</td>
<td>57.60</td>
<td>0.10</td>
<td>60.40</td>
<td>0.30</td>
<td>60.50</td>
<td>0.45</td>
</tr>
<tr>
<td>Scat. Coef. (Sq.m/Kg)</td>
<td>30.81</td>
<td>0.09</td>
<td>36.76</td>
<td>0.78</td>
<td>39.17</td>
<td>1.09</td>
</tr>
<tr>
<td>Abso. Coef. (Sq.m/Kg)</td>
<td>4.81</td>
<td>0.05</td>
<td>4.77</td>
<td>0.01</td>
<td>5.07</td>
<td>0.02</td>
</tr>
<tr>
<td>Opacity (%)</td>
<td>77.90</td>
<td>0.30</td>
<td>81.20</td>
<td>0.00</td>
<td>82.20</td>
<td>0.30</td>
</tr>
<tr>
<td>Tensile Index (Nm/g)</td>
<td>5.46</td>
<td>0.91</td>
<td>29.99</td>
<td>1.21</td>
<td>37.10</td>
<td>0.15</td>
</tr>
<tr>
<td>TEA (J/Sq.m)</td>
<td>0.190</td>
<td>0.05</td>
<td>1.09</td>
<td>0.02</td>
<td>1.58</td>
<td>0.15</td>
</tr>
<tr>
<td>Fines Content (%)</td>
<td>40.26</td>
<td>0.04</td>
<td>29.84</td>
<td>0.91</td>
<td>35.54</td>
<td>0.87</td>
</tr>
</tbody>
</table>

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Figure 34. Effect of Refining on Brightness of Quensel Unbleached CTMP.
Figure 35. Effect of Refining on Scattering Coefficient of Quensel Unbleded CTMP.
Figure 36. Effect of Refining on Absorption Coefficient of Quensel Unbleached CTMP.
Figure 37. Effect of Refining on Opacity of Quensel Unbleached CTMP.
Comparison of Effects of Refining on Brightnesses of Pulps

Figure 38 compares the effects of refining on the brightnesses of the two unbleached thermomechanical pulps included in this study. These pulps were Bowater Unbleached TMP and Quensel unbleached TMP.

The brightness of Bowater TMP increased significantly due to refining. The brightness of Quensel TMP increased initially with refining, but in the later stage it decreased. Raw material of Bowater TMP was loblolly pine while the raw material of Quensel TMP consisted of 55% white spruce, 35% lodgepole pine, and 10% balsam fir. It is possible that due to the differences in the fiber morphology and initial flexibility of fibers of the raw materials used for these pulps, development in s-values of these two pulps were different. Total increase in s-value of Bowater TMP was 61.5% of initial (unrefined) value while for Quensel TMP it was only 20%.

Since the pine fibers of Bowater TMP were stiff and thick-walled (4), probably they were more resistant to swelling and fibrillation than Quensel TMP. This is evident from the difference in refining time (105 minutes for Bowater TMP vs. 55 minutes for Quensel TMP) to achieve double digit freeness values. Fines content (obtained by fiber classification) of highly refined Bowater TMP was 68.08% and for Quensel TMP it was 50.76%.
Figure 38. Comparison of Effects of Refining on Brightnesses of Thermomechanical Pulps.

Legend.  
0 = Bowater, * = Quensel

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Thus, it can be concluded that, in the case of Bowater TMP, the increase in s-value was dominant over the increase in k-value (11.4% of original value). This resulted in an increase in the brightness. In the case of Quensel TMP, the increase in k-value (40.8% of original value) was dominant over the increase in s-value. That was why an overall decrease in the brightness was observed.

Figure 39 shows the comparison of effects of refining on the brightnesses of three unbleached chemithermomechanical pulps studied in this work. These pulps were: Pilot Plant CTMP, Tembec CTMP, and Quensel CTMP. Except for Quensel CTMP, the raw material for the other two pulps was spruce. For Quensel CTMP, spruce represented the largest portion of the raw material (55%).

