Calibrating NIR spectroscopy for extractive content of *E. bosistoana* stem cores

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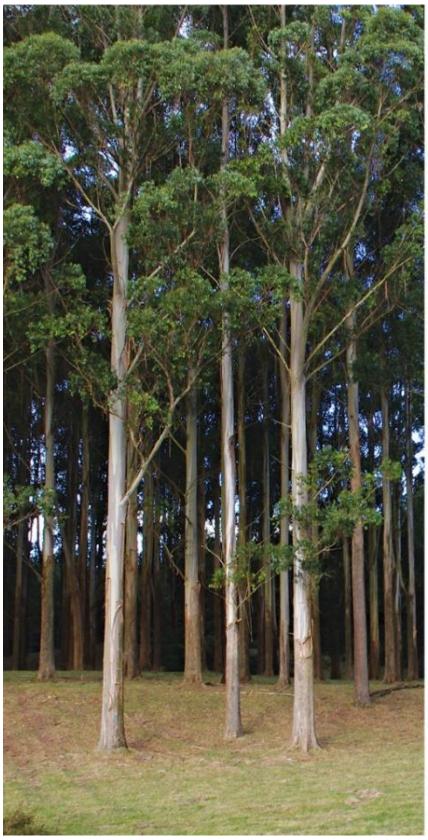

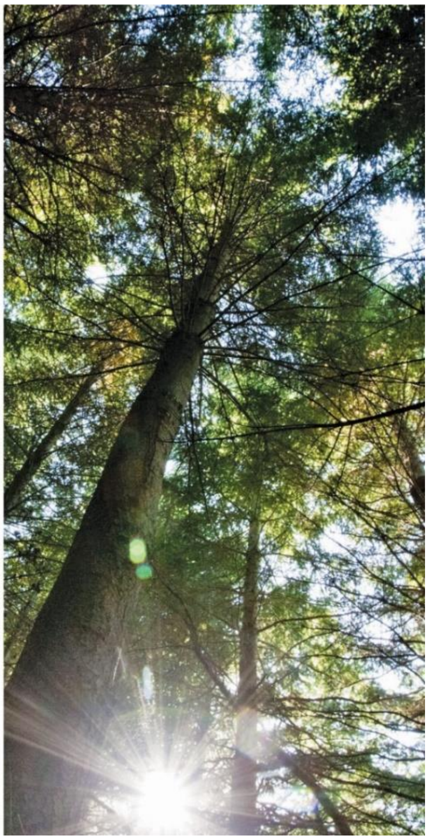

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INTRODUCTION

The New Zealand Dryland Forests Initiative (NZDFI) is working to establish a timber industry based on durable eucalypts (www.nzdfi.org.nz). A central part of the current work is a selection/breeding programme to ensure healthy trees with good wood properties. A key property for the produced timber is natural durability. Only heartwood can be naturally durable. Extractives (numerous smaller organic molecules) deposited by the tree in the heartwood are the main factor inferring natural durability.

A meaningful selection/breeding programme requires the assessment of many (i.e. thousands) of individuals. The time and labour requirements of standard durability tests based on fungal decay prohibit their use in a lean breeding programme. As the heartwood extractives are inferring natural durability, the extractive content offers a proxy measurement for natural durability. Heartwood with higher extractive content is likely to be more durable than heartwood with lower extractive content.

The chemical composition of a material (i.e. wood) can be assessed with infrared spectroscopy. Near Infrared (NIR) spectroscopy is used in the agricultural sector to determine product quality i.e. composition. NIR spectroscopy has the advantage of quick measurements (seconds) and little demands on sample preparation, i.e. it is possible to obtain spectra from solid samples. However, NIR spectroscopy is characterised by limited signal resolution (as it assesses vibrational overtones) and a strong influence of moisture. Furthermore it has been reported that the grain angle of solid wood is affecting NIR spectra (e.g. Schimleck et al. 2003).

The NZDFI breeding populations will be screened for heartwood. This requires extracting a (14 mm diameter) core from the trees and assessing heartwood quantity and quality. Heartwood quality is envisaged to be assessed by predicting extractive content from NIR spectra of the cores. However, the grain orientation is not always clearly defined on a cylindrical core and cores not necessarily pass through the pith, sometimes resulting in cores having heartwood only on the radial-longitudinal face.

