NEAR INFRARED SPECTROSCOPY IN THE FOREST PRODUCTS INDUSTRY

By Chi-Leung So, Brian K. Via, Leslie H. Groom,
Laurence R. Schimleck, Todd F. Shupe, Stephen S. Kelley, and Timothy G. Rials

Improving manufacturing efficiency and increasing product worth requires the right combination of actions throughout the manufacturing process. Many innovations have been developed over the last several decades to achieve these goals. Innovations typically work their way backwards in the manufacturing process, with an increasing level of monitoring occurring at the end of a production line. There exists, however, an ever-increasing array of tools available to forest products manufacturers that allow rapid assessment of material and product variables throughout the manufacturing process. A technology that shows great potential in all facets of material assessment is near infrared (NIR) spectroscopy.

The potential for NIR technologies has not gone unnoticed by the wood research community and there are scores of national and international laboratories developing appropriate applications. The rapid assessment of solid wood properties using NIR spectra is a fast-growing field that has broad implications in relation to wood quality and, ultimately, tree improvement. NIR as a means of online monitoring during the manufacturing process has also spurred many laboratories to examine potential applications for wood composites. It is probable that this type of monitoring will lead to increases in efficiency and profits.

NIR

NIR + MVA

These are two abbreviations that are creating exciting opportunities in the forest products industry.

Near infrared (NIR) spectroscopy involves the study of the interaction of electromagnetic radiation (in the NIR range) with materials. While our eyes can detect certain regions of the sun's radiation (i.e., visible region), the rest are invisible. Infrared radiation is just beyond what the human eye can see – NIR is the infrared region closest to the visible.

There are a range of spectroscopic techniques that can measure the chemical composition of a material; however, NIR spectroscopy can provide this information rapidly, with minimal sample preparation, and it is nondestructive. These attributes make it ideally suited to quality control and process monitoring applications.

Nevertheless, without multivariate analysis (MVA), the chemical information obtained from an NIR spectrum is limited. Together, a wealth of information can be acquired. MVA is a statistical tool that can be used to find relationships between one set of measurements that is cheap and easy to obtain (e.g., NIR spectra) and another set that is expensive and/or labor intensive to obtain (e.g., wet chemistry data or mechanical properties). With strong relationships, we can use the cheaper NIR data to rapidly predict the expensive measurements. For example, a commercial wet chemistry analysis is costly and may take days to obtain; this analysis can be achieved using NIR spectroscopy (with MVA) at a fraction of the time and cost.

BACKGROUND

The composition of a material and the manner in which it is assembled dictate structural performance as well as subsequent usefulness in a composite system. In the forest products industry, this fundamental information represents the basis for determining the chemical, physical, and mechanical properties of solid wood as well as composite components such as fibers, strands, or particles. The constant technological advancements over the years have led to improved methods for property assessment. In particular, the ever-changing face of computer technology has revolutionized the field of instrumentation, especially in terms of speed and accuracy. While this is applicable to traditional techniques for property assessment, it is particularly relevant to the development of NIR spectroscopy as a rapid assessment tool.

The NIR region extends from 780 to 2500 nm in which the spectra may be characterized by the assignment of the absorption bands to overtones and combinations of fundamental vibrations associated with C-H, O-H, and N-H bonds. The signals from these vibrations are similar, with resultant spectra consisting of many broad and overlapping bands, as opposed to the mid-IR (2500 to 25000 nm) region that consists of many sharp absorption bands highly characteristic to the sample. Mid-IR spectrometry is particularly suited to the structural elucidation and identification of organic compounds, but for quantitative analysis, mid-IR spectra are not as useful because they are highly influenced by instrumentation and sample preparation. However, NIR spectroscopy, with its lower cost instrumentation and rapid spectra collection (with little or no sample preparation), is ideally suited for quantitative analysis, and is particularly applicable to process monitoring and quality control applications.

The application of multivariate analysis (MVA) to NIR spectra has resulted in an upsurge of interest in NIR spectroscopy because it has reduced much of the problem of overlapping signals. Figure 1 shows the relationships between NIR spectra and some of the main constituents of wood: cellulose, lignin, and extractives. The application of MVA to NIR spectra first came to prominence with studies by Norris in the 1960s (e.g., Norris and Hart 1965) in which reflectance spectra were collected from wheat and other foodstuffs, revolutionizing the measurement of moisture in the food industry. Further studies were successfully conducted into

