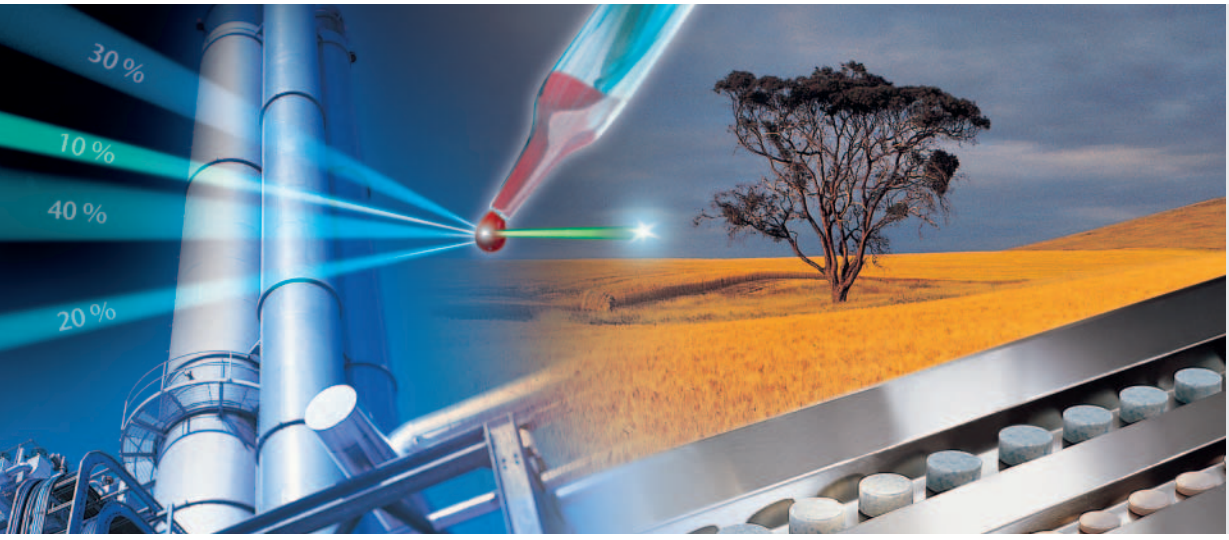
Polytec

Process Control Using NIR Spectroscopy



Field of Application

- A Aerospace
- B Audio & Acoustics
- C Automotive
- D Data Storage
- G General Vibrometry
- M MEMS & Microstructures
- **P Production Testing**
- S Scientific & Medical
- T Structural Testing
- U Ultrasonics

Polytec Spectrometers Measure Chemical and Physical Parameters in Laboratory and Process Applications

Near Infrared (NIR) spectroscopy is used for fast, reliable, and non-destructive measurements that simultaneously control manufacturing processes and product quality, assuring that final product specifications and quality are met. The functionality of the product can be determined in both a quantitative and qualitative manner, and measurements can be carried out on solid or liquid samples. Combining a high sampling rate with a flexible optical delivery method, Polytec-designed spectrometers utilize fiber-optic probes to fill a wide range of applications in process control.

NIR Spectroscopy – Simple and Flexible

Optical spectroscopy covers wavelengths from 200 nm to 25 µm (see Figure 1) and is divided into three important spectral ranges:

- Ultraviolet/visible: 200 – 760 nm
- Near Infrared : 760 – 2500 nm
- Mid-Infrared : 2500 nm – 25 µm

Photons in the Ultraviolet/Visible spectral range have enough energy to excite or ionize materials by raising the energy level of bound electrons. NIR radiation has less energy/photon but does excite molecular vibrations. Vibrational spectroscopy in the NIR range is used for processmonitoring and quality control. The NIR wavelengths enable a very flexible measurement setup and a high measuring rate. Another advantage of using the NIR spectral range is the low coefficient of absorbance, allowing relatively deep penetration of the radiation in the sample. Therefore, samples can be measured without substantial preparation and information on both surface and volume parameters can be acquired. Finally, these measurements are non-destructive and samples are not altered and can be reused. The advantages of NIR spectroscopy make integration into automated production processes much easier, and the availability of fiber-coupled measuring heads allows for flexible measurement set-ups.

