# Assessment of Some Wood Properties by Near Infrared Spectroscopy

by

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Assessment of Some Wood Properties by Near Infrared Spectroscopy

submitted by Samuel Ayanleye

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the degree of <u>Master of Science</u>

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# Abstract

Near-infrared spectroscopy (NIRS) is a suitable technique for characterizing many materials, including wood, and has been used to predict several wood properties. However, existing reports on this use of NIRS have paid little attention to the effect of wood surface condition and grain orientation. This study therefore used NIRS to assess wood density, modulus of elasticity, modulus of rupture, grain angle, and annual ring width, studying whether and how surface condition and grain orientation affected the measurement of these properties.

The research focused on using NIRS coupled with partial least squares regression (PLS-R) to predict the properties of two softwoods (Western hemlock and Douglas-fir). PLS-R models were calibrated and validated using the test-set validation method. The predictive accuracies based on grain orientation (quarter-sawn and flat-sawn) and wood surface condition (rough and smooth) were compared. Models developed using reduced wavelengths also showed the possibility of predicting these properties using a narrow spectral range.

The results of this study showed that calibrations based on mixed sets, which included both crosssections, were inferior to those based on these cross-sections separately. Promising predictive models were obtained for density ( $R_p^2 = 0.66$ ), modulus of elasticity ( $R_p^2 = 0.78$ ), and modulus of rupture ( $R_p^2 = 0.82$ ), with poor correlations for grain angle and annual ring width ( $R_p^2 \le 0.50$ ). Further, the rough surface predictions outperformed those from the smooth surface for all properties. The quarter-sawn sections also showed better predictive ability than the flat-sawn sections for both surface conditions. The only exception was for modulus of rupture, where the trend was reversed. The results therefore show the potential for using NIRS as a non-destructive technique to predict the properties of wood.

# Lay Summary

The main goal of this thesis was to assess the feasibility of using NIRS to predict the properties of two softwoods based on different surface conditions and grain orientations. NIRS is a rapid analysis technique useful for predicting wood properties. This technique has several advantages, including scant need for sample preparation, and the non-destructive nature of sampling. However, the reliability of this method depends on the accuracy of spectral acquisition and reference measurements. While plenty of research has used NIRS to assess wood properties, little attention has been paid to applying the method to two species in Canada: Western hemlock and Douglas-fir. This study therefore focused on using NIRS to predict the properties of these softwoods. Using the partial least squares procedure, I built NIRS calibrations to predict certain wood properties and observed the influence of surface condition and grain orientation on NIRS performance.

# Preface

I, <u>Samuel Ayanleye</u> affiliated to the Wood Physics and Drying Laboratory at the University of British Columbia, Point Grey Campus, conducted the work presented in this thesis. I was responsible for all the experiments, data collection, analyses, result interpretation and wrote the thesis.

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# Dedication

To the Ayanleye family, who has always been a continuous source of support and motivation to me during the tough moments of life. I will be forever thankful for your unconditional love towards me.

# **1.0 INTRODUCTION**

Wood is an important construction material that requires high degree of structural performance and reliability (Liang *et al.* 2016). Consequently, it has a broad range of applications (Nguyen *et al.* 2017). Wood is an anisotropic, hygroscopic, and heterogenous bio-based material with a porous structure that presents variations between species at both microscopic and macroscopic levels (Hans 2014). This variation is one of the most important challenges that wood product producers face when trying to accurately characterize quality for appropriate end product design and manufacturing (Schajer and Orhan 2006, Fujimoto *et al.* 2008).

Western hemlock is the single most plentiful tree species found on the coast of British Columbia (BC), making up 18% of the volume of BC's growing stock (Middleton and Munro 2001). Hemlock wood is of medium density, has moderate strength and stiffness, and is mostly used for construction purposes (Kenneth 2001). Douglas-fir (D-fir) makes up to 8.8% of the provincial growing stock (D-fir species profile n.d.) and grows along the BC coast and in the interior of the province. Coastal D-fir wood is known for its superior strength, stiffness, and higher density (Cherry *et al.* 2008) and is mostly used as dimensional timber, plywood, flooring, and in construction (Johnson and Gartner 2006).

Wood density ( $\rho$ ) influences its physical and mechanical properties and is regarded as an important measure of wood quality (Jozsa *et al.* 1989). The  $\rho$  within a tree increases radially from pith to bark and decreases vertically with height in the stem (Kiaei *et al.* 2015, Tsoumis 1991, Todoroki *et al.* 2012). The moduli of elasticity and rupture (*MOE* and *MOR*) are two important properties used in choosing wood as a structural material (Missanjo and Matsumura 2016). *MOE* is an indication of stiffness, while *MOR* is an indication of the strength of a board or structural member (Johnson and Gartner 2006). The annual ring width (*ARW*), which is composed of earlywood with thinner walls and latewood with thicker walls—and thus has a smaller lumen size and higher  $\rho$  (Fromm *et al.* 2001)—has been reported as an important variable in the prediction of *MOE* and *MOR* of wood (Haartveit and Flæte 2002). The slope of grain (*SOG*) relative to the length of a timber is another factor that contributes to understanding wood's strength properties. The strength varies with the orientation of the grain, making the measurement of *SOG* an important requirement for machine grading and structural analysis (Ingemi and Yu 2019).

Uncertainty about some of the properties of wood has led to the development of prediction methods by indirect observation—that is, obtaining measurements without damaging the material or altering its properties (Romer 2013). Measurements of  $\rho$  and *SOG*, which directly affect a product's mechanical properties, are important to the wood industry. Although traditional methods have been employed to estimate these properties, a suitable method that is non-destructive, swift, easy to implement within the production line, and requires minimal sample preparation is highly desirable.

Near infrared spectroscopy (NIRS) is the measurement of the wavelength and intensity of a material's absorption of near-infrared light. NIR light spans the 800–2500 nm range and is energetic enough to excite overtones and combinations of molecular vibrations to higher energy levels. NIRS is normally used for the quantitative measurement of functional groups, especially O-H, N-H, and C-O, containing physical and chemical information about a sample (Schimleck *et al.* 2001). The advantages of NIRS include contactless application, fast acquisition times, non-destructive sampling, and the potential for on-line or portable applications (Druckenmüller *et al.* 2018). NIRS has been successfully applied to estimate several properties of wood, such as moisture content (M), density ( $\rho$ ), and mechanical properties (Leblon *et al.* 2013, Acquah *et al.* 2018).

This study examines the use of NIRS to predict the density, bending strength, stiffness, grain angle, and annual ring width of hemlock and D-fir. The hypothesis is that NIRS will be a swift and effective tool to predict the aforementioned properties using multivariate analysis of the acquired spectra in real time. Such capability may allow for sorting before processing and/or property evaluation, thereby increasing product quality and decreasing processing costs.

# **1.1 Outline of thesis**

Chapter 1 introduces what the research was about and why it was carried out. Chapter 2 provides background information on the non-destructive evaluation of wood, as well as spectroscopy, specifically explaining the NIRS utilized in this study. It also explains Western hemlock and D-fir properties and applications, and describes in detail the application of NIRS technology in wood property assessment. Chapter 2 ends with a statement of the thesis' objective. Chapter 3 provides

information about the study material and methods: sample preparation, NIR measurements, procedures for testing wood properties (density, *MOE*, *MOR*, grain angle, and annual ring width), and the method of analysis. Chapter 4 presents the results of the study and discusses them in relation to the objective of the thesis. Finally, Chapter 5 draws general conclusions from this research and makes recommendations for further studies.

# 2.0. LITERATURE REVIEW

#### 2.1. Non-destructive Evaluation of Wood

Non-destructive evaluation (NDE) is a general term that encompasses various methods or techniques used to assess material properties, components, or entire process units without damaging the material in question. These techniques can also detect, characterize, or measure the presence and magnitude of natural and processing defects in wood (ASNT 2016). The importance of these methods increases as the timber industry continues to seek better ways to test wood products in a non-destructive manner. These approaches have also gained attention for the evaluation of complete wooden structures (Dunn 1992).

The instruments used for NDE may be quite diverse, and some of their benefits and limitations have been reported in detail (Bucur 2003, Niemz and Mannes 2012). NDE as a method of identifying the physical and mechanical properties of wood, from standing trees to in-place structures, does so without altering the material's end-use capabilities. The resulting information can be used to make decisions about appropriate applications and design changes (Ross *et al.* 1998). Research on NDE arose mainly from the need to provide methodologies or techniques for assessing wood and wood-based materials and thereby to promote better decision-making processes regarding the use of the products (Brashaw *et al.* 2009).

Historically, the wood community has developed and used various NDE techniques in applications such as sorting or grading structural products. Two common examples are machine stress rating (MSR) of timber and ultrasonic grading of veneer. In North America, MSR grading couples visual sorting criteria with NDE practices to assign a timber to an established grade (Galligan *et al.* 1977). Laminated veneer manufacturing facilities use stress wave NDE techniques for sorting incoming veneer into strength classes before final product assembly. These are established through empirical relationships between the travel velocity of induced stress waves through the veneer, and its strength (Sharp 1985). A detailed review of NDE techniques, especially those utilized for the assessment of wood properties, was conducted by Ross (2015).

# 2.2. The Physics of Light and Spectroscopy

## 2.2.1 Physics of light

Light is defined as any natural agent that stimulates the sight and improves the visibility of things (Sliney 2016). It has also been regarded as a fraction of the radiation that can excite the human visual system (Zwinkels 2015). Visible light is the electromagnetic radiation (EMR) within the electromagnetic spectrum that is easily observed by the human visual system (CIE 1987). In physics, "light" sometimes refers to EMR of any wavelength, whether it is visible or not (Smith 2006). The known properties of light include intensity, direction of propagation, frequency or wavelength, polarization, and speed (Uzan and Leclercq 2008). Like other EMR, light propagates as waves, and the energy imparted by these waves is absorbed, reflected and transmitted. The absorbed energy of these waves is called a photon and represents quanta of light.

For several centuries, scientists disagreed about whether light should be regarded as particles or waves. Sir Isaac Newton, after conducting various experiments in the 17<sup>th</sup> century, argued that the nature of refraction and reflection could only be explained if light were composed of particles, as waves would not travel in straight lines. Although many physicists subsequently attempted to articulate a wave theory of light, this concept was not accepted until Sir Thomas Young experimentally demonstrated wave interference (Young 1807). Physicists later reached a consensus that light could be characterized concurrently as both a stream of particles and a wave, depending on the type of experiment (Sliney 2016).

There are three main methods of dispersing radiation: refraction, diffraction, and interference. The interactions between particles such as protons and ions, and their interactions with other particles due to collision energy, is a field of study known as "spectroscopy."

# 2.2.2. Spectroscopy

The field of spectroscopy is concerned with studying the interactions between electromagnetic waves and matter. Light interacts with the atoms and molecules of matter in several ways, through absorption and emission. Measurement of these interactions permits us to identify specific wavelengths of light that interact with the atoms and molecules, and to measure the amount of light that is absorbed or emitted at certain wavelengths (Spectroscopy 2017).

Spectroscopy falls into two types: atomic spectroscopy (AS) and molecular spectroscopy (MS). AS comprises atomic absorption and atomic emission spectroscopy, while MS comprises mass spectroscopy, infrared spectroscopy, nuclear magnetic resonance, and ultraviolet and visible spectroscopy. In AS, every element has a distinct atomic structure characterized by a positively charged nucleus surrounded by the number of electrons necessary to maintain neutrality (Sahin 2019). In MS, EMR interacts with a material and produces a spectrum from which relevant compositional and structural information about the material can be deduced (Goodman 1994).

Spectroscopic experiments primarily use light sources that emit radiation of discrete and welldefined frequencies (Chu *et al.* 2018) and its data are represented by a range —for instance, a plot of the material's reflectance/absorbance as a function of wavelength/frequency (Sandak *et al.* 2016). This spectrum is indicative of the material's molecular structure and has unique properties, including band position, width, and intensity, which are all used to obtain information about functional groups or to monitor molecules under different conditions (Mollaoglu *et al.* 2018). The spectral response is used to either qualitatively determine the chemical constituents or quantitatively determine the amounts of specific compounds present in the material.

# 2.2.2.1. Infrared spectroscopy (IRS)

IRS is an analytical technique for the characterization of a wide range of materials and is commonly used in research and production for quality control purposes (Theophanides 2012). This technique involves the interaction between infrared radiation (IR) and matter and is mostly used to identify and quantify various organic compounds present in a material, via their selective IR absorption. The history of IRS dates back to the 19<sup>th</sup> century, and the first spectrometer was built in 1835. By the beginning of the 20<sup>th</sup> century, IRS had become a valuable tool for identifying and characterizing chemical compounds and materials (Theophanides 2012). Thereafter, it was applied to more complicated molecules such as proteins (Elliot and Ambrose 1950) and later, DNA (Colthup *et al.* 1990).

IRS is a form of vibrational spectroscopy. The absorption of IR radiation by matter results in molecular bond vibrations. Each molecule possesses unique vibrational characteristics, and the resulting IR spectra make vibrational spectroscopy a good method for characterizing molecular

structures. It is a reliable way to investigate functional, structural, and compositional changes in cells, tissues, and biological molecules. It is also one of the most important analytical techniques used to study almost all types of liquids, solutions, and pastes (Mollaoglu *et al.* 2018).

