Industrial Process Monitoring Requires Rugged AOTF Tools

Dr Jolanta Soos

Growth has been rapid in the use of spectroscopic methods to monitor industrial processes, both in production lines and for quality control and quality assessment. The most common applications are following the progress of chemical reactions, fermentation, or mixing processes and checking the purity of final or intermediate products. By far the most widely used spectroscopic tools are infrared (usually Fourier-transform IR) and nuclear magnetic resonance spectroscopy. However, because of the limitations of these methods, much analytical work in the pharmaceutical industry is still done with time-consuming chromatographic methods, such as liquid chromatography.

Recent advances in electro-optic technology and instrumentation have greatly increased the power and utility of near-infrared (NIR) spectroscopy. Specifically, the availability of rugged, all-solid-state smart spectrometers based on acousto-optic tunable-filter (AOTF) technology now makes NIR spectroscopy a faster, more reliable tool for applications as diverse as petro-chemicals, foodstuffs, pharmaceuticals, cosmetics, and perfumes. In addition to replacing other analytical techniques in these areas, it opens new possibilities in terms of high-speed, real-time, closed-loop process control.

NIR spectroscopy

The near-infrared (NIR) spectral region is loosely defined as the band of wavelengths from 0.7 to 2-3 micrometers. Many molecular vibrations have overtone frequencies that give rise to absorption bands in this spectral region. The wavelengths at which these vibrations occur for a particular chemical are a function of its structure and composition. NIR spectra can thus be used to identify molecular species and evaluate their concentrations or mole fractions in complex mixtures such as penicillin fermentation, blended gasoline, and formulated cosmetics.

There are two practical advantages of NIR over conventional infrared spectroscopy. First, most IR samples have to be specially prepared as this films or pastes. Direct observation of solution samples is difficult because infrared wavelengths are strongly attenuated by water, solvents, the analytes themselves, and even the glass walls of the reaction vessel. Second, remote monitoring is difficult because these wavelengths cannot be efficiently transmitted through optical fibers. For these reasons, it is difficult to use IR spectroscopy for real-time monitoring of syntheses and fermentations.

NIR spectroscopy has no such limitations; it can be used directly with aqueous and organic solutions, in both transmission and reflection. Also, it is well suited to fiber-based remote sensing using conventional, low-cost fiber optics.

On the down side, NIR spectra tend to consist of overlapped broad features with little or no true baseline. However, data analysis is simplified with software programs that rely on chemometrics. A typical NIR spectrometer contains, or is interfaced to, a microcomputer on which a chemometrics package is loaded. During a training phase, the instrument is presented with a range of "training" samples of known composition. The software correlates the recorded spectral data points with the known results, generating its own algorithms using partial least squares and principal component regression. During normal operation, the spectrometer then uses these algorithms to derive composition and/or concentrations from real data.

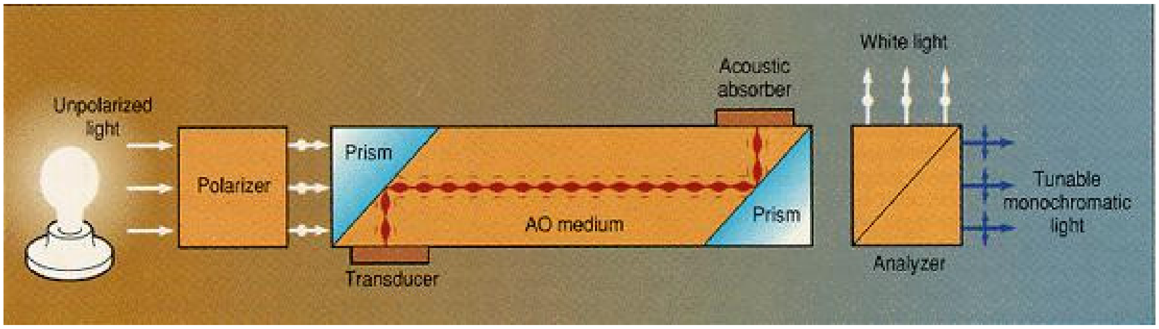

Figure 1. *In a collinear AOTF, light waves and acoustic waves interact to produce photons at the sum and difference energies.*

