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Wood Quality Assessment of *Pinus radiata* (radiata pine) Saplings by Dynamic Mechanical Analysis

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Keywords

Cell-wall composition, compression wood, dynamic mechanical analysis (DMA), *Pinus radiata*, wood quality

Abstract

The use of dynamic mechanical analysis was explored as a possible method of screening for wood quality in breeding programmes. Viscoelastic properties along the grain of wood from 18-month-old *Pinus radiata* saplings were measured using a humidity-controlled dynamic mechanical analyser. Storage modulus and tanδ were determined independently for opposite wood (OW) and compression wood (CW) in 25 trees in the temperature range from 10 to 45°C at 5°C intervals at three frequencies (0.1, 1 and 10 Hz) at constant moisture content of 9%. Storage modulus and tanδ were frequency and temperature dependent. The two wood types did not differ significantly in their storage modulus. But OW exhibited significantly higher tanδ values than CW. The relationship of viscoelastic properties with physical (acoustic velocity, basic density and longitudinal shrinkage) and chemical wood properties was explored. There was a strong correlation (R=0.76) between storage modulus and dynamic MOE (measured by acoustics). In addition, tanδ was positively correlated with longitudinal shrinkage. Monosaccharide compositions of the cell-wall polysaccharides and lignin contents were determined and showed significant differences in the relative proportion of major cell-wall components in OW and CW. Correlations between tanδ and xylose, originating from heteroxylans, and lignin content were found for CW, suggesting that the damping behaviour of cell-walls is controlled by the matrix between cellulose fibril aggregates.

Introduction

Pinus radiata (radiata pine) is widely grown as a plantation tree, especially in New Zealand (Walker 2013). Wood properties vary significantly between trees (Walker 2006) and a large part of this variability is under genetic control (Baltunis et al. 2007). Hence there are opportunities to improve wood properties through selective breeding (Harris 1981). Greater economic gains can be achieved if mass screening is performed at an early age, focusing on wood quality thresholds, below which sawn timber will have little value due to low stiffness and dimensional instability (Apiolaza et al. 2013). This relates particularly to the highly

variable, poor quality juvenile corewood i.e. the first formed rings at the base of the stem (Burdon et al. 2004). It also relates to the presence of compression wood (CW), which is formed in softwoods usually as a reaction to the stem being displaced from the vertical (Scurfield 1973). Compression wood is prevalent in juvenile corewood due to crown asymmetries and stem malformation found in seedlings and young trees (Apiolaza et al. 2008). Corewood constitutes around 50% of the merchantable timber in a 25 year old tree grown in New Zealand (Cown et al. 1991). Shorter breeding cycles should outweigh the lower accuracy of early selection (Apiolaza 2009). To screen effectively it is necessary to understand the underlying wood characteristics that most influence stiffness and dimensional stability.

Cellulose microfibril angle (MFA) was proven to be a better predictor of stiffness than basic density in young trees (Cave and Walker 1994). The cell-wall matrix of lignin and non-cellulosic polysaccharides, including hemicelluloses, are also thought to influence solid wood properties such as shrinkage (Cave 1972). At high MFAs, which occur in corewood and CW, the effect of the cell-wall matrix on wood properties was predicted to be particularly pronounced (Xu et al. 2011; Yamamoto and Kojima 2002). In compression wood, which has MFAs comparable to those in corewood (Wooten et al. 1967), higher longitudinal shrinkage can be attributed to differences in the structure and composition of the cell wall. In particular, a (1→4)-β-galactan, a matrix polysaccharide, has been implicated in high longitudinal shrinkage (Brennan et al. 2012; Floyd 2005).

For mass screening, tools are required which must be reliable, rapid and cheap. Acoustic velocity has proven to be an effective predictor of stiffness both in the field and laboratory, and is rapid and cheap (Huang et al. 2003). Acoustics have been used with very young fast-grown radiata pine clones (Lindström et al. 2002). However, quantification of the monosaccharide compositions of the cell-wall polysaccharides and lignin content classically determined by wet chemistry are much slower and more expensive. Therefore, practical and rapid methods are desirable to either correlate or directly measure these compositions. Dynamic mechanical analysis (DMA) relates mechanical properties like storage modulus and the damping factor (tanδ) to changes at the structural level in viscoelastic materials. Provided the measurements are carried out at constant moisture content, DMA measurements can be used to explore the hydroscopic polymers in the cell-wall matrix (Kelley et al. 1987). DMA measurements are reasonably quick and assess parameters associated with shrinkage i.e.

stiffness and the cell-wall matrix and are therefore worth exploring for assessing wood quality.

