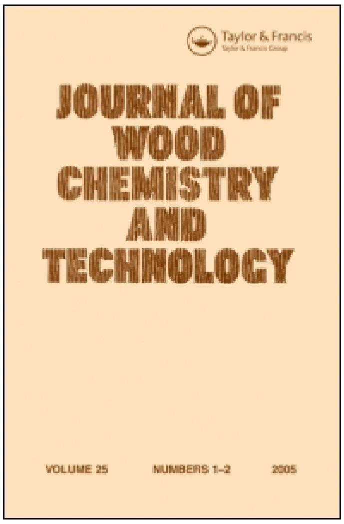

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Extending Near Infrared Reflectance(NIR) Pulp Yield Calibrations to NewSites and Species

Laurence R. Schimleck[a]; Peter D. Kube[b]; Carolyn A. Raymond[c]; Anthony J. Michell[d]; Jim French[e]
[a] Warnell School of Forest Resources, University of Georgia, Athens, Georgia, USA [b] Forestry Tasmania, CSIRO Marine and Atmospheric Research, Hobart, Tasmania, Australia [c] Forests NSW, Land Management and Technology Services, Tumut, NSW, Australia [d] CSIRO Forestry and Forest Products (retired), Victoria, Australia [e] Fibre Technology, Gunns Ltd., Burnie, Tasmania, Australia

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Extending Near Infrared Reflectance (NIR) Pulp Yield Calibrations to New Sites and Species

Laurence R. Schimleck
Warnell School of Forest Resources, University of Georgia, Athens, Georgia, USA

Peter D. Kube
Forestry Tasmania, CSIRO Marine and Atmospheric Research, Castray Esplanade, Hobart, Tasmania, Australia

Carolyn A. Raymond
Forests NSW, Land Management and Technology Services, Tumut, NSW, Australia

Anthony J. Michell
CSIRO Forestry and Forest Products (retired), Victoria, Australia

Jim French
Fibre Technology, Gunns Ltd., Burnie, Tasmania, Australia

Abstract: Recently, we demonstrated that the accuracy of pulp yield predictions for wood samples from a site (Gog) new to their calibration (Tasmania-wide *Eucalyptus nitens*) was greatly improved by adding five Gog samples to the calibration set. In this study we investigated the addition of Gog samples to the Tasmania-wide *E. nitens* set,

Samples used to build the Tasmania-wide *E. nitens* calibration and the *E. globulus* samples were provided by Gunns Ltd. *E. nitens* samples (Gog site) were provided by Forestry Tasmania. Financial support was provided by the CRC for Hardwood Fiber and Paper Science and the CRC for Sustainable Production Forestry. The authors gratefully acknowledge this support.
Address correspondence to Laurence R. Schimleck, Warnell School of Forest Resources, University of Georgia, Athens, GA 30602-2152, USA. E-mail: lschimleck@smokey.forestry.uga.edu

with the aim of further improving predictive accuracy. It was demonstrated that the addition of a single Gog sample to the Tasmania-wide calibration set was sufficient to greatly reduce predictive errors and that the inclusion of at least 3 Gog samples in the Tasmania-wide set was sufficient to give relatively stable predictive errors. The addition of different sets of 5 Gog samples to the Tasmania-wide calibration, however, caused predictive errors to vary between sets. The standard deviation of pulp yield for the prediction set (20 Gog samples) was important, with sets having the largest standard deviations giving the best predictive statistics. Finally, the Tasmania-wide *E. nitens* calibration was enhanced using samples from a different species (*Eucalyptus globulus*) and applied successfully to other *E. globulus* samples.

Keywords: *Eucalyptus globulus*, *Eucalyptus nitens*, kraft pulp yield, near infrared spectroscopy, NIR

INTRODUCTION

Near infrared (NIR) spectroscopy provides an inexpensive and rapid alternative to traditional laboratory methods for determination of wood chemistry.[1−7] Although these studies have shown that NIR spectroscopy can be used to reliably predict chemical wood properties, most studies have used samples from one or only a few sites.

