APPLICATION OF LOW CONSISTENCY REFINING OF PULP IN PRODUCING MULTI-PLY FOLDING BOXBOARDS

By

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A thesis submitted in conformity with the requirements for the degree of Master of Applied Science
Department of Chemical Engineering and Applied Chemistry
University of Toronto

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Abstract

In this study, the effect of using low consistency refined (LCR) pulp in the middle-ply furnish of folding boxboards (FBBs) is examined. LCR radiata reject pulp (LCRR) with a wide range of freeness values were blended with three commercial BCTMPs to form FBB middle-ply samples. The results indicated that increasing LC refining energy of radiata reject pulp improved the mechanical strength of samples up to the optimum freeness of about 400mL. Laboratory-made three-ply FBBs with varying LCRR contents in the middle-ply were also prepared and compared to those containing 100% commercial BCTMPs in the middle-ply. It was shown that FBBs containing 45% LCRR had better mechanical properties compared to those containing only commercial BCTMPs. Additionally, the use of HC-LC refined spruce-pine-fir (SPF) pulp as the middle-ply furnish in a three-ply FBB was investigated. It was found that sulphite pretreatment of HC-LC refined pulp had no clear effect on FBB properties.
Acknowledgement

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<tbody>
<tr>
<td>ANOVA</td>
<td>Analysis of Variance</td>
</tr>
<tr>
<td>ASR</td>
<td>Aspen, Spruce, Radiata pine</td>
</tr>
<tr>
<td>ATR-IR</td>
<td>Attenuated Total Reflectance Infrared Spectroscopy</td>
</tr>
<tr>
<td>BCTMP</td>
<td>Bleached Chemi-Thermo Mechanical Pulp</td>
</tr>
<tr>
<td>BEL</td>
<td>Bar Edge Length, km/rev</td>
</tr>
<tr>
<td>CD</td>
<td>Cross Direction</td>
</tr>
<tr>
<td>CIE</td>
<td>Commission Internationale de l'Eclairage</td>
</tr>
<tr>
<td>CSF</td>
<td>Canadian standard freeness, mL</td>
</tr>
<tr>
<td>CTMP</td>
<td>Chemi-Thermo Mechanical Pulp</td>
</tr>
<tr>
<td>DSF</td>
<td>Dynamic Sheet Former</td>
</tr>
<tr>
<td>FBB</td>
<td>Folding Box Board</td>
</tr>
<tr>
<td>FQA</td>
<td>Fibre Quality Control</td>
</tr>
<tr>
<td>HC</td>
<td>High Consistency</td>
</tr>
<tr>
<td>LC</td>
<td>Low Consistency</td>
</tr>
<tr>
<td>LCR</td>
<td>Low Consistency Refining</td>
</tr>
<tr>
<td>LCRR</td>
<td>Low Consistency Refining Reject</td>
</tr>
<tr>
<td>MD</td>
<td>Machine Direction</td>
</tr>
<tr>
<td>OCCAM</td>
<td>Ontario Centre for the Characterization of Advanced Materials</td>
</tr>
<tr>
<td>PPS</td>
<td>Parker Print Surface</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Full Form</td>
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<td>--------------</td>
<td>-----------</td>
</tr>
<tr>
<td>QRP</td>
<td>Quesnel River Pulp</td>
</tr>
<tr>
<td>SCT</td>
<td>Short span compression test, N.m/g</td>
</tr>
<tr>
<td>SE</td>
<td>Specific Energy, kWh/t</td>
</tr>
<tr>
<td>SEL</td>
<td>Specific Edge Load, J/m</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SPF</td>
<td>Spruce-Pine-Fir</td>
</tr>
<tr>
<td>TEA</td>
<td>Tensile energy absorption, mJ/g</td>
</tr>
<tr>
<td>TMP</td>
<td>Thermo Mechanical Pulp</td>
</tr>
<tr>
<td>TOF-SIMS</td>
<td>Time-of-Flight Secondary Ion Mass Spectrometry</td>
</tr>
<tr>
<td>UBC</td>
<td>University of British Columbia</td>
</tr>
<tr>
<td>WPI</td>
<td>Winston Pulp International</td>
</tr>
<tr>
<td>XPS</td>
<td>X-ray Photoelectron Spectroscopy</td>
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1 Introduction

1.1 Background

Pulping process is defined as converting the rigid structure of wood chips to a fibrous material, called pulp. Pulping methods are broadly divided into two categories: chemical pulping and mechanical pulping.

In chemical pulping, wood chips are cooked in a pressurized digester with appropriate chemicals to dissolve the lignin. The yield related to this method is relatively low, i.e. 40% to 55%. However, in mechanical pulping, wood chips break up by applying mechanical forces to produce wood fibres that include both cellulose and lignin, hence resulting in a high yield (typically about 95%).

A common mechanical pulping method is thermo-mechanical (TMP) pulping process. In this process, wood chips flow across bars and grooves of refiner disks and are disintegrated into individual fibres. The spacing between the refiner disks, i.e. gap, is in the order of a few fiber widths and is an important operational variable that affects both the pulp quality and the energy consumption [1]. Commercial TMP process usually involves several operations including converting wood to chips, chip pre-treatment, multi-stage refining, post-refining stage, latency removal, screening, and dewatering (Figure 1.1) [2]. In a typical TMP unit, following the refining stage, pulp is introduced to screens to separate the fibres according to their length. Adequately refined fibres pass through the screens as accept pulp while long coarse ones are separated as “reject”. The reject pulp is further refined in the reject refiner and is screened. The accept pulp from reject screens is subsequently added to the mainline pulp.

Although TMP pulping technology generates high quality papermaking fibers, it is very energy intensive. For instance, about 11% of the total electrical energy in British Columbia, Canada, is consumed by seven mechanical pulping facilities in that province. In a conventional TMP operation, approximately 80% of total energy consumption is related to the high consistency (HC) refiners. However, only a small portion of this energy is used to produce fibres and the remainder is converted to heat [3].
To address this issue, steam or chemicals could be applied on wood chips prior to the refiner. These pretreatments decrease the required energy for the refining process by either softening or removing lignin from the wood chips. As an alternative, several mills have replaced their mainline HC refiners with low consistency refining (LCR) that is known to consume as much as 20% less energy [4].

Figure 1.1 A typical thermo-mechanical pulping process with three stages of primary, secondary and tertiary refining [5]

1.2 Motivation

The global demand for paper-based packaging materials has been growing rapidly in recent years. Today, more than 40% of packaging materials are derived from paper. In many applications, paper is the preferred choice for packaging due to its good mechanical properties, ability to preserve goods from deterioration, good visual appearance and printability, and its environmental friendliness [6] [7].

FBB constitutes a large segment of paper packaging industry. Applications of folding boxboard are for packaging of health and beauty products, foods, pharmaceuticals, cigarettes, confectionaries, and graphical uses (Figure 1.2 b).
This paper grade includes multiple layers of chemical and mechanical pulps, with the middle ply furnish often consisting of chemi-thermo mechanical pulp (CTMP) (Figure 1.2 a). CTMP is known to enhancing the bending stiffness of FBB due to its high bulk.

Low consistency (LC) refining technology offers an opportunity to decrease the production cost of CTMP for FBB (and other) products by reducing the refining energy cost. As pointed out earlier, a natural application of this technology could as a replacement of the HC secondary refining stage (Figure 1.3 a). In this case, however, it may be possible to further reduce the refining energy by the chemical pretreatment of wood chips. An alternative scenario for implementing LCR in CTMP production is the refining of reject pulp. Due its higher efficiency and its fibre cutting action, LCR is expected to result in the shortening of long reject fibres while preserving bulk. Figure 1.3b illustrates a potential refining strategy for producing high bulk pulp
containing LCR reject (LCRR) pulp. In this scheme, reject pulp from HC screens enters a LC refiner and is subsequently screened. The accept pulp is either directly withdrawn to obtain a 100% LCRR pulp or blended with the mainline to obtain a HCR/LCRR blend.

Although the above refining strategies promise a significant reduction in the energy cost for the production of market CTMP, their successful implementation requires a systematic investigation of their effects on the pulp quality and product performance.

Figure 1.3 Schematics diagrams of two refining scenarios: (a) LC refining of mainline pulp, and (b) LCR of reject pulp
1.3 Hypothesis and Objectives

The overall objective of this thesis is to examine the application of LC refined CTMP pulp for the production of FBB grades. It is our hypotheses that:

1- The addition of LCR reject pulp to the middle ply of FBB can improve bulk and strength properties.

2- Using chemically pretreated HC-LC refined CTMP pulp in the FBB middle ply could improve FBB properties.

Accordingly, specific objectives of this study are:

1- Examine the effect of LCRR pulp addition to the middle ply furnish on bulk and mechanical properties of FBB.

2- Investigate the influence of sulphite pretreatment of wood chips prior to HC-LC refining on the middle ply furnish quality and FBB properties.

To achieve the above objectives, various board samples were prepared using a laboratory Dynamic Sheet Former (DSF) and were examined for their physical properties. Structure and chemical composition of these samples were also inspected at microscale using scanning electron microscopy and X-ray photo-electron spectroscopy.

1.4 Thesis Structure

The dissertation continues with an overview of the paper properties as well as mechanical refining technology in Chapter 2. The experimental methods utilized in this study are presented in Chapter 3. Chapter 4 contains analysis and in depth discussion of the experimental data. This chapter is divided into two separate parts which correspond to each of the two specific objectives of this project. In the first part, effect of using LCR reject pulp in the middle ply furnish on FBBs properties is investigated, and in the second part, a comparison between physical properties of lab-made 3 ply FBB samples containing chemically pretreated LC refined pulp is presented. Finally, Chapters 5 gives a summary of the contributions as well as recommendations for future research.
2 Literature Review

2.1 Introduction

This section provides the background knowledge for thermo-mechanical pulp (TMP) production and properties as well as low consistency refining process.

2.1.1 Fibre morphology

Wood fibre morphology and its chemical and physical properties depend on the wood species and its growing conditions. Fibre cell wall structure consists of circular layers with different chemical and physical characteristics. Figure 2.1 shows a typical cell wall structure of wood fibre.

Figure 2.1 Schematic representation of cell wall structure of wood fibre [9]

M= middle lamella, P= primary wall, S1= outer layer of secondary wall
S2= secondary wall, S3= inner layer of secondary wall, W= warty layer
The structure of fibre cell wall affects the fibre collapse and paper properties. Middle lamella is the layer that contains the most lignin and is surrounded by primary wall (P layer). The secondary wall (S) consists of outer, middle, and inner layers, namely S1, S2, and S3, respectively. Fibril arrangement in each of the S layers depends on the wood species and growing conditions [10].

Refining changes fibre morphology by cutting and peeling of the fibres. Generally, fibre cutting is not a desirable action however it can promote fines production that improves fibre-fibre bonding by filling the gap between the fibres. In addition, the presence of fines in the pulp furnish enhances the optical properties of paper by increasing the light scattering effects. Peeling, on the other hand, results in the reduction of the cell wall thickness and is caused by the fibre pulling action [11]. Peeling of the outer layer of fibres can also result in the external fibrillation that generally improves paper and board properties by enhancing the bonding strength. Other mechanisms that contribute to the fibre fibrillation include splitting of the inner cell wall that promotes fibre swelling and is referred to as the internal fibrillation, and the molecular fibrillation caused by the dissolving of the polymeric matrix components of the cell wall [12].

### 2.1.2 Physical properties of paperboard

Physical properties of paperboard depend on fibre characteristics, sheet structure, and finishing operations (e.g. coating and calendaring). These properties could be broadly divided into two categories:

1. Mechanical properties including strength, stiffness, compression strength. These properties are typically characterized by measuring tensile strength, stiffness, tearing resistance, ply bond strength, bending stiffness, compression strength, etc.

2. Appearance properties including surface structure, smoothness, and whiteness, printability, coating content, etc. These properties are quantified by measuring surface strength, brightness, whiteness, opacity, surface roughness, gloss, color, ink absorption, etc.

Both mechanical and appearance properties of paperboard depend on basis weight, thickness, density (bulk), sheet structure, fibre orientation, furnish composition. In addition, fibre characteristics such as fibre length, width, coarseness, strength, and stiffness as well as fibre-
fibre bond strength, fines content, and strength additives play important roles in the mechanical performance of board products. Meanwhile, optical properties of furnish (i.e. light absorption and scattering coefficients), filler content, coating weight and coating formulation influence the appearance of board.

The key properties for folding boxboard are bending stiffness, creasing, ply-bond strength and bulk. Bending stiffness and creasing of a FBB indicate its flexibility and the ability to resist the bending stress before rupture. On the other hand, ply bond in multi ply folding boxboard is important to prevent delamination of the plies during printing. High bulk is usually a desirable factor to improve the bending stiffness while using less amount of pulp.

Throughout this dissertation, we use several test methods to evaluate fibre characteristics as well as the board properties. These test methods are described below.

**Bulk**: Bulk is the inverse of sheet density. It is predominantly a function of furnish characteristics, such as fibre diameter, coarseness, transverse stiffness, and bending stiffness. Stiff coarse fibres with large diameters form a bulkier sheet. Bulk is also affected by forming conditions such that more severe wet pressing and calendaring result in a denser sheet with lower bulk. In multi ply folding boxboards, usually a high bulk chemi-thermomechanical pulp is used in the middle ply to impart the desired bulk and enhance the mechanical properties of FBB [13].

**Tensile Strength**: Tensile strength is defined as the tensile stress applied parallel to the specimen surface that causes rupture and failure of the material. Generally for paper products it is commonly expressed in terms of tensile index, i.e. the ratio of tensile strength and the sheet basis weight.

Tensile index is related to the fibre strength, fibre length, and bonding. Usually paperboards containing longer fibre in their furnish have higher tensile index. However, the fibre-fibre bonding is the most important factor contributing to the tensile results [14]. Tensile strength is also affected by the fibre orientation, i.e. more force is required to break a sample in the machine direction or MD (i.e. the direction that paper is made) than in the cross machine direction or CD (i.e. the direction perpendicular to the direction of paper fabrication). Tensile strength is an indication of the paperboard resistance to web breakage during printing and converting operations and also serviceability of the boards that are subjected to the direct tensile stress. Tensile strength could be also expressed in terms of the breaking length, i.e. the length of a
uniform width strip, beyond which, if such a strip were suspended by one end, it would break because of its own weight. Breaking length value varies from 500 meters for weak tissues to more than 8 kilometers for very strong Kraft paperboards [15].

Tensile properties of board is characterized by measuring the applied stress ($\sigma$) versus the strain ($\varepsilon$) of the sample:

\[
\sigma = \frac{F}{A}, \quad \text{Equation 1}
\]

\[
\varepsilon = \frac{\delta}{L}, \quad \text{Equation 2}
\]

$\sigma$ = normal stress on a plane perpendicular to the longitudinal axis of the specimen

F = applied load

A = original cross sectional area of paper

$\varepsilon$ = normal strain in the longitudinal direction

$\delta$ = change in the specimen’s length

L = original length of the sample

![Figure 2.2 An idealized stress-strain diagram, showing the failure point (star) [15]](image-url)
The stress-strain curve is a comprehensive way to represent the tensile test data (Figure 2.2). For example, other paper properties including stretch (or strain at break), modulus of elasticity, and tensile energy absorption (TEA) can be derived from this curve [15]. Stretch is the maximum tensile strain developed in the test specimen before rupture. In packaging papers, stretch indicates their performance under dynamic or repetitive straining and stressing conditions. According to Page’s equation, tensile strength is affected by both fibre properties and sheet characteristics [14].

\[
\frac{1}{T} = \frac{9}{8Z} + \frac{12 g C}{b P L (RBA)}
\]

Equation 3

Where \( T \) is the tensile strength (km), \( Z \) is the zero span breaking length of fibres (km), \( C \) is the fibre coarseness (kg/m), \( g \) is the gravitational acceleration (m/s\(^2\)), \( P \) is the perimeter of the fibre cross section (m), \( L \) is the length weighted average fibre length (m), \( b \) is the bond strength (Pa), and RBA is the relative bonded area that is estimated by [14]:

\[
RBA = (S_0 - S)/S_0
\]

Equation 4

\( S_0 \) and \( S \) are the optical scattering coefficients of the sheet and that of unbonded fibres. Equivalently, \( S_0 \) can be interpreted as the specific surface area of dry fibers and \( S \) as the specific surface area of the sheet (g/m\(^2\)).

