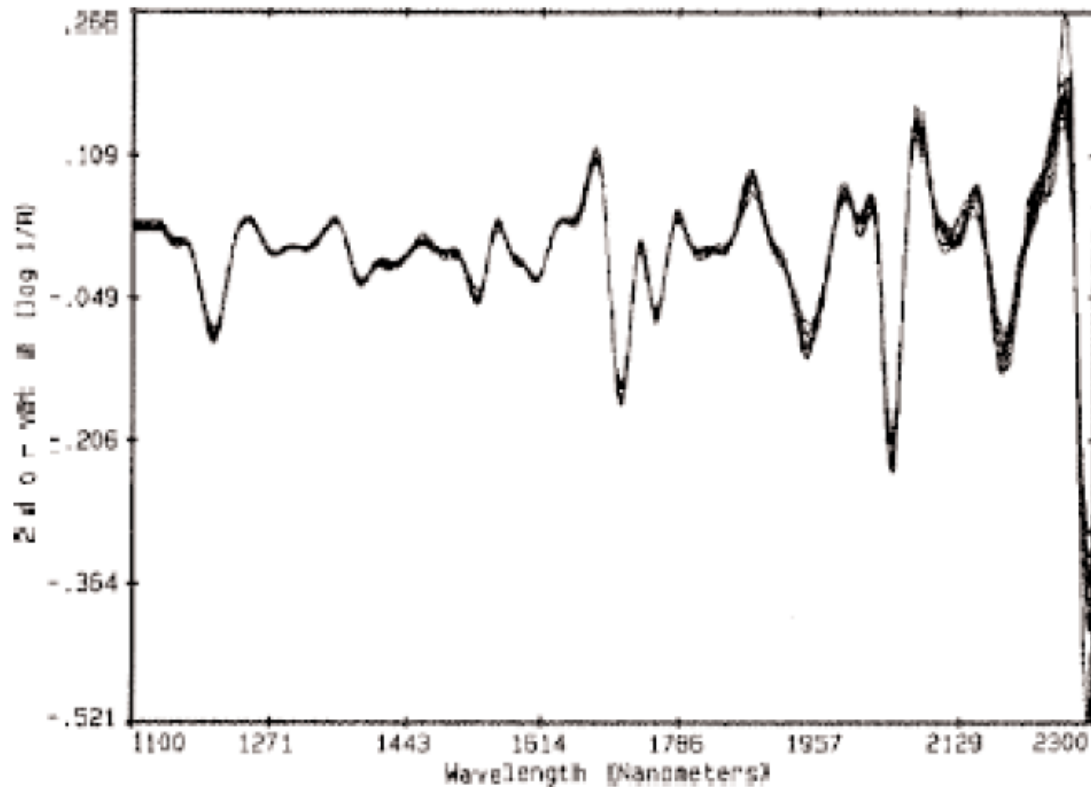


Determination of finish on nylon fibers by near-infrared spectroscopy



This Application Note demonstrates the feasibility of NIR spectroscopy to quantify the amount of (oil) finish on nylon fiber surfaces. Scattering differences that derive from the finish can be eliminated by using second-derivative spectra; partial-least squares (PLS) regression analysis was used to develop the calibration equation. NIRS offers real-time results – an analysis is done in less than 30 s – and requires neither sample preparation nor reagents.

Method description

Introduction

Finish, which is applied to textile fibers, serves two main purposes in the manufacture of textiles. The first purpose is to maintain cohesion of the fiber and prevent fiber fraying. A second purpose is to provide antistatic protection, thus reducing the likelihood of frayed fibers becoming entangled in machinery. Finally, the finish serves as a processing aid to lubricate the fibers as they are brought together.

Hence, in the production of nylon fibers, real-time quantitation of the finish levels is critical to fiber quality and processing efficiency. Therefore, it is vital to develop a method which can accurately and rapidly determine the amount of finish applied to the fiber.

In this study, near-infrared (NIR) spectroscopy is used to quantify the amount of oil finish applied to nylon fibers. Partial least-squares (PLS) regression analysis was used to develop the calibration equation.

Experimental

All NIR spectra were obtained on a Foss NIRSystems Model 6500 spectrophotometer, equipped with a Direct Contact Analyzer (DCA) module. Since this instrument is not available anymore, the NIRS XDS RapidContent Analyzer is recommended. An open platform design for easy and versatile sample presentation facilitates the analysis of virtually any solid material. This sampling method is advantageous since fiber samples are difficult to prepare for conventional laboratory analysis.

The concentration of the oil finish on the nylon fibers ranged from 0.18% through 1.73% (as determined by solvent extraction). Samples of the nylon fibers were packed into a standard sample cup with no sample preparation or attention paid to sample orientation. The sample cup was then placed onto the DCA sampling stage and a spectrum of the nylon fibers was obtained. Each sample was analyzed three times as the sample cup was rotated between each scan.