If NIR analysis is to be widely used for routine assessment of wood chemistry, there is a need to determine how to extend existing calibrations to new sample sites or to other related species. It is not possible to compare results across sites if a new and independent calibration is developed for each new sample site. In addition, the cost of undertaking the laboratory chemistry required to develop site-specific calibrations would become prohibitive as the number of sites increases. A more cost-effective solution is to determine how many samples from each new site need to be added to an existing calibration to ensure the calibration is effective and reliable when applied to samples from the new site.

The application of calibrations to samples from sites different to those used to build the calibration has rarely been investigated. Schimleck et al.[6] reported that the use of an existing *Eucalyptus nitens* (Deane and Maiden) Maiden (shining gum) calibration on a different site led to overestimation of pulp yields with the errors of pulp yield estimates being too large for the predicted pulp yields to be indicative of yields measured using laboratory pulping. More recently, Schimleck et al.[8] demonstrated that the accuracy of pulp yield predictions for samples from a new site were greatly improved by adding a small number (five) of the prediction set samples to their calibration set. Although the addition of 5 samples from the new site to the existing calibration set was a successful approach for decreasing prediction error several questions remained, including:

1. How many samples are required from a new site to successfully enhance the existing calibration?

2. Is there a difference between different sets of samples from the new site? If so, how should the sample set be selected?
3. Is it possible to obtain similar results if the existing calibration was applied to samples from a closely related species?

Answers to these questions were sought in this study.

MATERIALS AND METHODS

Sample Origins

Three separate sets of samples were in this study. The first was a large set used to build the main calibration model, and the second and third were smaller sets used to evaluate and enhance calibrations.

The main calibration model (referred to here as the Tasmania-wide calibration) used a total of 126 *E. nitens* whole-tree composite chip samples, of which each represented a single tree. Of these, 88 were taken from 5 sites in northern Tasmania (latitude of about 41°S) and 38 were from one site in southern Tasmania (latitude of about 43°S). These were typical cool-temperate *E. nitens* sites; moderately to highly productive (MAI of 20 to 30 m^3/ha at harvest age), with a rainfall of between 1100 mm and 1600 mm per annum, at an altitude of between 250 m and 500 m, and subject to frequent winter frosts and occasional snow falls. The trees were felled and 1 m long billets were removed from 3 heights (30, 50, and 70% of merchantable height). The billets were chipped and mixed for kraft pulping.

To evaluate an enhanced *E. nitens* calibration, 25 whole-tree samples were obtained from a separate northern Tasmanian site (the Gog site). These trees were originally sampled as part of a different study.[9] A total of 186 trees had been core sampled and assessed for basic density and cellulose content[10] at age 13 years, and the 25 trees were chosen to cover the range of both properties. Selected trees were felled and a 25 mm thick disc taken from 0, 10, 20, 30, 40, 50, 60, and 70% of total height. The discs were chipped and mixed for kraft pulping. Details of the Gog site are given elsewhere.[9]

Twenty-eight *Eucalyptus globulus* Labill. (Tasmanian blue gum) whole-tree samples obtained from trees grown in the Esperance Valley, south-eastern (SE) Tasmania (*c.* lat. 43.15°S; long. 146.50°E) were used to examine the application of calibrations across different species. All samples were from 8-year-old trees. The trees formed part of a detailed study into the growth of several eucalypt species that were considered, at the time of establishment, to be potentially suitable for intensive management.[11] One hectare sites were established in 1983 at elevations of 60, 240, 440, and 650 m by the then CSIRO Division of Forest Research (now CSIRO Forestry and Forest Products) in cooperation with the Tasmanian Forestry Commission (now Forestry Tasmania).[11] Turnbull et al.[11] provide information relating to site preparation, fertilizer treatment, and pest control. No *E. globulus* samples from the 650 m site were available for pulping as freezing temperatures and snow damage had resulted in death or slow growth of the seedlings.[11]

Determination of Kraft Pulp Yield

Gunns Ltd. staff assessed the kraft pulp yield of each whole-tree composite sample using the conditions described in Schimleck et al.[8] A representative sample of the whole-tree composite was removed for analysis by NIR spectroscopy.

