Prediction of non-recoverable collapse in *Eucalyptus globulus* from near infrared scanning of radial wood samples

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Abstract

Near infrared (NIR) spectroscopy calibrations was used to predict radial profiles of cellulose content, wood density, cellulose microfibril angle (MFA) and modulus of elasticity (MOE) in 20-year-old plantation *Eucalyptus globulus* to identify non-recoverable collapse zones associated with tension wood.

Radial (cambium-to-pith) wood cores were extracted at a height of 1.0 m from trees selected to represent a range of silvicultural treatments. Near Infrared (NIR) spectra were measured at 1 mm intervals along the radial-longitudinal face of each core after drying to 12% equilibrium moisture content (EMC) at 40°C. Tangential shrinkage was measured at 8 points along each core, following steam reconditioning and re-drying to 12% EMC.

Additional cores from 20 of the sample trees were collected. Radial profiles of density, MFA and MOE were obtained for wood strips prepared from these cores, using the SilviScan 3 wood assessment system. Trait profiles were matched to radial NIR scans of these cores, enabling the development of NIR calibrations using Partial Least Squares (PLS) regression. These, and an existing NIR calibration for cellulose content, were used to predict the radial profiles of the four wood properties for the first set of cores. Predicted wood properties were then related to actual tangential shrinkage measurements and the occurrence of visible bands of non-recoverable collapse.

A regression model was developed to reliably predict regions of non-recoverable collapse from NIR-predicted cellulose content and MOE. Micrography of stained wood sections indicated that the collapse was caused by the presence of tension wood.

Vorhersage von nicht rückverformbarem Kollaps in radialen *Eucalyptus globulus* Prüfkörpern mittels Nah-Infrarot-Spektrometrie

Zusammenfassung

Mittels NIR-Spektroskopie wurden radiale Profile des Zellulosegehalts, der Holzdichte, des Cellulose-Mikrofibrillenwinkels (MFA) und des Elastizitätsmoduls (MOE) von 20 Jahre alten Plantagen-*Eucalyptus globulus* erstellt, um nicht rückverformbare Kollapsbereiche in Verbindung mit Zugholz zu erkennen.

Radiale Bohrkern (vom Kambium zur Markhöhle) wurden aus Bäumen, die verschiedene waldbauliche Behandlungsverfahren repräsentieren, in 1,0 m Höhe entnommen. Nah-Infrarot (NIR)-Spektren wurden entlang der radialen/longitudinalen Fläche eines jeden Kerns in 1mm Abständen gemessen, nachdem diese bei 40°C auf eine Gleichgewichtsfeuchte (ECM) von 12% getrocknet worden waren. Nach Dampfkonditionierung und erneuter Trocknung auf 12% EMC wurde das tangential Schwindmaß an acht Stellen entlang eines jeden Kerns gemessen.

Aus 20 der untersuchten Bäume wurden zusätzliche Kerne entnommen. Radiale Dichte-, MFA- und MOE-Profile wurden an Holzstreifen aus diesen Kernen mittels des Holzanalysegeräts SilviScan 3 erstellt. Aus den Eigenschaftsprofilen und den radialen NIR-Scans dieser Kerne wurden mittels partieller Regression NIR-Kalibrierungen entwickelt. Diese sowie eine bestehende NIR-Kalibrierung des Zellulosegehalts wurden zur Bestimmung der radialen Profile der vier Holzeigenschaften des ersten Kernkollektivs verwendet. Die so bestimmten Holzeigenschaften wurden dann mit den aktuell gemessenen tangentialen Schwindmaßen und dem Auftreten sichtbarer Banden von nicht rückverformbarem Kollaps in Beziehung gebracht.

Es wurde ein Regressionsmodell entwickelt, mit dem die Bereiche mit nicht rückverformbarem Kollaps aus dem mittels NIR bestimmten Cellulosegehalt und dem Elastizitätsmodul zuverlässig bestimmt werden können. Mikroskopische Aufnahmen gefärbter Holzabschnitte wiesen darauf hin, dass der Kollaps durch das Auftreten von Zugholz verursacht wurde.

Introduction

Most *Eucalyptus globulus* plantations, which total more than 2 million ha worldwide, are managed on short (10- to 15-year) clearfall rotations to produce pulpwood (Potts et al. 2004), but there is increasing interest in managing plantations of this species for sawn timber and engineered wood products. However, processing *E. globulus* for these solid wood products can be compromised by the presence of tension wood. Tension wood produces high longitudinal growth stresses when it occurs at the stem periphery (Wahyudi et al. 1999; Washusen et al. 2003), causing log splitting and excessive distortion, poor sawing accuracy and board splitting, all of which reduce green board recovery. Bands of tension wood within the log cause non-recoverable collapse and high and variable transverse and longitudinal shrinkage during drying (Touza Vázquez 2001; Washusen 2009). This reduces the recovery and value of dried sawn products.

As defined by the International Association of Wood Anatomists Committee on Nomenclature (IAWA 1964), tension wood is characterized by the presence of an internal gelatinous layer in the fibres and an unlignified cell wall. The high shrinkage associated with tension wood that occurs during drying appears similar to collapse in normal wood, however, with the application of steam reconditioning these zones do not recover their original dimensions (Boyd 1977; Chafe et al. 1992; Kauman 1964). Washusen (2000) reported a comprehensive anatomical study of *E. globulus* wood which described non-recoverable collapse and confirmed the presence of associated tension wood by microscopic examination of stained wood sections.

