Rapid NIR analysis of chemical and mechanical properties for *Eucalyptus camaldulensis* at plantation in Thailand

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Introduction

Pulp and paper industries have played important economic role for Thailand development. Their productions are expanding significantly year by year. Especially, *Eucalyptus camaldulensis* is used as the main raw material for pulp and paper making in Thailand. Due to being a fast-growing tree, Eucalyptus can be easily grown in every climatic variable. Only for 3-5 ages, it can be used for papermaking. In order to improve the productivity of pulp yield, the pulp and paper industries require the supply of wooden chip with stable quality (i.e.; lignin content, cellulose content, density, length of fiber, etc.). However through the tree breeding programs, it is difficult to satisfy them as the wood is natural resources. The chemical components or physical properties of Eucalyptus have wide variety, which depend on the location, tree age or individuality.

Recently, near-infrared (NIR) spectroscopy has been recognized as one of the most powerful analytical techniques for the wood qualities. Several scientists (1-9) have studied the ability of NIR spectroscopy to analyze the wood properties such as kraft pulp yield, the content of cellulose, hemicellulose, lignin, glucose, glucan, xylose, xylan, acetyl, carbohydrate, uromic acid, hot water solution as well as fiber orientation, moisture content and pulp kappa number.

The purpose of this research is to investigate the feasibility of predicting chemical composition and mechanical properties using NIR spectroscopy as selection criteria in the tree breeding and close selection programs of *Eucalyptus camaldulensis* in Thailand.

Materials and method

Sample

The *Eucalyptus camaldulensis* used in this study were supplied by the Siam Pulp and Paper public company limited. The sample separated into two parts, one parts for the soda pulping and another part for the chemical and NIR analysis. Air dried wood chips made by Wiley Mill were ground to pass through a 40 mesh screen and retained on 60 mesh screen. The retained sample was collected for chemical analysis as wet lab. style and NIR analysis.

Near Infrared Spectroscopy: Proceedings of the 11th International Conference.

Chemical analysis

Quantitative analysis in term of holocellulose, lignin, alphacellulose, pentosan, extractive and ash in *Eucalyptus camadulensis* were performed on the basis of TAPPI standard method. Xylose, xylan, glucose and glucan were determined by High Performance Liquid Chromatography (HPLC) following the method of Wallis (10). The pulp yield at Kappa number 20 were presented by The Siam Pulp and Paper public company limited.

NIR analysis

Totally 55 samples of wood meal were prepared for NIR analysis. Each sample was kept 25°C prior to the NIR measurement. The NIR diffusely reflected spectra were obtained by scanning 20g of each sample in a standard close cup using IA500 spectrophotometer from Bran+Luebbe Co. A number of scans were collected and averaged over the range 1100-2500 nm at 2 nm for each measurement. Each sample was replicated and measured three times.

Calibration

The samples were divided into two sets of similarly distributed chemical contents. The calibration set having a wider range of the chemical values was used to develop the calibration model by means of MLR and PLS. The model was subsequently validated with the second set. In PLS analyses the cross validation was also performed for comparison. A number of pretreatment combinations were empirically selected to obtain the spectra prior to calibration which could be best described by the developed model. In this study, multiplicative scattering correction (MSC), smoothing, normalization, first derivation and second derivationve were employed as the pretreatment algoritm.

Results and discussion

The NIR spectra with a range of total yield and screen yield percentage are shown in Figure 1a and Figure 1b, respectively.

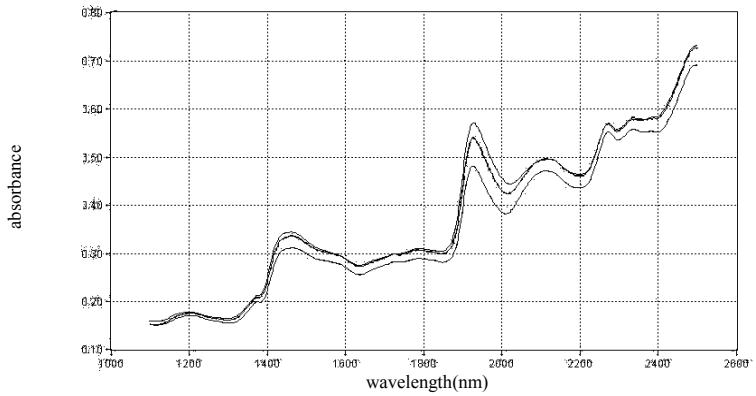

Figure 1a. NIR spectra with three different values of total yield.

The absorption bands are nevertheless difficult to choose distinctively as only broad bands are dominating. The MLR model pre-treated with a combination of smoothing and first derivative gave

the best result for total yield prediction (Table 1). The PLS model using the NIR spectra in a range 2000-2500 nm yielded a slightly better prediction (SEP=1.353%) with improved bias of –0.083 (Table 2). The MSC and first derivative were the best combination of pre-treatment used to convert the spectra prior to the calibration for the PLS model. As for the screen yield prediction, the PLS model was found to give better prediction than the MLR model. The spectra range arbitrarily selected in developing the PLS model for the screen yield was 1600-1800 nm and 2000-2500 nm which avoided the water band influence.

