

Chemical Analysis of Wood Chips in Motion Using Thermal-Emission Mid-Infrared Spectroscopy with Projection to Latent Structures Regression

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The opacity and random surface orientation of process streams of solids complicates the application of spectroscopy to on-line analysis of solid streams. We have used a specialized form of thermal-emission mid-infrared spectroscopy, called transient infrared spectroscopy, combined with PLS regression to analyze a moving stream of wood chips for lignin, hemicellulose, glucan, and extractives content. Sixty-eight softwood samples from 6 species were analyzed using one regression, and 28 hardwood samples from 14 species were analyzed with another. Root-mean-square errors of prediction for the properties varied from 0.8 to 1.9 wt %, depending on property and wood type, with lignin and hemicellulose being the most accurately determined and glucan being the least.

Process analytical spectroscopy is a rapidly growing area because of the potential benefits in time and cost savings, process efficiency, and safety.¹ Most process analysis techniques require sampling or sample conditioning, but these steps are the major sources of process analyzer problems.² In-line analysis, where the analysis location is in the process line itself, does away with sampling and conditioning but places substantial demands on the analysis technique. This is especially true for process streams of solid, noncontinuous material, such as wood chips, grain, and coal, where the material is opaque and the orientation of the process stream surface is random and constantly changing. We have examined applying thermal-emission mid-infrared spectroscopy combined with projection to latent structures (PLS, also known as partial least squares) to a moving stream of wood chips. The importance of wood feedstock properties to the quality of paper and other wood-based products makes in-line wood chip analysis a highly desirable goal. In addition, wood chips are a good prototype for other particulate solid streams to which our approach could be applied.

Conventional chemical characterization of wood and pulp is tedious because of the complex nature of the material. Such characterization methods, including sample preparation, can require substantial time to complete.^{3,4} Spectroscopic methods have therefore often been examined as potentially much faster alternatives. Starting in the 1980s with the advent of FT-IR spectroscopy, a number of authors tested FT-IR-based diffuse reflectance spectroscopy (DRIFTS) in the mid-infrared for examining pulp or ground wood^{5–10} and occasionally intact wood wafers.¹¹ Mid-infrared photoacoustic spectroscopy was also used to study intact wood.^{12,13} In the 1990s, with the rise in popularity of near-infrared (NIR) spectroscopy, NIR DRIFTS was used and compared to mid-infrared DRIFTS.^{14,15} Most recently, Fourier transform Raman spectroscopy has been used.¹⁶ All of these proved successful to some degree, but all involved sample preparation and were not intended for on-line application. Fourier transform Raman spectroscopy has been done on pulp samples with little or no sample preparation, but the higher lignin content in unprocessed wood would cause interference from fluorescence.¹⁷

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It is well known that many physical properties of paper correlate well with characteristics of the pulp from which it is made.^{5,18–22} Pulp properties also can be correlated with wood chip characteristics. (Both of these types of correlations have been demonstrated by Meder et al.⁶) The existence of significant correlations between spectral features of pulp and the paper product indicates that multivariate modeling can be used to derive quantitative relationships between the end product quality and on-line process parameters.^{23,24} An important objective of this work was to demonstrate that PLS multivariate calibration combined with spectral instrumentation can be exploited in process control. Unpublished exploratory work previously performed in our laboratories demonstrated that the information contained in the infrared spectra provides a continuous link between raw material and final product. (This continuous link consists of correlations between properties of each material, from wood, through pulp, to paper.) The speed, simplicity, and low cost of infrared analytical tools make them attractive candidates for exploitation in industry.