In most hardwood tree species, tension wood occurs when trees reposition their stem orientation to capture light as the stand develops, or in response to bending stresses applied by factors such as prevailing winds (Kubler 1988; Scurfield 1973). However, in some species, for example *Acacia mangium* and *E. globulus*, even apparently straight trees can develop tension wood (Wahyudi et al. 1999; Washusen and Ilic 2001). Tension wood was predominantly restricted to the lower 30% of tree height and increased in severity towards the base in straight trees in an 11-year-old *E. globulus* plantation in South Australia (Washusen 2002). The impact of tension wood on wood shrinkage depends on the frequency and distribution of tension wood fibres. In core samples from this plantation, when the percentage of tension wood fibres with thick gelatinous layers exceeded 10% over a 5 mm radial distance, there were identifiable zones of excessive shrinkage after reconditioning (Washusen and Ilic 2001).

Detection of tension wood by microscopic observation on the anatomy of xylem cells in stained wood sections is time-consuming, labour-intensive, and therefore expensive. Indirect methods that analyse wood physical and/or chemical properties at fine scale offer the prospect of detecting tension wood more rapidly and at lower cost. This would facilitate

systematic studies to evaluate the effects of site, silviculture and genetics on the occurrence of tension wood in *E. globulus* plantations managed for solid and engineered wood products. Tension wood is characterized by high cellulose content, low cellulose microfibril angle (MFA), high basic density and high modulus of elasticity (MOE) (Chaffey 2000; Chauhan and Walker 2011; Wada et al. 1995; Wardrop and Dadswell 1955).

The SilviScan wood microstructure assessment system combines scanning X-ray microdensitometry and X-ray diffraction to measure wood properties in prepared wood strips of 2 mm thickness. It has been used to determine the radial profile, at fine scale, of wood density, MOE and MFA (Evans 1994, 1999; Evans and Ilic 2001; Evans et al. 2000). Previous versions of SilviScan had been configured to measure cellulose crystallite width, which is an indicator of tension wood (Washusen and Evans 2001b). Maximum values of crystallite width greater than 3.6 nm are associated with the presence of tension wood (Washusen and Evans 2001a).

The effects of silvicultural treatments on the development of tension wood in an *E. globulus* plantation trial in Gippsland, Victoria were investigated using SilviScan. Radial profiles of cellulose crystallite width and MFA in 13-year-old trees were examined (Washusen et al. 2005). Tension wood, indicated by high crystallite width and low MFA, was found to be more prevalent in the treatments that had been heavily thinned to 200 trees ha⁻¹ at age 8 years than in unthinned and less heavily thinned treatments, but its occurrence was reduced by the application of fertilizer at thinning.

The association of wood chemical and physical properties with tension wood suggests that near infrared (NIR) spectroscopy could be used to detect tension wood occurrence. Diffuse reflectance NIR spectra consist mainly of overtone and combination bands corresponding to the fundamental stretching vibrations of O-H, N-H and C-H functional groups and reflect the chemistry of the sample (Michell 1995; Næs et al. 2002). NIR spectra can be collected from solid wood cores, wood meal, wood strips or wood chips. Regression calibrations are developed to associate particular wood properties with the spectra, for an appropriate sample population (Thomas 1994).

NIR spectroscopy has been used to predict wood cellulose content. A global NIR calibration for cellulose content was developed based on chemically analysed samples of wood meal from over 1800 samples from a number of *Eucalyptus* species (Downes et al. 2010). This global wood meal cellulose calibration was used to build a calibration to predict cellulose content based on spectra collected from the radial longitudinal surface (Downes et al. 2012). These calibrations displayed high explanatory power and low error when applied to a wide range of eucalypt samples. For example, the surface scanning calibration developed for *E. globulus* has an r^2 (percentage of variance explained) of 86% and root mean square error of cross validation (RMSECV) of 0.87% cellulose content (Downes et al. 2012). NIR calibrations to predict microfibril angle (MFA) in *Pinus radiata* and *P. taeda* (Schimleck et al. 2005b), basic

density in *E. globulus* (Schimleck et al. 1999) and modulus of elasticity (MOE) in *E. delegatenis* and *P. radiata* (Schimleck et al. 2002) have also been developed. SilviScan (Evans 1994) MFA and MOE data was used to build these NIR calibrations (Schimleck et al. 2005a). However, NIR calibrations for density, MFA and MOE developed to date have displayed lower levels of explanatory power compared to those for cellulose. This is to be expected, because variations in density, MFA and MOE are less directly related to basic wood chemistry and associated NIR spectra than is variation in cellulose content.

Low MFA and high cellulose content, density and MOE are known characteristics that are related to non-recoverable collapse, and it has been demonstrated that these wood properties can be predicted from NIR spectra. The study reported here examined whether bands of non-recoverable collapse in *E. globulus* could be reliably predicted from radial profiles of these wood properties, predicted by radial NIR surface scanning.

