Metrohm NIRSystems

Application Bulletin 413_1_EN

Analysis of textile using near-infrared spectroscopy

Branch

Textile

Keywords

Near-infrared spectroscopy, textile, nylon fibers, wool, finish oil, total solids, polypropylene treads.

Summary

This Application Bulletin details numerous NIR applications from the textile industry using NIRSystems. Feasibility studies are also addressed. Each application describes briefly the measuring systems used in the studies as well as the recommended systems and the test results.

Introduction

NIR spectroscopy has been long used in the textile industry to differentiate fiber types for carpet recycling. Blend analysis of different polymer fibers can be analyzed with NIR spectroscopy as well. Real-time analysis of the application of polyvinyl alcohol (PVA or PVOH) sizing to warp yarn has been done with NIR online process analyzers. Common fiber identified with NIR include: cotton/linen, merchandized cotton, acrylic, modified acrylic, acetate, triacetate, Nomex®, Kevlar® (K-29, K49, and K129), nylon-6, nylon-6,6, silk, polyester, cationic and disperse dyeable polyester, polypropylene, PVA and PVC.

Contents

No. 1: Monitoring oil finish on nylon fibers

Summary

This application shows a feasibility study for monitoring oil finish on nylon fibers. Seventeen samples were provided for the calibration procedure. Two calibrations were performed, one for the top finish (concentration range 0.2–1.7%), and the other for the bottom (concentration range 0.3–1.73%).

System

Model 5000, remote reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer DirectLight/NonContact	2.928.0310

Sampling

The spectral region covered ranged from 1100–2500 nm. The samples were analyzed by laying the fiber samples flat on a large Teflon square, placing the remote reflectance head over the sample, and scanning of the sample. A separate calibration was developed for the top finish and the bottom finish using PLS regression. For the bottom finish, seven PLS factors yielded a SEC of 0.1%. For the top finish, a calibration model with five factors yielded a SEC of 0.2%.

Results

The results indicate that NIR can be used for the analysis of oil finish on nylon fibers. In order to proceed with the actual application, a sampling method must be designed to ensure that enough nylon fiber material is presented to the instrumentation. Furthermore, a calibration set must be developed which minimizes the lag time between the primary analysis and the NIR analysis.

No. 2: Qualitative monitoring of heatset temperature of nylon 6-6, 6

Summary

This application demonstrates that NIRS is used to differentiate between four samples, two which were heatset and two which were not heatset.

System

Model 5000, remote reflectance module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region using a remote reflectance probe. Each sample was placed on a teflon pad, and the remote reflectance head was placed on the sample. The teflon pad was used to insure total reflectance.

Spectral differences were noticed between the heatset and non-heatset samples in the 2020–2130 nm regions. The intensities of these bands are seen to increase with increasing heatset. These spectral regions have previously been identified and successfully used to develop NIR heatset calibrations.

Results

The results indicate that NIR can be used to distinguish between heatset and non-heatset nylon 6–6.6 samples.

No. 3: Quantitative determination of bond and finish on nylon thread

Summary

This feasibility study presents the use of NIRS to monitor bond and finish on nylon thread samples. For bond, the concentration ranged from 0–11.6%. For finish, the range was 0–8.3%.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. The thread samples were cut into approximately one inch lengths and placed into a standard sample cup. The samples were very inhomogeneous. Therefore, the samples were scanned six times, with three repacks and a rotation of the sample in between. For bond, the most sensitive region is found near 1530 nm. A calibration was developed in at 1516 nm (SEC of 0.6%). For the silicone based finish, the most sensitive region appears near 1880 nm. A calibration was developed (SEC of 1%).

Results

The results indicate that NIR can be used to detect the variation in both bond and finish on thread. In order to reduce the sampling error, the samples were reloaded several times, which averaged out sample inhomogeneity inherent in this type of sample.

No. 4: Distinguishing between nylon, polyester, and polypropylene threads

Summary

NIRS is used in this application to qualitatively distinguish between nylon, polyester, and polypropylene threads.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region using a spinning sample module.

Spectral differences are easily viewed throughout the entire NIR spectrum. IQ2 was utilized in the 1150–2450 nm region for identification purposes. All samples were correctly identified. Some blended samples were also analyzed to determine their identity. Each of these samples was correctly identified, although a lower correlation, which was expected, was obtained.

Results

The results indicate that NIR can be used to distinguish between nylon, polyester, and polypropylene. Blend samples could also be distinguished. A better method to identify the blend samples would be to include varying blend ratio samples in the library as separate products.