Based on equation 3, long and strong fibres with large diameter and low coarseness will produce sheets with a higher tensile strength. Similarly, larger fibre-fibre bond strength and sheets with a greater RBA will have a higher tensile strength. Fibre-fibre bond strength is determined by molecular interactions and hence fibre surface chemistry at fibre-fibre joints, however, it is also affected by the fines content and the presence of strength additives. RBA is a measure of fibre-fibre contact in the sheet and can be improved by wet pressing.

Page’s equation is not valid for fibres with curl or kink. Curly fibres, for example, can improve the stretch to a significant extent, because they have to be straightened before carrying the applied load [16]. These fibres, however, have been reported to have negative influence on the tensile strength [17]. Fibre axial stiffness also affects the stretching properties of paper and board. Lower axial stiffness results in a higher fibre elongation under the applied load and hence
increases the stretch. Similarly, fibre orientation can affect the stretch because fibres are more readily stretched in their axial direction.

*Elastic Modulus* or *Young's Modulus* is the slope of stress-strain curve during the elastic deformation of the sample. Within the elastic region, load is a linear function of the deformation, and moreover by removing the applied load, sample will return to its original state. Earlier studies have shown that elastic modulus of sheet is a function of fibre length and width, elastic and shear moduli of fibre, and fibre-fibre bonding:

\[
E_p = \frac{1}{3} E_f \left( 1 - \frac{w}{LRBA} \sqrt{\frac{E_f}{2G_f}} \frac{w}{LRBA} \sqrt{\frac{2G_f}{E_f}} \right)
\]

Equation 5

Where \( E_p \) is the elastic modulus of paper, \( E_f \) is the axial elastic modulus of a fibre, \( G_f \) is the shear modulus of a fibre, \( w \) is the mean fibre width, \( L \) is the mean fibre length, and \( RBA \) is the relative bonded area [18]. Based on this equation, long fibres with high elastic and shear moduli and low width will produce sheets with a higher elastic modulus. In addition, according to this equation, increasing fibre-fibre bonded area will increase the elastic modulus of the sample. Beyond the elastic region, stress is no longer proportional to strain. In this case, by removing the applied load, sample will exhibit a permanent plastic deformation which is the main reason of misregister in web printing [13].

In addition to the above information, stress-strain curve can be used to calculate the *Tensile energy absorption (TEA)* i.e. the work required to stretch a sample to the point of rupture. TEA is determined by integrating the area under the stress-strain curve, hence higher stretch and higher elastic modulus result in a higher TEA. This parameter is an indicative of the ability of a sample to withstand a shock when subjected to sudden high tension. It also reflects the performance of a paperboard under dynamic and repetitive forces. High TEA is an important factor for multiwall bag papers evaluation due to their high stretchability requirements [19].

*Tear Strength*: Tear strength represents the resistance of a sample to tearing force, i.e. a force that is perpendicular to the plane of the sample. Tear index of a sample is calculated by dividing the tear strength of the specimen by its basis weight. In general, high tear strength is required for almost every packaging applications especially when the package is to be opened and closed several times, however low tear strength is desirable for pull tabs using as the openings [13].
Tear strength depends on several factors including fibre length, fibre strength and fibre bonding. Using a micromechanical model, Yan and Kortschot proposed the following expression for the tear strength of paper [20]:

\[
\text{Work} = \begin{cases} 
\frac{WR}{\pi \omega} \left[ \frac{\tau_f A_b L^2}{9 L_f} + \frac{\tau_f^2 A_b^2 L^3}{18 L_f^2 A_f E_f} \right] & \text{if } L_{\text{max}} \leq 2L_c \\
\frac{WR}{\pi \omega} \left[ \frac{\tau_f \sigma_f^3 A_f^3 L_f^2}{9 \tau_b^2 A_b L} + \frac{\tau_f \sigma_f^3 A_f^3 L_f^2}{3 \tau_b^2 A_b L} \ln \left( \frac{\tau_b A_b L}{9 \sigma_f A_f L_f} \right) + \frac{\sigma_f^2 A_f L}{6 \tau_b^2 A_b E_f L} - \frac{4 \sigma_f^2 A_f^2 L_f}{9 E_f L \tau_b A_b} + \frac{\sigma_f^2 A_f L}{3 E_f} \right] & \text{if } L_{\text{max}} > 2L_c 
\end{cases}
\]

Equation 6

Here, \( W \) is the basis weight, \( R \) is the width of the sheet, \( \bar{L} \) is the mean fibre length, \( L_{\text{max}} \) is the maximum fibre length, \( L_f \) is the free fibre length, \( A_f \) is the fibre cross section, \( A_b \) is the fibre-fibre bonded area, \( \omega \) is fibre coarseness, \( \tau_f \) is the frictional stress, \( \tau_b \) is the bond strength, \( E_f \) is fibre modulus, and \( \sigma_f \) is the fibre strength, \( L_c \) is the critical length of fibre above which some of the fibres may break during the tearing process. In addition to the above parameters, the presence of curly fibres is known to improve the tear index due to the stress distribution along the length of these fibres. Curled fibres tend to be pulled out more than being torn, hence the paperboard containing more curly fibres have high tear index but low tensile index [21]. Fibre orientation also affects the tearing resistance. Tear strength is generally larger in the CD than in the MD. Tearing resistance in usually used as a control parameter in board manufacturing, because it is an indication of fibre quality, including the fibre length and curliness as well as the refining level of fibres in terms of fibre bonding.

**Burst:** Burst strength represents the pressure at which a sample will rupture (Figure 2.3). Burst strength is a complex property that is affected by several other physical properties such as tensile strength, tensile strain and shear characteristics of the material. Hence, a high burst strength is an indication of a strong sample that is able to tolerate different types of applied stresses. Burst strength depends on the basis weight, furnish type, sheet formation and the use of strength additives [13]. Burst strength has been proposed to be proportional to average of MD and CD tensile strength values and the square root of strain in the machine direction. Assuming that paper obeys a linear anisotropic stress-strain law, burst strength can be determined from uniaxial tensile measurements in CD and MD directions according to equation 7 [15]:

\[ B = \frac{R}{\pi} \left[ \frac{\tau_f A_b L^2}{9 L_f} + \frac{\tau_f^2 A_b^2 L^3}{18 L_f^2 A_f E_f} \right] \]

12
\[ P_{\text{max}} = \frac{1}{3} \sqrt{6 \frac{h}{a}} \left( \sigma_{M,u} + \sqrt{\frac{\varepsilon_{M,u}}{\varepsilon_{C,u}}} \sigma_{C,u} \right) \sqrt{\varepsilon_{M,u}} \]  

Equation 7

Where \( \varepsilon_{M,u} \) and \( \varepsilon_{C,u} \) are the MD and CD strain at break and \( \sigma_{M,u} \), \( \sigma_{C,u} \) are MD and CD stress at break determined from MD and CD uniaxial tensile tests, \( a \) is the radius of circular boundary of clamp in burst test, and \( h \) is the thickness of the sheet.

Burst index is calculated by dividing the burst strength by the basis weight of the specimen. Nevertheless, burst index is found to depend on the basis weight, due to the effect of basis weight on sheet formation. Comparing the same basis weight multi-ply and a single ply boards indicated that burst index is higher with increasing the number of plies due to their higher stiffness and higher force requirement for deformation [22].

![Burst test](image)

Figure 2.3 bursting resistance test [7]

**Bending stiffness:** Bending stiffness is the ability of a sample to resist bending deformation. For packaging materials such as FBB, bending stiffness represents how well the packaged goods are protected from potential damages during storage, shipping and handling. Fibres characteristics such as stiffness, length and dislocation as well as bond strength affect the bending stiffness. Long stiff fibres increase bending stiffness while fibres with dislocation are bent more readily and hence decrease the bending stiffness [23].

Bending stiffness can be determined by measuring the bending deformation of a sample subjected to a bending force. In the two points method, the bending stiffness can be determined from the deformation, \( \Delta \) [24]:

\[ S_b = \frac{F \times L^3}{\Delta \times 3 \times b} \]  

Equation 8
Where,

\( S_b = \text{bending stiffness (N.m)} \)

\( F = \text{Force (N)} \)

\( L = \text{length (m)} \)

\( b = \text{width (m)} \)

\( \Delta = \text{the maximum deflection (m)} \)

Figure 2.4 Loading principle and beam conditions in two-point condition

It can be shown that the bending stiffness is related to the elastic modulus of the material according to:

\[
S_b = \frac{EI}{b} = \frac{E_t}{12} \]

Equation 9

In the case of a multi-ply board, the thickness of moduli of each layer as well as their position relative to the neutral plane of the board affects the overall bending stiffness of the sheet. In a special case of a symmetrical three layer structure where top and bottom layer have the same thickness and composition, the bending stiffness of the sheet is given by:

\[
S_b = \frac{E_2(t_2^3-t_1^3)}{12} + \frac{E_1(t_1^3)}{12} \]

Equation 10

Where \( E_1 \) and \( E_2 \) are the moduli of middle ply and outer ply of the board and \( t_1 \) and \( t_2 \) are their respective thicknesses.
From the above equations, the bending stiffness is a strong function of thickness. Hence, thickness (or equivalently bulk) is one of the most important factors affecting the rigidity of the sample.

The multi-ply structure of FBB also helps to improve the bending stiffness of board. During bending of a multi-ply board, the convex side is subjected to tensile stress while the concave side experiences compression (Figure 2.5). Therefore, using chemical pulp with long fibres can enhance the tensile and compression stiffness of the surface plies and improve the bending stiffness of FBB [13].

In packaging grades, bending stiffness is a key factor for paperboard performance. For example, solid bleached board (SBB) stiffness is relatively high per unit of their basis weight, however FBBs have higher stiffness while they are bulkier than SBBs.

**Figure 2.5 stress distribution during bending of three-ply board**

*Box compression strength:* Compression strength represents the ability of the packaging material to resist crushing due to stacking, transportation, and converting operations. Short span compression (SCT) is an established method for measuring the compression strength of paperboards that is based on applying a longitudinal compression force to the sample clamped at two sides (Figure 2.6).

Under compression stress, fibre network behaves like supporting columns, and start to bend and buckle until failure occurs [13]. Shallhorn et al., found that SCT of low and intermediate density boards (300-450 kg/m³) improved by increasing fibre elastic modulus, fibre wall thickness, and density and decreased by increasing fibre width and coarseness. For high density sheets (500-850
kg/m$^3$), however, the most dominant factor was axial compressive strength of fibres. They suggested that for paper made of ribbon-like fibres the SCT could be estimated from [25]:

$$\frac{1}{\sigma_c} = \frac{1}{\sigma_o} + \left[ \frac{C_w}{(2\alpha E_f t^3)} \right] \left( \frac{\rho_f}{\rho} - 1 \right)^2$$

Equation 11

Where $\sigma_c$ is the sheet compressive strength, $\sigma_o$ is the sheet compressive strength at limiting high intensity, $w$ is fibre width, $t$ is fibre thickness, $E_f$ is fibre modulus, $C$ is fibre coarseness, $\rho$ is the sheet density, $\rho_f$ is the fibre density, and $\alpha$ is an efficiency factor representing the inelastic buckling of fibres.

Figure 2.6 Compression loading on the sample in SCT test [25]

Initiation of the compression failure is typically attributed to the buckling of fibre segments. Using small deflection theory, the critical axial buckling stress, $\sigma_c$, can be expressed in term of fibre diameter, $w$, cell wall thickness, $t$, fibre elastic modulus $E_f$, and Poisson’s ratio of fibre, $\mu$ [26]:

$$\sigma_c = \frac{E_f t}{w \sqrt{3 (1-\mu^2)}}$$

Equation 12

Therefore, increasing sheet density, fibre modulus, fibre diameter and lumen diameter will increase critical buckling load of the fibre segments.

*Internal bond strength and ply bond strength:* The internal bond represents the degree of bonding between the fibres in a single ply sheet. A poor internal bond strength results in sheet splitting and delamination during the printing and coating processes. Internal bond strength is commonly determined using the Scott bond test that is a measure of the energy required to delaminate the sample [11]. Scott bond strength depends on fibre-fibre bond strength as well as the relative
bonded area. Scott bond strength can be improved by increasing sheet density (i.e. decreasing bulk) using higher wet pressing pressures by increasing the fines content and fibre flexibility through increasing the refining energy. For example, it has been reported that high density sheets made of Kraft pulp and low density samples made of TMP pulp had similar bond strength due to the presence of fibrils on the surface of mechanical pulp fibres as result of refining process [27].

2.1.3 Microscopic studies of paper and paperboard

Structure of paper and board is affected by furnish as well as pulping and papermaking conditions. Several microscopy techniques have been used in the literature to study the structure of paper and board. These techniques include Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS), Attenuated Total Reflectance Infrared Spectroscopy (ATR-IR), Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) and Atomic Force Microscopy (AFM). In this section, an outline of the principles and possibilities of the key instrumental techniques, SEM and XPS applied in this study is provided.

For example, in Figure 2.7 the effect of refining on fibre morphology, fine production, internal fibrillation, and external fibrillation can be seen. Similarly, using SEM, Sandin found that increasing the refining revolution of spruce CTMP resulted in increasing the fibrils and fine in the furnish. The pulp was refined mechanically in a PFI mill. Increasing the number of PFI mill revolutions is an indication of more refining of the pulp [28].

Figure 2.7 SEM pictures of fibres: a) Internal fibrillation, b) external fibrillation, and c) fine formation [29]
Other researchers used X-ray photoelectron spectroscopy to examine the surface chemistry of paper and paperboard. Dorris et al. [30] reported that the XPS spectra of wood fibre products consisted of C1s and O1s peaks. In general, the binding energy of C1s and its related peak could be due to various carbon bonding state, namely C1, C2, C3 and C4. C1 represents carbon linked to hydrogen or carbon (\(-\text{C-H} \text{ or } \text{C-C}\)), while C2 corresponds to carbon linked to a single oxygen (\(-\text{C-O}\)), C3 indicates carbonyl oxygen (O-C-O) or to two non-carbonyl oxygen (\(-\text{C}=\text{O}\)), whereas C4 can be attributed to a carbonyl or non-carbonyl (O-C=O) group. C4 does not appear in wood-derived material because the concentration of carboxyl groups on the pulp fibre surface is very low and undetectable by XPS [31]. C1 mainly shows the lignin and extractives content, while C2 and C3 contribute to carbohydrates. In the case of cellulose, XPS confirms that there are 83% C2, 17% C3 and 0% C1. Hemicellulose has similar peaks however extractives have the highest C1 percentage, i.e. 95%. On the other hand, lignin XPS spectra has about 50% C1, 48% C2, and 2% C3 [30]. The binding energy related to C1, C2, and C3 for cellulose are 285.0, 286.5, and 288.3 eV, respectively (Figure 2.9).
2.1.4 LC refining of pulp

A typical TMP process with LC refining includes the following stages: heating the prescreened and washed wood chips with steam, primary high consistency (20-40%) refining to convert the wood chips into individual fibres, secondary high consistency refining for further fiber development, and tertiary LC refining at 3-4% consistency. In this process, over 80% of the total energy consumed in the pulp mill is used up by the refiners. 75% of the total refining energy is devoted to HC refiners, 21% is used in the reject refiners, and only 4% of this energy is consumed by the LC refiners. Therefore, a shift toward the replacement of HC refining by LC refining is believed to result in significant energy saving in the mechanical pulping process.