Materials and Methods

Eucalyptus globulus plantation

Wood samples were collected from trees in an *E. globulus* silvicultural trial near Tostaree, Victoria, Australia (37.78° S, 148.18° E). The trial had been established to investigate the effects of silvicultural treatments on tree growth and log yield, and tension wood occurrence in the stand had been studied previously (Washusen et al. 2005).

The trial was planted in 1991 using *E. globulus* ssp. *globulus* seedlings (Waitawhile Track, Otway Ranges, Victoria seed source) at a spacing of 3.5 x 2.5 m (1143 trees ha⁻¹). The trial tested three thinning treatments (unthinned control (UT), and non-commercial thinning at age 8 years with residual stockings of 400 trees ha⁻¹ (NCT400) and 200 trees ha⁻¹ (NCT200) in factorial combination with two levels of fertilizer application at thinning (no fertilizer, and fertilizer providing 200, 62.5, 62.5 and 6 kg ha⁻¹ of N, P, K and S, respectively).

Sampling

The trees were sampled at age 20 years. From measurements of all trees in the silvicultural trial at this age, average diameters (over bark at 1.3 m height; DOB_{1.3}) increased from UT (29.5 cm), NCT400 (30.3 cm), to NCT200 (31.2 cm), with little effect of fertilizer on DOB_{1.3}.

Potential sample trees (live, single-stemmed, with less than 20° butt-sweep) were initially identified from the inventory of all trees within the trial to select matched sets of trees spanning a comparable diameter range (DOB_{1.3} 25-35 cm) across different silvicultural treatments (6 treatments x 18 trees), and additionally trees representing the wider diameter range in each treatment. In total, 180 trees were sampled (Table 1), but only 175

wood cores were used, because 5 cores broke during initial laboratory processing.

Radial, bark-to-pith, 12 mm diameter increment cores were taken from the south-western aspect of the sampled trees at ~1.0 m height. The south-western aspect was chosen because previous research on the within-tree distribution of tension wood at this site (Washusen et al. 2002) had indicated that tension wood was likely to be most prevalent at this aspect, consistent with strong winds being predominantly from the west and south-west.

The cores were taken using a motorized wood corer. Wood cores were discarded if they contained a knot or kino pocket, and a replacement core was taken from the same aspect of the same tree at ~ 0.9 m height. The first set of wood cores was taken in February 2011. The second set of wood cores was collected in July 2011 from a subset of these trees that were judged likely to display non-recoverable collapse based on preliminary predictions of wood properties and measurements of tangential shrinkage from the first set. The second set of cores was taken from the south-western aspect of the trees at ~0.9 m height.

Tangential shrinkage measurements

The first set of cores was planed on both longitudinal-radial faces to facilitate shrinkage measurements and NIR scanning. Tangential width measurements were made on every core using a digital spindle micrometer (Mitutoyo micrometer series 331-261). The micrometer had a range of 0-25 mm, a graduation of 0.001 mm, a 2 mm diameter anvil and spindle, and a ratchet stop to apply a constant force. The micrometer recorded measurements directly to a computer.

Green cores were delimited at 25%, 50% and 75% of their bark-to-pith length, dividing the core into four quarters. The mid-points of each quarter were marked (Fig1). Tangential widths and distances from bark were measured at each mid-point in the green condition, and the cores weighed.

The cores were dried as recommended by Washusen (2000), inside a laboratory kiln at a dry bulb temperature of 40°C and a wet-bulb temperature of 34.5°C. Samples were dried over 3 days to constant weight, corresponding to equilibrium moisture content (EMC) of about 12%. After drying, the cores were placed in a conditioning room (relative humidity 66.6% and temperature 22.5°C) to maintain 12% EMC.

The tangential width of the cores was re-measured at the marked mid-points of each quarter. In addition, the width at the point of observed maximum tangential shrinkage was measured in each quarter. That is, a total of 8 tangential widths were measured per core. The points of maximum tangential shrinkage were marked with a cross using a waterproof pencil (Fig2).

Tangential shrinkage at each mid-point and at the maximum shrinkage point in each quarter of the cores (relative to green condition) was measured again after reconditioning using a water vapour saturated air stream (95-100°C) for two hours followed by re-drying to about 12% MC. The cores were then oven dried at 103 +/-2°C, tangential shrinkage remeasured and oven dry weights measured to calculate the moisture content after reconditioning. Shrinkage values were normalized to 12% moisture content (MC) by using measured MC for each core in the following equation:

$$(1) \quad \frac{T_a}{T_{mar}} = \frac{MC_{ar} - 12}{MC_{12\%} - 12}$$

where:

T_a is the adjusted tangential shrinkage after reconditioning (mm);

T_{mar} is the measured tangential shrinkage after reconditioning (mm);

T_{mod} is the measured tangential shrinkage after oven drying (mm);

MC_{ar} is the moisture content of the core after reconditioning (%), and

$MC_{12\%}$ is the moisture content of the core at 12%.

The average of the four mid-point core widths prior to drying was used to calculate shrinkage at the points of maximum shrinkage (i.e. where widths were not measured before drying because the points had not yet been identified). The average standard deviation of all green mid-point tangential widths was 0.006 mm, small compared to the average width difference from green to dry condition (1.2 mm).

SilviScan analysis

The second set of cores collected for SilviScan analysis was dehydrated in ethanol (Evans et al. 2000) for 1 week. The cores were then conditioned in a room at 20°C and 40% humidity until their weight was constant. The moisture content of the samples was about 8%.

