Drying Behavior of Fractionated Fibers

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DRYING BEHAVIOR OF FRACTIONATED FIBERS

by

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This dissertation studies water removal from paper produced using fractionated fibers. One of the principal aims of fiber fractionation is to separate long fibers from fines and then to use these long fibers in paper grades that require high bulk and high water absorption. However, during the paper manufacturing process, the bulk of these materials may be reduced by mechanically pressing the paper to remove water. Also the drying rate of the high bulk paper may be lower than conventional paper due to changes in the resistance to heat transfer. The main objective of this dissertation is to further develop methods of measuring paper drying rates and determine the effect of different process variables on the drying rates.
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CHAPTER I
INTRODUCTION AND LITERATURE REVIEW

General Background

The objective of this study was to determine the effect of fiber fractionation on paper-drying rates and heat-transfer characteristics of both virgin and recycled fibers. The costs for fibers and for drying energy are the two most important costs in paper manufacturing. The fractionation process can be used to separate and recover a particular fiber-length range from a cheap fiber source, such as recycled fiber or sludge. These recovered fibers may be used to produce lower-cost paper products. An important aspect of the fractionation process that has not been previously studied is the relationship between fiber fraction and drying. This dissertation covers that gap in the research.

One application of fiber fractionation could be improving recycled sheet properties (Scott & Abubakr, 1994). Since recycled fibers lose flexibility, less water is removed during pressing; therefore, they require more drying time and energy. The quality of recycled fiber can be improved by separating long fibers from short fibers and fines (Scott & Abubakr; Howard, 1991). The long fibers can be used in high-quality products, and the short fibers can be used in lower-quality products (Howard; Kosta & Carsky, 1997). In a patent, (Pounder 1993) describes multi-layer papers and tissues. This patent states that the most important aspects of such products are strength, softness, and absorbency. Recycling increases fiber stiffness, reduces fiber-
fiber bonding potential, and decreases the paper's strength characteristics. Reasons for this decrease in paper strength include the inability to re-hydrate the fibrils; the loss of hemicellulose; the decrease in the fines, which are lost during the recycling process; and the reduction in fiber flexibility and permeability with respect to water (Smook, 1997). Hemicellulose contributes to bonding through the carboxyl groups. The carboxyl groups are more highly charged and form stronger bonds.

Koniarik (1984) stated that

To remove 1 kg of water from paper in the dryer section, about 1.5 kg of steam is required.” He also stated that “because of the cost of energy, it is important to remove mechanically as much water as possible. The drying depends on the moisture content and the partial pressure of vapor in the dryer section air. Also, the type of fiber, degree of refining, paper basis weight, type of the felts, quality of the cylinder surface, and other factors affect the drying process. (p. 68-72)

Abubakr, Scott, and Klungness (1995) studied the use of fiber fractionation to increase the utilization of mixed office waste. They studied the effect of fractionation on the mechanical properties of both the recycled fibers and the resulting paper. They concluded that the longer fibers’ fractions were less affected by recycling than were the shorter fibers’ fractions.

Oksanen, Buchert, and Viikari (1997) studied the deterioration of the fiber properties after recycling. They related deterioration in recycled fibers, or hornification, a loss of swelling capacity, to irreversible structural changes in the fibers during drying. The dried fibers showed loss of swelling and bonding capacity, which was confirmed by reductions in the water-retention value (WRV), sheet density, tensile strength, total pore volume, as well as an increase in fiber stiffness.
Since the loss of hemicellulose contributes to homification, the retention of hemicellulose in fine pores and interfibrillar spaces can reduce homification.

Karnis (1997) studied commonly used fractionation equipment. He reported that the basic task of fractionation was to separate fibers based on their length, and that separation by fiber flexibility was a secondary effect. Karnis listed the characteristics of the float-wash fractionator, the liquid-column fractionators, and the hydrocyclone. He said that:

The float-wash fractionator is a very efficient separator for mechanical pulp. The liquid-column fractionator uses plug flow to separate fibers on the basis of their length and the hydrocyclone, which is highly effective for mechanical pulps, separates fibers based on the balance between the apparent density of the particle and its surface area. For unbeaten chemical pulps this balance results in separation based on fiber coarseness or cell-wall thickness. For highly refined pulps such as mechanical pulps, separation is based on specific surface. The atomizer separates fibers based primarily on their diameter. (p. 480-488)

The U.S. Department of Energy's (DOE) National Renewable Energy Laboratory (NREL) ("Clean Wood Fractionation," 2000) researched a clean fractionation process to produce cellulose and feedstocks for making chemicals. It used an organic solvent and water to dissolve the lignin and hemicellulose in the wood. The lignin and the hemicellulose ended up in aqueous phases. Filtration removed the remaining cellulose, which was purified by bleaching processes.

Guss and Meier (1997) studied the features, operation, and economic advantages of fractionation to recover usable fiber from secondary-fiber systems, washer effluent, floated sludge, and excess paper-machine white water.
Vinson et al. (2001) developed a new technology based on the use of sequential centrifugal screens and hydrocyclones to continuously fractionate a fiber stream into two streams. This method produced two fiber streams with higher values than the initial stream. The authors applied this technique to mixed office waste; one of the produced streams had an improved relationship between softness and strength. It also showed a decreased need for deinking. The other stream showed improved strength and higher freeness compared to the feed stream.

Fiber length is an important parameter in paper making. For example, long fibers tend to produce paper with higher tear strength than short fibers. Thus the fractionation process has an impact on the quality of the recovered fiber. For example, Zhu and Tan (2005) studied fractionation phenomena in the flotation deinking processes. They collected fibers from different heights and analyzed their fiber-length distributions. They found that these fiber-length distributions had lower fractions of short fibers and higher fractions of long fibers than those in the original pulp sample. The length-weighted mean fiber length in the collected froth was 11% longer than the original fiber. They concluded that flotation deinking selectively rejected more fibers greater than 2 mm compared with the original pulp, and this significantly impacted the quality of recovered fibers.

Haggblom-Ahnger & Hautala (1996) prepared double-layer test sheets based on fractionated recycled fiber obtained from old corrugated boxes. The top ply consisted of clean, fractionated short fibers, and the back ply consisted of screened and cleaned long fibers (without dispersion and refining). The sheets were compared
to a reference sheet containing chemical pulp in the top ply and recycled fiber in the back ply prepared by conventional methods. The sheets were analyzed for bulk, stiffness, and internal bond; short-span compression testing; ring-crush testing; and visual inspection. Haggblom-Ahnger and Hautala’s results indicated that:

The optimally fractionated test sheet containing 100% old corrugated carton (OCC) had 6% higher bulk, 10% higher stiffness, 15% higher ring crush test (RCT) values, better strength properties, and higher short span compression test (SCT) values than the reference sheet. The test sheets appeared smoother and cleaner and the internal bond was maintained at an acceptable level. The parameters that influenced the fiber fraction and the reduction of contaminants were a sequence of pulping, coarse screening, and dispersion. (p. 386-391)

Moss and Retulainen (1995) investigated the effect of fines on the fiber-bonding properties of dry paper. They analyzed handsheets prepared from the long-fiber fraction of a spruce, with long fibers and 30% fines, and long fines and 30% kraft fines. They reported that:

Sheets made from 100% long fibers were bulky with poor strength when compared to other samples and the samples containing kraft fines were significantly stronger than those containing thermal mechanical pulp (TMP) fines. Fibers in networks to which fines had been added generally became flatter upon drying than those in networks consisting of long fibers alone. (p. 97-101)

Drying Curve

Paper drying can be defined as the reduction of the moisture content to less than 10% by a process of pressing and heating (Clark, 1985). The drying process results in drastic changes in the physical properties of both the fibers and the paper. For example, drying causes a partial collapse of the external micro-fibrils and a slight fiber curl. The external fibril collapse affects physical properties such as burst, tear,
bulk, and opacity. As the paper dries, the increasing surface tension forces pull the fibers together allowing hydrogen bonds to form. Drying then increases the fiber's cohesiveness and the paper-strength properties.

The drying time and the required energy are the most important factors in all drying processes. Figure 1 shows typical drying curves. Kuang, Thibault, Chen, and Grandjean (1995) found that the evaporation rate decreased with time as the paper sheet passed through the drying section. As the temperature of the dryer surface increased, a higher evaporation rate was obtained but with lower drying efficiency. A typical drying curve was divided into four periods: warm-up period, constant-rate period, first falling-rate period and second falling-rate period.

As drying proceeded, the continuous water film on the paper surface disappeared and water evaporation occurred within the paper's pores. The rate of drying depended on the rate of water leaving the surface. Once the paper surface was not uniformly wet, the drying rate decreased. This occurred at the beginning of the first falling period. As the drying rate decreased, a new energy balance had to be reached at the surface, with a slight increase in the sheet temperature, for drying to continue. Free water still existed at the surface, and dry patches still contained bound water. With further drying, the dry patches expanded until there was no free water remaining on the surface, and a rapid increase in paper temperature was observed. This occurred at the beginning of the second falling-rate period, in which the water was conducted to the surface by capillary flow (Zhe, Shiming, & Dengying, 1999).
Figure 1. Typical Drying Rate versus Moisture Content Curve. The AB segment represents the warm-up period. The BC segment represents the constant-rate period. The CD segment represents the falling-rate period, and the D segment represents the bound-water evaporation period.

Paper is hygroscopic porous material that absorbs water from the surrounding environment. The physical structure of the paper affects the way it dries. Four distinct drying segments are noticed in paper drying: the warm-up segment (from A to B in Figure 1), the constant-rate period (from B to C), the first falling-rate period (from C to D), and the second falling-rate period (from D to E).

Warm-Up Period

The warm-up period happened directly after the wet sheet touched the dryer hot surface. The sheet temperature started to increase quickly and the drying rate increased. This period was a transit segment, which prepared the equalization of the
mass-transfer rate to the heat-transfer rate. The most important change in this period was the increase in web temperature.

**Constant-Drying-Rate Period**

Section BC in Figure 1 represents a constant-drying-rate period. During this period the surface is covered with a continuous layer of free water. Drying mainly takes place by evaporation from the saturated surface. This involves diffusion of the water vapor through a stationary air film and into the bulk of the air. The movement of liquid is maintained by capillary force. It is also driven by other forces, such as liquid-solid matrix interfacial drag, Darcy resistance of liquid phase, the inertial force of the movement of the liquid film, viscous force, and gravity. At this point, internal moisture transfer to the surface and the evaporation at the surface are in equilibrium. Free water on the surface evaporates steadily and continuously. Therefore, the drying rate is determined by external conditions only, and a constant-drying-rate period is observed. The surface temperature is almost constant, and its value is very close to the wet-bulb temperature of the flowing air. The moisture content at the point between the end of constant drying rate period and the first falling are period is defined to be critical moisture content (CMC).