Benefits of NIR Spectroscopy

- Non-contact, non-destructive method
- Fast and automated measurement
- Easy and flexible process integration
- Precise and reproducible results
- No sample preparation

Theory of NIR Spectroscopy

The interaction of the electromagnetic radiation with the sample can be described by the following effects:

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Analytics
Application Note
Spectroscopy
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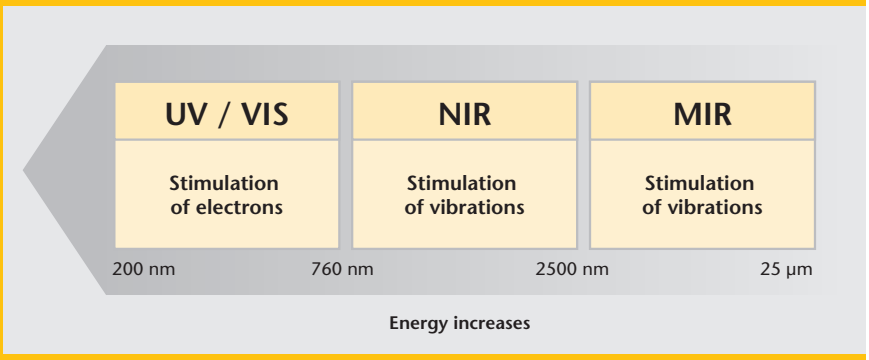

Figure 1: The wavelength range and common spectral divisions for optical spectroscopy.

- Absorbance/Reflection (Figure 2a)
  The absorbance spectra are used for the qualitative and quantitative determination of composition.
- Surface effects (Figure 2b)
  The evaluation of surface effects determine physical characteristics such as roughness and grain size.
- Interface effects (Figure 2c)
  Interference patterns in returned spectra can be used to determine thickness of thin layers.

Measurements can be done in transmission or reflection, and the resulting spectral response provide information about the sample. Statistical methods are applied to the observed spectra to extract the critical information often buried in the overlay of the effects just mentioned.

Spectra Evaluation Methods

Chemometrics is the application of statistical methods (e.g., principal components analysis or partial least squares) to extract information from chemical or spectroscopic data. There are qualitative and quantitative methods for conducting multi-component analysis. Quantification, for example linking sensor signal with substance concentration, is possible even without specific interactions. To calibrate the system, data sets with known concentrations of the substance in question are measured. The evaluation of these data sets in combination with appropriate reference analyses form the basis for the development of a chemometric model for the monitoring and control of the process.

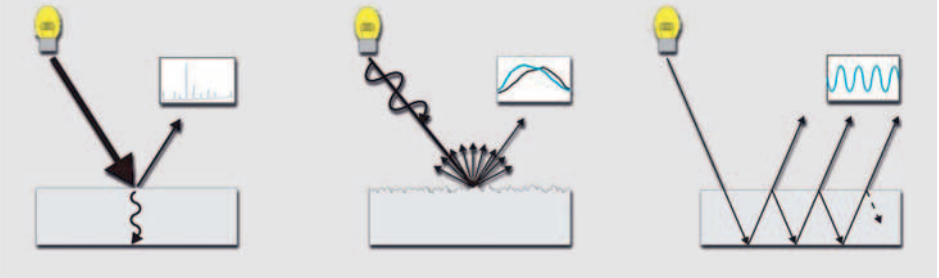

Figure 2: a) Absorption/reflection can determine composition; b) Surface effects can determine surface roughness and grain size; c) Interface effects can determine layer thickness.

Fields of Application

The wavelength of light employed in NIR spectroscopy excites vibrations of covalent molecular bonds. Therefore, it is suitable for the determination of water content in food and agrochemical products (renewable raw materials, feedstuff, corn, milk). Organic and pharmaceutical products can be examined with respect to their protein (N-H bonds) or fat content (C-H bonds).

In addition, various molecular structures and groups can be detected in polymers. An excellent example is given by the carboxylic groups (COOH). For these reasons, the method is commonly used in the chemical, pharmaceutical, and food industry for quality assurance and process control.

Polytec PSS Spectrometers and Modules

The PSS Polychromator (Figure 4), developed by Polytec, is the heart of PSS Spectrometer Systems, as well as OEM systems designed to customer specifications. Both state-of-the-art diode array illumination and the absence of moving parts make for a very compact and rugged design.

Polytec's PSS Spectrometers (Figure 5) are available either in a tabletop housing or in a 19" or 1/2 19" rack-mount unit. They can be connected using fiber-optics to various measurement set-ups, for example immersion probes or flow cells. Furthermore, user-friendly software supports many NIR technology applications for industrial process control. The robust construction of the PSS systems works for production line applications, while delivering reliable, real-time data. Optical multiplexers offer the option to control several measurement points simultaneously from one spectrometer.

Applications

NIR spectroscopy has numerous applications in laboratory analysis and process control. Some typical examples are described below.

Process Control of Biogas Plants

Without an appropriate measurement technique, biogas plants are often run

below capacity, in order to avoid instabilities in, and in extreme cases, failure of the fermentation process. The result is reduced energy output and possible operating losses. To achieve efficiency, reliable and precise measurement techniques are required, and automated process monitoring is required. With NIR probes, various process parameters can be quantified directly in the reaction vessel, and, thus, be used for process control. A lack or excess of certain components (e.g., acetic acid, as shown in Figure 6) has a strong impact on the fermentation process. But by monitoring and controlling concentration, optimal production efficiency can be achieved.