The IR range refers to the part of the electromagnetic spectrum between the visible and microwave regions, and it is divided into three regions: near-infrared, mid-infrared, and far-infrared (Anastasopoulou and Theophanides 1972). The IR energy excites the molecules at the surface of the material, and part of the energy is absorbed selectively and proportionally to the quantity of the excited molecules (Sandak 2013). Different molecular interactions, such as, C–H, O–H, and N–H, are stimulated and often result in phenomena such as stretching (symmetrical or non-symmetrical), scissoring, rocking, wagging, and twisting—phenomena that depend on factors such as the molecular structure, chemical composition, or physical properties of the surface measured (Coates 2000).

An instrument called a "spectrometer" measures the light that is reflected or transmitted from a sample and uses a detector to plot transmittance through the molecules against the wavelength of the radiation. The resulting spectrum is better visualized in graphs of transmittance/reflectance plotted against wavelength (Infrared Spectroscopy n.d.). This type of information can be obtained from a single experiment, and from small samples or powders.

### 2.2.2.2. Near infrared spectroscopy (NIRS)

NIRS is a non-destructive technique for analyzing organic materials that is rapidly growing in popularity. Although the use of spectrophotometers to measure spectra is an old technique, the NIR region of the electromagnetic spectrum was not properly utilized, as it was considered to contain non-structural information (Barton 2004). The earliest analytical application of NIRS was to determine the moisture content (M), crude protein, and oil concentrations of cereal grains (Ben-Gera and Norris 1968a); it was also used in the agriculture, food, paper, polymer, and textile industries (Ciurczak 1992, Barton 2004).

NIRS is a potentially rapid tool that can be used to get information about wood products in the industry. NIRS has been successfully applied in wood science and technology, as reported elsewhere (So *et al.* 2004, Tsuchikawa 2007, Leblon *et al.* 2013). It has been used to assess *M* 

(Hoffmeyer and Pedersen 1995, Adedipe and Dawson-Andoh 2008),  $\rho$  (Thygesen 1994, Schimleck *et al.* 1999), wood chemical properties (Schimleck and Evans 2003, Jones *et al.* 2005*a*), and mechanical properties (Gindl *et al.* 2001; Kelley *et al.* 2004a; Kothiyal and Raturi 2011). It has also been used to estimate the properties of normal and modified wood as well as wood composites (Schimleck *et al.* 2002, Blanco and Villarroya 2002, Li *et al.* 2010).

As solid wood is an opaque material, EMR has difficulty passing through thick samples, so NIR spectra are collected via reflection (Hans 2014). In reflectance spectroscopy, there is an interaction between the radiation energy sent by the source and the material, producing scattering, transmission, and absorption (Leblon *et al.* 2013). The radiation energy reflected from the sample is then measured. Details about reflectance and absorbance spectroscopy with their equations have recently been reported by Hans (2014). Detailed reviews of the application of NIRS to wood and forest products have been presented by So *et al.* (2004) and Tsuchikawa (2007).

# 2.2.2.3. Effect of surface roughness on NIR spectra

Wood is a porous material whose surface features are the result of machining conditions and the wood's anatomy (de Moura Palermo *et al.* 2014, Zhang *et al.* 2015). Surface roughness is difficult to control, as it is an important index for wood product quality that is directly affected by wood machining and bonding performance (Ozdemir and Hiziroglub 2007). The surface quality of wood is important in developing NIR-based models for predicting wood properties, as previously reported (Hoffmeyer and Pederson 1995, Costa *et al.* 2018). However, NIR-region spectral absorbance is reported to be inversely proportional to wood surface roughness (Schimleck *et al.* 2005a).

Previous studies have shown the influence of surface roughness on the NIR spectra of wood. Some authors found the predicted results of NIR-based models for smooth surfaces to be better than those for rough surfaces (Schimleck *et al.* 2005b, Liu *et al.* 2006). Contrasting results have also been reported (Costa et *al.* 2018, dos Santos *et al.* 2020), where rough surface calibrations outperformed those from smooth surfaces. Better predictions from samples of greater roughness could be attributed to the capture of more information by the EMR during NIR spectral acquisition (Costa *et al.* 2018).

# 2.3. Western Hemlock and Douglas-fir

# 2.3.1. Distribution, properties, and uses of Western hemlock

Western hemlock (*Tsuga heterophylla*) is the most abundant species along the coast of BC, and it also grows in the interior wet belt of the Rocky Mountains (Parish 1995). The standing volume of matured hemlock is about 1.3 billion m<sup>3</sup> and represents about 18% of the total growing stock in BC (Shahverdi 2015). The best climate for its growth is mild or humid weather that is characterized by frequent fog and precipitation during the growth season in the coastal region (Pojar *et al.* 1991). It grows in pure stands or in mixture with other species, in even-aged stands where the climate is cool and moist.

Hemlock is a strong wood which is suitable for structural applications (Rohrbach 2008). It also has a straight and even grain with a medium to fine, coarse, and lustrous structure. The sapwood and heartwood are often difficult to distinguish (Nourian 2018). In green wood, the sapwood appears to be a little darker due to the large amount of free water in it (*M* is sometimes over 200%). Once the free water is evaporated when the wood is dry, there is no colour difference between the two areas, making it impossible to visually separate them. However, the growth rings are distinct and uniform in width, albeit narrow, delineated by a band of darker latewood with a purplish to reddish-brown tinge that is visible to the eyes. Approximately two-thirds or more of the growth ring is occupied with earlywood, and the transition from earlywood to latewood is gradual (Hoadley 1990).





Western hemlock

ky mountain bougias-iir 🔛 bougias-



**Figure 2.1**: The distribution map of western hemlock and Douglas-fir in the Pacific Northwest (Natural Resources Canada, 2015).

Hemlock is also known for the presence of wetwood, which is a portion of the heartwood zone with higher *M* and  $\rho$  than adjacent parts. This zone is related to bacterial activity and is difficult to dry (Shahverdi 2015, Nourian 2018). The average  $\rho$  of hemlock is 470–490 kg/m<sup>3</sup> (Rohrbach 2008). The literature also stated an average *MOE* value ranging from 10,000 to 12,300 MPa and an average *MOR* value ranging from 75 to 81.1 MPa (Coast Forest Products Association 2003).

The strength and the nailing characteristics of hemlock have made it a popular construction material in North America and overseas (Shahverdi 2015). It is commonly used for general construction, decking, plywood manufacturing, stock lamination, and the production of glue-laminated and solid beams. Other uses of hemlock include the manufacture of doors, windows, and floors, and in applications where a high-grade softwood is needed (Parish 1995). It is also exported to Japan for posts and beams in roof rafters and traditional housing (Lazarescu and Avramidis 2012) and is the most important pulpwood species in BC (Softwood Export Council 2004, Rohrbach 2008).

## 2.3.2. Distribution, properties, and uses of Douglas-fir

Douglas-fir (*Pseudotsuga menziesii*) is one of the most popular softwood species in North America, originally named after a Scottish botanist (David Douglas) whom the Royal Horticultural Society in the late 1700s directed to study this tree (Hebda 1995). Two common native species of *Pseudotsuga menziesii* are *P. menziesii var. menziesii*, called coastal D-fir, and *P. menziesii var. glauca*, referred to as interior D-fir (Hermann 1982). The range of coastal D-fir extends from central BC (55°N) south along the Pacific Coast ranges into central California to a latitude of 34.44°N (Hermann and Lavender 1999), while the interior D-fir ranges from northern Mexico (19°N) to the north of BC (55°N) (Zustovic 2015).

D-fir is characterized by a straight or slightly wavy grain with a medium to coarse texture. The sapwood and heartwood of D-fir are not difficult to distinguish, unlike hemlock, as the sapwood is light in colour and the heartwood ranges from yellowish to reddish-brown with sharply defined bands (Leavengood 1998). Also, in contrast to hemlock, the transition from earlywood to latewood is abrupt (Osborne *et al.* 2016). D-fir wood is very hard and resistant to abrasion, making it very suitable for applications where wear is a factor, as in bridge parts and commercial buildings. An

average  $\rho$  of 450 kg/m<sup>3</sup>, and *MOE* and *MOR* values of 13,500 MPa and 88.6 MPa, have been reported for D-fir (Jozsa *et al.* 1998).

The higher strength of D-fir and the availability of large dimensions from old-growth trees make it a good choice of material for the construction industry. It has been identified as one of the finest timbers for heavy structural purposes, including glulam beams, laminated arches, and roof trusses (Douglas-fir Species Profile n.d). D-fir is used primarily in general construction and as dimension timber, plywood, and laminated veneer timber (Johnson and Gartner 2006, Ukrainetz *et al.* 2008).

#### 2.4. Selected Wood Properties

#### 2.4.1 Density

The density of wood ( $\rho$ ) is defined as the mass or weight per unit of volume (both at the same *M*), usually expressed in kilograms per cubic meter (kg/m<sup>3</sup>) or grams per cubic centimeter (g/cm<sup>3</sup>) (Haygreen and Bowyer 1996). Density is known to be influenced by the amount of water in the hygroscopic range, namely, 0 to 30% *M* (Antwi-Boasiako and Atta-Obeng 2009), which therefore allows for comparison of values only under the same *M* conditions (Kollman and Cote 1968).

Density is one of the most important properties of wood and is known to vary significantly within and between trees (Louzada 2003). This variation can be attributed to several factors, such as differences in cellular structure resulting from physiological and hereditary characteristics, and the presence of extraneous components (Rathgeber *et al.* 2006). For instance, the  $\rho$  of heartwood is usually higher than that of sapwood due to a higher concentration of extractives such as terpenes, resins, and polyphenols in heartwood (Hans 2014). In addition, past studies show that  $\rho$  variation in wood is usually affected by annual ring width (Fabris 2000) and varies from the earlywood to the latewood within rings (Gartner *et al.* 2002).

Density is considered important because it influences wood attributes such as shrinkage, swelling, and mechanical properties (Haygreen and Bowyer 1989), and because of its effect on the general quality of final products (Anjos *et al.* 2010). In many cases,  $\rho$  is particularly important to tree breeders and is usually evaluated when there is large within-species variation, a high degree of genetic control, and easy assessment (Santos *et al.* 2012).

### 2.4.2. Mechanical properties

The mechanical properties of wood reflect its ability to resist applied forces that tend to deform it in any manner. Wood with greater resistance to deformation has a higher strength, and the ability of wood to resist applied load depends on the magnitude and direction of the force in relation to its fiber orientation. Wood's mechanical properties therefore affect the quality of the products used in load-carrying applications (Haartveit and Flæte 2006).

Many methods have been identified for measuring the mechanical properties of wood, such as the modulus of rupture (*MOR*) and the modulus of elasticity (*MOE*). *MOR* is a measurement of the maximum stress that the material can endure before failure occurs and is measured in Newtons per square meter (N/mm<sup>2</sup>). *MOE* measures the deformation that results from a given level of stress before the point of total failure and is also measured in N/mm<sup>2</sup>. Apart from their importance in structural lumber, *MOR* and *MOE* are also valuable traits in standing trees because they reduce mortalities that results from stem failures and the uprooting of trees during harsh weather conditions (Lachenbruch *et al.* 2011).

Destructive and NDE techniques are commonly used to measure the mechanical properties of wood. The destructive technique is based on the direct estimate of the *MOE* and *MOR* of wood using static bending tests, where a load is applied at mid-span to a piece of wood supported at its ends (ASTM 2005). The NDE tests are based on propagation speed using either acoustic or ultrasonic waves, or on acoustic spectral analysis in which mechanical properties can easily be estimated. In studying the mechanical properties of wood, it is important to know that those relating to bending strength include, among others, compressive strength, shear strength, and *MOR*, while the properties relating to wood elasticity include *MOE*, plasticity, resilience, and Poisson coefficient (Andrade *et al.* 2010).

#### 2.4.3. Slope of grain (SOG)

The *SOG* of wood refers to deviations other than those due to spiral grain or its related phenomena, such as interlocked and wavy grain, and is commonly referred to as cross-grain in sawn timber (Anderson *et al.* 1945). As wood is highly anisotropic, *SOG* exerts a strong influence on its material properties (Bodig and Jayne 1982). For instance, at a *SOG* of 45°, Young's modulus and axial strength are reduced to 10–25% of the value at 0° (Kollmann and Cote 1968), whereas

longitudinal shrinkage increases considerably. Therefore, *SOG* remains a considerable factor when lumber is graded, especially for load-bearing applications (Piazza and Riggio 2008).

*SOG* has been previously evaluated using thermal anisotropy (Naito *et al.* 2000), X-ray computed tomography (Sepulveda 2001), transmission measurements (Malik *et al.* 2005), and microwave technology (Schajer and Orhan 2006). In addition, past researchers have proposed a *SOG* indicator using a rotating capacitance-type transducer sensor (McDonald and Bendtsen 1986, Samson *et al.* 1993). While measuring the *SOG* of wood is often difficult (Sepúlveda 2001), its potential for predicting the mechanical properties of timber has been established (Olsson *et al.* 2013, Viguier *et al.* 2015).

