Workshop on Commercial Application of IR Spectroscopies to Solid Wood

Philip J. Harris and Clemens M. Altaner (eds)
PREFACE

The workshop “Commercial Application of IR Spectroscopies to Solid Wood” was held on 11 June 2013 in the School of Biological Sciences, The University of Auckland, Auckland.

The workshop explored the issues that need to be covered before we can expect NIR to be taken up more widely by the forest industry. While the issues are general, those organising this workshop have a specific interest in addressing local issues and uncertainties.

We thank all the speakers and their companies/organisations for their support of this workshop. Their time in preparing material for the proceedings and their presence at the workshop was greatly appreciated.

We thank those who helped review and edit these proceedings, particularly Professor John Walker.

We acknowledge the sponsorship of the Solid Wood Initiative in this workshop together with our industry partners in the Compromised Wood programme, Forest and Wood Products Australia, Forestry Corporation of NSW, Future Forests Research, Proseed Ltd., The Radiata Pine Breeding Company, The University of Auckland and Weyerhaeuser (USA) Inc.

The cover image was kindly provided by S. Tsuchikawa and H. Kobori and shows a NIR based wood grader.

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TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Authors</th>
<th>Title</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Keith Mackie</td>
<td>Introduction</td>
<td>1-2</td>
</tr>
<tr>
<td>Richard Streamer</td>
<td>What NIR Instruments to Use</td>
<td>3-16</td>
</tr>
<tr>
<td>Marlon Reis</td>
<td>NIR for On-Line Measurement</td>
<td>17-26</td>
</tr>
<tr>
<td>Armin Thumm</td>
<td>NIR Implementations for the Solid Wood Industry Investigated at SCION</td>
<td>27-36</td>
</tr>
<tr>
<td>Geoff Downes, Manuel Touza, Maximillian Wentzel-Vietheer and Chris Harwood</td>
<td>NIR Detection of Tension Wood in <em>Eucalyptus globulus</em></td>
<td>37-48</td>
</tr>
<tr>
<td>Satoru Tsuchikawa and Hikaru Kobori</td>
<td>Using NIR for Grading Timber</td>
<td>49-54</td>
</tr>
<tr>
<td>Nicholas Davies and Clemens Altaner</td>
<td>Natural Durability of Redwood (<em>Sequoia sempervirens</em>) Heartwood</td>
<td>55-64</td>
</tr>
</tbody>
</table>
1. INTRODUCTION

Wood is a natural composite mainly of carbohydrate and lignin. Its structure is complex and comprises both structured and amorphous components that together are designed to withstand the ravages of biological and physical forces.

Due to varying genetics, the influence of site and silviculture, and the inherent variability in the distribution of different wood types (corewood, sapwood, heartwood etc.) across stems and from top to bottom means that lumber derived from trees is very variable in density, stiffness, durability and stability. This variation is particularly prevalent in fast grown plantation pines as planted in New Zealand.

The utilisation of wood in building structures (lumber, LVL etc.) demands that the physical properties of wood be known so that engineering factors can be assigned. In comparison to wood, competitive materials such as steel are highly uniform and ‘predictable’. Hence the segregation of wood products such as lumber based on their quality in order to reduce its variability is a significant factor in deriving value from plantation species such as radiata pine.

Many technologies are being used in wood segregation which measure stiffness, density, moisture content etc. For a considerable period of time NIR has been considered a ‘likely’ candidate on the basis that NIR can provide a (largely surface) chemical fingerprint of the wood.

2. NIR HAS UNDERDELIVERED

Much research has been undertaken testing the potential for NIR to measure and predict wood properties over the past 20 years. This work continues today but it is noteworthy that very few industrial applications of NIR in the solid wood processing industry are in place. Despite numerous applications being tested and showing promise in controlled laboratory conditions few have been translated to the rugged and demanding wood processing workplace.
By way of example Solid Wood Innovation (sponsor of this workshop) has for some nine years been trying to develop technology for predicting the in-service stability of lumber used in framing systems.

We know the stability of lumber is driven by both the chemical and physical variability in wood and that being able to successfully measure lengthwise shrinkage patterns in lumber would enable lumber stability to be predicted. Using small wood coupons and carefully surfaced/prepared samples, research using NIR gave highly promising results as shown in the table below. For both green and dry wood samples quite good coefficients of prediction were obtained.

<table>
<thead>
<tr>
<th>Model</th>
<th>Tangential shrinkage</th>
<th>Radial shrinkage</th>
<th>Longitudinal shrinkage (block)</th>
<th>Longitudinal shrinkage (pin)</th>
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<td>Green samples, NIR range</td>
<td>0.85</td>
<td>0.79</td>
<td>0.80</td>
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<td>Dry samples, NIR range</td>
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<td>0.75</td>
<td>0.77</td>
<td>0.75</td>
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</tbody>
</table>

Despite these promising results the NIR work never progressed beyond this point. In lumber manufacturing factors that are encountered include:

- Varying wood moisture content
- Varying surface finish (planed and rough-sawn)
- Varying surface ‘age’
- Requirement for scanning at lumber speeds of around 1000 meters per minute
- Rugged and demanding environment

When these factors are considered NIR has fallen by the wayside.

3. LOOKING AHEAD

The requirement for better segregation technologies in solid wood processing will increase, as customers demand higher performing products. The need for rapid, inexpensive and accurate wood quality assessment tools generically will remain.

To meet this demand a concerted effort needs to go into trialling NIR in a well-defined and relatively straightforward application where we can gain experience with the practical impediments outlined above.

SWI is committed to working with researchers by finding such commercial applications and setting suitable performance requirements that can be accessed for this information. What is important here is to discuss how each technology may be applicable to the measurement of solid wood.

4. AFFILIATIONS

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Abstract: Near Infrared (NIR) spectroscopy has been used for timber, pulp and paper applications for over 20 years. Despite a lot of R&D work on solid wood applications for both field and in-process use, the industry has not adopted NIR as a routine technique. Solid wood has proven to be a challenging sample type to analyse in real time by NIR as the modern timber mill processes logs at a very fast rate. The available instrumentation has, up until the recently, been too slow for on-line measurements in this environment. This paper describes the performance requirements for NIR systems for solid wood processing and discusses the various instruments available.

1. INTRODUCTION

Near Infrared (NIR) spectroscopy has been used for timber, pulp and paper applications for over 20 years. There are many installations worldwide for pulp and paper, medium density fiberboard (MDF) and particleboard but there are few installations for solid wood processing. This is despite a number of applications for solid wood being developed at the R&D level (on discrete, static samples) for both in-field use and on-line in the timber mill. Solid wood has proved to be a challenging sample to analyse on-line by NIR as the modern timber mill processes logs at a very fast rate. The available instrumentation has, up until the last few years, been too slow for real time measurements in this environment. Apart from speed, consideration also needs to be made for key instrument parameters such as robustness of the instrument hardware, scan range, transferability of data and instrument performance parameters.

2. KEY NIR INSTRUMENT PARAMETERS FOR PROCESSING SOLID WOOD

2.1 Portable / Lab / On-Line

There is a wide range of NIR instrumentation available for use in the field, in the lab or on-line. A lot of development in the technology has occurred over the last 5 years and some systems have good potential for solid wood applications. A number of key parameters need to be considered when choosing an instrument for a given application. As most potential NIR applications have been developed by research groups using high quality full wavelength range instruments, it is important to understand the requirements and potential trade-offs when looking at systems which are going to be used for routine use. The key parameters for consideration are discussed below.
2.2 Wavelength Range, Bandpass and Spectral Resolution

The NIR region covers the wavelength range from 700-2500 nm. There are many instrument technologies available which cover all or only parts of this range. These will be discussed later. NIR is characterised by combinations and overtones of fundamental mid-IR absorptions. These overtones or harmonics represent the same information repeated down the spectrum. This is characterised by a decrease in absorbance and a broadening of bands which may decrease sensitivity for some measurements. Therefore the spectral region which is used to develop an application needs to be matched to the appropriate instrument. Figure 1 shows the various absorption bands which are seen in the NIR region.

![Absorption Bands in the Near-Infrared](image)

Depending on the type of NIR instrument, bandwidth or spectral resolution may be fixed or variable. Instruments such as dispersive and diode array systems have fixed bandwidth. This is often around 10nm. Systems such as FT-NIR can have variable spectral resolution as high as 2 cm\(^{-1}\). For solid wood applications, narrow bandpass or high spectral resolution is not needed as the bands are very broad.
WHAT NIR INSTRUMENTS TO USE

The number of data points in the spectrum (sometimes incorrectly referred to as spectral resolution) is important. This again varies depending on the type of instrument and can be anything from 1 data point every 0.5 nm or up to 1 point every 10 nm or 20 nm. When choosing an instrument for solid wood applications this is a more important parameter than bandwidth or spectral resolution.

2.3 Scan Speed

The speed at which NIR instruments can collect spectral information varies greatly depending on the type of instrument. Typical scan rates can vary from 1 or 2 scans per second to as many as a 1000 scans per second. Each sample is generally scanned multiple times and the spectra co-averaged to improve signal to noise ratio. For measurements on static samples, where speed is not as critical, an analysis can typically take 10 to 30 seconds.

For on-line measurements in the timber mill the scan speeds need to be much faster as timber is processed at a very rapid rate. Logs are processed in seconds so the NIR data acquisition needs to match this speed. A few technologies are now available which can scan at very rapid rates which may be suitable for solid wood applications.

2.4 Fiber Optic Attenuation

On-line NIR systems often utilize fiber optics as the interface between the instrument and the sample. The fibers used in NIR are commonly made of an ultra-low OH silica and they exhibit a dramatic decrease in signal at the longer NIR wavelengths above 2200 nm. This reduced energy means that the wavelengths above 2200 nm cannot be used for on-line measurements. This is important to keep in mind when choosing systems for solid wood measurements or when developing calibrations based on lab instrument data if the ultimate aim is to use the data on-line with a fiber optic based system.