It is evident from Figure 39 that brightness values of all these pulps increased only modestly in the early part of the refining process. But in the later stage of refining, brightness values decreased. A similar trend was observed with s-values of these pulps (Tables 5, 9, and 11). This indicates that, initially, the fines generated due to refining possibly helped in increasing the scattering coefficients of the pulps. As refining progressed, fibrillation of fibers (spruce fibers are thin walled and flexible; further chemical pretreatment improved the fiber flexibility and enhanced the bonding properties) improved fiber bonding. This bonding effect decreased the scattering coefficients of the pulps.
Figure 39. Comparison of Effects of Refining on Brightnesses of Unbleached Chemithermomechanical Pulps.

Legend. Δ = Pilot Plant, 0 = Tembec, * = Quensel

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In the case of equal and opposite effects of fiber bonding and generation of fines, the scattering coefficient remained unaffected. Since the $k$-values of highly refined pulps were higher than their corresponding unrefined pulps, a combination of the decrease in $s$-values and increase in $k$-values resulted in the decrease in the brightness values of these pulps.

Figure 40 shows the comparison of the effects of refining on the brightness values of thermomechanical and its corresponding chemithermomechanical pulps (i.e., from the same raw material: 55% white spruce, 35% lodgepole pine, and 10% balsam fir; CTMP was given chemical pretreatment while TMP was not) received from Quensel River Pulp Company. Refining initially increased the brightness values of both pulps but, in the later stage a drop in the brightness values was noticed. The brightness drop of TMP was more pronounced than that of CTMP.

In general, the $s$-value and $k$-value of TMP were higher than CTMP. Because of the chemical pretreatment, the $k$-value of CTMP is lower than that of TMP. Since in the early stage of refining, the increase in $s$-values of both pulps was significant, an increase in the brightness values was observed. In the later stage of refining, an increase in $k$-values was dominant over the increase in $s$-values; therefore, a brightness drop was observed. The percentage increase in the $k$-value of TMP (lowest freeness pulp over
Figure 40. Comparison of Effects of Refining on TMP and CTMP from Same Raw Material (Quensel Pulp).

Legend.  0 = TMP,  * = CTMP
unrefined pulp) was higher (40.9%) than that of CTMP (11.9%). This was the possible reason for the higher brightness drop of TMP than that of CTMP due to refining.

Figure 41 compares the effects of refining on the brightnesses of the two bleached chemithermomechanical pulps (BCTMP) included in this study. These pulps were: Pilot Plant BCTMP and Quensel BCTMP. Different raw materials were used in making these pulps. The raw materials are given in Table 3.

Brightness values of both pulps decreased with refining. The scattering coefficients of both pulps decreased and the absorption coefficients increased with refining. A decrease in s-values indicates that BCTMP behaved more like chemical pulp. Chemical treatment before refining helped in improving the fiber to fiber bonding. The combined effects of an increase in k-values and a decrease in s-values resulted in the decrease of the brightness values of both pulps.

Figure 42 compares the effects of refining on the brightnesses of Quensel CTMP and corresponding BCTMP. Figure 42 shows that the brightness of unbleached CTMP increased in the initial stage of refining, but in the later stage it decreased. Brightness of BCTMP started decreasing from the very beginning. An increase in k-values was observed with both pulps. The increase in k-value of BCTMP was 120.9% (lowest freeness pulp over unrefined pulp) while it was only 11.8% for CTMP.
Figure 41. Comparison of Effects of Refining on Brightnesses of Bleached Chemithermomechanical Pulps.

Legend. 0 = Pilot Plant, * = Quensel
Figure 42. Comparison of Effects of Refining on Brightness of Quensel CTMP and Corresponding BCTMP.

Legend. 0 = Unbleached CTMP, * = Bleached CTMP

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The scattering coefficient of CTMP increased with refining but it decreased for BCTMP. The combined effects of significant increases in the k-value and decreases in the s-value resulted in a significant brightness loss of BCTMP. Increases in the s-value of CTMP helped in offsetting the effect of an increase in k-value on the brightness of CTMP. That was why the brightness loss of CTMP due to refining was nominal.

Explanation for the Changes in s- and k-Values

As mentioned in Chapter IV, fiber analyses were performed and kappa numbers were determined to attempt to explain the effects of refining on the scattering and absorption coefficients of various pulps used in this study.