In-line analysis performed in real time creates a new demand for rapid methods to evaluate process information. The infrared spectra of wood samples provide a rich information base from which statistical classification models can be developed. Spectral “fingerprints” can be used for species identification and could be useful in identifying unintentional feedstock mixing or in quantifying intentional blends. Similar juvenile wood “fingerprints” can be used for process optimization by adjusting the pulping severity to increase yield strength and reduce the load on the recovery boiler furnace. In addition to speciation and physical distinctions, multivariate calibration of infrared spectral data permits quantitative determination of carbohydrates, lignin, and extractives, and very good estimates of density. Axrup et al.²⁵ reported using NIR (800–1100 nm) reflectance spectroscopy combined with PLS to determine compositional properties of a moving stream of wood chips.

We report here on using transient infrared spectroscopy (TIRS) combined with PLS chemometrics as the approach for chemically analyzing moving wood chips. We have analyzed 96 different wood chip samples for their extractives, glucan, hemicellulose, and lignin content. TIRS uses the thermal infrared emission from a heated surface layer to acquire the desired infrared spectra.²⁶ A heated solid body produces thermal emission in the mid-infrared, but if the body is opaque, its emission spectrum is that of a blackbody, lacking compositional information. If the body is thin enough not to be opaque at mid-infrared wavelengths, however, its emission spectrum contains structure indicative of its composition, equivalent to the structure found by other spectroscopic methods.²⁷ Most solid material must be quite thin, no more than

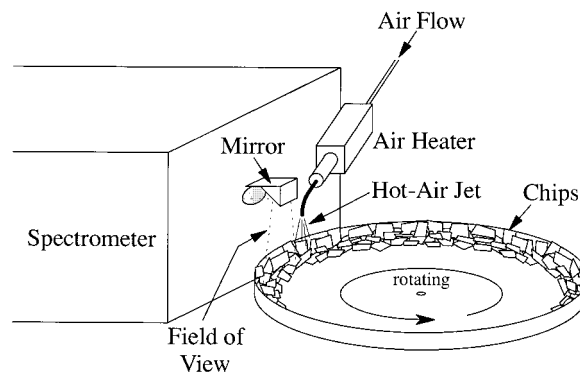


Figure 1. Experimental setup for TIRS of wood chips. Chips in a spinning platter pass through a jet of hot air and then through the field of view of an FT-IR spectrometer, which records the emission spectrum of the chips.

a few tens of micrometers thick, for good-quality emission spectra to be acquired. Obviously, physically thinning material is not feasible on line. TIRS circumvents this problem by making the sample material appear thin, without actually being so. TIRS uses a small jet of hot air to warm the surface of the moving process stream as it passes through the field of view of an emission spectrometer. The thin, heated surface layer emits much more strongly than the rest of the solid and its emission is structured rather than blackbody-like.²⁶ TIRS has broad potential applicability and has been tested in line on various process streams of continuous solids (i.e., films and sheet stock).²⁸ A short, qualitative test of TIRS applied to moving corn kernels has been reported,²⁹ but the present study is the first extensive, quantitative test of TIRS on a stream of noncontinuous (i.e., particulate) material. A brief report on the early stages of this study has already been published.¹² Previous work done in the laboratory of one of us (R.R.M.) and others⁶ indicates that the chemical composition (lignin, carbohydrates, moisture, density, extractives) of wood can be quantitatively determined using infrared spectroscopic techniques and state-of-the-art calibration modeling.^{15,30} Mathematical and statistical tools of PLS provide the method by which the spectra are converted into quantitative information.³¹

EXPERIMENTAL SECTION

The arrangement used to mimic TIRS on a wood chip conveyor is shown schematically in Figure 1. The wood chips were placed randomly along the inside rim of a 43-cm-diameter platter, which was spun to mimic the movement of chips along a conveyor. A Bomem MB100 Fourier transform infrared (FT-IR) spectrometer was fitted with a side port and an external 14-cm focal-length gold mirror to focus the field of view of the spectrometer down onto the stream of chips moving past on the platter. A jet of hot air was applied to the chips by an air heater (Leister 700 hot air tool) just upstream of the spectrometer field of view to produce the

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thin, heated layer TIRS requires. In an industrial application, the chips would pass the TIRS unit only once. In this laboratory arrangement, the chips spin repeatedly past the analyzer. To prevent the bulk of the chips from being warmed by repeated passes through the hot-air jet, a jet of cooled helium (not shown in the figure) was aimed onto the chips just downstream from the spectrometer field of view. In this way, the chips started out near room temperature each time they entered the hot jet.