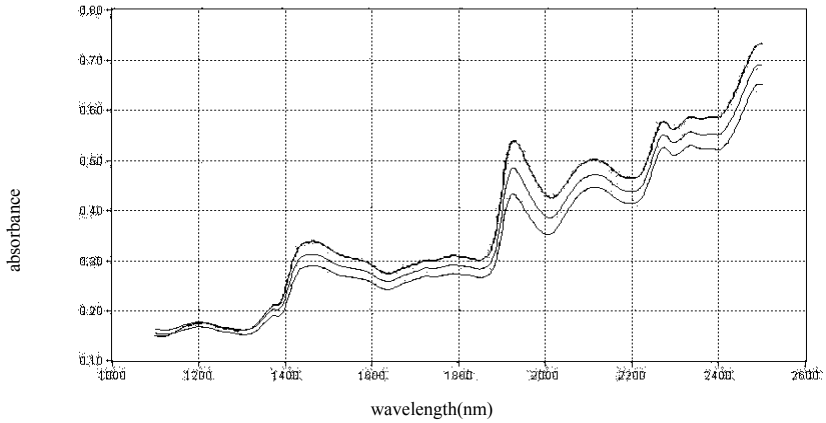

Figure 1b. NIR spectra with three different values of screen yield.

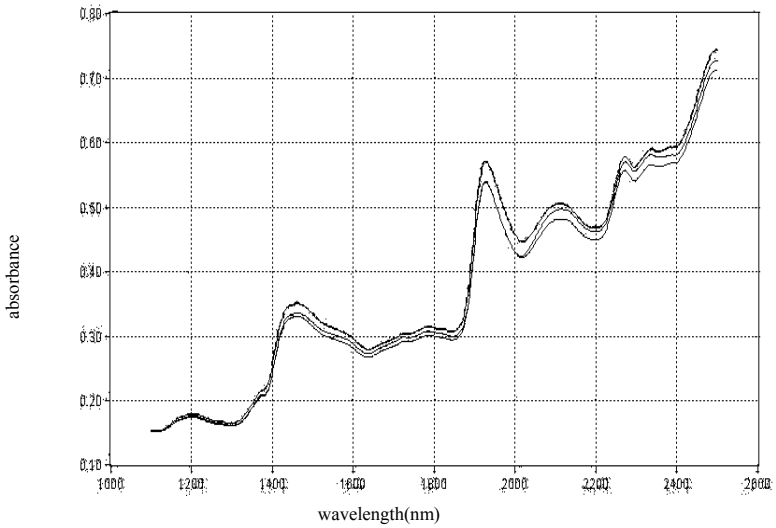

Figure 2 NIR spectra of wood sample with three different values of lignin content

Shown in Figure 2 are the NIR spectra (1100-2500 nm) of the wood meal with a range of lignin content. Slight improvement in prediction was offered by MLR model in terms of SEP over PLS model. Both models had their NIR spectra pretreated with a combination of smoothing and second derivative with MSC addition in case of PLS model.

The other models for calculating xylose, xylan and holocellulose were also developed with acceptable accuracy. Generally PLS model were found to be better than MLR models.

Table 1. The pretreatment combination, selected wavelengths and NIR calibration statistics of MLR regression models for optimally predicting each chemical constituents

Constituents	Pretreatment	Wavelength	Correlation (R)	SEC	SEP	Bias
%Total yield	Ab+Sm+1 De	1502 1607 2316	0.776	1.306	1.380	0.170
%Screen yield	Ab+Sm+No+2De	1148 1244 1482 1530 2500	0.881	1.133	1.650	0.190
%Lignin	Ab+Sm+2 De	1688 2180 2328	0.800	0.711	0.890	0.160

Ab = Absorbance Sm = Smoothing No = Normalization 1 De = First Derivetive 2 De = Second Derivative

Table 2. The chemical range, selected range(s) of wavelengths, pretreatment combination, validation method, number of factors and calibration statistics of PLS regression models for the optimal prediction of each chemical constituents

Constituents	Wavelength	Pre-treatment	Method	No. Factors	Calibration	Validation		
					SEC	Correlat-ion	SEP	Bias
%Total yield	2000-2500	Sm+MSC+1De	Test set	4	1.251	0.697	1.353	-0.083
%Screen yield	(1600-1800) +(2000-500)	Sm+MSC+1De	Test set	4	1.441	0.813	1.173	-0.056
%Lignin	(1600-1800) +(2000-500)	Sm+MSC+2De	Test set	4	0.434	0.853	0.521	0.119
%Glucose	1600-1800	Sm+MSC+1De	Test set	2	2.376	0.483	2.341	0.485
%Glucan	1100-2500	Sm+MSC+1De	Test set	3	1.912	0.598	1.919	0.760
%Xylose	1600-1800	Sm+MSC+2De	Test set	12	0.365	0.803	0.557	-0.127
%Xylan	2000-2500	Sm+MSC+1De	Test set	3	0.354	0.839	0.340	-0.126
%Pentosan	(1600-1800) +(2000-500)	Sm+MSC+2De	Test set	6	0.566	0.944	0.912	0.047
%Ash	1600-2500	Sm+MSC+2De	Cross validation	10	0.063	0.759	0.117	0.000
%Extractive	(1600-1800) +(2000-500)	Sm+MSC+1De	Cross validation	7	0.247	0.795	0.406	0.000
%Alpha cellolose	1600-1800	Sm+MSC+1De	Test set	3	0.918	0.654	1.060	1.056
% Holocellulose	2000-2500	Sm+MSC+2De	Cross validation	4	0.441	0.827	0.551	0.015

Sm = Smoothing 1De = First Derivative 2De = Second Derivative MSC = Multiplicative scatter correction

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