The following definitions are introduced:
Table 1
Definitions of Terms Used in the Mathematical Model to Describe Drying Behavior (Karlsson, 2000)

<table>
<thead>
<tr>
<th>Term</th>
<th>Symbol</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat of vaporization</td>
<td>$\Delta H_v$</td>
<td>KJ/Kg H$_2$O</td>
</tr>
<tr>
<td>Wrapping percentage</td>
<td>$\Phi_p$</td>
<td>%</td>
</tr>
<tr>
<td>Heat-transfer coefficient through condensate</td>
<td>$\alpha_s$</td>
<td>W/m$^2$.K</td>
</tr>
<tr>
<td>Thickness of dryer shell</td>
<td>$\delta_c$</td>
<td>m</td>
</tr>
<tr>
<td>Thermal conductivity of shell</td>
<td>$\lambda_c$</td>
<td>W/m.K</td>
</tr>
<tr>
<td>Fraction of heat flow from to air from steam</td>
<td>$a$</td>
<td></td>
</tr>
<tr>
<td>Heat-transfer coefficient from cylinder to web</td>
<td>$\alpha_k$</td>
<td>W/m$^2$.K</td>
</tr>
<tr>
<td>Length of drying cylinder</td>
<td>$L$</td>
<td>m</td>
</tr>
<tr>
<td>Cylinder diameter</td>
<td>$D$</td>
<td>m</td>
</tr>
<tr>
<td>Cylinder temperature</td>
<td>$T_s$</td>
<td>°F</td>
</tr>
<tr>
<td>Paper temperature</td>
<td>$T_p$</td>
<td>°F</td>
</tr>
<tr>
<td>Surface area of cylinder</td>
<td>$A$</td>
<td></td>
</tr>
<tr>
<td>Active cylinder surface</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Overall heat-transfer coefficient</td>
<td>$\alpha_s-p$</td>
<td>W/m$^2$.K</td>
</tr>
<tr>
<td>Heat flux through paper</td>
<td>$q_p$</td>
<td>W</td>
</tr>
<tr>
<td>Drying rate</td>
<td>$R_c$</td>
<td>Kg H$_2$O/s.m$^2$</td>
</tr>
</tbody>
</table>

The overall heat transfer coefficient through the condensed steam and the drier shell was developed according to equation 1.1 (Karlsson, 2000). This coefficient accounted for the amount of heat lost to the air from the condensed steam. Also it accounted to the wrapping percentage of the paper covers the drier cylinder. Multiplying this coefficient with the temperature difference between the steam temperature or the dryer temperature and the paper temperature will result in calculating the heat flux through the paper as shown in equation 1.2. Dividing the heat flux through the paper by the water heat of evaporation calculate the drying rate as shown in equation 1.3.
\[
\alpha_{s-p} = \left[\frac{1-a}{\Phi_p}\right] \times \left[\frac{1}{\frac{1}{\alpha_s} + \frac{\phi}{\lambda_c} + \frac{1-a}{\alpha_k \times \Phi_p}}\right]
\]

(Karlsson, 2000)

Heat Flux Through Paper = \left[\alpha_{s-p}(T_s - T_p)\right]

Drying Rate = \frac{\left[\text{Heat Flux through paper} \times \Phi_p \times A\right]}{\Delta H_v}

First Falling-Rate Period

When the free-water content is greater than the critical moisture content, the water phase is continuous. As long as the free-water content at the surface is less than the critical, the surface will form discontinuous wet patches. As the drying proceeds, wet areas on the paper surface decrease with decreasing surface-moisture content. Thus, the mass-transfer coefficient decreases with the surface free-water content, and the first falling-rate period starts. In the first falling-rate period, a new energy balance will be reached at the surface, accompanied by slowly rising surface temperature. Free water still exists at the surface, the dry patches still contain bound water, and the vapor pressure at the surface is determined by the Clapeyron equation No. 1.4.

Clapeyron Equation

Clapeyron equation is given by equation 1.4; it explains the coexistence of vapor and liquid phase coexist during drying in the first drying rate period.
\[ dG = Vdp - SdT \] (Salzman, W., (2005))

where

G: Gibbs free energy
V: Volume
P: Pressure
S: Entropy
T: temperature

Two phases in equilibrium at constant pressure and temperature have the same Gibbs's free energy so

\[ \Delta G = 0 \] \hspace{1cm} 1.4a

Or

\[ G_1 = G_2 \] \hspace{1cm} 1.4b

\[ dG_1 = dG_2 \] \hspace{1cm} 1.4c

By substituting \( dG \) from equation 1.4 into 1.4c

\[ V_1 dp - S_1 dT = V_2 dp - S_2 dT \] \hspace{1cm} 1.4d

Rearranging

\[ \left( \frac{dp}{dT} \right) = \left( \frac{\Delta S}{\Delta V} \right) \] \hspace{1cm} 1.5

For a phase transition occurring at constant temperature and pressure, the definition of entropy is:

\[ dS = \frac{dq}{T} \] \hspace{1cm} 1.5a

Implies that
The Clapeyron equation is valid along any phase co-existence line.

Clausius-Clapeyron Equation

When the Clapeyron equation is applied to liquid-vapor phase co-existence, several simplifying assumptions can be made. First, since the volume of a gas is much greater than the volume of a condensed phase:

$$\Delta V = V_{\text{gas}} - V_{\text{liquid}} \approx V_{\text{gas}}$$  \hspace{1cm} 1.6.a

Second, the gas is assumed to obey the ideal gas law:

$$V_{\text{gas}} = \frac{nRT}{p}$$  \hspace{1cm} 1.7

This allows the Clapeyron equation to be simplified as follows:

$$\frac{dp}{dT} = \frac{p\Delta H}{nRT^2}$$  \hspace{1cm} 1.8

$$\frac{dp}{p} = \frac{\Delta H}{nR} \frac{dT}{T^2}$$  \hspace{1cm} 1.8a

Third, $\Delta H$ is assumed to be independent of temperature and pressure, allowing indefinite integration over $p$ and $T$: 

$$\Delta S = \frac{\Delta H}{T}$$  \hspace{1cm} 1.5.b

Thus,

$$\frac{dp}{dT} = \frac{\Delta H}{T \Delta V}$$  \hspace{1cm} 1.6
\[ \ln p = \frac{\Delta H}{nR \left( \frac{-1}{T} \right)} + \text{constant} \]  

Choosing the constant of integration to equal \( \ln p_0 \), where \( p_0 \) equals one pressure unit, e.g., 1 Torr if pressure is measured in Torr, allows the pressure units to cancel and yields:

\[ \ln \left( \frac{p}{p_0} \right) = -\frac{\Delta H}{nRT} \]  

This equation is useful for determining \( \Delta H \) from a plot of \( \ln (p/p_0) \) vs. \( 1/T \).

If definite integration from \( p_1 \) to \( p_2 \) and from \( T_1 \) to \( T_2 \) is performed, the result is:

\[ \ln \left( \frac{p_2}{p_1} \right) = -\frac{\Delta H}{nR} \left( \frac{1}{T_2} - \frac{1}{T_1} \right) \]  

This equation is useful for determining the vapor pressure \( p_2 \) at temperature \( T_2 \) given \( \Delta H \) and the vapor pressure at one temperature (\( p_1, T_1 \)), e.g., the normal boiling point.

The drying rate in the first is defined mathematically by:

\[ R = -\frac{L_s \times dX}{A} \]  

\[ R = R_c \times \frac{X}{X_c} \]  

\[ t = \frac{L_s \times X_c}{A \times R_c} \times \ln \frac{X_c}{X_2} \]  

\[ L_s = x_1 \times A \times \rho_s = \text{Solids wt} \]
$X$ : Moisture Content at any time
$X_c$ : Critical Moisture Content (CMC)
$A$ : Drying Area
$t$ : time
$p_s$ : Cellulose Density 1550 kg/m$^3$

$$t = \frac{L_s \times X_c}{A \times R_c} \ln \frac{X_c}{X}$$  \hspace{1cm} 1.15

$$t = \frac{x_1 \times p_s \times X_c}{R_c} \ln \frac{X_c}{X}$$  \hspace{1cm} 1.16

$R_c$ : Drying Rate at $X_c$

Second Falling Zone

Capillarity is the ability of a narrow tube to draw a liquid upwards against the force of gravity. It occurs when the adhesive intermolecular forces between the liquid and a solid are stronger than the cohesive intermolecular forces within the liquid. The effect causes a concave meniscus to form where the liquid is in contact with a vertical surface (2006 Capillary action on Wikipedia). Capillary forces are the controlling factor that supplies water to the sheet surface where it evaporates.

Surface tension pulls the liquid column up until there is a sufficient weight of liquid for gravitational forces to overcome the intermolecular forces. The weight of the liquid column is proportional to the square of the tube's diameter, but the contact area between the liquid and the tube is proportional only to the diameter of the tube, so a narrow tube will draw a liquid column higher than a wide tube.

First: Mass-flow rate:

Capillary flow is described by Darcy’s law:
\[ q = -\left( \frac{k \times A}{\mu} \right) \frac{\Delta P}{\Delta L} \]  

1.17

\[ q : \text{Volumetric Flow Rate in The Capillary} \]

\[ k : \text{Permeability. darcy.} \ \frac{cm^2 \times cp}{s \times atm} \]

\[ A : \text{Area of Capillary Tube} \]

\[ q : \text{Flow Rate} \]

\[ \Delta P : \text{Pressure Drop} \]

\[ \Delta L : \text{Length of Capillary Tube} \]

\[ \mu : \text{Viscosity} \]

From Young-Laplace equation:

\[ \Delta P = \gamma \left( \frac{2}{D} + \frac{2}{D} \right) \]  

1.18

\[ \Delta P = \frac{4 \times \gamma}{D} \]  

1.19

\[ D : \text{Capillary diameter} \]

By substituting Young-Laplace in Darcy we get:

\[ q = -\left( \frac{4 \times A \times \gamma}{\mu} \right) \left( \frac{k}{D \times \Delta L} \right) \]  

1.20

Multiplying by the water viscosity we get:

\[ q \times \rho = -\left( \frac{4 \times A \times \gamma}{\mu} \right) \left( \frac{k}{D \times \Delta L} \right) \times \rho \]  

1.21

**Mass flow rate:**

\[ \frac{dQ}{dt} = h_s \times A \times (T_a - T_s) \]  

1.22

**Second: Heat-flow rate:**
\[
\frac{dQ}{dt} = \text{Heat Transfer Rate} \ [J / s]
\]

\( h_s \) : Heat Transfer Coefficient \([J / m^2 \cdot s \cdot ^\circ C]\)

\( A \) : Area

\( T_a - T_s \) : Air temp – Surface temp

Latent heat of vaporization of water is about 2260 kJ/kg.

By dividing equation 1.23 by the latent heat of vaporization, the rate of water evaporated because of heat transfer is found.

This magnitude should be greater than the mass-flow rate from equation 1.21, since it is assumed that all evaporation happens on the surface. All the water supplied to the surface by the capillary should evaporate once it reaches the surface. The rest of the energy will be either lost to the surrounding air or used to heat the sheet and the rest of the moisture within the sheet.

**Water Permeability Importance in Drying**

Water permeability of paper, or Darcian permeability, is one important factor for processes involving fluid flow through fibrous webs by affecting the product behavior under manufacturing processes such as infiltration, rewet, wicking, and drying. Permeability controls the amount of vapor and liquid flow through the paper during the pressing process (Nilsson & Larsson, 1968; Asklof, Larsson, Linderoth, & Wallstrom, 1964; Yih & McNamara, 1964; Lindsay, 1992).
Sheet permeability is affected by many factors, including refining level, yield, fines content, pH, and sheet formation. For example, Carlsson (1983); Carlsson, Lindstrom, and Floren (1983); and Ellis (1981) found that permeability decreased with increased refining; high-freeness pulp tended to have high permeability. Ellis found that sheets formed at low consistencies had lower permeability than sheets formed at higher consistencies. Gren (1972) examined the effect of yield on permeability; his finding was that a higher yield resulted in increased water permeability. Poppel and Lad (1977) examined the effect of chemical additives, refining, and composition on mixtures of spruce and beech pulps. They found that bleached pulps had four to five times the permeability of spruce pulps at identical freeness values.

Park, Venditti, Jameel, and Pawlak (2005) studied the drying behavior of softwood bleached kraft market pulp. They introduced the definition of hard-to-remove water (HRW) as the moisture ratio (g of water/ g of dry fiber) at the transition between the constant-drying period and a single falling-rate period. They studied the effect of fractionation on HRW and water retention value (WRV). They concluded that because fractionation causes loss of fines, it lowers both the HRW and the WRV. Their Thermogravimetric Analyze (TGA) drying curve included three periods: a warm-up period, a constant-drying period, and a single falling-rate period. They were not able to resolve the HRW into the two regions HRW[I] and HRW[II].

Cellulose fibers formed the paper-network structure, which could be described by different parameters such as continuity and tortuosity of the flow paths, formation,
pore volume, pore-size distribution, fiber thickness, and fiber conformability. Fiber length and fiber coarseness were often used as major parameters in modeling-paper formation, paper drying, and paper’s physical properties. The liquid water, water vapor, and volume fractions of the different components (fiber, liquid water, and water vapor) affected mass and heat transfer during drying (Karlsson, 2000).