Results and discussion

The log (1/R) spectra for the nylon fiber samples are shown in Figure 1. Although significant variations appear in these spectra, most of this variation is associated with differences in light-scattering from the material, rather than the changes in the concentration of the oil finish.

One method used to compensate for these scattering differences involves calculation of the second derivative of the log (1/R) spectral data. The second derivative spectra are shown in Figure 2. Much of the baseline variation seen in the absorbance spectra has now been minimized; therefore, the changes which appear in the second derivative spectra are attributed to actual chemical differences in the samples.

Figure 1
Oil Finish on Nylon

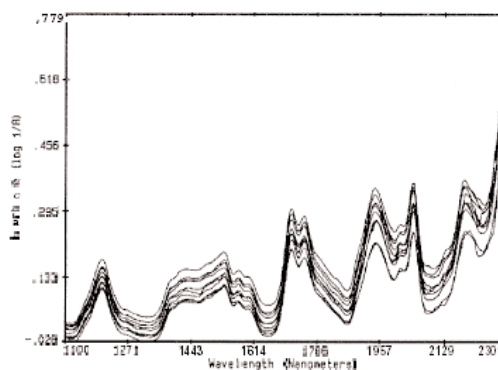
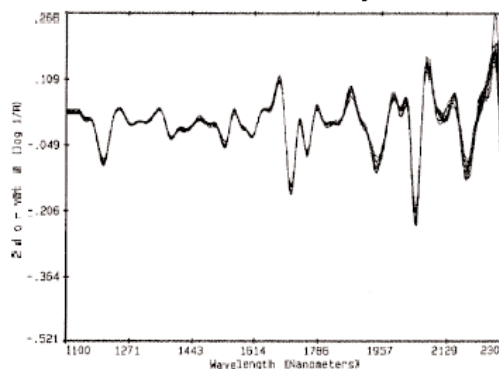


Figure 2
Second-derivative Spectra

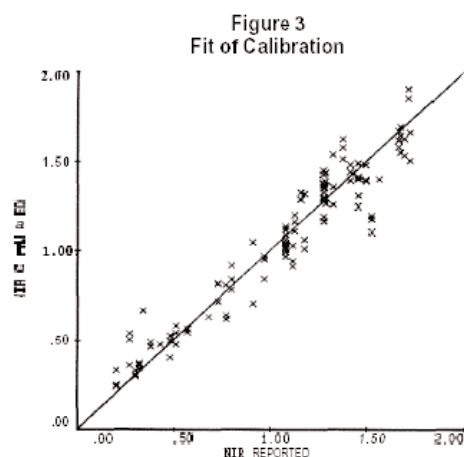


Due to the low level of finish on the fiber and the complexity of this system, a more rigorous calibration technique is required. Partial least-squares (PLS) regression is one chemometric technique that can use several spectral ranges for the development of the calibration equation. Unlike multiple linear regression, where one or two wavelengths are used to model a chemical constituent, PLS regression uses a series of wavelengths to describe the constituent.

Seven factors were required to accurately describe the finish on nylon fiber system. A factor is a mathematical expression describing the variation in a system. A correlation of 0.96 and a standard error of calibration (SEC) of 0.13% were obtained.

A scatterplot of NIR calculated results versus laboratory reported results is shown in Figure 3. Since NIR spectroscopy is not a primary method of analysis, the standard error associated with the NIR method can only approach the standard error of the primary method. The standard error of the NIR calibration closely approaches the accuracy (+ 0.1%) of the primary analytical method.

Method description



The PLS equation was tested using five validation samples not included in calibration development. The results from the analysis of these samples are shown in Table 1. The standard error of prediction (SEP) for this validation set was 0.07%. Since the SEP is of the same relative magnitude of the SEC, this indicates that the PLS calibration is a reasonable and acceptable one.

Table 1: Results from oil finish external validation set

Sample	Lab value	NIR value
A	1.06	1.00
B	1.32	1.34
C	1.26	1.22
D	1.08	1.07
E	0.84	0.75

Standard error of prediction = 0.07%

Conclusions

The results obtained in this study demonstrate the feasibility of using NIR spectroscopy to quantify oil finishes on nylon fiber surfaces. The standard error of the NIR results were comparable to the primary method of analysis (0.13% vs. 0.10%).

There are two major advantages of using NIR methodology for this application. First, NIR provides a more rapid method of analysis (40 s with reference and sample scanning) compared to the primary method of solvent extraction (typically 2 h). Therefore, more sampling can occur which provides greater process control information.

The second advantage is the fact that no sample preparation is needed. Consequently, there is no more need for solvents and solvent disposal, thereby decreasing the overall cost of producing and maintaining quality fibers.