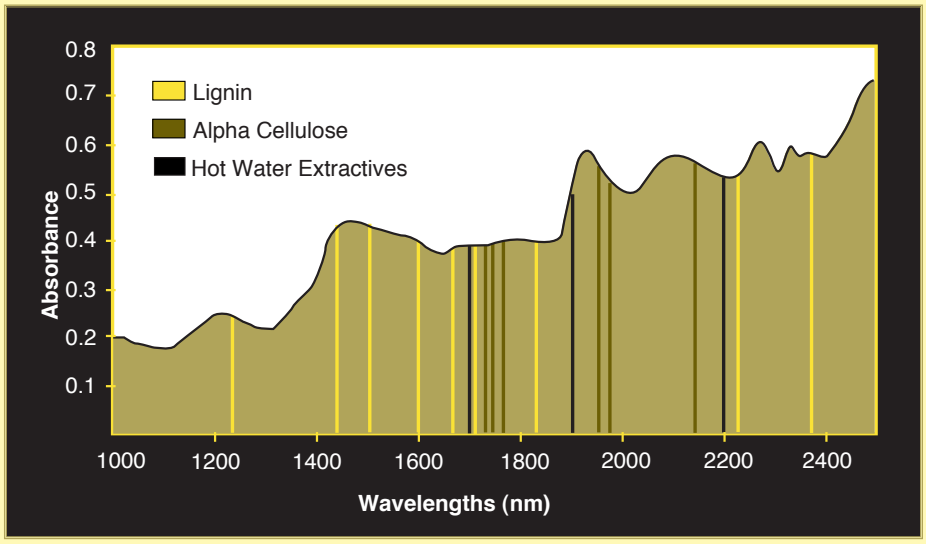

Figure 1. — Typical NIR spectrum of loblolly pine in the 1000 to 2500 nm region. Wavelengths associated with wood chemical constituents are highlighted.

other properties such as fat content (Ben-Gera and Norris 1968), which led to increased acceptance of this technology in the food and agricultural industries. Many years later, this technology was applied to the pulp and paper industry (Birkett and Gambino 1988, 1989; Wright et al. 1990; Wallbacks et al. 1991). Since then, its potential has been recognized throughout the entire forest products industry.

MVA utilizes chemical data in the development of calibration models for both qualitative and quantitative analyses. MVA techniques such as principal component analysis (PCA) and projection to latent structures or partial least squares (PLS) are often used for classification and prediction purposes. PCA involves a mathematical procedure that transforms a set of correlated variables into a smaller number of uncorrelated variables called principal components (or latent variables), orthogonal to each other. The first principal component accounts for as much of the variability in the data as possible, with each succeeding component accounting for as much of the remaining variation as possible. A PCA scores plot that compares different preservative treatments is shown in Figure 2. PLS is a regression technique that correlates subtle changes in spectra with independently measured material properties (Martens and Naes 1991). PLS can be used to describe the underlying latent structure in the predictor variables (e.g., NIR data) and response variables (e.g., moisture content) and generate a calibration that can be used for predictive purposes. The results of the calibration process are often presented in a plot of NIR-predicted property versus measured property; an example for wet chemistry data obtained from clearwood specimens is shown in Figure 3. PLS modeling also generates regression coefficients, or information on the chemical features that drive the calibrations. The regression coefficients can be used to relate chemical features in the spectra to the material properties.