Instrument Limitations

In terms of hardware, the key to successful NIR spectroscopy is making quantitatively accurate measurements of absorption or reflectance at a number of precisely known wavelengths. For rapid results, the instrument must be tuned from one wavelength to another quickly and reproducibly. For the production environment, the NIR instrument should also be rugged and immune to the effects of vibration, thermal shifts, dust, and general low-level abuse.

Until recently, there has been no instrument or even a base technology for building an instrument that could satisfy these important criteria. Traditional instruments, such as grating-based spectrometers and Fourier-transform devices, have relatively slow data-acquisition rates and issues of absolute wavelength calibration. Also, they are generally too delicate to deploy in direct process-control applications on the plant floor.

Benefits of AOTFs

Recently, several companies, including Brimrose, have been developing acousto-optic tunable filters (AOTF), all-solid-state wavelength-tuning devices. An AOTF is a specially prepared optical crystal, usually quartz or tellurium oxide, with a high-frequency transducer (vibrator) attached to one side. The device transmits only one wavelength of light according to the frequency of an applied RF (radio frequency) source. When RF power is applied to the transducer, it produces acoustic vibrations that flow through the crystal at the applied frequency. As light passes through the crystal, interaction between the light waves and sound waves causes the crystal to act as a narrow-line band pass filter.

The collinear AOTF is the easiest kind of tunable filter to understand (see Fig. 1). In this configuration, the RF transducer is mounted on the crystal so that acoustic waves travel longitudinally through the crystal, that is, in the same direction as the light waves. A nonlinear interaction in the crystal leads to an effect similar to frequency mixing, used to shift the frequency of laser beams. In simple terms, a photon of light (v1) combines with an acoustic phonon to generate a new photon (vac) of different energy: v1 +/- vac. This energy shift is extremely small given that v1 is typically 107 larger than vac.

Because the shifting takes place over a large path through the crystal (typically 1 cm), the incident light and diffracted light must have the same phase velocity (that is, they must be phasematched). Otherwise the diffracted waves generated at different points along the transmission path will be out of phase and destructively interfere. The incident and diffracted light have slightly different frequencies, however, because they will naturally travel at different velocities because of dispersion.

The collinear AOTF takes advantage of birefringence. The geometry of this crystal interaction is set up so that the diffracted light has its polarization rotated 90ƒ with respect to the incident light. For a given RF value (phonon energy), there is only one wavelength of light in which the velocities of the incident and shifted light are equal because of their orthogonal polarizations (the phase-matching condition). The only other thing necessary to make a functioning AOTF is to place appropriate polarizers on either end of the device so that the light traveling through the crystal is plane-polarized.

An all-solid-state commercial NIR spectrometer with a built-in AOTF functions well even in harsh industrial environments (see Fig 2). There are no moving parts, thus calibration cannot be affected by vibrations. In fact the wavelength of the transmitted light depends only on the applied RF frequency; in most instances, this signal is digitally generated and controlled for repeatable precision and accuracy.