Sample Preparation for NIR Spectroscopy

Sub-samples were removed from the whole-tree composite chip samples for NIR spectroscopy. These samples were dried at 30°C or less for 10 to 14 days to give a nominal moisture content of 10%. After drying, the samples were milled in a Wiley mill through a 1.0 mm screen. Gog composite chips were reduced to small fragments using a disc pulverizer and also milled in a Wiley mill through a 1.0 mm screen.

Near Infrared Spectroscopy

Wood meal was placed in a large NIRSystems sample cup (NR-7070). The NIR spectra were measured in diffuse reflectance mode from samples held in a spinning sample holder in a NIRSystems Inc. Model 5000 scanning spectrophotometer. Spectra were collected at 2 nm intervals over the wavelength range 1100–2500 nm. The instrument reference was a ceramic standard. Fifty scans were accumulated for each sample and the results averaged. After the spectrum had been obtained, the sample cup was emptied and repacked. A duplicate spectrum was obtained and duplicate spectra were averaged to give a single spectrum per sample.

Kraft Pulp Yield Calibrations

Kraft pulp yield calibrations were developed using the Unscrambler (version 8.0) software package (Camo AS, Norway) and second derivative spectra (left and right gaps of 8 nm were used for the conversion). The following calibrations were developed:

1. Tasmania-wide *E. nitens* (based on the 126 whole-tree samples) and enhanced using a variable number of samples from the Gog site. Initially 5 samples (those used to enhance calibrations reported in Schimleck

et al.[8] and selected to encompass the range of pulp yields) were used, followed by 4, 3, 2, and finally a single Gog sample. Two different calibrations were obtained using the single sample; one after adding a sample with a high pulp yield and the other after adding a sample with a low pulp yield. The samples used in the smaller enhanced calibrations were selected from the original 5, but selected so as to retain the extremes. All calibrations were applied to 20 Gog samples, with the 5 samples initially added to the Tasmania-wide set being excluded;

2. Tasmania-wide 50%+ (based on the 75 whole-tree samples with pulp yields greater than 50% in the Tasmania-wide set) and enhanced as described in number 1. The prediction set described in number 1 was used;
3. Tasmania-wide (based on the 126 whole-tree samples) and enhanced using different Gog samples. The Gog set was split into 5 groups of 5 samples allocated randomly. The calibrations were applied to 20 Gog samples but the composition of the prediction set varied between sets; and
4. Tasmania-wide *E. nitens* (based on the 126 whole-tree samples) enhanced using 4 Tasmanian *E. globulus* samples. The calibrations were used to predict the pulp yields of the remaining 24 *E. globulus* samples.

The calibration sets are summarized in Table 1.

Partial least squares (PLS) regression was used to create the pulp yield calibrations with full cross validation (i.e., leave-one-out) and a maximum of ten factors. The software recommended the final number of factors for each calibration unless otherwise indicated. Calibration performance was assessed using the standard error of calibration (SEC) (determined from the residuals of the final calibration), the standard error of cross validation (SECV) (determined from the residuals of each cross validation phase), the coefficient of determination (R^2), and the ratio of performance to deviation (RPD_c),[12] which was calculated as the ratio of the standard deviation of the reference data to the SECV. The standard error of prediction (SEP) gave a measure of how well the calibrations predicted the pulp yields of the test set samples. The predictive ability of calibrations was assessed by calculating the ratio of performance to deviation (RPD_p), which is the ratio of the standard deviation of the reference data to the SEP.[12]

Table 1. Statistical summary of the Gog and Tasmania-wide *E. nitens* calibration set and the Tasmania *E. globulus* calibration set

Sample set	Min.	Max.	Average	Std. dev.
Tasmania-wide *E. nitens* (126 samples)	45.6	54.5	50.3	2.1
Gog *E. nitens* (25 samples)	50.1	56.8	53.2	1.7
Tasmania *E. globulus* (28 samples)	50.0	58.3	53.8	2.0