#### 2.4.4. Annual ring width (ARW)

Wood structure is mainly composed of tubular fibre cells that are cemented together and normally created by the way the trees grow. Every growing season, two layers are formed on the outside of the trunk, resulting in earlywood and latewood. Annual ring width (*ARW*), defined as the sum of earlywood and latewood for that season, is important because of its correlation to wood mechanical properties (Alteyrac *et al.* 2006). *ARW* is also one of the most important factors that directly affect wood's physical and mechanical properties (Nathalie and Malo 2010, Ibrahim and Ayşenur 2016) and its durability (Takata *et al.* 2002).

Past researchers have reported the influence of *ARW* on the properties of wood. For instance, Karlman *et al.* (2005) showed in their work on different species of larch that an *ARW* up to 2.5 mm had the highest  $\rho$ , while there was a marked reduction in  $\rho$  where the *ARW* was wider than 3 mm. Likewise, Ethington *et al.* (1996) incorporated variations in *ARW* and observed that the *ARW* has a significant effect on the compressive strength perpendicular to the grain of the wood. Lastly, statistical analyses performed on five different species showed the effect of *SOG* and *ARW* on the shear strength and *MOE* of the species (Lang *et al.* 2000, 2002).

#### 2.5. NIRS Wood Assessment

### 2.5.1. Density

The first known study that reported the correlation between NIR spectra and  $\rho$  was presented by Thygesen (1994), where solid wood of Norway spruce was used to predict the  $\rho$  of wood. Several

studies have since reported that NIRS can be used to estimate the  $\rho$  of wood (Hoffmeyer and Pedersen 1995, Viana *et al.* 2009, Hein *et al.* 2009a).

A number of studies have reported good calibration of  $\rho$  using NIRS. Alves *et al.* (2012) reported PLS-R models for  $\rho$  based on X-ray micro-density data for each species of *Pinus pinaster* and *Larix* × *eurolepis*. The common model provided a residual prediction deviation (RPD) of 3.1, and the single models for *Pinus pinaster* and *Larix* × *eurolepis* provided RPDs of 3.5 and 3.2, respectively. Also, Fujimoto *et al.* (2012) used NIRS to estimate the  $\rho$  of wood independently of its *M* regardless the fact that the increase in *M* is reported to greatly affect the variation of NIR spectra (Fujimoto *et al.* 2012, Via *et al.* 2003). In the same way, Inagaki *et al.* (2010) developed a PLS-R model for the prediction of the air-dried  $\rho$  of *Eucalyptus camaldulensis* with an RPD of 3.8. In their experiment, they proved that the model is quite robust and stable by removing 40% of the samples during the cross-validation step and ended up with an RPD of 3.2. The use of NIRS and multivariate analysis to predict  $\rho$  has been fully established (Hoffmeyer and Pederson 1995, Via *et al.* 2003).

### 2.5.2. Mechanical properties

Several reports have previously described the use of NIRS to estimate the mechanical properties of wood (Schimleck *et al.* 1999, Fujimoto *et al.* 2008, Hein 2010). For instance, NIRS was used to predict the *MOR* of *Eucalyptus grandis* and *Eucalyptus urophylla* (Thumm and Meder 2001). In the same way, NIRS was also utilized to predict the strength and elasticity in compression parallel to the grain of wood with good correlation coefficients of 0.78 and 0.75 (Hein *et al.* 2009b). Past researchers have also modeled the *MOE* and *MOR* of *Pinus palustris* of different wood types—juvenile, mature, and pith (Via *et al.* 2003). The poor performance of the models was linked to the narrow range of values for the species utilized in the study. Similarly, others (Rials *et al.* 2002, Adedipe and Dawson-Andoh 2008) have applied NIRS to estimate the *MOE* and *MOR* of solid wood and wood composites. Hence, this technique has been established as a very effective, reliable, and practical method of estimating the mechanical properties of softwoods (Kelley *et al.* 2004a).

#### **2.6.** Chemometrics and Multivariate Calibration

Chemometrics has been fully explored for the extraction of physical, chemical, and mechanical information from spectral data by employing multivariate data analysis techniques in the estimation of wood properties. Multivariate analysis methods such as PCA, PLS-R, principal component regression (PCR), and soft independent modelling of class analogy (SIMCA) are useful in the extraction of relevant information from spectroscopic measurements. This is possible over a broad wavelength range, allowing all data to be used in the analysis (Leblon *et al.* 2013). The advantage of using multivariate analysis techniques is that the wavelength variables tend to be collinear, and the information extracted or mined is in the correlation patterns instead of individual data points (Eriksson *et al.* 2001). Most of the published studies and reviews on the application of NIRS in the forest products industry have applied chemometrics to predict several properties of wood and wood products from NIR spectra (So *et al.* 2004, Tsuchikawa 2007).

Several regression methods have been used to quantitatively derive information from reflectance or absorbance spectra. Some of the methods, such as MLR, support vector machine (SVM), PCR, and PLS-R, have been reported on (Steven *et al.* 2010, Atzberger *et al.* 2010, Vohland and Emmerling 2011). A detailed comparison of many of these methods has been attempted in the past (Viscarra Rossel and Behrens 2010). The PLS-R method has become one of the most popular for chemometrics in recent years (Wold *et al.* 2001) and was the method utilized in this study.

There are many kinds of multivariate data modelling, such as PCA modelling, which models only the X matrix. In PCA, a principal component model of the essential structure of the data is built to gain an overview of the data structure. Multivariate calibration is concerned with the X and Y matrices, with X being the independent variable (spectra) and Y being the dependent variable (property of interest). The multivariate model for (X, Y) is a logistic relationship between the empirical X and Y relations, and establishing and calibrating such a model is the first stage in multivariate modelling (Esbensen *et al.* 2002). The second stage is prediction.

In multivariate modeling, a set of known X and Y values are first used to develop the model, which is then used to predict the Y values from new X measurements (spectra which is also the predictor variable). The interesting point here is that the model makes it possible to use only X measurements (spectra) in future predictions instead of taking more Y measurements ( $\rho$ , *MOE*, *MOR*, among others) that are very expensive, time-consuming, laborious, dangerous, and in many cases destructive. These characteristics of the Y values make it desirable to replace them with X measurements, such as spectra, that are simple to use, faster than other methods, and cheaper to collect. NIRS could therefore be used to replace slow and cumbersome experiments such as physical, mechanical, and wet chemistry measurements.

The calibration data must meet several requirements, such as being representative of the future population from which the new X-measurements are to be sampled, and the measuring conditions should also be broadly similar. However, they should not be too similar, because the only difference will then be the sampling variance, that is, the variance due to two independent samplings from the target population. The determination of the predictive ability of a PLS-R model must be accompanied by the separation of the data sets of the property (y variable) being studied in the training or calibration set used in developing the model. An external or independent test set based on the calibration set is then used to validate the model and determine its predictive ability (Hauksson *et al.* 2001).

### 2.6.1. Effect of spectral pre-treatment on chemometrics

The use of spectral pre-treatments in chemometrics has been widely studied. For instance, derivatives were applied to absorption spectra to gain enhanced spectral information for the spectral ranges during a decomposition process (Ben-Dor *et al.* 1997). In the same way, min-max normalization, first derivative, and vector normalization after mean centering have been used to improve spectral quality (Udelhoven *et al.* 2003). Vasques *et al.* (2008) also applied various pre-treatments such as derivatives, normalization, and non-linear transformations on 554 soil sample spectra from Florida to estimate their organic carbon content. The effect of log 1/R transformation, first derivative, and SNV-DT on soil diffuse reflectance spectra (Stenberg and Viscarra 2010), untransformed spectra, and first and second derivative with gaps of one to 64 bands have also been analyzed to estimate several variables for airborne hyperspectral data (Hively *et al.* 2011).

A detailed review of the most common pre-treatments for near-infrared spectra in chemometrics was published in the last decade (Rinnan *et al.* 2009). Most of the work done on NIR analysis of wood and wood products has used simple spectral pre-treatments such as 1<sup>st</sup> or 2<sup>nd</sup> derivatives, or

multiplicative scattering correction. Other analytical techniques have been used to improve the quality of the models constructed using NIR spectral data (Axrup *et al.* 2000).

#### 2.6.2. Effect of grain orientation and surface roughness on NIRS calibrations

One of the factors that influences NIRS calibrations is wood anisotropy (Hans 2014). There are three sections in a piece of wood: transverse, radial (quarter-sawn, Qs), and tangential (flat-sawn, Fs). The effect of these sections on the NIR spectra of wood has been reported (Leblon *et al.* 2013). The sections of wood to be used for calibration must be carefully selected to suit the application so that the section from which spectra are collected contributes to the performance of NIR-based models. For instance, calibrations developed based on transverse sections to estimate the *M* of red oak lumber were better than those developed from Qs and Fs (Defo *et al.* 2007).

The transverse section has been reported to result in better predictions, which is attributable to the distinct anatomical differences within the growth rings. The differences between the measurements for the various sections could also be explained by the way scanning is carried out. With a transverse section, incident radiation directly interferes with free water in the cell lumen, which is not the case when scanning other sections, since the radiation first has contact with the cell wall (Tsuchikawa *et al.* 1996). The models based on Fs sections are known to have the poorest performance. This is because of the presence of only earlywood or latewood portions when scanning is done which thus affects the calibrations (Hans 2014).

An additional factor influencing NIRS calibration is surface roughness. Roughness of wood on transverse sections is usually higher than on Qs and Fs sections (Tsuchikawa *et al.* 1996). Also, transversal sections have tracheid cells with a longitudinal axis that is parallel to the direction of the NIR incident radiation, which results in greater absorbance compared to Qs and Fs surfaces (Fujimoto *et al.* 2008). Absorbance is higher for Qs than Fs samples because the former coincides with the longitudinal orientation of the medullary rays that form the tissue radiating between pith and bark (Leblon *et al.* 2013). Generally, an increase in surface roughness decreases the absorbance of NIR radiation (dos Santos *et al.* 2020).

#### 2.6.3. Principal component analysis (PCA)

PCA is a multivariate analysis technique that is often applied to spectroscopy for data reduction and clustering visualization. It decomposes a linear combination of original variables into a few principal components (PCs) (Yu *et al.* 2016). The PCs show the structure of the dataset and provide information on the main spectral components causing differences between samples (Sandak *et al.* 2016). PCA and PCR are common algorithms used for the calibration of spectrometers and the evaluation of unknown measurement spectra (Vogt and Tacke 2002). The calculation of PCs is often accompanied by singular value decomposition (SVD) of the data matrix (Press *et al.* 1992). The k calibration spectra contain n measurements at a time. In addition, the rows of the matrix  $V^T$ build up the preferred orthonormal basis of the vector subspace spanned by the calibration spectra (Vogt and Tacke 2002). This is represented mathematically as

$$M_{(KXn)} = U_{(KXK)} S_{(KXK)} V^{T}_{(KXn)}$$

$$\tag{1}$$

SVD and PCA are common techniques in the analysis of multivariate data (Wall *et al.* 2003) and were utilized in this study. A large dataset is widely used in many disciplines and applications, and methods are often required to drastically reduce the dimensionality of the data in an interpretable way to get information from such datasets. While many techniques have been identified for this purpose, PCA is the oldest and the most widely used (Jolliffe and Cadima 2016).

PCA is based on the principle of reducing the dimensionality of the dataset while preserving as much variability as possible (Jolliffe and Cadima 2016). The variability is preserved by finding new variables that are linear functions of those in the original dataset. The earliest literature on PCA dates from Pearson (1901), but not until computers became available decades later was it computationally feasible to use PCA on large datasets. It can be based on either the covariance matrix or the correlation matrix, and choosing between these analyses has been previously described (Jolliffe and Cadima 2016). In either case, the new variables (the PCs) depend on the dataset rather than being pre-defined basis functions and are therefore adaptive in the broad sense. The main uses of PCA are descriptive rather than inferential.

#### 2.6.4. Partial least squares regression (PLS-R)

PLS-R models have been widely used to extract useful information from spectroscopic data. A PLS-R model relates the spectral information to quantitative information about the measured samples (Swierenga *et al.* 1998). PLS-R has proven to be a popular and effective approach to solving several problems in chemometrics. The algorithms are resistant to overfitting and are easy to implement and tune, and their ability to produce inferences in high-dimensionality conditions makes them ideal for a kernel approach (Bennett and Embrechts 2003).

A PLS-R model is better at finding the relationship between input variables and output variables; a correlation model is then built using this procedure to predict the output variables by utilizing the input variables (Wang *et al.* 2015). Although the accuracy of PLS-R may not be significantly higher than that of PCR, it is considered better because there are fewer PCs in the final calibration (Naes *et al.* 1986). PLS-R is also preferred for its faster algorithms, higher precision, and more harmonious calibration models (Kalivas and Gemperline 2006).

While PLS-R is reliable in linear conditions, most industrial processes have explicit non-linear qualities that can be hard to ignore (Ding *et al.* 2013). Hence, a new method that can be utilized in highly non-linear processes, called the Kernel PLS (KPLS), has been proposed (Rosipal and Trejo 2001). The KPLS algorithm was introduced to speed up calibrations, handle very wide and tall data, and handle multivariate Y according to the nonlinear iterative PLS (NIPALS) standard, as reported by Andersson (2009). KPLS has also been shown to be numerically stable and one of the fastest algorithms for PLS (Alin 2009, Anderson 2009).

