Spectral Reflectance Applications with Miniature Fiber Optic Spectrometers

Miniature fiber optic spectrometers that interface to computers have been around for about 10 years. These small instruments are low-cost and therefore widely used in many different industrial applications across a variety of markets.

Example markets and applications include aerospace, agriculture, food and dairy, biomedical, pharmaceutical, chemical and petrochemical, cosmetics, educational, research, semiconductor and thin film measurement, lamp and lighting, LED and laser development, optical component characterization, paint color formulations, pulp and paper processing, telecom monitoring, fiber optic sensing, and UV process manufacturing.

In recent years, significant design improvements have been made to ruggedize miniature fiber optic spectrometers for industrial and portable applications. The following article will show, how US firm StellarNet has implemented several optical features and high speed USB-2.0 computer interfaces to enhance performance of such devices.

1 Spectrometer technology

Fig. 1 shows a UV-VIS spectrometer with improved optical performance due to a holographic aberration corrected concave grating. This type of optical design eliminates 2 mirrors, which minimizes stray light effects. The concave grating projects a flat field wavefront on the detector array, providing a uniform 1 nm resolution across the wavelength range 190-900 nm (using a 25 µm slit). The improved focus provides superior imaging of the raw spectrum and eliminates aberrations. The correction of aberrations, namely coma, spherical aberration, astigmatism and field curvature, makes concave gratings popular for many spectroscopy applications.

Holographic gratings have some significant advantages over ruled gratings. The most important advantage is reduced surface roughness. Reduced surface roughness decreases grating scatter and can improve detection limits. The performance of spectrometers and spectrographs is judged by system resolution, throughput, and system limit of detection. These criteria involve grating efficiency, system light collection capability, system stray light and design aberrations. The use of holographic concave aberration corrected gratings allows optimization of all these parameters with a single optic.

Additionally, the gratings do not require positioning adjustments, making the instrument insensitive to shock or vibration.

For similar reasons, the detector array is not mounted into a socket; it is soldered to a circuit card that is bolted to the optical spectrograph. This design configuration minimizes optical alignment drift that would otherwise occur with temperature fluctuations. For additional protection from environmental exposures, the optical spectrograph is mounted inside a rugged miniature enclosure.

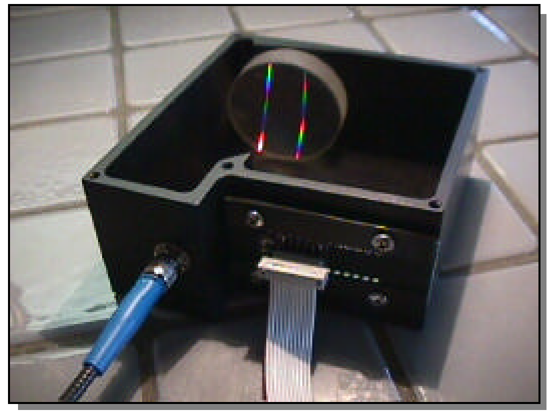

Fig.1: StellarNet's spectrometer optics, here shown in an EPP2000C UV-VIS spectrometer, consists of an aberration corrected concave grating with no mirrors and a securely mounted detector. There are no moving parts for portable applications that require vibration tolerance.

2 Detectors

Charge-coupled device arrays (CCDs) are primarily used for UV-VIS applications because of their high sensitivity in the 190-900 nm wavelength range, and their low dark current, which improves the spectrometers dynamic range. These linear array detectors typically have 2048 elements with each pixel being 14 µm wide x 200 µm tall.

There are some rather interesting applications which occur in the Near Infra-Red (NIR) 900-2200nm range, which use InGaAs Photo Diode Arrays (PDAs) instead of CCDs. The InGaAs PDAs, while more expensive, offer additional benefits. StellarNet has adapted both a 512 and 1024 pixel InGaAs PDA detector into its line of miniature fiber optic spectrometers.