RESULTS AND DISCUSSION

Enhanced Tasmania-wide Pulp Yield Calibration Using a Variable Number of Samples

The Tasmania-wide kraft pulp yield calibration was enhanced using a variable number of Gog samples ranging from five to one, and the performances of each of these models are reported in Table 2. The calibrations demonstrated similar statistics for eight factors (the number of factors recommended by the software varied from 4 to 8 and it was decided to use 8 factors for all calibrations to facilitate comparison). When applied to the separate test set the calibrations provided similar R_p^2 but SEP and RPD_p were variable. The SEP was close to one when 3 to 5 samples were used to enhance the Tasmania-wide calibration but increased as fewer Gog samples were added.

Schimleck et al.[8] demonstrated that removal of samples having pulp yields less than 50% from the Tasmania-wide set improved the predictive performance of the subsequent calibrations. Here we investigated what impact the addition of a variable number of Gog samples to the Tasmanian-wide (limited) set had on predictive statistics (Table 3).

The Tasmania-wide 50%+ enhanced calibrations had weaker statistics than those for the full set owing to the narrower yield range. When applied to the remaining 20 Gog samples the calibrations gave R_p^2 values that were lower and more variable than those obtained using the full set Tasmanian-wide enhanced calibrations. However, SEP and RPD_p were improved with the lowest SEP (0.93) and the highest RPD_p (1.84) being obtained when 5 Gog samples were added to the calibration set. Predicted yields for the remaining 20 Gog samples are shown in Figure 1a (Tasmanian-wide calibration, 5 Gog samples added) and Figure 1b (Tasmanian-wide 50%+ enhanced calibration, 5 Gog samples added).

Table 2. Summary of enhanced Tasmania-wide *E. nitens* kraft pulp yield calibrations based on a variable number of samples

Samples used to enhance calibration	Calibration					Prediction (w-tree)		
	#Factors	R^2	SEC	SECV	RPD_c	R_p^2	SEP	RPD_p
5 samples added	8	0.92	0.62	0.75	2.91	0.77	1.03	1.65
4 samples added	8	0.92	0.62	0.75	2.89	0.77	1.11	1.54
3 samples added	8	0.92	0.62	0.75	2.90	0.77	1.04	1.65
2 samples added	8 (7 rec.)	0.92	0.61	0.75	2.88	0.78	1.23	1.40
High sample added	8 (4 rec.)	0.92	0.62	0.81	2.66	0.78	1.33	1.29
Low sample added	8 (4 rec.)	0.92	0.61	0.79	2.68	0.78	2.02	0.85

All calibrations were applied to 20 Gog samples, the 5 samples initially added to the Tasmania-wide set were excluded.

Table 3. Summary of enhanced Tasmania-wide *E. nitens* kraft pulp yield calibrations based on a variable number of samples and with samples having pulp yields less than 50% excluded from the calibration

Samples used to enhance calibration	Calibration					Prediction (w-tree)		
	#Factors	R^2	SEC	SECV	RPD_c	R_p^2	SEP	RPD_p
5 samples added	8	0.83	0.51	0.74	1.66	0.73	0.93	1.84
4 samples added	8	0.83	0.51	0.75	1.63	0.73	0.96	1.79
3 samples added	8 (7 rec.)	0.84	0.49	0.73	1.68	0.67	0.99	1.73
2 samples added	8 (6 rec.)	0.84	0.48	0.78	1.54	0.69	0.94	1.82
High sample added	8 (4 rec.)	0.85	0.47	0.71	1.70	0.61	1.78	0.96
Low sample added	8	0.82	0.47	0.68	1.66	0.70	1.49	1.15

All calibrations were applied to 20 Gog samples, the 5 samples initially added to the Tasmania-wide set were excluded.

Examination of Figure 1a shows that the enhanced Tasmanian-wide calibration tended to underestimate the pulp yields of the 20 Gog samples whereas the Tasmanian-wide 50%+ enhanced calibration tended to overestimate the yields of the low pulp yield samples and underestimate the yields of the higher yielding samples. When ranking trees for pulp yield the calibration giving the highest R_p^2 (assuming SEPs were similar) would be preferred, hence, the enhanced Tasmanian-wide calibration would be favored.