The cores were cut to produce radial strips, 2 mm tangentially and 7 mm longitudinally (Evans 1994, 1999) using an adjustable twin-blade saw. The samples were analysed, commencing at the cambial end, using SilviScan-3 (Evans 1999, 2006; Evans et al. 2000). SilviScan measurements of MFA, density and MOE were obtained for all 20 cores, at 1 mm intervals.

NIR spectroscopy

NIR diffuse reflectance spectra were measured on one radial longitudinal face of each core dried to 12% MC prior to reconditioning, for the set of 175 samples used to measure tangential shrinkage, and for the 20 SilviScan strips.

A Bruker MPA FT-NIR spectrometer (Bruker Optik GmbH, Ettlingen, Germany) with a built in fibre-optic scanning attachment joined to a linear sample transport system was used to measure spectra at 1 mm intervals from bark to pith. The wave number range was between 10,000 cm⁻¹ and

4,000 cm⁻¹ (between 1000 nm and 2500 nm) at an 8 cm⁻¹ resolution. Each spectrum was an average of 16 scans.

Calibration development

Using the data obtained from the SilviScan analyses and the respective spectra from the set of 20 cores, calibrations for MFA, density and MOE were developed. Each NIR and SilviScan sampling interval was obtained relative to the cambial end enabling matching of each SilviScan data point to the corresponding NIR spectrum. The QUANT software (Bruker 2005) was used, employing partial least squares (PLS) regression (Næs et al. 2002). Statistical parameters including the coefficient of determination (R^2), root mean square error of the cross validation (RMSECV) and the number of principal components required (rank) were obtained for each calibration. All the SilviScan data available for each property (MFA, density, MOE) was used with the corresponding NIR spectra.

Predictions

In addition to the NIR calibrations for density, MFA and MOE, a previously developed cellulose calibration was applied to the spectra sets from the 175 wood cores before reconditioning, to predict these wood properties at 1 mm intervals. The cellulose calibration had been developed with NIR spectra obtained at 1 mm increments and then averaged to 10 mm intervals, so that the data could be matched to laboratory diglyme cellulose analyses (Downes et al. 2012). No laboratory cellulose validation was done on the samples used in this study.

Statistical analysis

At each measured tangential shrinkage point the distance from bark to pith was determined to match with the appropriate spectra from which cellulose content, MFA, density and MOE were predicted. For example, in core No. 001 (Table 2), the first measurement of tangential shrinkage was 21 mm from the bark. This was the original reference point in the quarter closest to the bark and the predicted wood properties from the associated NIR spectrum were matched to the measurement point. Table 2 shows the NIR predictions for the eight 1 mm scans of core No 001 that correspond with the eight marked points where tangential shrinkage after reconditioning was measured.

To give better representation to sites of non-recoverable collapse in the analysis, a data set was selected using stratification and accrual methods so that the tangential shrinkage had equal representation across the shrinkage range. This was done because points of high shrinkage after reconditioning that indicated tension wood presence were relatively few and the bulk of the data were considered to be within the shrinkage range for normal wood. The data were firstly allocated to 8 stratum cells, each with a shrinkage range of 2.5 %. Data points were then randomly selected from the whole data set until 10 points were accrued for each shrinkage range. As there were only 9 sample points with shrinkage over 20%, this stratum cell only had 9 points, thus making the total number of shrinkage

points chosen 79. This set was termed the equally weighted shrinkage data set. The second set included all of the tangential shrinkage points and therefore had far greater representation of normal wood. It was termed the full data set. Pearson correlations (Edwards 1976) were calculated for each data set to determine correlations between NIR-predicted wood properties and tangential shrinkage after reconditioning.

NIR-predicted wood quality traits that were significantly correlated with tangential shrinkage were included in a forward stepwise regression to predict tangential shrinkage after reconditioning, using IBM SPSS Statistics software version 19. Tests for both data sets were conducted for heteroscedasticity (Breusch and Pagan 1979) with the same software.

The best model developed from the regression analysis was applied to all the spectra obtained from the 175 core samples data set to analyse the strength in predicting tangential shrinkage across whole cores and to examine the correspondence between high predicted tangential shrinkage and measured non-recoverable collapse.

Histochemical analysis

To test whether the non-recoverable collapse was associated with tension wood, histochemical tests were conducted on anatomical sections taken from selected samples that presented non-recoverable collapse.

The transverse sections of the cores were cut into 1.5 cm long pieces in the radial direction with the non-recoverable collapse zones in the middle of the section. To differentiate between normal and tension wood fibres, a double staining procedure was conducted using a combination of safranin and alcian blue.

Sections of 18 μm thickness were cut using a microtome (American Optical Company Sliding Microtome) and firstly stained with 1% aqueous safranin and counter stained with 1% aqueous alcian blue. The samples were then viewed in a microscope (Olympus BX61 motorized microscope) with a camera attached to obtain digital images using the AnalySIS Pro Image software.

