HARRICK SCIENTIFIC PRODUCTS

No. 80312

application note

Fiber Optic Probes for Vis-NIR-MIR Diffuse and Specular Reflectance

Omni-Spec

Omni-Diff

FiberMate2

Figure 1. Fiber optic probes and coupler.

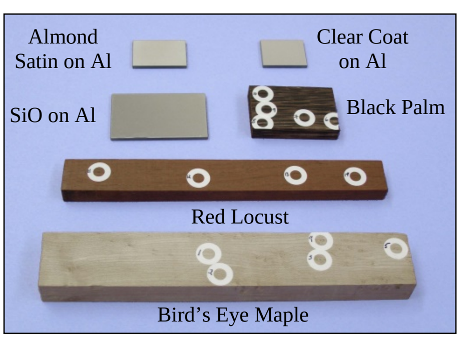


Figure 2. Samples. Wood samples shown with their binder reinforcements.

INTRODUCTION

Fiber optic reflectance probes have been used for remote measurements and analyses of large samples for many years. These fiber optic probes typically include optical components, such as lenses and fiber optics, built into the sampling head that limit their wavelength range.

New diffuse and specular reflectance probes have been developed using exclusively reflecting optics. This makes them suitable for use across a wide wavelength range when coupled with the appropriate fibers. An integrated camera allows real-time imaging and photographic documentation of the sample.

In this work, we examine several wood samples, using mid-IR and Vis-NIR diffuse reflection spectroscopy, to observe the changes in composition along the grain boundaries. In particular, black palm, red locust and bird's eye maple will be examined. In addition, several coatings on substrates will be studied by specular reflectance in the mid-IR and Vis-NIR.

EXPERIMENTAL

Measurements were carried out using the Omni-Diff and Omni-Spec fiber optic probes for diffuse and specular reflectance respectively (see Figure 1). These probes were connected to a commercial FTIR or UV/Vis/NIR spectrometer using 1.5m long fibers via the FiberMate2 coupler. Depending on the wavelength range under investigation, chalcogenide (CIR, 1000µm core dia.), silver halide (PIR, 900µm core dia.) or fused silica (1000µm core dia.) fibers were used.

The FTIR spectrometer used a DTGS detector and collected 128 scans at 8 cm^{-1} resolution. The spectrometer gain was set to 8 and its aperture was fully open. The UV/Vis/NIR spectrometer was set to double-beam mode with a data interval of 2.0nm, a SBW of 2.0 nm and fully open slit. An attenuator was used in the reference beam to balance the energy levels in the sample and reference beams. InfraGold™ and Spectralon™ were used as reference materials for the FTIR and Vis/NIR diffuse reflectance measurements respectively, and a front-surface aluminum mirror was used as a reference for the specular reflectance measurements. The Vis/NIR spectra were normalized for ease of interpretation. The FTIR spectra were smoothed and the spectra collected with PIR and CIR fibers were merged for each to provide a wider spectral range

For the diffuse reflectance measurements, the Harrick Omni-Diff diffuse reflectance probe was used to examine three different types of wood: Red Locust, Black Palm and Bird's Eye Maple. Binder reinforcements were placed on various positions on three pieces of wood to measure different parts of the grain (see Figure 2). The Omni-Diff camera was used to center the probe on each binder reinforcement prior to measurement, so the same area of the sample could be measured in both the FTIR and Vis/NIR. For each position measured, a photograph of the sample was also taken using just the illumination from the camera. Since the camera has a larger field of view than the area studied by the Omni-Diff, the images were cropped to show just the area examined. To determine the location and area of the approximately 1-2mm diameter spot illuminated by the spectrometer, the UV/Vis/NIR spectrometer was set for 'white light' and a photograph was taken of one sample. This was then used to create a template and crop the photographs of the samples accordingly in Adobe Photoshop

For the 45º specular reflectance measurements, the Harrick Omni-Spec was used to examine several coatings deposited on aluminum mirrors. The coatings included 500Å thick SiO, Krylon Satin Touch 3511 Almond Satin spray paint and Krylon Crystal Clear Acrylic Coating 1303.

Results and Discussion

I. *Diffuse Reflectance*

The diffuse reflectance spectra of the three different types of wood for both the Vis/NIR and FTIR spectral ranges are presented in Figures 3 through 5, along with photographs of the particular positions measured. Note that the Vis/NIR spectra of all the positions examined are shown, while the FTIR spectra display only two samples from each type of wood. Due to the low S/N of the Omni-Diff plus FiberMate2 used with a DTGS detector, minimal differences were observed at various positions along the grain.