The effect of moisture on NIR spectra can be mitigated by appropriate sample equilibration.

This study investigated the effect of grain angle on NIR spectra obtained from *E. bosistoana* cores.

Objective 1

Is the grain angle affecting extractive content assessments by NIR?

Objective 2

Can NIR be used to predict the grain angle on *E. bosistoana* cores?

METHODS

Grain angle

8 cores extracted from 7-year old *E. bosistoana* trees with heartwood were equilibrated to air dry conditions before taking NIR measurements. NIR spectra were collected with a Bruker Tensor 37 fitted with a fibre optics probe along the core in 5 mm intervals at 0, 45, 90, 135, 180, 225, 270, 315° grain angles (Figure 1). Data was recorded from 9000 to 4000 cm^{-1} averaging 32 scans. A total of 264 spectra on the 8 cores were acquired.

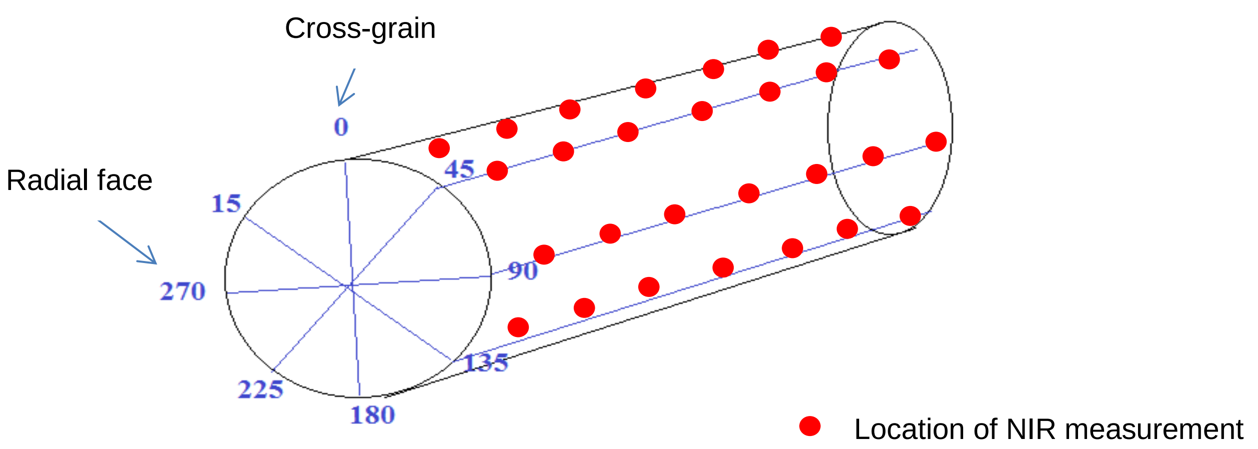

Figure 1: Sampling strategy on the cores.

Extractive calibration

Heartwood from 127 *E. bosistoana* trees of varying age was milled in a Wiley mill fitted with a 2 mm screen. NIR spectra were collected from these powders after equilibrating to air dry conditions. Then the ethanol soluble content (referred to as extractive content (EC)) of these powders was determined gravimetrically after extraction with a Thermo ASE (15 min static time, 70 °C, 100% rinse volume; 2 extraction cycles). EC for the samples was ranging between 0.96% and 14.92% with an average of 6.49%.

The 1st derivatives of the NIR spectra of powders were calculated using the Savitzky-Golay algorithm with a 2nd order polynomial and a window size of 15 before further analysis.

The software R was used for data analysis.

RESULTS

Figure 2 shows the NIR spectra obtained on different grain angles along 8 *E. bosistoana* cores. Some spectral regions are influenced more by the grain angle than others. For example a stronger influence of grain angle can be seen for the signals ~5100 cm^{-1} and ~4000 cm^{-1}. The effect of grain angle was less pronounced in the 1st derivative spectra (Figure 3).