No. 5: Measuring heatset temperature in nylon carpet

Summary

This NIR application shows a feasibility study to monitor heatset temperature in nylon carpet five samples of known heatset temperature, ranging from 185 °C to 205 °C, were provided. Eight unknowns were also provided.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region. A spinning sample module was used for analysis. Each sample was analyzed three times, repacking the sample between scans. A calibration was developed for heatset temperature at 2008 nm (SEC of 3 °C).

Results

The results indicate that NIR can be used to monitor heatset temperature in nylon yarns.

No. 6: Monitoring fiber blends for blend ratios, moisture and finish

Summary

NIRS is used to monitor fiber blends for blends ratios, moisture and finish. The samples provided represented either wool/rayon or nylon/acrylic blends of different types of wool.

System

Model 5000, reflectance detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region. The samples were placed in an elongated cell for analysis. The 2170 nm band is wool absorption, with no interference from rayon. For nylon and acrylic, the most significant region appears around 2050 nm. For moisture, the 1932 nm band was used (SEC of 0.5% for range of 1.7–13%). For finish, a calibration was developed at 2110 nm (SEC of 0.1% for range of 0.4–1.2%).

Results

The results indicate that the NIR spectrum can be used to determine the different components in textile blends, and therefore quantitative analysis of each component in the blends should easily be performed. The ability to monitor moisture and finish were also demonstrated. The results can be improved dramatically using calibrations developed individually for each type of material.

No. 7: Measuring finish on crimped and uncrimped fibers, and total solids in bath liquor

Summary

This application shows the measurement of the amount of finish on fiber (crimped or uncrimped) and the amount of total solids (TS) in bath liquor. Eight samples of uncrimped fibers, crimped fibers and bath liquor were provided.

System

Model 5000, reflectance detector module, transmission detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The spectra of the fibers were collected in reflectance mode and the liquors in transmission mode. Spectra on the fibers were collected using a coarse sample cell. The scan range was 1100–2500 nm. A poor correlation between finish on fiber and wavelength was obtained. However, the wavelength and the error were not reported. For TS, a calibration was developed at 1724 nm with a SEC of 0.05% for seven samples in the 0–2.24% range.

Results

The results indicate that NIR can be used to determine total solids in bath liquor. The results for finish on fiber indicate that it may not be feasible to monitor the finish. However, results may improve by averaging the data.

No. 8: Monitoring finish on fibers

Summary

This feasibility study was aimed to monitor finish on fibers from two different processes. First, bobbins were analyzed to develop a calibration, then more bobbins were analyzed to validate the calibration. Finally, routine samples were analyzed to test the validity of the calibration on real fiber samples.

System

Model 5000, transmission detector module, fiber optic setup module, interactance fiber and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer DirectLight/NonContact	2.928.0310

Sampling

The samples were analyzed in the 1100–2500 nm region using a fiber optic reflectance probe. Each bobbin was analyzed four times, repositioning the probe between scans.

For process 1, 30 samples were analyzed with finish concentration ranging from 0.5–1.7%. A PLS regression was performed in the 1100–1350 nm, and 1600–1850 nm regions (SEC of 0.1%, seven factors).

For process 2, 29 samples were analyzed with finish concentration ranging from 0.697–1.889%. A PLS regression was performed using the averaged spectra (SEC of 0.2%, three factors).

Results

The results indicate that NIR can be used to monitor finish on fiber for two different process runs.

No. 9: Monitoring finish oils on fibers

Summary

This application shows the use of NIRS for monitoring finish oil on fibers. Sixteen fiber samples were provided with finish content ranging from 0.136–0.825%. Also provided were the raw fiber, the base oil, and the emulsion.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region using an interactance reflectance probe. Each sample was analyzed six times, repositioning the probe between scans. Upon comparison of the raw fibers and the oil, spectral differences were identified throughout the 1350–2280 nm region. A PLS calibration was performed in this spectral region. Five factors were necessary to describe this chemical system (SEC of 0.06%). Upon external validation, the calibration model predicted well.

Results

The results indicate that NIR can be used to monitor finish oil on fibers.

No. 10: Monitoring percent finish on polyester

Summary

This feasibility shows the NIR's ability to monitor percent finish (fluorocarbon based material) on polyester. The finish is measured as percent fluorine. The sample concentration ranged from 0.08–0.367% fluorine.

System

Model 5000, reflectance detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm spectral range. A coarse sample cell was used for analysis. Each sample was analyzed in duplicate.