Results

Tangential shrinkage after reconditioning

The mean tangential shrinkage after reconditioning was 9.3%. Samples that had measured shrinkages $>15\%$ presented distorted surfaces and a visible collapse that can be described as non-recoverable, as the steam reconditioning had little or no effect on dimension recovery. In contrast, samples with tangential shrinkage values $<15\%$ exhibited normal shrinkage as there were no visible signs of collapsed surfaces. Sample points, either at the marked mid-points or the points of maximum shrinkage (Fig2), that had tangential shrinkage $>15\%$ comprised 3% of all the measurements (40 out of 1,386), but were present in 16% of the cores (28 out of 175).

Cores with maximum shrinkage exceeding 15% occurred in all six silvicultural treatments at approximately equal frequencies. Fertilizer application did not significantly affect the incidence of non-recoverable collapse (36% of the cores exhibiting non-recoverable collapse came from treatments without fertilizer). Table 3 shows the number of trees in each treatment having at least one instance of non-recoverable collapse in a core.

The measured points where tangential shrinkage exceeded 15% were used as reference points to assess whether the values of NIR-predicted cellulose content, MFA, density and MOE could be used to identify zones of tension wood.

Measurement of wood properties using SilviScan

Mean SilviScan density for the 20 cores selected for development of the calibrations was 783 kg m^{-3} , mean MFA was 10.3 and mean MOE 18.6 GPa, although the range of individual values for all three variables was very wide (Table 4). There was a moderate negative relationship between MFA and density ($r = -0.66$).

NIR-based predictions of wood properties

Cross-validation models for the radial scanning calibrations are shown in Fig.3. The component ranges for each property were; $2^\circ - 33.8^\circ$ MFA, $292 - 1216 \text{ kg m}^{-3}$ density and $5.3 - 36.7 \text{ GPa}$. The calibrations explained 62.8%, 64% and 73.8% of the variation in MFA, density and MOE respectively and the RMSECV was 2.42° for MFA, 96.3 kg m^{-3} for density and 3.0 GPa for MOE.

These calibrations, along with the existing calibration for cellulose content, were used to generate NIR-predicted radial profiles (Fig.4) of these properties for the first sample set of 175 wood cores. The range of predicted cellulose content varied from 35% to 60.6% with a mean of 43.8%. Cellulose content tended to generally increase from the pith to the cambium. The predicted MFA varied between 1° and 39° with a mean of 12° . This was higher than the average from the Tostaree SilviScan data calibration (10.3°).

The minimum predicted density was 177 kg m^{-3} and the maximum 1252 kg m^{-3} . Predictions below 600 kg m^{-3} were obtained from the pith ends of the samples in most of the cores. The mean predicted density was 766 kg m^{-3} , which was lower than the average density from the Tostaree calibration sample set (783 kg m^{-3}).

The predicted MOE presented some unusually low values, which were related to unusually high MFA predictions and very low cellulose percentages, both properties that are biologically related to MOE. Negative values and values less than 5 GPa were discarded. The mean measurement was 17.6 GPa, slightly lower than the values obtained in the Tostaree calibration (18.2 GPa).

Out of the 175 samples, 30 showed very low MFA and high predictions for density and MOE at one or more points along the sample.

Relationships between predicted wood properties and tangential shrinkage

The predicted cellulose content, MFA, density and MOE were examined in cores in which tangential shrinkage over 15% occurred at one or more of the eight measured points. These zones of non-recoverable collapse (high measured shrinkage) were typically associated with high predicted cellulose content (usually over 50%), density (above 960 kg/m³) and MOE (above 25 GPa), and low predicted MFA (under 7.5°), as seen in Fig. 4, where the peaks of the predicted wood properties were related to the measured non-recoverable collapse zone in the core.

The Pearson correlation matrices for the equally weighted and full data sets are given in Tables 5 and 6. NIR-predicted cellulose content, MFA, MOE and density were all significantly correlated with shrinkage to 12% MC after reconditioning (AR) in both data sets ($p < 0.01$). The equally weighted shrinkage data had stronger correlations with shrinkage than the full data set. There was a particularly strong correlation between shrinkage and cellulose content ($r = 0.76$). Tangential shrinkage also correlated well with MFA, density and MOE. There was a strong negative correlation between cellulose content and MFA, and a positive correlation between cellulose content and density and MOE. MFA, density and MOE were strongly inter-correlated.

Stepwise regression to predict tangential shrinkage after recovery

The stepwise regressions developed to predict tangential shrinkage are presented in Table 7. In both cases the dependant variable was tangential shrinkage at 12% moisture content after recovery.

Cellulose content was an important predictor of tangential shrinkage, especially in Model 1. MOE was also an important predictor in both models. In neither case did MFA or density add significantly to the explanatory power of the regression. A Breusch-Pagan test was performed using IBM SPSS Statistics software to test for heteroscedasticity. The results rejected heteroscedasticity at $p < 0.05$, indicating that no data transformation was necessary for the regression.

Model 1 is represented by the following equation:

Model 1

where:

Y is the predicted tangential shrinkage to 12% MC AR;

X₁ is the NIR-predicted cellulose content (%); and

X₂ is the NIR-predicted MOE (GPa).