2.5 Signal to Noise (S/N)

Signal to noise is a very important parameter for NIR as it affects analytical performance in terms of accuracy, precision and sensitivity of the measurement. Signal to noise can be improved by increasing the number of scans taken for each sample analysis. This also increases the time for measurement, which may not be desirable, particularly for on-line applications. Optimal signal to noise needs to be determined for each application, especially for applications where the component of interest is at low levels or where high precision is needed.

2.6 Sample Size / Sample Area

The properties of solid wood are quite variable both radially and longitudinally. When scanning samples by NIR, the sample size and sample area must be considered. In some process systems the area of illumination of the sample can be
changed. The selection of this parameter will be application dependant and needs to be determined accordingly. Too small an area may give quite variable results as even small variations in the sample properties will be seen. If the area of illumination is too large the sample variations will be “averaged” out and result in no variation being seen.

2.7. Ruggedness

For field or on-line applications the environment can be very harsh and variable. Unlike a laboratory instrument, a portable or process instrument needs to tolerate temperature fluctuations, humidity changes, vibration, dust and mistreatment by plant operators. A timber mill generally suffers from all of these variables.

On-line instruments are often packaged in NEMA 4x (IP65) enclosures which are splash and dust proof. These enclosures can also be made explosion proof and can have heating or cooling depending on the environment.

Many of the NIR instruments have moving parts to generate the NIR scan (eg dispersive monochromators and FT-NIR). These are susceptible to vibration and will affect the accuracy and repeatability of the system. Various methods of dampening vibration can be employed to protect the instrument. These are generally quite effective unless the vibration is severe.

Some instruments (eg diode array) have no moving parts and can tolerate quite high levels of vibration. These systems have limited scan ranges however and may not be suitable for the application.

2.8. Ability to Transfer Data / Calibrations

The calibration process for NIR is well documented and may require a large number of samples which have been analysed by traditional reference methods for the parameters of interest. These are then scanned on the NIR and chemometric modelling performed. This procedure can be expensive and is generally not something that can be performed by staff in the field or in a mill environment. The development of NIR methods for solid wood will generally be undertaken by research groups or skilled chemists in a lab environment. Calibrations developed by these groups will need to be transferred to other systems implemented in the field or in the mill. Therefore it is important to consider the ability to transfer data when choosing an instrument and developing methods. Some instrument types can transfer methods well, others cannot. Also, a poorly developed calibration may not transfer regardless of the instrument being used.

2.9. Price (instrument, installation and implementation)

The price for NIR instrumentation can vary greatly depending on the technology. It is important to remember to choose the right system for the job and not just to buy on price. A key part of the overall cost of a project is not just the price of the instrument however. The cost of implementation also needs to be factored in. This will vary depending on the application and includes the cost of calibration, and for
on-line installations, will depend on the level of automation required. In some cases the cost of implementation can be equal to or higher than the price of the instrument. It is very important to determine the economics for the overall project in terms of pay-back or return on investment. This can be quite high and needs to incorporate many factors. These include potential lab savings, time saving, product improvement, elimination of waste product, improvement in throughput etc.

3. NIR INSTRUMENT TECHNOLOGIES

As already mentioned there are now many NIR instrument technologies available. Of these, the technologies that have been used or may be useful for solid wood applications are shown below along with a brief description of the technology. It is not in the scope of this paper to discuss how each technology functions or examine all the available sampling options. There are many publications and manufacturers’ technical brochures that can be accessed for this information. What is important here is to discuss how each technology may be applicable to the measurement of solid wood.

3.1 Dispersive Monochromator (grating based technology)

This technology has the widest wavelength range available with some systems covering the visible and NIR region (ie 400-2500 nm). Dispersive systems are available in lab and on-line configurations and have been used for R&D, lab and process applications for some timber pulp and paper applications for many years. A schematic of a typical laboratory system is shown below (Figure 2).

Dispersive systems have high signal to noise and excellent wavelength accuracy and precision. Dispersive systems generally have fixed bandpass (approx. 8-10 nm) and some have data points every 0.5 nm.

Scan speeds for dispersive systems are typically around 1 to 2 scans per second. It is usual to take multiple scans of a sample (usually 8 or 16) to analyse 1 sample. The result for a given sample is usually produced in about 10 seconds. Therefore their applicability to on-line solid wood processing is limited.

Typical dispersive systems have a wide range of sampling accessories for lab units and on-line systems can be equipped with numerous fiber optic devices for measurement in various processes. Examples of typical dispersive systems are shown below in Figure 3. Dispersive systems do have moving parts, the most critical of which is the moving grating which produces the individual NIR wavelengths. These can be affected by high levels of vibration in the process environment. Therefore steps do need to be taken to dampen this out.

With most dispersive systems it is possible to transfer data and calibrations between the instruments of the same type with the same sampling system. This allows for easier implementation of future systems.
Figure 2. Schematic of a Pre-Dispersive NIR Monochromator

Figure 3. Typical Lab and Process Dispersive NIR instruments
3.2 FT-NIR

FT-NIR systems utilize some form of interferometer. This technology has been around for a number years and is similar to the technology used in FT-IR spectrometers. The layout of a typical FT-NIR instrument is show below in Figure 4. As with dispersive systems FT-NIR instruments can also cover a wide wavelength range but do not cover the visible region. The scan speed, signal to noise, wavelength accuracy and precision are comparable to dispersive systems. FT-NIR systems also have variable resolution and can be run at much higher spectral resolutions than dispersive systems. This is generally not required for most NIR measurements, however, as the absorption bands being measured are quite broad. This is particularly the case in solid wood. FT-NIR systems scan the entire spectrum simultaneously at a rate of about 1 to 2 scans per second. As with dispersive systems a number of scans are co-averaged to produce the spectrum used for sample measurement. As such their use for solid wood processing in an on-line timber mill application is limited.

![Figure 4. Schematic of a typical FT-IR/NIR instrument](image)

Typical FT-NIR instruments have a wide range of sampling accessories for lab units and on-line systems can be equipped with numerous fiber optic devices for measurement in various processes. Examples of typical FT-NIR systems are shown in Figure 5.

FT-NIR instruments also have moving parts. These can be affected by high levels of vibration in the process environment. Therefore steps do need to be taken to dampen this out.

With most FT-NIR systems it is possible to transfer data and calibrations between instruments of the same type with the same sampling system. This allows for easier implementation of future systems. Claims have also been made that data can be transferred to or from other instrument types.
3.3. Diode Array (also dispersive technology)

Diode array instruments have been on the market for some time in both lab and online forms. As with dispersive systems they use a grating to produce the NIR wavelengths. In diode array systems this grating is fixed and all wavelengths are produced at the same time. The diode array detector element measures all these wavelengths at one time and at a very fast rate. Scan times are in the order of 5 to 50 milliseconds so results can be generated in under a second. Diode array systems also have no moving parts and are insensitive to vibration. This makes them suitable for process measurements of solid wood.

A schematic of a typical diode array system (Figure 6) is shown on the next page along with an example of a diode array process analyser (Figure 7).

Diode array systems are available in numerous configurations that enable implementation for many sample types. There are some aspects of diode array systems which need to be considered. The main parameter is wavelength range. Diode array systems do not cover the full NIR spectral region. Common ranges are 1100-1650 nm, 800-1100 nm and 950-1650 nm. Therefore it is important to understand what spectral region is required for the measurement to be performed. The other aspect of diode array systems is the number of spectral data points collected. This can vary from 128, 256 or 512 data points across the spectral region measured depending on the number of detector elements or pixels in the diode array. For example, a system with a scan range of 1100-1650 nm (ie 550 nm range) using a 512 pixel array will have a data point every 1.1 nm. The number of data points can affect the accuracy, precision and sensitivity of the measurement.

The final aspect of diode arrays to consider is transferability. Great improvements have been made in the production of the diode arrays and they are far more reproducible now compared with the first systems developed. This combined with the higher number of pixels in the array makes calibration transfer between instruments possible.
Figure 6. Schematic of a diode array analyser

Figure 7. Typical diode array process analyser
3.4. Acoustic Optical Tunable Filters (AOTF)

AOTF systems use a crystal such as tellurium dioxide (TeO$_2$), which is driven at acoustic frequencies by a piezoelectric transducer. When vibrated at given frequencies, polychromatic light that enters the crystal is split into the NIR wavelengths to generate a spectrum. Figure 8 shows a schematic of a typical AOTF system. The wavelength range of AOTF systems is broader than diode array systems but still does not cover the wide range of monochromators or FT-NIR instruments. Typical wavelength ranges are 850-1700 nm, 900-1800 nm and 1100-2300 nm. The spectral resolution can vary from 2 to 10 nm and the number of data points can vary from 1 to 10 nm.

AOTF systems scan at a very fast rate, typically less than 100 microseconds. AOTF instruments generally have poor signal to noise however, so a number spectra are usually co-averaged to produce analytical results.

As with diode array systems, AOTF instruments do not have moving parts so they are relatively insensitive to vibration. These factors make them appear ideal for process applications that require high-speed measurements such as solid wood processing.
WHAT NIR INSTRUMENTS TO USE

AOTF systems are available in portable, laboratory and on-line configurations. Some typical AOTF systems are shown in Figure 9.

![Figure 9. Typical portable and process AOTF instruments](image)

For any given application the signal to noise, wavelength accuracy and precision and transferability of AOTF needs to be examined as these factors may reduce the effectiveness of these systems for solid wood applications.

3.5 Micro-Electro-Mechanical-Systems (MEMS)

MEMS is generic term for mechanical devices that are manufactured using silicon wafer technology derived from the semiconductor industry. As the name suggests the spectral engine of MEMS systems is quite small and the instruments manufactured using MEMS technology are also very small. MEMS systems are available in portable, lab and on-line systems.

One example of a MEMS system is the Digital Transform Spectrometer (DTS). The DTS MEMS chip is an array of programmable micro-mirrors which can be raised and lowered to form a programmable diffraction grating (Figure 10). These elements are analogous to piano keys with a gold mirror on top. If all of these elements are in their normal “up” condition, the surface reflects the light from each pixel. If an element is activated, it acts as a diffraction grating generating individual NIR wavelengths. The activation of each element is very fast so MEMS systems scan the wavelength region they are covering in about 600 milliseconds.