Both the Vis/NIR and FTIR spectra show expected characteristics from the lignans and water expected in wood. In the Vis/NIR spectra, the peak around 1936nm is due to an overtone of the C=O stretch possibly with a contribution from an H_2O overtone. The band at 1504nm is due to an N-H overtone, while the bands at 1244nm and 1604nm arise from C-H and ArC-H overtones respectively. The peak at 1238nm is due to a C-H overtone and the one around 1458 is due to an O-H overtone. In the FTIR spectra, the broad

Figure 3a. Vis/NIR Diffuse reflectance spectra measured from several sites on bird's eye maple.

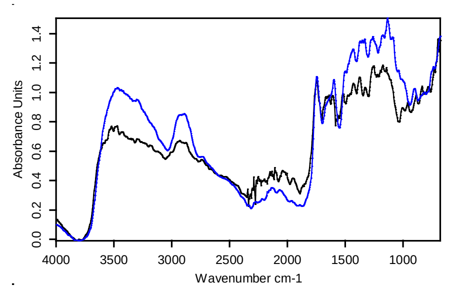


Figure 3b. FTIR Diffuse reflectance spectra measured from several sites on bird's eye maple.

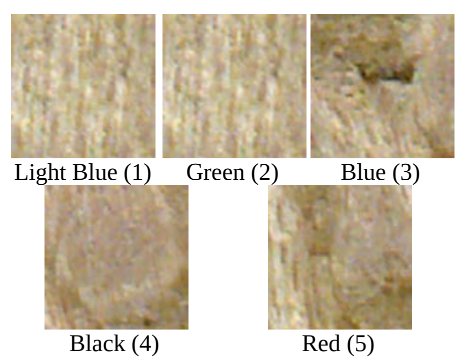


Figure 3c. Photographs of the sites examined in 3a and 3b.

Fiber Optic Probes for Vis-NIR-MIR Diffuse and Specular Reflectance

Figure 4a. Vis/NIR Diffuse reflectance spectra measured from several sites on black palm.

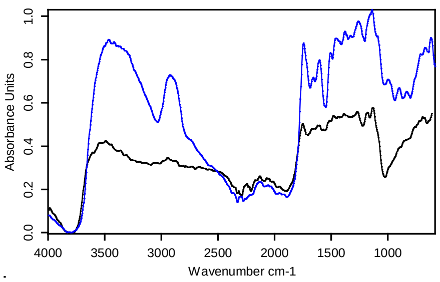


Figure 4b. FTIR Diffuse reflectance spectra measured from several sites on black palm.

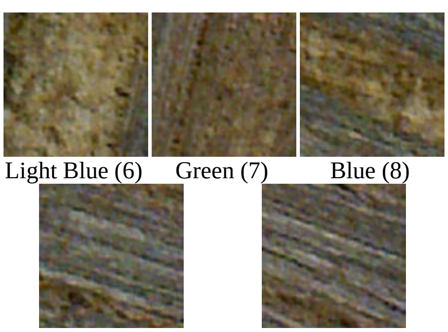
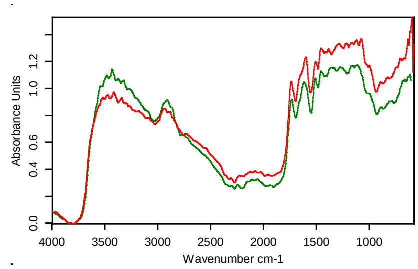


Figure 4c. Photographs of the sites examined in Figures 4a and 4b.

band near 3500cm^{-1}, combined with the peaks around 2100cm^{-1} and 1750cm^{-1}, indicate the presence of water within the sample. The band around 2900cm^{-1} is due to C-H stretches, as expected. The fingerprint region shows many additional peaks but they are difficult to distinguish as a result of the noise level in the spectra. For a more in depth study of these bands, a more sensitive MCT detector is required.

For the bird's eye maple sample (Figure 3) samples 1 and 2 were measured in areas that had uniform grain; samples 4 through 6 were recorded from knots in the wood. Two of the measurements on knots, samples 5 and 6, show a broader peak in the visible, as expected since the knots are typically darker than the rest of the grain. These also show a shoulder around 500nm, indicating that they are slightly more green in color than the rest of the areas examined. The infrared spectra show a slight overall reduction in intensity for the spectra of the knots and this may originate from a difference in surface texture. The C-H band around 2900cm^{-1} also appears slightly more intense relative to the –OH band near 3500cm^{-1}, which may indicate a lower water content in the knot.