The reason for higher energy efficiency of LCR in comparison to HCR is not well understood, however, it is believed to be caused by the differences in the fluid mechanics of pulp slurry in the refiners. HC refining works with a heterogeneous suspension that consists of three phase, namely wood fiber, steam, and water. This three phase mixture moves through the refiner disk by the motive power comes from feed screws.

On the other hand, in LC refining, pulp suspension is more homogenous and incompressible, and it is pumped through the refiner by an external force. In addition, the gap between the LC
refiner plates is smaller in comparison with the HC refiner, hence the power consumption is more stable. During LC refining, no steam will be produced and as a consequence there is sufficient water surrounding the fibers. The surrounding water transfers the refiner disks’ force to the fibre flocs. The imposed force leads to fibre- fibre contact, fibre lumen compression, and fibre swelling and internal and external fibrillation [33].

Morphological studies of HC and LC refined pulps have revealed significant differences in the fibre development between these processes. HC refining is known to reduce the fibre wall thickness and improve the external fibrillation while preserving the fibre lengths. On the other hand, LC refining causes more damage to the fibres that leads to more fiber cutting [4], [34]. It has been also reported that LC refining produces more straight and flexible fibres while HC refined pulp fibres tends to be more curled [35].

Klinga et al. and Kang et al. [36], [37], reported that at the same specific energy LC refining pulp has lower freeness values and shorter fiber length. Klinga et al. also concluded that LCR pulp results in higher tensile index and Z strength. Similar results were reported by Clark [38] and also Hafren et al. [39] who have shown the higher tensile strength for LC pulp than HC refined pulp. However according to Clark’s study LCR pulp had lower tear index than the HC refined one at the same given specific energy.

2.1.4.1 LC Refining conditions

Refining affects fiber characteristics by fibre cutting, fines formation, internal changes in fiber walls structure such as internal fibrillation, swelling and delamination. It also causes external fibrillation, curling or straightening the fibers. As a result of the above effects, refining makes fibers more flexible and improves fibre-fibre bonding hence increases the sheet density and mechanical strength. In addition, refining reduces the freeness, bulk and air permeability [40].

The amount of energy used in the refining process is characterized by the specific energy consumption (SE) expressed in terms of kWh or MJ per tones of pulp. Required energy for LC refining is consumed in two major parts: most of the energy is consumed for refining process or in other word for improving pulp properties. However, 20-50% of the total energy is used for overcoming the hydraulic and mechanical losses that is referred to “no-load” power. No-load power is defined as the required energy to operate the device when the gap size between the rotor
and stator refiner disks is large enough to eliminate all refining effects. The no-load power, $P_{\text{no-load}}$, indicates a threshold power beyond which fiber morphology will change. Other researchers defined no load power as the minimum energy required for rotating the rotator disk in pulp solution. Although both definitions are correct the former is commonly used in the industry [41].

Hence, SE is obtained as follows:

$$SE = \frac{P_{\text{total}} - P_{\text{no-load}}}{c \times F} = \frac{P_{\text{net}}}{m_{\text{fib}}},$$  \hspace{1cm} \text{Equation 13}$$

Where, $m_{\text{fib}}$ is the mass flow of dry fiber through the refiner, $c$ is pulp consistency in the feed, $F$ is the feed flow rate, $P_{\text{net}}$ is the net power delivered to the pulp in the refiner, and $P_{\text{total}}$ is the total power delivered to the refiner.

In addition to SE, refining intensity, i.e. the refining impacts, can significantly affect the pulp quality. Refining intensity depends on several variables including flow rate, specific energy, rotation speed and geometry of refiner disks, and gap size, and is commonly evaluated by the specific edge load (SEL) in J/m [41]:

$$\text{SEL} = \frac{P_{\text{net}}}{Z_{\text{st}} Z_{r} l_{c} n} = \frac{P_{\text{net}}}{L_{s}}, \hspace{1cm} \text{Equation 14}$$

Where $Z_{\text{st}}$ and $Z_{r}$ are the number of rotor and stator bars, $l_{c}$ is the common contact length of opposing bars (m), $n$ is the refiner disk speed (1/s), and $L_{s}$ is the cutting speed (m).

Roux and Joris [42] suggested that the above equation does not accurately take into account the effects of plate geometry on the refining outcome. Instead, based on bar width (B), groove width (W), inner radius ($R_{i}$), outer radius ($R_{o}$) (Figure 2.10) they defined Bar Edge Length (BEL) in km/rev by:

$$BEL = (2\pi)^{3} \frac{R_{o}^{3} - R_{i}^{3}}{3(W + B)^{2}}, \hspace{1cm} \text{Equation 15}$$
Accordingly, SEL can be expressed by [43]:

\[
\text{SEL} = \frac{P_{\text{net}}}{n.\text{BEL}} = \frac{P_{\text{total}} - P_{\text{no-load}}}{n.\text{BEL}}
\]

Equation 16

2.1.5 Chemical pretreatment

Wood chips usually are exposed to a pretreatment in mechanical pulping process. This action is done to soften the wood chips before refining and as a consequence enhance the refining and reduce the cost and energy requirement of the process. Most common pretreatments involve exposing wood chips to steam or chemicals. The former preconditioning is part of the conventional TMP process while the latter is referred to as the chemi-thermo-mechanical pulp or CTMP [44].

One approach for reducing the use of electrical energy in the TMP process is chip pre-treatment prior to refining. Various types of pre-treatment techniques have been evaluated throughout the history of TMP, including: mechanical pre-treatment (Frazier and Williams 1982) [45], sulphite pre-treatment (Atack et al. 1978) [46], alkaline hydrogen peroxide pre-treatment (Bohn and Sferrazza 1989) [47], oxalic acid pre-treatment (Akhtar et al. 2002) [48], enzymatic pretreatment (Peng et al. 2005) [49] and others.

Various chemical agents can be used for the pretreatment in CTMP process including sodium hydroxide, alkaline hydrogen peroxide [47], sodium sulphite [46], peracetic acid [50], sulfuric acid, and enzymes [49]. These chemicals affect cellulose, hemicellulose, lignin, and the other fibre contents through various pathways such as hydrolysis with alkali which generates new carboxylic groups, hydroxylation with peracetic acid which can attach new hydroxyl groups onto
the lignin structure, lignin oxidation by adding peroxide, and sulphonation by sodium sulphite which adds sulphonic groups onto the lignin structure [51].

Among the above chemical pretreatment methods, the most common ones are alkali-peroxide, hydrogen peroxide, and sulphonation. The alkali peroxide treatment includes using either sodium peroxide or hydrogen peroxide. Generally, alkali peroxide treatment breaks down the lignin molecular structure through the hydrolysis and oxidation reactions and causes delignification. While, pretreatment with hydrogen peroxide results in lower delignification and xylan solubilization. In the sulphonation pretreatment sodium sulphite reacts with the lignin network and breaks down its molecular structure, resulting in a pulp with good bleachability [52], [53].

Yuan et al. [54] investigated the effect sulphonation with and without addition of NaOH on Aspen fibre morphology and surface chemistry as well as pulp properties. They found that chemical impregnation resulted in more long fibres and less shives in the pulp which led to higher pulp brightness. Comparing chips pretreatment with sulphite and with only water (no-chemical), showed that refining with sulphite requires more energy to a given freeness and results in lower tensile index. However, combined NaOH and sulphite pretreatment resulted in the least energy requirement in the refining stage and significantly increased tensile index, but also decreased the bulk due to increase in fibre flexibility. Xu [55] conducted a study to compare alkali peroxide pretreatment and sulphonation and reported that with alkali peroxide higher brightness and tear strength were obtained. In studying the comparison of sulphonation and hydrogen peroxide pretreatment on hardwood pulps, Francis et al. [56] found that both of the pretreatments had similar effect on the tensile index, however, H$_2$O$_2$ caused higher brightness and increased the light scattering coefficient.

Although there are several studies on chemical pretreatment of various pulps there are few literatures on the effect of chemical pretreatment on LC refining process. Xu, compared the LC and HC refined pulps pretreated by alkaline peroxide and found that the tensile strength of the pretreated LC pulp was higher than that of pretreated HC pulp at a given specific energy. However, he also reported that refining the treated pulp at LC resulted in a lower tear strength comparing with the HC treated results [57]. Trocki studied the effect of alkaline peroxide pretreatment on the LCR pulp. In his work, whole log Hemlock chips were refined at HC and were subsequently treated by adding alkaline peroxide before being LC refined. He reported that
chemical pretreatment of the pulp yielded in refining acceleration that led to lower refining energy requirement to achieve the desired sheet strength [58].

2.1.6 Reject pulp

In mechanical pulping process, the first stage of refining is followed by a screening process to separate the dirt, shives, fibre bundles and long fibres. Fines and smaller fibres pass this screen and the remaining fraction, known as rejects, is separated. In a TMP process, about 20 to 50% of pulp is separated in the screening process as reject pulp and is sent to reject refiners for further refining. The refined reject pulp then gets feed back to the mainline for using as final production.

The fractionation choices available today are limited to pressure screening and centrifugal cleaning. Pressure screening which is more popular in mills, separate the fibres according to their fibre length and also their flexibility, whereas it has been suggested that centrifugal cleaning fractionate pulp regarding to their specific surface and cell wall thickness [59][60].

Reject pulp has higher proportion of long and coarse fibres but lower fines content. Long fibres of refined reject pulp result in higher tensile strength however due to the presence of coarse fibres it is expected that the surface properties such as smoothness and opacity can be deteriorated. Gao et al. reported that increasing reject refined pulp in a TMP furnish from 0% to 100% improved the tensile strength of the TMP sheet. However, higher reject refined pulp did not have a significant effect on smoothness and opacity which indicates that refining of reject pulp can overcome the surface properties deterioration as the result of using coarse reject pulp [61].

High proportion of coarse and long fibres in the reject pulp provides a great potential for the LC refining process due to the higher loadability and hence higher tensile index of the long fibres. Andersson et al. [62] stated that the potential for energy saving of long and coarse fibre enriched pulp is greater because the higher rupture strength of these fibres. He also reported that increasing long fibre concentration resulted in forming a stronger fibre network that led to the possibility of applying larger refining gap at a given specific energy. A small gap size in refiner extensively shortens the fibres and causes tensile strength reduction. Hence, larger gaps can improve the tensile index without fibre cutting [5]. Such a refining condition is considered to be the preferred way for balancing between fibre development and sheet strength properties [63].
Inspired by the chemical pretreatment of pulp, Bian et al. run a complete set of experiments to evaluate the effect of alkaline peroxide pretreatment on TMP reject pulp. They found that by using 5.9% alkaline peroxide the specific refining energy decreased up to 20% for a given freeness while tensile index was even improved. That is because of the higher fibre flexibility due to the chemical pretreatment [64]. Similar tensile improvement was reported by Lehto et al. who studied the effect of sulphonation prior to TMP reject refining. According to the fibre length results they could optimize the pulp to have high fibre length and low coarseness simultaneously by using gentle multi stages refining. They claimed that the tensile and Scott bond strength approached to those of chemical pulp whereas, tear index was far lower than chemical pulp values. [65].

2.2 Summary

Mechanical pulping is an outstanding pulping method due to the high yield, however high energy consumption in mechanical pulping limits its application in paper product manufacturing. Therefore, development of suitable technologies to reduce the required energy in mechanical pulping is one the priorities in mills. The most common technologies using to reduce the mechanical refining energy consumption are replacing HC by LC refining and pretreating wood chips with various chemicals. Although effects of these technologies are well studies on refined pulp characteristics and paper properties, it is not well known regarding their effect on reject pulp and application of the obtained pulp in producing multiply FBBs.

Chemical pretreatment of wood chips accelerates the refining due to the wood softening, however, using some specified chemicals such as alkaline, hydrogen peroxide, sulphite, etc. can improve the mechanical strength of the paper product while saving energy during the pulp refining. Combining chemical pretreatment of pulp following by LC refining leads to less refining energy requirement to obtain a specified physical strength.

LC refining of reject pulp is promising due to the higher fibre length of the rejects. Also, coarse fibres presenting in the rejects can be soften by using LC refining. Higher fines production during the LC refining can improve the paper product mechanical strength by increasing the relative bonded area of the fibres.
The key parameters that affect different paperboard mechanical strength, are fibre strength, fibre-fibre bonding, fibre orientation, and fibre length. In particular, according to the theoretical equations, some paperboard characteristics can be estimated by knowing these data as well as some physical strength of the boards including tensile properties.
3 Methodology

3.1 LC refining of pulp

According to the objectives of this study, two type of pulps were used for LC refining, 1) a commercial CTMP reject pulp, and 2) a sulphite pretreated pulp prepared in a pilot facility.

3.1.1 LCR of reject pulp

The reject pulp sample was a radiata pine CTMP provided by Winstone Pulp International (WPI) (Figure 3.1). The pulp was collected from the screen reject after the first HC refiner and was shipped to the Pulp and Paper Centre at the University of British Columbia for LC refining. The LC refining were performed using various gap size, plate geometries, input power values, refining intensity, and rotational speeds. Two sets of refining trials were performed on this pulp sample. In Phase I, the effect of freeness on the properties of middle ply was investigated, while in the Phase II the reject pulp sample was refined to a target freeness value and the effect of middle ply composition on the performance of a three-ply FBB was examined. Details of these refining trails are discussed below:

1) Phase I: In this phase, the radiata reject pulp was LC refined at two different intensities by changing the refiner plates, (Table 3-1). Selected plates were different in terms of their BEL; higher BEL value is related to more bars number of rotor and stator and leads to lower SEL values (Equation 16). Pulp was recirculated through the refiner and samples were collected after each pass. By increasing the number of passes, pulp was subjected to increased specific energy levels and freeness was progressively decreased (Table 3-1).

![Figure 3.1 Reject pulp sample as received](image-url)
Table 3-1 Refining conditions for Phase I

<table>
<thead>
<tr>
<th>Refiner Disk</th>
<th>BEL (km/rev)</th>
<th>SEL (J/m)</th>
<th>LCR pulp freeness (mL)</th>
<th>Pulp Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disk No. 1</td>
<td>5.59</td>
<td>0.32</td>
<td>715</td>
<td>SB0 (No load)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>624</td>
<td>SB1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>533</td>
<td>SB2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>375</td>
<td>SB3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>245</td>
<td>SB4</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>155</td>
<td>SB5</td>
</tr>
<tr>
<td>Disk No. 2</td>
<td>2.74</td>
<td>0.64</td>
<td>680</td>
<td>SA0 (No load)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>545</td>
<td>SA1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>416</td>
<td>SA2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>293</td>
<td>SA3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>176</td>
<td>SA4</td>
</tr>
</tbody>
</table>

Figure 3.2 Two refining disk: (a) BEL: 2.74, (b) BEL: 5.59 (km/rev)
In addition to LCR reject pulp, commercial aspen, spruce and radiata pine chemi- thermo mechanical pulp (BCTMP) were obtained and were used in the middle ply furnish.

Pulp properties including freeness and fibre characteristics; such as length, width and coarseness, were measured at the University of British Columbia (UBC) Pulp and Paper Center. The LC refined pulp was shipped to FPInnovations to make the middle ply of the FBBs. The furnish that was used for this phase was 25% of the LC refined reject pulp mixed with 25% aspen, 25% spruce, and 25% commercial radiata pine.

2) Phase II: For these experiments, LC refining trials were performed using tank-to-tank (i.e. one-pass) configuration at two different refining conditions, Table 3-2. Two samples with freeness of about 300 mL were prepared for board making experiments. LCR radiata reject pulp was combined with the three other commercial pulps with different ratios to evaluate the effect of using different amount of reject pulp on mechanical properties of folding boards.

As the control samples boards with 100% of each mentioned commercial BCTMPs for the middle ply and the same chemical pulp for top and bottom ply were made.

LCR reject pulp was combined with the three other commercial pulps with different ratios to evaluate the effect of using different amount of reject pulp on mechanical properties of folding boards.