3 Spectrometer-to-computer interface

Most new computers are now equipped with high speed USB-2.0 interfaces, replacing the older USB-1.0 standard, which was 40 times slower. USB-2.0 ports simplify peripheral installation without degrading data acquisition performance. E.g., with respect to EPP2000 spectrometers, the USB2EPP interface cable enables many new applications which require multiple channels and higher speed spectral data for process monitoring and control.

Most of the time, for spectroscopy measurements analytical PC software is used on a desktop or notebook computer. Typical measurement disciplines include:

- Spectroradiometry (200-1700 nm range, measurement of absolute light intensity and emission color using xy chromaticity),
- Spectrocolorimetry (400-700 nm range, measurement of reflected color values using CIELAB and delta E* for detection of color differences),
- Optical Spectrum Analysis (200-1700nm range, characterization of optical sample transmittance

or device emission shapes including peak, centroid, Full Width Half Max (FWHM)),

- and Spectrochemistry (190-2200 nm range, measuring chemical concentrations and reaction times using absorption or transmittance).

StellarNet's SpectraWiz measurement software provides all the above, and includes additional programs for operation directly from Excel-VBA and LabVIEW, which can be easily modified by the user.

4 Application examples

Spectral reflectance in the NIR can be used to chemically identify many materials in both powder and solid forms. In certain cases this technique can also be used for quantitative chemical analysis. There are numerous industries where this technology is being utilized to automate process applications such as material sorting or quality control monitoring of product packaging lines. Several examples are provided in the following graphs to illustrate application feasibility.

Fig.2: StellarNet EPP2000-NIR-InGaAs spectrometer used to capture sample reflectance spectra shown in the graphs.

4.1 Measurement details

The spectral data shown in the graphs was captured using an EPP2000-NIR-InGaAs miniature fiber optic spectrometer (**Fig.2**) operating in the 0.9-1.7 µm wavelength range. The instrument has a 25 µm slit to provide a 3.1 nm optical resolution. A fiber optic reflectance probe was attached to the spectrometer input using a 600 µm fiber optic cable.

The accessory has an integrated 30 watt halogen lamp with a platform on top to place samples for reflectance measurement. An integrated collimating lens provides a sample viewing distance of 75 mm. To simplify handling, powder samples were placed in glass or plastic containers.

A white reflectance standard was used to create a reference for the measurements. The sample spectrums are saved using absorbance units *AU*, where

$AU(l)$ = Log10((sample-dark)/(reference-dark))
(Equation 1)

The second derivative of each sample spectrum d^2AU/dl^2 is used to improve sample discrimination and then train neural networks.

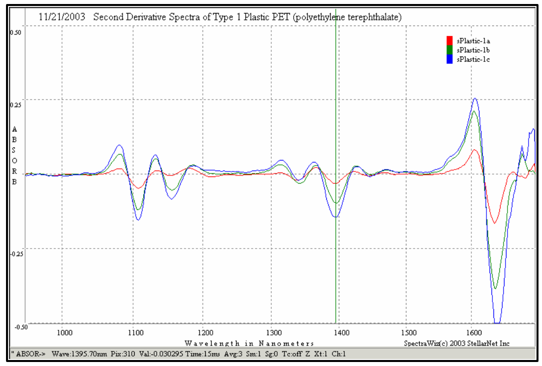

Fig. 3: Plastic type 1 - PET (polyethylene terephthalate)

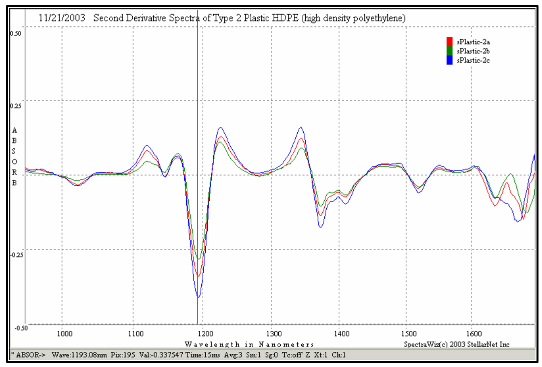

Fig. 4: Plastic type 2 - HDPE (high density polyethylene)

4.2 Identification of polymers

The graphs in **Fig. 3 - 7** show clearly that NIR reflectance can be used to improve sorting of recycled plastic material.