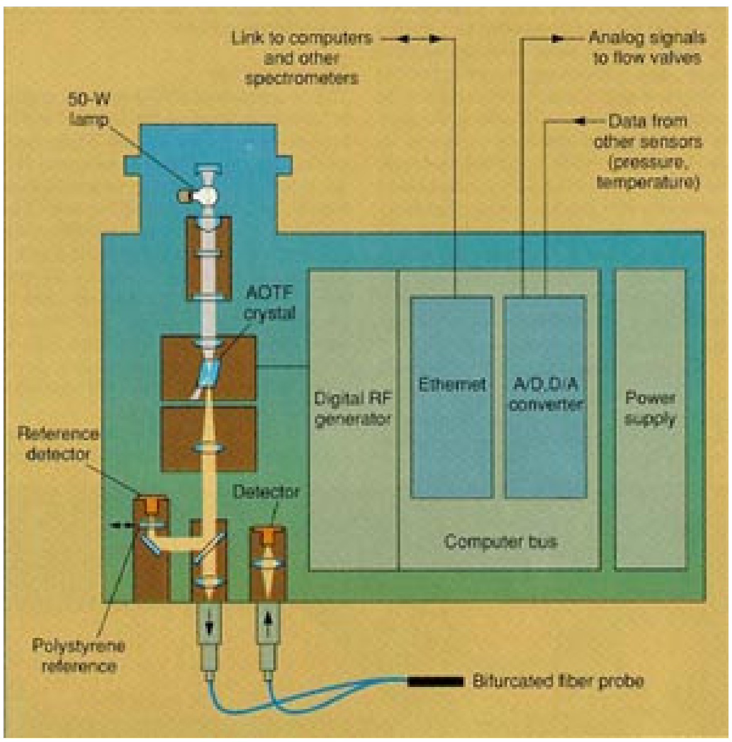

Figure 2. *AOTF-based spectrometer integrates wavelength-tuning crystal in compact, all-solid-state device. Sampling with a bifurcated fiber probe makes the instrument adaptable to process environments.*

High-speed data acquisition is another advantage. The slow data acquisition of traditional NIR instruments has not only been a drawback to laboratory use but has often precluded their use for real-time, closed-loop monitoring and control of industrial processes such as blending, mixing, and fermentation. With the capability of recording 4000 date points/second, even in random access mode, the AOTF makes NIR spectroscopy useful for these tasks.

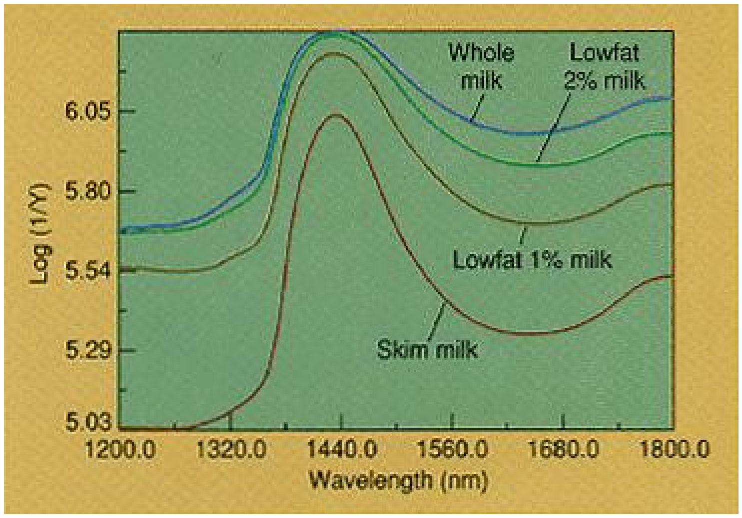

***Figure 3**. NIR reflectance spectra of various milks, recorded with a Brimrose Free Space spectrometer, indicate fat levels during processing.*