Wood cell walls are composed of cellulose microfibrils embedded in a matrix of non-cellulosic polysaccharides and lignin. Galactoglucomannans are the predominant non-cellulosic polysaccharides of softwoods, with heteroxylans being present in smaller amounts (Harris and Stone 2008). The heteroxylans are apparently associated with linear lignin molecules, whereas the galactoglucomannans are associated with more branched lignins (Lawoko et al. 2005). Furthermore, the galactoglucomannans are in closer proximity to the cellulose microfibrils than to the heteroxylans (Altaner et al. 2006; Salmén and Burgert 2009). The amount and type of lignin also differs in CW and opposite wood (OW - the wood formed in the stem opposite to compression wood), with CW having a higher content of lignin that contains *p*-hydroxyphenyl (H) units in addition to guaiacyl (G) units (Brennan et al. 2012).

The dynamic mechanical behaviour of wood has been recently reviewed by (Havimo 2009). As is typical for polymeric materials, three peaks in tanδ are observed between -200 and +200°C; the so called γ- (~-80°C), β- (~-50 to +50°C) and α-peaks (~+75 to +120°C). All three tanδ peaks shift to higher temperatures and decrease in magnitude with decreasing moisture content (Havimo 2009). For example the α-peak is found between ~+75 to +120°C under fully saturated conditions and ~+150 to +230°C in dry conditions. Water acts as a plasticiser and according to Entwistle (2005), energy losses arise due to stress-induced modifications in the hydrogen bonded network involving both water and matrix polysaccharides. The β-peak has been associated with the motion of adsorbed water (Kelley et al. 1987) and also the hydrophilic cell-wall hemicelluloses (Backman and Lindberg 2001). The α-peak is mostly attributed to the softening of lignin. However, Kelley et al. (1987) point out that the α-peak is complex and that hemicelluloses are likely to contribute to this peak. Irvine (1984) reported that the glass transition temperature of hemicellulose in *Pinus radiata* varied from 40ºC at 20% moisture content to 100ºC at 8%. This is in agreement with Olsson and Salmén (2004) who have made DMA measurements on xylan coatings on inert glass braids and found that glass transition temperature varied from 30ºC at 30% water content to 80ºC at 15%. The dynamic properties of cell-wall components and their relative volume fractions have been reported to be the determinant factors of viscoelastic properties of wood (Brémaud et al. 2013; Obataya et al. 1998). Additionally MFA, cellulose crystallinity and cell size have been reported to influence tanδ (Havimo 2009).

The present study explores the potential of using viscoelastic properties to characterise wood properties of CW and OW in tilted radiata pine saplings for wood quality screening. In particular, the effect was investigated of cell-wall composition, as determined using wet chemistry, on the physical wood properties.

Materials and methods

Samples for this study were a subset of a multi-family radiata pine breeding trial established in 2007 at Amberley, Canterbury, New Zealand, to explore very early screening of genotypes for superior stiffness and stability of juvenile corewood (Apiolaza et al. 2011). Twenty five 18-month-old radiata pine trees were tilted at an angle of 35° after one year of growth in the field to produce opposite wood (OW) on the upper side and compression wood (CW) on the lower side of the stem. The average diameter of these saplings ranged from 20 mm to 50 mm with an average of 36 mm. Bolts (100 mm long) were cut from the bases of the trees and sawn lengthwise to separate CW and OW segments. Density and stiffness of the segments were measured after conditioning them in a controlled environment room (20°C and 65% relative humidity); this resulted in ~12% moisture content. Densities were calculated from the dry and green masses and volumes, which were determined by immersion weighing in water. Acoustic velocity was determined from the resonance frequency of the longitudinal vibrations. The resonance frequency was obtained using the "WoodSpec" (Industrial Research Ltd., New Zealand) tool. Longitudinal shrinkage (green to air-dry) was determined from the original length and a precise measurement in the change in length ($\delta\ell$) by measuring the displacement between two spherical headed pins on opposite ends of the sample before and after drying under the conditions mentioned above. A more detailed description of the experimental procedures has been published by (Chauhan et al. 2013).

