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Prediction of Pulp Yield and Basic Density of *Eucalyptus spp.* using Near Infrared Spectroscopy (NIR)

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Objectives

- To develop NIR Spectroscopy calibrations of Basic Density and Pulp Yield.
- To analyze the NIR Spectroscopy calibrations in their different properties and evolution.
- To study the feasibility of this method to predict Pulp Yield and Basic Density with different species of the Uruguayan Forest Eucalyptus.
- To improve the efficiency of the Basic Density and Pulp Yield determinations.

Introduction

The *Eucalyptus* genus is used world wide for the kraft pulp production. In Uruguay -since the recent installation of big industries producers of cellulose- the species used are mainly *E. grandis, E. dunnii, E. maidenii* and *E. globulus.*

Pulp yield and Basic density are two of the main parameters considered for the profitability evaluation of tree plantations used in cellulose pulp production. However, the traditional assessment of these properties is quite time-consuming, particularly the pulp yield determination, which consists in simulating the industrial pulping by cooking a sample of chips representative of the whole tree.

Near Infrared Spectroscopy has been implemented as a higher-efficiency process for pulp yield and basic density determinations. Some of the problems implicit with the NIR Spectroscopy methodology have been overcome by instrumental development and computer processing breakthroughs.

The analyses in NIR spectroscopy include the measurement of spectra of a big enough amount of samples with a known pulp yield determined at a wished kappa number, the development of a model that can relate the NIR spectra with the pulp yield, and then the use of this model and the spectra of an unknown pulp yield sample to predict the value. The prediction of basic density by NIR spectroscopy involves a similar process.

Although NIR spectroscopy has been known in Uruguay for several years already, it aimed at other agribusiness purposes. Therefore, this work is the first approach to the use of the NIR spectroscopy method in the Uruguayan forest industry.

Methods

PULP YIELD CONVENTIONAL METHOD DETERMINATION

Table 1. Kraft pulping conditions

Cooking Type	A	B
Samples	83	55
Time to/at temperature	90 min / 50 min	50 min / 145 min
Cooking temperature	165 ºC	157 ºC
Sulphidity	25 %	25 %
Liquor ratio	3.5 g/g	3.5 g/g
H Factor	570	750

BASIC DENSITY CONVENTIONAL METHOD DETERMINATION

CHIPS & DISCS

of *E. dunnii, E. grandis, E. globulus ssp. globulus, E. globulus ssp. maidenii, E. biscostata* and *hybrids*

CALIBRATION

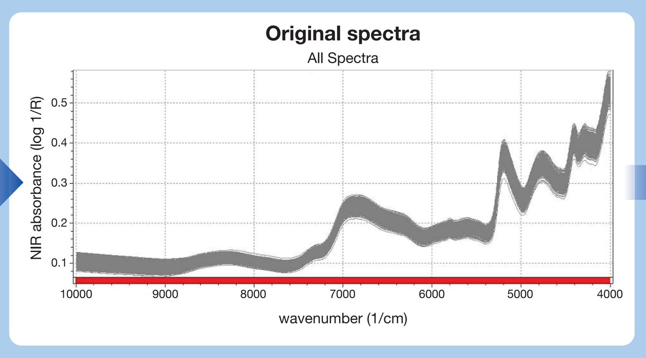
Table 3. Calibration and validation set of basic density (conventional method values)

	Calibration set	Validation (prediction) set
Nº of samples	127	35
Minimum	0.366 g/cm3	0.376 g/cm3
Maximum	0.642 g/cm3	0.637 g/cm3
Average	0.527 g/cm3	0.526 g/cm3
SD	0.049 g/cm3	0.049 g/cm3

Table 4. Calibration and validation set of pulp yield (conventional method values)

	Cooking Type A		Cooking Type A	
	Calibr. set	Validation (predict.) set	Calibr. set	Validation (predict.) set
Nº of samples	60	23	39	16
Minimum	43.7 %	44.9 %	53.0 %	53.1 %
Maximum	57.4 %	56.5 %	55.0 %	54.9 %
Average	50.8 %	50.9 %	54.1 %	54.1 %
SD	2.6 %	3.1 %	0.4 %	0.4 %

MILLING



Statistics (Simplification of spectra): PC, PLS, smoothing, derivatives, normalizations, etc.