<table>
<thead>
<tr>
<th>Refining Trial</th>
<th>Power (kW)</th>
<th>Speed (RPM)</th>
<th>BEL (km/rev)</th>
<th>SEL (J/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A4-T4</td>
<td>25</td>
<td>1200</td>
<td>2.74</td>
<td>0.45</td>
</tr>
<tr>
<td>A5-T5</td>
<td>25</td>
<td>1600</td>
<td>2.74</td>
<td>0.35</td>
</tr>
</tbody>
</table>

3.1.2 LCR of chemical pretreatment pulp

Spruce-Pine-Fir (SPF) wood chips provided by Quesnel River Pulp (QRP) were refined at Andritz’ pilot plant facility in Springfield, OH. Part of the wood chips was chemically treated by sulphite (labeled as “sulphite”) prior to refining. Chemical pretreatment conditions are listed in Table 3-3. Wood chips with and without chemical pretreatment (labeled as “no chem.”) were
pre-steamed and refined at high consistency of 35% using the ANDRITZ 36” pressurized single disc refiner (36-1CP refiner equipped with refiner plate pattern 36SA002). The refiner was operated with a pressure of 3.1 bar at 2300 rpm. The HC refined pulp samples were shipped to UBC pilot facility for LCR trials.

Table 3-3 Chemical Pretreatment condition of wood chips

<table>
<thead>
<tr>
<th>Furnish</th>
<th>Chemical treatment*</th>
<th>Steam pressure</th>
<th>RT pressafiner retention time</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPF</td>
<td>No-chem</td>
<td>20 psi</td>
<td>15 sec</td>
</tr>
<tr>
<td>SPF</td>
<td>Sulphite 2%, pH 8.4</td>
<td>20 psi</td>
<td>15 sec</td>
</tr>
</tbody>
</table>

*Stabilizers were added. DTPA (0.07-0.08%) in the sulphite treatments

For LC refining, pulp samples were diluted to 3.5% consistency and refined at 1200 RPM and 250 L/min to a target freeness of 400 mL. Each pulp was refined at two SEL values, namely 0.25 J/m and 0.60 J/m. These intensity levels were achieved by changing the plate pattern with BEL values of 5.59 km/rev and 2.74 km/rev, respectively, and by closing the refiner gap to achieve target power. Considering various refining conditions used in these trials, five pulp samples were selected for board making experiments as listed in Table 3-4.

Table 3-4 LC refining trials for chemical pretreatment experiments. 1200 rpm, 250 L/min. Sum SRE is the total specific refining energy

<table>
<thead>
<tr>
<th>Trial #</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre-treatment</td>
<td>No-chem</td>
<td>No-chem</td>
<td>No-chem</td>
<td>Sulphite</td>
<td>Sulphite</td>
</tr>
<tr>
<td>SEL (J/m)</td>
<td>0.25</td>
<td>0.25</td>
<td>0.6</td>
<td>0.25</td>
<td>0.6</td>
</tr>
<tr>
<td>BEL</td>
<td>5.59</td>
<td>5.59</td>
<td>2.74</td>
<td>5.59</td>
<td>2.74</td>
</tr>
<tr>
<td>Target HC Freeness (mL)</td>
<td>500</td>
<td>600</td>
<td>600</td>
<td>600</td>
<td>600</td>
</tr>
<tr>
<td>Actual HC Freeness (mL)</td>
<td>305</td>
<td>523</td>
<td>583</td>
<td>533</td>
<td>562</td>
</tr>
<tr>
<td>Target LC Freeness (mL)</td>
<td>400</td>
<td>400</td>
<td>400</td>
<td>400</td>
<td>400</td>
</tr>
<tr>
<td>Actual LC Freeness (mL)</td>
<td>214</td>
<td>402</td>
<td>402</td>
<td>462</td>
<td>366</td>
</tr>
<tr>
<td>Sum SRE (kWh/t)</td>
<td>55.91</td>
<td>47.63</td>
<td>64.02</td>
<td>52.89</td>
<td>67.50</td>
</tr>
<tr>
<td>Net power (kW)</td>
<td>27.09</td>
<td>27.52</td>
<td>35.62</td>
<td>27.62</td>
<td>32.78</td>
</tr>
</tbody>
</table>
3.1.3  Fibre Characterization

Fibre length, curl, width, kink, and coarseness are determined using Fibre Quality Analyzer (FQA, OpTest Equipment, Canada) at UBC Pulp and Paper Centre. In this equipment, a low consistency pulp sample passes through a high resolution imaging system. Multiple images are acquired to obtain distributions for various fibre characteristics. Both arithmetic average length and length weighted as well as weight-weighted fibre length are determined during the measurement:

\[
L_n = \frac{\sum n_i l_i}{\sum n_i} \quad \text{Equation 17}
\]

\[
L_l = \frac{\sum n_i l_i^2}{\sum n_i l_i} \quad \text{Equation 18}
\]

\[
L_w = \frac{\sum w_i l_i}{\sum w_i} \quad \text{Equation 19}
\]

Where, \( L_n \) = numerical average length

\( L_l \) = length-weight average length

\( L_w \) = weight-weight average length

\( n_i \) = number of fibres in the \( i \)th class

\( l_i \) = mean length of the \( i \)th class

\( w_i \) = weight (or mass) of fibres in the \( i \)th class

Curl is the gradual and continuous curvature of a fibre while kink represents the abrupt change in the curvature of a fibre (see Figure 3.3). Curl index is defined as:

\[
CI = \frac{l}{L} - 1 \quad \text{Equation 20}
\]

Where, CI is the curl index, \( l \) is fibre contour length, and \( L \) is the longest dimension.

Kink index is defined as the weighted sum of the number, \( N_{(x,y)} \), of kinks with angles between \( x \) and \( y \):

\[
\text{Kink index} = \left[ 2N_{(21-45)} + 3N_{(46-90)} + 4N_{(91-180)} \right] / L_{TOTAL} \quad \text{Equation 21}
\]
Here, $L_{\text{total}}$ is the total length of fibres used in the measurement.

In addition to the above parameters, FQA also measures the fines content in the pulp sample.

![Figure 3.3 Definition of fibre curl and kink](image)

3.2 Board making

Board making was performed at FPInnovations facility in Montreal using the Dynamic Sheet Former (DSF), originally made by Noram and modified by FPInnovations. Prior to preparing board samples, LC refined pulp was soaked for at least 3 hours, disintegrated using Noram CA350 hot disintegrator, and left in room temperature for about 8 hours. The prepared pulp sample was then diluted to 0.22% consistency. In Phase I, LCR reject pulp was mixed in equal portions with three commercial BCTMP pulps and was used to make the middle ply of FBB with a basis weight of 220 g/m$^2$ (see Table 3-5). While in Phase II, three-ply board samples were prepared with basis weights of 45, 220, and 45g/m$^2$ for top, middle and bottom ply, respectively. For this step of the experiments, top and bottom layer were made from a mixture of 50% northern bleached hardwood and 50% northern bleached soft wood chemical pulps specified in Table 3-7. Furnish used in preparing the middle ply sheets for phases I and II and multiply board samples are provided in Table 3-5 and Table 3-6. In phase II, an equal blend of aspen (1/3), spruce (1/3) and radiata pine (1/3) market CTMP was mixed with various proportions with LCR reject pulp as the middle ply furnish.
Table 3-5 Furnish of middle ply samples for Phase I. Radiata pine, spruce and aspen are market BCTMP.

<table>
<thead>
<tr>
<th>Pulp</th>
<th>Freeness (mL)</th>
<th>Brightness (%)</th>
<th>Percentage in the middle ply furnish, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCR reject</td>
<td>Varies</td>
<td>80</td>
<td>25</td>
</tr>
<tr>
<td>Radiata pine</td>
<td>380</td>
<td>75</td>
<td>25</td>
</tr>
<tr>
<td>Aspen</td>
<td>400</td>
<td>80</td>
<td>25</td>
</tr>
<tr>
<td>Spruce</td>
<td>350</td>
<td>75</td>
<td>25</td>
</tr>
</tbody>
</table>

Table 3-6 Middle ply furnish for Phase II. ASR is an equal blend of radiata pine, spruce and aspen market BCTMP pulps.

<table>
<thead>
<tr>
<th>Pulp</th>
<th>LCR Reject Pulp (%)</th>
<th>ASR (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A4-T4</td>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td>(SEL: 0.45 J/m)</td>
<td>35</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>55</td>
</tr>
<tr>
<td>A5-T5</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>(SEL: 0.35 J/m)</td>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 3-7 Top and bottom ply pulp properties

<table>
<thead>
<tr>
<th></th>
<th>NBHK*</th>
<th>NBSK**</th>
<th>HW/SW Mixture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Species</td>
<td>Maple</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Freeness, mL</td>
<td>380</td>
<td>525</td>
<td>411</td>
</tr>
<tr>
<td>Consistency, %</td>
<td>4.88</td>
<td>3.6</td>
<td>4.15</td>
</tr>
</tbody>
</table>

* Supplied by Domtar Espanola.
** 12.5% supplied by Domtar Espanola and 37.5% by Resolute St-Félicien

For chemical pretreatment samples, 100% of each five different pulps mentioned in Table 3-4 was used in the middle ply furnish. Basis weight for these samples were 50, 120, 40 g/m$^2$ for top,
middle, and bottom ply, respectively. Top and bottom ply were made with the same chemical pulp shown in Table 3-7.

Prior to preparing the board samples, 2 to 4 sheets were made to identify optimum drum speed, nuzzle angle, nuzzle pressure, number of passes of nozzle, and scooping time. Drum speed affects fibre alignment and sheet formation. Decreasing the drum speed resulted in a more uniform fibre orientation (less fibre alignment) but poorer formation. Nuzzle degree and pressure were therefore adjusted to obtain the desired fibre alignment and to improve formation. Scooping time can be important in the pressing section because insufficient scooping time can result in poor sheet structure due to fibre movement during the pressing operation. The target basis weight for each ply was achieved by adjusting the number of nuzzle passes.

Once the optimum DSF operation was established, 8 sheets were made for each board sample. The formed sheets were removed from the DSF drum and were pressed twice at 40 and 100 psi, respectively. These sheets were subsequently dried twice using a hot plat drier at 105°C, initially for 8 min and subsequently for 5 min. For all samples, the side of the sheet that was in touch with the wire during the sheet forming was marked as the bottom side.

3.3 Calendaring

Prepared boards were calendared to reduce the surface roughness. Samples were calendared at the same load to the target roughness of about 5µm PPS10 using a custom-made hard nip lab calendar at FPInnovations. For calendaring, the temperature, pressure and the speed were kept constant at 55 °C, 32 psi, and 410 m/min, respectively.

3.4 Board testing

All the tests were conducted at the standard conditions of 23°C and 50% humidity.

3.4.1 Fibre orientation

Fibre orientation was measured using L&W tester. This equipment measures the ultrasound velocity in every 30 degrees in the plane of the sample. The ratio of highest to lowest measured velocity is known as orientation intensity [66]. The desire value for the orientation intensity for
this study was 1 (i.e. fibres aligned randomly every direction), however, due to operational limitations, the actual values for fibre orientation intensity were between 0.9 to 1.9.

3.4.2 Tensile index

To measure the tensile strength a TAPPI standard test method T494 using Instron universal testing machine. For each board sample, 7 tensile specimens 15 mm in width and about 160 mm in length were prepared in both MD and CD directions. Tensile index, strain at break, tensile energy absorption (TEA), and modulus of elasticity were measured for these specimen.

3.4.3 Tear index

Tear strength is a measure of the required work to tear a specimen using a force perpendicular to the plane of the sample. In this study, Elmendorf method was used to measure the tear strength of board samples according to Tappi standard T414. The instrument consists of two holding clamps and a pendulum (sector arc). One of the clamps is movable and attached to the pendulum however the other one is stationary. A 76 mm-width sample is placed to the clamps in a way that the unclamped length is 63 mm. Then a 20 mm slit is made as it is shown in (Figure 3.4). When the pendulum, to which the movable clamp is attached, is moved from its fixed position, a perpendicular load will be applied to the surface of the paper. This force causes a shearing stress in the paper plane and leads to tearing the 20 mm-slit through the 43-mm length of the paper. When there is no specimen in the clamps the required energy for the specific pendulum rotation is known. However with the specimen in clamps the amount of rotation with this energy will be decreased and this decrease represents the amount of energy that is required for tearing the specimen.

For each board sample, three specimens were tested on both MD and CD direction. However, for some samples the results were not reliable due to the high tear strength values specially in the CD direction.
3.4.4 Burst index

Burst strength was measured according to Tappi standard T403 using REGMED Burst Tester. The specimen was tightened between the annular clamps on top a rubber diaphragm. The rubber diaphragm was subsequently expanded by applying hydraulic pressure until the specimen ruptured (Figure 2.3). The applied at the point of rupture in terms of psi was reported as the burst strength. For each specimen three tests were performed on the both wire side and top side. However, the final burst index that is reported in this study is the mean value of the both side measurement with three replicates divided by the basis weight of the sample.

3.4.5 Bending stiffness

L&W bending stiffness tester was used to measure the bending stiffness based on the two points load according to Tappi standard method T556. Ten specimens for both MD and CD were prepared with 8.0 cm × 3.8 cm dimension for each board sample. Then, one side of the sample was clamped and the other side was applied by an automatic force from the 50 mm distance and made the specimen to bend (Figure 3.5). The machine calculated the work that was done to bend the specimen at 5, 7.5, 15, and 30° in terms of mN.m. In the L&W Bending Tester, Taber stiffness can be calculated as follows:

Taber Stiffness (g.cm) = Taber 15°(mN.m) / 0.098066
3.4.6 Scott bond

Scott bond is a common method to quantify the delamination and ply bond strength of board. Scott bond is defined as the required energy to delaminate the specimen by using a pendulum with a control mass and velocity which causes a rotational tensile stress with a negligible sheer stress on the specimen.

Here, Scott bond was measured Hugen Internal Bond Tester was used according to the Tappi standard T569 (Figure 3.6). 25.4 mm wide and 140 mm long strips were cut in both MD and CD directions of the board samples. These strips were placed on the stationery base of a specimen preparation device that was covered with a double-sided tape. The strip was then cut into five specimens (25.4mm x 25.4 mm each) for testing (Figure 3.7) to have five replications for MD and CD.
3.4.7 Short span compression test

Short span compression test (SCT) was measured using L&W Compression tester (Figure 3.8) based on Tappi standard T826. For each DSF board sample, seven 15mm wide specimens were cut in both MD and CD directions. The lengths of the specimens were 100mm, approximately to be able to be hold between the two clamps of the STFI tester. By imputing the basis weight of the sample the machine start to apply a pressure force to the specimen until the failure occurred. The amount of force as well as the work required for the failure were recorded.
3.4.8 Roughness

Roughness is an important factor in printing since it is a determination of ink transformation on the paper surface. To measure the roughness of a paperboard sample, the airflow rate between the sample surface and a standard ring is determined. A larger airflow rate corresponds to a higher roughness value. In this dissertation roughness was measured for both sides of each sample using Parker Print Surf (PPS) roughness tester according to the Tappi standard method T575.

3.4.9 Brightness

Brightness is the amount of light reflected from the surface of the sample. The light that is using in brightness test is blue light with wavelength of 457 nm and 44 nm wide. Tappi standard method 451 was used for this test. For each board sample, brightness of three points were tested. The L&W instrument that was used to measure the brightness also provided the data of the Lab color space which indicates the real color of the sample based on the Commission Internationale de l'Eclairage (CIE) color coordinates, L*, a*, and b*. The theory behind this coordination is that two colors cannot be green and red at the same time or blue and yellow at the same time. Hence, L* indicates lightness, a* is the color variation between red and green coordinate, and b* is the on the yellow and blue coordinate [69].

3.4.10 Caliper

Caliper was measured according to Tappi Standard method T411 using Technidyne- profile plus machine, an automated caliper machine. For each FBB sample, five to six specimens were tested and the mean value used for caliper and bulk data.