Two parameters that help determine paper’s strength are the relative bonding area (RBA) and the fiber-fiber bond strength. The RBA is the ratio of the bonded area to the total internal area in the sheet. The bond strength is increased by external fibrillation. The RBA is increased by internal fibrillation, which increases the fiber’s flexibility. The fiber cross-sectional dimensions affect the collapse of fibers during production and hence water-retention ability. Wet fibers can collapse into flattened ribbons during paper production. Studies of pulps from softwoods have shown a linear relationship between fiber collapsibility and fiber-wall thickness. Thicker-walled fibers are stiffer and resist flattening during pressing and drying. Thinner fibers flatten during pressing and drying, which increases the sheet density and reduces the number and size of the voids. Recycling reduces fiber fibrillation, which in turn reduces the RBA.

The paper-shell contact coefficient (the contact between the paper and the metallic drying surface) was the most critical factor affecting the heat-transfer resistance between the shell and the paper (Cameron & Zwick, 2003). The contact coefficient depended on the water/solids ratio and decreased as the moisture ratio decreased. The literature has shown conflicting results on the effect of surface
temperature on the contact coefficient. For example, Wilhelmesson, Fagerholm, and Strenstom (1994) found no relation between the surface temperature and the contact coefficient, and Redfern (1963) reported that the contact coefficient increased with increasing surface temperature. In addition, Meinecke, Chau Huu, and Loser (1988) found that the contact coefficient decreased with increasing surface temperature.

Horn and Setterholm (1990) found that the majority of the variation in the strength properties of paper, such as burst strength and tensile strength, could be related to fiber length and cell-wall thickness, and that these properties were highly dependent upon fiber-to-fiber bonding. Generally, bursting and tensile strength of paper made from softwoods responded to the same fiber characteristics as hardwoods.

The adsorption isotherms related the surrounding relative humidity to the equilibrium moisture content. Paper was a hygroscopic material by nature. The hydroxyl (OH) groups on the cellulose surface had a great affinity for water. The binding of water to the fiber was confirmed to be a hydrogen bonding. The analysis was done by comparing the FTIR spectra of dry cellulose to wet cellulose (Clark, 1985).

The percentage of absorbed water depended on the fraction of non-crystalline cellulose because water could not permeate the cellulose crystalline fibers. When cellulose or other porous hygroscopic materials are dried, the rate of drying fall when the moisture content reached about 12%. This indicated the increase in bonding between the remaining water and the fiber (Ayers, 1956). Moisture hysteresis is the phenomena of having higher moisture content when decreasing the relative humidity.
to a certain point, then increasing the relative humidity to the same point. Absorbed water expands the structure of fibers and increases the internal surfaces to accept additional water that will be bonded by hydrogen bond.

Norminen (2006) studied the effects of refining levels on paper properties. He found that refining conditions had the greatest effect on the pulp properties. The fiber length was set between 0.6 and 1.1 mm by controlling the rotation speed, plate type, and refining mode. He reported that the lowest refining level and the highest fiber length resulted in the highest tensile index. The lowest refining intensity at the highest fiber length resulted in reduced fiber-bonding properties but improved the bonding properties of the shorter fibers' fractions and fines.

Rewatkar and Masliyah (1997) developed a process to fractionate fibers with minimum rotation speed in a rotating-cone fractionator (RCF). This RCF was constructed with a rough surface of sieve mesh, and a fiber suspension was fed onto the RCF's rotating conical surface. The effects of surface roughness were assessed using 12-, 20-, and 32-mesh screens. Fiber fractionation performance was assessed by measurements of fiber length. The data indicated that the flow characteristics were dependent on surface roughness, rotation speed, and feed-flow rate. "It was concluded that a rotating cone with a rough surface (20- and 32-mesh) provided better fiber fractionation at lower rotational speed and higher feed flow than a rotating cone with a smooth surface." Page 196-204.

In experiments carried out on old corrugated boxes, Waterhouse and Liang (1995) used fractionation and sodium hydroxide treatment to reverse the adverse
effects of drying on the fines’ fraction in recycled-fiber pulps. Fractionation was used to separate out the fine fraction, then the researchers treated the pulp with sodium hydroxide (NaOH). Finally, they evaluated properties of blends of treated fines and the OCC long-fiber fraction. They reported that:

NaOH treatment of OCC fines had a significant effect on pulp CSF, elastic, and strength properties. At 10% NaOH, freeness was almost equal to that of the unrefined long-fiber fraction. Water removal by wet-pressing also appeared to be independent of fines and NaOH levels. At both 15% and 30% fines addition, the in-plane elastic constant increased initially, reaching a maximum at approx. 1-2% NaOH, after which it decreased to just below the level of 0% NaOH treatment. (p. 103-116)

Poirier and Pikulik (1997) investigated the effects of various drying conditions, particularly drying temperature, on paper properties. They collected different paper samples like newsprint, fine papers, and LWC papers from the press section, the dryer section, and the calender stack of several commercial paper machines. Lab pressing and/or drying at room temperature was applied to some of the samples. Other samples were dried in a conventional cylinder dryer with surface temperatures from 60 °C to more than 100 °C, or in a hot-plate dryer at temperatures ranging from 80 °C to 140 °C. In a separate study, sheets were impulse-dried at 30 °C, 165 °C, and 190 °C; dried in a bench-top dryer; and calendered. Poirier and Pikulik found that:

Samples were characterized for brightness, light-scattering coefficient, and light-absorption coefficient. Results indicated that sheets containing a large fraction of mechanical pulp showed decreasing light-scattering coefficient and brightness with increasing drying temperature. This was attributed to the increased relative bonded area of the fibers and thermal plasticization of the wood polymers, which improved strength properties. Drying temperature had no significant effect on the optical properties of sheets containing mostly chemical pulp. (p. 1869–1879)
In a related study, Retulainen and Nieminen (1992) found that the addition of fines increased drying stress and tensile stiffness more than did starch addition.

Przybysz and Czechowski (1980) studied the mechanism of paper web drying, specifically web shrinkage during drying and the role of fines. They reported that “the removal of fines lowered the dryness and the shrinkage. Increasing web tension increased dryness values and diminished fines.” (p 83-87).

Krgovic (2004) found that significant loss of heat energy during paper drying was due to unconsidered factors in the heat-transmission phenomena. The drying sieve’s influence (including sieve weaving, tension force, and pressure rate between paper and cylinder) on heat transmission and drying rate was one of the main factors. Another factor was the influence of air temperature and speed over the efficiency of tissue-paper drying. It was determined that water evaporation may be increased by higher air temperature and speed in the drying box.

Poirier (1992) studied the properties of drying paper using superheated steam and hot air. Drying TMP sheets by superheated steam improved strength properties like burst index and tensile index, which were related to increased bonded area and decreased brightness. Kraft-pulp strength properties decreased and optical properties increased when compared to the properties of sheets dried in air. These decreased strength properties were attributed to a thermally induced drying-stress relaxation.

Ceragioli (1984) studied the effect of previously dried fiber on paper properties. He used bleached kraft long-fiber pulp from softwood and bleached kraft
short-fiber pulp from hardwood. His data showed that drying refined or unrefined pulp caused changes in the fiber structure when compared to never-dried pulp. Dried pulp had an a less swelling capacity or WRV, lower apparent density, lower tensile and bending strengths, increased air permeability, and improved opacity. It also appeared to be more fragile as shown by the decrease in the long-fiber fraction.

Ceragioli (1984) found that “the hardwood pulp showed decrease in tear strength, whereas the softwood pulp showed increase in tear strength. This opposite tear strength behavior may be due to the opposite behavior of these pulps as beating progresses.” (p. 171-179). The extent of disintegration produced a denser sheet with increased strength, the bending strength of long-fiber pulps was increased, and the tear strength of short-fiber pulps was also increased. The extent of disintegration of dried pulp caused an increase in the drainage index, especially for long-fiber pulps; fiber swelling capacity increased moderately.

Nordman, Hirvonen, Levlin, and Ebeling (1980) studied the influence of drying temperature on paper properties, and they developed mathematical models presenting the changes in optical, strength, and surface properties as a function of drying temperature. The most significant effect of temperature was observed for mechanical properties. Optical properties were also affected significantly by high drying temperatures. This effect was due to the softening of fibers containing different amounts of hemicelluloses and lignin. The researchers found that the increase in temperature for sheets with moisture content below 30% was insignificant.
CHAPTER II

EXPERIMENTAL DESCRIPTION

Source of Fibers

The fibers used in this research were southern softwood lap pulp, never-dried hardwood, and recycled fiber from old corrugated cardboard (OCC). The softwood fibers were taken from the pilot plant at Western Michigan University (WMU), the recycled fibers were obtained from the recycling room at WMU's Parkview campus, and the never-dried hardwood was acquired from International Paper in Michigan's Upper Peninsula. A species identification of the used fibers showed that the softwood fibers were pine, the hardwood fibers were maple and spruce, and the recycled fibers were mixed. The recycled fibers were re-pulped and screened using a 200 mesh to remove any fillers and fines before being evaluated by a fiber quality analyzer (FQA\(^1\)), which determined that 35% of the recycled fibers were softwood and 65% were hardwood.

Design of Experiments

Stategraphics Plus 5.1\(^2\) statistical analysis software was used to design the

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\(^1\) Fiber Quality Analyzer (FQA) model LDA02 manufactured by OpTest Equipment, Inc.
\(^2\) Stategraphics Plus version 5.1 from StatPoint, Inc.
experiments. The factors considered in this design were the basis weight, refining level, fiber length, and recycling. Two levels were tested: 1) 60 gsm, which is the bases weight of most writing paper and 2) 120 gsm, which is the bases weight of paperboard. The 60-gsm level was considered low and was denoted by -1, and the 120-gsm level was considered high and was denoted by +1. Two levels of refining were applied: 1) a low level (no refining), denoted by -1 and 2) a high level (in which the fibers were refined to 400 Canadian standard freeness (CSF)), denoted by +1. Three recycled cycles were applied: 1) a low level (no recycling), denoted by -1; 2) a first recycling cycle; denoted by 0; and 3) a second, high level of recycling, denoted by 1. Two moisture contents were tested: 1) a low level of 1 g-water/g-fiber, denoted by -1 and 2) a high level of 2 g-water/g-fiber, denoted by +1. Since the fractionation process produced four different fractions based on mean fiber length, the experiments contained four levels of fiber lengths. Each fiber fraction was named after the number of the mesh used to collect the corresponding fibers, and the levels were arranged from the longest fiber to the shortest fiber, in this order: mesh 14, mesh 30, mesh 50 and mesh 100.

Table 2 shows factors used in the experiments’ design, and Table 3 shows actual values of the levels used in the experiments’ design. Tables 4 and 5 show factors used in the experiments’ design for hardwood fibers and the actual values of levels used in the experiments’ design.
Table 2
Factors Used in the Experiments’ Design for Virgin Fibers

<table>
<thead>
<tr>
<th>Factor</th>
<th>Unit</th>
<th>Low factor</th>
<th>High factor</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Refining Level</td>
<td>CSF</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Recycling</td>
<td>Number of cycles</td>
<td>-1</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Moisture Content</td>
<td>g-water/g-fiber</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 3
Factors Used in the Experiments’ Design for Recycled Fibers

<table>
<thead>
<tr>
<th>Factor</th>
<th>Unit</th>
<th>Low factor</th>
<th>High Factor</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Refining Level</td>
<td>Refining Time</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Recycling</td>
<td>Number of Cycles</td>
<td>-1</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Moisture Content</td>
<td>g-water/g-fiber</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>
### Table 4
Factors Used in the Experiments' Design for Hardwood Fibers

<table>
<thead>
<tr>
<th>Factor</th>
<th>Unit</th>
<th>Low factor</th>
<th>High factor</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basis Weight</td>
<td>gsm</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Refining Level</td>
<td>CSF</td>
<td>-1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Recycling</td>
<td>Number of cycles</td>
<td>-1</td>
<td>1</td>
<td>3</td>
</tr>
</tbody>
</table>

### Table 5
Actual Values of Levels Used in the Experiments' Design

<table>
<thead>
<tr>
<th>Basis Weight, 60 gsm</th>
<th>120 gsm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content</td>
<td></td>
</tr>
<tr>
<td>1 g-water/g-fiber</td>
<td>2 g-water/g-fiber</td>
</tr>
<tr>
<td>Refining Level</td>
<td>Unrefined</td>
</tr>
<tr>
<td>Recycling</td>
<td>No Recycling</td>
</tr>
</tbody>
</table>

**Sample Preparation**

The experimental work was divided into five main categories: 1) fiber fractionation by a Clark classifier, 2) fiber characterization by an FQA, 3) handsheet-making by Noble and Wood’s machine, 4) drying measurements using a drying simulator and a thermogravimetric analyzer (TGA), and 5) paper characterization.