Model 1 explained 64% ($p < 0.01$) of the variance in tangential shrinkage when applied to the equally weighted data (Fig.5) and 35% when applied to the whole data set of measured shrinkage points (Fig.6). The vertical and horizontal lines in both graphs in Fig.5 and Fig.6 divide the data into quadrants that indicate the capability of each model to predict bands of non-recoverable collapse, assuming a 15% shrinkage threshold. The lower left quadrant indicates data points correctly identified as points of normal shrinkage and the quadrant at the top right indicates the data that are correctly identified as affected by non-recoverable collapse (over 15% tangential shrinkage). The data points in the two remaining quadrants were incorrectly identified by the model as either points of normal shrinkage (upper left quadrant) or points of non-recoverable collapse (lower right quadrant). Both prediction sets had a number of errors. Model 1 correctly identified 75% of the observed points with non-recoverable collapse in the equally weighed set. In the whole data set it correctly identified 55% of these points. At the high end of the predicted shrinkage, in the equally weighed set, out of the 29 measurements of shrinkage higher than 15%, 21 were under predicted and 8 over predicted, meanwhile from the whole set, from 40 measurements over 15%, 30 were under predicted and 10 over predicted.

Prediction of tangential shrinkage for the entire radial profiles

Model 1 was applied to the NIR radial profiles from the 175 cores, to predict wood properties at 1 mm resolution. Model 1 predicted that more than 10% of the data points would have shrinkage of over 15% at 12% MC AR.

The predicted radial profiles of tangential shrinkage were tested across individual wood cores with observed non-recoverable collapse bands to compare predicted and observed shrinkage patterns.

Fig.7 shows four examples of cores with bands of non-recoverable collapse. These cores all had measured non-recoverable collapse bands corresponding to the distinctive peaks of predicted shrinkage visible in the graphs.

Histochemical analysis of tension wood presence

Collapse zones of the four core samples shown in Fig.7 were selected for histochemical staining. Inspection of microscopic images confirmed the presence of tension wood, with cells having characteristically thick walls, small lumens and a clearly visible layer corresponding to gelatinous cellulose free of lignin, stained by alcian blue.

Discussion

It has been demonstrated that bands of non-recoverable collapse in *E. globulus* wood samples can be predicted from radial profiles of NIR-predicted wood properties determined from scanning of the radial-longitudinal wood surface after three days of drying under controlled conditions from the green to the air dry state (12% moisture content). NIR-

predicted cellulose content and MOE were the strongest predictors of tangential shrinkage and non-recoverable collapse. As with previous studies on *E. globulus* (Washusen 2000; Washusen and Ilic 2001), microscopy and histochemical analysis confirmed that zones of excessive non-recoverable collapse were anatomically identified as tension wood.

More work is required to determine the extent to which the NIR calibrations presented here can be extended across a range of site conditions and different eucalypt species so as to allow global calibrations for shrinkage to be developed, as has been done for prediction of cellulose content and Kraft pulp yield from woodmeal samples (Downes et al. 2011). When developing NIR calibrations for density, MFA and MOE the use of SilviScan is critical as it is the only cost-effective mean of generating the required calibration data at a suitable radial resolution (1.0 mm sampling intervals).

The variance explained by NIR calibrations for density, MFA and MOE developed for this study was similar to that of other NIR calibrations for these traits in eucalypt species, such as basic density from 8 year old *E. globulus* (up to R^2 of 0.77) (Schimleck et al. 1999), MFA (R^2 of 0.77) and MOE (R^2 of 0.90) in *E. delegatensis* (Schimleck et al. 2001) and MFA (R^2 of 0.69) and MOE (R^2 0.80) in *E. nitens* (Schimleck et al. 2006). Published calibrations describing the prediction of wood basic density from woodmeal samples (Downes et al. 2011; Schimleck et al. 1999) and solid wood (Schimleck et al. 2003) indicate root mean square errors of prediction of $\sim 25\text{-}30 \text{ kg m}^{-3}$. The calibrations presented here exhibited markedly higher standard errors. This probably arises from three sources:

Minor (less than 1 mm) mis-alignment between the radial NIR spectra with the SilviScan arising from slight differences in the starting point of the two scans

Although both scans were generated at 1 mm intervals, the width of the NIR illuminated region was 1.08 mm compared with the 1 mm footprint for SilviScan.

Collection of NIR spectra from the radial-longitudinal surface was matched with SilviScan data generated from X-rays transmitted through the 2 mm tangential thickness of the strip. Where orientation of density variation caused by the annual cycle of early/late wood, associated with growth rings, is not perfectly tangential, some measurement error, from the perspective of the NIR spectra, would arise.

It is possible that developing calibrations between SilviScan and NIR using wider sampling intervals (2 – 5 mm) might reduce the significance of these potential sources of error. The application of calibrations developed at these coarser resolutions to spectra collected at finer intervals also requires investigation. However the successful development of a surface-scanning cellulose calibration for *E. globulus* at 10 mm resolution (Downes et al. 2012; Downes et al. 2010) suggests that improvements in the calibrations for density, MFA and MOE may be feasible.

Considerable radial variation in wood properties was evident (Fig.4). Radial trends in cellulose content matched those reported by Downes et al (2012) who found that NIR-predicted cellulose content generally increased from pith to cambium for *E. globulus* plantations at three sites in Western Australia. In their study, a small proportion of trees had one or more distinct bands of high cellulose content and Kraft Pulp Yield, indicative of non-recoverable collapse zones. The radial increase in MOE from pith to cambium and the corresponding decline in MFA observed in the current study was consistent with that reported for 22-year-old plantation-grown *E. nitens* by Medhurst et al. (2012) who examined radial profiles with SilviScan.

