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FT-NIR Spectroscopy and Wood Identification

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Keywords

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Wood identification
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Softwood
Hardwood

Summary

In the present project, a total of 90 samples of 12 different species of wood were examined using the FT-NIR method. It was found that a comparison of test results is only possible if the samples have been prepared in the identical manner. Furthermore, once the measuring unit had been calibrated, it proved possible to correctly identify new samples of the species previously tested. The results indicate that it may be possible to differentiate between samples of a given wood species of different origins. This is of some interest because it would allow the region in which the wood originated to be verified when the wood is certified.

Introduction

The application of infrared spectroscopy in the field of wood research is not new: Initial descriptions of wood and lignin based on their infrared spectra were published by Kratzl and Tschamler as far back as 1952. Mitchell (1969) described woods of taxonomic interest, and Chow (1972) characterized four softwood species with the aid of infrared methods. Kar-kin and his group applied this technique in a number of publications to describe the lignin of softwoods and hardwoods, and in particular that of birch (1981). Wienhaus *et al.* (1988) were able to draw a distinct line between softwoods and hardwoods on the basis of their differences in composition. In their work building on these foundations, Niemz *et al.* (1989, 1990) attempted on the one hand to determine the wood species themselves, and on the other hand to establish the content of bark, needles, formaldehyde, hardwood and softwood of tableted chip mixes. The latter posed no problems, but for the identification of the wood species these authors lacked the necessary mathematical-statistical method which would have allowed the goal to be attained with the aid of a cluster analysis.

Since recently, Fourier-transformed infrared analyses have become affordable and are now a permanent feature of, for example, lignin chemistry. Faix (1991) succeeded in applying the method to establish a classification system allowing the lignin spectra of seven genera of plants to be allocated to three main groups. This made it possible to obtain a semi-quantitative classification of a wide variety of lignins in practical applications. It must be pointed out, how-

ever, that this has still not eliminated the need for wet preparation of the samples in this area, and the measuring range is around wavelengths above approx. 2500 nm.

Measurements based on near infrared (NIR) cover the range below 2500 nm. Borga *et al.* (1992) applied these spectra to examine wet-stored round pine timber, and they compared the storage time with the quality of the different water types used. However, they could only determine 19 measurement points (1445 to 2348 nm) with their unit, and they used PLS (Partial Least Squares Regression) to process the data. The FT-NIR unit used for the present work, on the other hand, is capable of determining 500 measurement points in the NIR range (1000 to 2500 nm), and data processing is performed by the further developed Buhler Chemical Analytic Package (BCAP) software. This program package combines Principal Component Analysis (PCA), Partial Least Squares Regression (PLS) and Principal Component Regression (PCR). The objective of the present work was to establish whether it is possible to apply NIR spectroscopy (FT-NIR) to determine a given wood species and possibly its origin.

In the commercial production of chemicals, pharmaceuticals, plastics, cosmetics, printing ink, paints and foodstuffs (Seiler and Walder-Trenka 1992; Paduch 1992), the raw materials, the intermediates and the finished products are all subjected to continuous quality checks. This is necessary in order to ensure the consistency of product quality. In order to allow these inspections to be performed at reasonable cost, the efficiency of testing procedures must be increased

and the number of expensive costly wet analyses reduced. Now, quality assurance is precisely one of the fields in which the FT-NIR spectrometer proves to be of invaluable service. In large companies, the application of spectrometers in research, development and production has already become routine.

Buhler Ltd of Uzwil, Switzerland has developed the NIRVIS spectrometer system, which enables FT reflectance measurements to be performed in the NIR range. The NIR spectra, which contain information about the chemical composition and surface-specific physical properties of samples, are used by this system for detecting certain substances. The unit is primarily applied for the identification and quality control of substances containing C=O-, C-H, N-H-, O-H groups. The analytical process is based on state-of-the-art computing methods in conjunction with Fourier Transform spectroscopy. The main advantage of this approach over other systems operating in the NIR range is its user-friendly chemometric software, which permits a quick comparison of unknown spectra against calibration curves. Other benefits include the minimum training requirement involved, the fact that virtually no sample preparation is needed, and shorter analysis times. Moreover, the unit is easy to transport, rugged and flexible (fibre optics). This allows tests to be conducted also outside the laboratory (for example on site at the goods receiving point).