## 2.6.5. Validation of chemometrics model

NIR calibration models are often used for the correlation of either raw or pre-processed spectra with one or more physical-chemical properties of a set of samples. As complicated as it sounds, numerous well-developed calibration techniques have proven to work with most NIR applications, and these techniques are usually included in chemometric software packages (Agelet and Hurburgh 2010).

An adequate validation of calibration models is a crucial step to determine the suitability of models to predict new samples; this is the main reason for developing NIR calibrations. There are different

approaches to estimate the suitable number of PCs to be included in the calibration. One of the most popular is cross-validation (CV), a technique used in the assessment of how the results of a statistical analysis will generalize to an independent data set. The goal of CV is to test the model's ability to predict new data that were not included in the calibration to avoid overfitting or selection bias (Cawley and Talbot 2010). It is often used for the basic assessment of calibration performance. A description of this method can be found elsewhere (Seni and Elder 2010).

Another approach used in the validation of chemometric models is called test-set validation, which is considered the ultimate way of testing calibration performance. Here, two-thirds of the samples are regarded as the training set and one-third as the validation set; only the training set is used in the calibration, while the validation set is used for the prediction of new samples based on the PLS-R models tuned with the calibration dataset (Sandak *et al.* 2016). This method (test-set) is regarded as the most conservative validation for testing models on a representative independent test-set, especially when the purpose is to establish calibration models that can predict quantities (Westad and Marini 2015). The quality of the predictive model can be measured in several ways, some of which have been described (Martens and Naes 1991, Schimleck *et al.* 2001).

# 2.7. Objective

This study aims to examine the ability of NIRS to predict specific properties (density, grain angle, annual ring width, and mechanical properties) of Western hemlock and D-fir. The study attempts to develop robust models for within- and between-species variation based on PLS-R to evaluate the predictability based on orientation—that is, quarter-sawn, flat-sawn, and surface roughness (rough and smooth). The results are expected to provide the wood industry with information on using NIRS for swift and accurate analysis of wood properties and on the influence of surface conditions when assessing wood properties.

# **3.0 MATERIALS AND METHODS**

#### **3.1. Sample Preparation**

Kiln-dried lumbers of Western hemlock and coastal D-fir were obtained from Interfor-Acorn Sawmill, Surrey, BC and Western Forest Products Sawmill, Ladysmith, BC, respectively. Sixty quarter-sawn ( $Q_s$ ) and sixty flat-sawn ( $F_s$ ) samples 1220 mm in length and 50 x 100 mm in cross-section were selected for each wood species. These samples were further sawn into dimensions of 25 mm × 90 mm × 410 mm (longitudinal [L]) by a sliding table saw (T75, MARTIN Woodworking Machines Corp., USA) and conditioned at a temperature and relative humidity of 20°C and 65% *H*. A schematic of the cutting pattern of samples is shown in Figure 3.1.



Figure 3.1: Schematic of the cutting pattern of samples.

One of the longitudinal-tangential surfaces of the  $Q_s$  samples and one of the longitudinal-radial surfaces of the  $F_s$  samples were then planed in order to have two types of surface conditions: one rough and the other smooth (Figure 3.2). This was done to examine the effect of surface condition on NIR spectra. NIRS measurements were done on the rough and smooth surfaces of all  $Q_s$  and  $F_s$  samples. Thus, for each type of surface condition, a total of 240 samples were analysed. Two different species and two orientations were considered in this study, resulting in four combinations (two wood species x two orientations), for a total of 240 samples of mixed wood type (60 for each combination).



Figure 3.2: (a) Hemlock rough, (b) D-fir rough, (c) hemlock smooth, and (d) D-fir smooth.

Each NIRS sample, with dimensions of 25 x 90 x 410 mm (thickness, width, and length, respectively) was converted into two strips (samples) of 25 mm x 25 mm x 410 mm (length) for destructive *MOE* and *MOR* measurements (Figure 3.3) using an MTS dynamic material testing machine (model 810, MTS Systems Corp, Minnesota, USA), while 25 mm x 25 mm x 25 mm (length) samples were prepared for density determination (Figure 3.4). A total of 480 samples were prepared.



Figure 3.3: Cutting pattern of samples for *MOE* and *MOR* measurements.



Figure 3.4: Cutting pattern of samples for  $\rho$  measurements.

Table 3.1: Sample ca	ategorization for NIR	analysis, density	, and mechanical	properties.
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Species	Orientation	NIR	$\rho$ , <i>MOE</i> and	SOG and ARW
		Analysis	MOR	
Hemlock	Quarter-sawn (Rough	60	120	60
	& Smooth)			
	Flat-sawn	60	120	60
	(Rough & Smooth)			
Douglas-fir	Quarter-sawn (Rough & Smooth)	60	120	60
	Flat-sawn	60	120	60
	(Rough & Smooth)			
Total		240	480	240

N.B.: Only 120 samples were prepared for each wood species for the NIR analysis because one surface was rough and the other was smooth.

# **3.2. NIRS Measurements**

Wood sample NIR spectra were acquired using an NIR spectrometer system, which consisted of a light source, an optic fiber, a fiber spectrometer (ASD Lab Spec<sup>®</sup> Pro, Analytical Spectral Devices Inc. Boulder, CO, USA), a sample holder, and a computer.

The optic fiber connected to the spectrometer was oriented at 30° above the sample surface at a distance of 130 mm and aligned in parallel with the longitudinal axis of the sample. The samples were illuminated with a tungsten halogen bulb (ASD Pro Lamp) oriented perpendicular to the sample surface. The distance between the sample surface and the bulb was 168 mm, resulting in an NIR spot size of approximately 80 mm (Figure 3.5). For each sample, four spectra were collected with four subsets that consisted of 50 scans (to reduce the noise), which were averaged into a single spectrum. The averaged spectra for the samples were used to predict the wood properties.



1.Light source 2.Optic fiber 3.Spectrometer 4.Computer 5.Specimen holder. 6.Specimen



All samples used in this study were scanned in the full wavelength range of 350–2500 nm at intervals of 1 nm to cover the visible spectroscopy (Vis) and NIR spectroscopy ranges. The effects of reduced spectral ranges (900–1900 nm, 1100–2500 nm, 1300–2300 nm) on spectral information were also assessed. A piece of commercial microporous Teflon was chosen as the reference material, and a background reference scan was taken every 30 minutes to correct for potential drift over time.
The reference spectra were measured and stored before the test sample spectra collection. Two readings were taken (top and bottom face) at quarter points per sample to give four spectra (two rough and two smooth spectra per sample). The two spectra per surface were averaged to give a single spectrum, and a total of 960 spectra (4 x 240 samples) were acquired for the experiment. All NIRS measurements were taken in a lab location where the temperature ranged between 20 -  $25^{0}$ C and relative humidity between 40 - 55%. Effort was also made for all NIRS measurements to be completed in a short time span to avoid *M* fluctuations of the samples. The set-up of the NIR system used for spectral acquisition is shown in Figure 3.6.



Figure 3.6: The NIR system setup used for spectral acquisition (1. light source, 2. optic fiber, 3. spectrometer, 4. specimen).

# **3.3.** Determination of Wood Density $(\rho)$

The wood density ( $\rho_M$ ) is defined as the ratio between the mass and the volume of the sample at a given *M* (Williamson and Wiemann 2010). The samples used for  $\rho$  were conditioned to a target equilibrium moisture content ( $M_{emc}$ ) of 13.7% and 13.3% (for hemlock and D-fir) in a walk-in chamber (Constant Temperature Control Limited, Aurora, ON, Canada) at a temperature and relative humidity of 20°C and 65% *H*, respectively. The difference in the  $M_{emc}$  of the species

could be attributed to the wood chemistry and specimen moisture history which thus affects the sorption properties of wood (Glass *et al.* 2014). The initial volumes and weight of samples were measured using a digital caliper and digital balance, respectively.

The  $\rho$  of the samples was calculated by

$$\rho_M = W_M / V_M \tag{2}$$

where,  $\rho_M$  (kg/m<sup>3</sup>) is the density, *W*(kg) is the sample weight, and *V*(m<sup>3</sup>) is the volume of the sample at the respective *M* for the species.

#### 3.4. Determination of *MOE* and *MOR*

The mechanical tests were done via a 3-point static bending machine loaded with a 10 kN load cell, a span of 360 mm, and loading rate of 1.3 mm/min according to ASTM D143-94. The samples were conditioned at a temperature of 20°C and 65% H in a walk-in chamber (Constant Temperature Control Limited, Aurora, Ontario, Canada), and *MOE* and *MOR* were determined using an MTS dynamic material testing machine (model 810, MTS Systems Corp, Minnesota, USA) (Figure 3.7). A total of 480 strips were used for the mechanical tests, and spectral information for the mechanical properties of wood was also collected on the samples for NIR analysis.



Figure 3.7: Material Test System (model 810) for measuring *MOE* and *MOR*.

The MOE and MOR of the samples were calculated by

$$MOR (N/mm^2) = \frac{3PL}{2bh^2}$$
(3)

$$MOE (N/mm^2) = \frac{PL^3}{4\Delta bh^3}$$
(4)

where P(N) is the maximum load (*MOR*), L (mm) is the span length, P(N) is the applied load at elastic limit (*MOE*),  $\Delta$  (mm) is the deflection at elastic limit, b (mm) is the sample width, and h (mm) is the sample depth or thickness.

# 3.5. Annual Ring Width Measurements

*ARW* is important in the manufacturing of wood products because of its correlation to the strength properties of wood (Alteyrac *et al.* 2006). The samples used for the NIR test were conditioned at 20°C and 65% *H* for the *ARW* measurements, and the visual inspection of the *ARW* were carried out on them.

The rings were counted over a length of 90 mm and reported as number of rings per distance (mm). A total of 240 samples were assessed, and all measurements were done visually with a ruler. The *ARW* in mm was calculated by

$$ARW(mm) = \sum_{i=1}^{n} \frac{Wi}{n}$$
(5)

where Wi (mm) is the  $i^{th}$  annual ring width and n is the number of rings on the Qs and Fs samples used for the NIRS test.

# 3.6. Slope of Grain Measurements

The *SOG* is defined as the angle between the wood grain (i.e., the direction of the wood fibres) and the main axis of lumber (Gindl and Teischinger 2002). The *SOG* was measured with a shopbuilt scratch gauge with a pivoting angle for inscribing a light groove parallel to the grain (Figure 3.8 left), and a MITE-R-GAGE adjustable Lexan protractor (Figure 3.8 right) was used to measure the *SOG* with respect to specimen edge, measurable to 0.5°. *SOG* measurement via this method is called apparent or surface rather than machine-measured internal *SOG*.

The samples used for the NIR test also had their *SOG* measured only on the smooth surface of the samples rather than the rough surface to increase the accuracy of the *SOG* measurements. For each specimen, measurements were carried out at three points: the front quarter end, the middle point and the rear quarter end. The groove made had a minimum length of 300 mm so that the average *SOG* could be measured. A total of 240 samples were tested.



**Figure 3.8**: Measuring the *SOG* of wood (left: shop-built scratch gauge; and right: MITE-R-GAGE adjustable Lexan protractor).

# 3.7. Multivariate Data Analysis

Once all spectra were collected, multivariate calibrations were developed for each wood property by PLS-R, and the analysis was performed using Unscrambler® version 10.5.1 (CAMO, Norway) software to find possible correlations between the X components (spectra) and the Y components (response variables). Also, the NIR data were mean-centered and normalised before the PLS-R analysis. As a rule, in multivariate modelling, the first stage is to subtract the average from each variable, which is referred to as mean centering. This ensures that all results will be interpretable in terms of variation around the mean.

The spectral data were randomly split into calibration and prediction sets consisting of 36 and 24 samples in each combination of species and surface conditions. Samples of each combination were then divided into two data sets according to the sample set partitioning, based on the joint x-y distance (SPXY) algorithm (Galvão *et al.* 2005). Three-fifths (36 samples in each combination) was used as the calibration set and the remaining two-fifths (24 samples) as the prediction set. Thereafter, the samples in the calibration and prediction sets of each combination were merged into the final calibration (144 samples) and prediction (96 samples) sets, respectively. Sample calibrations were carried out using a mixed set with 960 datapoints (4 datapoints x 240 samples), which included both types of cross-sections, and another set with 480 datapoints (4 datapoints x

120 samples), which consisted of samples from Qs and Fs sections, separately. The methods used for the pre-treatments of spectra before model development included Savitzky-Golay (SG) smoothing, standard normal variate (SNV), and first- and second-order derivatives. NIR reflectance data are usually transformed into log of inverse reflectance (log 1/R) (absorbance); this is often done to ensure good linear modelling.

The development of the models was based on the spectrum of wavelengths 800–2500 nm (full NIR range). Reduced wavelength regions were also used to build models to check the influence of defined spectral ranges on model quality. Reducing the range allowed for the potential use of lightweight, portable, and inexpensive spectrometers for predicting wood properties. The best models were selected based on the coefficient of determination of calibration ( $R_c^2$ ) and the coefficient of determination of prediction ( $R_p^2$ ), which reflect the ability of the model to predict new samples, and RPDp, which uses the standard deviation of the reference data used in the prediction and the standard error of prediction (SEP).