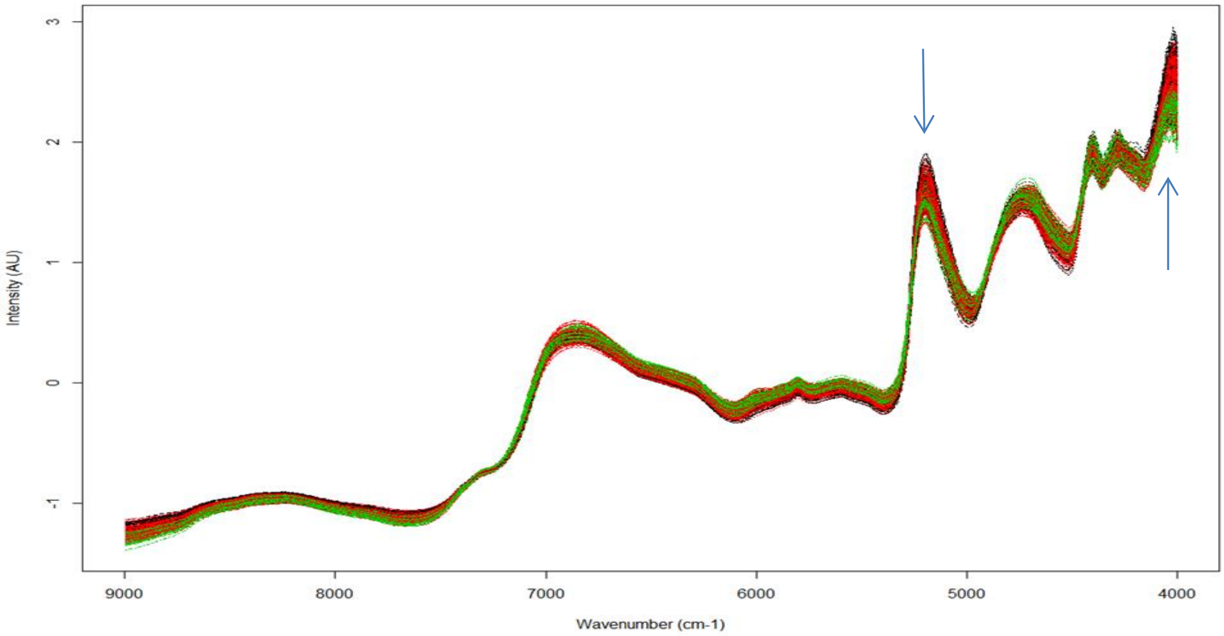

Figure 2: Full SNV normalised NIR spectra from *E. bosistoana* cores. Black: cross-section (0° and 180°); green: radial face (90° and 270°); red: 45° grain angle (45°, 135°, 225° and 315°).

