Estimation of clear wood properties by near infrared spectroscopy

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Introduction

Rapid, cost-effective methods of measuring wood quality are extremely important to tree improvement programmes where it is necessary to test large numbers of trees. Non-destructive sampling of a forest can be achieved by using increment cores generally taken at breast height. At CSIRO Forestry and Forest Products (CSIRO-FFP) methods for the rapid, non-destructive measurement of wood properties (SilviScan-1 and -2 and acoustic methods) and wood chemistry (near infrared spectroscopy) based on increment cores and other samples have been developed.

Recently, a series of experiments have been conducted at CSIRO-FFP that have used near infrared (NIR) spectroscopy to estimate several economically important wood properties such as density, wood stiffness (longitudinal modulus of elasticity— E_L) and microfibril angle (MFA). Experiments were conducted on a hardwood and a softwood species, the two species combined and samples representing a number of different species from around the world, with the aim of developing calibrations that could be applied to increment core samples. In this paper the results of these experiments are reported.

Materials and methods

Sample selection

Eucalyptus delegatensis: 50 dried clear wood (defect-free) samples of mature *E. delegatensis* trees from native forest in East Gippsland, Victoria, Australia.

Pinus radiata: 50 dried clear wood samples of mature *P. radiata* trees from Tallaganda, Canberra, Australia. Schimleck *et al.*^{1,2} describes the origin of the *E. delegatensis* and *P. radiata* samples in detail.

Mixed species: 59 samples that had an extremely wide range of wood properties and included many commercially important timber species from around the world. Schimleck *et al.*³ gives details of the wood properties of these species.

Wood properties

Dried clear wood samples were equilibrated to approximately 12% moisture content and cut to 20 mm × 20 mm transversely and 300 mm longitudinally. Dimensions and masses of these samples were used to calculate their average air-dry densities (D_{stick}). Dynamic elastic modulus was determined using the natural frequency of vibration along the fibre direction.⁴ After measurement of E_L , a small strip (2 mm tangentially, 7 mm longitudinally, ~ 20 mm radially) was cut from one end of each sample

for MFA analysis (by scanning x-ray diffractometry) on SilviScan- 2^5 and NIR analysis. Dimensions and masses of the SilviScan-2 test samples were used to calculate their average air-dry densities (D_{strip}).

Near infrared spectroscopy

NIR diffuse reflectance spectra were obtained from the radial/longitudinal face of each sample using a NIRSystems Inc. Model 5000 scanning spectrophotometer. Samples were held in a custom-made holder.¹ A mask was used to ensure that a constant area was tested. The spectra were collected at 2 nm intervals over the wavelength range 1100–2500 nm. One spectrum was obtained per sample. Second derivative spectra were used for the development of calibrations. A segment width of 10 nm and a gap width of 20 nm were used for the conversion.

Calibration

Calibrations were developed for each wood property using partial least squares (PLS) regression. The *E. delegatensis* and *P. radiata* sample sets were divided, at random, into calibration and prediction sets as follows.

E. delegatensis: calibration set 70 samples, prediction set 34 samples.

P. radiata: calibration set 70 samples, prediction set 34 samples.

Hardwood–softwood (*E. delegatensis* + *P. radiata*): calibration set 140 samples, prediction set 68 samples.

Mixed species: all samples were used to develop calibrations that were then tested on selected samples from the *E. delegatensis* (50 samples) and *P. radiata* (49) sample sets. One *P. radiata* sample was omitted due to fungal staining.

NSAS software (version 3.52)⁶ was used to develop calibrations using four cross-validation segments and a maximum of ten factors. In most cases the number of factors recommended by the software was used. For the *E. delegatensis* D_{strip} calibration, eight factors were recommended but only five factors are reported, as the extra factors marginally improved the standard error of prediction. For the mixed species set the calibrations reported gave the best results in prediction on the *E. delegatensis* and *P. radiata* samples.

Calibration statistics

The measure of how well a calibration fits the data is the standard error of calibration (*SEC*). The measure of how well the calibration predicts the trait of interest for a set of unknown samples that are different from the calibration set is given by the standard error of prediction (*SEP*).

Results and discussion

Individual species

Good calibrations were obtained for each physical property examined using NIR spectra obtained from the *E. delegatensis* and *P. radiata* samples (Table 1). Coefficients of determination (R^2) ranging from 0.77 for MFA to 0.93 for D_{stick} were obtained for *E. delegatensis* and R^2 ranging from 0.69 for D_{stick} to 0.94 for D_{strip} were obtained for *P. radiata*. *SEP* results were similar to the *SEC*'s, indicating that calibrations based on NIR spectra of solid wood can be used to rapidly predict solid wood properties in a prediction set.

E. delegatensis and P. radiata

The calibrations obtained for each solid wood property, using the combined *E. delegatensis* and *P. radiata* sample set, were also good (Table 2) with R^2 similar to those of the individual species. The

Table 1. Summary statistics of solid wood property calibrations developed for the *E. delegatensis* and the *P. radiata* sample sets. $D_{stick} = air$ -dry density of the stick samples, $D_{strip} = air$ -dry density of the strip samples, $E_L = longitudinal modulus$ of elasticity of the stick samples, MFA = average microfibril angle of the strip samples.

