

Double stitched provides more strength

Production control of fibres using FT-NIR



Figure 1: IRPrestige-21 with NIR integration sphere and sample holder

When it comes to modern fibres, this saying seems truly outdated, even if the requirements with respect to comfortable wear and durability of yarns and fibres are continually increasing. But this goes hand in hand with the continuous developments of new fibre coatings which enable fast and reliable production processes in mechanical looms. By coating a fibre with polymers, its surface become smoother and its resistance to tearing is increased.

In order to guarantee a constant quality during the fibre production process, a fast and precise analytical method is needed and

this is offered by the FT-NIR technique. Shimadzu's IRPrestige-21, a powerful FTIR spectrophotometer, covers the entire infrared wavelength range (MIR [standard], NIR and FIR [optional]).

Using the NIR integration sphere (Pike Technologies, see Figure 1) with integrated Indium-Gallium-Arsenic detector, which is available as an accessory, the time- and materials-consuming routine analytical method is simplified resulting in increased productivity. A complex sample preparation process is generally not required.

In NIR spectroscopy, parameters such as particle size, particle size distribution, phase (solid or liquid), compression, temperature and other physical and chemical characteristics have a significant influence on the sample spectrum and therefore must be taken into account during evaluation of the spectra and sample preparation.

In all cases, the IRPrestige-21 with the NIR integration sphere

measures the back-reflected light. Transparent materials or films only generate very weak absorption signals that can be strongly enhanced via a reflecting mirror oriented towards the sample surface as the NIR light is directed back into the integration sphere. This double transmission (via the sample surface, reflected by the mirror back to the sample surface and subsequently into the integration sphere) is an elegant way of analysing the layer thickness of transparent samples. This state-of-the-art technique is also referred to as transreflectance.

The NIR spectra obtained generally show broad bands that can seldom be individually assigned. The sample is therefore better characterised by the totality of the spectrum. Using calibration models, the required selectivity can be attained. Figure 2 shows a spectrum of a coated fibre sample obtained using the IRPrestige-21 with integration sphere. The fibre sample was positioned in the desired orientation on the sample window of the integration sphere and placed inside the

PLS Calibration Report		
Algorithm	PLS I	
Number of components	2	
Number of references	16	
Range [cm ⁻¹]	4,000.00 - 8,700.00	
Pretreatment	PLS Calibration	
	Base line: Auto Zero	
Centered data	Yes	
Components	A	B
Number of factors	3	3
Correlation coefficient	0.99427	0.99492
MSEP*	0.01071	0.00949
SEP*	0.10347	0.09743

Table 1: Summary from the calibration report of the IRsolution software 1.10

*MSEP: Mean Square of Prediction

**SEP: Standard Error of Prediction

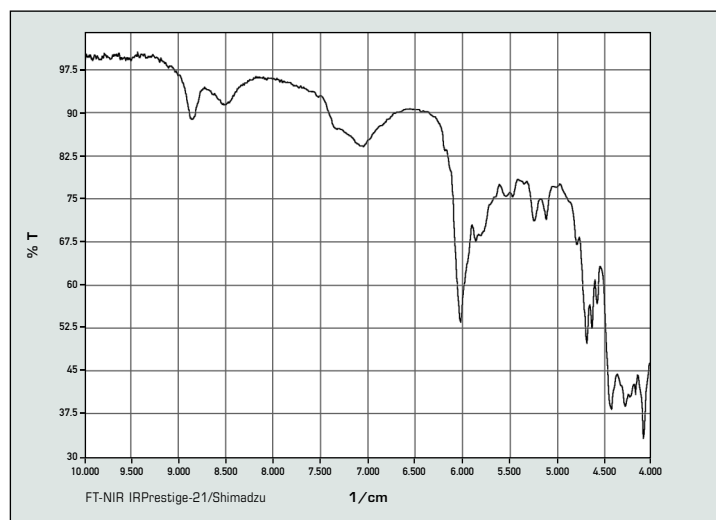


Figure 2: NIR spectrum of a coated fibre sample, measured in diffuse reflection mode. 40 spectra were averaged, 8.0 cm^{-1} resolution

sample holder (the fibre is positioned on a card).

The overlapping NIR absorption bands in the spectrum require a multivariate mathematical procedure such as partial least squares (PLS) in order to take full advantage of the benefits of the high signal to noise ratios, irrespective of the overlapping bands. This procedure does not require the selection of suitable wavelengths as this concerns a full-spectrum analysis. To increase the robustness of the calibration it may only be necessary to exclude several non-variable ranges in the spectrum.

The PLS method is especially suitable in cases where a large number of standard spectra (reference spectra) are available, but very little information on the NIR absorption behaviour of the compounds.