EVALUATION OF CALIBRATION

Table 2. NirCal 5.2 Q-Value Calculation

Weights	Formula for value	General Term
10	Number of C-Set spectra with residual too big	Rejection of known
1	Number of V-Set spectra with residual too big	Rejection of unknown
2	Abs (SEE-SEP) / (Abs (SEP) + 1.0)	Relative consistency
2	Abs (V-set BIAS) / Abs (Range)	Weighted BIAS
1	1 – V-set Regression	Validity
1	Abs (C-Set Regression – V-Set Regression)	Comparability
1	SEP / Abs (Range)	Precision
1	Abs (RSS)/Abs (Range)	Weighted Accuracy

C-Set, Calibration Set; V-Set, Validation Set; SEE, Standard Error of Estimation of the C-Set; SEP, Standard Error of Prediction; RSS, V-Set Residual error Sum of Squares

Results and Discussion

BASIC DENSITY

Table 5. Evolution of the basic density calibrations

Cal Nº	Samples in C-set	Samples in V-set	Q value	Pre-treatment	C-set regression coefficient	V-set regression coefficient
1	55	12	0.73	db1	0.87	0.85
2	70	17	0.81	mf, db1	0.90	0.91
3	76	22	0.85	sa3, ncl, db1	0.94	0.94
4	116	30	0.84	ds2	0.92	0.94
5	127	35	0.84	ds2	0.92	0.94

db1 - first derivative BCAP; mf - multiplicative scatter correction full; sa3 - smooth average three points, ncl - normalization by closure; ds2 - second derivative Taylor 3 points segment5 gap5 smoothing; snv - standard normal variate

Table 6. Comparison of pretreatments in the final Calibration and Validation sets

Pre-treatment	Q value	Factors	SEC (g/cm^3)	SEP (g/cm^3)	C-set regression coefficient	V-set regression coefficient
ds2	0.84	9	0.019	0.017	0.92	0.94
mf	0.82	10	0.020	0.017	0.91	0.94
None	0.82	10	0.020	0.017	0.91	0.94
ncl	0.80	8	0.022	0.018	0.89	0.93
snv	0.80	8	0.022	0.018	0.89	0.93

snv – standard normal variate; note: all the calibrations were made in the wavelength range of 4000 – 10000 cm^{-1}

The conventional method error has been reported by the laboratory to be 2.8 %, which means a range of 0.010 to 0.018 g/cm^3 (standard deviation of the samples is 0.049 g/cm^3). Similarly, the NIR method has a SEC of 0.019 g/cm^3 and a SEP of 0.017 g/cm^3.

Figure 2 shows an overestimation of the basic density in the range 0.400 to 0.450 g/cm^3 along with an underestimation of high basic density samples (0.600 to 0.650 g/cm^3 range).

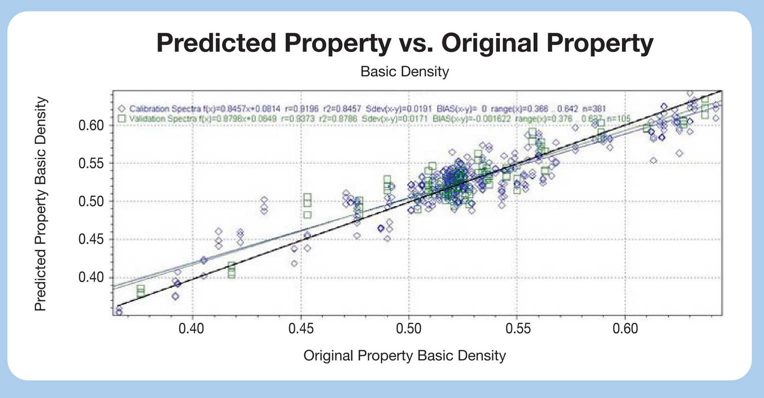


Figure 2. NIR Spectroscopy predicted basic density versus conventional method determined basic density.

KRAFT PULP YIELD

Cooking Type A

Table 7. Evolution of the pulp yield calibrations (Cooking Type A)

Cal Nº	Samples in C-set	Samples in V-set	Q value	Pre-treatment	C-set regression coefficient	V-set regression coefficient	Wavelenght range (cm^{-1})
1	38	13	0.67	db1	0.97	0.98	4000-9000
2	45	13	0.74	snv,db1	0.96	0.97	4000-10000
3	49	14	0.74	db1, ncl	0.96	0.97	4000-10000
4	56	16	0.64	ncl, db1	0.94	0.97	4000-10000
5	60	23	0.72	db1, ncl	0.94	0.96	4000-9000

Table 8. Comparison of pretreatments in the final Calibration and Validation sets (Cooking Type A)

Pre-treatment	Q value	Factors	SEC (%)	SEP (%)	C-set regression coefficient	V-set regression coefficient
db1, ncl	0.72	8	0.9	0.9	0.94	0.96
db1	0.71	12	0.9	0.9	0.93	0.96
ncl, db1	0.70	11	0.9	0.9	0.94	0.96
sa3, ncl, db1	0.70	10	0.9	0.9	0.93	0.96
None	0.70	10	1.0	1.0	0.92	0.95

sa3 – smoothing average 3 points; note: all the calibrations were made in the wavelength range of 4000 – 9000 cm^{-1}

Fewer samples were available, thus having less spectra variability that can be related to the pulp yield in the whole range.
The 0.9 % of SEP compared to the conventional method error (stated as 0.46 % of pulp yield) is not small enough to predict an unknown sample with sufficient precision, however, the ratio SEP/SD is 0.33, indicating an acceptable result.
More samples have to be added, principally in the extremes of the total range.