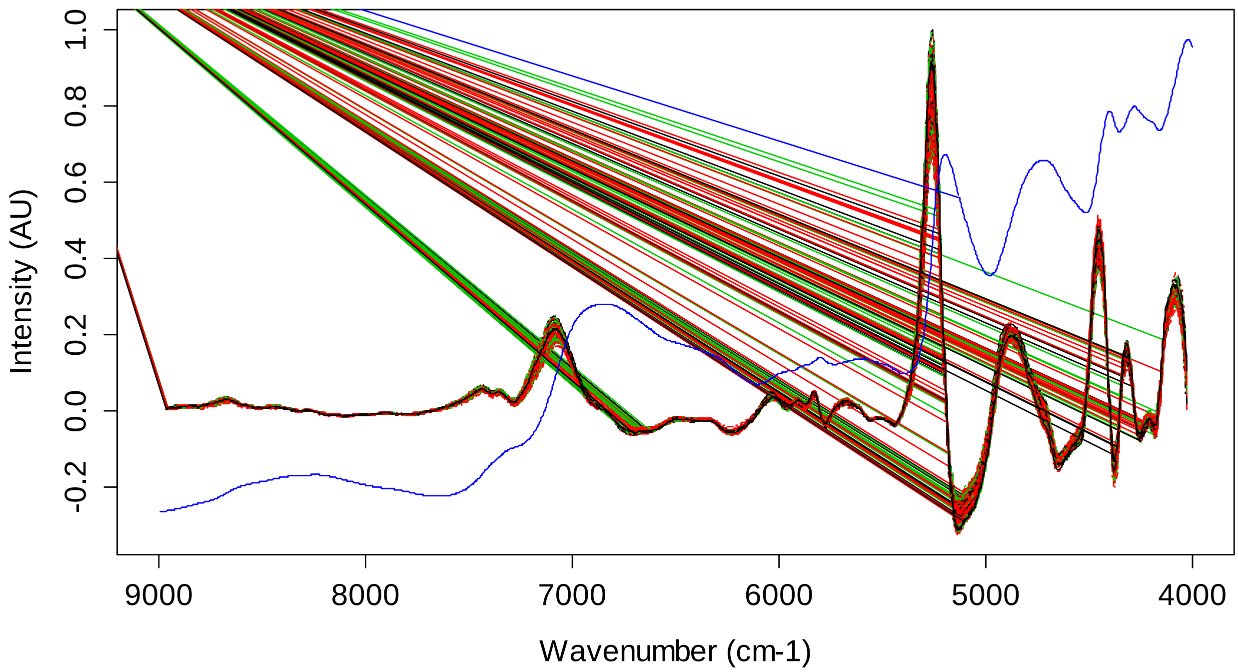

Figure 3: 1st derivative spectra from *E. bosistoana* cores. Black: cross-section (0° and 180°); green: radial face (90° and 270°); red: 45° grain angle (45°, 135°, 225° and 315°). Blue: average full SNV normalised NIR spectra.

Objective 1

Figure 4 shows the residual standard error (RSE) of EC prediction of the *E. bosistoana* heartwood spectra (black) for each wavenumber using the calibration data. Figure 4 also shows the p significance value of grain angle using this wavenumber to predict the EC on the cores (red). A low RSE indicates a good prediction of EC while a high p significance value indicates that this wavenumber is independent of the grain angle. The wavenumber predicting EC most accurately was 4528 cm^{-1} with a RSE of 1.18%. However, the effect of taking spectra on the cross or radial face of the cores was slightly significant ($p = 0.049$). The wavenumber predicting EC with the lowest RSE (1.28%) and showing no influence of grain orientation ($p = 0.428$) was found to be 4516 cm^{-1} (grey). Figure 5 shows the calibration for the 4516 cm^{-1} signal.