The FT-IR spectrometer was fitted with a wide-band liquid nitrogen-cooled MCT detector, and its normal infrared source was replaced by a liquid nitrogen-cooled cold source. The spectrometer scanned at 1.50 cm/s optical path difference velocity and 8-cm⁻¹ resolution, taking ~0.8 s for each scan. Each spectrum consisted of 128 coadded scans, requiring 100 s to acquire. The spectrometer viewed an 8-mm-wide swath of the wood chip stream centered 19 cm from the center of the platter. The platter spun at 89 rpm, giving the wood chips a linear speed of 1.78 m/s (350 ft/min) through the spectrometer field of view. At this speed, the platter revolves 148 times during each spectrum acquisition, but as long as the cooled helium jet is set properly, the results are very similar to cases where any point in the material stream passes only once past the instrument. If the cooling jet is not completely effective in removing the deposited heat, the bulk of the material stream warms, and the spectrometer observes the structured TIRS emission atop an increased-temperature blackbody spectrum, which does not degrade the quality of the spectra but could complicate the PLS modeling. We have reported several tests of the TIRS instrument on materials other than wood where the tested stream consisted of continuously fresh material.^{28,32}

The hot air tool nozzle was 15 mm wide and 3 mm tall. The tip of the nozzle was 4 mm upstream from the leading edge of the spectrometer field of view and 19 mm above the face of the platter. Because of the random positions of the wood chips, the chip surfaces varied from 1 to 17 mm below the nozzle. The hot air tool ran at between 230 and 290 W with an air flow of 0.47 L/s. The data presented here were gathered in several sessions over a period of 17 months, and the setup shown in Figure 1 was broken down and reassembled between sessions, resulting in minor variations in the physical arrangement. The power level of the tool was adjusted to produce roughly constant total emission intensity from the samples from session to session, but no other compensation was made for the data being gathered discontinuously. The cooling jet consisted of helium chilled by passage through a liquid nitrogen bath sprayed from a 1-mm nozzle roughly 2 cm above the top of the chips at a flow rate of 0.11 L/s.

Several computational techniques have been used to develop multivariate statistical models for quantitative calibration of spectral techniques. However, the most advanced approach developed has been based upon partial least-squares projection to latent structures. PLS is a multivariate data analytic method that relates the systematic information in a matrix **X** to the information in a matrix **Y** with the purpose of predicting **Y** from **X**. In this case, the first step consists of obtaining several spectra of materials with known compositions. The **X** matrix consists of the absorption spectra (intensity versus wavenumber) for several

materials with varying chemical composition. The **Y** matrix consists of the known (independently determined) concentrations or measures of the properties of concern (e.g., lignin, carbohydrates, and extractive content). The statistical technique PLS simultaneously calculates multivariate projections of the predictor variables (spectral intensities) and the dependent variables (concentrations) so that the projection scores of the two data blocks are maximally correlated. In this way, a quantitative expression of the relationship (multivariate calibration curve) between the two matrices is obtained. Models derived in this way can then be used to extract the predicted concentrations of unknown samples for which the spectra have been obtained.