Subsequently a 35 (longitudinal) x10 (tangential) x1.5 (radial) mm sample was prepared from each specimen close to the cambium for DMA. Samples were conditioned until of constant weight in a humidity controlled chamber over a saturated aqueous solution of potassium nitrate maintaining 50% relative humidity at 20˚C that corresponds to ~9% moisture content in the sample as estimated according to (Stamm 1964).

The specimens were stressed in radial direction as an end-loaded single cantilever with the effective span length of 17.5 mm in a DMA Q800 (TA Instruments, Delaware) equipped with a humidity controlled chamber. Samples were clamped on their tangential face with a

clamping torque of ~1 Nm. Displacement amplitude of 15 μm was applied to the free end and properties were measured from 10 to 45°C at 5°C intervals at 0.1, 1 and 10 Hz. The humidity control was programmed to achieve a minimal cell-wall water loss during the experiment. Initially, the relative humidity in the chamber was maintained at 50%, and with every 5°C increase in temperature, humidity was programmed to increase by 1% and equilibrated for 30 minutes to maintain the constant moisture content of the sample. Samples were weighed before and after each DMA run to monitor any change in moisture content. The change in sample weight after the DMA run was no more than 0.5%, corresponding to a moisture content change from 9% to 8.4%.

The lignin contents and monosaccharide compositions of the wood polysaccharides were determined using a scaled-down two-stage sulphuric acid hydrolysis of milled samples modified from TAPPI standard T-249 cm-85 (1985). The washed and dried residues were weighed to give the Klason lignin contents. The neutral monosaccharides and uronic acids released were separated and quantified using high-performance anion-exchange chromatography with pulsed amperometric detection as described in (Brennan et al. 2012).

Statistical analysis of the data was performed with the R software package (R Development Core Team 2011). ANOVA was used to determine the significance (α=0.05) of the effect of wood type (OW and CW) on the wood properties applying a linear model and assuming that the residuals were identical and independently normally distributed. Linear regression analysis was performed using tanδ values to predict physical properties and chemical components.

Results and discussion

DMA

Changes in storage modulus and tanδ with temperature at three frequencies in OW and CW at 9% moisture content are given in the range 10 to 45°C in Fig. 1. The tanδ measurements at low frequency indicate that the curves are complex with a minor β-peak at ~15°C sitting on the side of the major α-peak centred above 45°C. This is consistent with observations for other softwoods at air-dry condition, for which tanδ peaks have been described at these temperatures (Backman and Lindberg 2001; Kelley et al. 1987). Measurements of tanδ over temperature require adjustments of the relative humidity to keep the moisture content of the samples constant (Stamm 1964) and previously reported tanδ measurements other than at

oven-dry or full saturation are often confounded by moisture loss. The β-peak is highly dependent upon moisture content and can become indistinguishable at lower moisture contents (Havimo 2009). Increasing frequency not only shifts the α-peak to higher frequencies but also increases its intensity (Havimo 2009). Therefore it is possible that the better resolution of the β-peak at low frequencies observed here is a result of the reduced intensity of the α-peak. Furthermore the increased cross-linking of CW lignin should result in a higher softening temperature (Olsson and Salmén 1997). However, Placet et al. (2007) reported a lower glass transition temperature for green CW tested in the transverse direction compared to normal wood, an observation counter intuitive to the present experiment for CW at 9% moisture content (Fig. 1).