Quality Monitoring in the Food Industry

Water, fat and protein content determines cost and quality in food production. These parameters can be determined simply and quickly using Polytec's NIR technology, allowing the corresponding production and processing steps to be controlled and optimized. As an example, the monitoring of the ethanol content in different beer brands can be determined by NIR spectroscopy.

NIR for Identifying Plastics

Polymers display characteristic spectra (Figure 8). Hence, identifying PE, PET, PP, PS, and so forth, is another area of application for NIR technology. High measurement speed, as well as confidence in identification, is the basis for industrial application in sorting and recycling facilities. During the production of polymers, quick and easy quality assurance regarding water content (for example), is made possibly by NIR spectroscopy.

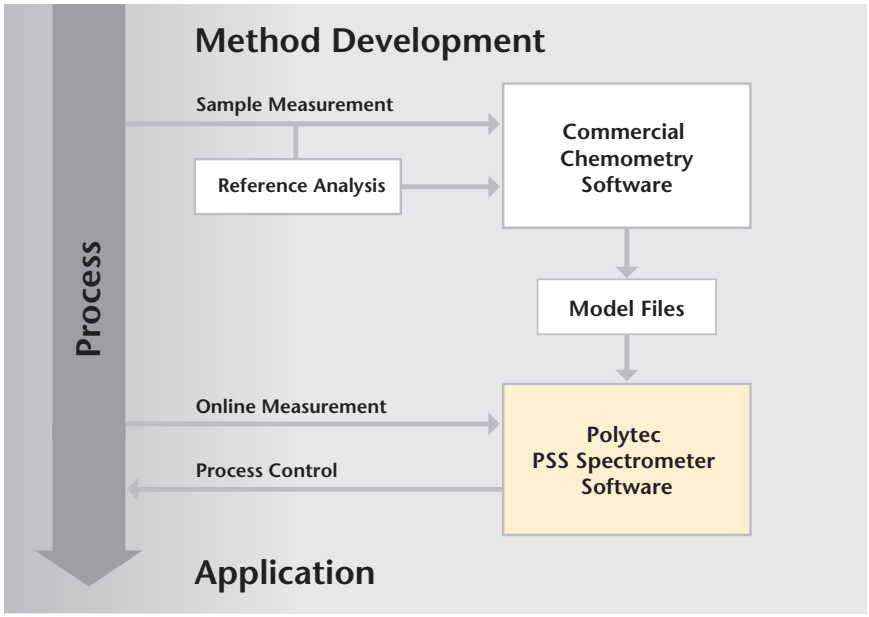

Figure 3: Process integration of NIR technology

Determination of Layer Thickness

Using NIR spectroscopy, layers ranging from 100 nm thick up to 100 µm can be measured without contactby the use of thin film interference patterns (Figure 9). This holds true even for layers that are not transparent in the visible spectral range, e.g. lacquers.

Quality Control of Tablets, Capsules and Pharmaceutical Substances

Using fiber-optic probes, pharmaceuticals can be analyzed quickly and easily – even in the production facility. Due to the extremely high measurement speed of PSS systems, each and every capsule or tablet produced can have its active ingredient content verified, thus giving 100 % quality control.

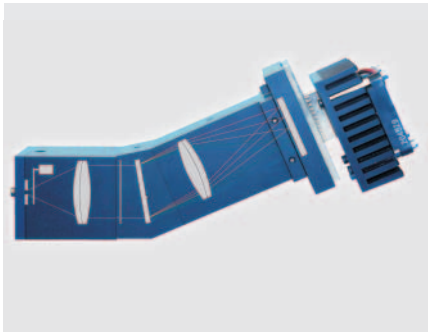

Figure 4: PSS Polychromator.

Figure 5: Polytec PSS Spectrometer with fiber optics.

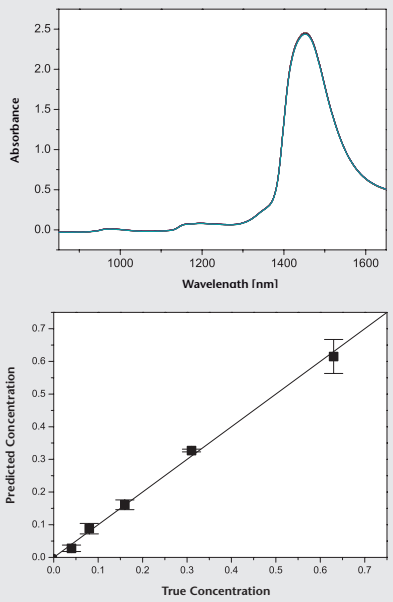

Figure 6: Monitoring of process parameters in a biogas plant.