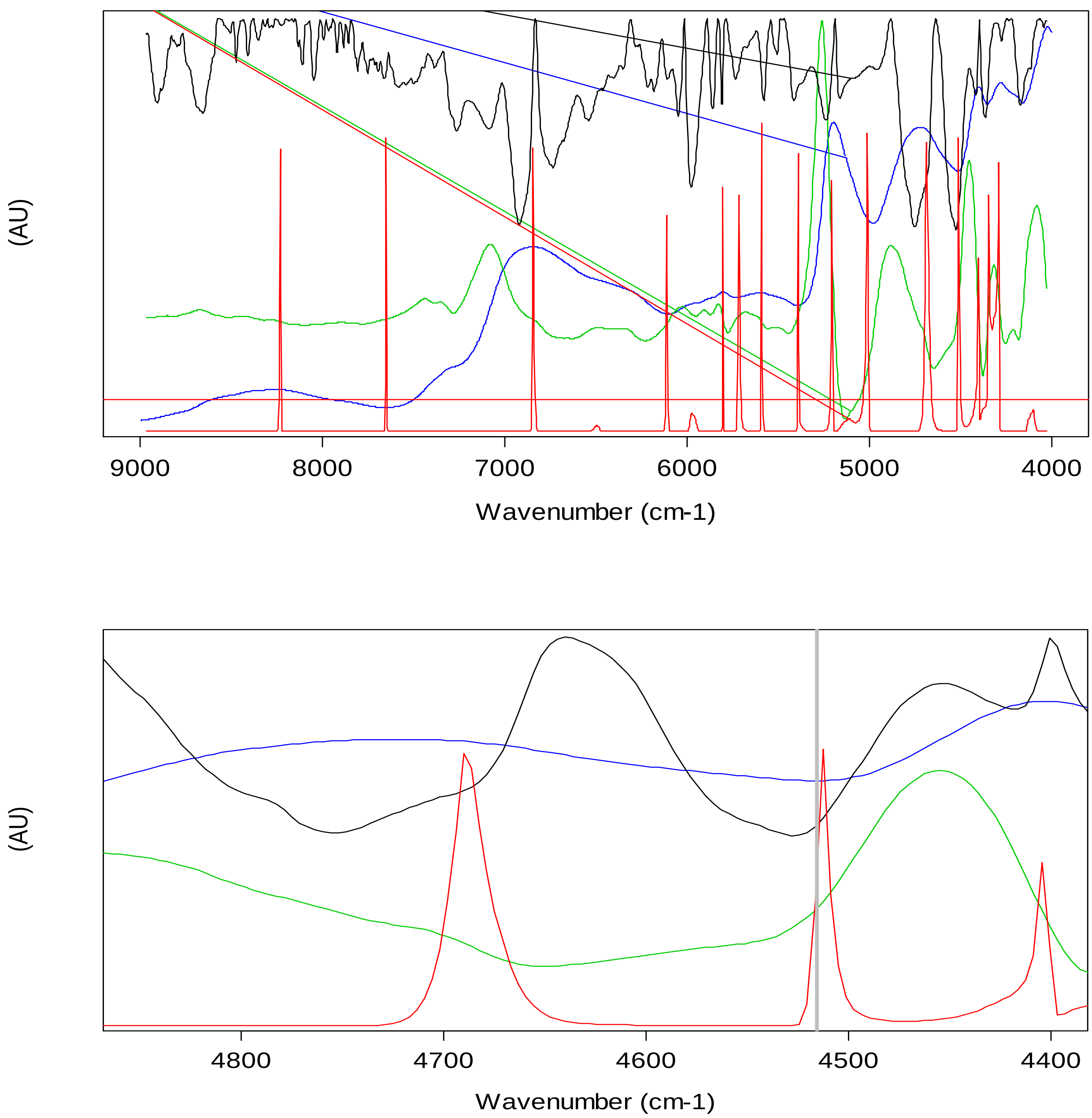

Figure 4 Influence of grain angle and extractive content on NIR spectra of *E. bosistoana*. Top full spectra; bottom selected region. Black: residual standard error of the calibration spectra to EC at any wavenumber. Red: significance of grain angle (0° to 90°) of the EC prediction at any wavenumber; red horizontal line: 0.1 significance of grain angle on EC. Blue: average full SNV normalised NIR spectra from cores. Green: average 1st derivative spectra of calibration spectra. Grey: selected wavenumber for predicting EC. AU: arbitrary units.

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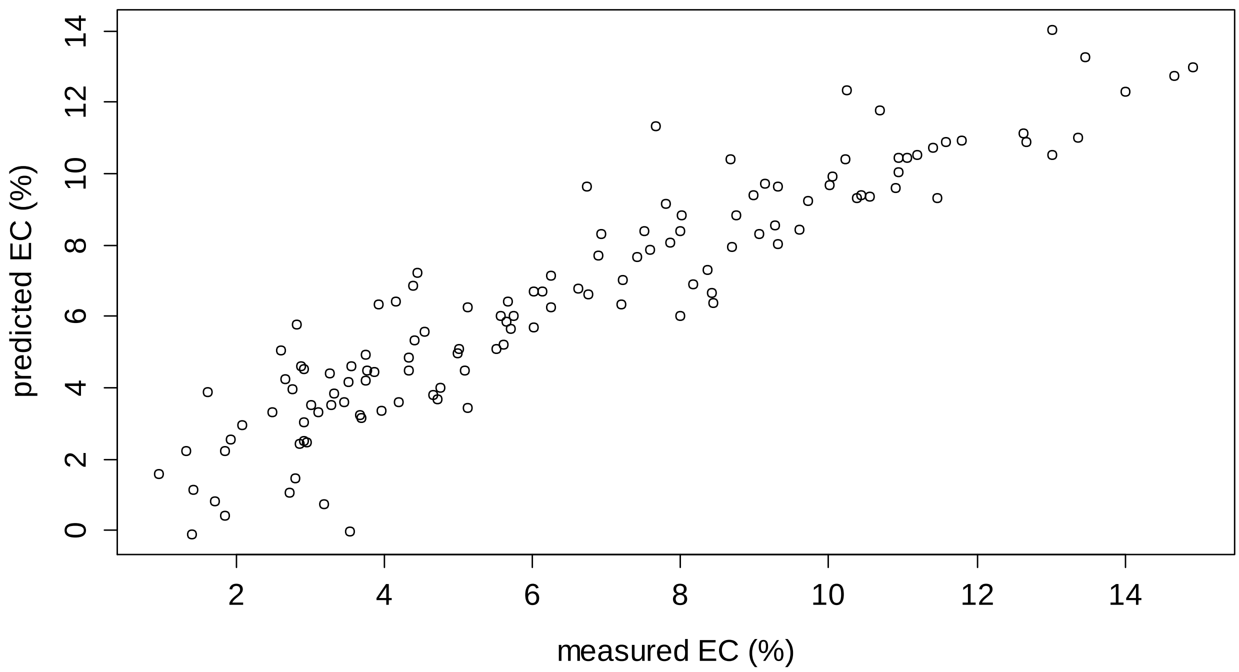