The results reported in Tables 2 and 3 indicate that even a single Gog sample, when added to the Tasmania-wide calibration set, can have a large positive influence on the predictions made by the calibration. When the Tasmanian-wide calibration was initially applied to all 25 Gog samples[8] predictive errors were large (SEP = 4.60, $RPD_p = 0.37$) despite a reasonable R_p^2 (0.70). The addition of a single sample was sufficient to greatly reduce the error (by 71% if the high pulp yield Gog sample was added to the full set) but at least 3 samples were required for the RPD_p become relatively stable.

Enhanced Tasmania-wide Pulp Yield Calibration Using Different Gog Samples

While we demonstrated that the addition of a small number of Gog samples to the Tasmania-wide calibration set can enhance predictive accuracy, the addition of different Gog samples to the Tasmania-wide set has not been examined. Table 4 reports calibrations and predictions for Tasmania-wide calibrations enhanced using 5 different sets of Gog samples (note the 20 samples for each prediction set were different).

The calibrations for the enhanced Tasmania-wide sets using different samples were similar regardless of which set was added. RDP_c varied from 2.81 to 2.98 owing to variation in the standard deviation of the calibration

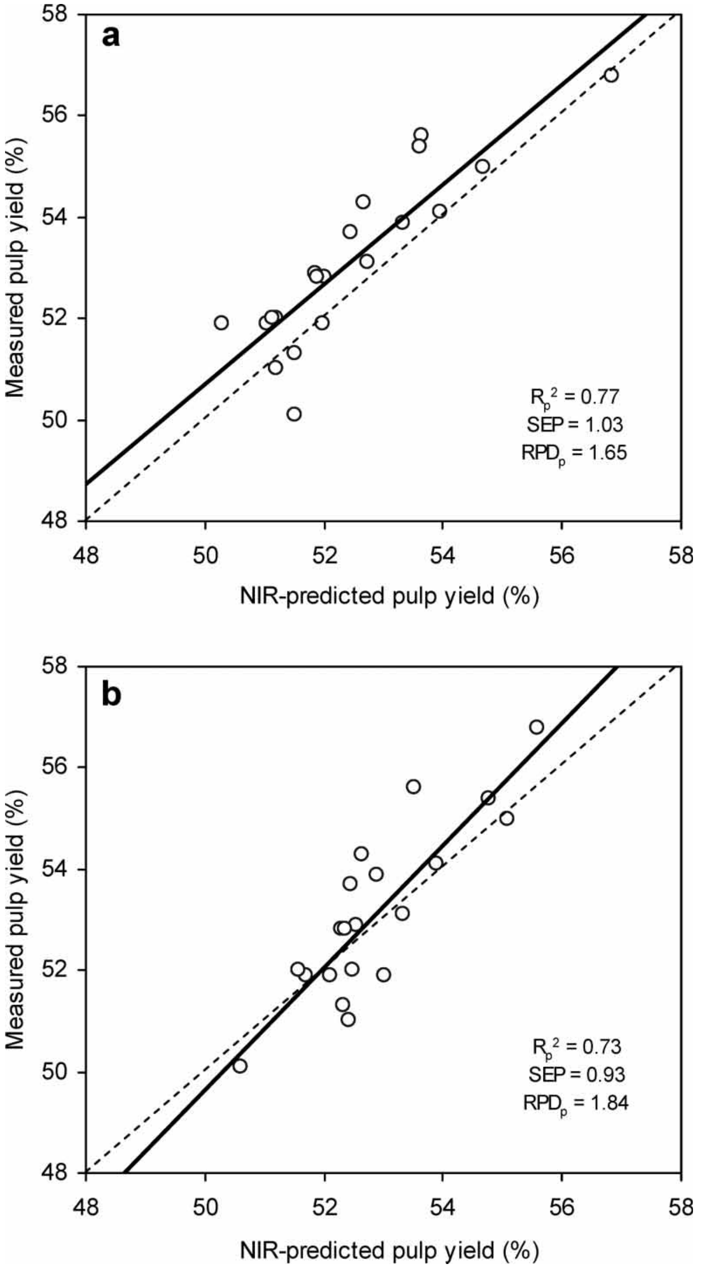

Figure 1. Relationships between measured pulp yield and NIR-predicted pulp yield for 20 Gog *E. nitens*. Predictions were made using (a) enhanced Tasmania-wide pulp yield calibration and (b) Tasmania-wide 50%+ enhanced calibration. Note the regression line has been plotted and the thin broken line represents the line of equivalence.

Table 4. Summary of enhanced Tasmania-wide *E. nitens* kraft pulp yield calibrations based on 5 different sets of Gog samples

Samples used to enhance calibration	Calibration					Prediction (w-tree)		
	# Factors	R^2	SEC	SECV	RPD_c	R_p^2	SEP	RPD_p
Set 1 added	8	0.92	0.62	0.75	2.98	0.74	0.83	1.86
Set 2 added	8	0.91	0.62	0.75	2.81	0.82	0.95	1.93
Set 3 added	8	0.92	0.62	0.75	2.85	0.84	0.77	2.35
Set 4 added	8	0.92	0.63	0.76	2.86	0.81	0.76	2.34
Set 5 added	8	0.92	0.63	0.77	2.83	0.83	0.98	1.57

The calibrations were applied to 20 Gog samples but the composition of the prediction set varied between sets.