The first step in sample preparation was to re-pulp the fibers using a British disintegrator, shown below in Figure 2. Thirty grams of oven-dry pulp was mixed in three liters of water and disintegrated in each batch. After disintegration, a Mead refiner, shown in Figure 3 below, or PFI type refiner, shown in Figure 4 below,
was used to refine the pulp to 400 CSF. The refining level was measured according to TAPPI standard T 227 (TAPPI, 2004b) using a CSF tester such as the one shown in Figure 5. After that the Clark classifier, shown in Figure 6, was employed to fractionate the fiber into four fractions. The fractionator in the classifier was equipped with screen meshes of numbers 14, 30, 50, and 100. TAPPI standard T 233 (TAPPI, 2004c) was followed. The fractionation process was repeated until enough quantity of each fraction was collected. The fiber length was measured using an FQA, model LDA02, shown in Figure 7. Then handsheets with the specific basis weight were prepared using Nobel and Wood’s machine, shown in Figure 8. TAPPI T 205 (TAPPI, 2004a) and T 272 (TAPPI, 2004d) standards were followed in producing the handsheets. Formed samples were pressed and saved for the drying experiments.

Figure 2. Laboratory British Pulp Disintegrator Used to Repulp Dried Fibers
Figure 3. Laboratory Mead Refiner Used to Refine the Pulp

Figure 4. Laboratory PFI Refiner Used to Refine the Pulp
Figure 5. Canadian Standard Freeness Tester Used to Measure Refining Level

Figure 6. Clark Classifier Used to Fractionate the Fiber
Figure 7. Fiber Quality Analyzer Used to Characterize the Fibers

Figure 8. Nobel and Wood’s Machine Used to Make the Handsheets

The sample preparation and measurement schematic diagram is shown in Figure 9 below.
Methods of Characterization

Drying Behavior

Drying rates and surface contact coefficients were measured using the drying laboratory apparatus designed by Dr. John Cameron (Cameron & Zwick, 2003). A schematic diagram is shown in Figure 10; see Appendix A for more details. The basic principle of this measurement is to dry a known area of the wet paper sheet to a hot surface.
The apparatus was designed to simulate a commercial drying cylinder with a 3-ft. radius and about half an inch thick (1.27 cm). The tension on a commercial fabric dryer is in the range of 1.5 to 2.5 kN/m. The felt tension of the laboratory apparatus was about 1.1 kN/m which is in the recommended TAPPI range for newsprint machines (0.5 kN/m to 1.7 kN/m) (TAPPI, 1989).

![Diagram of drying set-up](image)

Figure 10. Drying Set-Up Used for Measuring Drying Behavior (TAPPI, 1989)

The drying surface was preheated to 125 °C using a heating pad. The heating was then stopped and the heater side temperature decreased to 120 °C, which resulted in both sides of the drying surface having the same temperature. The temperatures of the heater side and the shell side were measured using type E thermocouples. The thermocouples were connected to a data-acquisition board manufactured by Computer Measurements. The data stored in the system were analyzed using Visual Basic modules, and Microsoft Excel 11 was used to calculate and display the drying rates and heat fluxes.
In separate experiments, southern softwood virgin fiber and recycled fiber were refined to 400 CSF using a Mead refiner. A Clark classifier was used to fractionate the pulp into four fractions of mesh numbers 14, 30, 50, and 100. Fiber length was measured using the FQA. Handsheets were prepared using a Nobel and Woods handsheet machine. Different levels of fiber coarseness were used to produce single-layer handsheets with a base weight of 60 gsm. The drying behaviors of the handsheets were tested using drying equipment and a thermogravimetric analyzer (TGA)\(^3\), shown in Figure 11. More details about the TGA are in Appendix B.

Figure 11. Schematic Diagram of TGA

\(^3\) Thermogravimetric Analyzer (TGA) model Q500 manufactured by Texas Instrument, Inc.
A TGA system (Q500) was also used to study the drying behavior of the virgin and recycled fiber with a heating rate of 10 °C/min until 90 °C was reached. Then the drying procedure was run isothermally at 90 °C for 30 minutes. Nitrogen gas was used at a flow rate of 40 ml/min for balance gas and 60 ml/min for sample gas. The weight change in time was recorded. The data were further processed using TA Instruments Universal Analysis 2000 V4.2E software. A mercury intrusion porosimeter\(^4\) was used to measure the pore-size distribution of the handsheets; see Appendix C. The WRV were measured based on TAPPI useful method number UM 256 with modified rotational speed of 3000 rpm.

The adsorption isotherms of the sheets made of fractionated fibers were studied using a CARON 6030 environmental chamber\(^5\). The isotherms were constructed at 25 °C, 35 °C, and 60 °C, and with a relative humidity starting at 30%, going to 90%, and then going back to 30%. Sample weight was recorded as a function of time at each temperature and relative humidity until a constant weight was obtained before moving to the next point.

**Mechanical Properties**

A tensile test was carried out according to TAPPI standard T 494 (TAPPI, 2004f) using an Instron tensile machine\(^6\). A tear test was carried out according to TAPPI standard T 414 (TAPPI, 2004e).

\(^4\) Mercury Porosimetry AutoPore model IV 9500 manufactured by Micromeritics.
\(^5\) Humidity Chamber Model 6030 by CARON Products & Services, Inc.
\(^6\) Instron model number 2501 by Instron Corporation.
Physical Properties

A Parker Print Surf (PPS)\(^7\) was used to measure smoothness and porosity according to TAPPI standard T 555 – om 99 (TAPPI, 2004g). The sheets were measured at a pressure level of 500 kPa with soft packing. The mean value of 20 different readings was reported.

\(^7\) Parker Print Surf Model 90 by Messmer Instrument Ltd., UK.
CHAPTER III

RESULTS AND ANALYSIS

Fiber Fractionation and Characterizations

**Softwood and Recycled Fibers**

A comparison of the fractionation of virgin and recycled fibers is shown in Figure 12. The virgin fibers tended to be longer and coarser. The level of virgin fibers collected in mesh 14 was higher than that of recycled fibers collected with the same mesh. Almost the same fiber percentage was collected in mesh 30, 50, 100, and 200 (less virgin fiber was lost). Since the percentage of lost recycled fiber (about 20%) was higher than lost virgin fiber (about 5%), the lost fibers can be considered fillers or fines that passed mesh 200. Such a result was expected because of the use of corrugated boxes that were recycled an unknown number of times and/or refined at unknown levels.

Analysis of the fiber lengths of each fraction, shown in Figure 13, indicated that the recycled fibers were about 0.5 mm shorter than the virgin fibers. Fiber lengths were in the range of 0.9 mm to 3.0 mm for the virgin fibers and from 0.6 mm to 2.5 mm for the recycled fibers.
Fractionation with Clark classifier produced populations of fibers with different mean fiber lengths and coarseness values. The shorter fibers were thinner and thus had lower coarseness values compared to the longer fibers. Mesh 14 collected about 40% of the original fiber, with a mean fiber length of approximately
2.75 mm and a coarseness of 0.5 mg/m. Fewer fibers were collected at higher mesh numbers, and these fiber fractions contained shorter mean fiber lengths and lower coarsenesses values, as shown in Figure 14.

![Figure 14. Fiber Coarseness for Each Fraction. The Fiber Coarseness Decreased with Increasing Mesh Number or Decreasing Fiber Length](image)

The coarseness values of the softwood fractionated fibers were in the range of 0.17 to 0.54 mg/m, as shown in Figure 14. It was found that the tensile strength increased from 1.35 to 1.55 kN/m with decreasing coarseness values from 0.54 to 0.16 mg/m. The lower-coarseness fibers consisted of fibers with thinner walls, which produced stronger sheets, as shown in Figure 15.
Figure 15. Relationship Between Fiber Length and Fiber Coarseness

Figure 16 shows the relationship between the coarseness values of the fiber and the tensile strength of the handsheet made of that fiber. Handsheets made of the thinner, shorter fibers had the highest tensile strength of 1.55 KN/m. The thicker and coarser the fiber, the lower the tensile strength of the handsheets. Fibers of 0.2 mg/mm coarseness resulted in handsheets with 1.37 KN/m tensile strength, and fibers of 0.55 mg/mm coarseness made 1.35 KN/m tensile strength handsheets.

The break length was calculated for the handsheets made of softwood-fractionated fibers. The shorter, thinner fibers had the highest break length of about 2650 meters. The longer, thicker fibers had the lowest break length of about 2300 meters. As the fibers became shorter and thinner, break length got higher. This is shown in Figure 17.
Figure 16. Effect of Coarseness on Sheet Tensile Strength

Figure 17. Breaking Length of Fractionated Softwood Fibers as a Function of Their Fiber Lengths
Thicker fibers or higher-coarseness fibers had higher porosity and roughness, as shown in Figures 18 and 19, porosity and roughness was measured using parker print surf as described in chapter II under physical properties. The changes in the physical properties of each fraction were also expected to affect the drying behavior of each fraction since the increase in roughness and porosity increased the area exposed to drying and introduced a higher void volume for water.

![Graph](image_url)

Figure 18. Effect of Fiber Coarseness on Sheet Roughness Measured Using Parker Surf Machine

The effect of refining on unfractionated softwood fiber lengths is shown in Figure 20. Refining reduced the fiber lengths, and increasing the refining level (adding more time) further reduced fiber length. The fiber length decreased rapidly during the first 40 seconds, then leveled in the next 60 seconds; after that the fiber length decreased rapidly again.
Figure 19. Effect of Fiber Coarseness on Sheet Parker Print Porosity

Figure 20. The Effects of Refining on Unfractionated Softwood Fiber Length
As mentioned earlier, refining reduced fiber length due to the fiber cutting and fines generation. Refining also resulted in opening the fiber structure through internal and external fibrillation. These changes in the fiber structure had other effects on the drainage ability of the fiber. The drainage ability, which is the fiber’s ability to lose water, decreased with more refining. As the refining time increased, the degree of CSF decreased. Figure 21 shows the relationship between refining time, using Mead refiner to refine softwood fibers, and the degree of freeness. As the refining time increased, the degree of freedom and the drainage decreased.

![Figure 21. The Effect of Refining of Softwood Using Mead Refiner on Drainage or Canadian Standard Freeness (CSF)](image)

**Never Dried Hardwood Fibers**

On the other hand, when the never-dried hardwood fibers were fractionated, fibers were collected only in mesh 50 and mesh 100. About 15% of the unrefined
hardwood fibers were collected in mesh 50, 15% in mesh 100, and 15% in mesh 200. After refining, 45% were collected in mesh 50, 15% in mesh 100, and 10% in mesh 200, as shown in Figure 22.