Figure 5: Measured versus predicted EC for *E. bosistoana* using the 4516 cm^{-1} signal. RSE = 1.28%; R^2 = 0.87.

No statistical significant difference between the measurements at 0° (cross-section), 45° and 90° (radial face) grain direction were found when using the signal at 4516 cm^{-1} to predict the extractive content from the spectra of the 8 cores (Figure 6).

Therefore it was concluded that grain orientation is not affecting the assessment of extractive content on cores by NIR. The implication is that uncertainties of grain in the cores, either due the difficulty to locate the 'up and down' orientation of the tree in the core or due to grain variation inherent to the tree (i.e. spiral/interlocked grain), do not impact on the accuracy of the method.

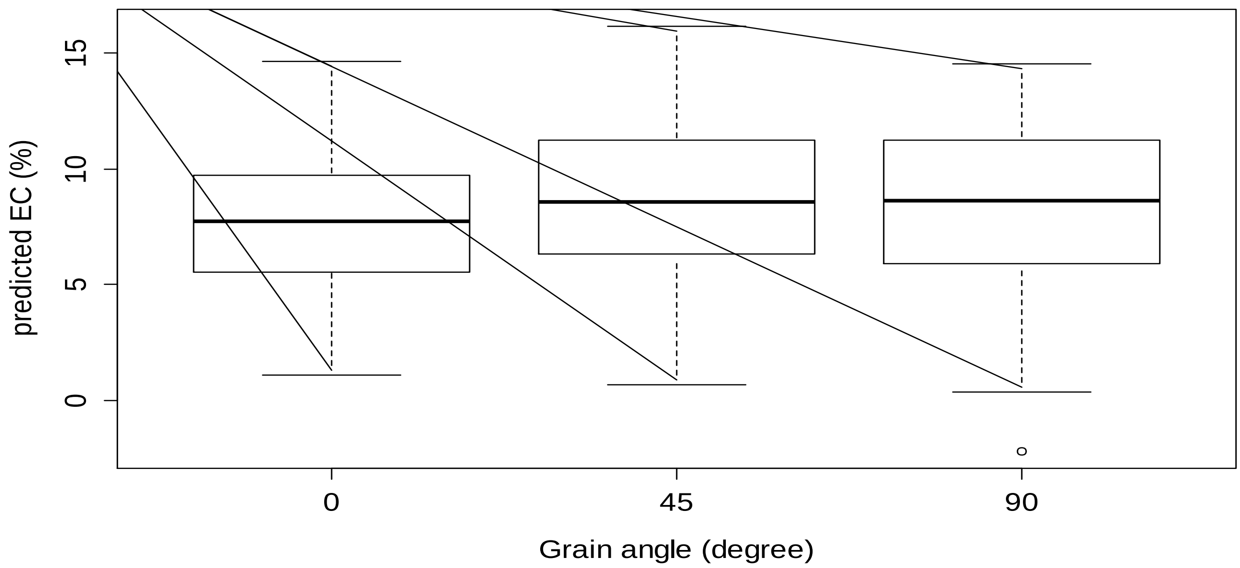

Figure 6: Predicted extractive contents for multiple measurements on 8 *E. bosistoana* cores dependent on grain angle.

Objective 2

The NIR spectra were also used to predict the grain angle using multivariate statistical analysis. The model was able to differentiate between cross-section and radial face (Figure 7). But its high residual error of ~25° rendered NIR on cores unsuitable to assess grain deviations inherent to the trees. A confounding effect would have been the presence of inherent grain deviations in the cores used for this experiment. However, grain deviations were not obvious in the samples and are generally below 10°.