The two wood types differed significantly in tanδ values (Table 1 and Fig. 2). Lower damping in CW was consistent with an earlier study (Brémaud et al. 2013). As MFA is positively correlated with tanδ the lower damping in CW is likely to be caused by the different proportions or structures of the cell-wall polymers, or the anatomical features of the tracheids (Brémaud et al. 2013). This can also be inferred from the negative correlation between acoustic velocity squared, which is inversely related to MFA, and tanδ in OW and CW (Table 2) and the absence of a significant difference in acoustic velocity between OW and CW (Table 1). At comparable specific modulus (storage modulus/density) tanδ in CW was lower than in OW (Fig. 3). Olsson and Salmén (1997) reported that the mobility of lignin increased with the amount of methoxyl groups. As CW lignin is more abundant, more condensed and bears fewer methoxy groups than OW lignin, its mobility will be reduced. Furuta et al. (2010) suggested that the thermal softening temperature of water-swollen wood is more affected by change in quantity and quality of lignin than by differences in the hemicelluloses.

CORRELATION BETWEEN DMA AND WOOD PROPERTIES

The summary statistics of measured physical and chemical compositions for CW and OW are shown in Tables 1 and 4, respectively. The values were similar to those reported earlier (Apiolaza et al. 2011; Brennan et al. 2012; Chauhan et al. 2013). Consistent with these reports, OW and CW of two-year-old *Pinus radiata* differed significantly in all measured wood properties except acoustic velocity. The absence of a significant difference in acoustic

velocity between OW and CW could be explained by the fact that both wood types have a comparable MFA in young trees (Donaldson et al. 2004; Wooten et al. 1967).

Correlation of viscoleastic properties with physical properties and chemical compositions are given in Tables 2 and 3, respectively. A strong correlation between storage modulus measured by DMA and dynamic MOE measured by acoustics and immersion weighing was found (Table 3). The higher storage modulus of CW can be attributed to its higher basic density as the storage modulus normalised for basic density did not differ significantly between the two wood types.

The correlation of DMA measurements to physical properties or chemical compositions depended on the temperature and frequency of the measurements (Table 2 and 3). DMA is capable of predicting longitudinal shrinkage and dynamic MOE in OW and CW. For wood quality selection at an early stage especially the high correlation (R = 0.78) between tanδ and longitudinal shrinkage in OW is useful, which is less strongly correlated to acoustic velocity or stiffness (Chauhan et al. 2013). Correlations between viscoelastic properties and chemical composition of wood were fewer and predominantly found for CW. The lignin content in particular but also the xylose and 4-*O*-methylglucuronic acid content are correlated with tanδ (Table 3). These two monosaccharides are derived from the heteroxylans (4-O-methylglucuronoarabinoxylans).

The cell wall 'matrix' has been suggested to influence wood properties (Xu et al. 2011; Yamamoto and Kojima 2002). More specifically it has been shown that lignin is the most viscoelastic cell wall polymer especially at low moisture content (Akerholm and Salmén 2004). Heteroxylans have been shown to be in spatial and mechanical association with lignin forming a lignin-heteroxylan complex between the cellulose microfibril aggregates (Altaner et al. 2006; Salmén and Burgert 2009). This is consistent with the here reported results which show a correlation between heteroxylan contents and tanδ in compression wood (Table 3).

Conclusion

The damping behaviour of compression wood was, apart from MFA, influenced by the quantity of lignin and heteroxylans. Other cell-wall components, i.e. cellulose fibrils surrounded by galactoglucomannans, seemed to have no significant effect in the observed temperature range.

DMA measurements correlated strongly with longitudinal shrinkage and dynamic MOE of wood from *Pinus radiata* saplings. Tanδ was also correlated with cell-wall composition in CW samples, where there were correlations with lignin and xylose contents. The measurement of acoustic velocity is an effective way of predicting wood quality in saplings of *Pinus radiata*. However if lignin (or heteroxylan) contents or a higher accuracy of longitudinal shrinkage assessment is required, measurements of tanδ could be a viable alternative.

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Declaration

The authors declare that they have no conflict of interest.