3.4.11 Microscopic tests

**SEM:** SEM tests were done by the Hitachi SU 3500 located at the Ontario Centre for the Characterization of Advanced Materials (OCCAM) in the Department of Chemical Engineering and Applied Chemistry at the University of Toronto.

Small specimens were cut from each board sample, and then they were mounted by double sided tape followed by coating with Bio-RAD PS3 gold sputter coater.
XPS: XPS spectra of middle ply samples were obtained to develop a base line for the chemical composition of FBB samples. XPS spectra were obtained using a Thermofisher Scientific ThetaProbe (Thermofisher Scientific, E. Grinstead UK) housed at the Ontario Centre for the Characterization of Advanced Materials (OCCAM) located in the Department of Chemical Engineering and Applied Chemistry at the University of Toronto.

Small rectangular specimens were cut out from the centre of each sample and were attached to the specimen platen using a double-sided adhesive tape. A monochromatic Al Kα X-ray excitation source (1486.6 eV) was used with a spot size of 400 μm. Survey spectra were obtained (pass energy – 200 eV, 10 scans, step size 1 eV, 50 ms dwell time), followed by the C1s and O1s peaks at higher energy resolution (pass energy – 50 eV, 50 scans, step size 0.1 eV, 50 ms dwell time). Charge compensation was applied using the combined e⁻/Ar⁺ flood gun. All data acquisition and work-up was performed using the software that came with the instrumentation (Avantage 5.926, Thermofisher Scientific, E. Grinstead UK). Spectra were charge shifted to place the C2 (C-O) peak at 286.7 eV. Sensitivity factors supplied with the instrument for quantification.

3.5 Statistical analysis

Two statistical analysis methods, namely analysis of variance (ANOVA) and Tukey analysis, were used in this study to examine accuracy and meaningfulness of the data and to analyze results.

Analysis of variance, ANOVA, is the quantitative investigation to specify the differences among group means. In other word, analysis of variance shows whether the means of all groups are the same or different. In this analysis, the meaningful difference of the means at a given significant level, α, is specified with P-value in a way that P-value > α shows the group means difference is not significant and vice versa. In this study, the significant level of 0.05 was selected for ANOVA analysis.

In addition to ANOVA, Tukey analysis was performed to investigate the difference between 2 groups’ mean.

The above statistical analyses were performed using R-studio Version 0.99.484 software provided by RStudio, Inc.
4 Results and Discussion

This chapter is divided into two main parts according to the objectives of the project: first, the use of LCR reject pulp in the middle ply of FBB is discussed and then the effect of sulphite pretreatment of LCR pulp on the FBB properties is examined. For both topics, other than the pulp characteristics and physical properties of the boards, SEM and XPS results will be presented and at the end a preliminary economic analysis for using LCR pulp in the middle ply of FBB will be discussed.

4.1 LC refining of reject pulp

Here, first the effect of refining conditions on the LCR reject pulp was examined and then the influence of using different LCR reject percentage on properties of FBB was studied.

4.1.1 Effect of refining conditions on middle-ply properties

For a given refiner, pulp quality (as characterized by freeness) is typically controlled by adjusting the specific energy. Figure 4.1 illustrates the relationship between the specific energy and Canadian standard freeness for pulp samples used in this study.

![Figure 4.1 Canadian standard freeness against specific energy for LCR reject pulp samples](image-url)
According to Figure 4.1, at a given specific energy, freeness reduction was faster at higher SEL (i.e. higher refining intensity). At higher refining intensities, higher load is applied to the refiner bars resulting in the formation of more fines and short fibres that lower freeness.

### 4.1.1.1 Fibre orientation

In machine-made paperboards, fibre orientation depends on the draws (i.e. speed differences between adjacent sections of the board machine) and jet to wire speed ratio. However, for the laboratory board samples made using the dynamic sheet former, fibre orientation is determined by the velocity difference between the rotating drum and the jet leaving the nozzle. The larger the velocity difference the larger the fibre orientation. Fibre orientation of the board samples, as measured by the ultrasonic fibre orientation tester, are presented in Table 4-1.

**Table 4-1 Fibre orientation of middle ply samples**

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SEL: 0.32 J/m</td>
<td>SEL: 0.64 J/m</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample</td>
<td>Freeness (mL)</td>
<td>MD/CD ratio</td>
<td>Sample</td>
<td>Freeness (mL)</td>
<td>MD/CD ratio</td>
</tr>
<tr>
<td>SB0</td>
<td>715</td>
<td>1.93</td>
<td>SA0</td>
<td>680</td>
<td>1.56</td>
</tr>
<tr>
<td>(No load)</td>
<td></td>
<td></td>
<td>(No load)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SB1</td>
<td>624</td>
<td>0.99</td>
<td>SA1</td>
<td>545</td>
<td>1.39</td>
</tr>
<tr>
<td>SB2</td>
<td>533</td>
<td>1.12</td>
<td>SA2</td>
<td>416</td>
<td>1.94</td>
</tr>
<tr>
<td>SB3</td>
<td>375</td>
<td>1.57</td>
<td>SA3</td>
<td>293</td>
<td>1.85</td>
</tr>
<tr>
<td>SB4</td>
<td>245</td>
<td>1.23</td>
<td>SA4</td>
<td>176</td>
<td>1.69</td>
</tr>
<tr>
<td>SB5</td>
<td>155</td>
<td>1.68</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 4.1.1.2 Fibre characteristics

Fibre characteristics results from FQA showed that fines percentage increased and fibre length shortened upon the rise in the refining energy. Higher intensity refining also led to more fines production in the furnish. According to Table 4-2, at the almost same freeness values fines production was higher with applying more refining intensity due to the more energy expanded to the fibres.
Fibre kink and fibre curl illustrate the bending of the fibres. Curl index provides the information regarding the degree of non-straightness of the fibre, while kink index indicates the magnitude of sharpness and abruptness of fibres bending.

Table 4-2 FQA results of the middle ply samples (LW: Length weighted)

<table>
<thead>
<tr>
<th>Fibre property</th>
<th>SB0</th>
<th>SB1</th>
<th>SB2</th>
<th>SB3</th>
<th>SB4</th>
<th>SB5</th>
<th>SA0</th>
<th>SA1</th>
<th>SA2</th>
<th>SA3</th>
<th>SA4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freeness (mL)</td>
<td>715</td>
<td>624</td>
<td>533</td>
<td>375</td>
<td>245</td>
<td>155</td>
<td>680</td>
<td>545</td>
<td>416</td>
<td>293</td>
<td>176</td>
</tr>
<tr>
<td>Length LW (mm)</td>
<td>1.644</td>
<td>1.599</td>
<td>1.476</td>
<td>1.240</td>
<td>1.228</td>
<td>1.103</td>
<td>1.653</td>
<td>1.516</td>
<td>1.386</td>
<td>1.24</td>
<td>1.034</td>
</tr>
<tr>
<td>Curl Index LW(mm)</td>
<td>0.05</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>Kink index (1/mm)</td>
<td>0.47</td>
<td>0.41</td>
<td>0.37</td>
<td>0.4</td>
<td>0.41</td>
<td>0.44</td>
<td>0.44</td>
<td>0.36</td>
<td>0.4</td>
<td>0.4</td>
<td>0.43</td>
</tr>
<tr>
<td>Mean width (µm)</td>
<td>37.1</td>
<td>37.2</td>
<td>37.2</td>
<td>35.9</td>
<td>34.8</td>
<td>33.7</td>
<td>37.6</td>
<td>37.2</td>
<td>36.6</td>
<td>35.9</td>
<td>34.6</td>
</tr>
<tr>
<td>Coarseness (mg/m)</td>
<td>0.319</td>
<td>0.312</td>
<td>0.299</td>
<td>0.266</td>
<td>0.239</td>
<td>0.219</td>
<td>0.330</td>
<td>0.320</td>
<td>0.297</td>
<td>0.266</td>
<td>0.254</td>
</tr>
</tbody>
</table>

4.1.1.3 Tensile properties

Tensile strength is one of the most significant properties for paper and board products. Tensile strength depends on the individual fibres strength as well as the fibre-fibre bonding strength. Refining action improves the fibre-fibre bonding by increasing fibre collapsibility as well as fines content, and consequently increases the tensile strength. Here, to account for variations in the basis weight of lab-made samples, tensile index (Nm/g) which is the tensile strength (N/m) divided by the basis weight of the sample (g/m²) is reported.
According to Figure 4.2, tensile index improved in MD with decreasing the freeness values, i.e. increasing the refining energy. This is expected due to the larger fines production by applying more refining energy (i.e. lower LCR reject pulp freeness) and consequently higher fibre-fibre bonding strength. It should be noted that the freeness values reported in Figure 4.2 are those of LC refined reject pulp, however, as discussed before, furnish for the middle ply samples was a mixture of LCR reject pulp with three commercial BCTMPs (see Table 3-5).

For CD direction, however, there appeared to be an optimum freeness value at around 400 mL for both SEL levels. As pointed out earlier, increasing the LC refining energy of reject pulp increases fines content (and hence fibre-fibre bond strength) that is expected to have a positive effect on the tensile index. However, continuous increase in the bond strength results in more fibre breakage and less fibre pull-out during the tensile fracture process and could ultimately cause a reduction in the tensile index. This effect is more pronounced in the CD direction due to the fibre orientation effects, hence leading to the decreasing trend in the CD tensile index in Figure 4.2 as freeness falls below about 400 mL. Accordingly, the optimum LC refining energy in terms of tensile index of the middle ply samples was about 400 mL and applying more refining
energy to the reject pulp had no significant positive effect on the tensile properties of the samples.

Stretch, TEA, elastic modulus, and breaking length of the middle ply samples are provided in Figure 4.3. Breaking length is an alternative way to describe the tensile strength of paper and hence followed the same trend as in Figure 4.2. Stretch at break generally followed a similar trend to that of tensile index as well, except CD stretch values were higher than those of MD, due to lower elastic modulus in CD direction. TEA, a measure of energy used for the tensile fracture of the sample, generally increased with the refining energy (i.e. decreasing LCR reject pulp freeness) and the refining intensity (i.e. SEL).

Elastic modulus of sheet is not only a function of fibre characteristics, namely fibre width, elastic modulus, and moment of inertia [70] [71], it is also affected by the fibre-fibre bonding and sheet structure. By increasing SEL, elastic modulus in both CD and MD directions increased sharply, likely due to more fines generation at higher SEL and hence better fibre-fibre bonding. For SEL of 0.64 J/m, elastic modulus in the MD direction had an increasing trend with increasing the LC refining energies (decreasing freeness of LCR reject pulp), once again likely due to improved fibre-fibre bond strength. However, for SEL of 0.32 J/m, elastic modulus first decreased by increasing the refining energy and then increased. The initial decrease was likely due to the decrease in the fibre orientation ratio. In the CD direction, higher refining energies of LCR reject pulp initially improved the modulus of elasticity presumably due to increased fibre-fibre bond strength. However, at higher refining energies, the positive effect of fines content was likely masked by reduction in the fibre coarseness (that could lower fibre modulus of elasticity) and increase in fibre kink.
(a) Breaking Length, km vs. LCR reject pulp freeness, mL

(b) Stretch, % vs. LCR reject pulp freeness, mL

- CD, SEL: 0.32 J/m
- CD, SEL: 0.64 J/m
- MD, SEL: 0.32 J/m
- MD, SEL: 0.64 J/m
Figure 4.3 Effect of LCR reject pulp freeness on the other tensile properties middle ply board: a) breaking length, b) stretch, c) TEA, and d) elastic modulus
4.1.1.4 Tear index

Tear strength depends on the fibre strength and the internal bonding strength of the sheet. During the tear test, fibres are either pulled out or break at the crack line. Fibre breakage generally requires less energy than fibre pull out. If fibre-fibre bonding is not strong enough, fibres will be readily pulled out and the tear strength would be low. On the other hand, if the fibre-fibre bond strength is too high, the rupture will occur by fibre breakage that will also result in a low tear strength value.

Figure 4.4 shows the MD tear index of middle ply samples in this study. MD tear index appeared to reach an optimum at LCR reject pulp freeness of about 400 mL. Moreover, refining intensity had no significant effect on the MD tear index of samples.

![Figure 4.4 Tear index of two different SELs plotted against the LCR pulp freeness](image)

The results for the CD were generally higher than those for MD direction, however, these data are not included here. As seen in Figure 4.5, in CD tear tests, fracture lines were very close to the edges of the sample or propagated in the MD direction. Therefore, CD tear data were considered to be unreliable.
Burst index

Burst strength indicates the rupture resistance of packaging material. As seen from Figure 4.6, burst index at both refining intensities exhibited a similar trend with changing the freeness values. According to Figure 4.6, burst index for the middle ply samples increased by increasing the refining energy of LCR reject pulp and reached a plateau at freeness values below 400 mL. Although this effect is small, according to statistical analysis, it is statistically significant. While, refining intensity did not have a significant effect on burst index.

As discussed in Chapter 3, burst strength can be estimated from tensile strength, board properties including the basis weight and thickness, and the burst test geometry (see Equation 7). Figure 4.6 shows that the predicted burst index closely followed the experimental data for both refining intensities.
Bending stiffness

The bending stiffness of paperboard characterizes its resistance to bending deformation. In the case of FBB, bending stiffness is mainly determined by the middle-ply properties and affects the end use of FBBs as a packaging material. Bending stiffness is affected by thickness and density among other factors. At same thickness, denser sample will have a higher bending stiffness due to higher modulus of elasticity.

As expected, increasing the refining energy (i.e. reducing freeness) of LCR reject pulp resulted in a sharp reduction in the caliper of middle-ply samples (Figure 4.7). However, caliper appeared to be unaffected by the changes in the SEL as it was governed by the final freeness of LCR reject pulp. The decrease in the board caliper would be expected to cause decrease in its bending stiffness, as normally bending stiffness is related to the board thickness to the third power. However, according to Figure 4.8, Taber stiffness in the MD direction showed a continuous increase, likely due to better fibre-fibre bonding at larger fines content. This was not the case for CD bending stiffness where showed a maximum at freeness values between 300-400 mL before decreasing at higher refining energy levels (i.e. smaller freeness) of LCR reject pulp. As expected, the observed trend for bending stiffness is similar to that of elastic modulus (Figure 4.3)
d), as according to equation 10, bending stiffness is expected to be proportional to the elastic modulus.

Figure 4.7 Caliper of middle-ply samples for two different SELs plotted against the LCR reject pulp freeness

Figure 4.8 Bending stiffness of middle-ply samples at two different SELs at both MD and CD direction plotted against the LCR reject pulp freeness
4.1.1.7 Scott bond

Scott bond test mostly depends on the inter-fibre bond strength, which is affected by the fibre collapse, fines content in the furnish, fibre orientation, fibre surface chemistry and the presence of strength additives. LCR refining of pulp had a positive effect on Scott bond strength, as shown in Figure 4.9. CD Scott bond values were generally higher than those for MD. This is expected since more fibres are aligned perpendicular to the delamination direction in CD Scott bond test compared to that of MD. Fibres aligned perpendicular to the delamination direction require a greater energy for debonding compared to those aligned in the delamination direction.