Portable MEMS devices have already been examined for solid wood measurements in the field with some success. An example of one of these devices is shown in Figure 11.

The wavelength range of MEMS systems is limited. Typical wavelength ranges are 1000-1800 nm or 1600-2400 nm. The spectral resolution can vary from 2-10 nm and the number of NIR data points can vary from 1-10 nm. AOTF systems scan at a very fast rate, typically less than 100 micro seconds. The analysis time is usually around 3 to 10 seconds. As with diode array systems, AOTF instruments do not have moving parts so they are relatively insensitive to vibration. AOTF systems scan at a very fast rate, typically less than 100 microseconds. The analysis time is usually around 3 to 10 seconds. As with diode array systems, AOTF instruments do not have...
moving parts so they are relatively insensitive to vibration. As with diode array and AOTF systems, MEMS devices need to be assessed for their suitability for solid wood measurements based on the application of interest.

For any given application the signal to noise, wavelength accuracy and precision and transferability of MEMS devices needs to be examined as these factors may reduce the suitability of these systems for solid wood applications.
WHAT NIR INSTRUMENTS TO USE

4. CONCLUSIONS FOR SOLID WOOD APPLICATIONS

From the above discussion it can be seen that the choice of an NIR instrument for solid wood measurements will depend on many factors. Table 1 summarizes the key instrument features discussed above and solely with the application to solid wood applications in mind. The ranking is out of 5 and is based on the opinion of the author. It is not the purpose of this paper to make any recommendations as to which instrument should be used for solid wood applications. However these features should be kept in mind when trying to determine what system should be considered when determining a suitable system.

<table>
<thead>
<tr>
<th></th>
<th>Range</th>
<th>Resolution</th>
<th>Speed</th>
<th>Sampling</th>
<th>Ruggedness</th>
<th>S/N</th>
<th>Transfer-ability</th>
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</tbody>
</table>

*Table 1 Comparison of key parameters of NIR instrument technologies.*

As many applications for solid wood have already been developed at the R&D level the processing industries, such as timber mills needs to determine what it wants from an NIR and how much they are willing to pay for it. The R&D community can then determine which NIR instruments may be suitable for these applications. Commercial NIR companies and their engineering partners can then decide if there are economically justifiable projects worth pursuing which can be of benefit to all concerned.

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6. AFFILIATIONS

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NIR FOR ON-LINE MEASUREMENT

MARLON REIS

NIR FOR ON-LINE MEASUREMENT

Experiences developing on-line measurements in meat processing plants

Abstract: A common problem found in biological industries is the variability in the raw material entering the process. In certain cases, this variability is outside specification and can affect the quality of the final product. Thus, the ability to detect the suitability of the raw material early or even before entering in the process allows the out-of-spec material to be managed, preventing inconsistencies in the final product. Near Infra-Red Spectroscopy (NIR) has been widely used in biological industries to measure key properties and so support decision making during the processing of biological materials. However, its development has not always been straightforward. This work reviews the development of NIR for applications in the meat industry and refers to two studies, which show the importance of the design of the data collection on the development of a practical NIR application.

1. INTRODUCTION

A series of biochemical and structural changes take place in the carcass muscles after the animal is slaughtered. These changes lead to rigor mortis. Then, a new series of changes take place, which combined with those pre rigor changes will define the meat quality attributes in the muscles from that carcass. Several factors (pre and post slaughter) affect these changes, some are manageable during the carcass processing, but others are not. Hence, inconsistencies in the final meat quality of the carcass can occur. A strategy to deal with these inconsistencies is to identify abnormal carcasses early on in the process and manage them towards a desire set of attributes. To be successful this strategy requires the carcass to be evaluated within a time frame that allows it to be managed towards a desired use. This represents a typical problem found in process monitoring, where the raw material enters the process in a variable state and its quality needs to be assessed in a timely manner. In this case, measuring technologies play the important role of providing reliable information, in a timely fashion, to support decision making.

In NIR spectroscopy a beam of electromagnetic radiation is shone onto the sample leading to changes in the vibrational status of molecules due to the radiation-sample interaction (Barton, 2002; Pasquini, 2003). NIR allows high penetration of the incident radiation into the sample allowing the non-invasive detection of the radiation backscattered, or transmitted through the sample. For complex solid samples such as meat, the radiation-sample interaction involves mainly two principles: scattering and absorption, the former is related to the microstructure of the sample and the later results from the chemical composition of the sample. This allows NIR to be used to study/monitor meat attributes, which depend not only on the meat’s chemical composition but also on structural components.

A large number of studies have investigated the NIR technology for this purpose, showing variable results (Prevolnik et al., 2004; Prieto et al., 2009;
Weeranantananaphan et al., 2011). This work reviews the development of NIR for an *in plant* application, where NIR is considered as a replacement technology for an existing system for carcass classification already in use at the plant. Thus the main focus of the development process is to achieve a performance similar to or better than the existing technology. The aim of this work is identify the key factors that allowed this challenge to be overcome, leading to an application with potential to be used *in plant*.

2. METHODS

A common practice in the New Zealand meat industry is to process carcasses using hot boning, where removal of fat and lean muscle from the carcass takes place before *rigor mortis*, with the muscles still warm. Meat with ultimate pH (pH achieved after 24 hours post slaughter) lower than 5.8 is considered as having normal pH, presenting desirable eating quality attributes (Braggins, 1996; Dransfield, 1981; Gill and Newton, 1981; Purchas and Aungsupakorn, 1993; Silva et al., 1999; Viljoen et al., 2002; Watanabe et al., 1996). However measurement of ultimate pH on hot boned muscles is not feasible, as cuts are already packed, and in many cases frozen, by the time the ultimate pH is reached after 24 hours *post mortem*. Currently, a wet chemistry based method to estimate the ultimate pH from samples excised from the carcass prior to boning is used in some New Zealand abattoirs. The method is based on the relationship between *pre rigor* glycogen content and ultimate pH (Young et al., 2004). It has been reported that NIR is able to predict glycogen content in *pre rigor* meat with reasonable accuracy ($R^2 = 0.70$) from spectra collected in a laboratory environment NIR (Rosenvold et al., 2009). As NIR is a non-invasive technique able to be used in plant providing a rapid assessment of the carcass (less than 30 sec.), not requiring consumable or excision of samples, it was proposed as replacement for wet chemistry based method. In this work two studies investigating this possibility are reviewed, one pilot study ($n = 90$) (Lomiwes et al., 2010) and the second involving data collected from 657 carcasses (Reis and Rosenvold, 2013), both carried out in a commercial environment.

2.1. Study 1 ($n = 90$)

In this study samples and spectra were collected on-line from 90 animals: 30 steers; 30 bulls; and 30 cows, following routine processing conditions of the plant in a single day (Lomiwes et al., 2010). While the majority of steers and cows presented an ultimate pH below 5.7, bulls present in general higher ultimate pH. Combined, these categories of animals would provide a range of variation in ultimate pH, allowing the development of predictions (or classification) models. Although limited in the number of animals, this study involved a comprehensive collection of samples and measurement of pH at the time NIR spectra were collected (45 minutes post-mortem) and 48 hours later, as well as quantification of glycogen on samples collected when NIR spectra were obtained. A series of chemometric approaches were used and are described in details in (Lomiwes et al., 2010).
2.2. Study 2 (n = 657)

This study involved a more comprehensive data collection, including data collected from 657 carcasses during three different weeks, comprising cows, bulls, steers and heifers. The data collected were split between calibration and validation (~50% each) data sets representing the main known sources of variation, as shown in Figure 1. Partial least squares method (PLS) was used to fit the models, where NIR spectra are calibrated against ultimate pH values. Then the predictions are compared against the threshold pH of 5.8. The performance of the model(s) is evaluated by calculating the percentage of samples correctly classified above or below the threshold pH of 5.8 (Reis and Rosenvold, 2013).

![Figure 1. Distribution of carcasses in Study 2 according calibration and validation data sets (Cal and Val, respectively). Low and High refer to the ultimate pH as being below or higher than 5.8, respectively. The height of each corresponds to the average ultimate pH of carcasses and its colour indicates the day when the spectra were collected.](image)

3. RESULTS

3.1 Study 1

In Study 1, two approaches were considered to predict the ultimate pH:

- Indirect, where the model was fitted to predict glycogen and then the predictions from NIR spectra were used to forecast ultimate pH; and Direct, where ultimate pH is predicted directly from NIR spectra.
Different animal categories (steers, cows and bulls) were combined in single data set to achieve the range of variation in ultimate pH necessary to fit PLS based models.

In both approaches (direct and indirect) the ability to classify the carcasses as below or above the threshold pH of 5.8 was not achieved (Lomiwes et al., 2010). It was observed that:

- There was a confounding factor between animal category and processing conditions, where the speed of animal processing was different across the three categories (Figure 2).
- The models were not able to handle the confounding factor between animal category and ultimate pH (Figures 3 and 4).
- There was a distinction between NIR spectra from bulls and steers (Figure 5) and this was not related with attributes of the measured carcasses.

Figure 1. Distribution of carcasses according to time (in minutes) from start of the data collection. The height of each bar is arbitrary.

The results from Study 1 indicated that: a range of variation on ultimate pH should be included within each animal category; that the same animal category should be monitored under different processing conditions, i.e., different chain speeds; and specific models for each animal category could improve the results.
Figure 2. Hierarchical cluster analysis (HCA) of spectra in Study 1 displayed as a dendrogram on the right. The heatmap graph represents a set of variables of each carcass regrouped based on the dendrogram. On the far left the number attributed to each group is shown. pH45min and Temp45min correspond to the pH and temperature measured when NIR spectra were collected for that carcass. Glycogen corresponds to glycogen content from sample collected at the same time as the NIR spectra were collected. Ultimate pH corresponds to the pH measured in the muscle 48 hours later, while Dress, CarcassesWt and Live Wt correspond to the weight of carcass and animal at different stages of the processing.
3.2. Study 2

In Study 2, a more comprehensive data set was obtained (Figure 1). This resulted in a variety of carcasses sizes monitored at different shifts, where the time from
slaughter for the carcass to reach the NIR was between 20 minutes to 40 minutes, depending on the shift and animal being processed (Reis and Rosenvold, 2013). Hence, NIR was evaluated against different sources of variation (animal category, carcass size and chain speed).