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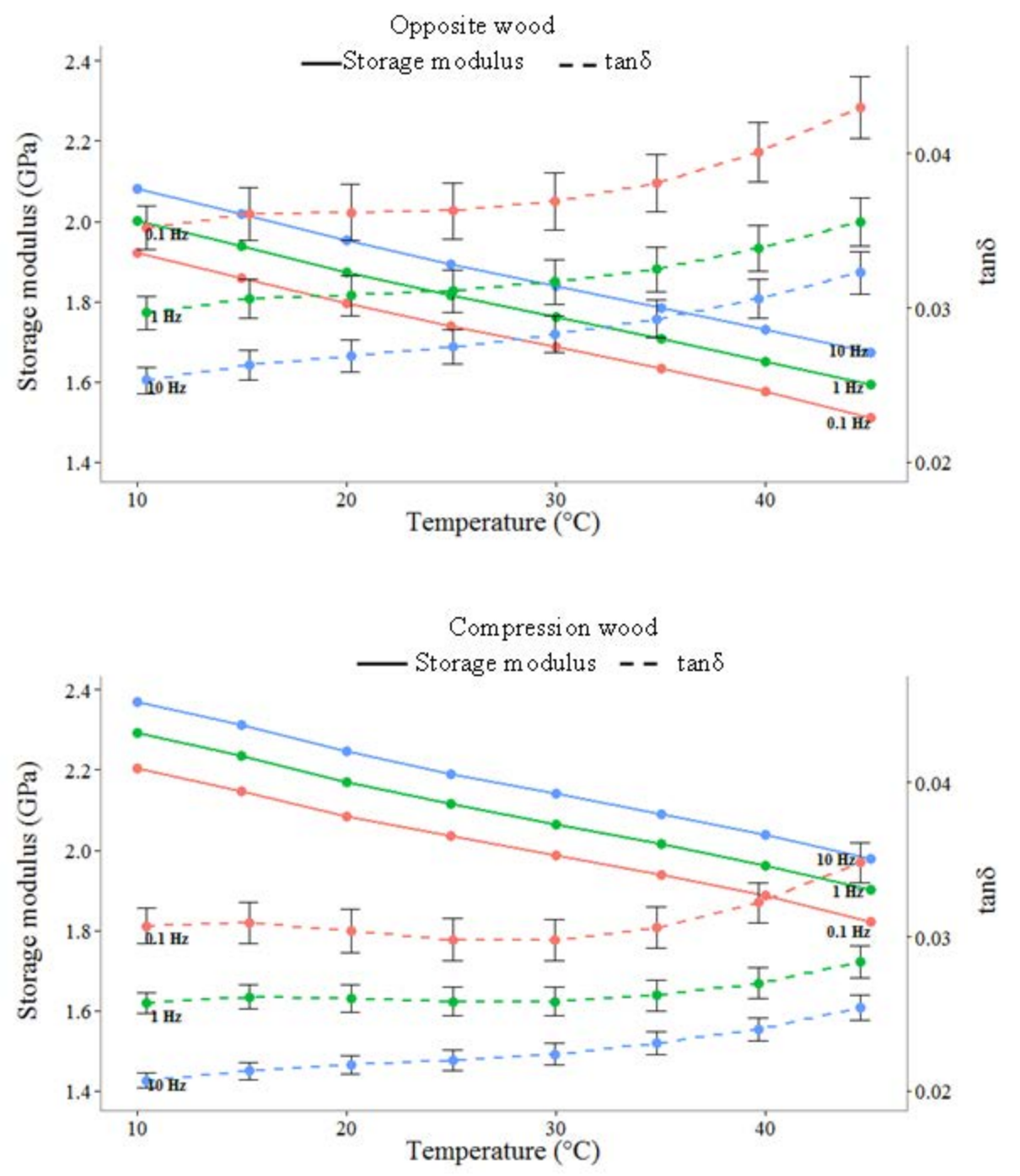