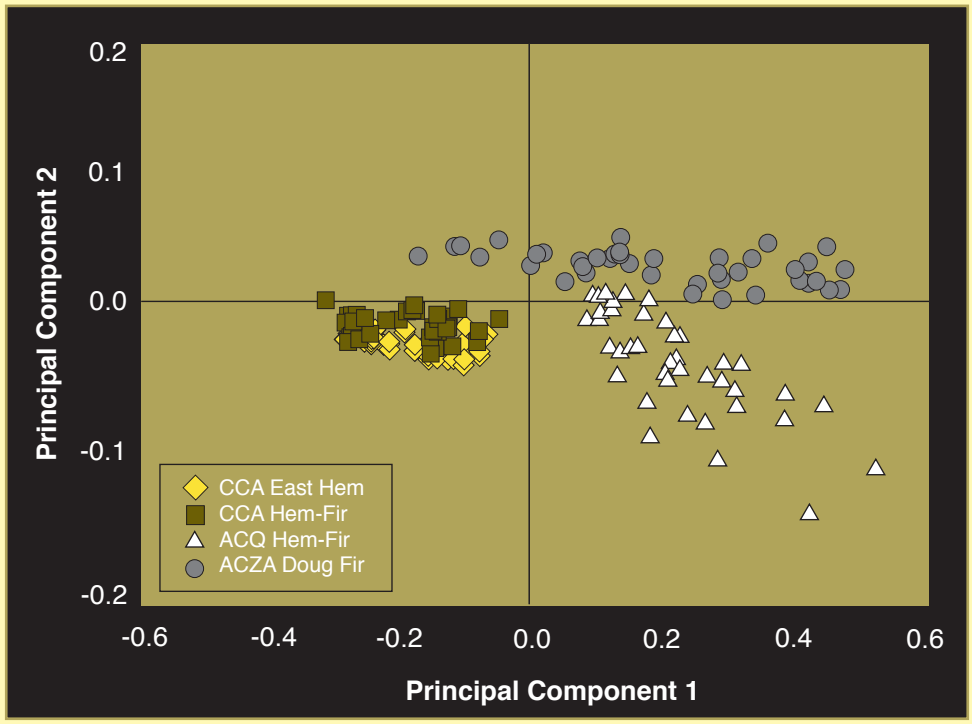

Figure 2. — Principal component analysis scores plot distinguishing wood treated with chromated copper arsenate, ammonium copper quat, and ammonium copper zinc arsenate preservatives.

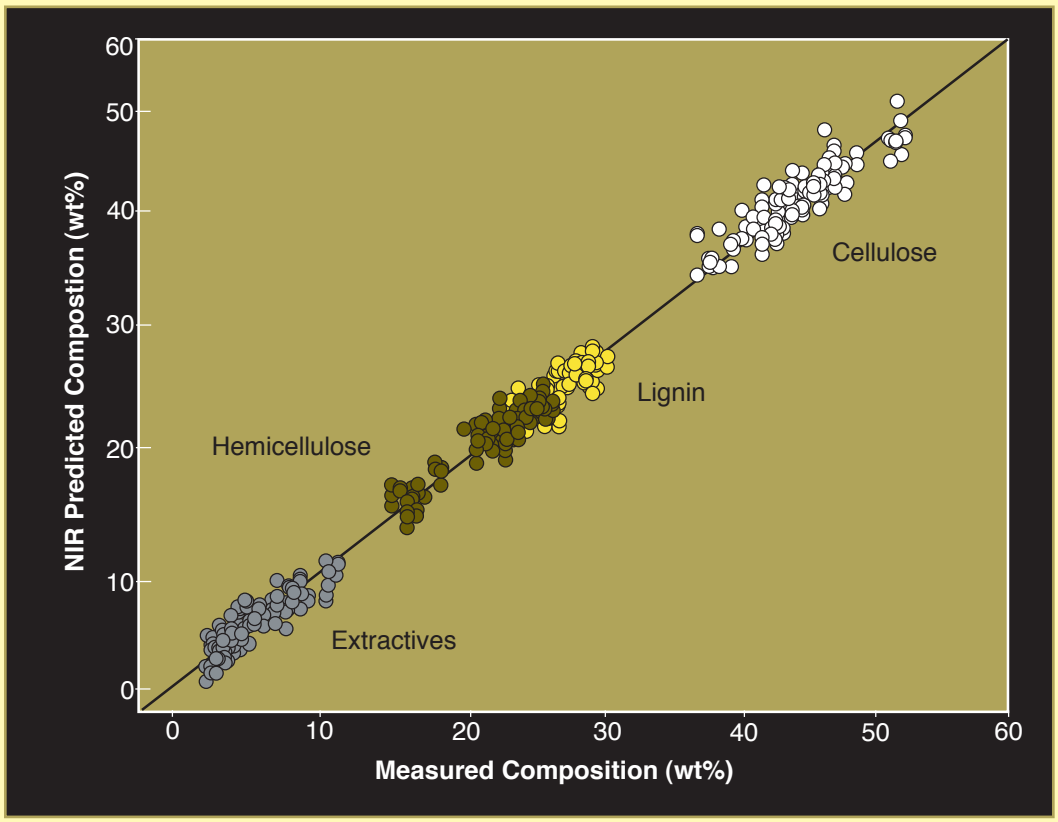

Figure 3. — NIR-predicted vs. measured plot of chemistry composition for milled loblolly pine samples.

Spectral preprocessing techniques are often applied to remove extraneous information from the NIR data with the purpose of producing strong, robust models. Traditional methods include first and second order derivatives, multiplicative scatter correction (MSC), and standard normal variate (SNV) transformation. These are often used to remove baseline offsets and slopes from spectra. The choice of pretreatment should be considered on a case-to-case basis, and is often a matter of trial and error. However, care must be taken not to lose relevant information during processing. Newer techniques involve the use of latent variables in the removal of unwanted variation in the data. Orthogonal signal correction (OSC) is one such method, involving the removal of latent variables orthogonal to the response used for calibration (Wold et al. 1998).