Statistical analysis provided correlations between the wood properties and the spectra for the full and reduced spectral ranges. For each of the wood properties, several data sets were created, including the raw spectral data and the first- and second-derivative data. The regression coefficients plots were also used to check the wavelength contributing most to the models, models were developed based on those variables, and comparisons were made with the full spectrum. Outliers were detected using the influence plot generated by PCA modelling, and they were removed before re-modelling to improve the predictive ability of the final model.

# 4.0 RESULTS AND DISCUSSION

### 4.1 NIRS Calibration

Wood property calibrations were performed using the test-set validation method, regarded as the best way to validate a model. It is based on testing the model on a subset of the available samples that were not included in the computation of the model components (Sandak *et al.* 2016). During the NIR calibration stage, the models were developed based on the training set, and some samples (40%) of the dataset were randomly selected to represent the entire population (Galvao *et al.* 2005). These samples, which are sometimes called the *validation* or *test set*, were then used to determine the ability of the model to predict new samples (Hauksson *et al.* 2001).

Validation of the chemometric model is important for determining modelling and prediction errors, outliers, an optimal number of PLS components, and trends (Tounis 2009). The number of factors or PCs used in the model development was selected by observing the response of the residual Y-variance with additional factors. The number of factors used in the final calibration is crucial to avoid overfitting, a phenomenon where too many factors are included and a fraction of noise is modelled (Agelet and Hurburgh 2010), and underfitting, when too few factors are included with higher bias and lower variance (Gowen *et al.* 2011). The optimal number of PCs used in this study was suggested by the Unscrambler® software.

The best calibration model was selected using high  $R^2$ , reduced error, and rank (number of PCs). All PLS-R models used in this study were based on the KPLS algorithm. The KPLS is a samplecentric approach where the relationship between every sample is characterized by a kernel function that maps the data into higher dimensional space, where the linear regression is performed (Mora and Schimleck 2010). A full description of its derivation can be found elsewhere (Rosipal and Trejo 2001).

The pre-treatment of spectra is also an important step in NIRS calibration, done to improve the model quality. SG smoothing using a 25-point filter width and a second-order polynomial was used during model development (Savitzky and Golay 1964, Gaspar *et al.* 2009). A common pre-treatment known as the derivatives—such as first derivative, which removes any offset difference, and the second derivative, which removes any slope effect in the data—were also applied to

improve spectra quality (Rinnan 2014). The derivatives used in this study were calculated by applying the SG algorithm using 17 smoothing points and a second-order polynomial (Schwanninger *et al.* 2004).

#### **4.2 Density and Mechanical Properties**

#### 4.2.1 Density based on calibrations for all samples

The  $\rho$  ranged from 377–725 kg/m<sup>3</sup> and 418–794 kg/m<sup>3</sup>, with averages of 491.97 kg/m<sup>3</sup> and 552.89 kg/m<sup>3</sup> for hemlock and D-fir, respectively. Others have found similar results for hemlock (Gonzalez 1990, Nourian 2018) and D-fir (Jozsa and Kellogg 1986, Gonzalez 1990). As expected, higher  $\rho$  values were observed for D-fir than for hemlock (Softwood Species Profile n.d). The variations in the  $\rho$  of the wood could be linked to the differences in the amounts of cell wall and extraneous materials present per unit of volume (Henze 2006). For  $\rho$  modelling, 144 and 96 samples of both species were used for the calibration and prediction sets. The descriptive statistics for this property from the calibration and prediction sets are shown in Table 4.1.

	Calibration Set $(n = 144)$				Prediction Set $(n = 96)$					
Property	Mean	Min	Max	SD	CV	Mean	Min	Max	SD	CV
$\rho$ (kg/m <sup>3</sup> )	507	377	725	60	11.83	546	389	794	72.21	13.23
MOE (MPa)	5608	1587	13247	2095	37.36	6199	1601	11792	2270	36.63
MOR (MPa)	63	18	110	18	28.57	70	33	114	17.33	24.76
SOG (°)	8.9	1.5	16.7	3.2	35.96	9.2	1.3	16	3.4	36.96
ARW (mm)	1.8	0.1	12.6	1.8	100	1.7	0.2	13.1	1.6	94.12

Table 4.1: Descriptive statistics of the wood properties from the calibration and prediction sets.

Min is the minimum value, max is the maximum value, SD is the standard deviation, and CV is the coefficient of variation.

All  $\rho$  models gave moderate predictions, with the  $R_c^2$  ranging from 0.46–0.60 and the  $R_p^2$  ranging from 0.51–0.63 (Table 4.2). The effect of pre-treatment on model quality was observed, especially when SG smoothing with a 25-point filter and a second-order polynomial were used (Savitzky and Golay 1964). The  $R_p^2$  improved when SG smoothing was applied to the spectral data (Table 4.2), which is in line with others' work (Gaspar *et al.* 2009) where pre-treatments improved the model performance. A typical graphical representation of a raw spectrum is shown in Figure 4.1.



Figure 4.1: A typical graphical representation of a raw spectrum.

The  $\rho$  models in this study were considered robust with few factors (no more than five). This is interesting because past authors (Sandak *et al.* 2016, Schimleck *et al.* 2018) reported that fewer factors in a PLS-R model is a better strategy for avoiding data overfitting. Although standard normal variate (SNV) as well as first and second derivatives were applied to the raw data, no improvement in the predictive ability of the model was shown. This was not surprising, as the best correlations were previously obtained from raw spectra (Andrade *et al.* 2010). Overall, results based on rough surfaces were better than those from smooth surfaces. This could be explained by the fact that EMR on a rough surface captures more information than on a smooth surface during scanning, which contributes to the predictions (Costa *et al.* 2018).

The PCA was used to distinguish between the grain orientations of the two softwoods (hemlock and D-fir) and to reveal any unusual variation in the PCA model. The PCA scores plots (Figure 4.2) show the four classes of samples earlier described with the first two components, PC1 and PC2 (the highest model contributors), which explained 96% of the variation in the raw data. It can be seen that some samples are clustered together, meaning they are similar and highly correlated with respect to the PCs, while those far apart from one another are negatively correlated (Figure 4.2).



**Figure 4.2**: PCA plot for raw spectra obtained from a rough surface (top) and smooth surface (bottom), where DF: Douglas-fir flat-sawn, DQ: Douglas-fir quarter-sawn, HF: Hemlock flat-sawn, HQ: Hemlock quarter-sawn.

For instance, PC1 describes more of the variation in the Fs samples, while PC2 shows the variation in the Qs samples for the rough surface (Figure 4.2, top). In the same way, for the smooth surface, both PC1 and PC2 fully describe the variation in grain orientations for the two species (Figure 4.2, bottom). A few samples are left out of the eclipse and are considered to have contributed little to the PCA model (Figure 4.2).

This showed that NIRS coupled with PCA analysis is a useful tool for identifying and differentiating between the *Qs* and *Fs* samples of the two softwoods, which agrees with previous findings (Everard *et al.* 2012). The plots showing the relationship between NIR predicted and measured values for the best models are shown in Figure 4.3 for both surfaces. The closeness of the data points to the target line (black line) is often used to determine the model's goodness of fit. These plots show the strength of correlation between the calibration data (blue dots) and validation data (red dots) for the property measured.

The calibrations in this study also involved selecting the optimal spectral range for the prediction of  $\rho$ . This is considered important to remove the spectral regions that make little contribution to the predictive ability of the models. It has been reported that not all spectral regions contribute to the predictive ability of models, hence the need for a narrow spectral range to optimize the model (Leardi *et al.* 2002, Mehmood *et al.* 2012). Although spectral ranges of 1100–2500 nm and 1300–2300 nm were used to develop calibrations, no model performance improvement was observed.

The  $R_p^2$  of  $\rho$  for the rough (0.57–0.63) and the smooth (0.51) surfaces were obtained. Although these values are lower than what has been reported for the  $\rho$  of *Pinus taeda* ( $R_p^2$  of 0.69; Schimleck *et al.* 2002), they are higher than the  $\rho$  predictions found in another study (Jones *et al.* 2007) for the same species. In this study, the rough surface predictions were considered best for  $\rho$  due to the high  $R_p^2$  values and reduced prediction errors. This meant that better predictions could be obtained for the  $\rho$  of hemlock and D-fir by taking the NIRS readings on the rough surface of lumber. The summary of the calibration statistics for  $\rho$  of the softwoods is shown in Table 4.2.

The difference in the calibration statistics for these studies could be attributed to the range of  $\rho$  observed and the species used. It has also been shown that mixed species calibrations may not be as accurate as those based on single species, such as in the aforementioned study (Schimleck *et al.* 