Materials and Method

Six different wood species each were tested in two series to determine the extent to which they could be distinguished.

Series 1: The sample volume for each wood species consisted of 2 to 5 samples with unprepared surfaces.

Series 2: The sample volume for each wood species consisted of a total of 11 sawed wood cubes (1 to 5 cm³). Three each had unprepared surfaces, and eight each were boiled in a 1:1 glyce-

rol-water mixture and additionally cut across the grain in a traversing microtome so as to produce an even surface (boiling times: *Jacaranda* approx. 2 hours (h); *Pinus sp.* approx. 6h; *Cedrela* and *Carapa* approx. 12h; *Laetia* approx. 70h). Apart from the wood species themselves, also the cellulose (bleached pulp) and lignin (lignosulphonic acid powder) contents of the samples were measured, which are both substances involved in papermaking. The samples were measured across the cross-sectional surface with the aid of the NIRVIS system by means of diffuse reflectance using fibre optics. The reflectance spectra in the range of 4500 to 10000 cm⁻¹ or 1000 to 2200 nm depend not only on the chemical composition of the sample, but also on the sample surface (quality and roughness). The surface quality of wood (cut or sanded) is therefore of primordial importance, which is why in the 2nd series eight samples of each wood species were tested with prepared (=standardized, ie boiled and cut) surfaces.

The NIRVIS spectrometer

Working principle

The measuring range is of crucial importance. Near infrared is adjacent to the visible light range and is assigned to the wavelengths between 800 and 2500 nm. In the near infrared (NIR) range, the only oscillations occurring are harmonic and combination oscillations of certain base frequencies. The NIR spectra result exclusively from oscillations with high base frequencies. They are the C=O, C-H, N-H and O-H absorption frequencies in the upper wavelength range of medium infrared (MIR), whose first upper harmonics are already located in the NIR range. The chemometric software of the system allows a maximum information content of the spectra to be utilized, the spectra being characterized by considerable overlapping of bands.

Analytical method

The novel feature of this analytical method is the Buhler Chemical Analytic Package software it uses. In addition to the Principal Component Analysis with a library module for the identification of diverse substances and the "cluster analysis" module for the classification of very similar substances or product grades, this software package includes two different calculation routines for quantitative analyses: Principal Component Regression and Partial Least Squares Regression.

In our tests, the qualitative model using cluster analysis was applied. The input values of PCA are the calibration spectra of known (=identified) samples. In PCA, related substances exhibit an approximately equal loading of the factors. When the spectra

Series	Scientific name	German/French trade name	English trade name	Family
1	<i>Andira inermis</i>	Rebhuhnholz	Angelin	<i>Papilionaceae</i>
	<i>Liriodendron tulipifera</i>	Tulpenbaum	Tulip poplar	<i>Magnoliaceae</i>
	<i>Manilkara bidentata</i>	Balatabaum	Bulletwood	<i>Sapotaceae</i>
	<i>Ocotea rubra</i>	Tabasco	Determa	<i>Lauraceae</i>
	<i>Pouteria guianensis</i>	Jaune d'oeuf	Asepoko	<i>Sapotaceae</i>
	<i>Trattinickia rhoifolia</i>	Olu	Thin-skin ulu	<i>Burseraceae</i>
2	<i>Carapa guianensis</i>	Demerara-Mahagoni	Crabwood	<i>Meliaceae</i>
	<i>Cedrela odorata</i>	Westindische Zeder	Red cedar	<i>Meliaceae</i>
	<i>Jacaranda copaia</i>	Kopaia	Futi	<i>Bignoniaceae</i>
	<i>Laetia procera</i>	Bois toucan	Warakairo	<i>Flacourtiaceae</i>
	<i>Pinus ponderosa</i>	Gelb-Kiefer	Pitch pine	<i>Pinaceae</i>
	<i>Pinus silvestris</i>	Waldföhre	Scots pine	<i>Pinaceae</i>