Under these parameters, better performance was achieved, especially for the non-bulls model (fitted with data from cows, steers and heifers) which had overall rates of correctly classified samples of 90% for high ultimate pH and 89% for normal ultimate pH (ultimate pH<5.8) in the validation data set (Reis and Rosenvold, 2013). Figure 6 and 7 compare the prediction of NIR performance for prime (steers and heifers) and bulls against the existing technology in use at the plant (RapidpH™). In both technologies a future prediction of ultimate pH is made from measurement taken up to one hour after post mortem, when rigor has not yet been reached. Thus it is expected that these measurements are affected by a series of upstream conditions affecting the rigor process, such as variations in the stimulation, temperature, as well as natural biological variation from animal to animal (Hwang et al., 2003). Therefore variation between the prediction of ultimate pH and its measured value is not only due to instrumental and sampling variation, but also due to variation associated to the carcass processing. Hence, NIR showed equivalent performance, being unaffected by the variation associated with changes in the speed carcasses are processed. This suggests that NIR has the potential to replace wet chemistry based evaluation of ultimate pH, at least for prime animals (steers + heifers).

Figure 5. Rate of well classified prime (steers and heifers) carcasses in the validation data set (n=232) from Study 2 predicted with RapidpH™ or NIR (Reis and Rosenvold, 2013).
4. CONCLUDING REMARKS

The second study represents an important step towards the development of in plant NIR based application, as it achieved a performance similar to the existing technology when tested in actual abattoir conditions. Altogether, the results of the two studies reviewed in this work identifies as a key factor for the development of NIR for an in plant application the ability to capture representative data sets spanning as many as possible sources of variation, affecting directly or indirectly NIR spectra. While a representative data set will always be required for the development of reliable calibration for NIR, the identification of sources of variation is not always obvious. In this case, the first study (a pilot study), although presenting poor performance for the models, allowed the identification of potential sources of variation, which was an important contributing factor for the design of the second study. Thus, pilot studies carried out to identify feasibility of NIR for a given application should be planned to capture sources of variation and its results should be investigated thoroughly. i.e., the feasibility analysis should not be restricted to evaluation of performances measures (e.g. $R^2$), but should also involve the analysis of factors that can be managed to allow the development of the NIR system.

5. ACKNOWLEDGMENT

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NIR IMPLEMENTATIONS FOR THE SOLID WOOD INDUSTRY INVESTIGATED AT SCION

Between 1996 and 2013

Abstract This paper gives a high-level overview of NIR applications which were investigated at Scion for the implementation in the solid wood processing industry. Whilst many quality issues of the solid wood processing industry could be addressed by NIR technology, to date no NIR application has achieved commercialisation.

1. INTRODUCTION

In the forestry sector NIR technology has had some commercial success in the area of pulp and paper as a process monitoring tool (Antti et al., 1996; Dahlquist et al., 1999) and to determine the pulp yield of (standing) trees (Schimleck et al., 2005). It is also used as a tool by tree breeders to measure heritability (Raymond et al., 2001). NIR technology has however to date not led to any commercially viable tools for the solid wood industry. Scion has over the years investigated various NIR opportunities for wood processors. This article is giving an overview of the issues addressed and the outcomes.

2. NIR IMPLEMENTATIONS

2.1. Internal checking

The issue: upon kiln-drying, radiata pine lumber can develop intra-ring checks which typically occur in the area of the heart/sapwood boundary. This is a highly undesirable trait which renders lumber unsuitable for remanufacturing. Identifying trees susceptible to intra-ring checking early-on makes it possible to direct them away from a remanufacturing process stream.

NIR opportunity: identify susceptible trees based on assessment of core samples or log ends.

Equipment: discs, simulating log ends, were investigated with a portable NIR spectrometer from ASD (Field Spec Pro, USA). Ground material, representing material obtained from cores, was investigated with a benchtop Foss 6500 instrument. Both instruments were using the NIR region 1100 – 2500 nm.

Outcome: NIR could not reliably detect trees susceptible to internal checking. This indicates that internal checking is not strongly encoded in the chemistry of wood.
2.2. Green timber stiffness and stability

The issue: A sawmill is typically unable to determine whether a piece of lumber is fit for purpose until it has been cut into structural timber of a certain size (e.g., 4x2, 6x2) and kiln dried. At this point most of the effort, and cost, has already occurred.

NIR opportunity: Determine stiffness and stability of potential future products based on first opening surface (cant), i.e., before a cant is broken down into specific products. Unlike ultrasound, NIR could give spatially resolved stiffness and stability values across a cant. Based on these, a decision can be made which part of the cant (if any) can be cut into a structural product and which part can be used for other purposes, e.g., remanufacturing.

Equipment: A Bruker Matrix-F instrument was used to scan 4.8 m cants on the center line (pith to pith) and then at 50 mm offsets to either side (Figure 2). The instrument was moved along the whole lengths of the cant while spectra were accumulated.

Outcome: The correlation between predicted stiffness based on the cant and the actual cut lumber was $r^2 = 0.57$. This was sufficient to correctly segregate timber into a class which would achieve MGP8 and a class which is below MGP8. MGP8
means lumber with an average stiffness of 8 GPa when measured by machine stress grading. This is a common class for structural timber. NIR was not able to predict the stability of timber in terms of warp (crook, twist, bow) after drying.


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2.3 Veneer stiffness

*The issue:* the manufacture of plywood panels requires the face gluing of individual veneers to form a panel. The overall strength and stiffness of the final panel is a result of the strength and stiffness of the individual veneers, particularly the two face veneers. To create plywood of consistent stiffness it is necessary to measure the stiffness of the individual veneer sheets before lay-up. This is typically done by employing an ultrasound measurement. The quality of the ultrasound measurement can however be heavily influenced by moisture and density of the veneer sheet.

*NIR opportunity:* if an NIR based system can reliably measure the stiffness of veneer sheets it would be a superior technology compared to ultrasound.

*Approach and equipment:* veneer sheets were scanned at industrial operational speeds by passing them underneath a NIRSystem DirectLight spectrometer on a
conveyor belt. Each veneer sheet was then cut into smaller sheets and used to create 6-ply plywood samples (Figure 3). These plywood samples were then used to determine the stiffness of the sheet. Veneer stiffness was consequently regressed against the NIR scan data.

Outcome: the stiffness measurements by NIR showed a substantial improvement in accuracy compared to the ultrasound measurements (if ultrasound measurements are uncorrected for moisture and density).


![Figure 3. Assembly of plywood test samples from a single veneer sheet.](image)

2.4. Resin features in (and below) timber surfaces and on log ends

The issue: the presence of resin defects severely affects appearance grade lumber. Currently, wood products like weather boards or mouldings etc with obvious resin features are identified and rejected near the end of the production process.

NIR opportunity: ideally, logs that are severely affected by resin defects such as streaks, shakes or pockets need to be segregated prior to breakdown to remove the worst logs from further processing. This could be achieved by scanning log ends for resin features. Also, later in the production process, visual grading could be replaced by an NIR scanner to reliably locate detrimental resin features on or near the surface of a future wood product.
Approach and equipment: a range of defined portions of an artificial resin placed on a wooden background were presented to an NIR probe (Foss 6500 with remote probe). This information was used to create a model which can predict the severity of a resin feature.

For a spatially resolved approach wooden discs containing resin features were scanned with a hyperspectral camera (Specim, ImSpector N17E). A resin model was established by allocating each individual pixel to be either a “resin pixel” or a “non-resin pixel”.

Outcome: NIR can clearly distinguish between wood and resin based on the substantially different chemistries of these materials. NIR can also detect resin just below the surface which may be undetectable by the human eye. Consequently an NIR probe was able to detect resin features on the surface of lumber and allocate them into classes of varying severity. A spatially resolved NIR scanner was able to visualise resin features on the end of wooden discs representing the end of logs (Figure 4).

Further information: Detection of resin on log ends via RGB and NIR imaging, WQI report no. APP 76; Resin Detection: Pre-screening trial of potential technologies, WQI report no. APP 48 (contact: keith.mackie@wqi.co.nz).

Figure 4. A NIR scanner can reliably determine resinous areas on a disc even if it has been contaminated with an oily footprint, dirt, and a spray paint mark.

2.5. Durability

The issue: naturally durable timbers can have a high variability in their resistance to rot fungi. This makes it difficult to use them with confidence in challenging situations.

NIR opportunity: NIR could be used as a scan/segregation technology to distinguish between timber with high and low resistance to rot.
Equipment: a Bruker MPA spectrometer with a fibre optic probe was used to obtain several spot measurements on the radial-longitudinal face of redwood blocks (25 x 15 x 50 mm², heartwood). The blocks were also scanned with a hyperspectral camera (Specim, ImSpector N17E).

Outcome: NIR spectra were correlated against mass loss due to fungal decay. NIR models (from camera and benchtop instrument) were able to segregate the heartwood into values of high and low natural durability.


2.6. Stability

2.6.1 Drying stability

The issue: structural timber is cut into products in a green state and then dried. After drying, a certain amount of timber will exhibit distortion which makes it unsuitable to be sold as the intended product.

NIR opportunity: the susceptibility of lumber to distort could be detected before break-down of a log. In that manner, logs with a high likelihood to produce timber with distortion can be segregated and dried differently or cut into a different product.

Approach and Equipment: the end of a cant (cant = the biggest possible box that can be cut out of the round log) was sawn to produce nominal 24x24x300 mm coupons. Each coupon was numbered and could be mapped back to a position within the cant section. The transverse (end) faces of the coupons were scanned in the green state immediately after sawing using a CDI portable NIR spectrometer at a spectral resolution of ~2 nm. The spectral range was 1100 - 2200 nm. A single spectrum was taken from each transverse end of the coupons.

Longitudinal shrinkage of the individual coupons was measured using calipers. Coupon length was measured green and after oven drying at 60 °C to constant mass.