NIR scanning of the set of 175 cores followed by shrinkage prediction using Model 1 predicted a greater number and higher proportion of sample points with non-recoverable collapse than what was measured with the microtome. This can be explained by the greater frequency of tension wood measurements in the large data set. The data used to develop Model 1 had a single measurement of shrinkage taken at the point of highest shrinkage within each collapse band. In the NIR-predicted data set, there were usually several measurement points indicating collapse within individual bands. At positions where predicted cellulose content and MOE were above 50% and 25 GPa respectively, non-recoverable collapse was typically found.

The proportions of trees with non-recoverable collapse across the six different fertilizer and thinning treatments combinations did not differ significantly in this study. This contrasts with the findings of Washusen et al. (2005), who sampled the same plantation five years after thinning and fertilizer application and showed that fertilizer application significantly reduced the development of tension wood, as indicated by high cellulose crystallite width at high thinning intensity. In the current study here the proportions of trees with collapse were low, making it difficult to detect treatment effects through comparison of proportions of trees affected.

This method offers the prospect of being able to survey the extent of non-recoverable collapse in standing *E. globulus* trees by radial NIR scanning of wood core samples, relatively quickly and at modest cost. This could be applied in studies that aim to better understand the effects of site, silviculture and genetics on the occurrence of non-recoverable collapse and associated tension wood that may limit the solid-wood utilization of this species (Washusen 2009). However, additional work is clearly required to understand the relationship between the levels of predicted occurrence of collapse in radial samples with product quality outcomes for defined wood processing systems. To cause severe drying degrade in actual boards (Touza Vázquez 2001; Washusen 2009), it is likely that predicted non-recoverable collapse bands would have to be at least 5 mm in radial extent; this needs to be verified experimentally.

Conclusion

Zones of high NIR-predicted cellulose content, density and MOE, and low NIR-predicted MFA were closely associated with bands of non-recoverable collapse in radial wood samples of *E. globulus*. This was confirmed by a regression analysis indicating that bands of non-recoverable collapse could be reliably predicted from NIR-predicted cellulose content and MOE. Non-recoverable collapse occurred when cellulose content and MOE were above 50% and 25 GPa respectively. These thresholds can be used with core sampling strategies and radial NIR scanning as a relatively low cost and effective means of identifying zones of excessive non-recoverable shrinkage associated with tension wood occurrence in *E. globulus*.

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Tables

Table 1: Number of sample trees in each silvicultural treatment (UT = Unthinned, NCT400 = Non-commercial thinning 400 trees ha⁻¹, NCT200 = Non-commercial thinning 200 trees ha⁻¹; F = fertilizer)

Tabelle 1 Anzahl der untersuchten Bäume in Abhängigkeit der verschiedenen waldbaulichen Behandlungsverfahren (UT = nicht durchforstet, NCT400 = nicht kommerzielle Durchforstung 400 Bäume pro ha; NCT200= nicht kommerzielle Durchforstung 200 Bäume pro ha, F = Düngung)

Treatment	DOB _{1.3}			Total
	< 25 cm	25-35 cm	> 35 cm	
UT	10	18 + 7	1	36
UTF	15	18 + 2	1	36
NCT400		18 + 5	4	27
NCT400F		18 + 6	3	27
NCT200		18 + 0	9	27
NCT200F		18 + 3	6	27

Table 2: Example of measured shrinkage data at eight points on a core and predictions of wood properties from matched NIR surface scans (core No 001)

Tabelle 2 Beispiel der an acht Stellen an einem Kern gemessenen Schwindmaße und der aus den NIR-Messungen hergeleiteten Holzeigenschaften (Kern Nr. 001)

Distance from bark (mm)	Shrinkage 12% EMC (%)	Cellulose (%)	Density (kg/m ³)	MFA (°)	MOE (GPa)
21	11.1	43.3	794	10.4	20.5
24	10.7	48.3	789	13.1	16.8
52	9.3	46.1	760	10.6	19.1
56	8.9	44.1	816	9.9	21.0
84	8.1	38.5	606	16.2	11.3
92	7.4	42.1	737	13.4	15.2
111	7.2	46.3	683	14.3	14.0
124	6.8	44.0	657	14.0	14.0

Table 3: Number of trees in each treatment with shrinkages >15% and <15% (UT = Unthinned, NCT400 = Non-commercial thinning to 400 trees per ha, NCT 200 = Non-commercial thinning to 200 trees per ha, F = Fertilizer applied)

Tabelle 3 Anzahl der Bäume in Abhängigkeit der verschiedenen waldbaulichen Behandlungsverfahren mit Schwindmaßen <15% und >15% (UT = nicht durchforstet, NCT400 = nicht kommerzielle Durchforstung 400 Bäume pro ha; NCT200= nicht kommerzielle Durchforstung 200 Bäume pro ha, F = Düngung)

Treatment	Number of trees with shrinkage <15%	Number of trees with shrinkage >15%	Percentage of trees with shrinkage >15%
UT	32	2	7.1
UTF	29	6	21.4
NCT200	19	7	25.0
NCT200F	21	6	21.4
NCT400	26	1	3.6
NCT400F	20	6	21.4
Total	147	28	16.0