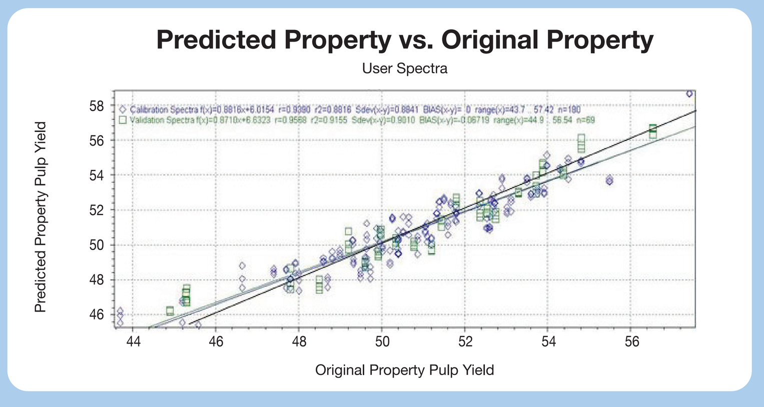


Figure 3. NIR Spectroscopy predicted pulp yield versus conventional method determined pulp yield (Cooking Type A).

Cooking Type B

Table 9. Comparison of pretreatments in the final Calibration and Validation sets (Cooking Type B)

Calibration method	Wavelenght range	Pre-treatment	Q value	Factors	SEC (%)	SEP (%)	C-set regression coefficient	V-set regression coefficient
PCR	5000-7144, 7404-10000	snv	0.53	8	0.3	0.3	0.59	0.61
PLS	4000-10000	None	0.53	12	0.4	0.3	0.57	0.61
PCR	4000-10000	None	0.53	10	0.4	0.3	0.56	0.62
PCR	5000-10000	snv	0.53	9	0.3	0.3	0.60	0.59
PCR	5000-7144, 7404-10000	None	0.52	9	0.4	0.4	0.57	0.56

PCR – Principal Component Regression

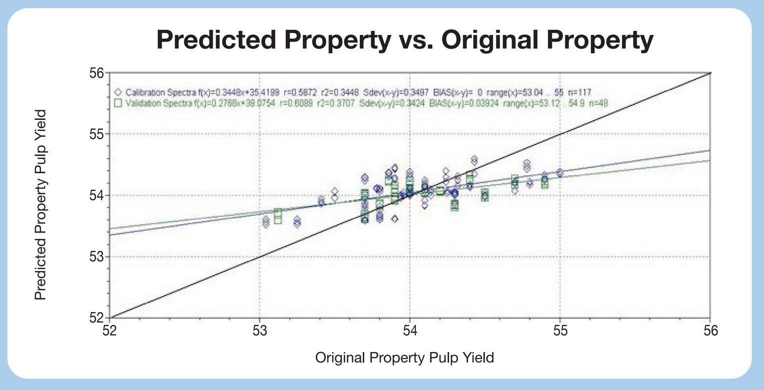


Figure 4. NIR Spectroscopy predicted pulp yield versus conventional method determined pulp yield (Cooking Type B).

The parameters to evaluate the calibration are not acceptable. The SEC and SEP values are similar to the standard deviation, revealing the futility of the calibration. It's worth mentioning that the Cooking Type B is composed mainly by *Eucalyptus globulus* –unlike Cooking Type A–, indicating that probably the lack of variability is the major responsible of this problem. Taking into account that the minimum error of the NIR method is the error of the conventional method (0.46 % in pulp yield), in order to have a useable NIR calibration of this cooking type, not only variability inside the range must be achieved, but also the range must be extended.

Conclusions

- NIR Spectroscopy calibrations of pulp yield and basic density of *Eucalyptus spp.* from Uruguay are achievable with satisfactory results, regardless of their origin, species or age.
- Preference between different calibration pretreatments is based mainly on a statistical Q-value.
- Basic Density calibration is acceptable with a SEP similar to the average error of the conventional method.
- Cooking Type A Kraft Pulp Yield calibration gives useable results.
- Cooking Type B Kraft Pulp Yield calibration is unsatisfactory.
- More samples have to be added to the three calibration sets.

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