Property	Roughness	Spectral Range	$R_c^2$	$R_p^2$	SEC	SEP	RPD
ρ (All)	Rough	Raw Data (Full Range)	0.46	0.57	44.42	48.97	1.5
<u>``</u>		Smoothed data	0.44	0.63	44.95	45.74	1.6
	Smooth	Full range	0.60	0.51	38.55	54.79	1.3
ρ	David	Raw Data (Full Range)	0.52	0.53	45.58	40.59	1.5
(Qs)	Kougn	Smoothed (1100–2500)	0.52	0.57	45.49	41.61	1.4
	Smooth	Raw Data (Full Range)	0.64	0.56	39.36	43.70	1.4
		Raw Data (1100–2500)	0.74	0.63	33.56	40.23	1.5
$\rho$ (Es)	Rough	Raw Data (Full Range)	0.32	0.63	42.53	52.67	1.1
(13)		Raw Data (1100–2500)	0.60	0.66	32.62	52.28	1.1
	Smooth	Raw Data (Full Range)	0.30	0.59	43.23	54.12	1.5
	~~~~~	Raw Data (1300–2300)	0.47	0.59	37.76	54.14	1.5

**Table 4.2**: Calibration statistics for  $\rho$  based on all samples (n = 240) and *Qs* and *Fs* samples (n = 120).

2010). Likewise, the poor performance of the  $\rho$  models could be attributed to the variation in  $\rho$  for different sections of wood along the stem, as compared to mechanical properties (Acquah *et al.* 2018). Again, the generally poor predictive statistics of  $\rho$  could be ascribed to the diverse genetic makeup of the samples in the prediction set. Previous researchers have also revealed that more precision and accuracy are needed in testing procedures (spectral acquisition and reference methods) to improve the calibration statistics from NIRS (Hein 2010).

A direct comparison of the prediction regression coefficients generated from the PLS-R model was also performed. Comparisons were made between the regression coefficients of  $\rho$  predicted for both surface conditions (rough and smooth). Some of the absorption bands related to the major wood components (cellulose, hemicelluloses, and lignin) were found to have contributed to  $\rho$  predictions, which is in line with the work of other researchers (Fujimoto *et al.* 2008). Similarly,

the cellulose- and lignin-associated wavelengths were correlated with  $\rho$ , suggesting that wood chemistry influences  $\rho$  independent of baseline shift (Hein 2010).



**Figure 4.3**: The relationship between measured and NIR-predicted values of  $\rho$  (kg/m<sup>3</sup>), based on rough (top) and smooth (bottom) surfaces for two softwoods (hemlock and D-fir). The blue and red dots indicate the calibration and prediction sets, respectively.

The results of the regression coefficients showed that similar wavelengths contributed to the  $\rho$  prediction of both surfaces. The important wavelengths found to have contributed to  $\rho$  included:

1480, 1484–1493, 1534, 1550, 1540, 1579, 1580–1597, 1793, 2110 (due to crystalline and semicrystalline cellulose; Schwanninger *et al.* 2011), 1471, and 1724 (due to the presence of hemicelluloses; Fujimoto *et al.* 2007, Fackler and Schwanninger 2010), and 1447, 1448, and 1672– 1674 (due to the presence of lignin; Mitsui *et al.* 2008). The wavelengths contributing to the predictions were not surprising, as past authors have also found the spectral range of 1100–1700 nm to contain the most important information for predicting wood properties (Schimleck *et al.* 2003, Todorovic *et al.* 2015). The specific assignments of NIR spectral features to different wood components have been reported by several authors (Bokobza 2002, Tsuchikawa and Siesler 2003).

### 4.2.2. Density based on calibrations for the Qs and Fs samples

For the modelling of  $\rho$ , 72 and 48 samples of both species were used for the calibration and prediction sets, respectively. Most of the models for  $\rho$  gave moderate predictions, with  $R_c^2$  ranging from 0.52–0.74 and 0.32–0.60 for the *Qs* and *Fs* sections, respectively. The  $R_p^2$  ranged from 0.53–0.63 and 0.59–0.66 for the former and latter, respectively. A comprehensive summary of the model statistics is provided in Table 4.2.

For the rough surface, the *Qs* section yielded better predictions ( $R_c^2$  of 0.52 and  $R_p^2$  of 0.53) compared to the *Fs* section (Table 4.2). The rough surface prediction was considered best due to the SEP (40.6 kg/m<sup>3</sup> for *Qs* section and 52.3 kg/m<sup>3</sup> for *Fs* section) and the RPD (1.5 for *Qs* section and 1.1 for *Fs* section). Several pre-treatments were applied, but no significant improvement was found in the model performance. The optimal spectral range for  $\rho$  prediction was also investigated in this study. The spectral range of 1100–2500 nm was used to develop the  $\rho$  model, and slight improvement was observed in the predictions for the grain orientations (Table 4.2). While the  $\rho$  prediction values are considered reasonable, better results have been reported by other researchers (Schimleck *et al.* 2002). A likely reason for this discrepancy may be related to the single species used for calibrations in their study. This is because single-species calibrations give better results than mixed-species calibrations such as those used in this study (Alves *et al.* 2012).

The smooth surface predictions were also similar to those from the rough surface (*Qs* section reported better statistics). The  $R_c^2$  ranged from 0.64–0.74 and 0.30–0.47 for the *Qs* and *Fs* sections, respectively. In the same way, the  $R_p^2$  ranged from 0.56–0.63 for the *Qs* section and was 0.59 for the *Fs* section. A similar spectral range (1100–2500 nm) also improved the calibration for the grain

orientations based on the smooth surface (Table 4.2). Overall, calibrations based on the Qs section were better than those from the Fs section, which is also in line with past work (Gindl *et al.* 2001).



**Figure 4.4**: PCA plot for the best model based on the rough surface and quarter-sawn section (top) and the smooth surface and quarter-sawn section (bottom).

The difference in the spectral information of the surfaces, especially for  $\rho$ , has been previously explained (Fujimoto *et al.* 2008) and was attributed to the interaction between the chemical and

anatomical properties of wood. Another reason could be the higher exposure of parenchyma cells in the *Qs* section compared to the *Fs* section, which may have affected calibrations (Hein *et al.* 2009a). The best prediction for  $\rho$  was observed when the rough surface was utilized. The highest prediction value from the  $\rho$  calibrations was found by utilizing the *Fs* section, a finding similar to a past report (Ribeiro 2009). The potential for predicting the  $\rho$  of wood has been revealed in past work (Mora *et al.* 2008), where strong correlations ( $R_p^2 = 0.80-0.84$ ) were reported between the measured values and NIR-predicted values for softwood.

A PCA was also used to distinguish between the *Qs* and *Fs* sections of the two softwoods. The PCA scores plots of the best model explained 98% of the variation in the data for both rough and smooth surfaces (Figure 4.4). For the rough surface, PC1 showed more variability in the *Qs* samples, which were clustered together (93% variation explained) than did PC2, where the samples were far apart and explained only 5% of the variation in the data (Figure 4.4, top). In the same way, the PCA obtained from the smooth surface explained more variation (97% for PC1, 1% for PC2) in the model with samples found to be highly correlated to one another (Figure 4.4, bottom). Even though some of the samples overlap (falling between PC1 and PC2), the results of the PCA model shows that 93% of the data collected from the *Qs* section of both species could be separated with certainty, especially when they fell outside the overlapping zone (Figure 4.4).

The plots showing the relationship between NIR predicted and measured values for the best models selected are shown (Figure 4.5) for both surface conditions. From the figures, it can be seen that the data points are not close to the target line, and the calibration and validation data are not tightly packed together. This shows that the predictions obtained for the  $\rho$  of wood were only moderate. The regression coefficients of the grain orientations for the rough surface were similar, suggesting that the same wavelengths contributed to  $\rho$  prediction. Similar wavelengths described earlier in this thesis contributed to the  $\rho$  prediction of the smooth surface. The assignment of bands that contributed to  $\rho$  prediction has been described earlier in this thesis (Section 4.2.1).



**Figure 4.5**: The relationship between measured and NIR-predicted values of  $\rho$  (kg/m<sup>3</sup>), based on the rough (top) and smooth (bottom) surfaces (quarter-sawn section) for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

# 4.3 Modulus of Elasticity

# 4.3.1 MOE based on calibrations for all samples

The *MOE* values ranged from 1587–6182 MPa and 5532–13247 MPa for hemlock and D-fir, respectively. Similar values have been reported for the *MOE* of D-fir (FPL 2010, Mousavi 2016),

although higher values were found for hemlock (Rohrbach 2008, FPL 2010). The reason for this could be the lower density values (Johnson and Gartner 2006) and the possible effect of knots observed in a few samples, which might have caused decreases in the *MOE* values (Koman *et al.* 2013). Higher *MOE* values were observed for D-fir than for hemlock, as expected. This could be explained by the reduction in its M (13.3) as compared to hemlock (13.7), as previously reported in this thesis (Section 3.3). Past researchers have also revealed that an increase in the *MOE* of wood could be linked to the decrease in its M below the fibre saturation point (FSP) (Skaar 1988, Güntekin and Aydin 2013). For the modelling of *MOE*, 144 and 96 samples were used for the calibration and prediction sets, respectively. The descriptive statistics for this property from the calibration and prediction sets are shown in Table 4.1.

The raw spectra were used to develop calibrations for *MOE*, and moderate predictions were obtained. An  $R_c^2$  of 0.69 and an  $R_p^2$  of 0.57 were reported for the rough surface, whereas lower values with high prediction errors were observed for the smooth surface (Table 4.3), while a reduced spectral range (1100–2300 nm) gave the best prediction for the rough surface with six factors, which makes the model less robust. The best model ( $R_p^2$  of 0.67) with reduced prediction error was observed when the spectral range was 1300–2300 nm for the smooth surface. These values are considered reasonable due to the robustness of the model and the fact that the model was validated using separate samples.

Pre-treatments improved the model quality for the rough surface, whereas no improvement was observed for the smooth surface. The first derivative slightly improved the prediction value (from 0.57 to 0.60) and reduced the prediction error (Table 4.3). Improvement in the model quality with reduced spectral range was not unexpected, especially in the spectral range of 1000–2500 nm. It has been established that this range contains the most distinct spectral information on the first overtone and combination bands that make up the NIR spectra (Kelley *et al.* 2004b). Reducing the spectral range for calibrations allows for the use of lightweight, portable, and inexpensive spectrometers for field applications (Hedrick *et al.* 2004). The calibration statistics are summarized in Table 4.3.

Most studies using independent samples rather than cross-validations to validate prediction models generally indicate that cross-validations are overly optimistic (Brown *et al.* 2005, Brunet *et al.* 

2007). However, the PLS-R models developed for the *MOE* of agro-based particleboards by NIRS have yielded  $R_p^2$  values ranging from 0.42 to 0.62 (Hein *et al.* 2011), which are lower than the results in this study. A ratio of performance to deviation (RPD) of between 1.3 and 1.6 was reported, making it suitable for initial screening. NIR spectra, especially from complex materials like wood and panel products, are driven not only by the chemistry of the material but also by the solid structure (Gierlinger *et al.* 2004).

Property	Roughness	Spectral Range	$R_c^2$	$R_p^2$	SEC	SEP	RPD
		Raw Data (Full Range)	0.69	0.57	1166	1538	1.5
MOE (All)	Rough	1 <sup>st</sup> Derivative	0.74	0.60	1070	1486	1.5
		Smoothed (1100-2300)	0.64	0.71	1250	1263	1.8
		Raw Data (Full Range)	0.58	0.41	1364	1779	1.3
	Smooth	Reduced (1300–2300)	0.66	0.67	1229	1358	1.7
		Reduced (1100-2500)	0.75	0.64	1049	1416	1.6
MOE (Qs)	Rough	Raw Data (Full Range)	0.77	0.78	1149	955	2.1
	Smooth	Raw Data (Full Range)	0.85	0.68	907	1160	1.7
		Raw Data (900–1900)	0.84	0.72	963	1087	1.8
MOE (Es)	Rough	Raw Data (Full Range)	0.67	0.57	991	1719	1.5
(1.3)		Raw Data (1000–2500)	0.72	0.63	917	1590	1.6
	Smooth	Raw Data (Full Range)	0.75	0.69	856	1453	1.7

**Table 4.3**: Calibration statistics for *MOE* based on all samples (n = 240) and *Os* and *Fs* samples (n = 120).

The RPD values were used to evaluate the model performance in this study. All RPD values for *MOE* met the 1.5 criteria, except for those based on raw spectra for the smooth surface, where the RPD was 1.3 (Table 4.3). It has been shown that NIRS can be used as an initial screening tool with an RPD of approximately 1.5 (Schimleck *et al.* 2003, Acquah *et al.* 2018). It has also been suggested that an RPD between 1.5 and 2.5 is enough for estimating wood properties (Hein 2010).



**Figure 4.6**: The relationship between measured and NIR-predicted values for *MOE* (MPa), based on the rough surface (top) and smooth surface, 1300–2300 nm (bottom) for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

The regression coefficients showing the important variables for these models were observed. The important wavelengths identified for *MOE* prediction are 1212–1225, 1480, 1477–1484, 1484–1493, and 1586–1596, which have been ascribed to cellulose, while 1471 is related to hemicelluloses and 2200 to lignin. For small clear specimens, high correlations for the *MOE* and *MOR* were found at the absorption bands due to -OH and -CH in the semi-crystalline or crystalline regions in cellulose. These results explain that an increase in the semi-crystalline or crystalline regions of cellulose is related to an increase in the *MOE* and *MOR* (Fujimoto *et al.* 2007). The plots showing the relationship between NIR predicted and measured values for the best models are shown in Figure 4.6 for both surface conditions.

The results obtained in this study using NIRS to predict the *MOE* of wood were better than those reported by Acquah *et al.* (2018), with cross-validated models for predicting this property for new samples. The  $R_p^2$  reported was 0.45 for *MOE*, even though the samples were taken from many trees (450). The errors reported in their study were also higher than what were obtained in this study. Higher errors are usually associated with predicting an independent test set from calibrated models, because how worse a model will perform is considered when applied to the test set not originally used in model training. Overall, the results based on the rough surface were better than those based on the smooth surface (Table 4.3).

#### 4.3.2 MOE based on calibrations for Qs and Fs samples

For the modelling of *MOE*, 72 and 48 samples of both species were used for the calibration and prediction sets, respectively. Most of the models for *MOE* gave strong correlations of  $R_c^2$ , ranging from 0.77–0.85 and 0.67–0.75 for the *Qs* and *Fs* sections, respectively. The  $R_p^2$  ranged from 0.68–0.78 and 0.57–0.69 for the two sections, respectively. These results are in line with the *MOE* calibration statistics previously reported for spectra collected from similar grain orientations (Thumm and Meder 2001, Andrade *et al.* 2010, Kothiyal and Raturi 2011).

