Prediction of pulp yield and cellulose content from wood samples using near infrared reflectance spectroscopy

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ABSTRACT *Wood assessed in powder form by near infrared reflectance spectroscopy (NIRS) was found to be positively and significantly correlated with common laboratory techniques for total pulp yield (r = 0.668) and cellulose content (r = 0.730). The amount of wood required per sample is approximately 5.0 g. This allows for nondestructive sampling of genetic and silvicultural trials.*

KEYWORDS
Cellulose
Chemical analysis
Kraft pulping
Nondestructive tests
Spectroscopy
Yield

The determination of pulp yield and cellulose content is an important step in evaluating raw materials for pulping. Cellulose content is especially useful when an organization is involved in more than one type of pulping (groundwood, kraft, dissolving, etc.). However, few organizations can devote the time and money necessary to obtain data from a sufficient number of samples to determine pulp yield and cellulose content.

In a tree breeding program, for example, hundreds of trees may have to be sampled in a genetic trial to find out which families are superior for pulp yield. This assessment could take the entire resources of a pulping laboratory, which then would be unable to respond to daily demands for quality control information.

The use of near infrared reflectance spectroscopy (NIRS) allows for small wood samples to be rapidly analyzed to predict pulp yield and cellulose content with an acceptable degree of precision. NIRS has been used to analyze forage and grains (*1–3*). The technique is being used by the South African Sugar Association Experimental Station to predict nitrogen content in sugar cane leaves (*4*). Wessman *et al.* (*5*) have found NIRS to be effective for determining the nitrogen and lignin content of foliage from forest and prairie species.

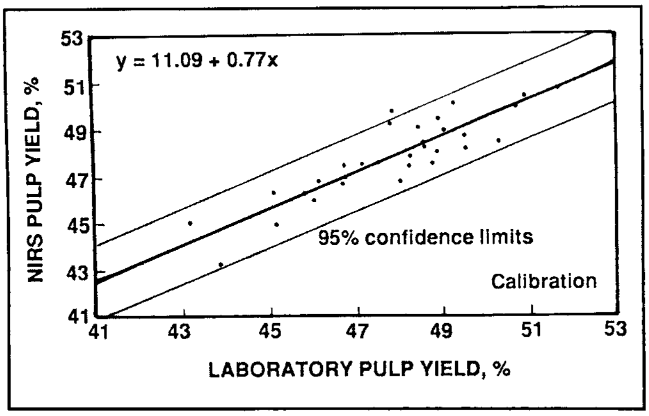
In the field of pulp and papermaking, the NIRS technique has been found to accurately predict kappa number from handsheets (*6, 7*). The use of diffuse reflectance Fourier-transform infrared spectrometry (FTIR) has also been shown to accurately predict lignin content in unbleached pulps (*8*). Garbutt *et al.* (*9*) found that NIRS could be used to predict lignin and cellulose content in wood samples of *Eucalyptus grandis* Hill ex-Maiden. The trees for that study were felled, though the quantity of wood required (less than 5.0 g) does allow for nondestructive sampling using an increment borer. In many genetic and silvicultural trials, this is a significant advantage. Further discussion of FTIR can be found in a recent review by Michell (*10*).

Materials and methods

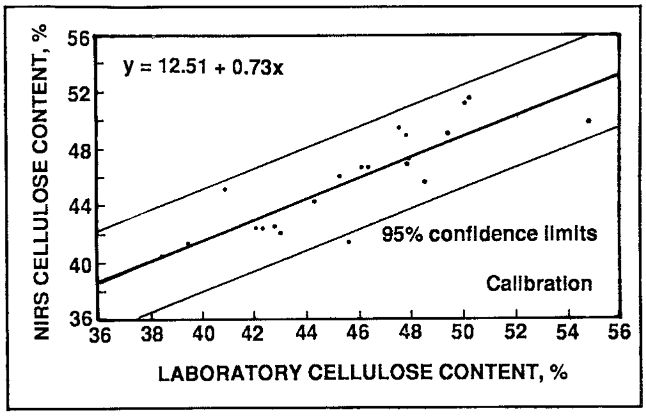
The instrument we used was a Technicon Infra-Analyzer interfaced with a personal computer. The description of the NIRS methods in use at Sappi's Research and Development Laboratory has been reported (*6, 7*). In our study, a "closed cup" fitted with a glass cover was used to hold the sample.