Species	Parameter	Factors	R^2 (calib. ⁿ)	SEC	R^2 (pred. ⁿ)	SEP
E. delegatensis	D _{stick} (kg m ³)	7	0.93	25.9	0.92	30.6
	D _{strip} (kg m ³)	5	0.92	26.4	0.91	31.9
	E _L (GPa)*	4	0.90	1.5	0.88	1.6
	MFA (deg)	3	0.77	1.3	0.74	1.7
P. radiata	D _{stick} (kg m ³)	2	0.69	22.9	0.71^{*}	21.8
	D _{strip} (kg m ³)	7	0.94	12.5	0.85	17.3
	E _L (GPa)	2	0.82	1.0	0.75	1.1
	MFA (deg)	3	0.78	1.9	0.64	2.1

*Note 69 samples were used to develop the *E. delegatensis* E_L calibration and two samples were deleted from the *P. radiata* prediction set when D_{uick} was estimated

Table 2. Summary statistics of solid wood property calibrations developed for the E. delegatensis and
<i>P. radiata</i> sample set.

Parameter	Factors	R^2 (calib ⁿ)	SEC	R^2 (pred ⁿ)	SEP
D _{stick} (kg m ³)	8	0.97	27.0	0.95*	30.8
D _{strip} (kg m ³)	7	0.97	28.3	0.95	31.4
E _L (GPa)	6	0.92	1.5	0.89	1.5
MFA (deg)	8	0.86	1.6	0.71	1.9

*Note two samples were deleted from the prediction set when D_{stick} was estimated

SEC's were similar to those obtained for *E. delegatensis*. These results suggest that it may be possible to develop general calibrations based on samples from a number of species of a single genus or samples from a number of different genera, even if the species exhibit large differences in wood chemistry and anatomy.

Mixed species

Good calibrations were obtained for density and E_L using the mixed species sample set (Table 3). R^2 were slightly lower than those determined using individual species and standard errors were higher. The wide range in the mixed species set has contributed to the large standard error. The calibration developed for MFA had a lower R^2 than was obtained using the *E. delegatensis* and *P. radiata* sets.

The mixed species calibrations provided good estimates of density (stick and strip) and E_L when applied to the *E. delegatensis* and *P. radiata* sets. Predictions of E_L for the *P. radiata* samples were in close agreement with the reference data (Figure 1). Predicted E_L for the *E. delegatensis* set had an *SEP* much higher than the mixed species *SEC*. Six *E. delegatensis* samples had E_L values beyond the range of the mixed species calibration (1.7 to 23.8 GPa) and were poorly predicted. If these samples were re-

	Mixed species calibration		E. delegatensis		P. radiata		
Parameter	# of factors	R^2	SEC	R^2	SEP	R^2	SEP
D _{stick} (kg m ³)	4	0.82	76.2	0.72	65.8	0.67	39.4
D _{strip} (kg m ³)	4	0.83	74.0	0.75	65.4	0.79	40.0
E _L (GPa)	7	0.79	2.14	0.76	3.00	0.75	1.18
MFA (deg)	5	0.52	2.53	0.54	2.19	0.63	3.10

Table 3. Summary statistics for the mixed species calibrations and of predictions made by these calibrations on the *E. delegatensis* and *P. radiata* sample sets.

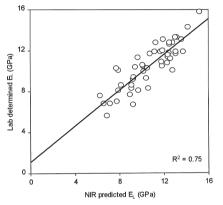


Figure 1. Relationship between laboratory determined E_L and NIR predicted E_L for the *P. radiata* samples. Predictions were made using the seven-factor mixed species E, calibration.

moved, the *SEP* fell to 2.14 GPa ($R^2 = 0.75$). The results indicate that mixed species calibrations, encompassing wide variations in wood anatomy, chemistry and physical properties, can be used for ranking trees.

The selected mixed species samples represent an extreme range and it is unlikely that such variation would be required in practice. For most applications, samples from a small number of species would suffice. It is expected that refinement of calibrations through appropriate sample selection would provide improved calibration statistics, owing to less variation in the calibration set and more accurate predictions of the physical properties of test samples.

An important aspect in the success of this work was that samples used for D_{stick} and E_L determination were well represented by the strips used for calibration. For the *E. delegatensis* samples the R^2 for the linear relationship between

 D_{stick} and D_{strip} was 0.98. For *P. radiata* the R^2 was lower (0.75) but three samples had strip and stick densities that were quite different; exclusion of these samples improved the R^2 to 0.88 (two of these samples were removed from the prediction set for the estimation of D_{stick}). For the mixed species set the R^2 was 0.99.

The results indicate that NIR spectroscopy may be calibrated to estimate several wood properties. The calibrations could be used to estimate the properties of core samples, which would be particularly important to tree improvement programmes. Spectra collected from the prepared surface of cores could be used to provide pith to bark profiles or average properties.

Conclusions

Experiments reported in this paper indicated that useful calibrations for several solid wood properties can be developed using NIR spectroscopy, when appropriately characterised samples are available. NIR spectroscopy offers a rapid and non-destructive alternative to traditional methods of analysis and is applicable to large-scale, non-destructive forest resource assessment and to tree breeding and silvicultural programmes.

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