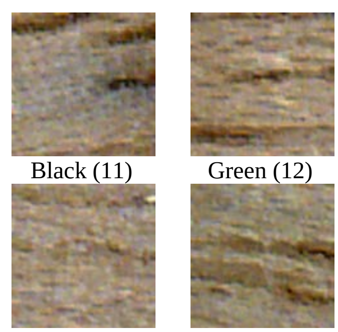


Figure 5c. Photographs of the sites examined in Figures 5a and 5b.

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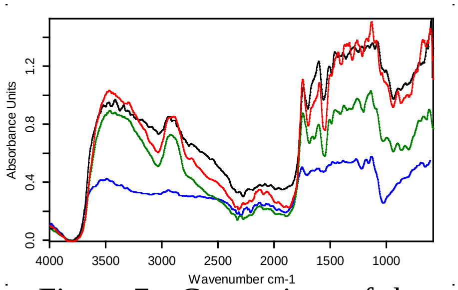

Figure 7. Comparison of the FTIR diffuse reflectance of several different woods: maple (red), palm (blue and green) and locust (black).

The black palm Vis/NIR spectra (Figure 4a) show much more spectral and color variations between locations on the sample than the other two types of wood. Samples 9 and 10 have a broad shoulder around 625nm, indicating that they are redder or darker than other portions of the sample. The FTIR spectra (Figure 4b) show a marked change in overall reflectivity as a function of position, probably due to surface finish. Both the Vis/NIR and FTIR spectra of the red locust show very little variation across the grain. The primary differences in the spectra could easily be due to slightly different surface finish.

Figures 6 and 7 compare the spectra from the three types of wood in the Vis/NIR and FTIR respectively. Due to the large variation in the spectrum of the black palm, two samples are included herein for reference. Based on the overall intensity of the bands in the visible region, it is clear that the bird's eye maple is the lightest color wood. Because of the large local variations in color, it is difficult to tell based solely on the Vis/NIR spectra whether the black palm is lighter or darker than the red locust. The FTIR spectra show some differences in water content and surface finish.

II. *Specular Reflectance*

The Vis/NIR and FTIR spectra of several coatings on aluminum mirrored surfaces are shown in Figures 8 and 9, respectively. The two Krylon spray paints exhibit similar mid-infrared spectra. From the FTIR spectra, both exhibit spectral bands in the 2850-3000cm^{-1} region which are characteristic of alkane C-H stretches and in the 1710-1750 cm^{-1} region from the carbonyl group. Both also have peaks around 3400cm^{-1}. Since this band in the Almond Satin is broad, it is probably due to an OH stretch. The narrower band in the acrylic may be from an NH stretching vibration. From the Vis/NIR spectra, both have CH combination bands around 2288nm and CH overtones at 1730nm. The visible spectrum also shows a broad peak that starts at the 560nm and ends at the short wavelength cut-off the fiber, with shoulders at 500nm and 470nm, indicating that this sample is colored with hints of blue and green. Based on the differences in these spectra, it is clear that these two paints have different compositions.

The other spectra shown in Figures 8 and 9 is that of a 500Å SiO coating on an aluminum mirror. The FTIR spectra show the distinctive SiO band at 1212cm^{-1}. In addition, there is a weak broad band at 3350cm^{-1}

and a band at 2286nm from moisture on the surface or bound Si-OH. The spectral features in the 350nm to 950nm region are due to the differences in reflectivity of the aluminum coatings on the sample and reference.

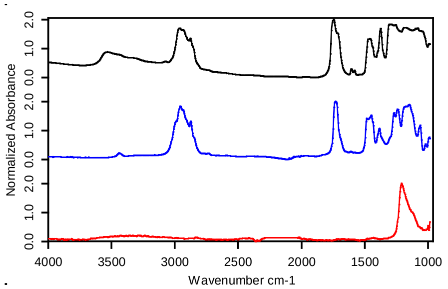

Figure 9. FTIR specular reflectance from several different coatings on aluminum: 500Å SiO (red), Clear Coat (blue) and Almond Satin (black).

CONCLUSION

In this work, several wood samples were examined by diffuse reflectance using the Omni-Diff probe in the mid-IR and Vis/NIR. Spectroscopic differences were observed in color, moisture content and surface finish. In addition, a selection of coating substrates was examined using the Omni-Spec probe over the same wavelength range. Suitable spectra were obtained to compare coatings and qualitatively analyze.

These results demonstrate that these new diffuse and specular reflectance fiber optic probes are suitable for use across a wide wavelength range when coupled with the appropriate fibers. For the mid-infrared, an MCT detector would be preferred over a DTGS for achieve higher signal-to-noise performance.