The samples used for the NIRS calibration of pulp yield were from a total of 29 trees from 14 different pine species, as previously described (*11*).

1. NIRS calibration of total pulp yield for pine wood samples



2. NIRS calibration of cellulose content for pine wood samples



I. Pulping conditions

Maximum temp., °C	170
Time to 170°C, min	110
Time to 170°C, min	60
Ratio, liquor to o.d. wood	5.4:1
Active alkali, % as Na_2O on wood	22
Sulfidity, %	26

II. Calibration of laboratory determinations for pulp yield and cellulose contents with NIRS

Trait	*No. of samples*	*Filter values, nm*	*Correlation coef.*	*Coef. of variance*
Pulp yield, %	29	1680, 1940 1982, 2100, 2208	0.876**	1.92
Cellulose content, %	21	1722, 1734, 2230, 2310, 2236	0.853**	4.58

Each tree was felled, and the height was taken to a top diameter of 8 cm. The tree was divided into 10% height intervals and sawn into bolts. A disc about 25 mm in thickness was sawn from the bottom of each bolt. These discs were kept separate by individual tree and were chipped. The dried chips were mixed, and a sample of 10.0 g was removed.

The sample was ground until it had reached a powder form in a water-cooled Ika A10 Analytical Mill fitted with an A15 radial cutter blade. The powder was then screened through a 250-μm screen before being placed in the sample holder and being subjected to NIRS. The entire wood sample was utilized through a process of repeated grinding and screening.

From each tree, 100 g of air-dried chips were pulped using laboratory kraft cooking conditions (**Table I**). The wood samples used for determining cellulose content were from 21 trees, including 14 pine hybrids and *Pinus taeda* L., *P. elliottii* Engelm. and *P. patula* Schiede and Deppe that were 18 years of age. The wood was removed from these trees in the form of increment cores with a diameter of 25 mm. Similar sample preparation for NIRS was applied to these samples. The Kurschner method (*12*) was used to determine the cellulose content from 1.0 g of wood from each tree. The method involves an acid extraction to dissolve all noncellulosic material (hemicellulose, lignin, extractives) followed by the determination of the polysaccharides in the residue. Subtraction of the polysaccharides from the residue results in a rectified cellulose value. The whole procedure requires about 16 h.

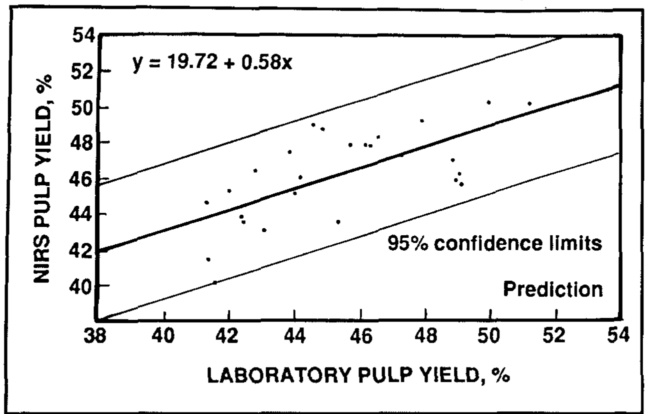
Results

The results of the NIRS calibration are given in **Table II**. The correlation coefficient (r) was significant for both pulp yield (**Fig. 1**) and cellulose content (**Fig. 2**). This indicates that NIRS can be used to predict these two properties from small wood samples. NIRS requires only a few minutes to perform an analysis, compared to several hours for traditional methods. Once the NIRS instrument has been calibrated, the technique allows us to predict the pulp yield and cellulose content from a large number of samples in a short period of time.

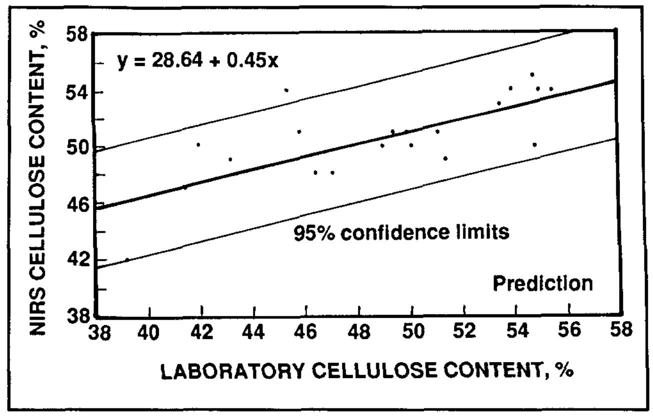
One trial was undertaken to test the predictive value of NIRS for pulp yield and cellulose content. The trial encompassed 26 trees and included almost equal representation of 20-year-old *P. taeda*, *P. patula*, and *P. elliottii*. Wood was taken from these trees as 25-mm increment cores. The laboratory methods used to determine pulp yield and cellulose content and to prepare the NIRS samples were similar to the methods described elsewhere (*11*).

