Prediction of kraft pulp yield in eucalypts: an inter-lab study

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Introduction

One of the commercially important properties of trees is the Kraft pulp yield (KPY) which is a measure of the percentage recovery of pulp fibre following Kraft pulping with sodium hydroxide. The determination of KPY can be employed at several intervention points along the value chain for:

i. generation of phenotypic data to enable selection of elite families/provenances in breeding trials;[1,2]
ii. assessment of the average pulp yield in a standing forest compartment as part of a pre-harvest analysis to aid harvest planning;
iii. assessment of wood chips being delivered to a client for quality control purposes;
iv. measurement of KPY in wood chip feedstock as chips are conveyed into the digester to allow process optimisation via a feed-forward system.

The measurement of KPY is a costly, destructive and time-consuming process. Generally whole trees are destructively sampled, with stem cross-sectional discs taken at intervals up the tree and chipped for laboratory pulping, using approximately 5 kg of dry-weight chips. Similarly grab samples may be obtained from chip piles prior to export. The laboratory pulping itself is a slow process with *ca*. five to six samples (trees/chip samples) per day being analysed. As a consequence it is a very costly process—the pulping component alone can cost several hundred US dollars.

Consequently NIR spectroscopy offers an excellent technique for the rapid prediction of KPY. Research over the last two decades has developed calibration models for a number of commercial species globally[3–7] and recently a global model for the prediction of KPY in Eucalypt species has

been developed.[8] This calibration has in excess of 800 samples in the model, which represents a calibration investment of over $1 m in reference data alone. In this paper we describe an inter-laboratory assessment of KPY calibration with a view to sharing data for calibration purposes.

Materials and methods

Seventy (70) samples of ground (16-mesh) Eucalyptus wood were selected from the total sample set of over 1,200 samples in CSIRO's global KPY model. NIR spectra were recorded on four Bruker MPA instruments at four laboratories involved in NIR prediction of KPY within the Southern Hemisphere, namely (in alphabetical order): CSIRO (Australia); ITC (India); Sappi (South Africa) and the University of Tasmania (Australia). Spectra were recorded at each laboratory using local parameter settings, but typically encompassed at least the range 10,000–4,000 cm^{-1} at 8 cm^{-1} resolution. Partial least squares regression was performed using raw spectra and preprocessing with 1st and 2nd derivative Savitzky-Golay transforms (15 point window, 2nd order polynomial) on the range 4,000–10,000 cm^{-1} (1000–2500 nm). Cross-validation (random 20 segment) was used for model calibration with a maximum of 10 factors allowed, with optimum number of factors chosen automatically to be the first local minimum in residual variance. All analyses were performed using *The Unscrambler* v9.8 (Camo, Norway).

Each sample had corresponding Kraft pulp yield values obtained from laboratory pulping conducted at the CSIRO facility in Clayton, Victoria, Australia. The Kraft cooks were carried out using 3-litre pressure vessels in an electrically heated air bath. All the cooks used the same conditions but the active alkali charge was varied to achieve the desired kappa number (wood charge = 400 g OD, liquor wood ratio = 4:1, sulfidity = 25%, time to temperature = 105 minutes, cooking temperature = 165 °C, H-Factor = 1300). All samples were pulped in triplicate.

Results and discussion

Results are presented in a random order to remove the identity of individual laboratories. The PLS calibration results show good agreement between instruments (Table 1, Figure 1) with calibrations requiring between five and seven principal components to converge for 2nd derivative data (raw

Table 1. NIR calibration statistics, (2nd derivative, 15-point Savitzky-Golay, 2nd order polynomial). Random laboratory assignment.

Lab	PC	Calibration			Prediction		
		RMSEE	*R2*	*RPD*	*RMSEP*	*R2*	*RPD*
A	6	1.4	0.84	2.6	2.1	0.68	1.72
B	5	1.1	0.91	3.2	2.6	0.48	1.37
C	7	1.5	0.80	2.2	2.6	0.45	1.38
D	6	1.4	0.83	2.5	2.2	0.61	1.60
All	7	1.2	0.88	2.3	1.8	0.76	1.45

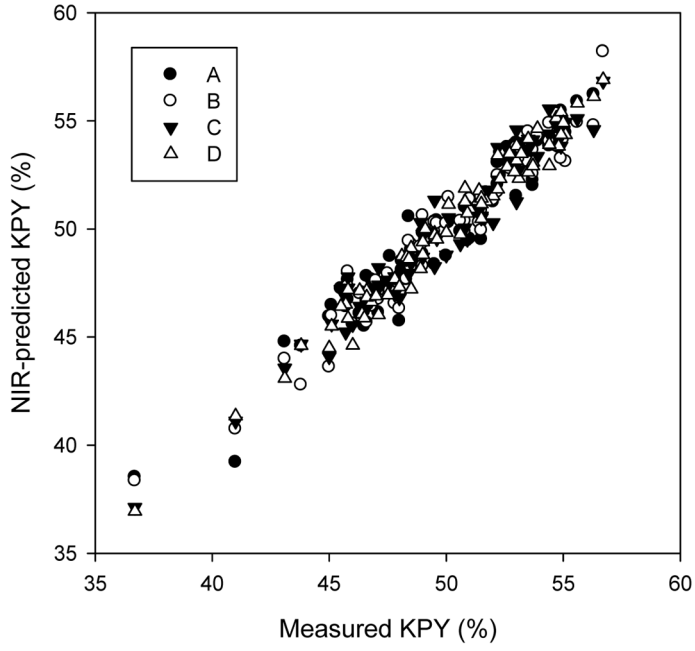

Figure 1. NIR-predicted vs measured plot (cross-validation) of KPY for the four laboratories.

and 1st derivative data not presented). This shows that individually the calibration models developed for all four laboratories are similar.

Table 2 shows the error in prediction made by cross-predicting KPY for each data set using calibration models derived from the four individual laboratory models. This shows that for the most part that there was little bias between the calibration models although there was a larger error in RMSEP between labs A and D; whether the model for A is used to predict data from D or *vice versa*. The results do however show that reliable cross-validated calibration models can be developed on all instruments, irrespective of the individual instrument used. This suggests that there were systematic differences between the laboratories. The combined model developed from all spectra however, shows that these differences were minimal, indicating that it should

Table 2. RMSEP values for data sets predicted using various individual models.

		Calibration model			
		A	B	C	D
Predicted set	A	1.6	2.4	3.9	5.2
	B	2.6	1.8	2.9	3.0
	C	2.9	1.8	1.8	3.7
	D	4.4	3.2	4.0	1.7

be possible to use spectra generated on one or several instruments to develop a single calibration model without the need to physically ship samples between laboratories.

Conclusion

The results show that the use of NIR will allow the prediction of KPY in samples of hardwoods for rapid screening or ranking of material genetic or clonal trials. Improved cross-correlations could be expected from the analysis of a larger number of varied samples. It is possible for NIR laboratories with good instrument operation to be able to calibrate instruments using data from other instruments within the cluster, thereby minimizing the considerable cost of obtaining reference data, which can cost upwards of $1,000 per sample to obtain. The sharing of reference calibration samples will also increase the geographic and species variability that would otherwise be difficult to obtain.

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