Some operations melt and remold used plastic containers into low-grade building materials. It is too complicated to identify plastic container types by the number that could be found inside a triangle on the container bottom. If high-quality recycled plastic material could be achieved, then it would be possible to make better use of such re-constituted plastic and improve profitability for recycling operations.

Several samples of each plastic type were measured, each having different shapes and pigments (i.e. colors). Each spectra was captured in 45 milli-seconds, averaging 3 spectral scans. As can be seen from the graphs, each of the different plastic types[1] are easily identifiable despite their color differences. Only small variances can be seen within a specific type which is related to the container's shape. Color has no effect in the NIR, except that black plastic objects have significantly lower reflectance.

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For signal processing we use sample absorbance values instead of reflectance values. A zero actually represents no absorbance by the sample, thus 100% reflectance. This is set using a white reflectance standard. Additionally, the absorbance values have a built-in log scale (see equation 1) which improves dynamic range in processing the shape and in spectral identification.

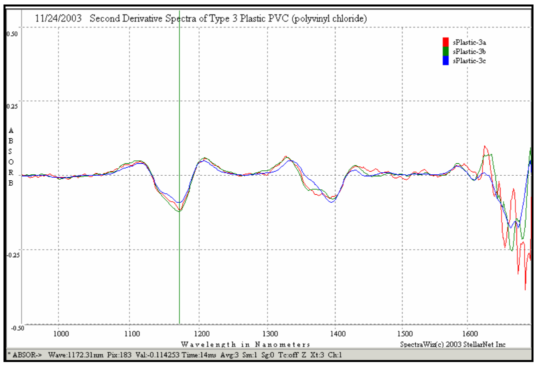

Fig.5: Plastic type 3 - PVC (polyvinyl chloride)

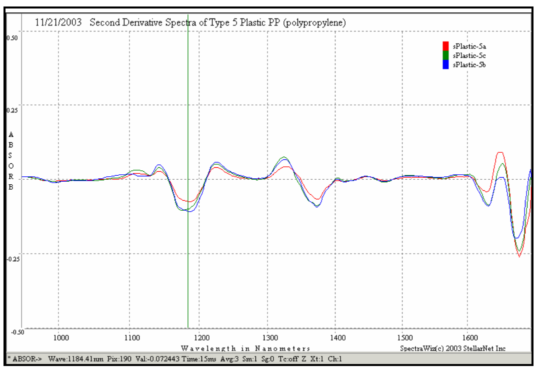

Fig.6: Plastic type 5 - PP (polypropylene)

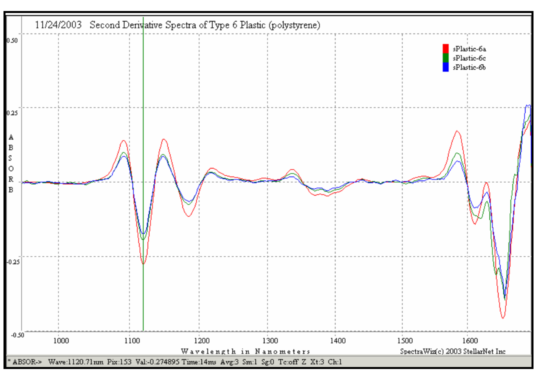

Fig. 7: Plastic type 7 – PS (Polystyral)

The sample absorbance dataset is then converted to second derivative spectral shapes. The final spectral dataset is used for training neural networks to control high speed plastic sorting machinery.