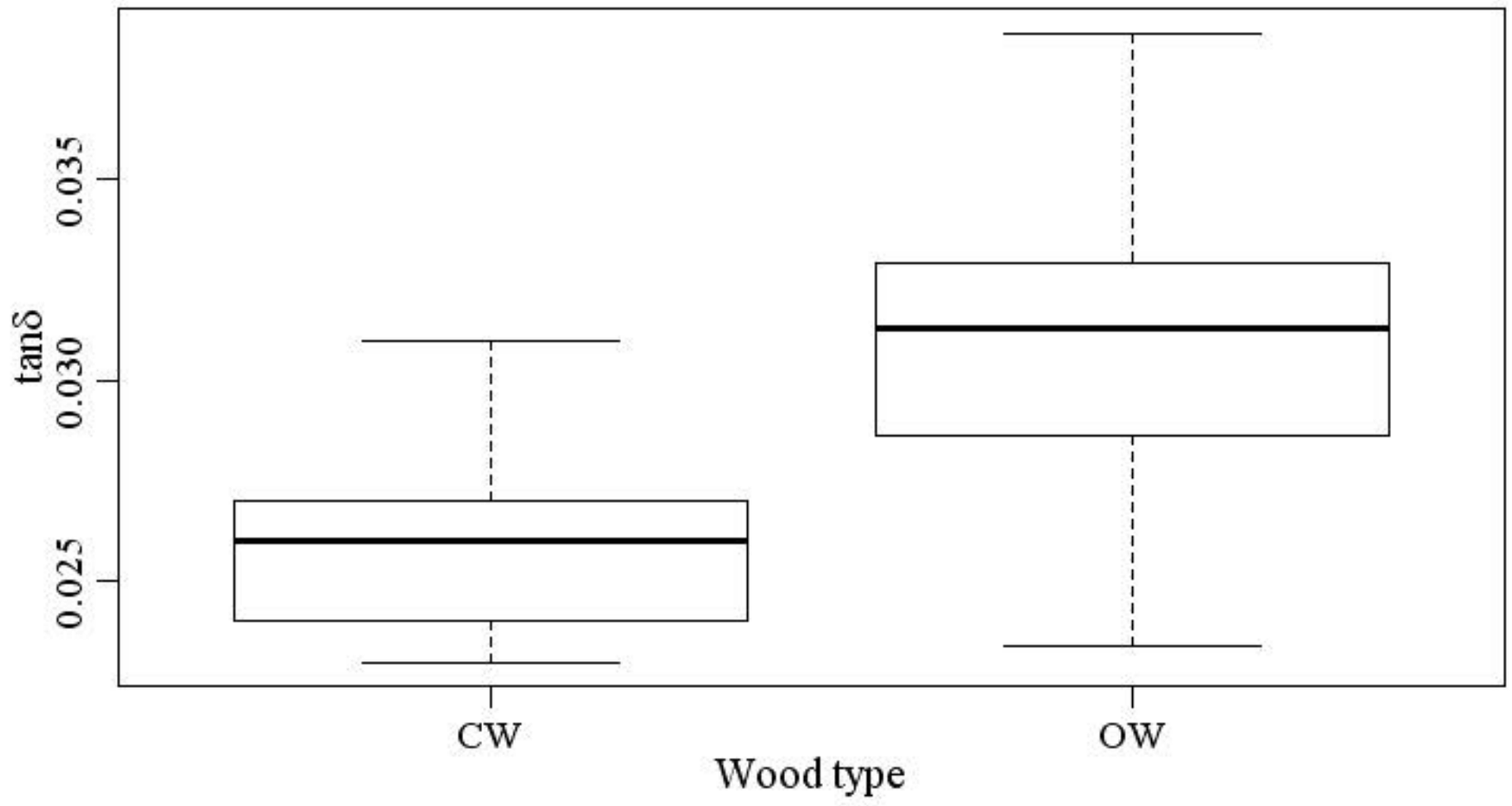
Figure caption