Raw TIRS spectra were obtained from each wood chip sample in duplicate (by mixing and replacement onto the rotation platter between spectra). The raw spectra of all samples were gathered between 501 and 2002 cm⁻¹ and stored at 390 equal intervals (of 3.85 cm⁻¹). The matrix of spectra (**X**-matrix, 136 or 56 spectra by 390 raw emission intensities) was transferred to a data file compatible with the Unscrambler multivariate calibration software (Camo, Inc.). The wood chip chemical compositions for model calibration were determined at NREL or provided by our industrial collaborators using the standard ASTM chemical methods for biomass, E1690-95 (ethanol extractives), E1721-95 (acid-insoluble residue), E1756-95 (total solids), E1757-95 (preparation of biomass for compositional analysis), E1758-95^{e1} (carbohydrates by HPLC), and E1821-96 (carbohydrates by GC).⁴ The known chemical compositions (lignin, extractives, glucan, hemicellulose) of the chip samples were added as the **Y**-matrix to the Unscrambler data files. Normalized spectral data files were constructed from raw spectra by subtracting the background signal and taking the ratio of the spectra to a reference blackbody spectrum. The results of calibration models for both raw and normalized spectra were obtained. While the normalized spectra provide a conventional appearance for interpretation (see Figure 2), the raw spectra provide identical statistical information for calibration modeling without pretreatment. Therefore, all calibration models discussed here were constructed from raw emission spectral intensities without preprocessing of any kind.

All calibration models were constructed by simultaneously obtaining the best prediction model for all four chemical components of the samples (so-called multiple block or PLS-2 modeling). Initially, hardwood and softwood samples were combined to produce a single calibration for 20 species. However, it is well known that lignins from hardwoods and softwoods are structurally distinct, while the differences among hardwoods or among softwoods are much smaller. Therefore, spectra from hardwoods and softwoods were ultimately placed in separate data sets for calibration modeling, which gave better precision than the combined model. The softwood model was constructed from 10 PLS factors while the hardwood model used 11 PLS factors. This number of required factors is expected for multispecies models used to predict four chemical constituents. All models were constructed using the statistical full cross-validation procedure. In a cross validation, each sample spectrum is held back (not included) in the model construction. That is, all of the other spectra were used to construct the model from which the held-back sample was predicted. Thus, the accuracy plots shown in Figures 3–6 show individual prediction points plotted against the

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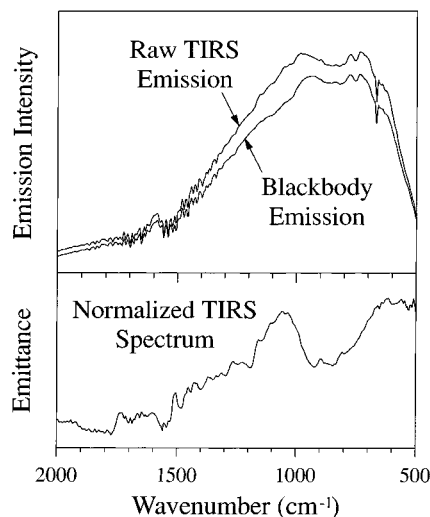


Figure 2. TIRS spectra of wood chips. Upper panel: The raw TIRS spectrum of southern yellow pine wood chips is compared to a 58 °C blackbody emission spectrum. Lower panel: The same TIRS spectrum as in the upper panel after normalization.

true or measured values based on models constructed from data on samples other than the one under scrutiny. This procedure allows for a more parsimonious use of the data collection and permits a robust accuracy assessment.

RESULTS

Figure 2 shows typical TIRS spectra acquired from the wood chips. In the upper panel, a raw TIRS spectrum of southern yellow pine chips is compared to a raw blackbody spectrum from carbon black powder heated to 58 °C, as recorded by the same spectrometer. The differences are modest but correspond to the desired wood spectrum. Raw emission spectra are usually normalized to compensate for any background signal, for spectrometer response, and for the decrease in intensity with increasing wavenumber that is characteristic of thermal emission. Normalizing involves subtracting off any background signal and taking the ratio to a reference (blackbody) spectrum.³³ The lower panel of Figure 2 shows the normalized version of the raw spectrum in the upper panel. The normalized spectrum shows the same peaks observed in more conventionally acquired infrared spectra of wood.^{6,8,13} The specific wood components and their vibrational modes that give rise to these peaks have been identified by others.¹³ Although spectroscopists usually study normalized spectra, PLS analysis can be done directly on the raw TIRS spectra, and the analyses described herein were performed using the raw spectra.