Table 4: SilviScan mean maximum and minimum values for the 20 wood cores

Tabelle 4 Mittelwerte, Größt- und Kleinstwerte der 20 Bohrkerne bestimmt mittels SilviScan

	Density (kg/m ³)	MFA (°)	MOE (GPa)
Mean	783	10.3	18.6
Max	1216	47.5	36.7
Min	293	2.0	5.4

Table 5: Pearson correlation matrix results for shrinkage at 12% MC AR, cellulose content, MFA, density and MOE for the equally weighted shrinkage data set. Level of significance: * = $P < 0.05$, ** = $P < 0.01$, *** = $P < 0.001$. N = 79

Tabelle 5 Pearson Korrelationskoeffizienten zwischen dem Schwindmaß bei 12%

Holzfeuchte nach Dampfkonditionierung, dem Cellulosegehalt, dem

Mikrofibrillenwinkel, der Dichte und des E-Moduls auf Basis der gleich gewichteten

Schwindmaße. Signifikanzniveau: * = $P < 0,05$, ** = $P < 0,01$, *** = $P < 0,001$. N = 79

	Cellulose	Density	MFA	MOE
Tangential Shrinkage	0.76**	0.67**	-0.67**	0.69**
Cellulose		0.66**	-0.65**	0.67**
Density			-0.85**	0.92**
MFA				-0.97**

Table 6: Pearson correlation matrix results for shrinkage at 12% MC AR, predicted cellulose content, MFA, density and MOE for the full data set. Level of significance: * = $P < 0.05$, ** = $P < 0.01$, *** = $P < 0.001$. N = 1,386

Tabelle 6 Pearson Korrelationskoeffizienten zwischen dem Schwindmaß bei 12%

Holzfeuchte nach Dampfkonditionierung, dem Cellulosegehalt, dem

Mikrofibrillenwinkel, der Dichte und des E-Moduls auf Basis aller Daten.

Signifikanzniveau: * = $P < 0,05$, ** = $P < 0,01$, *** = $P < 0,001$. N = 1386

	Cellulose	Density	MFA	MOE
Tangential Shrinkage	0.45**	0.51**	-0.52**	0.55**
Cellulose		0.38**	-0.42**	0.45**
Density			-0.83**	0.91**
MFA				-0.97**

Table 7: Forward stepwise multiple regressions for predicting tangential shrinkage after reconditioning to 12% MC using the equally weighted shrinkage data set (Model 1) and using the full data set (Model 2)

Tabelle 7 Schrittweise multiple Regressionen mit Vorwärtsselektion zur Bestimmung des tangentialen Schwindmaßes nach Rücktrocknung auf 12 % Holzfeuchte anhand der gleich gewichteten Schwindmaße (Modell 1) und anhand aller Daten (Modell 2)

Model 1	Beta	p-value
Constant (intercept)	-26.11	
Cellulose content	0.679	<0.001
MOE	0.382	<0.001
Model 2		
Constant (intercept)	-3.917	
Cellulose content	0.192	<0.001
MOE	0.274	<0.001

Figure Captions

Fig.1: Core marked into 4 quarters with respective mid-points marked with a dot

Fig.2: Dried core sample with 8 marked measured points. Crosses indicate observed points of maximum tangential shrinkage points. Dots indicate the mid-points of each quarter

Fig.3: Results of the cross validation for MFA (a), density (b) and MOE (c)

Fig.4: Variation of predicted cellulose content, density, MFA and MOE from bark to pith in core No. 209

Fig.5: Plot of the observed and predicted tangential shrinkage to 12% MC AR using the regression Model 1 applied on the equally weighed data set

Fig.6: Plot of the observed and predicted values of tangential shrinkage to 12% MC AR for the regression Model 1 applied on the whole data set

Fig.7: Variation of predicted tangential shrinkage in 4 cores indicating non-recoverable collapse

Abb.1 In vier Teile unterteilter Kern. Der Punkt gibt die Mitte eines jeden Viertels an

Abb. 2 Getrockneter Prüfkörper mit acht markierten Messstellen. Die 4 Punkte und 4 Kreuze kennzeichnen die Stellen mit dem höchsten erkannten Schwindmaß. Die Punkte stellen die Mitte eines jeden Viertels dar.

Abb. 3 Zusammenhang zwischen mit SilviScan gemessenen und bestimmten Werte des a) Mikrofibrillenwinkels (MFA), b) der Dichte und c) des Elastizitätsmoduls (MOE)

Abb. 4 Verlauf des bestimmten Zellulosegehalts, der Dichte, des Mikrofibrillenwinkels und des Elastizitätsmoduls von der Rinde zur Markröhre in Kernnr. 209

Abb. 5 Zusammenhang zwischen gemessenem und mittels Regressionsmodell 1 und dem gleich gewichteten Datensatz bestimmtem tangentialen Schwindmaß nach Rücktrocknung auf 12 % Holzfeuchte

Abb. 6 Zusammenhang zwischen gemessenem und mittels Regressionsmodell 1 auf Basis aller Daten Datensatz bestimmtem tangentialen Schwindmaß nach Rücktrocknung auf 12 % Holzfeuchte

Abb. 7 Verlauf des bestimmten tangentialen Schwindmaßes bei 4 Kernen, der auf nicht rückverformbaren Kollaps hinweist

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