set, which ranged from 2.11 to 2.23 as different sets of Gog samples were added to the Tasmania-wide set. When applied to the remaining 20 Gog samples the predictive performance was variable. The set 3 enhanced calibration (pulp yield range = 51.3 to 53.1%) gave the best predictions (Figure 2a) with a strong R_p^2 (0.84) and good RDP_p (2.35). The weakest R_p^2 (0.74) was obtained using the set 1 enhanced calibration (pulp yield range = 51.2 to 56.8%), whereas the lowest RDP_p (1.57) (Figure 2b) was obtained using the set 5 enhanced calibration (pulp yield range = 50.1 to 55.4%). The standard deviation of pulp yield for the various prediction sets is important. For example, the set 3 enhanced calibration predicted yields of samples with a standard deviation of 1.81, whereas the set 5 enhanced calibration was applied to a set with less variation (standard deviation = 1.54).

The predictive performance of 4 of the 5 calibrations was superior to the Tasmania-wide enhanced calibration reported in Table 3 of Schimleck et al.[8] Samples selected to enhance calibrations can have a large influence on how well the subsequent calibration performs hence the selection of the best possible samples for calibration enhancement is important. Without knowledge of the pulp yields of the samples the only options for selecting samples is to predict their yields using an existing calibration or to use their NIR spectra. These approaches are discussed in Schimleck et al.[13] who used both methods to select samples for calibration development. The selection of spectrally unique samples for calibration (or for addition to an existing calibration) relies on a neighborhood concept. In this study WinISI II[14] was used to identify spectrally unique samples. When a neighborhood H of 1.6 was used, 5 samples were identified as unique and these were used to enhance the Tasmania-wide calibrations. The calibration and its performance when used to predict the pulp yields of the remaining 20 Gog samples are shown in Figure 3a and 3b, respectively.

Calibration statistics were strong and similar to those reported in Table 4 and the calibration performed well when applied to the test set. R_p^2 and SEP