Outcome: NIR spectra were regressed against the shrinkage results of the coupons. Shrinkage severity (actual and predicted) was illustrated graphically using a colour scheme (Figure 5). Whilst it was not possible to use the predicted shrinkage data to estimate the distortion of individual boards recovered from the scanned cants, the predicted shrinkage information was sufficient to identify cants with a high likelihood of producing a high number of distorted boards.

Further information: NIR Log End Scanning, WQI report no. STA32 (contact: keith.mackie@wqi.co.nz).
Figure 5. Comparison of shrinkage maps based on measured and predicted data (white area is missing value). Values in the boxes are measured and predicted longitudinal shrinkage (%), respectively.
2.6.2 In-use stability

*The issue:* timber which is initially straight may exhibit dimensional instability when in use. This is the typical cause of sticking doors and windows.

*NIR opportunity:* the susceptibility of lumber to distort in-use could be detected before such lumber is used in an unsuitable application. There would also be the opportunity to sell timber with a stability “guarantee”.

*Approach and Equipment:* 4x2 lumber was scanned along its length on the radial as well as the tangential surface using a Foss 6500 instrument with a remote probe which was suspended above the lumber. The lumber was moved underneath the probe by a conveyor belt whilst scans were accumulated along its length (Figure 6).

The lumber was then exposed to cycles of humidity and temperature changes and the ensuing distortion was measured. NIR data (radial and tangential separately) from each piece of lumber was then regressed against the distortion data.

*Outcome:* there was no correlation between NIR data and distortion data. It is likely that the NIR scanning approach was flawed. Distortion is the result of a change in properties across a piece of lumber. By averaging across the whole width and length of a piece of lumber the necessary information for predicting distortion is likely to be lost.

*Figure 6.* Kiln-dried lumber is scanned along its length with a Foss 6500 NIR spectrometer to assess its susceptibility to distort in-use.
NIR IMPLEMENTATIONS FOR THE SOLID WOOD INDUSTRY

3. REFERENCES


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NIR Log End Scanning, WQI report no. STA32 (contact: keith.mackie@wqi.co.nz)

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NIR DETECTION OF TENSION WOOD IN E. GLOBULUS

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NIR DETECTION OF TENSION WOOD IN EUCALYPTUS GLOBULUS

Abstract: NIR predictions of cellulose content and stiffness (MOE) from spectra collected from the radial longitudinal surface of Eucalyptus globulus were found to be good indicators of non-recoverable collapse associated with tension wood formation. Tension wood (TW) is a key property limiting the diversification of E. globulus into a range of solid wood uses. Detection of TW is typically laborious and expensive, although some potential for using x-ray diffraction for detection has been identified previously.

Radial sections from 25 quarter-sawn boards cut from Spanish plantation-grown E. globulus trees were scanned to generate radial profiles of NIR-predicted wood properties at 1mm increments. These boards manifested a wide range of non-recoverable collapse, from no collapse to several severe collapse bands. The correspondence between the radial variation in NIR-predicted wood properties and collapse is demonstrated, and the potential commercial application of the approach discussed.

1. INTRODUCTION

This application of Near Infrared Spectroscopy (NIRS) to tension wood detection is based on two decades of research into the use of NIR for the assessment of commercially important wood properties in standing trees. The broad history of NIRS as a non-destructive evaluation (NDE) tool for assessing wood property variation in standing trees has been covered by Schimleck (2008) and Tsuchikawa (Tsuchikawa, 2007; Tsuchikawa and Schwanninger, 2011). Here, the intent is to briefly cover the development of NIRS that resulted in the opportunity for developing an application related specifically to the detection of tension wood in Eucalyptus globulus.

1.1 Development of a Commercial NIR Calibration of Kraft Pulp Yield

Once the proof-of-concept application of NIRS to the prediction of kraft pulp yield (KPY) and cellulose content (CC) in eucalypt woodmeal had been established, the need was then to develop a calibration to the point where it was commercially useful, and more important, trusted by industry. Downes et al. (2009, 2010, 2011) describe the development of larger multi-site and multi-species calibrations for these properties that have been through multiple iterations of development, validation and expansion. These calibrations have been utilised in a range of commercial and research applications (Freeman et al., 2009; Thumma et al., 2009; Southerton et al., 2010; Stackpole et al., 2010; Freeman et al., 2011; Freeman et al., 2013).
1.1 Radial Scanning (NIR)

The value of NIRS measurement of KPY lies not only in its speed and low cost, but in the small sample size required. The commercial measurement of KPY by traditional means requires up to 5 kg of oven-dry wood, and consequently destructive sampling. In contrast an NIRS measurement can be made on samples as small as 1gm. This then provides a basis for measuring the radial variation in KPY using increment cores samples through the dividing of the radius into smaller sections and grinding of these to woodmeal (Figure 1).

![Diagram of calibration process](image)

*Figure 1. Calibration data for the radial NIR scanner was generated using predicted values from woodmeal samples based on large, well-tested calibrations. A single radial strip was separated into 10 mm increments as shown and ground into woodmeal, which was then used for NIR spectra collection and laboratory cellulose content determination (Downes et al. 2010).*

The development of SilviScan in the early 1990s opened a new era in NDE of physical wood properties (Evans et al., 1995; Evans et al., 1996; Evans et al., 2000; Evans, 2006), providing a low-cost basis for obtaining detailed, high-resolution data on radial variation in physical wood properties such as density, microfibril angle (MFA) and stiffness. It had always been the intention to develop a radial-scanning NIR system as part of the suite of technologies called SilviScan. To that end numerous studies have shown that NIR models, utilising SilviScan data for calibration, can predict density, MFA, and wood stiffness (Schimleck et al., 2001; Schimleck and Evans, 2002a; Schimleck and Evans, 2002b; Schimleck et al., 2002a; Schimleck et al., 2002b). Thus NIR calibrations on intact increment core samples were shown to be feasible. A radial-scanning NIR (Meder et al. 2010) was used by Downes et al. (2010) to demonstrate that radial variation in KPY and cellulose
content could be predicted from surface scans on the longitudinal-radial face of radial wood samples of *E. nitens* at sampling intervals of 1 - 5 mm. This enabled radial profiles of these wood properties to be examined. The calibrations for this application were developed using the principle illustrated in Figure 1, i.e. the KPY values used as calibration data for spectra collected from the intact radial surface were produced by NIR predictions of the woodmeal obtained from the well-tested calibration described by Downes et al. (2011). This is the only current means of obtaining a KPY value from such a small sample. As a check that this approach gave reliable radial profiles of cellulose and KPY, actual laboratory diglyme cellulose contents (Wright and Wallis, 1998) were determined on the ground section samples and compared with cellulose content predicted from NIR surface scans. Cellulose content is highly correlated with KPY (Downes *et al.*, 2011).

The application was expanded to *E. globulus* (Downes *et al.*, 2012) in a study that examined radial variation in KPY for three contrasting sites and two thinning treatments. Radial increase in KPY from pith to bark was demonstrated (Figure 2), as well as the tendency for more productive, higher rainfall sites to have higher KPY. It was evident in some of these trees, as well as in other unpublished data, that some cores can exhibit peaks of high cellulose content, evident in tree 12 from the unthinned treatment at the Carpenters site (Figure 2).

### 1.2 Tension Wood

These developments created the opportunity to explore the application of NIRS to the detection of tension wood (TW) both in standing eucalypts and sawn timber. Tension wood is characterized by the presence of a thick, cellulose-rich, G-layer with low microfibril angle in affected wood fibres (Wardrop and Dadswell, 1955; Goswami *et al.*, 2008). Thus one would typically expect TW to exhibit high cellulose, wood density and MOE together with low MFA.

Tension wood is a major cause of sawn timber degrade in eucalypts, contributing to non-recoverable collapse and stability problems during sawing and timber drying (Washusen and Ilic, 2001) as well as quality (density) variations in the final products. Various options to establish a NDE method for quantifying the presence of TW in wood samples were evaluated (Washusen and Evans, 2001a; Washusen and Evans, 2001b; Washusen and Evans, 2002). The ability of SilviScan 2 to quantify cellulose crystallite width was used to assess the spatial distribution of TW in a single *E. globulus* stem (Washusen *et al.*, 2002), and as a broader-scale screening tool (Washusen *et al.*, 2005) to examine large numbers of trees. While successful in identifying TW occurrence, verified against histochemical methods, changes in the SilviScan methodology, such that cellulose crystallite width could no longer be determined, prevented further progress with this approach.
Figure 2. Radial patterns of predicted KPY in each BH radius for the six site-by-treatment combinations. In each plot the mean trajectory is shown (thick line). Radial trends are expressed as a percentage of the radius. Arrows indicate high cellulose peaks in tree 12 possibly indicative of tension wood occurrence (Downes et al., 2012).
The potential of radial-scanning NIR on air-dried core samples to identify zones of non-recoverable collapse (NRC) in increment cores taken from *E. globulus* trees and determine the degree of association between collapse bands and TW was evaluated by Wentzel-Vietheer et al. (2013). As well as a qualitative assessment, the objective was to identify quantitative thresholds for indicator wood properties, that could be subsequently evaluated as a means of identifying collapse-prone wood related to degrade in sawn timber. This study identified NIRS-predicted cellulose content and MOE as the best predictors of non-recoverable collapse, and these collapse zones were associated with the presence of tension wood, assessed by histochemical observations. This study of increment cores taken from over 170 trees successfully demonstrated the application of NIR to NRC prediction, and identified thresholds of predicted cellulose (>50%) and predicted MOE (>25 GPa) as indicators of NRC.

1.3 Collapse Detection Validation

The study reported here applied the radial NIR calibrations used by Wentzel-Vietheer et al. (2013), to the prediction of NRC in sawn boards of *Eucalyptus globulus* trees grown in Spain. The area of *E. globulus* plantations in the Iberian peninsula exceeds 1 million hectares, with the wood being mainly used for pulp production. Recently there has been a growing interest in diversifying into high quality solid wood products. The objective was to test the hypothesis that NRC in quarter-sawn boards could be detected by peaks of cellulose content and MOE predicted by NIR spectra collected from the air-dry radial, longitudinal surface prior to final drying and reconditioning.