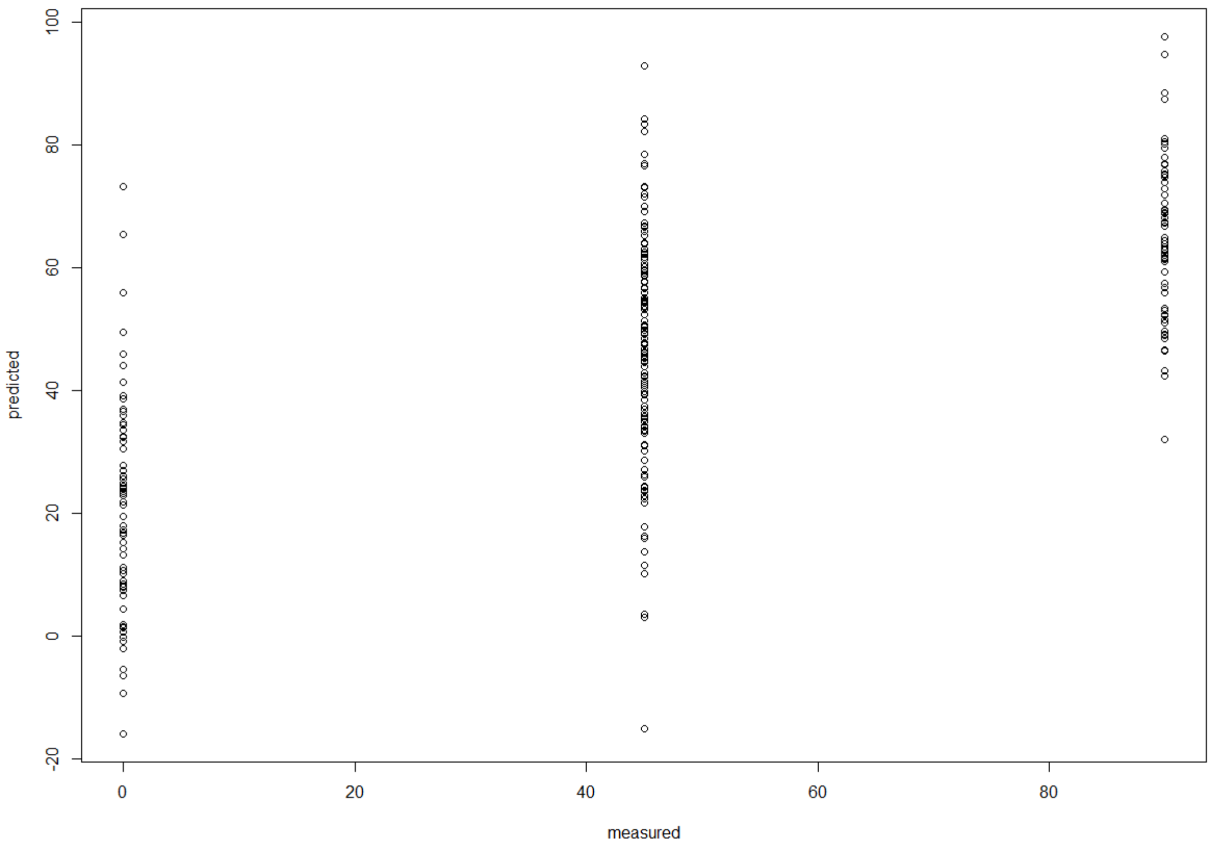

Figure 7: Measured and predicted (from NIR spectra) grain angle (°) in *E. bosistoana* cores.

CONCLUSION

1. The extractives content of heartwood of *E. bosistoana* can be assessed independently of grain angle by NIR spectroscopy. This assists the use of NIR spectroscopy to screen *E. bosistoana* for heartwood quality.
2. NIR spectroscopy is not able to predict grain deviations on cores with accuracy suitable for screening purposes.

REFERENCES

Schimleck, L. R., C. Mora and R. F. Daniels (2003). "Estimation of the physical wood properties of green *Pinus taeda* radial samples by near infrared spectroscopy." Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 33: 2297-2305.

APPENDICES

Appendix 1:

Distribution of predicted extractive contents in heartwood of 8 cores from 7-year old *E. bosistoana* trees. Note: variation within cores is expected as the extractive content varies along the radial profile.