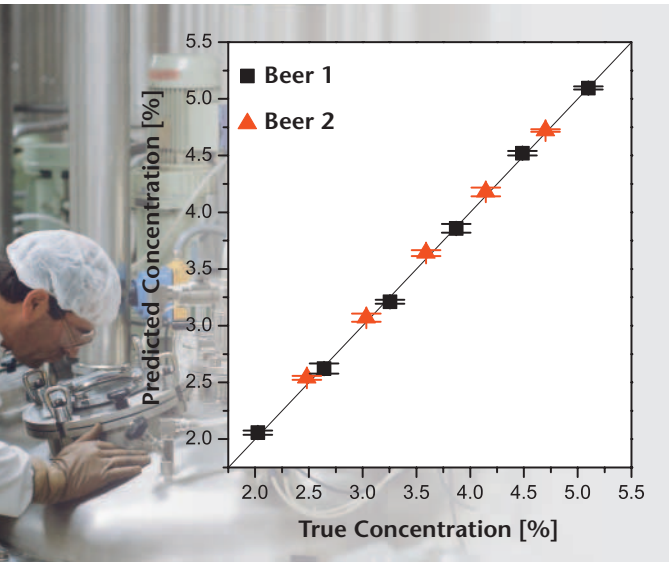

Figure 7: Ethanol monitoring in different beer brands.

NIR – Process and Multi-Component Analysis in the Chemical Industry

Using special probes connected directly to pipelines and reaction vessels, it is easy to monitor water, alcohol and total organic carbon.

Quantitative determination of all components in a reaction is decisive for monitoring and controlling many processes. With the aid of suitable probes which are directly connected to extruders, reactors or pipelines, it is possible to determine important parameters (e.g. viscosity, density, composition) from the NIR spectra in real time.

Production Control for Paper

NIR spectroscopy is highly suitable for in-line moisture control of paper rolls. In addition, a wide range of customer-specified applications or coatings can be monitored and controlled.

Conclusions

NIR spectroscopy can evaluate many different parameters, enabling a multitude of process control applications. The combination with statistical evaluation methods opens up multi-component analysis, as well as qualitative and quantitative assessments of material compositions. Due to different principles of interaction between light and matter, not only can chemical composition be measured, but layer thickness and surface properties as well.

NIR spectroscopy is an important tool that can be used to simultaneously optimize process control and product quality, delivering an improved product to the market.

More Information

For detailed information, please contact your local Polytec sales/application engineer. Visit our website at **www.polytec.com/st** to download product data sheets, or send an e-mail to **ST@polytec.de** with a specific request.

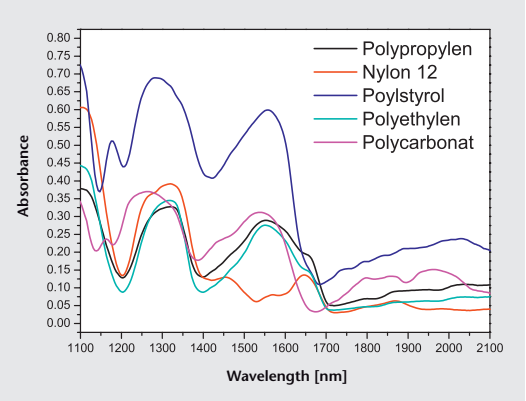

Figure 8: Identification of different polymers by NIR spectra.

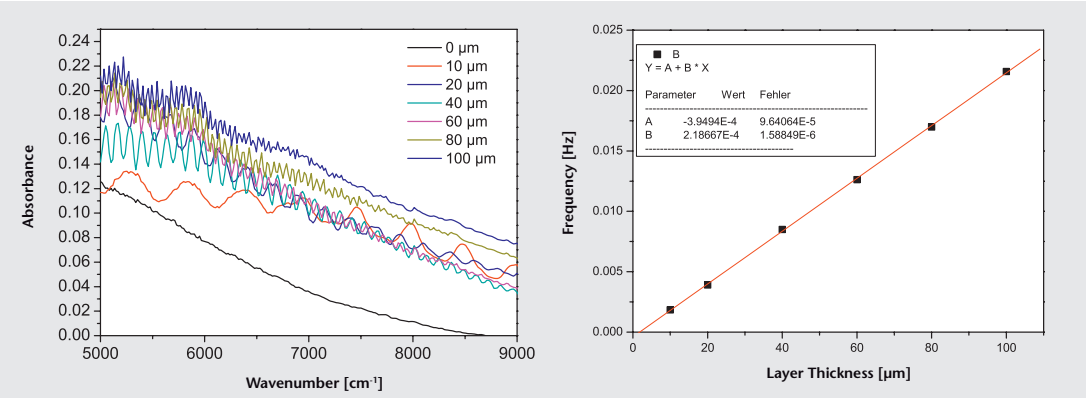

Figure 9: Interference pattern (left) and layer thickness measurement (right).

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