Fiber analyses were performed by using a Kajaani Fiber Analyzer and fines contents of different pulps at different freeness levels were determined. The fines content of different pulps at different freeness levels are presented in Tables 5 through 11 in earlier discussion. Corresponding scattering coefficients are also given in the same tables. In general, these results indicated that unrefined pulps had higher fines content than their corresponding refined pulps. As discussed in chapter IV, this discrepancy might have arisen from the fact that refining generated many small fines which were beyond the measurement capability of the Kajaani Fiber Analyzer. The instrument's lower limit
of measurement is 20 micrometers. Probably this was the reason that a strong correlation between fines content and the scattering coefficient did not exist.

Generally the absorption coefficients (k) of the pulps increased with refining. The increase was, however, noticeable only with TMP and BCTMP.

Parsons (22) found an increase in the k-value of bleached spruce sulfite pulp with beating. Parsons fractionated chemical and groundwood pulps using softened water and concluded that fines were very sensitive in catching and retaining the dirt. Parsons hypothesized that this was the possible reason that the pulps with high fines content had high k-values.

Since in this research, the chelating agent (EDTA) was used during refining, the effect of metals ions on brightness may be assumed negligible. In this study the following reasons may be considered as the contributing factors in increasing the k-value:

1. According to Stieg (44), fines smaller in size than one-half the wavelength of light do not contribute to the scattering power. They may increase the absorption of the incident light. Therefore smaller fines may increase the k-value of the pulp.

2. Fines have a large surface area and if colloidal or ionic foreign particles coming with the pulp and/or water adsorb on these fines, their overall effect on k-value may be significant.
3. Refining of the pulp may expose the lignin on the surface and hence may affect the k-value. To study this phenomenon, kappa numbers of some pulps were determined. The pulps of highest and correspondingly lowest freeness were included in this experiment. Two determinations were made at each freeness level of the pulps. Mean and standard deviation values were calculated. Unbleached pulps from the following sources were used for kappa number determination: (a) TMP from Bowater Inc., SC; (b) TMP from Quensel River Pulp Company, Canada; and (c) CTMP corresponding to (b).

The kappa numbers of these pulps are given in Table 12.

Results from Table 12 indicate that the kappa number of Quensel thermomechanical pulp was higher at low freeness than at high freeness. But the standard deviation for highly refined pulp was high. No significant difference in the kappa numbers of unrefined and highly refined Bowater thermomechanical pulp was observed. This is similar to the case with Quensel chemithermomechanical pulp.

Therefore, no definite explanation for the changes in k-values could be based on kappa numbers.
Table 12
Kappa Numbers of Unrefined and Corresponding Refined Pulps

<table>
<thead>
<tr>
<th>Pulp Source</th>
<th>Pulp Type</th>
<th>Freeness (C.S.F.)</th>
<th>Kappa Number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mean</td>
</tr>
<tr>
<td>Bowater Carolina</td>
<td>Unbleached</td>
<td>690</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>TMP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bowater Carolina</td>
<td>Unbleached</td>
<td>55</td>
<td>156</td>
</tr>
<tr>
<td></td>
<td>TMP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quensel Canada</td>
<td>Unbleached</td>
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<td>163</td>
</tr>
<tr>
<td></td>
<td>TMP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quensel Canada</td>
<td>Unbleached</td>
<td>32</td>
<td>208</td>
</tr>
<tr>
<td></td>
<td>TMP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quensel Canada</td>
<td>Unbleached</td>
<td>735</td>
<td>182</td>
</tr>
<tr>
<td></td>
<td>CTMP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quensel Canada</td>
<td>Unbleached</td>
<td>62</td>
<td>170</td>
</tr>
<tr>
<td></td>
<td>CTMP</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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CHAPTER VI

SUMMARY OF RESULTS

Refining of Bowater TMP increased the brightness significantly. This was a Southern pine TMP. Increase in the scattering coefficient had more influence than the increase in the absorption coefficient on the brightness.

Refining of BCTMP decreased the brightness mainly because of increase in the absorption coefficient. A decrease in the scattering coefficient was also observed.

Spruce TMP and CTMP did not show increases in brightness with refining similar to pine, because of morphological differences of fibers. Thin walled and long spruce fibers did not increase the scattering coefficients of the pulps comparable to southern pine due to improved bonding upon refining.