2. METHODS

Quarter-sawn boards of *Eucalyptus globulus* from a commercial manufacturer, which exhibited a range of collapse features upon drying (Figure 3), were selected for study. These boards had not been heated above 60°C or steam reconditioned and were expected to display both recoverable and non-recoverable collapse. A 100 mm radial strip sized 10 mm in the tangential and longitudinal directions was cut from these boards by CIS-Madera and sent to the CRC Forestry Ltd. in Hobart, Tasmania for NIR evaluation. The collapse bands were not evident in the radial strips, enabling a blind test for the detection of collapse by NIR, although each strip could be precisely keyed to its position in the board, for subsequent matching of radial NIR and board shrinkage profiles.

2.1 Radial NIR Scanning

NIR spectra were obtained from both sides of each radial strip (50 scans at 2 mm sampling intervals) using a custom-built scanning system attached to a Bruker FT-NIR instrument (Downes *et al.*, 2010). Spectra were collected between 10,000 and 4,000 cm\(^{-1}\) with an optical resolution of 8 cm\(^{-1}\). Existing NIR calibrations
Figure 3. 25 radial strips were prepared from the above 25 boards which exhibited a range of collapse features. Circles and arrows indicate affected portions.
(Downes et al., 2012; Wentzel-Vietheer et al., 2013) were used to predict cellulose content and MOE for each 2mm interval using the Bruker QUANT procedure within the OPUS 5.5 software package (Bruker, 2005).

2.2 Data Processing and Analysis

Each radial profile of cellulose content and MOE was stored in a MYSQL database and accessed by custom procedures written in R (http://cran.r-project.org/). These scripts rescaled the radial profiles such that positions were expressed as a percentage of the total radius. The percentage of each radius that exceeded predicted MOE of 25 GPa and a predicted cellulose content of 50% was calculated. Radial patterns were compared to the cross sections of the sawn boards from which the samples were prepared and the association between cellulose content and MOE and collapse zones assessed visually.

Following NIR analysis all samples were reconditioned in a thermostatic bath by exposure to steam for two hours to determine whether collapse was recoverable.

3. RESULTS AND DISCUSSION

Of the 25 radii studied (Figure 3), 14 had regions where collapse was clearly evident. Many strips showed some degree of irregularity in the tangential width. Visually the zones of severe collapse could be associated with a darker colour, indicative of higher wood density.

Based on the NIR-predicted values of cellulose content and MOE (Figure 4), many samples had regions where cellulose content and MOE exceeded the prescribed threshold values (cellulose content >50% and MOE > 25 GPa) identified in a previous study (Wentzel-Vietheer et al., 2013). On this basis substantial non-recoverable collapse would be expected to occur in 6 of the samples (5, 8, 9, 10, 34, 39), while a further 7 might be expected to exhibit some NRC (3, 13, 14, 20, 21, 26, 27, 33). Of these only seven samples had greater than 5% of the total radial length affected.

These detected regions corresponded closely with the visually observable regions of collapse in the sampled boards. Figure 5 illustrates the synchronicity of excessive collapse and the corresponding wood properties in two affected samples. However, the presence of collapse in samples such as 4, 6 and 18 was not associated with particularly high cellulose or MOE. Subsequent reconditioning of the samples indicated that the collapse in these samples recovered (Figure 6).
Figure 4. The radial profiles of the 25 radial strips are shown for cellulose content (red) and MOE (blue). Both sides of each strip were scanned and indicated as “A” and “B” profiles. The dotted horizontal line indicates the threshold identified for tension wood. When both series exceed this line for more than 3 consecutive millimetres, non-recoverable collapse is predicted to be present.
Figure 5. (a) Sample 10 exhibited wide bands of collapse along with the approximate location of the sample prepared for NIR radial scanning (blue box). (b) Sample 20 also showed collapse but in a narrower band. The horizontal dashed line shows the threshold values for cellulose and MOE.

Figure 6. Samples that did not show NIR-predicted evidence of non-recoverable collapse, indicated by high cellulose and MOE, generally recovered collapse upon steaming (top). This is in contrast to those that did show NIR-predicted evidence of NRC (bottom).
4. CONCLUSIONS

Radial scanning predictions of cellulose content and MOE based on NIR scanning of the radial-longitudinal face were successfully used to identify zones of non-recoverable collapse in *Eucalyptus globulus* boards. Values of cellulose content and MOE in excess of identified thresholds indicated zones that exhibited collapse that did not recover with steam reconditioning.

The application of this approach to non-destructive evaluation of radial wood samples may be a useful way of screening for the presence and severity of non-recoverable collapse. The method can be applied to radial increment core samples, enabling non-destructive assessment of NRC in standing trees. This raises the prospect of investigating site, silviculture and genetic effects on the severity of NRC occurrence in *Eucalyptus globulus*. Appropriate sampling strategies need to be developed to provide accurate and quantitative assessments of the levels of NRC that would adversely affect commercial processing.

5. ACKNOWLEDGEMENTS

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NIR DETECTION OF TENSION WOOD IN _E. GLOBULUS_


47


6. AFFILIATIONS

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Abstract The feasibility of placing a NIR system into a wood grader has been demonstrated where NIR is used to grade planed boards for moisture content and modulus of elasticity. The predictive models for both wood characteristics achieved sufficient prediction accuracy.

1. INTRODUCTION

Near infrared (NIR) spectroscopy, which is based on the absorption within the 800 – 2500 nm range, representing overtones and combinations of fundamental molecular vibrations, has been applied for the non-destructive estimation of various properties of organic material. NIR has a potential to predict not only chemical properties, but also mechanical properties of wood, where there are relationships between mechanical properties and chemical components (Tsuchikawa and Schwanninger 2013).

Considering the practical use of NIR to predict lumber properties, it is necessary to acquire spectra from the surface of wood while the lumber passes under the detector at high speed. We investigated the prediction of modulus of elasticity, bending strength, density and moisture content by FT-NIR spectrophotometry with spectra obtained from moving surfaces. NIR spectra collected from wood moving at a speed of 10 m/min were able to predict wood properties with good accuracy (Fujimoto et al. 2010a, 2010b). However, an increase of the speed of NIR spectral acquisition is required for the practical use in sawmills.

We recently designed a NIR spectrophotometer with a linear image sensor for high speed acquisition (120 m/min) of the NIR spectra form moving wood (JP 2011-17565). The accuracy predicting moisture content (MC) and modulus of elasticity (MOE) with this machine was investigated.

2. METHODS

2.1. Design of NIR spectrophotometer

We designed a dispersive type spectrophotometer to measure NIR spectra from wood surfaces passing with high speed under the detector on a belt conveyor (Figure 1 and 2). An InGaAs linear image sensor with a grating unit was used to detect the reflectance spectra. Halogen lamps with collective lenses were arranged circularly.
The illuminating area of wood surface was approximately 40 mm in diameter. The detector was placed in the centre of the circularly arranged halogen lamps.

Figure 1. NIR spectrophotometer based in an InGaAs linear image sensor with a grating unit able to acquire spectra in the range from 872 nm to 1718 nm. Halogen lamps with collective lenses were arranged circularly around the detector. The illuminating area of wood surface was approximately 40 mm in diameter.

Figure 2. On-line NIR measurement system mounted over a conveyor belt moving at 120 m/min.
2.2. **NIR spectral acquisition**

Diffuse-reflectance spectra of radial-tangential surfaces were acquired ranging from 872 nm to 1718 nm at 1 nm resolution. The spectrophotometer was mounted over a flat belt conveyor which was running at a speed of 120 m/min. The fixed exposure time was 5 msec and 10 scans were averaged into a single spectrum. As the sample was transported under the spectrophotometer during the measurement these average spectra represented a larger area of the sample. 2 averaged spectra were automatically acquired on each 1000 mm long sample. Totally 306 averaged spectra were collected.

2.3. **Sample, MC, and MOE**

A total of 20 Hinoki (*Chamaecyparis obtusa*) boards with dimensions of 26 (radial) × 114 (tangential) × 1000 (longitudinal) mm were prepared from planned boards. All samples were fully saturated by water-impregnation for 3 weeks subsequently dried at ambient temperature (25-30°C) until their moisture content reached 25%. Then a set of weight measurement (MC), NIR spectra acquisition and bending tests (MOE) were repeatedly performed on a daily basis during further air-drying at ambient temperature until the samples reached the equilibrium moisture content of ~12%.

3. **RESULTS**

Our previous machine was able to acquire spectra within 5 sec what equates to 700 mm in length at a moving speed of 10 m/min. The new machine is 12 times faster and has a speed which is sufficient to be introduced into a sawn mill. The cross validated result of PLS predictive models for MC and MOE are summarised in Figure 3. The spectra were converted into the 2nd derivative before being used in the PLS analysis. Numbers of latent variables used were 5 for MC and 3 for MOE, respectively. The standard error of prediction (SEP) for assessing MC was 0.63%. This is of similar accuracy to previous investigations on *Larix kaempferi* (Fujimoto et al. 2010a) and the present study benefited from a wider range of MC values. The accuracy of the model for MC showed sufficient prediction accuracy to be useful in a commercial setting, as indicated by the ratio of the standard error of prediction to the sample standard deviation (RPD).

NIR-spectra obtained from wood surface moving at a speed of 120 m/min were able to predict MOE with a SEP of 0.89 GPa. This model was of slightly better quality as those obtained in earlier studies with *Larix kaempferi* (Fujimoto et al. 2010a, b). However, the predictive model for MOE was less accurate than that for MC as indicated by RPD but still adequate for initial screening purposes. It needs to be kept in mind that a reduction in MC below fiber saturation point causes and
increase in MOE. Therefore the range of MOE values used for generating the MOE model in this study is larger than the range of MOE at a given MC. Therefore, due to this confounding effect it is possible that the MOE model accuracy might be too optimistic. However, this effect is negligible as the variation of MOE between samples by far exceeded variation for one sample at different MC. Furthermore timber is used at air dry conditions and as equilibrium MC is variable between samples air-dry MOE is affected likewise.