Several pre-treatments were applied to improve the model quality but showed no effect. Thus, all models for *MOE* were based on raw spectra. For the rough surface, the results based on the *Qs* section yielded better calibration statistics than the *Fs* section (Table 4.3). A good  $R_c^2$  of 0.77 and an  $R_p^2$  of 0.78 were reported for the *Qs* section, while an  $R_c^2$  of 0.67 and an  $R_p^2$  of 0.57 were reported for the *Fs* section. These results show that the former provides better predictions for *MOE* than the

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latter, which is similar to past reports (Thumm and Meder 2001, Zhao *et al.* 2009). The *Qs* section showed the potential for predicting new samples better (RPD > 2.0). As reported previously for agricultural materials, an RPD between 2.0 and 2.5 indicates very good prediction (Mouazen *et al.* 2005). Reducing the spectral range (from 1000 to 2500 nm) slightly improved the predictions, but only for the *Fs* section ( $R_p^2$  from 0.57 to 0.63; see Table 4.3).

Similar predictions to the rough surface were also observed for the smooth surface, where the Qs section reported better statistics than the Fs section. Reasonable predictions were observed for the Qs ( $R_c^2 = 0.85$ ,  $R_p^2 = 0.68$ ) and Fs ( $R_c^2 = 0.75$ ,  $R_p^2 = 0.69$ ) sections. The same trend was observed for the prediction error of the Qs (1160 MPa) and Fs (1453 MPa) sections, respectively. It was evident that better predictions were obtained from the Qs section. Calibrations based on the Qs and Fs sections previously reported (Kothiyal *et al.* 2014) showed that the former had the stronger calibrations. The difference in the accuracy of prediction could be linked to the surface condition of the sample, since earlywood and latewood are better represented in the Qs section than the Fs section. The resulting spectrum from the former usually has a better representation of the total wood characteristics (Thumm and Meder 2001) than the latter, in which calibrations depend on whether earlywood or latewood was present when spectra were collected (Dahlen *et al.* 2017).

The narrow spectral range (900–1900 nm) created for the *Qs* section improved the model ( $R_p^2$  increased from 0.68 to 0.72), and the SEP dropped from 1160 to 1087 MPa. Spectral ranges of 900–1900 nm and 1000–2500 nm also improved the model quality. Reducing the spectral range is deemed important because it is less expensive than full spectral analysis (Yu *et al.* 2009). The aforementioned spectral ranges are considered effective for predicting the *MOE* of wood. Overall, the *Qs* section reported better statistics than the *Fs* section for rough and smooth surface conditions, as shown in Table 4.3. The highest predictive value for *MOE* was obtained from the *Qs* section ( $R_p^2 = 0.78$ ) based on the rough surface, which agrees with past work when calibrations were developed using the same section (Fujimoto *et al.* 2008).

The models for *MOE* in this study were constructed with no more than five factors. This is in contrast with previous claims (Fujimoto *et al.* 2008) that six to eight factors are needed to build good models that predict mechanical properties for small clear samples. The calibrations presented in this study demonstrate that NIRS has the potential to predict the mechanical properties of wood

samples with considerable accuracy. NIRS has also been used to predict the mechanical properties of wood-based materials such as medium-density fibreboard, with an  $R_p^2$  of 0.80–0.82 (Rials *et al.* 2002).



**Figure 4.7**: The relationship between measured and NIR-predicted values for *MOE* (MPa), based on the quarter-sawn (top) and flat-sawn (bottom) sections for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

The PCA was used to distinguish between the grain orientations of the two softwoods and show variations in the data. The PCA scores plots of the best model selected explain 98% and 96% variation in the data for the rough surface, and 99% and 96% explained for the smooth surface. The plots showing the relationship between NIR predicted and measured values for the best models selected for the *Qs* and *Fs* sections are shown in Figure 4.7. The wavelengths contributing to the *MOE* prediction of the former and latter are similar to those reported earlier in this thesis (Section 4.3.1). The assignment of NIR peaks has been reported by several authors (Via *et al.* 2003, Tsuchikawa and Siesler 2003).

#### 4.4 Modulus of Rupture

#### 4.4.1 MOR based on calibrations for all samples

The *MOR* assessed in this study ranged from 18–83 MPa and 54–114 MPa for hemlock and D-fir, respectively. Similar values have been reported for the *MOR* of hemlock (Rohrbach 2008) and D-fir (FPL 2010, Mousavi 2016). Higher *MOR* values were observed for D-fir than for hemlock, which is similar to past reports (FPL 2010). This could be attributed to the difference between the *M* and  $\rho$  of the species. Past reports have revealed that a lower *M* and a higher  $\rho$ , as observed in D-fir, is accompanied by an increase in the mechanical properties (Tsoumis 2020). This could have contributed to the higher *MOR* values observed in D-fir. For the modelling of *MOR*, the same number earlier described (Table 4.1) were used for the calibration and prediction sets.

Several pre-treatments were applied, with no significant improvement observed, so raw spectra were used to build the model for both surface conditions. For the rough surface, moderate predictions ( $R_p^2 = 0.56$ , SEP = 12.24 MPa) were reported, while lower values ( $R_p^2 = 0.42$ , SEP = 13.64 MPa) were reported for the smooth surface. The 1300–2300 nm range based on first-derivative spectra gave a good prediction for the smooth surface, with  $R_c^2$  increasing from 0.55 to 0.74 and  $R_p^2$  from 0.42 to 0.66. The SEC dropped from 12.05 to 9.15, while the SEP went from 13.64 to 10.76 MPa (Table 4.4). Previous works have revealed similar values for the *MOR* of lumber (Schimleck *et al.* 2018). These findings also agree with the work of others, where similarities were found in the prediction of lumber and small clear samples (Fujimoto *et al.* 2008).

Property	Roughness	Spectral Range	$R_c^2$	$R_p^2$	SEC	SEP	RPD
MOR (All)	Rough	Raw Data (Full Range)	0.68	0.56	10.18	12.24	1.4
(Au)		Smoothed	0.68	0.57	10.25	12.10	1.4
		Raw Data (Full Range)	0.55	0.42	12.05	13.64	1.3
	Smooth						
		1 <sup>st</sup> Derivative (1300–2300)	0.74	0.66	9.15	10.76	1.6
MOR (Os)	Rough	Raw Data (Full Range)	0.77	0.52	9.52	11.48	1.4
		Raw Data (Full Range)	0.87	0.57	7.20	10.72	1.5
	Smooth	Raw Data (1100–2500)	0.85	0.65	7.68	9.85	1.6
MOR	Rough	Raw Data (Full Range)	0.77	0.76	7.42	9.87	1.9
( <b>F</b> s)		Smoothed (1300–2300)	0.66	0.82	9.05	8.61	2.2
		Raw Data (Full Range)	0.72	0.67	8.24	11.14	1.7
	Smooth	Raw Data (900–1900)	0.73	0.71	7.97	11.10	1.7

**Table 4.4**: Calibration statistics for *MOR* based on all samples (n = 240) and *Os* and *Fs* samples (n = 120).

However, stronger correlations ( $R_p^2 = 0.81-0.86$ ) were previously reported for small clear samples of *Pinus taeda*, regardless of whether the cross-section or radial surface was rough or sanded (Schimleck *et al.* 2005b). Fujimoto *et al.* (2007) also reported an  $R_p^2$  of 0.84 when the *MOR* of hybrid larch was predicted using NIRS. The lower values that were observed in this study could be attributed to not separating the samples into juvenile and mature wood. This could have contributed to poorer calibrations, as samples containing a wide variety of juvenile and mature wood have been reported to have lower calibration performance (Dahlen *et al.* 2017). Another reason for the lower values could be the range of values of *MOR* investigated in this study, since a wide range of values has been linked to improved model quality (Gindl *et al.* 2001). The calibrations for *MOR* were lower than those for *MOE*, which could be because the  $\rho$  of wood samples strongly affects the *MOR* rather than the *MOE* (Fujimoto *et al.* 2007). The relationship between *MOE* and *MOR* and several properties of wood have been described in detail (Kliger *et al.* 1995). Overall, the smooth surface gave better predictions than the rough surface, a reverse trend to the models built for  $\rho$  and *MOE*.

The important wavelengths identified for *MOR* prediction were 1212–1225, 1480, 1477–1484, 1484–1493, 1586, 2110 (due to cellulose), 1907, 1910, 2134 (due to hemicelluloses), and 2200 (due to lignin; see Figure 4.8). Significant correlations for the *MOR* were found at the absorption bands due to the -CH in hemicelluloses, meaning that increase in hemicelluloses is related to an increase in the *MOR* of wood (Fujimoto *et al.* 2007). Some wavelengths show negative correlations at bands in the 1868–1981 nm range, which are associated with water and known to play an important role in the NIR spectrum of wood (Thygesen and Lundqvist 2000, Bokobza 2002). All band assignments for the spectra of wood can be found elsewhere (Schwanninger *et al.* 2011).

Plots showing the relationship between NIR predicted and measured values for the best models for both surfaces are shown in Figure 4.9. NIRS coupled with PLS-R was found to be a suitable technique for predicting the  $\rho$ , *MOE*, and *MOR* of wood. Overall, the best prediction was obtained for *MOE*, followed by *MOR* and then  $\rho$ , which is in accordance with what was reported in previous studies (Haartveit and Flæte 2006).



1300 1330 1360 1390 1420 1450 1481 1511 1541 1571 1601 1632 1662 1692 1722 1752 1783 1813 1843 1873 1904 1934 1964 1994 2024 2055 2085 2115 2145 2175 2206 2236 2266 . X-Variables (MOR, Factor-5, B0:8.30663)

**Figure 4.8**: Regression coefficients based on raw data for the rough surface (top) and the smooth surface (bottom).



**Figure 4.9**: The relationship between measured and NIR-predicted values for *MOR* (MPa), based on the rough surface (top) and smooth surface (bottom) for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

# 4.4.2 MOR based on calibrations for Qs and Fs samples

For the modelling of *MOR*, 72 and 48 samples of both species were used for the calibration and prediction sets, respectively. Most of the models for *MOR* gave strong correlations, with  $R_c^2$ 

ranging from 0.77–0.87 and 0.66–0.77 for the *Qs* and *Fs* sections, respectively. The  $R_p^2$  ranges were from 0.52–0.65 and 0.67–0.82 for the former and latter, respectively. Similar reports for *MOR* calibrations have been found in the literature (Fujimoto *et al.* 2007). A summary of the calibration statistics is presented in Table 4.4.

Pre-treatments were applied to improve the model quality, but raw spectra gave the best predictions for the rough surface. The models based on the *Fs* section reported better performance than the *Qs* section (Table 4.4). A good  $R_c^2$  of 0.77 and a moderate prediction value ( $R_p^2 = 0.52$ ) were obtained for the *Qs* section, while the *Fs* section yielded an  $R_c^2$  of 0.77 and a higher value of  $R_p^2$  (0.76). The results obtained for the *Qs* section are considered moderate, as very low prediction value ( $R_p^2 =$ 0.44 and RPD = 1.1) have been reported (Andrade *et al.* 2010). The values obtained for the *Fs* section are similar to those in past reports (Fujimoto *et al.* 2008, Andrade *et al.* 2010). Reducing the spectral range to 1100–2500 nm, 900–1900 nm, and 1300–2300 nm did not affect the model performance for either grain orientation. An exception was found when smoothed data in the range of 1300–2300 nm was used to predict the *MOR* for the *Fs* section, which slightly improved the  $R_p^2$ from 0.76 to 0.82 and reduced the SEP from 9.87 to 8.61.

A similar trend in the results for the rough surface was observed for the smooth surface where the *Fs* section yielded better statistics than the *Qs* section. A good  $R_c^2$  of 0.87 and an  $R_p^2$  of 0.57 for the *Qs* section and an  $R_c^2$  of 0.72 and  $R_p^2$  of 0.67 for the *Fs* section were obtained. Models based on the latter were chosen as the best due to the high  $R_p^2$  and RPD values (Fujimoto *et al.* 2008). Pre-treatments such as smoothing, SNV, first derivative, and second derivative reduced the model quality. Reducing the spectral range to 1000–2500 nm for the *Qs* section slightly improved the predictive ability ( $R_p^2$  from 0.57 to 0.65) and reduced the error (SEP from 10.72 to 9.85; see Table 4.4). For the *Fs* section, the prediction slightly improved in the 900–1900 nm range ( $R_p^2$  increased from 0.67 to 0.71, while the SEP dropped from 11.14 to 11.10). The improvement in the model performance due to the reduced spectral range is useful for predicting wood's mechanical properties (Liang *et al.* 2016).

All RPD values for *MOR* meet the 1.5 criteria except for a model based on the rough surface and *Qs* section, where the RPD was 1.4 (Table 4.4). A considerably higher RPD of 2.2 was observed

for the best model selected for the Fs section, which is in line with the work of Fujimoto *et al.* (2008) for the same grain orientation. It has been established that a model must have an RPD greater than 1.5 to be considered as a preliminary screening tool (Hein *et al.* 2009a). Overall, the Fs section yielded better results than the Qs section for both surface conditions, and the best prediction for *MOR* was observed when the rough surface was utilized.

The PCA was used to distinguish between the grain orientations of the two softwoods and show variations in the data. The PCA scores plots of the best model selected explain 95% and 99% of the variation in the data for the grain orientations based on a rough surface. The PCA for the smooth surface explains 99% and 98% of the variation in the data for both grain orientations. Plots showing the relationship between NIR predicted and measured values for the best models selected for the *Qs* and *Fs* sections are shown in Figure 4.10. The interpretation of PCA and PLS-R plots has been reported earlier in this thesis (Sections 4.2.1 and 4.2.2).

The significant wavelengths contributing to *MOR* prediction for both grain orientations were similar to those reported earlier in this thesis. The bands at 1674 and 1684 nm were found to be negatively correlated to *MOR*. These bands have previously been assigned to extractives and lignin (Michell and Schimleck 1996); the lignin content of wood has been reported to influence NIR absorbance (Schimleck *et al.* 1997). The relationship between mechanical properties such as *MOR* and NIR spectra could be attributed to how lignin content and composition affected NIR absorbance (Gindl *et al.* 2001). The absorption bands related to cellulosic features were found to be the most important contributors in model building for *MOR*, which is in accordance with what others have reported (Fujimoto *et al.* 2008).



**Figure 4.10**: The relationship between measured and NIR-predicted values for *MOR* (MPa), based on the rough surface (flat-sawn) (top) and smooth surface (quarter-sawn) (bottom) for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

# 4.5 Slope of Grain (SOG)

### 4.5.1 SOG based on calibrations for all samples

The *SOG* assessed in this study ranged from  $1.33-16.67^{\circ}$  for hemlock and  $1.67-15^{\circ}$  for D-fir. For the modelling of *SOG*, the same number of samples as described above (see Table 4.1) were used for the calibration and prediction sets.

*SOG* is highly important for load-bearing applications because of its influence on wood's *MOE* and *MOR*. PLS-R analysis coupled with NIR absorbance spectra were used to determine the wood's *SOG*. The NIR spectra were taken from the smooth surface to increase the accuracy of prediction. Raw spectra were used to develop calibrations but gave poor predictions, with an  $R_c^2$  of 0.13 and an  $R_p^2$  of 0.21. An SEC of 2.99 and an SEP of 3.03° were obtained (Table 4.5).

Property	Roughness	Spectral Range	$R_c^2$	$R_p^2$	SEC	SEP	RPD
SOG (All)	Smooth	Raw Data	0.13	0.21	2.99	3.03	1.1