Refining of Quensel River TMP (55% white spruce, 35% lodgepole pine and 10% balsam fir) did not increase the brightness as was the case with corresponding CTMP in spite of the fact that the increase in the scattering coefficient was significant. The reason for the decrease in brightness was that the increase in the absorption coefficient outweighed the increase in the scattering coefficient. CTMP showed an increase in brightness with refining because of less increase in the absorption coefficient.
Kajaani fiber analyses showed higher fines content for unrefined pulps than corresponding refined pulps. This was because of the inability of the Kajaani Fiber Analyzer FS 100 to characterize fines in the fraction below 0.2 mm and to detect the fines smaller than 0.02 mm. Refining may have developed more fines in this region that were not detected.

Any definite explanation for the increases in k-values due to refining could not be based on kappa numbers because no significant change in kappa number was observed with refining.

Refining increased the opacity of TMP and CTMP. Increases in the scattering and absorption coefficients were the reason for increases in opacity.

Tensile indices and tensile energy absorption (TEA) values of all pulps increased with refining because of increased bonding of fibers.
CHAPTER VII

CONCLUSIONS

From the experimental data obtained in this study the following conclusions may be drawn:

1. Refining of southern pine TMP was effective in increasing the brightness because of significant increases in s-value of this pulp with refining. This behavior was not observed with spruce TMP because the increase in s-value of this pulp with refining was not as pronounced as it was with pine TMP.

2. Both the scattering and absorption coefficients of pulps normally were increased by refining. If increases in the k-value were more dominant than increases in the s-value, the brightness of pulp decreased and vice versa. In some cases s-values decreased with refining, which was attributed to increased fiber to fiber bonding.

3. Thermomechanical pulps had higher scattering coefficients than the corresponding chemithermomechanical pulps made from a mixture of white spruce, lodgepole pine, and balsam fir. But an increase in k-values due to refining was also higher for TMP than the corresponding CTMP.

4. Brightness of BCTMP decreased with refining. Refining possibly exposed the inner dark surfaces of fibers
which resulted in an increase in k-value. Thus, the decrease in brightness which was observed could be explained.

5. Opacity of TMP and CTMP increased with refining because the increases in the s- and k-values had positive effects on opacity.
CHAPTER VIII

SUGGESTIONS FOR FURTHER STUDY

Further experimental research is recommended in the following areas:

1. This study should be performed with hardwoods, southern pines (i.e. slash, longleaf and shortleaf), western hemlock and Douglas fir.

2. Refining should be done on a pilot plant scale (using disc refiner) to verify the refining results obtained in this study using a laboratory beater.

3. Some other means of fiber analysis should be used for determination of fines content which can characterize fines below 0.2 mm and detect the fines smaller than 0.02 mm.

4. Some other method should be developed to check the exposed surface area of fines.

5. Effect of refining consistency on the brightness of pulp should be determined.
LITERATURE CITED


25. Giertz, H. W., "Understanding the Role of Fines".


Appendix A

Strength Determination of Sodium Sulfite Liquor
Appendix A

Strength Determination of Sodium Sulfite Liquor

For the preparation of the liquor, calculated amount of sodium sulfite was dissolved in the calculated amount of water by thorough agitation and slow addition. Well mixed solution was titrated with 0.1N Iodine solution using starch solution as an indicator.

The reaction was as follows:
\[ \text{Na}_2\text{SO}_3 + \text{I}_2 + \text{H}_2 \rightarrow \text{Na}_2\text{SO}_4 + 2\text{HI} \]

1 ml of I₂ = 0.003203 gm SO₂ \( \frac{126.04 \text{ Na}_2\text{SO}_3}{64.06 \text{ gm SO}_2} \)
or, 1 ml of I₂ = 0.006302 gm of Na₂SO₃

\[ \% \text{ Na}_2\text{SO}_3 = \frac{\text{gm of Na}_2\text{SO}_3}{\text{gm of solution weighed}} \times 100 \]

Volume of iodine consumed in titration for 20 ml sodium sulfite solution was 117.4 ml.

Therefore,
Strength of sodium sulfite solution = 117.4 X 0.006302 X 5
\[ = 3.7\% \]
Appendix B

Description of Sunds Defibrator
Appendix B

Description of Sunds Defibrator

Figure 43 shows the schematic diagram of Sunds Defibrator. Following is its description.