Fig. 1 Variation in mechanical properties with temperature at three different frequencies in opposite wood (top) and compression wood (bottom) in *Pinus radiata* saplings at 9% moisture content. Average of 25 measurements. Error bars displaying the 95% confidence interval for the tanδ curves. The large variation arises from different intercepts of the curves, and not the shape of the curves which is statistically well described by a cubic model.

Fig. 2 Box and whisker plot for tanδ at 1 Hz and 25°C for compression wood and opposite wood from *Pinus radiata* saplings (n=25). Box: 25th and 75th percentiles; bars: min and max values; horizontal line: median

Fig. 3 Relationship between damping coefficient (tanδ) and specific storage modulus at 1 Hz and 25°C in opposite wood and compression wood in saplings of *Pinus radiata*


Opposite wood
Storage modulus
tanδ
Storage modulus (GPa)
tanδ
Temperature (°C)
0.1 Hz
1 Hz
10 Hz
Compression wood
Storage modulus
tanδ
Storage modulus (GPa)
tanδ
Temperature (°C)
10 Hz
1 Hz
0.1 Hz


tanδ
0.035
0.030
0.025
CW
OW
Wood type

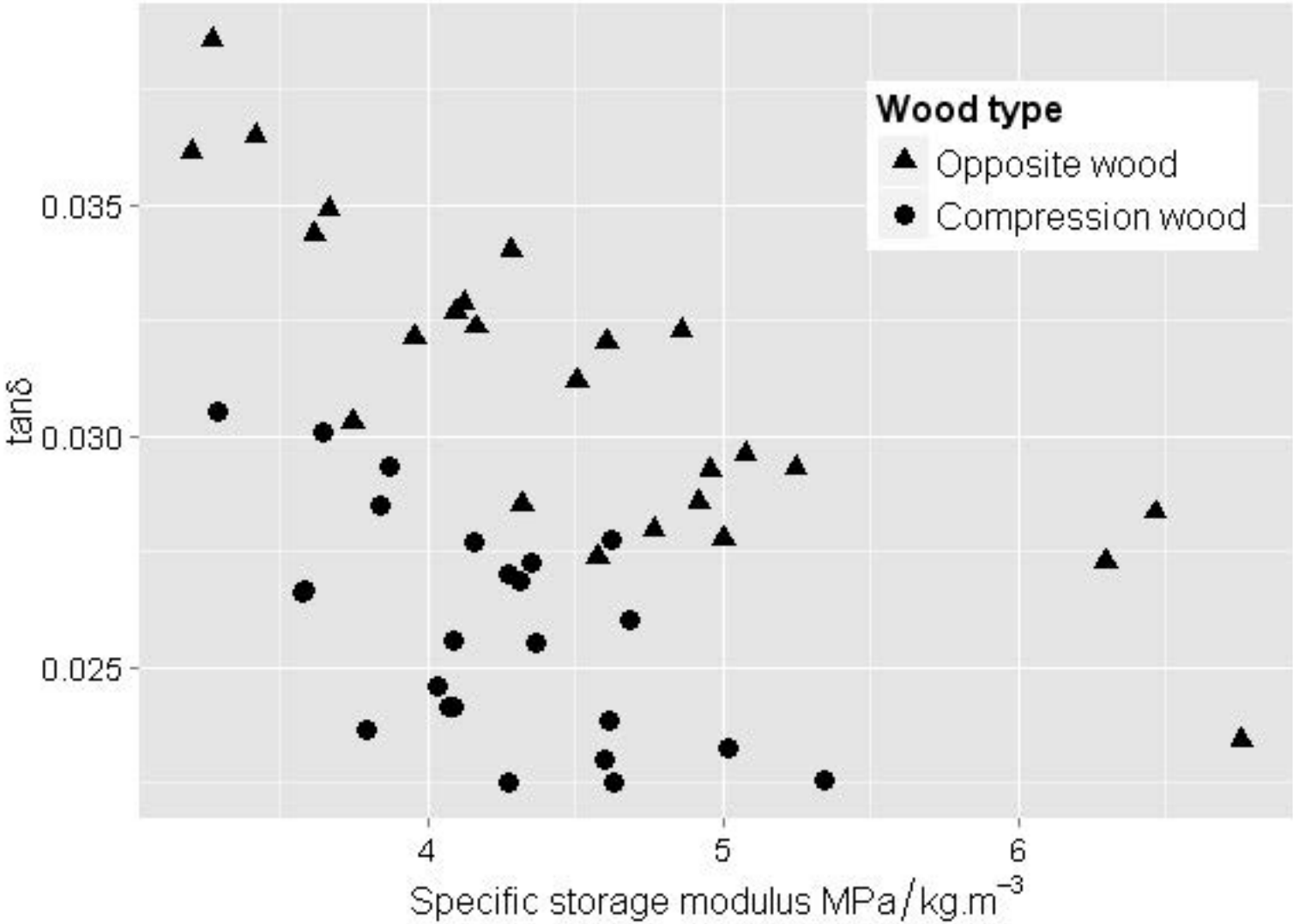

Wood type
Opposite wood
Compression wood
tanδ
0.035
0.030
0.025
4
5
6
Specific storage modulus MPa/kg.m^{-3}

Table 1. Summary statistics of physical and viscoelastic properties for opposite wood (OW) and compression wood (CW) in young *Pinus radiata,* n=25. In each row, mean values with a common lowercase letter are not significantly (α=0.05) different.