WOOD QUALTIY

Companies, government, and non-profit organizations internationally are beginning to focus more on wood quality. Wood quality has not been an important component of many tree improvement programs because of the inability to rapidly measure wood chemistry, fiber morphology, and mechanical and physical properties. Instead, improvement of stem straightness, removal of reaction wood, increasing disease resistance, and increasing volume have been the major focus of genetic improvement (Maynard 1976, White et al. 1993, Li et al. 1999, Land et al. 2001). Silviculture has likewise focused on volume improvement by controlling spacing, fertilization, ground vegetation, thinning, and site preparation (Larson et al. 2001). NIR spectroscopy provides a new tool that could potentially be used in tree improvement programs to rapidly assess wood quality of standing trees.

SilviScan, developed at CSIRO, Australia (Evans 1994, 2002), utilizes x-ray diffraction, x-ray densitometry, and image analysis to measure (both directly and indirectly) a range of wood properties from one increment core. Density, stiffness, microfibril angle, and tracheid properties such as tracheid diameter, coarseness, and wall thickness can all be measured. SilviScan data have been used to calibrate an NIR instrument to predict many of the properties measured by SilviScan (Schimleck et al. 2001a, 2001b; Schimleck and Evans 2002a, 2002b, 2004) and poten-

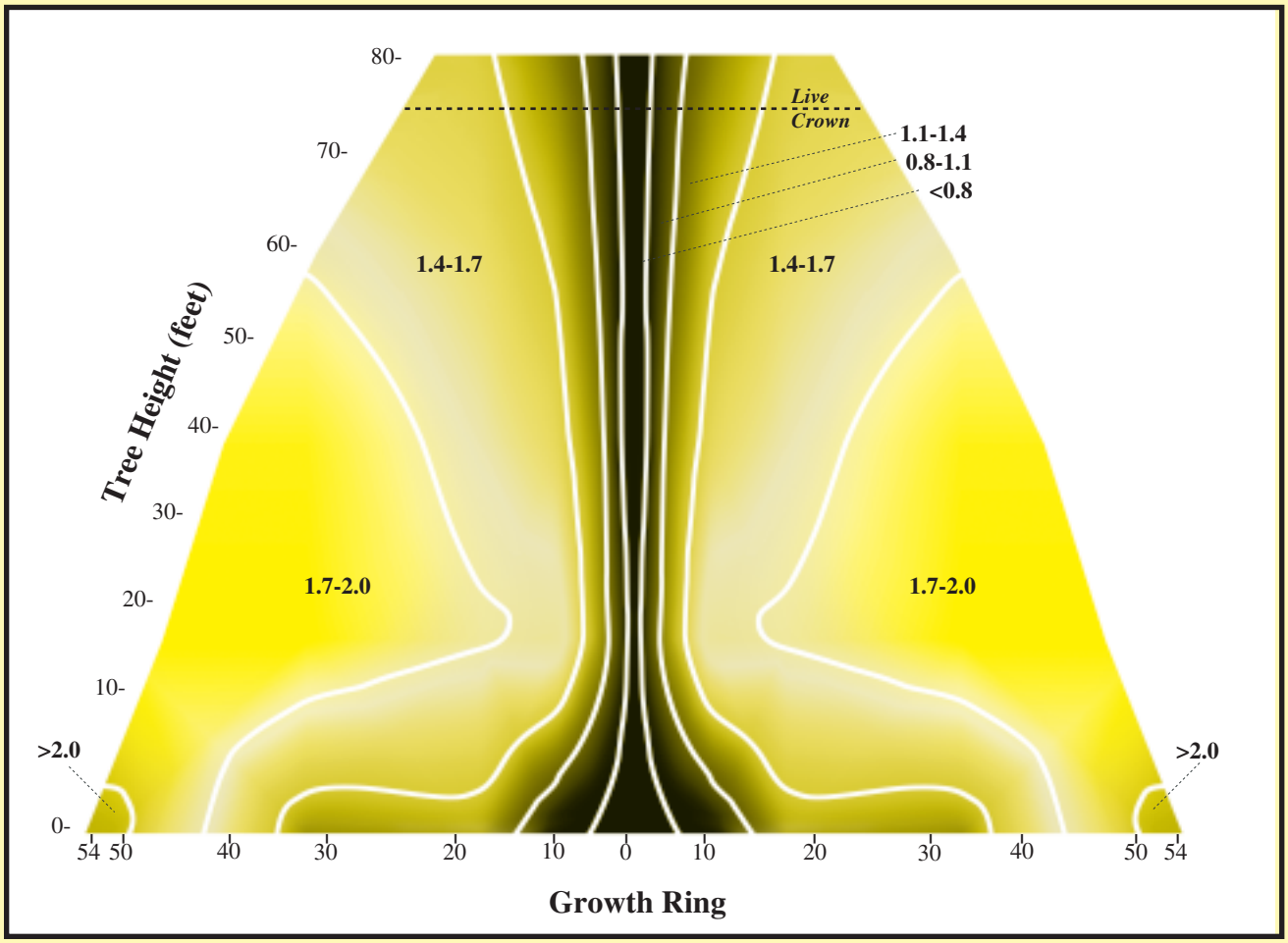

Figure 4. — Wood property map showing the variation of modulus of elasticity within a 55-year-old loblolly pine tree. Values shown are in 10^6 psi.

tially NIR spectroscopy could be used to complement SilviScan analysis.