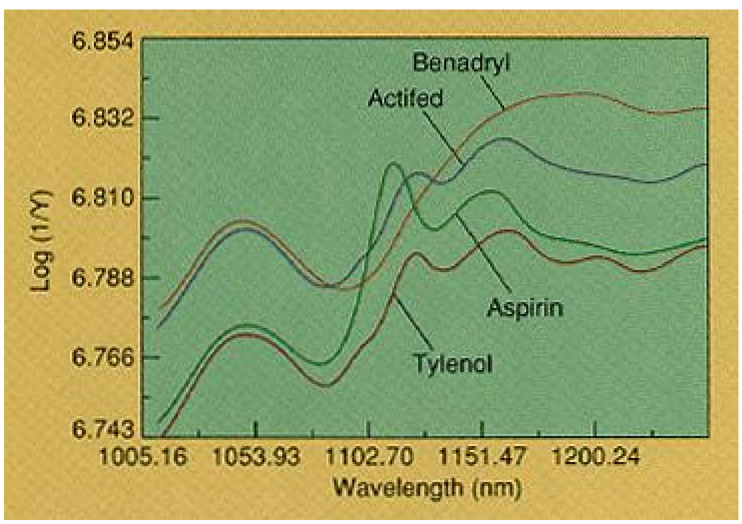

***Figure 4**. NIR reflectance spectra of common nonprescription tablets sealed in a glass bottle, recorded with a Brimrose Free Space spectrometer, reveals differences in tablet composition.*

Real-Time Process Control

NIR instruments with an on-board computer can perform tasks beyond merely acquiring and analyzing spectra. For example, instruments are now available with both an A/D board and Ethernet hardware and software. Resident software allows the user to set up closed-loop operation where the instrument will directly control flow valves and other equipment either through the A/D board or Ethernet links in response to the analyzed data. Alternatively, the results of NIR data can be continuously fed to a central plant computer.

In an ideal automated plant setup, networked spectrometers would be on-line in both the plant and the laboratory. As new processes and protocols are developed, the requisite software training would take place in the lab and analysis algorithms automatically downloaded to the on-line instruments.

Petrochemicals

Accurate measurement of octane number is important to petroleum companies. Too low an octane number means the blend cannot be sold, and too high an octane number effectively reduces the supplier's profit margin. Octane measurement normally involves burning a pint of gasoline in a specially designed test engine. The gas is rated according to its knock intensity, relative to reference fuel blends. The process takes 20 minutes.

NIR spectroscopy has been shown capable of measuring octane within 0.1 octane number in real time (<0.1 second). Consequently, a number of refineries are starting to install fiber-based NIR monitoring instruments to control the actual blending process as it is occurring.

Foodstuffs and beverages

The various food and beverage industries are increasingly turning to NIR spectroscopy to increase profitability by reducing scrap product. NIR is ideal for tasks such as monitoring the amount of alcohol in a fermentation or the amount of moisture in "long-shelf-life" baked goods.

An interesting application involves determining the amount of fat in milk products to make sure a batch meets its specified value (skim milk, 1%, 2%, and so forth; see Fig. 3). Again, speed is the major benefit over alternative technologies such as chromatography. NIR enables product composition to be monitored in real time on the production line, instead of using captured samples in a quality-control laboratory.

Pharmaceuticals

Probably no industry requires such tight process- and quality-control monitoring as pharmaceuticals. This requirement, coupled with strict FDA regulations, leads to extremely cautious adoption of new methods and standards. Even here, NIR spectroscopy is starting to have an impact, spurred in part by FDA-approved chemometrics software such as Pirouette (from Infometrix, Seattle, WA).

NIR Spectroscopy has already been demonstrated capable of monitoring streams of prepackaged tablets. NIR spectroscopy can be used to confirm (or deny) the nature of a particular drug (in liquid or solid form), even when the drug is enclosed in a tamper-proof, sealed glass bottle. The reflectance spectrum of bottled aspirin tablets shown in Fig. 4 was recorded merely by pointing the fiber probe from an NIR instrument at the bottle.

AOTF technology, together with inexpensive microprocessors and powerful software, have rapidly brought NIR spectroscopy out of the lab and into the production line of industries as diverse as oil refineries and commercial bakeries. As the benefits of this technology become more widely known, and user familiarity and confidence with this approach increase, new applications for NIR spectroscopy are sure to be developed. Furthermore, this marriage of electro-optics and microprocessors is bound to result in other instruments and techniques that will benefit production technology.