SOG (Qs)	Smooth	Raw Data (Full Range)	0.15	0.21	2.64	2.84	1.1
SOG (Fs)	Smooth	Raw Data (Full Range)	0.38	0.17	2.03	2.16	1.1
		Raw Data (1000–1960)	0.36	0.24	2.06	2.16	1.1

**Table 4.5**: Calibration statistics for *SOG* based on all samples (n = 240) and *Qs* and *Fs* samples (n = 120).

The regression coefficients show the relationship between *SOG* and absorbance spectra. The correlation was variable over the observed wavelength range in this study, with positive and negative values, which is in accordance with past research (Gindl and Teischinger 2002). The PCA plot explains the variation in the data (96%) (Figure 4.11), with PC1 explaining more variability in the samples (93%) than PC2 (3%). A plot showing the relationship between NIR predicted and measured values is provided in Figure 4.12, where it is evident that the correlation between the

calibration and validation data was weak and the model is a poor fit (data points far from the target line).

The pre-treatments earlier described in this thesis were applied to the spectra but yielded no improvement in model quality. Reducing the spectral range based on observation of the regression coefficient also resulted in no improvement. The calibration statistics reported in this study were very low. This may be due to the method used to measure the *SOG* of wood. More precise methods and equipment should be used in future studies to check the predictive value of *SOG* using NIR. Another reason for the poor prediction may have been the range of *SOG* assessed in this study. A very strong correlation between NIR and the *SOG* of wood has been reported, with an  $R_p^2$  of 0.80 (Gindl and Teischinger 2002). The range of observed values may have contributed to the calibration performance, as the range of *SOG* in their study was 0–40° compared to 1.33–16.67° in this study. A wide range of values has been reported to improve NIRS calibrations.



Figure 4.11: PCA plot based on raw spectra for SOG.



**Figure 4.12:** The relationship between measured and NIR-predicted values for *SOG* (°), based on the raw spectra for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

### 4.5.2 SOG based on calibrations for Qs and Fs samples

For the modelling of *SOG*, 72 and 48 samples of both species were used for the calibration and prediction sets, respectively. All models for *SOG* gave poor correlations, with  $R_c^2$  ranging from 0.15–0.38 and  $R_p^2$  from 0.17–0.24 for the *Qs* and *Fs* sections, respectively.

Raw spectra were used to develop calibrations, as pre-treatments failed to improve the model performance. The results based on *Fs* section were considered better ( $R_p^2 = 0.24$ ) than those based on *Qs* section ( $R_p^2 = 0.21$ ). An optimal spectral range of 1000–1960 nm has been reported to yield good predictions for *SOG* (Gindl and Teischinger 2002). This range slightly improved the model for the *Fs* section ( $R_p^2$  increased from 0.17 to 0.24). The poor predictions obtained for *SOG* may be due to the narrow range of values and the precision of the equipment used. A wider range of values and more accurate measurements should be considered in future research.

The PCA was used to distinguish between the grain orientations of the two softwoods. The PCA scores plots of the best model selected explain 97% and 99% of the variation in data for the Qs and Fs sections, respectively. The plot of the relationship between NIR predicted and measured values for the best model, selected based on the latter, is shown in Figure 4.13. For the regression

coefficients, negative correlations were observed at bands between 1432 and 1613 nm, which have been associated with cellulose, hemicelluloses, and lignin (Schwanninger *et al.* 2011). This is also similar to previous findings (Gindl and Teischinger 2002).



**Figure 4.13**: The relationship between measured and NIR-predicted values for *SOG* (°), based on the flat-sawn section for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

### 4.6 Annual Ring Width (ARW)

#### 4.6.1 ARW based on calibrations for all samples

The range of *ARW* observed in this study was 0.66-7.40 mm for hemlock and 0.10-13.10 mm for D-fir. For the modelling of *ARW*, the same numbers of samples as described in Table 4.1 were used for the calibration and prediction sets.

Raw spectra were used to develop calibrations for the *ARW*. Raw spectra gave poor predictions  $(R_p^2 = 0.18)$  for the rough surface and worse results for the smooth surface  $(R_p^2 = 0.04)$ . Pretreatments were applied but did not improve the model's accuracy. However, reducing the spectral range to 800–1400 nm improved the  $R_c^2$  from 0.36 to 0.44 and reduced the calibration error from 1.40 to 1.31 mm for the rough surface (Table 4.6). The reason for these suboptimal results might be the narrow range of values observed in this study.

Property	Roughness	Spectral Range	$R_c^2$	$R_p^2$	SEC	SEP	RPD
ARW	Rough	Raw Data (Full Range)	0.36	0.18	1.40	1.43	1.2
(All)		Reduced (800–1400) *	0.44	0.19	1.31	1.45	1.2
	Smooth	Raw Data (Full Range)	0.10	0.04	1.67	1.58	1.0
ARW (Qs)	Rough	Raw Data (Full Range)	0.56	0.34	1.06	0.81	1.2
	Smooth	Raw Data (Full Range)	0.54	0.25	1.08	0.81	1.2
		Smoothed	0.67	0.30	0.95	0.84	1.2
ARW (Fs)	Rough	Raw Data (Full Range)	0.27	0.09	1.62	1.81	1.1
		Raw Data (800–1400)	0.44	0.24	1.42	1.78	1.2
	Smooth	Raw Data (Full Range)	0.02	0.04	1.88	2.01	1.0

**Table 4.6**: Calibration statistics for *ARW* based on all samples (n = 240) and *Qs* and *Fs* samples (n = 120).

The poor calibration statistics observed in this study could also be attributed to the method used in determining the *ARW*. No past studies were found in the literature to serve as a basis of comparison. Further studies should be done using more accurate measurements and a wide range of reference values to improve calibration performance. The PCA plot for the best models selected explains the variation in the data (96% and 96%) for both surface conditions. The plots showing the relationship between NIR predicted and measured values for both surface conditions are provided in Figure 4.14.


**Figure 4.14:** The relationship between measured and NIR-predicted values for *ARW* (mm), based on the rough surface (top) and smooth surface (bottom) for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

## 4.6.2 ARW based on calibrations for Qs and Fs samples

For the modelling of *ARW*, 72 and 48 samples of both species were used for the calibration and prediction sets, respectively. Most of the models for *ARW* gave fair calibration values, with  $R_c^2$  ranging from 0.02–0.67 and  $R_p^2$  from 0.04–0.34 for the *Qs* and *Fs* sections, respectively.

Pre-treatments were applied to improve the model performance, but no effect was observed. For the rough surface, results based on the *Qs* section gave fair predictions ( $R_p^2 = 0.34$ ) compared to the *Fs* section ( $R_p^2 = 0.09$ ). Reducing the spectral range to 800–1400 nm and 1300–2300 nm failed to improve the model's performance except when the range of 800–1400 nm was utilized for the *Fs* section ( $R_p^2$  increased from 0.09 to 0.24, and SEP dropped from 1.81 to 1.78).

The smooth surface predictions were similar to those based on the rough surface. The *Qs* section gave a better prediction value ( $R_p^2 = 0.25$ ) than the *Fs* section ( $R_p^2 = 0.04$ ). SG smoothing also improved the prediction for the former ( $R_p^2$  from 0.25 to 0.30). A comprehensive report on the influence of pre-treatments on model performance has been published (Rinnan *et al.* 2009). Overall, the *Qs* section outperformed the *Fs* section for both surfaces. The best prediction for *ARW* was observed when the rough surface was utilized.

Calibrations were also developed using a CV method with 10 segments to test the predictive ability of the *ARW* model. The combination of SNV and second derivative was applied to the spectra to improve the model performance for the *Qs* section, and a reasonable prediction was obtained, with  $R_c^2 = 0.77$  and  $R_{cv}^2 = 0.50$ . This further shows it is possible to predict the *ARW* of wood using NIR with more accurate measurements and a wide range of values. Although new samples were not predicted, a CV method has been used by several researchers to determine the predictive ability of models (Gindl *et al.* 2001, Kobori *et al.* 2015).

The PCA was used to distinguish between the grain orientations of the two softwoods. The plots showing the relationship between NIR predicted and measured values for the best models selected based on the *Qs* section are provided in Figure 4.15. As observed from the regression coefficients generated by the PLS-R models, the relationship between the *ARW* and absorbance was highly variable, changing from positive to negative over the investigated wavelength range. The bands at

2200–2300, 1877–1930, and 1687–1700 nm were found to be fairly correlated with *ARW*. All band assignments for the NIR spectra of wood can be found elsewhere (Schwanninger *et al.* 2011).



**Figure 4.15**: The relationship between measured and NIR-predicted values for *ARW* (mm), based on the rough surface (top) and smooth surface (bottom) in the quarter-sawn section for the softwoods. The blue and red dots indicate the calibration and prediction sets, respectively.

## 5.0 CONCLUSION

NIRS coupled with PLS-R was found to be a suitable technique for the prediction of wood properties. While a good number of studies have utilized this technique to estimate several properties of wood, the current study focused on two coastal softwoods (Western hemlock and Douglas-fir), which have not received much attention from past researchers and thus make this essentially new research. The results of this study demonstrate that NIRS has the potential to predict some wood properties, although poor calibrations were developed for the *SOG* and *ARW* of wood, which could have been due to the narrow range of values investigated and the testing procedures. Differences between the calibration models for the separate sets (the Qs and Fs sections) of the two species utilized had not previously been explored and hence were the focus of this study.

Two sets of predictive models were developed in this thesis: one included all the samples (n = 240) and another consisted of samples from the *Qs* and *Fs* sections separately (n = 120) for both surface conditions (rough and smooth). Different surface conditions were used to simulate the actual situation in a sawmill, where the surface quality of lumber varies due to factors such as roughness, grain, and knots. The models that included all samples were inferior to the ones with separation of the samples into *Qs* and *Fs* sections for the assessed properties. For the first set, the best prediction was obtained for *MOE*, followed by *MOR* and then  $\rho$ , with poor results for *SOG* and *ARW*. The rough surface predictions on the smooth surface. The second set of predictions followed the same trend, with the best predictions obtained in the order of *MOE*, *MOR*, and  $\rho$ , and poor results for *SOG* and *ARW*. For this set, the *Qs* section gave better results than the *Fs* section for both surface conditions (rough and smooth), except for *MOR* based on a rough surface, which showed the opposite trend. The *Fs* section reported better results for *SOG* based on the smooth surface. Most of the models reported in this thesis met the 1.5 RPD criteria for a model to be considered as a preliminary screening tool.

The models developed using the reduced spectral ranges also showed slight improvement in model performance compared to those based on the full NIR range. These findings therefore show the feasibility of using portable, lightweight, and inexpensive spectrometers for predicting wood

properties by NIRS. The results of this study also show that the application of pre-treatments to the NIR spectra helped to optimize the model. It was further concluded that the same wood chemistry associated with wavelengths related to  $\rho$  was also found to be related to *MOE* and *MOR*. This could have been the result of the strength of the relationship established between these properties; hence, this study found that the same underlying chemical constituent that contributes to  $\rho$  prediction is also responsible for both *MOE* and *MOR*. The contributions of these polymers to mechanical properties have been studied extensively but are rather controversial. It is not entirely possible to comprehensively interpret the relationship between chemical components and mechanical properties. However, the absorption bands related to cellulosic features are certainly the main contributors to building robust models for predicting the mechanical properties of wood.

## 6.0 FUTURE RESEARCH

In light of this investigation, and acknowledging the constraints and limitations of the experimental design and available equipment, the following are suggested as areas for future research related to the use of NIRS as a non-destructive evaluation method to assess wood properties.

- The lower statistics reported in this thesis could have been a result of the samples used when developing calibrations; specifically, mixed-species calibrations could have caused the lower calibration statistics. Therefore, single-species calibrations with diverse samples should be considered in future studies to improve the quality of the model.
- Although clear wood samples were used in this study, a negligible number of samples were found to have small knots, which could have affected the calibrations. Studies should be conducted to determine the differences in calibrations for wood of higher and lesser grades.
- 3. One of the most limiting factors in NIRS calibration is the accuracy of the reference data. Several attempts have been made to determine the best analytical conditions (sample preparation and spectrum acquisition technique) that minimize scatter in NIRS methods. This is important because not even the most powerful NIRS calibration software can give reliable predictions from low-quality spectra. Therefore, work and time need to be spent on spectral collection and reference measurements to improve the overall statistical models from NIRS calibrations.
- 4. The NIR spectra in this study were collected in a static condition. However, it would be helpful to understand the influence of measurement conditions on calibrations. Combining the NIR spectra from moving measurement conditions and those from static conditions could improve the performance of the model.
- 5. NIRS calibrations based on samples with a wide range of values could be done to build more robust and reliable models. This is because smaller datasets and a narrower range of properties could compromise the model's quality more than the limitations inherent to particular species.
- 6. Several studies have linked the use of reduced wavelengths in NIRS studies to much lower cost, which necessitates the prediction of wood properties with narrow spectral ranges. So,

more studies should be carried out to assess calibration performance within narrow spectral ranges.

- 7. Further studies with well-defined grain orientation are also needed to get better results. Only two sections (*Qs* and *Fs*) of the three found in wood were investigated in this study. A study that utilized all three sections, that is the transverse, *Qs*, and *Fs* sections, should be considered. The direction of scan—for instance, with the transverse section, where variations in wood properties can be accurately captured—contributes to better predictions.
- 8. Finally, other technologies such as hyperspectral imaging, which combines NIRS and an imaging system and allows the accurate measurement of wood properties, should be explored.

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