![Graph showing percentage of collected hardwood in mesh 50, mesh 100, and mesh 200 before and after refining.](image)

**Figure 22.** Percentage of Collected Hardwood in Mesh 50, Mesh 100 and Mesh 200 Before and After Refining

![Graph showing fiber length before and after refining.](image)

**Figure 23.** Never Dried Fiber Length Before and After Refining

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A comparison between the refined and unrefined fractionated hardwood fiber lengths is shown in Figure 23. The average unrefined fiber length collected in mesh 50 was 0.65 mm. Refining increased this value to 0.75 mm. Mesh-100 unrefined mean fiber length was 0.5 mm; this value increased to 0.6 mm after refining. Although a reduction in the fiber length as a result of fractionation was expected, in this case an increase was noticed. This was due to the effect of refining on opening the structure of the fibers and the removal of fines through the fractionation process. The presence of fines would have decreased the measured average fiber length.

The effect of refining on the drainage ability of hardwood is shown below in Figure 24. The hardwood was refined using a PFI refiner, and its drainage ability was decreased from 700 ml (CSF) to about 400 ml (CSF) after 3000 revolutions. As was concluded from the measured fiber lengths, this was enough to open the fibers’ structure without cutting them. Fines produced due to the refining were either collected in or passed through mesh 200. It is easier to control the effect of PFI refiner over mead refiner especially when shorter fibers need to be refined.

**Drying Behavior**

The drying curves for the fractionated handsheets, shown in Figures 25 through 28, were divided into four periods, marked with numbers on the figures: 1) sheet warm-up, 2) constant-drying rate, 3) first falling rate, and 4) second falling rate. Each period represented a different drying behavior in the drying process. The first period, warm-up, which occurred directly after the contact between the drying
cylinder and the wet sheet, had the highest heat-transfer rate. Here there was a rapid increase in the temperature of the wet sheet and a decrease in the drying rate until

Figure 24. The Effect of Refining on Hardwood Drainage

equilibrium is reached, at which point the drying moved into the second period. In the second drying period, a constant heat-transfer rate was observed. The sheet had an even distribution of moisture, and the evaporation rate was constant. The capillaries supplied water to the surface at the same rate the water evaporated. Once the capillaries' ability to supply water to the surface decreased, the drying rate started to decrease. The moisture content at this point was called critical, and a new drying period occurred in which the paper temperature started to increase, moisture was distributed unevenly on the sheet, and dry patches appeared. The drying in this period occurred in the large capillaries. This stage continued until the water in the large
capillaries had dried; then the resistance increased and the temperature of the paper increased rapidly. At this stage the last period of drying started, the water evaporated from the fine capillaries, and the chemically bound water was released and evaporated.

![Figure 25. Drying Rate as a Function of Time for Fiber Separated in Mesh 14](image)

Analysis of Figure 25 through 28 shows that shorter fibers have less surface water and higher HRW[II] as will be seen later.

The drying behavior of the different fiber fractions, followed by the TGA, indicated two distinct falling-rate periods, as shown in Figure 29. This experiment was repeated with different initial moisture levels, and results were similar, with two falling-rate periods always occurring. The drying behavior and water-retention value of two additional recycling stages of the fractionated fiber were also determined. The
Figure 26. Drying Rate as a Function of Time for Fiber Separated in Mesh 30

Figure 27. Drying Rate as a Function of Time for Fiber Separated in Mesh 50
ratio of the moisture content per oven-dry fiber (unit mass) was used to describe the two falling-rate periods. The first point, the moisture ratio at the boundary between the constant-drying period and the first falling-rate period, was the basis for the hard-to-remove water total (HRW[T]). The hard-to-remove water one (HRW[I]) was the water removed during the first falling-rate period. The second point was the moisture ratio at the boundary between the first falling-rate period and the second falling-rate period. The hard-to-remove water two (HRW[II]) was the water removed from the second falling-rate period to complete dryness. This point included water in the fine capillaries and chemically bound water.
Figure 30 shows that handsheets made with coarser fibers had larger pore volume, while shorter, thinner fibers produced finer pores. Figure 31 shows the relation

![Drying Curve Using the TGA. The Four Drying Stages Are Shown With the First and Second Falling Rates Clearly Differentiated](image)

between water-retention value (WRV) and fiber coarseness. WRV increased as the fiber coarseness decreased. Recycling reduced the water-retention values of all fractions and further recycling reduced WRV even more. The shorter fibers were the most affected by recycling.

Figure 32 shows the relationship between HRW(T) and fiber length. HRW(T) increased as the fiber became shorter and finer. Recycling reduced the HRW(T) for
the shorter, finer fibers, but had little effect on the HRW(T) of the longer, thicker fibers. Again, further recycling reduced the HRW(II) more.

Figure 30. The Average Pore Diameter of the Handsheets Made of Fractionated Fibers Measured Using Mercury-Intrusion Porosimetry

Figure 33 shows only the water removed in the first falling-rate period (HRW[I]), unlike Figure 32, which shows (HRW[T]); (HRW[T] = HRW[I] +HRW[II]). Most of HRW[T] was removed as HRW[I] in the first falling-rate period, as shown in Figure 33. Recycling reduced the amount of water removed during the first falling-rate period and increased the HRW[II] as shown in Figures 33 and 34. The HRW[II] increased as the fibers became shorter and thinner. Recycling increased the HRW[II] for the shorter fibers but had little effect on the longer, thicker fibers, as shown in Figure 34.
Another way of presenting the data shown in Figures 32 to 34 is to represent the percentage of HRW(I) to HRW(T), as shown in Figure 35, and the percentage of HRW(II) to HRW(T), as shown in Figure 36. Between 85% and 95% of the HRW(T) was removed as HRW[I]. The percentage of HRW[I] to HRW[T] increased as the fiber length decreased, with the shortest fiber fraction having the highest percentage of HRW[I]. Recycling reduced the percentage of HRW[I] to HRW[II] for all fiber fractions and had more effect on the shorter fiber fraction than the longer fraction.

Figure 36 shows the effect of recycling on the percentage of HRW[II] for the four fiber fractions. Recycling reduced the percentage of HRW[II] and had a significant effect on the shorter fiber fractions.
To interpret the above results, pore-size distributions were measured. Figure 37 shows the effect of recycling on pore size. Recycling increased the number of the small pores, and this increased the HRW(II). Figure 38 shows the effect of recycling on pore diameter of sheets produced from mesh 50 and mesh 100. Recycling

![Graph showing the amount of water removed during the first and second falling-rate periods](image)

Figure 32. Amount of Water Removed During the First and Second Falling-Rate Periods, (HRW[T])

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Figure 33. Amount of Water Removed During the First Falling Rate Period, (HRW[I])

Figure 34. Amount of Water Removed During the Second Falling-Rate Period, (HRW[II])
Figure 35. Percentages of Water Removed During the First Falling-Rate Period, (HRW[I] to HRW[T])

Figure 36. Percentages of Water Removed During the Second Falling-Rate Period, (HRW[II] to HRW[T])
decreased the average pore diameter but increased the total volume of the pores.

Analysis of the drying behavior of fractionated virgin fiber using the drying equipment shows that as the fibers became shorter, less water evaporated in the falling rate. The same percentage of water dried in the constant-drying zone. TGA shows that more water dried when moving from longer fibers to shorter fibers in both the constant-drying zone and falling-rate zone (Figure 39).

Recycled fibers' drying behavior is shown in Figure 40. When using the drying equipment, more water dried as the fiber length decreased and less water dried in the constant-drying zone.

![Graph](image.png)

**Figure 37.** Total Pore Volume of Handsheets Produced From Mesh 50 and Mesh 100 Fibers Fraction
Figure 38. Effect of Recycling on Pore Diameter of Handsheets Produced From Mesh 50 and 100 Fibers Fractions
Figure 39. Comparison of the Drying Percentages in Each Drying Segment for the Fractionated Virgin Fiber Using the Drying Equipment and the TGA. Here, Period 1 Represents the Initial Heating Period, period 2 Represents the Constant-Drying Rate Period, Period 3 Represents the First Falling-Rate Period, and Period 4 Represents the Second Falling-Rate Period.

Figure 41 shows a comparison of the drying behavior of the virgin and recycled fibers, both with the drying equipment and the TGA. The same percentage of water was removed from the recycled and virgin fibers when the initial moisture content was at the high level. This behavior was noticed in both the drying equipment and the TGA. When the initial moisture content was at the low level, the TGA drying curve for the virgin fibers showed less water removed in the first two periods and more water removed in the last two periods. When the drying equipment was used at
Figure 40. Comparison of the Drying Percentages in Each Drying Segment for the Fractionated Virgin Fiber Using the Drying Equipment and the TGA. Here, Period 1 Represents the Initial Heating Period, Period 2 Represents the Constant Drying Rate Period, Period 3 Represents the First Falling-Rate Period, and Period 4 Represents the Second Falling-Rate Period.

the low level of initial moisture content, the virgin fibers had more water removed in the first period, less water removed in the second period, more water removed again in the third period, and finally less water removed in the fourth period.

The critical moisture content (CMC) is the point at which surface water evaporates and embedded water starts to evaporate. In the current study critical moisture content increased as the fibers became shorter (Figures 42 and 43). Virgin-fiber CMC increased from 40% to 50% (Figure 42). For the recycled fiber, CMC increased from 15% to 45% as the fibers became shorter (Figure 43).
Factors that controlled the drying rate depended on the nature of the fiber. Figure 44 represents the ratio of the moisture content to CMC versus time. The two drying zones were determined, and the drying rate for the first falling-rate zone increased as the fibers became shorter. The drying rate in the second falling-rate zone decreased with shorter fibers. The same behavior was observed for the virgin fiber, and the data are shown in Figure 34. More time was needed for complete drying of fractionated virgin fiber as the fibers became shorter, and fractionated recycled fiber showed the same time for complete drying (Figures 44 and 45).
Linear correlations were found between HRW(T) and WRV and between HRW(I) and WRV (Figures 46 and 47). No relationship, however, was found between HRW(II) and WRV (Figure 48). WRV consists of HRW(T) and other water that may still be on the surface of the sheet and fiber.

The maximum drying rate for the fractionated recycled fiber was found to be 4.4 g/cm².sec. This value was obtained with moisture content of 0.8 g-water/g-fiber, as shown in Figures 49 and 50. On the other hand, fractionated virgin fiber showed an optimum drying rate of 4.2 g/cm².sec with moisture content of 0.7 g-water/g-fiber and 1.2 g-water/g-fiber, as shown in Figures 51 and 52. Recycled fibers 3 mm long and virgin fibers 1.7 mm long had the highest drying rate, as shown in Figures 53, 54, 55, and 56.
Figure 43. Critical Moisture Content of the Fractionated Recycled Fiber as a Function of the Mesh Number
Figure 44. Comparison of the First and Second Falling-Rate Periods for the Fractionated Recycled Fiber
Figure 45. Comparison of the First and Second Falling-Rate Periods for the Fractionated Virgin Fiber
Figure 46. The Relationship Between HRW(T) and WRV of Softwood Fractionated Fibers

Figure 47. The Relationship Between HRW(I) and WRV of Softwood Fractionated Fibers
Figure 48. The Relationship Between HRW(II) and WRV of Softwood Fractionated Fibers
Figure 49. 3-D Representation of the Drying Rate as a Function of Fiber Length and Moisture Content for Recycled Fiber

Figure 50. Contour Map for Figure 49: Relationship Between Fiber Length, Moisture Content, and Drying Rate
Figure 51. 3-D Representation of the Drying Rate as a Function of Fiber Length and Moisture Content for Virgin Fiber

Figure 52. Contour Map for Figure 51: Relationship Between Moisture Content, Fiber Length, and Drying Rate

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Figure 53. 3-D Representation of the Drying Rate as a Function of Fiber Length and Time Content for Recycled Fiber

Figure 54. Contour Map for Figure 53: Relationship between Fiber Length, Drying Time, and Drying Rate
Figure 55. 3-D Representation of the Drying Rate as a Function of Fiber Length and Time Content for Virgin Fiber

Figure 56. Contour Map for Figure 55: Relationship Between Fiber Length, Drying Time, and Drying Rate
Figure 57 shows the drying time for each drying period of handsheets produced from unrefined fractionated hardwood. The drying times in the warm-up period and the constant-drying-rate period were the same for different fractions and different basis-weight sheets. The drying time for shorter fibers was higher than the drying time for longer fibers. In the first and second falling-rate periods, the drying time was higher for the longer fibers, and it was higher for the sheets with the higher basis weight.