Spectra were acquired from 68 different bark-free softwood samples from 6 different species and from 28 bark-free hardwood samples from 14 different species. The softwood suite contained 30 Douglas fir, 6 hemlock, 21 loblolly pine, 4 ponderosa pine, 4 southern yellow pine, and 3 white fir samples. The hardwoods included 10 eucalyptus samples, 3 acacia samples, 3 mixed southern hardwood samples (i.e., each sample contained multiple species), and one sample of each of bay, blackjack oak, cherry, hickory, magnolia, post oak, red oak, sourwood, sweet gum, white oak, water oak, and yellow poplar.

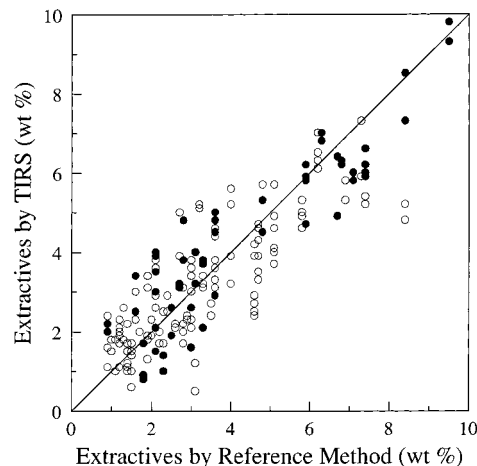


Figure 3. Single-elimination cross validation of TIRS-PLS analyses with reference method analyses for extractives content in (●) hardwoods and (○) softwoods.

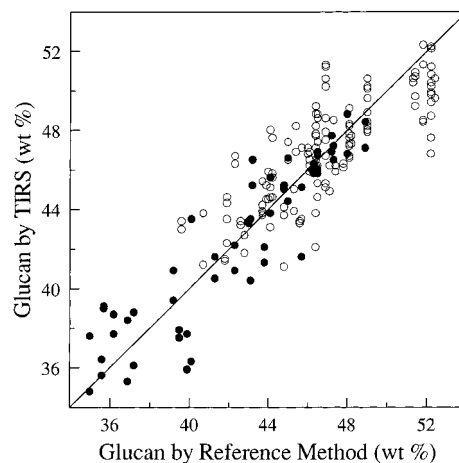


Figure 4. Single-elimination cross validation of TIRS-PLS analyses with reference method analyses for glucan content in (●) hardwoods and (○) softwoods.

Extractives, glucan, hemicellulose, and lignin were simultaneously determined from a single PLS model, but two models were developed, one for all softwoods and one for all hardwoods. Figures 3–6 show TIRS-PLS single-elimination cross validations for the four components. In each figure, the open circles are softwoods and the solid circles are hardwoods. Table 1 gives a summary of the statistics for the analyses. RMSEP is the root-mean-square error of prediction, and R is the correlation coefficient relative to the ideal line shown in each figure.

Glucan has the greatest RMSEP for both softwoods and hardwoods, but it also spans the greatest range of values. All of the RMSEP values are less than 2 wt %. Given that the precision of the reference analysis methods is approximately 1 wt %, this shows that TIRS-PLS is capable of determining the chip composition. The errors associated with the reference methods are discussed in detail elsewhere.³⁴ One source of variation in the TIRS-PLS measurements is the “presentation” of the sample to the TIRS analyzer. The strength of TIRS is that it senses only the near-surface region of a sample. This allows TIRS to analyze