Clearwood mechanical properties are a function of density, wood chemistry, and microfibril angle, and thus, NIR spectroscopy can be used to measure the stiffness of increment cores with good accuracy (Schimleck et al. 2001a, Schimleck and Evans 2002a). However, Via et al. (2003) found that its ability to predict strength and stiffness decreased when going from mature to juvenile wood in longleaf pine. Thus, age from the pith needs to be considered in determining the precision of a calibration equation for genetics trials.

The variation of stiffness both within and between trees can be large. Interactions such as live crown, specific gravity, and microfibril angle result in a three-dimensional maturation process that affects subsequent mechanical properties. The range and variation of properties within a tree can be visually described in the form of wood property maps (Evans et al. 1995, 1999, 2000; So et al. 2002), as illustrated in Figure 4. These maps show how growth conditions affect property variation within a tree and can be utilized for the application of silvicultural treatments. However, the construction of such maps requires much time and expense with traditional test methods; NIR spectroscopy can be used to predict fundamental wood properties with excellent results in a much smaller time frame. Further advances in technology using the visible-NIR region will lead to the production of handheld portable field units (Fig. 5) capable of assessing all major wood properties on green lumber (Meglen and Kelley 2003a, 2003b).

NIR spectroscopy could be a boon to tree improvement with respect to variables such as tracheid length, microfibril angle, wood mechanical properties, or wood chemistry. Data collection for these variables is uneconomical, because genetic trials commonly exceed 1,000 trees per study. Fortunately, the variables have been shown to correlate with NIR spectra. Spectroscopic measure-

ments were able to account for 70 to 77 percent of the variation of selected tracheid length index measurements on 48-year-old disks of Norway spruce (Hauksson et al. 2001). The spectra accounted for 66 to 95 percent of the variation for microfibril angle (Schimleck et al. 2001a, Schimleck and Evans 2002b, Kelley et al. 2004b). Several studies have also reported strong calibrations for cellulose, lignin, and extractives (Birkett and Gambino 1988, Schultz and Burns 1990, Wright et al. 1990, Kelley et al. 2004b). The strength and stiffness of a number of different species could also be predicted with NIR (Gindl et al. 2001, Hoffmeyer and Pedersen 1995, Kelley et al. 2004a, Thumm and Meder 2001).

PULP AND PAPER

Pulp yield is an important parameter for the paper industry and is a function of cellulose, density, extractives, and lignin content. Each wood constituent has a different capacity to resist chemical degradation, thus influencing the variation in residual pulp per volume of wood. The perpetual goal is to increase cellulose and density and decrease the lignin content and extractives. NIR spectroscopy has been used to determine pulp yield in both eucalypt and pine species with an acceptable level of accuracy (Birkett and Gambino 1988, Wright et al. 1990, Michell 1995).

Using pine, Antti et al. (1996) found that the NIR technique is a good predictor of porosity, paper caliper, tensile strength, paper density, tensile index, and scattering coefficient. The ability to predict stretch, burst strength, and burst index is less conclusive. Lignin (or kappa number), cellulose, glucan, uronic acids, viscosity, brightness, and degree of polymerization can be accurately estimated with NIR spectroscopy (Birkett and Gambino 1989, Schultz and Burns 1990, Wright et al. 1990, Axrup et al. 2000, Fardim et al. 2002). Measurement of lignin and xylan in black liquor can also be performed with almost 100 percent of the variation accounted for (Andersson and Wilson 2003). Excess residual lignin in the black liquor is undesirable as it is an indication that lignin may have precipitated back onto the pulp at the end of the cook. The ability to quantify the residual lignin in the form of control charts allows better monitoring of the pulp and paper manufacturing process.

An additional objective of building control charts is to monitor the variation of a parameter in addition to the mean value. The dispersion of a parameter can be critical in indicating a process change. One very important parameter that mill personnel monitor is kappa number, which is an indication of how much lignin is left in the pulp after digesting. An increase in the dispersion of the kappa number within a pulp batch is undesirable and may be due to improper mixing of the raw material or uneven cooking in the digester. NIR spectroscopy has been successfully used to measure the kappa number dispersion, accounting for over 90 percent of the variation (Antti et al. 2000).