Infeed Hopper

It is made of stainless steel and is provided with metering screw. It has flanged inlet of 12"X30". It also has four 1/2" steam inlets. The metering screw has a conical shaft and 4" flight diameter.

Specifications:

- Hopper volume : 0.14 cu.m
- Volume of the screw : 0.00062 cu.m./pitch.
- Speed of the screw : 2 - 40 rpm.

Screw Feeder

Screw feeder is designed to feed against steam pressure and liquor. It is equipped with plug pipes, feed screw, throat housing, bearing assembly, and is provided with positive feeding arrangement. Cover plates with drain funnel surround the throat section. Screw feeder is mounted on a steel hinged base. When the chips are compressed into a plug, a pressure seal is formed between screw feeder and preheater.
Figure 43. Schematic Diagram of Sunds Defibrator.
Specifications:
Volume of screw : 0.00024 cu. m/pitch.
Speed of screw : 6 - 58 rpm.

Preheater

The cooking time is maintained by the speed of the discharge screw.

Specifications:
Working pressure : max. 1722 kPa
Working temperature : max. 208°C
Total volume of preheater : 0.262 cu.m
Effective volume (bottom of impregnator): 0.11 cu.m
Volume of impregnator screws : 2X0.00062 cu.m/pitch
Volume of impregnator : 0.0015 cu.m
Speed of impregnator screws : 60 HZ 18rpm
Volume of discharge screw : 0.00062 cu.m/pitch
Blow back air cylinder 12.7 cm dia. : min. pr. 482 kPa
Speed of discharge screw : 1.5 - 25 rpm
Speed of agitator : 60 HZ 4.5 rpm

Defibrator 300 Feed Screw

This is 4" diameter full flight stainless steel screw with a packing box. The feed screw housing is provided with a 7.6 cm vent line (steam return pipe) to the preheater for the generated steam. The vent line has a 5.1 cm flanged connection for steam relief valve.
Specifications:

Volume of screw : 0.00062 cu.m/pitch
Speed of screw : 60 HZ 300 rpm.

Defibrator 300 CD

The defibrator is equipped with a "disc and cone" refining element and is known as type 300 CD. The two zones are adjustable independently from each other. The refining disc diameter is 12" + 4" cone (300 mm + 100 mm). The discs are encased in a stainless steel housing with a split cover. The main body is also split horizontally for access and servicing. The housing is designed for a maximum working pressure of 1722 kPa.

The defibrator 300 CD is provided with three flanged outlets (right-left-bottom). The side flange, left is provided with a blow valve by which the rate of pulp flow is controlled to two different chests. The side flange, right is provided with a hand operated blow valve which controls the flow rate of pulp to the sampling point in the digester room. The bottom outlet is used for atmospheric refining.

The disc clearances for the flat and conical zones of refiner are independently adjustable by separate handwheels mounted on the refiner housing. Disc clearances are adjusted manually with the help of handwheels. For the flat zone, one revolution of handwheel equals to 0.2 mm movement.
of rotor. For the conical zone one revolution of handwheel equals to 0.78 mm change in discs clearance. Arrangements can also be made for water and/or chemical addition into the refining zone. The main shaft is equipped with a stainless steel hardfaced wear sleeve and is supported by SKF type ball bearings.

The digester is controlled by a Foxboro Fox 300 process control computer. The computer logs all collected variables continuously which can be recalled at any time.

Specifications:
Refining disc diameter : 12" + 4" (300 mm + 100 mm)
Speed : max. 3600 rpm
Connected power : max. 150 kW
Working pressure : max. 1722 kPa.
Appendix C

Determination of Pulp Yield
Appendix C

Determination of Pulp Yield

Pulp production rate ($P$) = 2.4550 kgs / min.
Blow pulp consistency ($C$) = 16 %
Chip feed rate ($I$) = 0.6818 kgs / min.
Chip moisture ($M$) = 37 %
3.7 % Sodium sulfite addition rate ($S$) = 0.01233 kgs / min.

$$
\text{CTMP Yield} = \left[ \frac{PC}{I(100-M)} + S \right] \times 100
= \left[ \frac{2.4550 \times 0.16}{0.6818 \times (1.00 - 0.37)} + 0.01233 \right] \times 100
= 92.7\%
$$