4.3 Beverage additives and pharmaceutical samples

Additional interesting spectral samples captured with an NIR reflectance accessory are shown in **Fig. 8 - 10**. The samples were placed in a glass beaker, then onto the reflectance apparatus. The first graph shows the absorbance spectra for beverage sweeteners. Can you tell which sample tastes the sweetest?

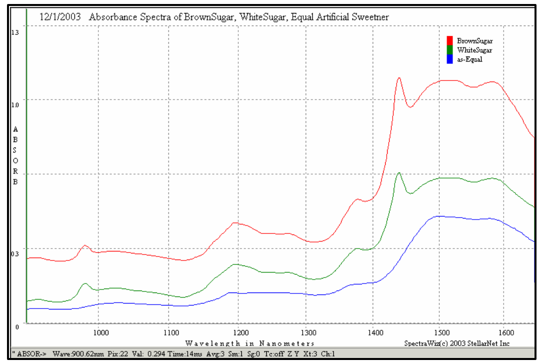

Fig. 8: Absorbance spectra of powdered beverage sweeteners: brown and white sugar plotted along with an artificial sweetener named Equal, which contains dextrose and aspartame.

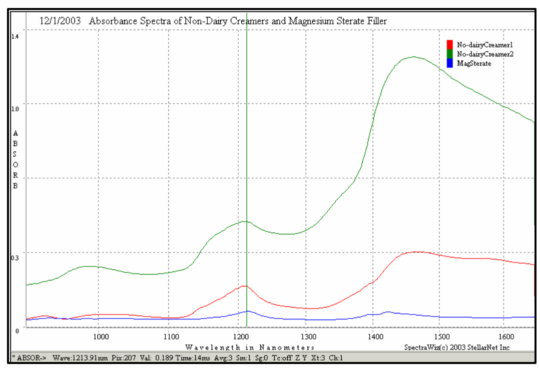

Fig.9: Absorbance spectra of typical coffee creamers, sweet and non-sweet. The taller spectrum is a sweet French vanilla in liquid form. Notice the similarity to the brown sugar shown in Fig. 8. The red spectrum is a powder creamer that is not sweet. The blue graph is Magnesium Sterate, a white powder filler with no sweet flavor at all.

In both the beverage additives and pharmaceutical samples, it is interesting to observe that their chemical makeup is revealed by the reflectance spectra, making them easy to identify. For example this technology could be used to verify pill contents during an automated bottling process, where each sample travels down a small conveyer belt as it passes by the reflectance detection area.

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[1] Plastic type 4 was unavailable for sampling

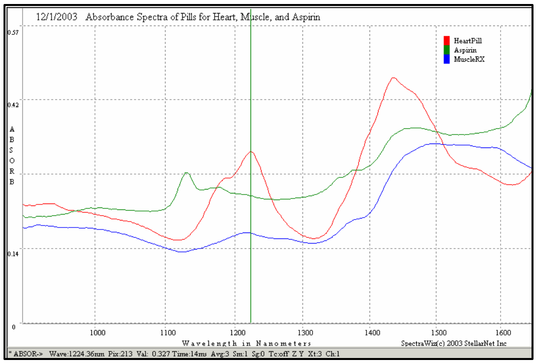

Fig.10: Absorbance spectra of typical pills taken by people for various bodily remedies: heart arrhythmia capsules, a muscle relaxer, and standard aspirin headache tablets.

5 Conclusion

We have shown that spectral reflectance in the NIR works to identify sample chemistry for many different markets. This type of technology can be used as a fast and powerful tool to enhance manufacturing quality and process control. It is easy to use for a variety of products, because there is no sample preparation and the spectral data can be acquired instantaneously. Using miniature fiber optic spectrometers for spectral measurement is the key to developing new low cost, high technology applications.

References:

[1] *Spectroscopic Identification of OrganicCompounds*, CRC Press 1995
[2] *Chemometrics in Analytical Spectroscopy*, Royal Society of Chemistry 1995
[3] Polymer Surfaces, *Diffuse Reflectance Spectroscopy*, John Wiley & Sons Ltd. 1998

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