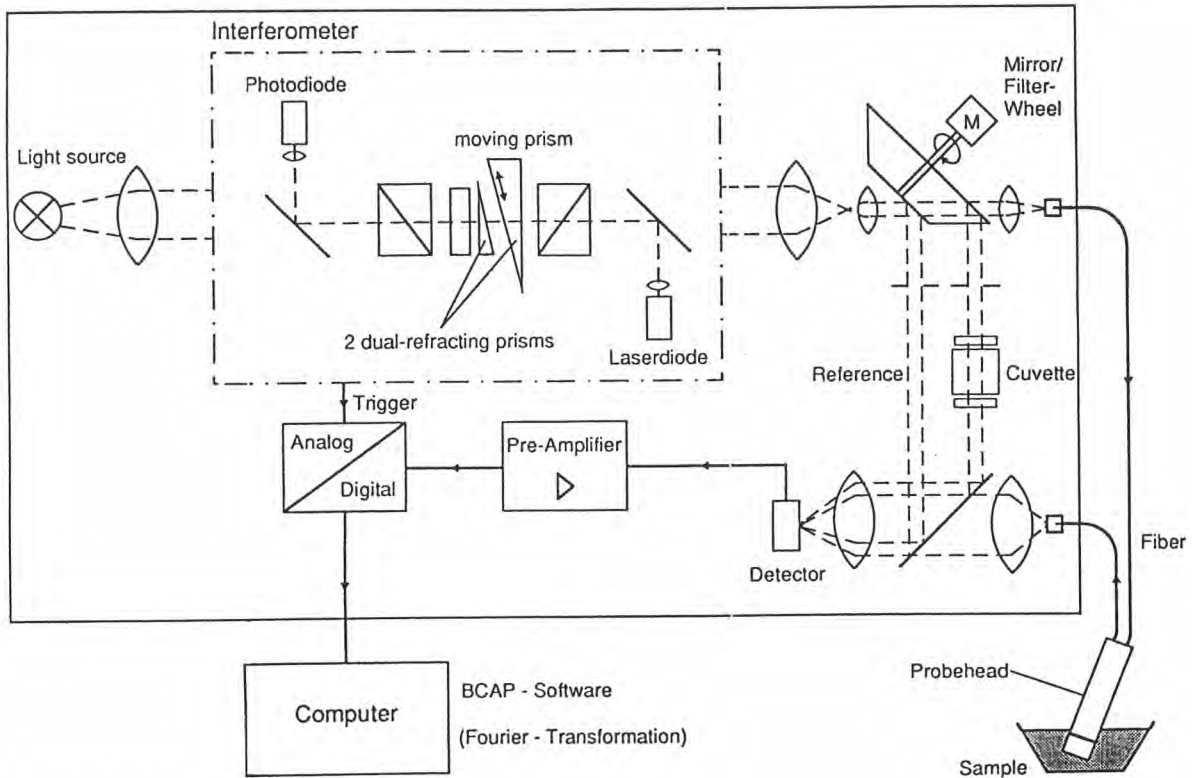


Fig. 1. Working principle of the Buhler NIRVIS unit.

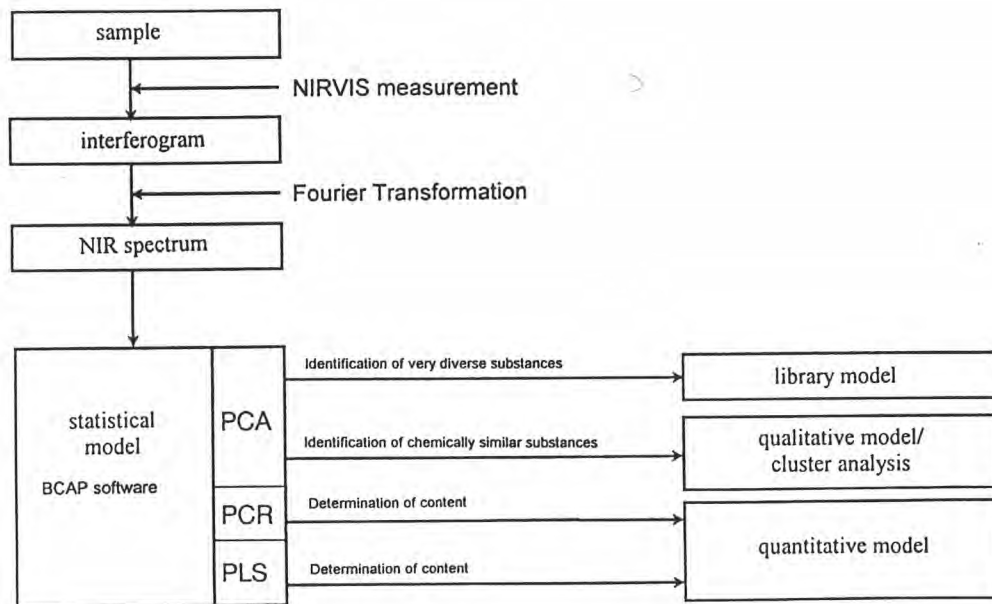


Fig. 2. Buhler NIRVIS analytical method.