One of the largest thrusts in the paper industry is to try to quantify the quality of the raw material going into the mill. Moisture and density information is needed to determine how much wood a mill is actually purchasing, since much pulpwood is purchased on a weight basis. Moisture meters measuring the absorbance or reflectance in the NIR range are available and currently measure the moisture content in chips going into the digester. The NIR range is very sensitive to moisture and therefore it can be measured independent of density. The base-

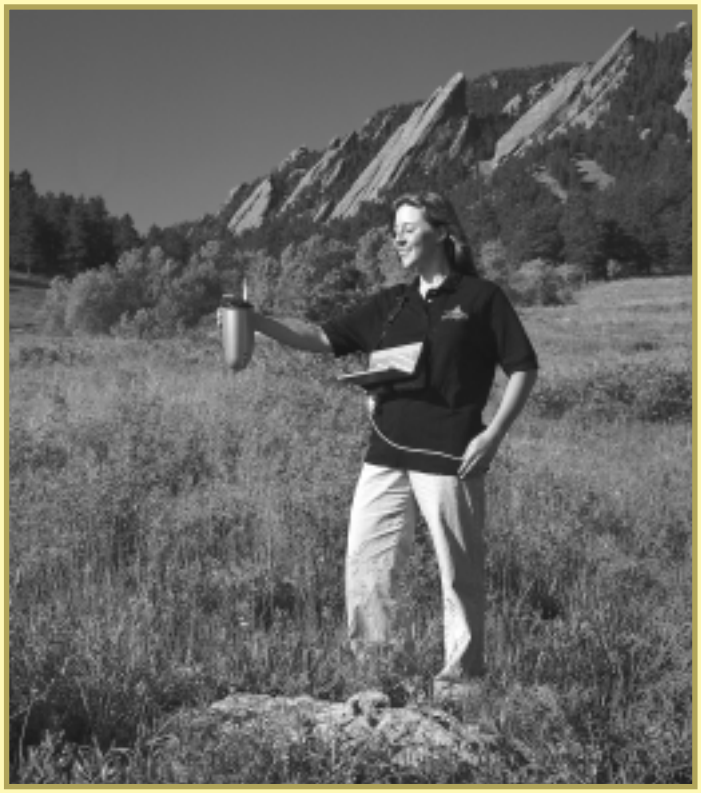

Figure 5. — Field monitoring using an ASD FieldSpec handheld portable spectroradiometer. *Photo courtesy of Analytical Spectral Devices.*

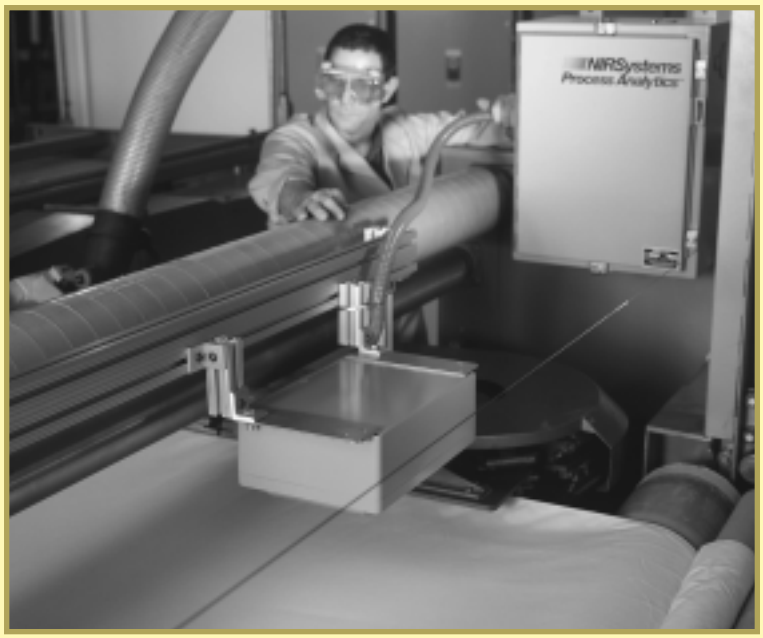

Figure 6. — Integrated NIR instrument for online process monitoring. ***Photo courtesy of Foss NIRSystems.***

line shift in absorbance is primarily dominated by density differences and this can be minimized by the use of derivative transformations. One of the main advantages of NIR is the ability to independently measure the density and moisture above the fiber saturation point; pin meters and CT scanners cannot differentiate between density and moisture variation. NIR can also be used to monitor the properties of sheets rolling off the production line, as demonstrated in Figure 6.

WOOD COMPOSITES

There is a diverse range of wood composites being manufactured for a variety of structural and nonstructural applications. The material properties of these wood composites are fundamentally determined by the properties of the individual components. While property variations in solid wood are studied at the cellular level, composite properties are studied with respect to the fibers, strands, and particles, as well as the adhesive resin. NIR spectroscopy has been used to study composite properties both within the laboratory and in manufacturing environments.