Thus, the pulp yield of pilot plant unbleached CTMP was 92.7%.
Appendix D

Correction of Opacity for Basis Weight
Appendix D

Correction of Opacity for Basis Weight

1. Express $R_0$, $R_{\text{inf}}$, and TAPPI Opacity ($C_{0.89}$) of the specimen in decimal form.

2. Calculate scattering power $sW$ with the following equations:

$$a = 0.5 \left( \frac{1}{R_{\text{inf}}} + R_{\text{inf}} \right)$$
$$b = 0.5 \left( \frac{1}{R_{\text{inf}}} - R_{\text{inf}} \right)$$
$$x = \frac{1 - a R_0}{b R_0}$$

Scattering power $sW = \frac{0.5}{b} \ln \left( \frac{x + 1}{x - 1} \right)$

Here, $W$ is basis weight of specimen.

3. Since scattering power changes in proportion to the basis weight, therefore:

New $sW' = \frac{\text{Old } sW \text{ (new basis weight)}}{\text{ (old basis weight)}}$

The $a$ and $b$ values do not change with basis weight because $R_{\text{inf}}$ is independent of basis weight.

4. Using $b$ and $sW'$ values calculate $R_0'$ as following:

$$Z = 2bsW'$$
$$N = e^Z$$
$$x' = \frac{(N+1)}{(N-1)}$$
$$R_0' = \frac{1}{(x'b + a)}$$
5. Calculate corrected opacity as following:

\[ R_{(0.89)} = R_0' + 0.89 \left[ \frac{(R_{INF} - R_0')}{(1 - R_0' R_{INF})} \right] R_{INF} \left(1 - \frac{0.89 R_0'}{R_{INF}} \right) \]

Corrected TAPPI Opacity = \( C_{0.89} = \frac{R_0'}{R_{(0.89)}} \)

The above mentioned equations were obtained from Technidyne Corporation.
Appendix E

Calculation of Scattering (s) & Absorption (k) Coefficients
Appendix E

Calculation of Scattering (s) & Absorption (k) Coefficients:

1. Express $R_0$ and $R_{INF}$ in decimal form.
2. Calculate $sW$ in the following manner:
   \[ a = 0.5 \left( \frac{1}{R_{INF}} + R_{INF} \right) \]
   \[ b = 0.5 \left( \frac{1}{R_{INF}} - R_{INF} \right) \]
   \[ x = \left( 1 - a.R_0 \right) / b.R_0 \]

Scattering power $Sp = (0.5 / b) \ln \left( \frac{x + 1}{x - 1} \right)$
Scattering coefficient $s = 1000 Sp / W$
Absorption power $Ap = (a.Sp) - s.W$
Absorption coefficient $k = 1000 Ap / W$

Here, $W =$ Basis weight (gram / m²),
$s =$ Scattering coefficient (m² / Kg), and
$k =$ Absorption coefficient (m² / Kg).

These equations were taken from Tappi Method - Opacity of Handsheets (T 425 om-86).
Appendix F

Handsheet Properties of Various Pulps
## (a) Average Properties of Various Pulps at Different Freeness Levels