are represented as points in a two- or three-dimensional factor plot, the spectra of identical or very similar products will be located closely to one another as a result of their almost equal loadings, whereas spectra of other substances or product grades are farther apart. The appearance of "clouds" formed by similar spectra, or in other words the occurrence of coherent sets of points in the loading space, is called "clustering". When a new and

unknown sample is measured and comes to lie – with regard to relevant factors – within a cluster defined by calculated boundaries, the product can immediately be allocated to a certain class and thereby be identified. Unlike the library module, which serves for verifying the identity of very diverse substances and which will not be discussed here, cluster analysis allows very slight differences in the spectra to be detected and utilized. In order to

achieve an appropriate degree of reproducibility, a maximum number of different measurement points and samples are required for calibration.

Results and Discussion

Interpretation of the spectra

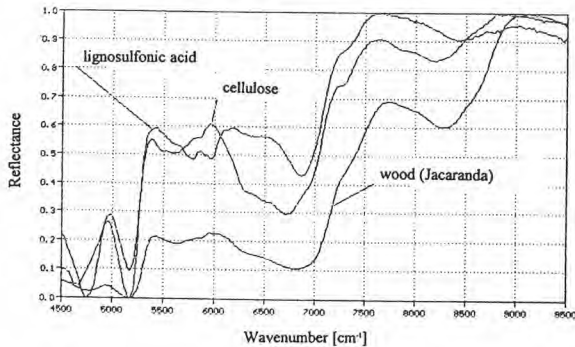


Fig. 3. NIR spectra of bleached pulp, lignosulphonic acid and various wood samples of *Jacaranda copaia* (series 2).

Wood consists of approx. 45–55% cellulose, approx. 15–30% lignin and 15–40% other constituents. The complicated composition of wood produces a spectrum which, unlike the spectra of pure substances, shows flatter and broader peaks (Fig. 3). Pronounced peaks are located at approx. 5200 cm^{-1} and 6900 cm^{-1} , which are attributable to hydroxyl (-OH) and carbonyl (-C=O) groups (water and cellulose). The peaks at approx. 5800 cm^{-1} and approx. 8200 cm^{-1} are caused by CH absorptions.

Surface quality

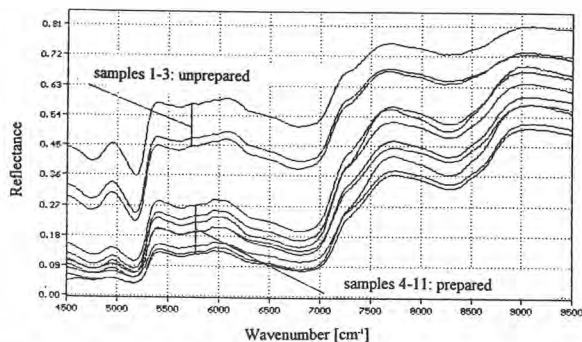


Fig. 4. 11 spectra of *Jacaranda copaia*. 1–3: unprepared samples. 4–11: prepared samples (series 2).

Figure 4 shows that the three unprepared samples exhibit markedly higher reflectance values than the treated samples, in particular in the lower wavenumber range. Besides the surface quality, this is attributable in particular to the modified chemical composition of the samples caused by preparation (boiling!), and to the associated change in the colour of the wood. The purpose of boiling is to plasticize the

wood, in particular the intercellular layer, in order to allow the specimens to be more readily cut to size. The boiling medium used is either distilled water or a mixture of 1 part of distilled water and 1 part of glycerol. The thermal process (Maillard reaction) causes the colour of the wood to become darker (browning), resulting in a higher absorption of the beam of light and therefore in a lower reflectance value.