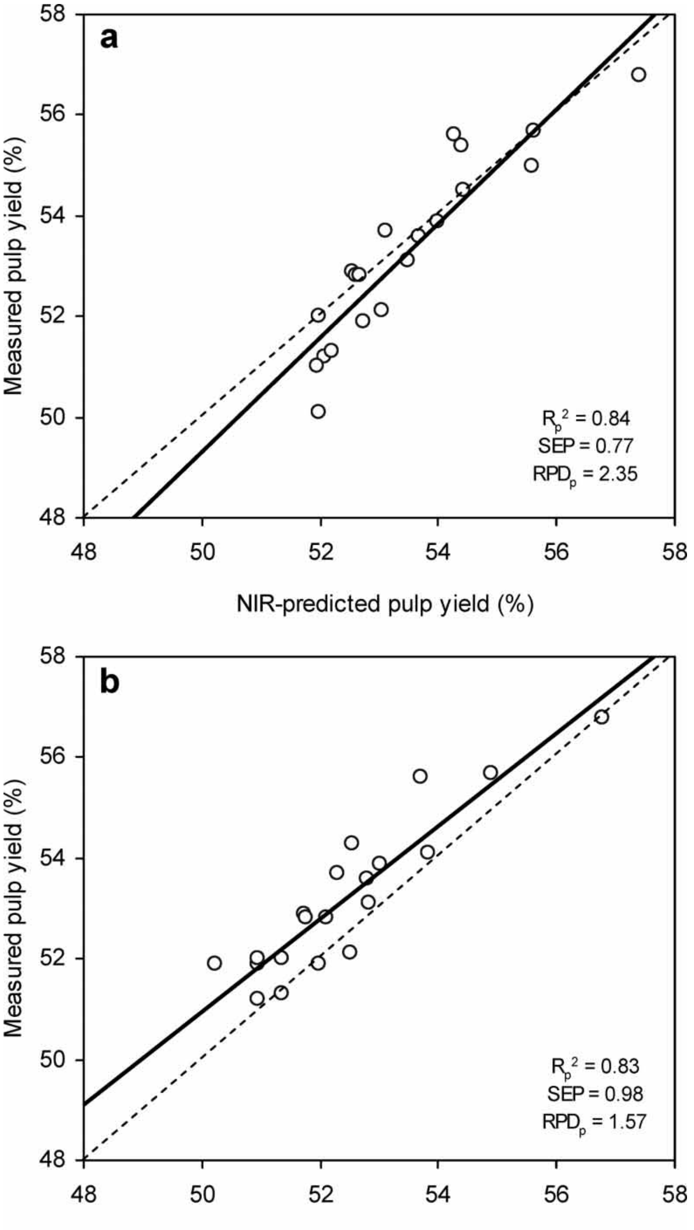

Figure 2. Relationships between measured pulp yield and NIR-predicted pulp yield for 20 Gog *E. nitens*. Predictions were made using Tasmania-wide pulp yield calibrations enhanced using 5 Gog *E. nitens* (a) set 3 added and (b) set 5 added. Note the regression line has been plotted and the thin broken line represents the line of equivalence.