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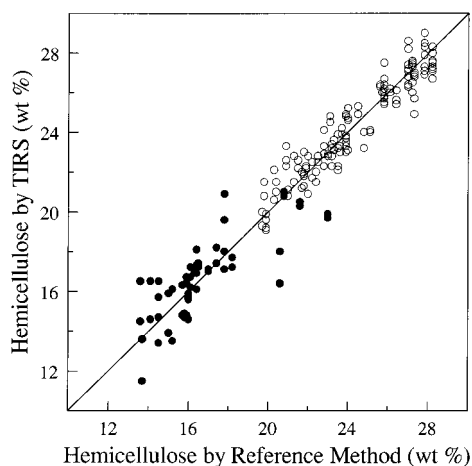


Figure 5. Single-elimination cross validation of TIRS-PLS analyses with reference method analyses for hemicellulose content in (●) hardwoods and (○) softwoods.

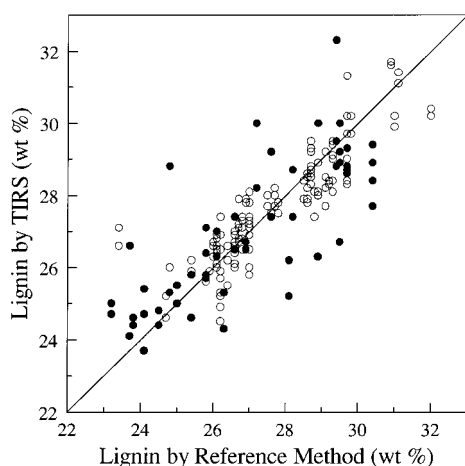


Figure 6. Single-elimination cross validation of TIRS-PLS analyses with reference method analyses for lignin content in (●) hardwoods and (○) softwoods.

opaque materials, but it also means that TIRS does not examine all of the sample material. If different faces of a heterogeneous sample are tested with TIRS, different analyses can result. With this in mind, duplicate TIRS spectra were acquired for each sample, as described in the Experimental Section, with the sample being taken out of and reloaded into the rotation platter between measurements. A single reference method analysis for each component was done on each sample, so the data points in the figures generally occur in vertically offset pairs, the size of the offset being the difference between the two TIRS-PLS analyses on different presentations of the sample. The rms difference given in Table 1 is the root-mean-square average of these differences between duplicate measurements of the same sample. The rms

Table 1. TIRS-PLS Analysis Statistics

property		range of values (wt %)	RMSEP (wt %)	<i>R</i>	rms difference ^a (wt %)
softwoods (68 samples, 136 spectra)	extractives	0.9–8.4	1.1	0.79	0.5
	glucan	39.6–52.4	1.9	0.79	1.4
	hemicellulose	19.7–28.2	0.8	0.94	0.7
	lignin	23.4–32.0	0.8	0.88	0.4
hardwoods (28 samples, 56 spectra)	extractives	0.6–9.6	1.0	0.91	0.8
	glucan	35.0–48.9	1.7	0.91	2.1
	hemicellulose	13.6–23.0	1.4	0.79	1.3
	lignin	23.2–30.4	1.4	0.78	1.6

^a The rms difference is the root-mean-square difference between pairs of measurements made on the same sample reloaded into the sample platter. See text.

difference is always a significant fraction of the RMSEP and even exceeds the RMSEP in two cases. This suggests that the variation in the sample presentation may account for a significant portion of the RMSEP. Of course, this is the TIRS version of the sampling problem that occurs whenever the properties of a large volume of heterogeneous material are to be determined by examining some fraction of the volume. TIRS generally has good sampling statistics because it observes the flowing stream continuously, averaging over a large surface area, but it does rely on the sample surface being representative of the sample bulk.

CONCLUSION

The chemical properties of a moving stream of wood chips have been successfully determined using transient infrared spectroscopy coupled with PLS regression. Root-mean-square errors for determining the major components of wood vary from 0.8 to 1.9 wt %. In the present study, the PLS-based chemical analysis was determined off line after the spectra had been gathered, but the analysis could be done in line in real time once a PLS regression model has been calculated.

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