Clusters obtained by the qualitative model

Different samples of *Jacaranda copaia* (series 2)

In the factor plot (Fig. 5), the spectra are represented as points (numerical values) based on the loadings of selected factors from the Principal Component Analysis. The difference between unprepared and prepared samples is evident here, too: every spot in the diagram stands for a specific range of spectra. All 10 samples measured are easy to distinguish, which may indicate differences of origin (which it was however not possible to determine unambiguously when the samples were taken). Sample 8 shows clearly how measurements taken at different points of a given sample may produce fairly wide variations while still allowing unambiguous distinction from the other samples. This demonstrates in an impressive way the capability of cluster analysis to detect extremely slight differences in the spectra which are no longer visible to the naked eye.

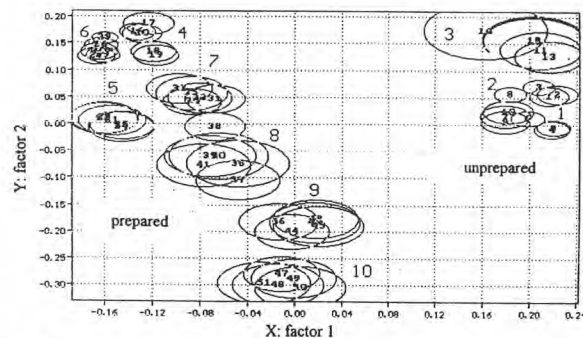


Fig. 5. 2-D factor plot of the prepared and unprepared samples of *Jacaranda copaia* (series 2).

Different wood species of series 1 (unprepared) and 2 (standardized surfaces)

Although the NIR spectra of the 1st series (Fig. 6) are very similar, the 3-D cluster analysis of the identical wood species (Fig. 7) shows pronounced differences. This can also be seen in Figure 8 for the wood species of series 2. Despite this fact, it proved impossible to achieve a 100% success rate in the determinations because of the limited volume of calibration data available. Additional tests based on a larger volume of spot samples would produce more unambiguous results.

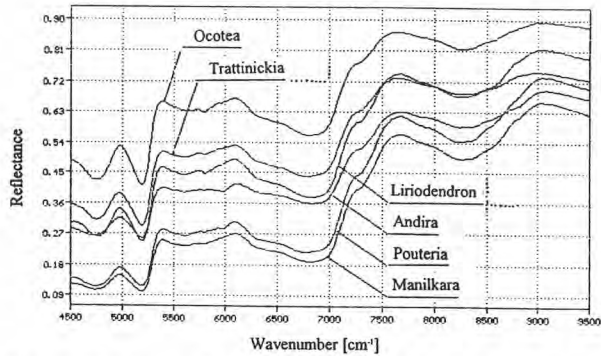


Fig. 6. NIR spectra of the 6 wood species of series 1.

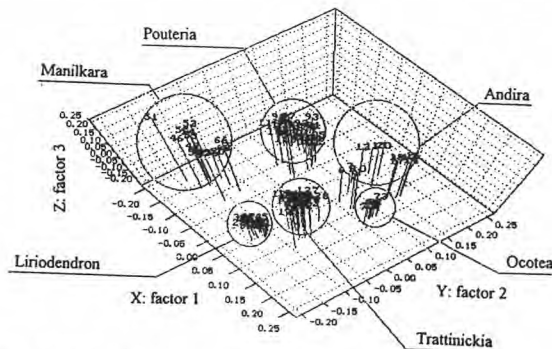


Fig. 7. 3-D plot of the 6 wood species of series 1.

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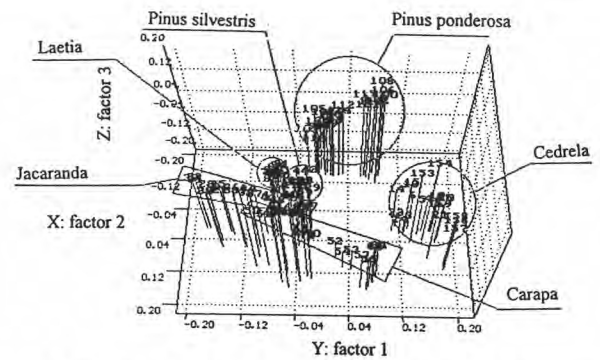


Fig. 8. 3-D plot of the 6 wood species of series 2 (prepared samples only).

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