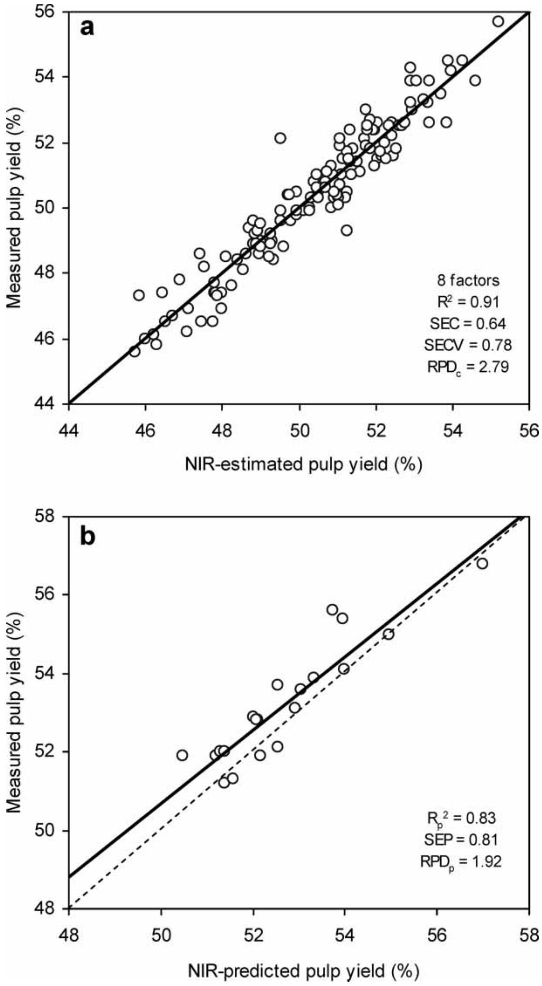

Figure 3. (a) Relationship between measured pulp yield and NIR-estimated pulp yield for the Tasmania-wide calibration enhanced using 5 Gog *E. nitens* samples identified using WinISI II software. Note the regression line has been plotted. (b) Relationship between measured pulp yield and NIR-predicted pulp yield for 20 Gog *E. nitens* obtained using the Tasmania-wide calibration enhanced using 5 Gog *E. nitens* samples identified by WinISI II. Note the regression line has been plotted and the thin broken line represents the line of equivalence.

were similar to the best predictive results reported in Table 4, whereas the RPD_p (1.92) was lower. The lower RPD_p is a consequence of the remaining samples having a smaller standard deviation (1.55) than sets 3 (1.81) and 4 (1.77). The results demonstrate that WinISI II provides a suitable method for selecting representative samples.

Table 5. Summary of the performance of the Tasmania-wide *E. nitens* calibration when used to predict the pulp yield of 24 Tasmania *E. globulus* samples and the performance of the Tasmania-wide *E. nitens* calibration (on the 24 *E. globulus*) after being enhanced with 4 *E. globulus* samples.

	Calibration					Prediction (*E. globulus*)		
	# Factors	R^2	SEC	SECV	RPD_c	R_p^2	SEP	RPD_p
Tasmania-wide *E. nitens*	6	0.91	0.64	0.76	2.76	0.81	1.23	1.60
Tas-wide *E. nitens* (+4 *E. glob*)	6 (7 rec.)	0.90	0.71	0.80	2.77	0.86	0.98	2.01

The 4 *E. globulus* used to enhance the calibration were excluded from the test set.

Application of the Tasmania-wide *E. nitens* Pulp Yield Calibration to a Different Species

We demonstrated that the enhanced Tasmania-wide calibration can be successfully applied to samples from the same species grown at a different location but have not explored the application of it to a different species. Table 5 reports the performance of the Tasmania-wide *E. nitens* calibration when used to predict the pulp yield of 24 Tasmania *E. globulus* samples and the performance of the Tasmania-wide *E. nitens* calibration (on the 24 *E. globulus*) after adding 4 *E. globulus* samples to it.

The Tasmania-wide *E. nitens* calibration performed reasonably well when applied to the Tasmania *E. globulus* set giving a good R_p^2 (0.81) and a SEP of 1.25 ($RPD_p = 2.50$). The same calibration enhanced using 4 *E. globulus* samples performed very well, giving an R_p^2 of 0.86, a SEP of 0.98 and an RPD_p of 2.01. The results reported here demonstrate that the Tasmania-wide calibration can be enhanced using samples from a different species (*E. globulus*) and applied successfully to other *E. globulus* samples. Some *E. nitens* samples in the Tasmania-wide sample set were from the same location as the *E. globulus* samples and predictive errors for the Tasmania-wide *E. nitens* calibration may have been larger if the *E. globulus* samples were from a different site.

CONCLUSIONS

The addition of a single Gog sample to the Tasmania-wide *E. nitens* calibration set was sufficient to greatly reduce predictive errors when the calibration was used to predict pulp yield of the Gog samples. At least 3 Gog samples were required for addition to the Tasmania-wide set to give relatively stable predictive errors. Predictive errors varied depending on what set of 5 samples was used to enhance the Tasmania-wide *E. nitens* calibration. The standard deviation of pulp yield for the remaining 20 Gog samples (forming the prediction set) was important, with sets having the largest standard deviations giving the best predictive statistics. The Tasmania-wide *E. nitens* calibration can be enhanced using samples from a different species (*E. globulus*) and applied successfully to other *E. globulus* samples.

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