![Graph showing drying time comparison](image)

Figure 57. Drying Time of Each Drying Period for Unrefined Fractionated Hardwood Sheets at two different Basis Weight

Figure 58 shows the effect of refining on the drying time of each drying period. The effect of handsheet basis weight for refined fractionated fibers on drying times is also shown. Comparing Figure 58 to Figure 57 shows that refining increased the drying time of the fiber due to the changes in fiber structure caused by the refining
process. Handsheets of 60-gsm basis weight made of mesh-50 and mesh-100 refined fiber had the same drying time. When the basis weight increased to 120 gsm, mesh-50 refined fibers needed more time for each drying segment, unlike the mesh-100 unrefined fibers, which needed more drying time at 120 gsm for only the first and second falling-rate periods.

![Graph showing drying time for different fiber types and basis weights](image)

Figure 58. Drying Time of Each Drying Period for Refined Fractionated Hardwood Using PFI Type Refiner for 15 Seconds

**Isotherms**

The adsorption isotherms of handsheets made of fractionated virgin fibers showed that, for all relative-humidity values, the equilibrium moisture content decreased as the temperature increased, and increased as the fiber length decreased. This is shown in Figures 59, 60, and 61.
Figure 59. Adsorption Isotherm for Virgin Fractionated Fiber at Surrounding Temperature of 25 °C
Figure 60. Adsorption Isotherm for Virgin Fractionated Fiber at Surrounding Temperature of 35 °C

Figure 61. Adsorption Isotherm for Virgin Fractionated Fiber at Surrounding Temperature of 60 °C
Recycled-fiber isotherms are shown below in Figures 62, 63, and 64. The recycled fibers exhibited the same behavior as softwood fibers: decreasing equilibrium moisture content with increasing temperature, and increasing equilibrium moisture content with decreasing size. It was also found that the equilibrium moisture content of the recycled fibers was higher than that of the softwood fibers.

Figure 62. Adsorption Isotherm for Recycled Fractionated Fiber at Surrounding Temperature of 25 °C
Figure 63. Adsorption Isotherm for Recycled Fractionated Fiber at Surrounding Temperature of 45 °C
Figures 65 through 67 show the isotherms of handsheets made of fractionated hardwood. As the temperature increased the isomers with the longer fraction had a higher equilibrium moisture content of 25 °C, and those with the shorter fraction had a higher equilibrium moisture content of 60 °C. The equilibrium moisture content of the two fractions was similar at 35 °C.
Figure 65. Adsorption Isotherm for Never-Dried Hardwood Fractionated Fiber at Surrounding Temperature of 25 °C

Figure 66. Adsorption Isotherm for Never-Dried Hardwood Fractionated Fiber at Surrounding Temperature of 35 °C
Fiber Fractionation Modeling

A system specific mathematical model that describes the relationship between fiber-length distribution and the normal fraction distribution was developed. This model based on using Clark classifier with screen mesh number of 14, 30, 50, 100 and 200 only. Further investigations will be needed to validate and generalize this model. In this model the fiber-length distribution of the fractions was estimated when the fiber-length distribution of the source fiber was known. What was needed to estimate the fractions' fiber-length distribution was the mean fiber lengths of each fraction. This model was applied theoretically by employing the available equipment to any
cases that were not able to achieve in a limited equipped laboratory, then using the model to estimate the rest of the data.

Below is the description of the mathematical model that related the fiber-length distribution to the fiber-length distribution of its fractions.

1. Fractionation of the fiber using a Clark classifier and calculation of the mass percentage of each fraction \((X_i)\).

2. Characterizing each fraction and the source fiber using an FQA.

3. Fitting the data from each fraction into normal distribution function:

\[
P(X) = \frac{1}{\sigma \sqrt{2\pi}} \exp\left(-\frac{(x - \mu)^2}{2\sigma^2}\right)
\]

where \(\mu\) is the mean and \(\sigma\) is the standard deviation, as shown in Figures 61 and 62.

![Process Capability for Length](image)

Figure 68. Normal Distribution Fitting Using STATGRAPHICS Plus 5.1
Those functions are:

\[ P(X) = \frac{1}{0.94\sqrt{2\pi}} \exp\left( -\frac{(x - 0.52)^2}{2 \times 0.94^2} \right) \]

Mesh 14 = \[ \frac{1}{1.81\sqrt{2\pi}} \exp\left( -\frac{(x - 2.63)^2}{2 \times 1.81^2} \right) \]

Mesh 30 = \[ \frac{1}{1.2\sqrt{2\pi}} \exp\left( -\frac{(x - 2.52)^2}{2 \times 1.2^2} \right) \]

Mesh 50 = \[ \frac{1}{0.82\sqrt{2\pi}} \exp\left( -\frac{(x - 1.59)^2}{2 \times 0.82^2} \right) \]

Mesh 100 = \[ \frac{1}{0.44\sqrt{2\pi}} \exp\left( -\frac{(x - 0.79)^2}{2 \times 0.44^2} \right) \]

Mesh 200 = \[ \frac{1}{0.3\sqrt{2\pi}} \exp\left( -\frac{(x - 0.43)^2}{2 \times 0.3^2} \right) \]
4. Adjusting each distribution by multiplying it by its fraction mass percentage calculated in step 1. Those adjusted functions are presented in Figure 63.
5. Calculating the summation of all the adjusted-fraction distribution functions. \( P_t(X) = \sum X_i P_i(X) \). \( X_i \) : Mass Percentage. \( P_i \): Distribution

\[
P_t(X) = 0.01062 + \frac{0.908}{2.96\sqrt{\pi/2}} \exp\left(-2 \times \left(\frac{X - 1.988}{2.96}\right)^2\right)
\]

Figure 71. Fiber-Length Normal Distribution of the Source Fibers and the Summation of the Fractionated Fibers

6. Finally finding the ratio between the summation of the adjusted distribution and the distribution of the source fibers.

\[
Ratio = \frac{1}{0.94\sqrt{2\pi}} \exp\left(-\frac{(x - 0.52)^2}{2 \times 0.94^2}\right)
\]

\[
\frac{0.01062 + \frac{0.908}{2.96\sqrt{\pi/2}} \exp\left(-2 \times \left(\frac{X - 1.988}{2.96}\right)^2\right)}{0.94\sqrt{2\pi} \exp\left(-\frac{(x - 0.52)^2}{2 \times 0.94^2}\right)}
\]

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CHAPTER IV

DISCUSSION

The Clark classifier satisfactorily separated fibers into coarse and fine fractions based on the differences between their mean fiber lengths and coarseness values. The fine fraction produced sheets with higher strength properties, but these sheets were denser. The finer fibers were also more flexible than the coarse fibers. Fiber rigidity reduced the chance that the fibers would make fiber-fiber bonds, and that resulted in a lower tensile strength but greater bulk. Coarseness also increased the porosity and the roughness of the sheet. A higher percentage of recycled fibers than virgin fibers were lost because of fractionation. Analysis of the fiber length showed similarity.

When the drying equipment was used, less water dried from the virgin fractionated fiber in the falling rate as the fiber length decreased. The same drying percentage was noticed in the constant-drying zone. The TGA produced more drying as fibers got shorter. The same behavior was noticed in the constant-drying zone.

Drying recycled fractionated fibers with the drying equipment produced a shift from the constant-drying zone to the bound-water zone as drying proceeded from longer fibers to shorter fibers. (Longer fibers contain more surface water than shorter ones; shorter fibers, on the other hand, have more bound water.) The TGA
produced the same drying percentage in the falling-rate zone as in the constant-drying zone.

It was found that the higher the moisture content the higher the surface water and the lower the CMC. As the paper basis weight increased the CMC also increased, but CMC decreased when sheet thickness increased. As the bound-water content increased the CMC also increased.

When the sheet moisture content was equal to or less than that of the CMC, the sheet characteristics controlled the drying behavior. The two drying zones (the first falling-rate period and the second falling period) were determined clearly with both the TGA and the drying equipment. The drying rate in the first falling period increased as the fiber became shorter, but decreased in the second falling period as the fiber became shorter. More time was needed to complete drying in the falling zones as the fiber became shorter. This behavior was the same for both recycled and virgin fiber.

Handsheets made from these fibers showed different drying behaviors for WRV, HRW[T], HRW[I], and HRW[II]. Free water filled the pores in the handsheets and formed a film on the surface of the fiber. HRW[I] represented the point at which capillaries could not supply water at the rate necessary to maintain a uniform surface moisture level. HRW[II] represented a second break point in the drying curve and included chemically bound water and water contained in fine capillaries. The sum of HRW[I] and HRW[II] equaled HRW[T].
The handsheets had a pore-size distribution; they also had a capillary-pressure curve, not just a capillary pressure. In the current study, the pore size for each fiber fraction was measured as an average value of all the pores between the fibers that formed the sheet structure and the pores within the fibers. The total HRW[I] and HRW[II] were higher for the finer fractions. Handsheets produced from shorter fiber fractions contained more numerous and smaller pores compared to those produced from longer fractions. The ability of the sheet to hold water within its structure increased as the pore volume increased.

Recycling was found to increase HRW[II] and decrease HRW[I] (due to the decrease in the number of large capillaries), which also, therefore, decreased HRW[T]. Recycling decreased the average pore volume, which decreased the amount of water held within the pores. Because of higher capillary pressure the water in these smaller pores was held more tightly than in the larger pores. Total pore area increased with recycling because the number of the small pores increased, which then increased HRW[II].

One other reason for this decrease was the effect of recycling on external fibrillation. As the sheet dried, some of the fibrils did not easily rewet and so remained attached to the fiber. If these fibrils held water within their structure, then the HRW[T] would decrease as the number of recycling stages increased.

With higher moisture content, both recycled and virgin fibers had the same drying behavior. The highest drying rate of 4.4 g/cm²/sec was calculated using a statistical mathematical model. This rate was obtained with a moisture content of 0.8
g water/ g fiber for the recycled fiber and 0.7 and 1.2 g water/ g fiber for the fractionated virgin fiber. Recycled-fiber length of 3 mm and virgin-fiber length of 1.7 mm had the highest drying rates.

The equilibrium moisture content increased with increasing relative humidity and decreased with increasing temperature for the softwood fibers and the recycled fibers. Finer fibers had higher equilibrium moisture content because of their higher percentage of non-crystalline cellulose. By extending the adsorption isotherm to 100% relative humidity, the resultant equilibrium moisture content represented the bound-water content. Bound water has a vapor pressure lower than that of liquid water at the same temperature. The bound-water data from the adsorption isotherm confirmed the TGA-produced bound-water findings. On the other hand, hardwood-fiber equilibrium moisture content showed a different response to increasing temperature. The longer fraction had a higher moisture content at lower temperatures, and this shifted as the temperature increased.
CHAPTER V
CONCLUSIONS

The major results from this study are summarized below:

- The lower-coarseness (thinner) fibers were shorter, had longer drying times, and produced sheets with higher tensile strength, lower roughness values, lower porosity, higher critical moisture content, higher WRV, higher HRW[T], higher HRW[I] and lower HRW[II]. As the fiber length decreased, the amount of surface water decreased while the bound water increased and this resulted in a decrease in the drying percentage for both the constant zone and the first-falling rate period and an increase in the drying percentage in the second falling-rate period.

- Fiber length had no effect on the percentage of drying or the drying time during the sheet-warm-up stage.

- TGA provides an accurate method to study fiber-drying behavior when the appropriate procedure is followed. A TGA technique was developed to identify the two falling-rate periods that occurred during paper drying. This technique identified HRW[T] and distinguished between HRW[I] and HRW[II].