![Figure 3. Accuracy of PLS calibration for MC and MOE obtained from NIR spectra measured on tangential-longitudinal surfaces moving at 120 m/min for hinoki (Chamaecyparis obtusa).](image)

5. ACKNOWLEDGEMENT

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7. REFERENCES
NATURAL DURABILITY OF SEQUOIA SEMPERVIRENS HEARTWOOD

NICHOLAS DAVIES AND CLEMENS ALTANER

NATURAL DURABILITY OF REDWOOD (SEQUOIA SEMPERVIRENS) HEARTWOOD

Prediction by IR spectroscopy

Abstract
Mid-range and near infrared spectroscopy were used to predict the extractive content and the fungicidal activity of these extractives as indicators of the natural durability of redwood (Sequoia sempervirens) heartwood. Spectra of solid and milled wood were taken at 0% (dry) and ~12% MC. The samples were extracted using the solvents ethanol and acetone and the extractives were added to an agar solution. The white rot fungus Trametes versicolor was grown on the agar containing the extract and the diameters of the colonies were measured daily for one week. A limited number of wood samples were available but key regions of the spectra which are most significant for predicting natural durability in Sequoia sempervirens could be identified. Spectra obtained from milled samples provided the highest correlations, whereas spectra taken from a radial-tangential or radial-longitudinal surface performed best without milling. Better predictions of the extractive contents were obtained with milled samples at ~12% MC than with dry samples.

1. INTRODUCTION

Redwood (Sequoia sempervirens (D. Don) Endl.) heartwood is classed as being naturally durable (AS5604:2005). However, large variation in durability has been reported both between (Jones et al. 2011) and within trees (Clark and Scheffer 1983), ranging from very durable to moderately/non-durable. As natural durability is a key characteristic of the products it is used for (Cornell 2002), a timber supply with a consistently high natural durability is required to meet the customers’ demands. This can be achieved by grading the timber according to its natural durability and/or by improving the germplasm used in plantations (Cown 2008). Both require a reliable, quick, and cheap way of assessing natural durability, although the demand for the methodology to be quick and cost effective is greater when grading timber in a sawmilling operation.

Natural durability is commonly measured by field or laboratory tests (AWPC 2007). Field tests, especially for durable timber, require decades. A representative sample is exposed to in-ground test conditions and the failure of individual samples is assessed periodically until a defined fraction has failed. In laboratory tests small samples are placed on a highly active wood decay fungus in controlled conditions which are optimised for fungal growth. Natural durability is assessed as the mass lost after several weeks of incubation. Many variations of these methods are used to assess the natural durability of timber but they all suffer from being destructive, highly variable and time consuming, whereas in a sawmilling operation the results need to be non-destructive, repeatable and instantaneous.
Natural durability of timber is largely due to the wood extractives, which are compounds of low molecular weight that are deposited into the wood during heartwood formation (Hillis 1987; Taylor et al. 2002). Heartwood extractives comprise numerous organic compounds of which some possess good fungicidal, bactericidal or insecticidal properties (Rowe 1989). As the activities of the individual compounds differ greatly, it is not only the quantity of total extractives but also the composition of the extractives, i.e. the relative abundance of individual compounds, that controls natural durability. Both quantity and composition of extractives are variable both between and within trees (Anderson 1961; Bito et al. 2011).

Infrared spectroscopy (IR) provides information on the chemical makeup of a sample. It is non-destructive and can give results within fractions of a second. The challenge is to obtain quantitative information from the semi-quantitative IR spectra. This is commonly achieved by chemometrics – the building of predictive multivariate statistic models from an extensive calibration set and this has been used for numerous wood properties, directly or indirectly related to its chemical composition (Tsuchikawa 2007; Tsuchikawa and Schwanninger 2013). Heartwood extracts are chemically distinct from wood cell wall polymers, which makes it easier to predict the total quantity of extracts by infrared spectroscopy. However, as IR spectroscopy measures functional groups, it cannot resolve individual extractive compounds, as is possible by chromatography. It is worth noting that chromatographic methods are rather time and resource consuming. Furthermore they can only assess a fraction of the extracts and therefore do not assess the entirety of the compounds present. Mid-range IR has been used to assess the relative proportions of particular extractive classes, i.e. extracts of similar chemical structure, in different wood samples (Caron et al. 2013).

Near-IR has been used to predict the amounts of extracts and the natural durability of wood (e.g. Michell and Schimleck 1996; Gierlinger et al. 2003; 2004; Lestander and Samuelsson 2010; Bush et al. 2011; Jones et al. 2011; Taylor et al. 2011). Although the amounts of extracts can usually be assessed reliably, the prediction of natural durability is associated with more uncertainty (Bush et al. 2011). The following factors contribute to the larger error in the prediction of natural durability by NIR:

- Physical factors (like density or permeability), which are not necessarily reflected in the spectra, can influence natural durability (Taylor et al. 2002).
- Minor differences in the molecular structures of individual extractive compounds can have huge impacts on their biological activity (Balogh and Anderson 1965; Bito et al. 2011) but are not necessarily resolved in the spectra.
- Field and laboratory tests which are used to measure natural durability suffer from noise and rely on numerous replicates (Råberg et al. 2005). As natural durability data on individual pieces of wood are needed for the calibration of NIR the error in the calibration data persists in the chemometric models.
Natural Durability of *Sequoia sempervirens* Heartwood

- Mass loss and extractions are usually performed on samples of larger size than IR measurements. Therefore small scale variations in the extractive contents of solid wood are not necessarily represented in the calibration data.

2. METHODS

2.1. Material

Heartwood from 21 *Sequoia sempervirens* trees was milled dry to pass a 2 mm mesh. *Trametes versicolor* (L.) Lloyd (ICMP 18215) was obtained from the International Collection of Micro-organisms from Plants (Landcare Research, New Zealand). Fungal growth was assessed using a base of malt extract agar (Merck) containing 30.0 g/l malt extract, 3.0 g/l soymeal peptone and 15 g/l agar-agar.

2.2. Extractions

The dry milled wood samples were extracted either with ethanol or acetone in an Accelerated Solvent Extractor (ASE350 / Thermo Scientific) in 33 ml cells under the following settings: 2 cycles; 70 °C; 15 min static time; 50 % rinse volume. Extractive content was measured gravimetrically with an aliquot of the extracts after drying at 105 °C.

2.3. Fungal assay

Agar was prepared containing 4.8 % w/v malt agar and extract solution from the equivalent of 4.8 % [w/v] dry wood. Solvents containing the extractives were evaporated in a hot water bath until no solvent odour was noticeable. Evaporation losses were compensated with distilled water, never allowing the extracts to become dry. Agar was added, autoclaved (121 °C, 10 min) and poured into 50 mm diameter Petri dishes. Controls containing the equivalent amount of solvent or water only underwent the same procedures as the agars containing wood extracts. A small fungal inoculum was placed at the centre of the agar and grown at 25 °C. Two perpendicular diameters of the fungal colony were measured using callipers approximately every ~24 h over the course of a week. The absolute growth rate (mm/h) was calculated by fitting a linear regression for the averaged diameter against time for each Petri dish. Five replicates for each extract were assessed.

2.4. IR spectroscopy

Six NIR spectra were recorded with an integrating sphere from a radial-tangential, radial-longitudinal and tangential-longitudinal face of solid wood blocks each. Each NIR spectrum was obtained from 32 scans in the 12000-4000 cm⁻¹ range at 16 cm⁻¹ resolution.
Near and mid-range IR spectra of milled unextracted wood were taken in duplicate using an integrating sphere or an ATR sampling accessory, respectively. Mid-range IR spectra were obtained from 32 scans in the 4000-400 cm\(^{-1}\) range at 4 cm\(^{-1}\) resolution. NIR spectra were obtained with the settings described above.

The replicate spectra were averaged as no qualitative difference between the replicates could be found. NIR spectra were measured at 0% MC (dry) after storage in a desiccator over dry silica gel and ~12% MC after equilibration in the laboratory atmosphere.

2.5. Chemometrics

Data analysis was performed using R (R Core Team 2013). Models were calculated from area normalised spectra using the ‘plsr’ function with ‘leave-one-out’ validation of the ‘pls’ package (Mevik and Wehrens 2007). As the fungal growth decreased with increasing extract content the fungicidal activity was expressed as a negative growth rate.

3. RESULTS

We have characterised heartwood samples from 21 *Sequoia sempervirens* trees for their ethanol and acetone extractives content (EC) as well as the fungicidal activity of the acetone extract against the white-rot fungus *Trametes versicolor* on a wood mass basis (Table 1). Before extracting the samples NIR spectra on the 3 principal faces of the solid samples as well as NIR and mid-range ATR spectra of the powdered wood were obtained.

<table>
<thead>
<tr>
<th>Ethanol EC (%)</th>
<th>Acetone EC (%)</th>
<th>Acetone TV (mm/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>5.4</td>
<td>1.4</td>
</tr>
<tr>
<td>Max</td>
<td>12.8</td>
<td>5.1</td>
</tr>
<tr>
<td>Mean</td>
<td>8.9</td>
<td>2.6</td>
</tr>
</tbody>
</table>

3.1. IR spectra

Figure 1 shows the averaged mid-range ATR spectra for the dry *S. sempervirens* heartwood samples, the variance between them and the correlation of the signal at the individual wavenumbers to the extractable matter as well as the fungicidal activity of the acetone extract against the *T. versicolor*. The similarity of the
3 correlation spectra indicted that the amounts of extracts were closely linked to the fungicidal activities and could serve as an indication of natural durability. This similarity also indicated that the ethanol and acetone soluble compounds are of similar chemical structure.