Two statistical tests (*13*) were undertaken to compare the NIRS and laboratory methods (**Table III**). First, the data were compared by calculating the correlation coefficients for predicting total yield (**Fig. 3**) and cellulose content (**Fig. 4**). Second, the rankings of each tree by the two methods were compared using Spearman's rank correlation coefficient (*13*). This statistical technique is especially important when comparing trees to be included in a tree breeding program. Thus, NIRS must indicate those trees that

3. NIRS prediction of total pulp yield for pine wood samples



4. NIRS prediction of cellulose content for pine wood samples



III. Correlation coefficients and Spearman's correlation coefficients for comparing NIRS and laboratory methods

Trait	*No. of sample*	*Corre-lation coef.*	*Spearman's rank cor-relation coef.*
Total yield, %	26	0.668	0.616
Cellulose content, %	21	0.730	0.653

are the most suitable and that are the least suitable as obtained by conventional laboratory methods.

The coefficients of variation for predicted total yield and cellulose content were 4.98% and 6.91%, respectively. The coefficient of variation for total yield is less than that obtained for micropulping discs for whole tree analysis (*11*). The positive and statistically significant values for the correlation coefficient and the Spearman's rank correlation coefficient indicate that NIRS can be used to predict total yield and cellulose content with acceptable precision.

Conclusions

While the correlation coefficients are positive and statistically significant, the current NIRS calibration must be modified to improve its predictive ability. First, there may be a need to develop a separate calibration for each species under consideration: certainly the NIRS calibration used for pines will not work for eucalypts. Second, the calibration should include enough samples to give a normal distribution of values. As laboratory data are produced, small wood samples from the same trees can be used to update the NIRS calibration. Finally, NIRS should be calibrated to predict lignin content from small wood samples.

Work is in progress for both pine and eucalypt wood samples to increase the precision and uses of NIRS. At our company, we are currently using NIRS to sample individual trees in genetic trials, in order to use pulp yield or cellulose content as a selection criterion in a tree breeding program.□

Literature cited

1. Shenk, J. S., Landa, I., Hoover, M. R., and Westerhaus, M. O., Crop Science 21(3): 355(1981).
2. Shenk, J. S., Westerhaus, M. O., and Hoover, M. R., J. Dairy Science 62(5): 807(1979).
3. Marten, G. C., Halgerson, J. L., and Cherney, J. H., Crop Science 23(1): 94(1983).
4. Meyer, J. H., Proceedings of the South African Sugar Technologists' Association, Mt. Edgecombe, Natal, 1983, p. 1.
5. Wessman, C. A., Aber, J. D., Peterson, D. L., and Melillo, J. M., Can. J. For. Res. 18(1): 6(1988).
6. Birkett, M. D. and Gambino, M. J. T., Tappi J. 72(9): 193(1989).
7. Birkett, M. D. and Gambino, M. J. T., Paper Southern Africa (Nov./Dec.): 34(1988).
8. Berben, S. A., Rademacher, J. P., Sell, L. O., and Easty, D. B., Tappi J. 70(11): 129(1987).
9. Garbutt, D. C. F., and Donkin, M. J., Proceedings of Technical Association of the Pulp and Paper Industry in Southern Africa, Institute for Commercial Forestry Research, Pietermaritzburg, Natal, 1988, p. 229.
10. Michell, A. J., APPITA 41(5): 375(1988).
11. Wright, J. A., Shaw, M. J. P., Raubenheimer, S., and Hadebe, W., Tappi J. 72(4): 191(1989).
12. Savard, J., Besson, A., and Morize, S., Analyse Chimique des Bois Tropicaux, Centre Technique Forestier Tropical, Nogent-sur-Marne (Seine), France, 1954.
13. Snedecor, G. W., Statistical methods, 5th edn., Iowa State University Press, Ames, Iowa, 1956.

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