This technique has been used to determine the stiffness of radiata pine veneers (Meder et al. 2002). Testing of small laminated veneer lumber (LVL) samples was performed for calibration purposes, and the results showed the potential for NIR spectroscopy as a tool for stiffness evaluation prior to lay-up of plywood and LVL panels. An example of this is shown in Figure 7. Similarly, NIR has been used to characterize various properties of medium density fiberboard (MDF), including physical (density) and mechanical (modulus of elasticity, modulus of rupture, internal bond) properties (Rials et al. 2002a). Further studies compared the effect of reduced wavelength models (Rials et al. 2002b) and showed very little loss in predictive ability when compared to the full spectral range. This is essential for the development of rapid, low-cost instruments for process monitoring and quality control applications.

Metso Panelboard has used NIR technology as the basis for PanelPro™, a system developed for use in the production of MDF (Carlsson et al. 2002). Online monitoring of fiber properties, such as moisture content, fiber length distribution, and shive content, was used to improve the quality of the resulting fiberboard. The same technology has also been applied to particleboard manufacture for characterizing wood chips in terms of maturation, particle size, and species distribution, leading to the development of the BoardModel™ process modeling system by Casco Products, AB (Engstrom and Hedqvist 1999, Johnsson et al. 2000). This system utilizes NIR spectra for assessing the raw material quality, both in process monitoring and board quality predictions (for modulus of rupture, internal bond, and thickness swelling). Process variables, such as resin content, density, and press settings, are also incorporated into the modeling and adjusted to optimize the manufacturing process for the quality of the raw material used.

LUMBER PRODUCTS

Careful process monitoring is essential in all board manufacturing processes, especially with the possibility of significant variations in the quality of the wood raw material and high levels of resin consumption. NIR technology along the production line can be effectively used to optimize the whole process. Spectra collected from the raw material (feed-forward control) and the finished board (feedback control) can be linked with the desired board properties to provide the required process vari-

Figure 7. — Process monitoring for the assessment of veneer in the manufacture of plywood using a portable NIR spectrometer.

ables. This results in less variation in board quality and substantial cost savings.

The engineering limits imposed by codes make softwood lumber an ideal subject for rapid mechanical property assessment. The stiffness of softwood lumber can be used to upgrade the lumber value usually controlled by visual grading. When a piece has exceptional stiffness, it can be grouped into a higher value category than determined by visual grading alone. The stress-rating machine is perhaps the most valuable innovation to measure this parameter. The deflection can be measured and the stiffness estimated by applying a stress. This machine is unlikely to be completely replaced by NIR technology due to its ability to measure stiffness directly. However, the ability to predict stiffness independent of moisture, as well as additional parameters, may make NIR spectroscopy another attractive technique that increases profits and minimizes variability.

A potential application of this technology may also occur at various stations within a sawmill. One study involved the scanning, processing, and tracking of 180 radiata pine cants (Meder et al. 2003). The cants were scanned on the surface and then broken down into individual pieces of lumber. The NIR spectra were sensitive to surface properties of the cant, which made prediction of lumber stiffness possible but somewhat variable. Determining the stiffness of the cant would improve the product mix within the mill. The results of this study suggest that NIR technology can be used to estimate the value of the lumber from cants, but it is perhaps more suited for smaller dimension lumber since only the absorbance at the surface can be measured. This, and other work, showed that the mechanical properties of wood could be predicted even when the moisture content of the wood varied over a wide range (Meder et al. 2003; Meglen and Kelley 2003a, 2003b).

Another area of technological improvement using NIR spectroscopy is the kiln-drying station. Each charge of lumber could be measured to estimate how much moisture needs to be removed, due to the ability of this technique to measure the moisture and density with good accuracy. Kiln schedules could be tailored to the total density and moisture volume in the charge. The distribution of moisture and density would also be important since all pieces need to be dried below 19 percent moisture content.

THE MANUFACTURING ENVIRONMENT

NIR instrumentation coupled with fiber optics is fundamental for process monitoring applications and has allowed the collection of spectra in both harsh environments and inaccessible locations. While spectra collection in the laboratory can be in a controlled environment, this is generally not the case in a manufacturing environment, especially in the forest products industry.

Temperature and humidity have been shown to influence spectra response, with temperature having a greater impact than moisture content. In addition to changes in relative humidity, the response of NIR absorbance is compounded by moisture and temperature interactions. This is attributable to the fact that as temperature increases, the average number of hydrogen bonds at each O-H site tends to increase (Thygesen and Lundqvist 2000). This results in increased adsorption of moisture-dependent wavelengths (Swierenga et al. 2000). A change in temperature thus influences spectra at any wavelength sensitive to moisture. The reported wavelengths influenced by temperature change are 970, 1450, and 1930 nm (Swierenga et al. 2000, Thygesen and Lundqvist 2000).