Figure 1. Mid-range ATR IR spectra of 21 S. sempervirens milled heartwood samples. Average spectra (Mean), variance between the samples (Var) and the correlation of each wavenumber to ethanol (Corr EC ethanol) and acetone (Corr EC acetone) extractable matter as well as the fungicidal activity of the acetone extract against T. versicolor on a wood mass basis (Corr TV acetone). Top: OH and CH region; bottom: fingerprint region.
Wavenumbers with positive correlation with the extractive content were associated with aromatic structures (aromatic skeletal vibrations at 1600 cm\(^{-1}\) and 1510 cm\(^{-1}\)), whereas negative correlations were observed for polysaccharide-associated wavenumbers (e.g. OH-stretching at 3500-3200 cm\(^{-1}\) and in the fingerprint region at 1150-970 cm\(^{-1}\)) (Schwanninger et al. 2004). The strong positive correlation with the extractive content in the region between 3100 and 3000 cm\(^{-1}\) could have arisen from baseline variations; however aromatic CH stretching signals are present here. It is worth noting that the positive correlations in the C=O region at 1760 cm\(^{-1}\) and 1700 cm\(^{-1}\) could correspond to signals of lignans, with the former assigned to \(\gamma\)-lactone structures (Willför et al. 2004). Certain norlignans have been associated with the natural durability of \textit{S. sempervirens} (Balogh and Anderson 1965) and the related species \textit{Cryptomeria japonica} (Bito et al. 2011); however these are lacking \(\gamma\)-lactone structures.

Figure 2 shows the spectra of milled \textit{S. sempervirens} heartwoods at 0 and 12% moisture content (MC) in NIR region by analogy with Figure 1. Again signals associated with aromatic structures (e.g. ~6000 cm\(^{-1}\) and ~4650 cm\(^{-1}\)) (Michell and Schimleck 1996; Schwanninger at al. 2011) showed a positive correlation with the extractive contents and fungicidal activities of the samples. These signals were also present in the loadings of PLS models developed to assess mass loss of \textit{S. sempervirens} heartwood against \textit{Coniophora puteana} (Jones et al. 2011).

The well-described water signals at ~5200 cm\(^{-1}\) and ~7000 cm\(^{-1}\) were prominent in the spectra taken from the samples at ~12% MC. The correlations with the investigated sample properties followed a similar pattern at 0% and ~12% MC, with the latter generally less strong and apparently more variable.

NIR spectra taken from the 3 principal planes of the solid wood samples were similar to those of the milled wood but correlations were generally weaker, particularly when using the tangential-longitudinal face (data not shown). This may indicate that radial variability in extractive content occurred within the small wooden blocks, which was captured when examining the transverse or radial-longitudinal but not the tangential-longitudinal faces.

### 3.2. Chemometric models

The limited number of samples made it impractical to split the data into a calibration and validation dataset. Therefore the models are not usable for a quantitative prediction of \textit{S. sempervirens} heartwood properties. The different models however give an indication of the influence of the sample form and relevant spectral regions on the predictability of extractive content in \textit{S. sempervirens} heartwood and fungicidal activity of the acetone extract against \textit{T. versicolour}. The accuracy of the models used to predict natural durability-related wood properties of \textit{S. sempervirens} heartwood was generally not high (around 50 to 100% of the standard deviation) (Table 2). This quality of prediction was similar to that of models developed to predict mass loss of \textit{S. sempervirens} by \textit{C. puteana} (Jones et al. 2011).
Figure 2. Average NIR spectra of 21 S. sempervirens milled heartwood samples. Average spectra (Mean), variance between the samples (Var) and the correlation of each wavenumber to ethanol (Corr EC ethanol) and acetone (Corr EC acetone) extractable matter as well as the fungicidal activity of the acetone extract against T. versicolor on a wood mass basis (Corr TV acetone). Top: 0% MC; bottom: ~12% MC.
### Table 2. Summary of models using IR spectra to predict extractive ethanol (Ethanol EC) and acetone (Acetone EC) soluble matter as well as the fungicidal activity of the acetone extract against *T. versicolor* on a wood mass basis (Acetone TV). Values are given for models built from spectra obtained from samples at 0% and 12% MC. Mean (Σ) and standard deviation (STD) of the 21 samples are given. RMSEP: residual mean square error of prediction; NC: number of components.

<table>
<thead>
<tr>
<th>MC: 0%/-12%</th>
<th>Ethanol EC (%)</th>
<th>Acetone EC (%)</th>
<th>Acetone TV (mm/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Σ</td>
<td>8.9</td>
<td>2.6</td>
<td>0.420</td>
</tr>
<tr>
<td>STD</td>
<td>2.3</td>
<td>1.1</td>
<td>0.067</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample form</th>
<th>Wavenumbers (cm⁻¹)</th>
<th>RMSEP</th>
<th>NC</th>
<th>RMSEP</th>
<th>NC</th>
<th>RMSEP</th>
<th>NC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>4000-400</td>
<td>1.3</td>
<td>3</td>
<td>0.56</td>
<td>3</td>
<td>0.035</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>3130-2995</td>
<td>1.0</td>
<td>2</td>
<td>0.59</td>
<td>3</td>
<td>0.032</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>1664-1487</td>
<td>1.7</td>
<td>4</td>
<td>0.63</td>
<td>6</td>
<td>0.027</td>
<td>4</td>
</tr>
<tr>
<td>Powder</td>
<td>12000-4000</td>
<td>1.7/1.2</td>
<td>4/4</td>
<td>0.75/0.70</td>
<td>6/4</td>
<td>0.034/0.039</td>
<td>4/5</td>
</tr>
<tr>
<td></td>
<td>8000-6000</td>
<td>1.3/1.0</td>
<td>3/4</td>
<td>0.57/0.56</td>
<td>6/5</td>
<td>0.029/0.032</td>
<td>4/4</td>
</tr>
<tr>
<td></td>
<td>6171-5978</td>
<td>1.5/1.5</td>
<td>5/5</td>
<td>0.58/0.56</td>
<td>5/4</td>
<td>0.030/0.032</td>
<td>5/4</td>
</tr>
<tr>
<td>Solid - transverse</td>
<td>12000-4000</td>
<td>1.6/2.1</td>
<td>6/4</td>
<td>0.95/0.91</td>
<td>2/4</td>
<td>0.043/0.036</td>
<td>5/5</td>
</tr>
<tr>
<td></td>
<td>8000-6000</td>
<td>1.5/2.4</td>
<td>4/0</td>
<td>0.85/0.84</td>
<td>5/5</td>
<td>0.038/0.049</td>
<td>5/3</td>
</tr>
<tr>
<td></td>
<td>6171-5978</td>
<td>1.7/2.3</td>
<td>6/4</td>
<td>0.72/0.65</td>
<td>3/4</td>
<td>0.036/0.050</td>
<td>5/3</td>
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<tr>
<td>Solid - radial</td>
<td>12000-4000</td>
<td>2.1/1.5</td>
<td>2/2</td>
<td>1.03/0.87</td>
<td>2/3</td>
<td>0.059/0.045</td>
<td>2/3</td>
</tr>
<tr>
<td></td>
<td>8000-6000</td>
<td>1.7/1.7</td>
<td>3/2</td>
<td>1.00/0.92</td>
<td>3/5</td>
<td>0.044/0.045</td>
<td>4/3</td>
</tr>
<tr>
<td></td>
<td>6171-5978</td>
<td>1.6/1.6</td>
<td>5/3</td>
<td>0.89/0.85</td>
<td>4/3</td>
<td>0.042/0.059</td>
<td>5/3</td>
</tr>
<tr>
<td>Solid - tangential</td>
<td>12000-4000</td>
<td>2.4/2.3</td>
<td>2/2</td>
<td>1.09/0.93</td>
<td>5/4</td>
<td>0.069/0.053</td>
<td>0/4</td>
</tr>
<tr>
<td></td>
<td>8000-6000</td>
<td>2.2/2.4</td>
<td>3/0</td>
<td>0.94/0.94</td>
<td>3/3</td>
<td>0.065/0.056</td>
<td>3/3</td>
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<tr>
<td></td>
<td>6171-5978</td>
<td>1.8/2.4</td>
<td>5/0</td>
<td>0.96/0.93</td>
<td>5/5</td>
<td>0.053/0.057</td>
<td>5/3</td>
</tr>
</tbody>
</table>

NIR spectra from milled samples yielded better models than those from solid samples. This may indicate considerable variation in extractive content in the small wood blocks, which was best captured by milling. Other possibilities such as the surface preparation of solid samples may also have an effect on the spectra. Among the models built from solid wood NIR spectra, those including the radial direction (i.e. transverse or radial-longitudinal faces) were superior. This may indicate that most variation in the extractive content was present in the radial direction. Radial variation in wood properties, extractive content and natural durability is well described but usually on a scale of centimetres rather than millimetres, as indicated here (Sherrard and Kurth 1933, Clark and Scheffer 1983, Seehann 1984). Millimetre
scale variation in extractive content is apparent in the streaky appearance of heartwood from species such as *Microberlinia brazzavillensis* (zebrawood) or *Dacrydium cupressinum* (rimu). In agreement with the present study Schimleck et al. (2005) reported that there was no general conclusion as to whether NIR calibrations for radial-longitudinal or transverse faces are superior, but rather that this depended on the wood characteristic calibrated for.

An influence of moisture on the model quality was observable when wider spectra regions were used, whereas when focusing on a narrow region around 6000 cm\(^{-1}\), which is essentially free of water signals, no influence was noticed. The better quality of models obtained using a wider spectral range for the milled samples at equilibrium moisture content in the laboratory atmosphere indicated that the extractives influenced the equilibrium moisture content.

Most information in the NIR spectra on the extractive content in *S. sempervirens* heartwood was present around 6000 cm\(^{-1}\). Models based on the 6171-5978 cm\(^{-1}\) spectral region were often more accurate than those using a wider spectral range. Signals around 6000 cm\(^{-1}\) were found to be characteristic of aromatic extractives (Michell and Schimleck 1996). A narrower spectral range has the advantage of faster measurements and the potential use of cheaper equipment. However, a larger data set than that analysed here would be needed.

Mid-range ATR IR spectra of milled samples gave more accurate models than NIR (Table 2). As the sampling is more cumbersome compared to NIR, it is not practical to use this in an industrial environment but it might be used in a breeding programme or to establish larger calibration data sets for NIR.

4. AFFILIATIONS

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5. REFERENCES