- Recycling decrease both WRV and HRW[T]; this decrease was due to the decrease in HRW[I] and the increase in HRW[II]. The greatest effect of recycling was seen after the first recycling stage; later recycling stages had
diminishing effects on HRW[T], HRW[I], HRW[II], and the WRV. Recycling had more effect on the HRW[T] of the shorter fiber fractions that it did on the longer, coarser fibers.

- Recycling increased the number of fine pores, which increased the HRW[II].
- Recycled fibers dry faster than virgin fibers because recycling affects the structure and composition of the fibers.

The major observations from this study are summarized below:

- The level of initial moisture content affects the drying behavior of the fiber. With a low level of moisture content there may not be a constant-drying zone.
- Virgin fibers have critical-moisture content from 40% to 55%, whereas recycled fibers have critical-moisture content from 15% to 45%.
- A 3-D statistical model showed that 1.7-mm-long recycled fibers with moisture content of 0.8 g water/g fiber had the highest drying rate and that 2.3-mm-long virgin fibers with moisture content of 2.1, 1.3, and 0.75 g water/g fiber had the highest drying rate of 4.4 g/cm²·sec.

It's recommended to study the following further:

- The effect of fractionation and recycling levels on the percentage of non-crystalline cellulose to the crystalline cellulose. The existence of differences in the crystallinity explains the differences in drying behavior and equilibrium moisture content.
- The system specific mathematical fractionation model developed in this study required a further study to inspect and develop a general model.
Appendix A

Drying Set-Up
The drying setup system was designed by Dr. John Cameron of Western Michigan University (Cameron & Zwick, 2003). The setup was built to simulate a 6-foot-diameter commercial drying cylinder. The fabric tension was within TAPPI-recommended limits, and it was achieved using the weight of the system pressing against the fabric during drying. The system consisted of an electrical heater, a 0.5-inch-thick stainless steel-304 plate, drying fabric, and three thermocouples. One thermocouple transferred the temperature from the paper, one from the shell, and one from the heater. The signal from the thermocouples was transferred digitally through a data-acquisition card. The three data streams with time transferred to a computer to be analyzed according to the explicit method described by Incropera and Dewitt (1990). Operating the equipment involved preheating it to 125 °C, then turning the heater off until the temperature reached 120 °C. This allowed equilibrium between the heater side and the shell side. At that time the test was started by lowering the top part of the equipment onto the fabric holding the sample. The data collection continued for about five minutes. After that the software asked for information about the wet weight, dry weight, and area of the sample. An analysis of the data was then displayed in Excel.
Figure A-72. The Environment of the Analysis Software in Excell 11 Macro

Below is the sub-macro written by Dr. John Cameron to the instant temperature from the three thermocouples. This macro was run by clicking on the “QuickTemp” button in the software environment.

Sub QuickTemp()
' quick_temp Macro
' Macro recorded 2/4/99 by Cameron
' Keyboard Shortcut: Ctrl+t
ProgStat% = cbTIn(0, 20, Celius, Temp1, FILTER)
Dim i As Integer
Time1 = Timer
For i = 1 To 100
  ProgStat% = cbTIn(1, 20, Celius, Temp1, FILTER)
  ProgStat% = cbTIn(0, 21, Celius, Temp2, FILTER)
  ProgStat% = cbTIn(0, 22, Celius, Temp3, FILTER)
Next i
时光2 = Timer
dt = (time2 - Time1) / 100
Worksheets("sheet1").Select
Range("b7").Value = dt
Range("b8").Value = Temp1
Range("b9").Value = Temp2
Range("b10").Value = Temp3
Range("a7") = "del_T's"
Range("a8") = "Heater 'C"
Range("a9") = "Shell 'C"
Range("a10") = "Paper 'C"
End Sub

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Following is the sub-macro written by Dr. John Cameron to collect and analyze the drying data. This macro was run by clicking on the “Record Temp” button in the software environment. This calculation method was based on the explicit method described by Incropera and Dewitt (1990).

Option Base 1
Sub GetTemperature()
  Dim Temparray() As Single
  Const max As Integer = 10000 'number of data points
  Const nodes As Integer = 25 'number of nodes in shell
  ' Calculated based on Basis Weight
  HFlos = 165 ' Energy lost due to heating fabric'
  HPC = 1# ' Fraction of heat transfer to paper used for drying
  Dim Fo As Single
  Dim k As Single
  Dim dt As Single
  Dim dx As Single
  Dim x As Single
  Dim Cp As Single
  Dim thi As Single
  Dim tsi As Single
  Dim TimeStart As Single
  Dim TimeEnd As Single
  Dim u As Integer
  Dim v As Integer
  Dim t As Single
  Dim rho As Single
  Dim alpha As Single
  Dim step As Single
  Dim i As Integer
  Dim j As Integer
  Dim Jave As Integer
  Dim n As Integer
  Dim nn As Integer
  Dim Temp As Single
  Dim TempI As Single
  Dim TimeII As Single
  Dim count As Integer
  Dim Temp(1 To max, 1 To 4)
  Dim Htem(1 To max, 1 To 11)
  Dim Array1(1 To max, 1 To 4) As Single
Dim sum1 As Single
Dim sum2 As Single
ReDim Temparray(l To max, 1 To 4)
Dim Temp2() As Single
Dim Qflux(1 To (max + 10), 3) As Single
Dim q As Single
Dim TemA(max + 1, nodes) As Single
Dim Temp1(nodes, max + 1) As Single
Dim dweight As Single
Dim wweight As Single
Dim area As Single
Dim DR As Single
n = nodes
Worksheets("sheet1").Select
With Range("A14:Ay10016")
.Clear
End With
' initialize board

ProgStat% = cbTIn(0, 20, Celsius, Tempi, FILTER)
ProgStat% = cbTIn(0, 21, Celsius, TempII, FILTER)
ProgStat% = cbTIn(0, 22, Celsius, TempIII, FILTER)

' set initial time and measure temperature
' Temperature Tempi = Heater
' Temperature TempII = Shell
' Temperature TempIII = Paper
TimeStart = Timer

For count = 1 To max Step 1
TempA = 0
TempB = 0
TempC = 0
For nn = 1 To 10
ProgStat% = cbTIn(0, 20, Celsius, Tempi, FILTER)
TempA = TempI / 10 + TempA
ProgStat% = cbTIn(0, 21, Celsius, TempII, FILTER)
TempB = TempII / 10 + TempB
ProgStat% = cbTIn(0, 22, Celsius, TempIII, FILTER)
TempC = TempIII / 10 + TempC
Next nn
Temparray(count, 1) = TempA
Temparray(count, 2) = TempB
Temparray(count, 4) = TempC
Next count
TimeEnd = Timer
'Start Heat flux routine here'
'Set Boundary Conditions at first data point
tsi = Temparray(1, 2)
thi = Temparray(1, 1)
'calculate delta time
t = (TimeEnd - TimeStart)
dt = t / max
'Calculate Constants
ktemp = 273.15 + (tsi + thi) / 2
k = 14.9 + 0.017 * (ktemp - 300.001) 'stainless 304'
rho = 7900
Cp = 477 + 0.38 * (ktemp - 300.001)
x = 0.0127
dx = x / (n - 1)
alpha = k / (rho * Cp)
'check constants
Fo = alpha * dt / (dx ^ 2)
MsgBox Fo, , "Fo must be less than 0.5"
'Create Time array
For i = 1 To max
Temparray(i, 3) = i * dt
Next i
'average temperatures
For i = 1 To 10
Tem(i, 1) = Temparray(i, 1)
Tem(i, 2) = Temparray(i, 2)
Tem(i, 3) = Temparray(i, 3)
Tem(i, 4) = Temparray(i, 4)
Next i
For i = 11 To max - 11
For j = -5 To 5
Tem(i, 1) = Temparray(i + j, 1) / 11 + Tem(i, 1)
Tem(i, 2) = Temparray(i + j, 2) / 11 + Tem(i, 2)
Tem(i, 3) = Temparray(i + j, 3) / 11 + Tem(i, 3)
Tem(i, 4) = Temparray(i + j, 4) / 11 + Tem(i, 4)
Next j
Next i
'Write temperature and time data to spreadsheet
Worksheets("sheet1").Select
With Range("c15:f10015")
.Value = Tem
End With
' Set initial conditions
' Dimension as distance and time
TemA(1, 1) = tsi
step = (thi - tsi) / (n - 1)
For i = 1 To n - 1
  TemA(1, i + 1) = tsi + i * step
Next i
'Determine Temp distribution in plate at each time t=p*dt (p > 1)
' Use thermocouple readings to get the temp at the front and
' back surfaces. Use numerical technique to determine internal
'Temp distribution. Calculate heat flux and store in array'
'Set Boundary Conditions
For i = 1 To (max - 1)
  TemA(i, 1) = Tem(i + 1, 2)
  TemA(i, n) = Tem(i + 1, 1)
Next i
For i = 1 To (max - 1)
  For j = 2 To n - 1
    TemA(i + 1, j) = (Fo * (TemA(i, j - 1) + TemA(i, j + 1))) + (1 - 2 * Fo) * TemA(i, j)
  Next j
Next i
For i = 1 To max - 2
  ' heat flux
  Qflux(i, 1) = 1 * k * (TemA(i, 2) - TemA(i, 1)) / dx
  ' heat flux including temperature change
  Qflux(i, 2) = -1 * ((rho * dx * Cp) / (2 * dt) * (TemA(i + 1, 1) - TemA(i, 1))) + Qflux(i, 1)
  ' Total heat transferred w/m^2
  Qflux(i + 1, 3) = Qflux(i, 2) * dt + Qflux(i, 3)
Next i
' write heat fluxes to worksheet Qflux is basic heat flux, Qflux2 is corrected heat flux, and Qflux3 is total energy transferred
Worksheets("sheet1").Select
With Range("z14:ax10015")
  .Value = TemA
End With
With Range("g14:i10014")
  .Value = Qflux
End With
'
' Calculation of Paper Shell Contact Coefficient
' Entering Sheet Data
" Input the date
' Input the run number
Temp = InputBox("Enter run number as #")
rnumber = CSng(Temp)
' Input the wet weight of the paper
Temp = InputBox("Enter the weight of wet paper in grams")
wweight = CSng(Temp)
iwweight = wweight
' Input the dry weight of the paper
Temp = InputBox("Enter the weight of dry paper in grams")
dweight = CSng(Temp)
' Input the Sheet Area
Temp = InputBox("Enter the area of the paper in sq meters")
area = CSng(Temp)
Range("a2") = "run number"
Range("a3").Value = rnumber
Range("b2") = "DATE"
Range("b3") = Date
Range("c2") = "wet weight"
Range("c3").Value = iwweight
Range("d2") = "dry weight"
Range("d3").Value = dweight
Range("e2") = "area"
Range("e3").Value = area
Bweight = dweight / area
Range("f2") = "Basis Weight g/M^2"
Range("f3").Value = Bweight
' Input Target Basis Weight
Temp = InputBox("Enter target basis weight")
TBW = CSng(Temp)
Range("f4") = "Target Basis Weight"
Range("f5").Value = TBW
' Input CSF
Temp = InputBox("Enter the refining level in CSF")
CSF = CSng(Temp)
Range("g2") = "CSF"
Range("g3").Value = CSF
' Input Fiber Type
Temp = InputBox("Enter the fiber type 1=HW, 2=SW")
Fiber = CSng(Temp)
Range("h2") = "Fiber Type, HW = 1 and SW = 2"
Range("h3") = Fiber
Range("h4") = TimeEnd
Range("h5") = TimeStart
'
For i = 2 To max - 1
If (Tem(i, 2) - Tem(i, 4)) = 0# Then
    Htem(i, 1) = 0#
Else: Htem(i, 1) = Qflux(i, 2) / (Tem(i, 2) - Tem(i, 4) + 0.00000000001)
End If
' Calculation of Sheet Moisture Content
Hlos = -0.06732 * TBW + 15.35
Htem(i, 2) = (wweight - dweight) / dweight
dryR = (((HPC * Qflux(i, 2) - Hlos * Tem(i, 4)) * dt - HFlos * (Tem(i, 4) - Tem(i - 1, 4))) / area) - (dweight * (1.33 + Htem(i, 2) * 4.184) * (Tem(i, 4) - Tem(i - 1, 4))) / (2508 - 2.45 * Tem(i, 4))
If dryR < 0# Then dryR = 0#
    wweight = wweight - dryR
Htem(i, 3) = dryR / (dt * area)
' Htem(i, 3) g/M^2
' Paper Balance
' Paper Balance Based on Calculated Heat Flux and Temperature of Paper
Htem(i, 4) = ((dweight * (1.33 + Htem(i, 2) * 4.184)) * (Tem(i, 4) - Tem(i - 1, 4)) + dryR * (2508 - 2.45 * Tem(i, 4))) / area
Htem(i, 5) = Htem(i - 1, 5) + Htem(i, 4)
' Paper Paper Balance Based on Wet and Dry Weights and Temperature of Paper
' Assume that the vaporization occurs at 295 K and the vapor leaves at paper temperature
Htem(i, 6) = (((Tem(i, 4) - Tem(l, 4)) * (dweight * 1.33) + (iwweight - dweight) * 2449 + ((wweight - dweight) * (Tem(i, 4) - Tem(l, 4)) * 2.09))) / area
' Energy Balances
'
' Steel Balance
'
Htem(i, 7) = 0.0127 * 7900 * 477 * (TemA(ll, 1) + TemA(ll, 2) - TemA(i, 1) - TemA(i, 2)) / 2#
'
' Ratios
' Paper (htem(i,9), htem(i,10) and steel htem(i,11))
Htem(i, 9) = Htem(i, 5) / (Qflux(i, 3) + 10 ^ -20)
Htem(i, 10) = Htem(i, 6) / (Qflux(i, 3) + 10 ^ -20)
Htem(i, 11) = Htem(i, 7) / (Qflux(i, 3) + 10 ^ -20)
Next i
'write data
' With Range("K15:u10015")
.Value = Htem
End With
Range("f6") = "dt"
Following are data samples obtained by the equipment and analyzed by the above software.