A PLS calibration was performed in the 1200–2400 nm region (SEC of 0.02%, six factors).

Results

The results indicate that NIR can be used to monitor the fluorocarbon based finish on polyester. The only questions which remain concern the sampling approach, and ways to minimize the error associated with this approach.

No. 11: Determination of the amount of polyester in a polyester/cotton blend

Summary

This application shows a quantitative analysis of the amount of polyester in a polyester/cotton blend. Thirty-five samples were received which covered the range of 44–57% polyester.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

NIR spectra were collected in reflectance mode, using the spinning sample cup modular attachment. Samples were scanned in the 1100–2500 nm wavelength region. Three scans were collected for each sample, with the sample cup being emptied and refilled for each scan. A calibration was developed at 1660 nm (SEC of 1%) for quantitating the amount of polyester in the blend.

Results

The results indicate that NIR can be used to quantitatively determine the percentage of polyester in polyester/cotton blend samples.

No. 12: Monitoring the amount of PBI during the batch production of fire retardant fabric

Summary

In this application, NIRS is used to monitor PBI in fire retardant fabric. The PBI concentration ranged from 21–30%.

System

Model 5000, reflectance detector module, horizontal setup module; or Model 5000, liquid sampling system was used for this application. These analyzers are no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed using a horizontal setup module. The thick, viscous liquid samples were analyzed in a plastic vial, sealed with a mylar sheet. The samples were analyzed in the 1100-2500 nm range in reflectance. Four scans of each sample were collected. The samples were also analyzed in transmission mode using a 1 mm cuvette. A calibration for the reflectance data was performed at 1128 nm. A SEC of 1% was obtained (too high SEC, and absorbance values greater than 1 absorbance unit). Regression on the transmission data at 1662 nm yielded a SEC of 0.2%.

Results

The results indicate that NIR can be used to monitor in fire retardant batch process.

No. 13: Monitoring polybenzimidazole (PBI) on fire retardant materials

Summary

This feasibility study was aimed to monitor polybenzimidazole (PBI) on high performance fibers used in fire retardant materials. Thirteen samples were provided for this study with PBI concentrations ranging from 0.5–0.76%.

System

Model 5000, reflectance detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. A coarse sample cell was used for analysis. Each sample was analyzed five times, repacking the sample between scans. A unique absorption for the PBI was located at 1686 nm (the high performance fiber did not contribute to the spectra). A least-squares regression was performed at 1686 nm (SEC of 0.1%).

Results

The results indicate that NIR can be used to monitor the percent PBI in high performance fibers. Improvements in the accuracy measurements would require additional samples with reproducible primary laboratory values. The precision of the measurement was found to be significantly better than the primary staple extraction.

No. 14: Determining moisture and oil content in wool samples

Summary

This application shows that NIRS can be used to determine moisture and oil content in wool samples. The moisture content ranged from 16.28–17.44%, while the oil content values were from 0.57–0.59%.

System

Model 5000, reflectance detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm range in reflectance mode. A coarse cell was utilized for analysis. For moisture analysis, two spectral regions are utilized: 1440 and 1920 nm. Calibration equations were developed at 1920 nm (SEC of 0.02%) and 1438 nm (SEC of 0.04%). For oil analysis, a least-squares regression was performed at 2266 nm (SEC of 0.001%). This wavelength region corresponds to the combination of O-H and C-C stretches found in fats and oils.

Results

The results indicate that NIR can be used to monitor the moisture and oil content in wool samples.

No. 15: Monitoring the levels of total solids in bath liquors

Summary

This feasibility study was aimed to use NIRS for monitoring the levels of total solids in bath liquors. During the course of this study, it was necessary to determine the additives which may be an interference. The total solids concentration ranged from 0.5–5.0%, while the additives ranged from 30 ppm to 20%.

System

Model 5000, liquid sampling system, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100–2500 nm region. The spectrum of the bath liquors, DMAc antifoam and dyes were measured in transmittance, using a 0.5 mm pathlength cuvette cell. The NIR spectra of the softener, sorbitan monopalmitate, and sodium bicarbonate were measured in reflectance, in a standard powder cell. For TS, a calibration was developed at 2288 nm (SEC of 0.3%, majority of samples were at 2.5% level). To correct for the interference from the softener, a second term was added to the calibration model (2288 and 2234 nm, SEC of 0.2%).

Results

The results indicate that NIR can be used to accurately predict total solids in bath liquors. The observable interference due to the softener was easily reduced by adding a second wavelength. In addition, the other additives were not an interference for total solids.