<table>
<thead>
<tr>
<th>Raw Material</th>
<th>Pulp Origin and Type</th>
<th>Pulp Yield (%)</th>
<th>Relining Time (Min.)</th>
<th>Freeness (ml CSF)</th>
<th>Brightness (%)</th>
<th>Opacity (%)</th>
<th>Absp. Coeff. (m²/kg)</th>
<th>Scatt. Coeff. (m²/kg)</th>
<th>Tensile Index (Nm/g)</th>
<th>TEA (J/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spruce</td>
<td>PILOT PLANT</td>
<td>92.7</td>
<td>1</td>
<td>530</td>
<td>30.3</td>
<td>0.50</td>
<td>96.3</td>
<td>0.14</td>
<td>18.79</td>
<td>23.44</td>
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<tr>
<td></td>
<td>UNBLEACHED CTMP</td>
<td></td>
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<tr>
<td>Spruce</td>
<td>PILOT LABORATORY</td>
<td>91.0</td>
<td>1</td>
<td>455</td>
<td>37.8</td>
<td>0.45</td>
<td>93.2</td>
<td>0.22</td>
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<tr>
<td>55/35/10</td>
<td>QUENSEL RIVER</td>
<td>90.0</td>
<td>1</td>
<td>505</td>
<td>74.4</td>
<td>0.41</td>
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<td>1.60</td>
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<td>TEMBEC CANADA</td>
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<td>1.32</td>
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### Notes
- TEA: Tensile Energy Absorbed
- Raw Material: The type of raw material used for pulp production.
- Pulp Origin and Type: The specific type of pulp, such as PILOT or UNBLEACHED CTMP.
- Pulp Yield (%): The percentage yield of the pulp.
- Relining Time (Min.): The time required for pulp refining.
- Freeness (ml CSF): The amount of water required to break down the pulp into a sheet.
- Brightness (%): The brightness of the pulp sheet.
- Opacity (%): The opacity of the pulp sheet.
- Absp. Coeff. (m²/kg): Absorption coefficient.
- Scatt. Coeff. (m²/kg): Scattering coefficient.
- Tensile Index (Nm/g): Tensile strength.
- TEA (J/m²): Tensile Energy Absorbed.
(b) Results Obtained by the Duplications of the Work Done Under (a)

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<th>Retting Time Min.</th>
<th>Freeness ml CSF Mean</th>
<th>Brightness % Mean</th>
<th>Opacity % Mean</th>
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<th>Scatt. Coeff. m2/Kg</th>
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## (c) Properties of Pulps Before and After Circulation in Valley Beater Without Load

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<th>Brightness %</th>
<th>Opacity %</th>
<th>Absp. Coeff. m²/Kg</th>
<th>Scatt. Coeff. m²/Kg</th>
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(d) Variations in Brightness Within Duplicated Handsheets

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<tr>
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<td>118 0.30</td>
<td>56.0 0.30</td>
<td>55.8 0.41</td>
<td>55.9 0.48</td>
<td>56.1 0.33</td>
<td>55.6 0.28</td>
<td>55.9 0.17</td>
</tr>
<tr>
<td>TMP</td>
<td>38 0.26</td>
<td>51.9 0.26</td>
<td>52.0 0.47</td>
<td>52.7 0.62</td>
<td>52.5 0.50</td>
<td>53.6 0.29</td>
<td>52.5 0.61</td>
</tr>
<tr>
<td>QUENSEL</td>
<td>745 0.37</td>
<td>57.8 0.37</td>
<td>57.8 0.42</td>
<td>57.6 0.47</td>
<td>57.7 0.28</td>
<td>57.8 0.40</td>
<td>57.7 0.07</td>
</tr>
<tr>
<td>RIVER</td>
<td>330 0.88</td>
<td>60.7 0.88</td>
<td>61.0 1.04</td>
<td>60.4 0.48</td>
<td>60.7 0.70</td>
<td>60.6 1.01</td>
<td>60.7 0.18</td>
</tr>
<tr>
<td>UNBLCHD</td>
<td>176 0.56</td>
<td>60.7 0.56</td>
<td>60.8 0.80</td>
<td>61.0 0.60</td>
<td>61.1 0.52</td>
<td>60.8 0.69</td>
<td>60.9 0.15</td>
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<tr>
<td>CTMP</td>
<td>68 0.30</td>
<td>60.1 0.30</td>
<td>60.1 0.44</td>
<td>60.1 0.54</td>
<td>60.2 0.38</td>
<td>60.0 0.67</td>
<td>60.1 0.06</td>
</tr>
</tbody>
</table>
Appendix G

Results of One-way ANOVA
Appendix G

Results of One-way ANOVA

Independent Variable = Refining Time
Dependent Variable = Brightness
Degrees of Freedom = 3