Figure A-73. Drying Rate as a Function with Time
Figure A-74. Moisture Ratio as a Function with Time

Figure A-75. Paper-Shell Contact Coefficient as a Function with Moisture Content
Figure A-76. Shell and Paper Temperature and Heat Flux Versus Time
Appendix B

Thermogravimetric Analysis (TGA)
Thermogravimetric analysis (TGA) (Inspiratech 2000 Ltd and ORTON (2004)) is a technique through which measurements of weight change as a function of time or temperature are done. This analysis provides information about moisture and/or volatiles content, composition, thermal stability, and reaction kinetics.

As materials are heated, they lose weight because of moisture evaporation or because of chemical reactions that produce gases, or they gain weight because of reactions with surrounding gases.

Performing a TGA is simple. The material to be tested is placed in a platinum ban. The ban is attached to an analytical balance that can measure 1/1000 mg, and the balance and the empty ban are tarred before the sample is introduced. A computer attached to the TGA controls the measurement process and displays the data.
Appendix C

Porosity Meter/Mercury Intrusion [63]
This technique permits the measurement of pore volume and size by forcing mercury to penetrate the open porosity. Mercury porosimetry is applied over a capillary-diameter range of 0.003 μm to 360 μm. Mercury is forced into the pores by applying an increasing controlled pressure. A mercury-intrusion porosimetry test involves placing a sample into a container, evacuating the container to remove contaminant gases, and allowing mercury to fill the container. This creates an environment consisting of solid, liquid, and vapor mercury. During the experiment the pressure is increased and the volume of mercury penetrated is detected by a capacitive system. The decreasing volume of mercury in the sample holder represents the pore volume. The penetration pressure is directly related to the pore-access size by a well-known mathematical model, the Washburn equation:

\[ R = \frac{-2 \gamma \cos(\theta)}{P_c} \]

where:

\( \gamma \) : surface tension of pure mercury (480 dyne/cm)

\( \theta \) : contact angle between mercury and the solid (average value 140°)

\( P_c \) : mercury-penetration equilibrated pressure

\( R \) : pore radius

The mercury porosimeter measures only the applied pressure and the volume of mercury intruded into or extruded from the sample bulk. The pressure is increased toward atmospheric pressure while the volume of mercury enters larger openings in the sample. When pressure has returned to ambient, pores of diameters less than 12
micro-meters will be filled. The sample container is then placed in a pressure vessel for the remainder of the test.
Appendix D

Fractionation Modeling, Paul (2001)
Table D.1

Fiber Analysis Summary for the First Softwood Trial

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Function</th>
<th>Mean Length (mm)</th>
<th>Std. Dev.</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>Normal Distribution</td>
<td>0.6888</td>
<td>1.042</td>
<td>17,139</td>
</tr>
<tr>
<td>Mesh 14</td>
<td>Normal Distribution</td>
<td>2.652</td>
<td>1.335</td>
<td>9,181</td>
</tr>
<tr>
<td>Mesh 30</td>
<td>Normal Distribution</td>
<td>1.807</td>
<td>0.9944</td>
<td>13,411</td>
</tr>
<tr>
<td>Mesh 50</td>
<td>Normal Distribution</td>
<td>1.152</td>
<td>0.6941</td>
<td>16,225</td>
</tr>
<tr>
<td>Mesh 100</td>
<td>Normal Distribution</td>
<td>0.7328</td>
<td>0.3860</td>
<td>16,747</td>
</tr>
<tr>
<td>Mesh 200</td>
<td>Normal Distribution</td>
<td>0.2800</td>
<td>0.3240</td>
<td>18,939</td>
</tr>
</tbody>
</table>

Figure A-77. Fiber-Length Normal Distribution of Fractionated Fibers, Softwood Trial 1
Figure A-78. Adjusted Normal Distribution of Fractionated Fibers’ Length, Softwood Trial 1

Figure A-79. Normal Distribution of Summation of Adjusted Fiber-Length Distributions and Normal Distribution of Source Fiber, Softwood Trial 1
Table D.2

Fiber Analysis Summary for the Second Softwood Trial

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Function</th>
<th>Mean Length (mm)</th>
<th>Std. Dev.</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>Normal Distribution</td>
<td>0.5155</td>
<td>0.9387</td>
<td>17,139</td>
</tr>
<tr>
<td>Mesh 14</td>
<td>Normal Distribution</td>
<td>2.630</td>
<td>1.811</td>
<td>9,181</td>
</tr>
<tr>
<td>Mesh 30</td>
<td>Normal Distribution</td>
<td>2.520</td>
<td>1.195</td>
<td>13,411</td>
</tr>
<tr>
<td>Mesh 50</td>
<td>Normal Distribution</td>
<td>1.594</td>
<td>0.8201</td>
<td>16,225</td>
</tr>
<tr>
<td>Mesh 100</td>
<td>Normal Distribution</td>
<td>0.7943</td>
<td>0.4376</td>
<td>16,747</td>
</tr>
<tr>
<td>Mesh 200</td>
<td>Normal Distribution</td>
<td>0.4263</td>
<td>0.2944</td>
<td>18,939</td>
</tr>
</tbody>
</table>

Figure A-80. Fiber-Length Normal Distribution of Fractionated Fibers, Softwood Trial 2
Figure A-81. Adjusted Normal Distribution of Fractionated Fiber Length, Softwood Trial 2

Figure A-82. Normal Distribution of Summation of Adjusted Fiber-Length Distributions and Normal Distribution of Source Fiber, Softwood Trial 2
Table D.3

Fiber Analysis Summary for the Third Softwood Trial

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Function</th>
<th>Mean Length (mm)</th>
<th>Std. Dev.</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>Normal Distribution</td>
<td>0.5443</td>
<td>0.9514</td>
<td>13,229</td>
</tr>
<tr>
<td>Mesh 14</td>
<td>Normal Distribution</td>
<td>2.774</td>
<td>1.749</td>
<td>7,947</td>
</tr>
<tr>
<td>Mesh 30</td>
<td>Normal Distribution</td>
<td>2.556</td>
<td>1.162</td>
<td>14,835</td>
</tr>
<tr>
<td>Mesh 50</td>
<td>Normal Distribution</td>
<td>1.4735</td>
<td>0.8402</td>
<td>14,604</td>
</tr>
<tr>
<td>Mesh 100</td>
<td>Normal Distribution</td>
<td>0.7724</td>
<td>0.4368</td>
<td>12,483</td>
</tr>
<tr>
<td>Mesh 200</td>
<td>Normal Distribution</td>
<td>0.4514</td>
<td>0.3006</td>
<td>12,636</td>
</tr>
</tbody>
</table>

Figure A-83. Fiber-Length Normal Distribution of Fractionated Fibers, Softwood Trial 3
Figure A-84. Adjusted Normal Distribution of Fractionated Fiber-Length, Softwood Trial 3

Figure A-85. Normal Distribution of Summation of Adjusted Fiber-Length Distributions and Normal Distribution of Source Fiber, Softwood Trial 3
Table D.4
Fiber Analysis Summary for the First Hardwood Trial

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Function</th>
<th>Mean Length (mm)</th>
<th>Std. Dev.</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>Normal Distribution</td>
<td>0.4343</td>
<td>0.3454</td>
<td>10,724</td>
</tr>
<tr>
<td>Mesh 14</td>
<td>Normal Distribution</td>
<td>0.3788</td>
<td>0.3870</td>
<td>8,640</td>
</tr>
<tr>
<td>Mesh 30</td>
<td>Normal Distribution</td>
<td>0.9084</td>
<td>1.051</td>
<td>4,668</td>
</tr>
<tr>
<td>Mesh 50</td>
<td>Normal Distribution</td>
<td>0.8735</td>
<td>0.2674</td>
<td>14,414</td>
</tr>
<tr>
<td>Mesh 100</td>
<td>Normal Distribution</td>
<td>0.6061</td>
<td>0.2220</td>
<td>13,459</td>
</tr>
<tr>
<td>Mesh 200</td>
<td>Normal Distribution</td>
<td>0.4057</td>
<td>0.2105</td>
<td>10,983</td>
</tr>
</tbody>
</table>

Figure A-86. Fiber-Length Normal Distribution of Fractionated Fibers, Hardwood Trial 1
Figure A-87. Adjusted Normal Distribution of Fractionated Fiber-Length, Hardwood Trial 1

Figure A-88. Normal Distribution of Summation of Adjusted Fiber-Length Distributions and Normal Distribution of Source Fiber, Hardwood Trial 1
### Table D.5

Fiber Analysis Summary for the Second Hardwood Trial

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Function</th>
<th>Mean Length (mm)</th>
<th>Std. Dev.</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>Normal Distribution</td>
<td>0.4333</td>
<td>0.3463</td>
<td>12,705</td>
</tr>
<tr>
<td>Mesh 14</td>
<td>Normal Distribution</td>
<td>0.3335</td>
<td>0.3455</td>
<td>6,981</td>
</tr>
<tr>
<td>Mesh 30</td>
<td>Normal Distribution</td>
<td>0.7126</td>
<td>0.9367</td>
<td>7,864</td>
</tr>
<tr>
<td>Mesh 50</td>
<td>Normal Distribution</td>
<td>0.8542</td>
<td>0.2703</td>
<td>12,732</td>
</tr>
<tr>
<td>Mesh 100</td>
<td>Normal Distribution</td>
<td>0.6018</td>
<td>0.2201</td>
<td>13,805</td>
</tr>
<tr>
<td>Mesh 200</td>
<td>Normal Distribution</td>
<td>0.3982</td>
<td>0.2071</td>
<td>18,084</td>
</tr>
</tbody>
</table>

![Figure A-89. Fiber-Length Normal Distribution of Fractionated Fibers, Hardwood Trial 2](image)

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Figure A-90. Adjusted Normal Distribution of Fractionated Fiber-Length, Hardwood Trial 2

Figure A-91. Normal Distribution of Summation of Adjusted Fiber-Length Distributions and Normal Distribution of Source Fiber, Hardwood Trial 2
REFERENCES