Figure 4.9 Scott bond of middle-ply samples at two different SELs for both MD and CD direction plotted against the LCR reject pulp freeness

4.1.1.8 Short span compression

Compression tests represent the ability of the packaging material to crushing due to stacking, transportation, and converting operations. Figure 4.10, shows the short span compression (SCT) results for middle-ply samples. SCT represents the ability of the board to transfer the load in the planar direction under compression. Hence, it is a function of sheet thickness, elastic modulus, fibre-fibre bonding, and fibre orientation. Thicker sheet, higher elastic modulus, and stronger fibre-fibre bond increase the SCT.
According to the results, for SEL of 0.64 J/m, MD-SCT decreased sharply after the first LC refining step (i.e. at freeness of 624 mL) and subsequently increased by further reduction in the freeness of LC reject pulp. The initial decrease in SCT was likely due to the decrease in the sheet caliper (Figure 4.7) while the subsequent increasing trend in SCT by increasing the refining energy (i.e. lowering the LCR reject pulp freeness) could have been caused by the increase in the board elastic modulus (Figure 4.3 d) and fibre-fibre bond strength (Figure 4.9). For comparison, there was a smaller decrease in SCT at SEL of 0.32 J/m after the first step of refining. In that case, however, decrease in sheet elastic modulus (see Figure 4.3 d) was likely responsible for the observed trend since sheet thickness remained nearly constant after the initial refining step. It should be noted that the CD-SCT results for the no-load conditions (samples containing SA0 and SB0 reject pulp samples) were not reliable due to their low SCT values and hence are not reported here.

Figure 4.10 Short span compression of middle-ply samples for two different SELs at both MD and CD direction plotted against the LCR reject pulp freeness

4.1.1.9 Roughness

The production of FBB usually involves the application of a top ply (and often a bottom ply) on the middle ply sheet. Hence, the roughness of middle ply could affect the final roughness of
multi-ply FBB product. According to Figure 4.11, as expected, surface roughness of middle ply samples decreased by increasing the refining energy of reject pulp likely due to increased fines generation and fibre cutting as well as decrease in fibre stiffness (increase in fibre collapsibility). This decrease in roughness was more pronounced for SEL of 0.64 J/m where more fibre cutting and more fines are expected.

![Figure 4.11](image-url)

Figure 4.11 Roughness of two different SELs at both top and bottom side of the board plotted against the LCR pulp freeness.

Figure 4.11 also shows that the bottom side of the boards, that is the side which was in contact with the wire, had higher roughness due to the wire marks on the sheet.

**4.1.1.10 Brightness**

Although in a multi ply FBB, the middle ply is sandwich between at least two other plies (i.e. top and bottom), the optical properties of middle ply, i.e. whiteness, brightness and color, could affect the final quality of FBB.
From Figure 4.12, in general, there appeared to be an increase in brightness at higher refining energies. This could be attributed to the increase in fines content at higher refining energy. Fines are known to scatter light effectively due to their large specific surface area and hence improve the optical properties of paper and board. However, no clear trend was observed for increasing the refining energy at the same SEL. Statistical analysis showed that there was no significant difference between the samples with different refining intensity.

### 4.1.1.11 Bulk

In a multi ply FBB, the middle ply is composed of high bulk furnish to impart the desired stiffness to the final product. In the present study, the key motivation for introducing coarse LCR reject pulp to the middle ply of FBB was to further enhance bulk and hence the bending stiffness of the board product. According to Figure 4.13, bulk of middle ply samples decreased steadily with increased refining energy of LCR reject pulp. It should be noted that increasing refining energy (i.e. lower freeness of LCR reject pulp) reduced the bulk regardless of the refining intensity. However, there was little difference in bulk between the two refining intensities at a given freeness.
4.1.1.12 Sheet structure

The surface morphology of the samples was investigated using scanning electron microscopy. By examining a large number of micrographs (Figure 4.14 and Figure 4.15) it became apparent that by increasing the refining energy (decreasing LCR reject pulp freeness) fines progressively bridge between fibres and fill the surface pores. This improves fibre-fibre bonding and reduces surface roughness. Comparing Figure 4.14b and Figure 4.15c (with freeness values of 544 mL and 533 mL, respectively) it is evident that at the same LCR reject pulp freeness, higher refining intensity (i.e. higher SEL) resulted in more fibre-fibre bridging, likely due to the generation of more fines.
Figure 4.14 SEM micrographs of middle ply samples containing LCR reject pulp with various refining energy at SEL of 0.64 J/m: a) SA0, b) SA1, c) SA2, d) SA3, and e) SA4. For LCR reject pulp codes see Table 3-1.
Figure 4.15 SEM micrographs of middle ply samples containing LCR reject pulp with various refining energies at SEL of 0.32 J/m: a) SB0, b) SB1, c) SB2, d) SB3, e) SB4, and f) SB5. For LCR reject pulp codes see Table 3-1.
4.1.1.1 X-ray photoelectron spectrometry (XPS)

XPS spectra of middle ply samples were obtained to develop a base line for the chemical composition of FBB samples.

Figure 4.16 shows an example of XPS results for sample SB4 (see Table 3-1 for sample specifications). As expected, the XPS results showed that the middle ply samples were mainly composed of carbon and oxygen. Elemental composition data (Table 4-3) revealed that higher intensity refining (samples SB0 to SB4) on average had a higher oxygen content. This suggests that high intensity refining likely removed more lignin from the outer layer of fibre wall, resulting in an increase in O/C ratio [72]. The high resolution XPS (pass energy: 48eV) spectrum for middle ply sample containing SB4 reject pulp provided in Table 4-3 shows that the dominant carbon bonding was C1sA. Moreover, by increasing the refining energy, the O/C ratio generally decreased. As seen in Figure 4.14 and Figure 4.15, the surface of middle ply samples containing LCR reject pulp at higher specific energies (lower freeness values) is increasingly covered by fines. Fines are known to contain a higher percentage of lignin and extractives that have a lower O/C ratio [72].

![Figure 4.16 XPS spectra for sample SB4](image-url)
According to the literature the carbon bond distribution of cellulose is 18, 56, and 26 % for C1s, C1sA, and C1sB, respectively, and the O/C ratio is about 0.83 [73]. Lower O/C ratios of the board samples in this study is an indication of lignin removal during the refining. In addition, according to Leduc et al. [74], bleaching of CTMP increases the O/C ratio to about 0.5.
### Table 4-3 Carbon bonding distribution

<table>
<thead>
<tr>
<th></th>
<th>C1s</th>
<th>C1s A</th>
<th>C1s B</th>
<th>C1s C</th>
<th>O/C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[C-C / C-H]</td>
<td>[C-O]</td>
<td>[C=O / O-C-O]</td>
<td>[O-C=O]</td>
<td></td>
</tr>
<tr>
<td>CTMP+8% Na₂SO₄ +1% NaOH  [31]</td>
<td>34.2</td>
<td>59.4</td>
<td>6.4</td>
<td>NA</td>
<td>0.47</td>
</tr>
<tr>
<td>CTMP+4% Na₂SO₄+1% NaOH [31]</td>
<td>39.8</td>
<td>54.2</td>
<td>6.0</td>
<td>NA</td>
<td>0.46</td>
</tr>
<tr>
<td>Unbleached CTMP [74]</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>0.43</td>
</tr>
<tr>
<td>Bleached CTMP with NaOH [74]</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>0.51</td>
</tr>
<tr>
<td>Bleached CTMP with Mg(OH)₂ [74]</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>0.49</td>
</tr>
<tr>
<td>This study</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SB0</td>
<td>26.64</td>
<td>55.43</td>
<td>14.99</td>
<td>2.94</td>
<td>0.52</td>
</tr>
<tr>
<td>SB1</td>
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<td>52.14</td>
<td>16.12</td>
<td>3.75</td>
<td>0.51</td>
</tr>
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<td>55.50</td>
<td>12.14</td>
<td>3.72</td>
<td>0.47</td>
</tr>
<tr>
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<td>12.14</td>
<td>3.72</td>
<td>0.48</td>
</tr>
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<td>51.46</td>
<td>14.17</td>
<td>5.28</td>
<td>0.47</td>
</tr>
<tr>
<td>SB5</td>
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<td>54.76</td>
<td>15.33</td>
<td>7.47</td>
<td>0.47</td>
</tr>
<tr>
<td>SA0</td>
<td>27.84</td>
<td>53.28</td>
<td>13.40</td>
<td>5.49</td>
<td>0.55</td>
</tr>
<tr>
<td>SA1</td>
<td>25.15</td>
<td>50.86</td>
<td>16.45</td>
<td>7.54</td>
<td>0.54</td>
</tr>
<tr>
<td>SA2</td>
<td>25.93</td>
<td>50.01</td>
<td>14.52</td>
<td>9.55</td>
<td>0.50</td>
</tr>
<tr>
<td>SA3</td>
<td>24.69</td>
<td>53.83</td>
<td>14.80</td>
<td>6.69</td>
<td>0.49</td>
</tr>
<tr>
<td>SA4</td>
<td>22.69</td>
<td>54.13</td>
<td>19.09</td>
<td>4.10</td>
<td>0.49</td>
</tr>
</tbody>
</table>

4.1.2 Multi-ply FBB containing LCR reject pulp

As discussed in Chapter 3, three-ply lab-made FBB samples were prepared with the middle ply containing a mixture of LCR reject pulp and three commercial BCTMPs, namely radiata pine, spruce and aspen (Table 3-6). Two LCR reject pulps prepared with a target freeness of 300mL at
refining intensities of 0.45 J/m and 0.35 J/m were used in this study. The middle ply furnish composition is provided in Table 3-6. The top and bottom plies composed of a mixture of hardwood and softwood Kraft pulps provided by FPInnovations, Table 3-7.

4.1.2.1 Fibre Characteristics

Fibre characteristics for the middle ply furnish containing various percentages of LCR reject pulp are provided in Table 4-4. By increasing the percentage of LCR reject pulp, as expected the mean fibre length decreased and the amount of fines increased at both refining intensities.

Table 4-4 FQA results for the middle ply furnish. SEL is the refining intensity of the reject pulp.

*ASR is an equal blend of aspen, spruce and radiata BCTMP.

<table>
<thead>
<tr>
<th>LCR Reject + ASR*</th>
<th>% LCR Reject (SEL: 0.45 J/m)</th>
<th>% LCR Reject (SEL: 0.35 J/m)</th>
<th>Aspen</th>
<th>Spruce</th>
<th>Radiata</th>
<th>ASR**</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25               35             45</td>
<td>25             35             45</td>
<td>100</td>
<td>NA</td>
<td>NA</td>
<td>11.2</td>
</tr>
<tr>
<td>Fines (%)</td>
<td>10.76            11.64          12.05</td>
<td>10.71          11.78          11.95</td>
<td>12.14</td>
<td>NA</td>
<td>NA</td>
<td>11.2</td>
</tr>
<tr>
<td>Mean fibre length (mm)</td>
<td>1.262          1.240           1.228</td>
<td>1.275          1.251          1.219</td>
<td>1.195</td>
<td>0.7</td>
<td>1.56</td>
<td>1.40</td>
</tr>
<tr>
<td>Curl index (mm)</td>
<td>0.07            0.06           0.05</td>
<td>0.07           0.05           0.05</td>
<td>0.04</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Kink index (1/mm)</td>
<td>0.67            0.52           0.53</td>
<td>0.64           0.58           0.49</td>
<td>0.42</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Mean width (µm)</td>
<td>28.2            29.9           29.5</td>
<td>28.6           29.45          29.9</td>
<td>30.2</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Coarseness (mg/m)</td>
<td>0.233           0.246           0.239</td>
<td>0.247          0.242           0.234</td>
<td>0.225</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

** Fibre characteristics for ASR are estimated based on rule of mixtures.
4.1.2.2 Fibre orientation

The fibre orientation results obtained by L&W tester shows that although fibres were mostly aligned in MD, all the MD/CD ratios between 1.1 and 1.66. Changes in the fibre orientation was a result of variations in the speed difference between the jet (leaving the nozzle) and the rotating drum in the Dynamic Sheet Former.

Table 4-5 Fibre orientation of the three ply FBB samples containing LCR reject pulp. Top and bottom ply furnish as in Section 3.2.

<table>
<thead>
<tr>
<th>Pulp</th>
<th>Content (%)</th>
<th>MD/CD ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCR reject*</td>
<td>25</td>
<td>1.23</td>
</tr>
<tr>
<td>A4-T4</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>1.35</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1.42</td>
</tr>
<tr>
<td>LCR reject*</td>
<td>25</td>
<td>1.66</td>
</tr>
<tr>
<td>A5-T5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>1.48</td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>1.10</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1.50</td>
</tr>
<tr>
<td>Aspen BCTMP</td>
<td>100</td>
<td>1.37</td>
</tr>
<tr>
<td>Spruce BCTMP</td>
<td>100</td>
<td>1.29</td>
</tr>
</tbody>
</table>

*Balance of the furnish is ASR, i.e. an equal mixture of aspen, spruce and radiata pine market BCTMP

4.1.2.3 Physical properties of the FBBs vs the LCR reject pulp percentage

By increasing the LCR content of the middle ply caliper and bulk increased (Figure 4.19a and b). Lower coarseness of LCR reject fibres increases the average fibre coverage (i.e. number of layers) of the middle ply. A larger number of layers combined with the larger fibre diameter resulted in an overall increase in the caliper and hence bulk. Increasing LCR reject fibre content
also increased the sheet roughness (Figure 4.19 c) up to 45% LCR reject pulp content. Surface roughness generally is affected by the fibre diameter, fibre collapse, fines content, as well as forming conditions. Reject fibres have larger diameters than ASR fibres and hence their addition to the middle ply initially resulted in a higher roughness values. However, at high LCR reject content, fines increasingly filled the surface pores and hence reduced the roughness.

Figure 4.19d shows that optical properties of middle ply affected the final brightness of the FBB samples. LCR reject pulp had a lower brightness than the ASR pulp, sheet brightness decreased steadily with LCR reject content in the middle ply.
Figure 4.19 Effect of LCR reject pulp percentage on (a) Caliper, (b) bulk, (c) roughness, and (d) brightness of FBB samples

Tensile properties of FBB samples are provided in Figure 4.20. Since top and bottom plies had the same furnish and basis weight in all samples, the observed trends in the physical properties of FBB samples were due to changes in the middle ply furnish. In general, there was a larger variability (standard deviation) in the measured physical properties that reflects the inherent variability in the preparation of three-ply FBB samples in the lab. Despite this fact, results showed that with increasing LCR reject pulp percentage from 0% to 45% the tensile index exhibited an increasing trend at both refining intensities in MD and CD directions, indicating a positive deviation from the rule of mixtures. This is likely because of the higher fines content in the middle ply and hence better fibre-fibre bonding as LCR reject pulp content increased (Table 4-4). For board samples containing low intensity LCR reject pulp (SEL=0.35 J/m), further increase of the reject pulp content in the middle ply to 100% had no statistically significant effect on the MD and CD tensile index values. This may be due to the competing effects of increased fines amount and decreased coarseness on one side versus shorter and wider fibres on the other side (see equation 3). Short wide LCR fibres have are expected to be less deformable and hence do not bond well to adjacent fibres and decrease the RBA. Meanwhile, LCR reject fibres are
short and are more likely to be pulled out than break hence decreasing the tensile strength. On the other hand, the decreased in the mean coarseness and increase in the fines content with higher percentages of LCR reject pulp were expected to have positive effect on the tensile index of the sheet. Statistical analysis showed that there was no significant difference between the samples with two different LCR intensities.

Earlier studies suggest that elastic modulus is proportional to the tensile strength of samples made of the same furnish with varying degrees of fibre orientation and wet pressing [75]. However, according to Figure 4.20b, by increasing LCR reject pulp percentage from 0% to 45%, elastic modulus in both CD and MD directions showed a slight decreasing trends. A closer look at equations 3 and 5 shows that both tensile strength and elastic modulus depend on the product of fibre length and relative bonded area, i.e. L×RBA. By increasing the LCR reject pulp content in the middle ply, average fibre length decrease while RBA increases (due to higher fines contents). Based on equation 5, the decreasing trend of elastic modulus suggested a net decrease in the L×RBA. In the case of tensile index, decrease in L×RBA at higher LCR reject pulp contents appears to have been more than compensated by the decrease in fibre coarseness (see equation 3), therefore the increasing trend in the tensile strength as LCR reject increased from 0% to 45%. Figure 4.20b also shows that at lower refining intensity (SEL of 0.35), elastic modulus appeared to follow the rule of mixtures reasonably well.