	Compression wood		Opposite wood	
Properties	Mean	CV%	Mean	CV (%)
Storage modulus (GPa)*	2.1_a	15	1.8_b	28
tanδ*	0.026_a	9	0.031_b	11
Acoustic velocity squared $(km/s)^2$	4.90_a	10	4.97_a	12
Basic density (kg/m^3)	412_a	12	309_b	11
Dynamic MoE (GPa)	2.5_a	17	2.1_b	21
Longitudinal shrinkage (%)	1.0_a	27	0.7_b	35
Volumetric shrinkage (%)	9.5_a	24	15.8_b	23

*Measured at 25˚C, 1Hz and 9% moisture content

Table 2 Significant correlation between viscoelastic and physical properties in opposite (OW) and compression wood (CW) of young *Pinus radiata*. The significant Pearson's correlation ($p<0.05$) along with frequency (Hz) / temperature (˚C) at which the maximum correlation was found is given in brackets. NA: no significant correlation at any frequency temperature combination

	Wood type	Acoustic velocity squared ($km^2\ s^{-2}$)	Basic density ($kg\ m^{-3}$)	Dynamic MOE (GPa)	Longitudinal shrinkage (%)	Volumetric shrinkage (%)
Storage modulus (GPa)	OW	0.65 (0.1/45)	0.63 (1/15)	0.79 (0.1/45)	-0.45 (0.1/45)	NA
	CW	NA	0.56 (10/10)	0.74 (0.1/35)	NA	NA
tanδ	OW	-0.83 (10/10)	-0.41* (1/15)	-0.75 (0.1/10)	0.78 (10/10)	0.41* (10/10)
	CW	-0.63 (1/15)	NA	-0.53 (0.1/10)	0.45 (10/45)	NA

*Only significant at given frequency temperature combination; all other correlations are significant at all frequency and temperature combinations.

Table 3 Significant correlation between viscoelastic and chemical components in opposite (OW) and compression wood (CW) of young *Pinus radiata*. The significant Pearson's correlation ($p<0.05$) along with frequency (Hz) / temperature (˚C) at which the maximum correlation was found is given in brackets. NA: no significant correlation at any frequency temperature combination; GalA: galacturonic acid; 4OMeGlA: 4-*O*-methylglucuronic acid.

	Wood type	Lignin	Arabinose	Xylose	Mannose	Galactose	Glucose	GalA	4OMeGlA
Storage modulus (GPa)	OW	NA	NA	NA	NA	NA	NA	NA	NA
	CW	0.42 (0.1/45)	NA	0.41 (0.1/30)	NA	NA	NA	NA	0.42 (0.1/30)
tanδ	OW	NA	0.44 (0.1/45)	NA	NA	NA	NA	NA	NA
	CW	-0.81* (1/25)	NA	-0.50 (0.1/15)	NA	NA	NA	NA	-0.57 (0.1/15)

* Significant for all frequency and temperature combinations; all other correlations are significant for some frequency and temperature combinations only.

Table 4 Summary statistics for the chemical components (lignin content and monosaccharide compositions of the cell-wall polysaccharides) in opposite wood and compression wood of *Pinus radiata* saplings, n=25. In each row, mean values with a different lowercase letter are significantly (α=0.05) different. GalA: galacturonic acid; 4OMeGlA: 4-*O*-methylglucuronic acid.

	Compression wood		Opposite wood	
Lignin content & monosaccharide components	Mean	CV (%)	Mean	CV (%)
Lignin	37.1_a	4	27.7_b	4
Arabinose	1.1_a	16	1.7_b	13
Xylose	5.7_a	10	9.3_b	10
Mannose	6.9_a	12	10.1_b	8
Galactose	11.7_a	14	3.5_b	22
Glucose	33.0_a	7	39.5_b	5
GalA	0.8_a	11	1.1_b	14
4OMeGlcA	0.1_a	19	1.7_b	12