In order to create a suitable calibration model, standards are required that reflect the expected variance of the analytical samples.

Chemometrics of a fibre sample series

The NIR spectra obtained were calibrated on two production parameters A and B. Parameter A describes the production rate and

B the percentage uptake of the polymer. Both parameters provide an indirect indication of the coating process of the fibres with the polymers or polymer blends. For calibration, a wavenumber range between 8700 and 4000 cm^{-1} was selected, as this range correlates strongly with the two predetermined parameters A and B.

To improve the PLS calibration, all spectra were centered and baseline corrected using the IRsolution software. Figure 4 shows the option Calc vs. Input that provides a fast overview on the quality of the calibration. The IRsolution software provides the reference parameters versus the spectroscopically obtained parameters. Outliers are indicated with a cross.

In addition to the various graphical representations, as shown in Figure 4, the software also displays analysis results in tabular form. A summary is shown in Table 1. For the 16 fibre samples analysed, a correlation coefficient of 0.99427 for A and 0.99492 for B was obtained, which reflects the high precision of NIR measurement.

Advantages

In comparison with routine analytical methods, FT-NIR spectroscopy delivers accurate and

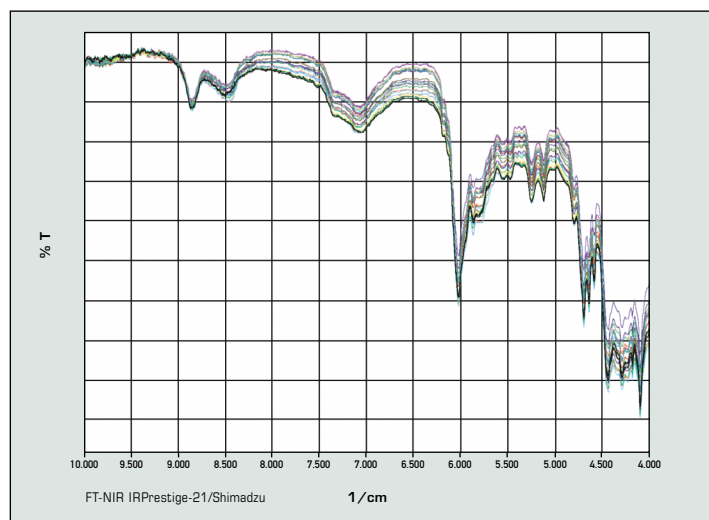


Figure 3: NIR spectra of 16 differently treated fibre samples, each resulting from averaging 40 spectra, 8.0 cm^{-1} resolution

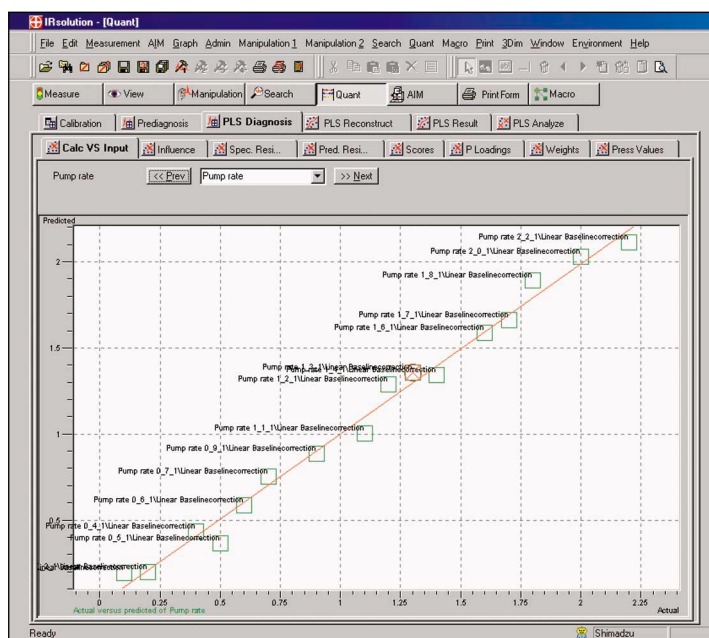


Figure 4: Screen shot of the option Calc vs. Input in the IRsolution software, the red cross indicates outliers. A data name is assigned to each data point

reproducible results with less effort. Using the IRPrestige-21 with the NIR integration sphere and integrated InGaAs detector, meaningful NIR spectra can be obtained easily and quickly without the need for sample pretreatment.

In addition to the quantitative fibre measurements presented here, it is also possible to measure many types of compounds directly in their glass containers, provided that the container walls are not too thick. For quantitative

evaluations, however, a very thin type of glass with a reproducible layer thickness can be used.

Non-homogeneous samples (for instance foods such as flour) can be spectroscopically analysed and quantitatively evaluated with very high precision using the optionally available rotating sample- and container holder.

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