Sheet stretch depends on two factors: 1) the stretch potential of the fibres, and 2) the fibre-fibre bonding [76]. Better bonding allows fibres to stretch closer to their maximum potential. At SEL of 0.35 J/m, Figure 4.20d shows that there was no significant difference between the stretch of FBB samples containing only ASR in the middle ply and those where the middle layer was 100% LCR reject pulp. In this case, despite variations in the experimental data, stretch appears to closely follow the rule of mixtures. Additionally, changing SEL from 0.35 J/m to 0.45 J/m appeared to have little impact on the stretch of FBB samples while as expected CD stretch was higher than that of MD stretch due to fibre orientation anisotropy effects. Similar trends were observed for TEA in MD and CD.
Figure 4.20 Effect of LCR reject pulp percentage on the tensile properties for three ply samples:
  a) Tensile index, b) elastic modulus, c) TEA, and d) stretch
Figure 4.21 Effect of LCR reject pulp percentage for three ply samples on (a) Burst index, and (b) Tear index MD, and (c) Scott bond

Similar to the tensile index, burst index (Figure 4.21a) initially increased by the addition of LCR reject pulp before decreasing again. This trend can be explained based on equation 7 that suggests burst index is proportional to the average MD and CD tensile stiffness values, MD stretch, and bulk. Figure 4.21a also shows that unlike tensile index, FBB samples containing LCR reject pulp with higher refining intensity had a lower burst index. This could be explained by the lower caliper of FBB samples made of high intensity refined reject pulp. According to equation 7, burst index is proportional to the sample thickness, therefore sheets with smaller caliper are expected to have proportionally lower burst index. Similarly, MD tear index appeared to improve by the addition of up to 45% LCR reject pulp likely due to the improved bond strength with higher fines content. Increasing the fines content beyond 45% resulted in a decrease in the MD tear probably due to the decrease in fibre length. As seen from equation 6, for short fibres \( L_{\text{max}} < L_c \) tear strength is more than proportional to fibre length. CD tear index was also measured, however due to the high CD tear strength of the samples these results were unreliable.

Scott bond strength (Figure 4.21 c) also showed an initial improvement by adding more LCR reject pulp in the middle ply furnish up to 45% LCR reject pulp content and subsequently. In the case of multiply FBB, Scott bond is a measure of the ply bond strength. Higher LCR reject pulp
content resulted in a higher amount of fines and that is expected to improve. In addition, larger fines content resulted in smoother middle ply surface (see Figure 4.14) that is expected to improve contact and hence inter-ply bonding. On the other hand, LCR reject fibres are short and wide and hence are expected to be less deformable. This could justify the ultimate decline of Scott bond as LCR reject in the middle ply increased to 100%. Figure 4.21c also shows that improvement in the Scott bond strength was more pronounced in the CD than MD. In MD Scott bond test, more fibres are aligned in the delamination direction and these fibres require less energy for debonding and delamination.

According to Figure 4.22a, addition of LCR reject pulp to the middle ply furnish did not change the board MD-SCT significantly. However, for both SEL values, CD-SCT increased with changing the LCR reject percentage from 0% to 35% and subsequently leveled off. In an oriented sheet where fibres are predominantly aligned in the MD than CD, elastic modulus of the sheet is larger in the MD direction (Figure 4.20b). Therefore, under compressive stress, sheet deforms more readily in the CD direction and hence could buckle at a lower load. In CD-SCT test, more fibres experience transverse loading that could result in fibre bending and fibre-fibre bond breakage. Therefore, in this case, increasing the LCR reject pulp content could have a positive effect on the SCT by reinforcing fibre-fibre bonds due to increasing the fines content.

Figure 4.22b illustrates that increasing LCR reject pulp percentage continuously improved Taber stiffness of the boards. As discussed before, bending stiffness of paper is proportional to caliper to the third power. By increasing the LCR pulp content, caliper increased that resulted in an increase in the Taber stiffness increased.

According to equation 10, most of the bending stiffness for multi-ply structures comes from the skin. Therefore, stiffness of middle ply accounted for less than 8% of the total bending stiffness. More detail calculations are provided in appendix section.

It should be noted that in this study creasing test equipment was not available, however, increasing creasing of the boards by increasing the LCR reject pulp is estimated due to stiffness improvement that is in relation to the folding resistance.
Figure 4.22 Effect of LCR reject pulp percentage on (a) SCT, and (b) Taber stiffness
4.1.2.4 Physical properties of the FBBs made with different mechanical pulp
DSF made boards containing 100% of commercial BCTMP including, aspen, and spruce were tested and compare with the FBB boards made using LCR reject pulp. Physical properties of these samples are provided in Table 4-6.

Table 4-6 Physical properties of FBB samples containing 100% LCRR, 100% aspen and 100% spruce BCTMP in the middle ply.

<table>
<thead>
<tr>
<th></th>
<th>LCRR</th>
<th>Aspen</th>
<th>Spruce</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile index-MD</td>
<td>25</td>
<td>26</td>
<td>33</td>
</tr>
<tr>
<td>Tensile index-CD</td>
<td>22.4</td>
<td>20.2</td>
<td>25.2</td>
</tr>
<tr>
<td>Stretch-MD</td>
<td>2.3</td>
<td>1.6</td>
<td>2.4</td>
</tr>
<tr>
<td>Stretch - CD</td>
<td>2.4</td>
<td>2.1</td>
<td>2.5</td>
</tr>
<tr>
<td>TEA- MD</td>
<td>411</td>
<td>294</td>
<td>538</td>
</tr>
<tr>
<td>TEA-CD</td>
<td>367</td>
<td>302</td>
<td>413</td>
</tr>
<tr>
<td>Elastic modulus- MD</td>
<td>395</td>
<td>548</td>
<td>424</td>
</tr>
<tr>
<td>Elastic modulus- CD</td>
<td>286</td>
<td>372</td>
<td>341</td>
</tr>
<tr>
<td>SCT- MD</td>
<td>11.1</td>
<td>13.4</td>
<td>12.3</td>
</tr>
<tr>
<td>SCT- CD</td>
<td>9.6</td>
<td>9.8</td>
<td>10.3</td>
</tr>
<tr>
<td>Taber stiffness- MD</td>
<td>345</td>
<td>274</td>
<td>317</td>
</tr>
<tr>
<td>Taber stiffness- CD</td>
<td>250</td>
<td>185</td>
<td>219</td>
</tr>
<tr>
<td>Scott bond- MD</td>
<td>85</td>
<td>81</td>
<td>59</td>
</tr>
<tr>
<td>Scott bond-CD</td>
<td>118</td>
<td>85</td>
<td>68</td>
</tr>
<tr>
<td>Tear index- MD</td>
<td>9.9</td>
<td>6.5</td>
<td>13.7</td>
</tr>
<tr>
<td>Burst Index</td>
<td>1.87</td>
<td>1.46</td>
<td>2.59</td>
</tr>
<tr>
<td>Bulk</td>
<td>2.52</td>
<td>1.89</td>
<td>2.19</td>
</tr>
<tr>
<td>Caliper</td>
<td>772</td>
<td>604</td>
<td>717</td>
</tr>
<tr>
<td>Roughness- top</td>
<td>4.8</td>
<td>4.8</td>
<td>5.1</td>
</tr>
<tr>
<td>Roughness- bottom</td>
<td>4.9</td>
<td>5.1</td>
<td>5.3</td>
</tr>
<tr>
<td>Brightness- top</td>
<td>72.5</td>
<td>83.7</td>
<td>79.9</td>
</tr>
<tr>
<td>Brightness- bottom</td>
<td>72.9</td>
<td>83.3</td>
<td>79.9</td>
</tr>
</tbody>
</table>
The results for tensile index and SCT shows that although increasing the LCR reject pulp can improved these properties, using 100% of LCR reject pulp somewhat reduced the strength. The DSF boards made with 100% of aspen and spruce had higher tensile and SCT strength (Table 4-6), however the statistical analysis shows there is no significant difference between the DSF board samples made with aspen, while spruce is significantly higher strength for SCT. Tensile statistical analysis demonstrates that the commercial pulps considerably have higher tensile index and confirms that some actions are required to be taken to strengthen the tensile properties of the boards using the LCR reject pulp in the future.

Taber stiffness as well as Scott bond strength was higher by using the LCR reject pulp in comparison to the commercial pulps. Although Figure 4.21c shows that increasing LCR reject pulp from 45% to 100% reduce the Scott bond, the board strength is still higher than the 25% LCR reject as well as the commercial pulps, Table 4-6.

In addition, commercial pulps acted differently for each test. For example, tear and burst index of boards made with spruce were significantly higher than the ones with aspen, whereas for some other tests they were almost similar. According to Table 4-6, aspen bulk was lower than the other samples, however comparing spruce and 100% LCR reject pulp gave almost similar bulk values. Hence, slight decreasing of bulk by increasing LCR reject pulp is basically due to increasing aspen in the furnish. Furthermore, increasing LCR reject pulp significantly reduced the brightness of the three ply FBBs. This is due to the high brightness of aspen and spruce; with increasing the LCR reject pulp and decreasing aspen and spruce percentage the brightness of boards decreased, however, this problem can be solved by coating the boards in the future. Roughness values of the boards were almost similar due to the same calendaring conditions used for all the boards and the boards made with LCR reject pulp even shows lower roughness values than the commercial pulps.

From all the data, it can be concluded that the reasonable strengths and surface properties for all tests with LCR reject pulp confirms that it is a good replacement pulp for the middle ply of FBBs than the commercial pulps due to using LC refining which leads to lower energy consumption as well as using the reject pulp which leads to save the material.
4.2 LC refining of chemical pretreated pulp

In this section, the use of chemically pretreated HC-LC refined SPF pulp as the middle ply of FBB is examined. Details of the refining conditions and the pretreatment of the wood chips are presented in Section 3.2. Board samples are labeled after the refining trial code as in Table 3-4.

Figure 4.23 provides a better idea of the effect of chemical pretreatment on the refining operation of SPF. This graph shows that for every 1 kWh increase in the specific refining energy, freeness dropped by about 2.5 mL except for the A8-T1 that showed a slower rate of decrease of freeness (namely 1.6 mL/kWh/t), which is the sample that had a lower freeness level of the others. It is worth noting that A10-T1 and A11-T1 as well as A10-T2 and A11-T2 samples had the same refining conditions but were subjected to different degrees of chemical pretreatments.

![Figure 4.23 Effect of LC refining specific energy and intensity as well as wood chip chemical pretreatment on the freeness of SPF pulp.](image)

4.2.1 Fibre characteristics of the chemically pretreated and regular pulp

FQA results indicate after second stage LC refining more fines were generated and fibre length was reduced regardless of the refining intensity and chemical pretreatment. It is worth noting that after first stage HC refining, fibre length for chemically treated and untreated pulps were quite similar (1.81 mm to 1.88 mm) while fines content was lower for chemically treated pulps (~9.5%
for untreated pulp vs. ~8.8% for chemically pretreated pulp). LC refining of chemically pretreated pulp at higher intensity resulted in less fibre cutting (higher fibre length) and less fines formation, while the opposite was true for SPF pulp without chemical pretreatment. Interestingly, LC refining had no significant effect on the fibre width but reduced fibre coarseness. This apparently contradictory result could be due to the error in fibre width measurement due to the limited optical resolution of FQA. Regardless, final coarseness of chemically pretreated pulps and untreated pulps were quite similar (within 10%). In addition, as expected, LCR straightened fibres and reduced curl and kink of samples. Comparing the primary HCR samples shows that after the HC refining, A8-T1 sample had a lower freeness, larger fines content and shorter fibre length. Chemically pretreated pulp samples, A10-T1 and A11-T1, had lower fines content after the first stage HC refining. However, second stage LC refining increased the fines content of chemically pretreated pulps more than untreated pulps. Sulphite pretreatment led to about 5% (in absolute terms) more fines generation and about 8% more reduction in fibre length. However, according to Figure 4.23, LC refining action cannot be accelerated by using sulphite pretreatment, because upon applying almost the same specific refining energy the freeness of no-chem and chemically treated samples reduced by the same amount. In addition, for higher SEL, sulphite pretreatment resulted in less fines generation.

Table 4-7 FQA results for the SPF pulp samples before (no load) and after LC refining

<table>
<thead>
<tr>
<th></th>
<th>A8-T1 No chem SEL: 0.25 J/m,</th>
<th>A10-T1 No chem SEL: 0.25 J/m,</th>
<th>A10-T2 No chem SEL: 0.6 J/m,</th>
<th>A11-T1 Sulphite SEL: 0.25 J/m,</th>
<th>A11-T2 Sulphite SEL: 0.6 J/m,</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freeness (mL)</td>
<td>No-load LCR</td>
<td>No-load LCR</td>
<td>No-load LCR</td>
<td>No-load LCR</td>
<td>No-load LCR</td>
</tr>
<tr>
<td>Fine percentage LW (%)</td>
<td>305 214</td>
<td>523 402</td>
<td>583 402</td>
<td>590 462</td>
<td>562 366</td>
</tr>
<tr>
<td>Length LW (mm)</td>
<td>1.52 1.44</td>
<td>1.83 1.68</td>
<td>1.88 1.56</td>
<td>1.84 1.53</td>
<td>1.81 1.65</td>
</tr>
<tr>
<td>Curl Index LW (mm)</td>
<td>0.06 0.04</td>
<td>0.05 0.04</td>
<td>0.05 0.04</td>
<td>0.05 0.04</td>
<td>0.05 0.04</td>
</tr>
<tr>
<td>Kink index (1/mm)</td>
<td>0.86 0.64</td>
<td>0.69 0.62</td>
<td>0.68 0.47</td>
<td>0.65 0.43</td>
<td>0.63 0.52</td>
</tr>
<tr>
<td>Mean width (µm)</td>
<td>30.3 30.7</td>
<td>30.6 30.8</td>
<td>30.6 31.0</td>
<td>31.7 32.1</td>
<td>31.8 31.8</td>
</tr>
<tr>
<td>Coarseness (mg/m)</td>
<td>0.25 0.23</td>
<td>0.28 0.26</td>
<td>0.31 0.27</td>
<td>0.28 0.24</td>
<td>0.27 0.26</td>
</tr>
</tbody>
</table>
4.2.2 Fibre orientation of FBBs making with chemical pretreated and regular pulp

Fibre orientation results (Table 4-8) show that for most FBB samples fibres were orientated nearly randomly in the sheets. In the limiting case when MD/CD fibre orientation ratio is equal to 1, physical properties in MD and CD directions on average will be equal.

Table 4-8 Fibre alignment results for FBBs made containing HC-LC refined SPF pulp

<table>
<thead>
<tr>
<th>Sample</th>
<th>MD/CD ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>A8-T1</td>
<td>1.12</td>
</tr>
<tr>
<td>A10-T1</td>
<td>1.04</td>
</tr>
<tr>
<td>A10-T2</td>
<td>1.23</td>
</tr>
<tr>
<td>A11-T1</td>
<td>1.16</td>
</tr>
<tr>
<td>A11-T1</td>
<td>1.08</td>
</tr>
</tbody>
</table>

4.2.3 Physical properties of FBBs containing chemically pretreated pulp

In this section, physical properties of FBB samples containing SPF pulp, with and without chemical pretreatment, are presented.