At extreme temperatures, 95° to 105°C, the relation between temperature and spectra has been shown to be nonlinear and the temperature

should either be accounted for in the model or the sensitive spectra could be removed from the wavelength set (Wülfert et al. 2000). Neither method proved superior at those temperatures. However, between -20° to 20°C, inclusion of temperature as an independent variable in the model was successful in that it accounted for a significant portion of variation (Thygesen and Lundqvist 2000). As a result, temperature variation within the calibration data adds another component to the model (Hodges and Krishnagopalan 1999, Ferré and Brown 2001). This suggests that tight control of temperature within the laboratory could result in a reduction of factors (or principal components) needed in PLS regression to achieve the same standard error of calibration.

When temperature is relatively stable, humidity changes can be increasingly important. This is especially relevant since wood has a different sorption isotherm in desorption than adsorption. As a result, different drying or adsorption histories between samples will result in differing wood moisture contents, even though both samples have equilibrated to the same temperature and relative vapor pressure. Delwiche et al. (1992) found that for wheat starch and microcrystalline cellulose, NIR spectra were very sensitive to sorption history. In fact, their model could accurately predict moisture content differences even when samples were equilibrated to the same relative vapor pressure and temperature. Since wood is a hygroscopic material, this means that all specimens should be exposed to identical sorption history so they exhibit minimal moisture variation under equilibrium conditions.

DECAY

A current issue in the wood preservation industry is the removal and disposal of treated wood. It is important to be able to identify the type of preservative treatment present in wood marked for disposal. NIR spectroscopy has been used for the identification of various inorganic preservatives in timber, such as those containing arsenic, copper, and boron (Feldhoff et al. 1998). Furthermore, MVA techniques have been applied to NIR data (So et al. 2004) and PCA was able to distinguish between wood treated with various types of preservatives (Fig. 2). In addition, the levels of arsenic, chromium, and copper from different assay zones were also predicted. It was concluded that NIR spectroscopy had the potential for use in both field-portable and online applications such as the sorting and recycling of treated wood. The need for sorting technologies to distinguish treated waste wood has become greater now that the decision to voluntarily phase out the use of chromated copper arsenate for virtually all residential applications has come into effect.

Decay in wood can be monitored with NIR spectroscopy (Kelley et al. 2002) and the mechanical properties of treated wood and wood that has been exposed to decay organisms can also be predicted (Hedrick et al. 2003, Kelley 2003).

FUTURE APPLICATIONS

NIR spectroscopy has great potential as a powerful tool for improving production efficiency and profitability for many wood processing applications. Several of these have been discussed in this article. Further research is needed to develop NIR technology that can properly function under harsh industrial settings. Critical to the industrial application of this technology will be the ability to function in an environment with dynamic temperature and to collect data from material with variable moisture contents.

The application of NIR spectroscopy to the forest products arena is still relatively new. Although qualitative calibration equations can be easily produced, more effort is needed in determining the underlying functions of the spectra and how those relate to property estimation. Such information is important if more robust calibration equations are to be developed. A better understanding of the fundamental relationships would also aid the interpretation of NIR spectra for research applications. Studies into the use of reduced wavelength regions and/or larger spectral intervals have shown promising results, comparable to that of the full spectral wavelength. This will allow for faster processing of data, a requisite for process monitoring applications. Reduced wavelength regions allow the introduction of smaller, lower cost instruments suitable for field studies.

The last decade has yielded many breakthroughs that added to our knowledge base of NIR applications and provided further insight into the understanding of wood properties and processing. Future funding opportunities in this area of research appear to be great. The benefit for the forest products industry is likely to be even greater.

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The authors are, respectively, Postdoctoral Researcher, School of Renewable Natural Resources, LSU Agricultural Center, Baton Rouge, LA 70803; Ph.D. Candidate, School of Renewable Natural Resources, LSU Agricultural Center; Project Leader, USDA Forest Service, Southern Research Station, 2500 Shreveport Highway, Pineville, LA 71360; Assistant Professor, Warnell School of Forest Resources, University of Georgia, Athens, GA 30602; Associate Professor, School of Renewable Natural Resources, LSU Agricultural Center; Principal Scientist, National Bioenergy Center, National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401; Director, Tennessee Forest Products Center, University of Tennessee, 2506 Jacob Drive, Knoxville, TN 37996. This paper is published with the approval of the Director of the Louisiana Agricultural Experiment Station.