<table>
<thead>
<tr>
<th>Pulp Name</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-value</th>
<th>r²</th>
<th>F-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pilot Plant Unbleached CTMP</td>
<td>12.3138</td>
<td>4.1046</td>
<td>17.01</td>
<td>0.93</td>
<td>0.0097</td>
</tr>
<tr>
<td>Laboratory Bleached Pilot Plant CTMP</td>
<td>43.8800</td>
<td>14.627</td>
<td>4.01</td>
<td>0.75</td>
<td>0.1064</td>
</tr>
<tr>
<td>Quensel Canada Bleached CTMP</td>
<td>234.820</td>
<td>78.273</td>
<td>6.57</td>
<td>0.83</td>
<td>0.0502</td>
</tr>
<tr>
<td>Bowater Carolina Unbleached TMP</td>
<td>51.9937</td>
<td>17.331</td>
<td>43.19</td>
<td>0.97</td>
<td>0.0017</td>
</tr>
<tr>
<td>Tembec Canada Unbleached CTMP</td>
<td>29.5100</td>
<td>9.8367</td>
<td>1.17</td>
<td>0.47</td>
<td>0.4258</td>
</tr>
<tr>
<td>Quensel Canada Unbleached TMP</td>
<td>15.7400</td>
<td>5.2467</td>
<td>11.53</td>
<td>0.90</td>
<td>0.0194</td>
</tr>
<tr>
<td>Quensel Canada Unbleached CTMP</td>
<td>10.8450</td>
<td>3.6150</td>
<td>19.81</td>
<td>0.94</td>
<td>0.0073</td>
</tr>
</tbody>
</table>
Appendix H

Description of Kajaani Fiber Analyzer FS 100
Appendix H

Description of Kajaani Fiber Analyzer FS 100

Instrument Design

This instrument, introduced by Kajaani Automation Inc., has mainly following four components:

1. An optical measuring unit consisting of a halogen lamp light source and photocell detector,
2. A low pressure vacuum pump and chamber to collect the analyzed fibers,
3. A microprocessor based unit serving as the calculating device, and
4. A control keyboard for entering operation instructions and a printer.

Principle of Operation

Figure 50 shows the measurement principle of Kajaani Fiber Analyzer FS 100. An aqueous suspension of fibers is passed through the capillary tube.

Since the light source is located on one side of capillary and the detector is positioned on the opposite side, the image of the fiber is projected onto the detector with the aid of system optics. This image provides the information about the lengthwise dimension of the fiber.
Figure 44. Measurement Principle of the Kajaani Fiber Analyzer FS 100.

**Operation**

A very dilute aqueous suspension of the pulp is prepared in such a way that visually all the fibers and fines look completely dispersed. This suspension is poured into the funnel where it is kept continuously suspended with the help of the agitator.

Fibers are drawn through the capillary due to the vacuum created by the vacuum pump and thus measurement of fiber length is done. Measuring range is set before the fiber count begins.
Operation Specifications

Capillary diameter | 0.2 mm
Measuring channels | 24
Sensitivity | 20 micrometers
Speed | max. 50 fibers / s
Measuring ranges

Fiber length measurement resolutions, mm

Results

FS 100 prints out the distribution and cumulative frequency functions as well as information concerning the average fiber length of fibers either mathematically or as a weighted average by length. Fibers smaller than or equal to 0.21 mm are characterized as fines. For short fibered pulps, 0 - 2.4 mm range is chosen for measurements.
Appendix I

Graphs of Tensile Index and TEA of Various Pulps
Appendix I

Graphs of Tensile Index and TEA of Various Pulps

Figure 45. Effect of Refining on Tensile Index of Pilot Plant Unbleached CTMP.

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Figure 46. Effect of Refining on TEA of Pilot Plant Unbleached CTMP.
Figure 47. Effect of Refining on Tensile Index of Pilot Plant Laboratory Bleached CTMP.
Figure 48. Effect of Refining on TEA of Pilot Plant Laboratory Bleached CTMP.

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Figure 49. Effect of Refining on Tensile Index of Quensel Bleached CTMP.
Figure 50. Effect of Refining on TEA of Quensel BCTMP.
Figure 51. Effect of Refining on Tensile Index of Bowater Unbleached TMP.
Figure 52. Effect of Refining on TEA of Bowater Unbleached TMP.
Figure 53. Effect of Refining on Tensile Index of Tembec Unbleached CTMP.
Figure 54. Effect of Refining on TEA of Tembec Unbleached CTMP.
Figure 55. Effect of Refining on Tensile Index of Quensel Unbleached TMP.
Figure 56. Effect of Refining on TEA of Quensel Unbleached TMP.
Figure 57. Effect of Refining on Tensile Index of Quensel Unbleached CTMP.
Figure 58. Effect of Refining on TEA of Quensel Unbleached CTMP.
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