Bulk for FBB samples are presented in Figure 4.24. A8-T1 had the lowest bulk due to larger amount of fines that could fill the pores in the middle ply of the FBB and hence increase its density (i.e. reduce bulk). Among other samples, sulphite pretreated pulp prepared at refining intensity of 0.25 J/m (A11-T1) produced a sheet with the highest bulk. This may be attributed to the low coarseness and larger diameter of SPF fibres treated under the above conditions.
Figure 4.24 Effect of LCR chemical pretreatment and LCR refining condition on (a) bulk, and (b) Caliper.
Based on Figure 4.25, effect of chemical pretreatment on the tensile strength appeared to be dependent on the refining intensity. Comparing A10-T1 and A11-T2 had very similar fibre properties, while the latter had lower fines content. In spite of this, A11-T2 had a higher tensile strength (as well as MD modulus, TEA and stretch) than A10-T1. It is possible that removal of lignin by sulphite pretreatment resulted in more efficient bonding at the fibre-fibre joints. ANOVA and Tukey analysis of data presented in showed that pulp freeness was the main factor in determining tensile properties of FBB samples. For A8-T1 sample (with the lowest pulp freeness) tensile properties were significantly higher than other FBB samples. Although fibre length of the middle ply furnish for this sample was lower, lower coarseness and higher fines content in the furnish (see Table 4-7) likely resulted in a higher tensile strength. In contrast, the CD-tensile strength of all samples were nearly the same. A similar trend was observed for TEA, elastic modulus, and stretch. Figure 4.25 also shows that there was a slight improvement in MD tensile strength and MD modulus after sulphite pretreatment of pulp, however, these improvements were not statistically significant. The average tensile strength increased by 1.6~2.3 N.m/g, depending on the LC refining intensity. This improvement might be attributed to the fibre softening and lower coarseness values as a result of chemical pretreatment (see Table 4-7) [54].
(b)

Elastic Modulus, N.m/g

Sample

No-chem, SEL:0.25
No-chem, SEL:0.25
No-chem, SEL:0.6
Sulphite, SEL:0.25
Sulphite, SEL:0.6

MD
CD

(c)

TEA Index, mJ/g

Sample

No-chem, SEL:0.25
No-chem, SEL:0.25
No-chem, SEL:0.6
Sulphite, SEL:0.25
Sulphite, SEL:0.6

MD
CD
Figure 4.25 Effect of chemical pretreatment and LCR refining condition on the tensile properties:
   a) Tensile index, b) elastic modulus, c) TEA, and d) stretch
The results for the burst index shows a higher strength for the sample with the lowest freeness value, i.e. A8-T1. Higher burst index is a result of higher MD tensile strength and MD stretch of this sample (see equation 7). Among other samples, A11-T1 had the largest burst index, likely due to its higher bulk.

Figure 4.26 shows that sulphite pretreatment had no significant effect on the tear strength of samples. However, degree of refining was once again had the most effect on the MD tear resistance of board, i.e. sample containing A8-T1 pulp had lower tear index likely due to the shorter fibres (see equation 6)
Figure 4.27 Effect of chemical pretreatment and LCR refining condition on (a) Scott bond, (b) SCT, and (c) Taber stiffness

Chemical pretreatment had no clear effect on the Scott bond of FBB samples (Figure 4.27). A8-T1 had the highest Scott bond strength due to its higher fines content that improved contact and bonding between middle ply with the top and bottom plies. In addition, Figure 4.27 suggests that in the case of SPF pulp without chemical pretreatment, refining intensity had little effect on the Scott bond strength. This is in contrast to the LC refining of reject radiata pine (see previous section) where higher SEL resulted in a slight increase in the Scott bond strength. For chemically pretreated pulp, Scott bond strength significantly improved with increased refining intensity, likely due to longer fibres. Moreover, the relatively lower Scott bond strength of A11-T1 is likely due to its higher bulk (lower density) that is known to have adverse effect on RBA. Figure 4.27 also shows that Scott bond for MD and CD were similar that is due to the near-uniform fibre orientation distribution in these board samples.

Based on Figure 4.27b, at similar final freeness values, MD-SCT improved upon chemical pretreatment likely due to the larger fibre width (see equation 12). However, refining intensity had no notable effect on this property. This is consistent with the results obtained for LCR reject pulp where SEL had little effect on the MD and CD SCT. Once again, FBB samples containing
A8-T1 (low freeness pulp) in the middle ply, performed the better and had the largest MD-SCT value due to its higher MD modulus. In contrast, CD-SCT remained practically unaffected by either refining intensity, chemical pretreatment, or middle ply pulp freeness.

The largest value of Taber stiffness was measured for FBB sample containing the high intensity LC refined chemically pretreated pulp (A11-T2). As explained in section 2.1.2, Taber stiffness is proportional to the elastic modulus of paper and the third power of caliper. Caliper did not significantly change with varying chemical pretreatment and refining intensities. However, A11-T2 sample had a relatively large fibre length and, as inferred earlier, had likely more efficient fibre-fibre bonding due to its lower lignin content. Longer fibres and better bonding both could improve the bending stiffness of the board. In comparison, Taber stiffness of the sample with the lowest freeness (A8-T1) was significantly lower than the others due to it lower caliper.
Roughness and brightness tests were done on top and bottom side of the board samples. Bottom side that was in contact with the wire in DSF had a slightly higher roughness. Higher fines content in A8-T1 caused lower roughness; and since fines can improve brightness, hence it was expected that A8-T1 has the highest brightness among the other samples. While the results show that sulphite-pretreated samples’ brightness are higher due to the delignification by sulphite impregnation.

4.2.3.1 XPS results of chemically pretreated samples

Wood chips were treated by adding sodium sulphite prior to HC refining. Therefore, to investigate these elements and also their carbon bonding distribution, XPS test was performed on board sample. The specimens tested were selected from the samples included only top and middle ply, and XPS was performed on the exposed surface of middle ply, since approximate surface depth of 5 to 10 nm can be achieved by XPS. A curve-fitting program was used to obtain the information about the binding of each element. O/C ratio for all samples were almost similar, however sulphite pretreatment slightly increases O/C ratio due to more lignin removal. High resolution XPS for atom C specified that most of the carbon bonding is in forms of C-O. Higher
C1sA amount in the pretreated samples confirms that more lignin removal happened for these samples.

Table 4-9 High resolution XPS for FBBs made by chemical pretreated and no treated pulp

<table>
<thead>
<tr>
<th>Sample</th>
<th>C1s</th>
<th>C1sA</th>
<th>C1sB</th>
<th>C1sC</th>
<th>O/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>A8-T1</td>
<td>36.38</td>
<td>49.94</td>
<td>11.18</td>
<td>2.50</td>
<td>0.470</td>
</tr>
<tr>
<td>A10-T1</td>
<td>33.06</td>
<td>47.19</td>
<td>15.40</td>
<td>4.34</td>
<td>0.467</td>
</tr>
<tr>
<td>A10-T2</td>
<td>37.85</td>
<td>48.65</td>
<td>11.46</td>
<td>2.03</td>
<td>0.477</td>
</tr>
<tr>
<td>A11-T1</td>
<td>35.46</td>
<td>51.57</td>
<td>11.13</td>
<td>1.84</td>
<td>0.480</td>
</tr>
<tr>
<td>A11-T2</td>
<td>35.93</td>
<td>50.74</td>
<td>11.58</td>
<td>1.75</td>
<td>0.482</td>
</tr>
</tbody>
</table>

XPS results (Figure 4.29) for Sulphur atom indicated the presence of Sulphur at binding energy of 169 (eV) that is an indication of S-O bonding in $SO_4^{2-}$ species. This result confirmed the presence of metal sulphate in pulp furnish in the pretreated samples. Moreover, areas of the above peaks are the same for the two chemically treated samples, suggesting that the sulphite amount was similar for these samples.

Figure 4.29 - XPS results for S element in the samples
High-resolution XPS analyses for S element in the pretreated samples were done to have a precise chemical identification of this element. The s2p spectra data illustrates the presence of s2p1, s2p1A as well as s2p3A and s2p3 that are representative of s2p\(^{1/2}\) and s2p\(^{3/2}\), respectively. s2p\(^{1/2}\) and s2p\(^{3/2}\) are related to the 2p orbital showing the presence of two electrons in 2p\(^{1/2}\) and four electrons in 2p\(^{3/2}\). According to the spin orbital coupling, the intensity ratio of 2p\(^{3/2}\) to 2p\(^{1/2}\) peak is 2:1. Based on the four doublet peaks shown in Figure 4.30 and the NIST XPS database [77], different sulphur species are probable to be available in the samples, however the binding energies higher than 167 eV might be assigned to sulphate.

s2p1A and s2p1 peaks, around 161 eV, are not clear in A11-T1 and A11-T2 XPS results that confirms the low probability of atomic sulphur in the surface.

Finally the binding energy of 163 eV which is related to s2p3 might be because of disulphide, sulphide or unbound thiol [78]. Thiol is carbon-bonded sulfhydryl compound, –C–SH or R–SH, which in this case R represents cellulose. However, it is almost impossible to produce cellulose thiol without adding some catalyzers and improving the oxidation of the compounds.

![Figure 4.30 – s2P scan for (a) A11-T2 and (b) A11-T1 sample](image)

<table>
<thead>
<tr>
<th>Compound type</th>
<th>160</th>
<th>163</th>
<th>166</th>
<th>169</th>
<th>172</th>
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</thead>
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<td>S</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Sulphide</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sulphite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sulphate</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 4-10 XPS standard data ranges
4.3 Economic analysis of the project

In the introduction section it was mentioned that about 80-90% of total electrical energy of a mill devotes to the pulp refining. WPI mill is producing 550 tons of pulp per day and about 100 tons of this pulp is reject pulp. For this project, the received pulp was refined up to 600 kWh/t at high consistency refining at WPI. The results showed that board properties were improves by increasing the refining energy, however refining to achieve the freeness values between about 375-115 mL did not change the board properties significantly. Hence, refining up to 375 mL that is related to the 200 kWh/t specific energy may be considered as the optimum specific refining energy. Moreover, the required freeness value for the middle ply of FBBs is around 400 mL that corresponds to a LC specific refining energy of 200 kWh/t. Hence, rejects of HC refining required at least another 200 kWh/t of LC refining to reach the optimum Canadian standard freeness (CSF) value. Considering 100 tons per day of reject pulp after the first stage HC refining, 20% energy saving by replacement of LC instead of HC refining in the second stage, and finally the electricity price in New Zealand leads to $116,800 annual saving for LC refining of reject pulp:

Amount of energy saving: 36,500×200×0.2= 1,460,000 kWh/year
Amount of cost saving: 1,460,000×8/100= 116,800$/year

QRP produces 380,000 tons of pulp annually. Using HC- LC refining for the mainline pulp (instead of HC-HC refining) can save significant amount of energy and hence cost. Since the pretreatment did not have any effect on the board properties, the chemical reagent expenses are not included in this calculation:

Amount of energy saving: 380,000×57.6×0.2= 4,377,600 kWh/year
Amount of cost saving: 4,377,600 ×12.5/100= 547,200$/year

QRP uses 20% of their total pulp in producing FBB, hence the cost saving for this part is:
Amount of cost saving in producing FBBs: 547,200×20/100= 109,440$/year
Table 4-11 cost analysis for two used pulps in this project

<table>
<thead>
<tr>
<th>Company</th>
<th>Electricity price in the mill region (cents/ kWh)</th>
<th>Annual pulp production (tons)</th>
<th>HC SRE (kWh/t)</th>
<th>LC SRE (kWh/t)</th>
<th>Annual saving ($)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WPI (New Zealand)</td>
<td>8</td>
<td>36,500</td>
<td>600</td>
<td>200</td>
<td>116,800</td>
</tr>
<tr>
<td>QRP (Canada)</td>
<td>12.15</td>
<td>100,000</td>
<td>700</td>
<td>57.6</td>
<td>547,200</td>
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</tbody>
</table>
5 Conclusion

5.1 Application of LC refining in TMP production

The analysis of physical properties of the DSF-made middle ply FBB samples containing 25% LCR reject radiata pulp and 75% commercial BCTMPs suggested that increasing LC refining of reject pulp, i.e. decreasing the freeness values to about 400mL, improved the board properties. However, further LC refining energy application to freeness decrease to 155 mL had no significant positive effect on board properties. Replacing 25% of the middle ply furnish with the LC refined reject pulp improved the middle ply properties by introducing fines. This effect was evident in the SEM images that showed improved fibre-fibre bonding due to the fines presence in the furnish.

Analysis of laboratory-made three ply FBBs showed that increasing LCR reject radiata pulp percentage in the middle ply from 0% to 45% increased the fines content of the furnish and led to improvement of tensile index, burst index, tear index, Scott bond, and Taber stiffness. Although increasing LCR reject pulp from 0 to 100% in the middle ply furnish showed an increasing trend in FBBs’ bulk, tensile index, stretch, burst index, tear index, and Scott bond declined by using 100% of LCR reject pulp due to the short and wide reject radiata pulp fibres that made them less flexible. Comparing the boards made with 100% of each commercial pulps and the one containing LCR reject pulp showed that the latter sample boards were comparable with the commercial ones in physical properties. Additionally, changing SEL from 0.35 J/m to 0.45 J/m appeared to have little impact on mechanical properties of FBB samples.

Sulphite pretreatment of middle ply furnish prior to HC-LC refining stages had a relatively minor effect on the properties of laboratory-made three ply FBB samples. Instead, middle ply furnish freeness was more significant in affecting the properties of FBB. Sample with the middle ply pulp with lower freeness value of 200 mL are much stronger than the target freeness value of 400mL.

Cost analysis showed that replacing HC-HC by HC-LC radiate reject refining could lead to 1,460,000 kWh/year and 116,800$/year energy and cost saving respectively. In addition, using
HC- LC refining for the mainline pulp of QRP mill (instead of HC-HC refining) showed 4,377,600 kWh/year energy saving that is equal to 547,200$/year cost reduction.

5.2 Future works

This work could be extended to examine various reject pulp samples with different characteristics to better examine factors such as the fibre length, coarseness, etc. Also, a wider range of chemical pretreatments in terms of degree and type of treatment, including alkaline, and hydrogen peroxide, etc., could be investigated. Therefore, it should be noted that the results obtained in this study, is restricted to one type of reject pulp (i.e. Radiata pine reject pulp), and one type of chemically pretreated pulp (SPF).

An exact range of freeness needs to be considered with the sulphite pretreatment versus no chemical using of middle ply pulp using in FBBs to eliminate the effect of freeness on the final product properties.

In this project, using LCR eject pulp and LCR chemical pretreatment was studied separately, however it is very useful to use chemical pretreatment for the reject pulp prior to LC refining in producing multi ply FBBs.

In this study, creasing and folding endurance tests were not available. However, since these properties are important factors in producing FBBs, it is suggested to do theses test for different LCR reject ratios in the middle ply and with different chemical pretreatments.

Different additives can be used in furnish to enhance the board physical properties. Hence it is suggested to use Chitosan, starch, micro fibre cellulose, etc. to reinforce the FBBs.

An assessment of the process with using paper machine instead using DSF is to be considered to compare the results in the mill scale.

A more detailed economic analysis regarding of the wood chips price, transferring, chemical reagents expense is suggested.
References


Finland, 2008.


Appendix

According to equation 10, middle ply strength has low contribution in the Taber stiffness, however, since the total caliper of the board can change Taber stiffness significantly, higher bulk of middle ply bulk improves this property.

The middle ply contribution in total Taber stiffness for the boards containing different LCR reject pulp was determined as follows:

\[
\text{Middle ply contribution, } \% = \frac{S_b}{E_1(t_1^2)} \times 100
\]

\( S_b \) values are taken from Figure 4.22 b, \( E_{\text{middle}} \) was estimated using Figure 4.20b, and \( t_{\text{middle}} \) was determined as follows:

\[
t_{\text{middle}} = t_{\text{total}} - t_{\text{top}} - t_{\text{bottom}}
\]

Where, \( t_{\text{top}} \) and \( t_{\text{bottom}} \) were equal and they were determined by testing the caliper of a 45 gsm single sheet made by the chemical pulp.

<table>
<thead>
<tr>
<th>LCCR percentage (%)</th>
<th>LCRR</th>
<th>( S_b ) (g.cm)</th>
<th>( S_b ) (N.m)</th>
<th>( t_{\text{total}} ) (µm)</th>
<th>( t_{\text{middle}} ) (µm)</th>
<th>( E_{\text{middle}} ) (N/m²)</th>
<th>Middle ply contribution in total stiffness (%)</th>
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<tbody>
<tr>
<td>SEL:0.35 J/m, MD</td>
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