Metrohm NIRSystems

Analysis of polymer using near-infrared spectroscopy

Branch

Plastics and polymers

Keywords

Near-infrared spectroscopy, polymer, monomer, polymerization, polypropylene, polyethylene, PET, resin, polyols, polyesters, polyolefin, hydroxyl number, hydroxyl value, acid value, density, melt index, additive, carboxyl end group, carboxyl number,

Summary

This Application Bulletin presents some examples of NIR applications and feasibility studies using NIRSystems in the polymer industry. It includes analysis of various parameters in different types of sample. Hydroxyl number is one of the common parameters that can be rapidly determined using NIR spectroscopy. Determinations of hydroxyl number in different value ranges and in different types of polyols were studied. Each application here briefly describes the measuring system as well as the recommended instruments and the study results.

Introduction

Due to NIR analysis requiring no sample preparation and being nondestructive, many polymer and plastic attributes can be measured rapidly inline, online, atline or offline for qualitative as well as quantitative parameters. NIR spectroscopy is sensitive to the O-H bond absorption, therefore hydroxyl number determination is a common application. Thermoplastics production, raw material purity, and moisture content can be analyzed with NIR spectroscopy. The disappearance of double bonds can be monitored real time in reactions using inline NIR process analyzer. Residual solvents, monomers and additives are possible to be analyzed. NIR analysis also allows for the determination of physical properties such as molecular weight, degree of branching, tacticity, melting point, particle size verification, density, and viscosity. NIR spectroscopy is an excellent tool for determining the incoming raw materials and reaction endpoints, reducing over-processing of product and improving production consistency.

Contents

No. 1: Monitoring hydroxyl number in polyols ranging from 78.25 down to 38.48

Summary

This study shows the NIR application for monitoring hydroxyl number in polyols. Samples with hydroxyl number ranging from 78.25 down to 38.48 were used for calibration.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The spectral region used for this study ranged from 1100 to 2500 nm. The samples were analyzed in transmission mode, using a 4 mm cuvette. The samples were warmed to a constant temperature (approximately 32 °C). Two regions of the spectra show sensitivity to monitoring hydroxyl number: 2040 and 1430 nm. A calibration for OH number was performed at 2036 nm (SEC of 0.8 OH number). Another calibration was performed at 1429 nm (SEC of 2 OH number).

Results

The results indicate that NIR can be used to determine hydroxyl number in polyols. The precision of the NIR method has also been demonstrated.

No. 2: Determining hydroxyl number in polyols ranging from 299.33 down to 289.35.

Summary

NIR spectroscopy was used to determine hydroxyl number in polyols. Thirteen samples were provided with hydroxyl numbers ranging from 299.33 down to 289.35. Four unknown samples were provided for calibration validation purposes.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The spectral analysis was performed in transmission mode, using a 4 mm cuvette. The only sample preparation required for this study involved the warming of the samples to a constant temperature (approximately 40 °C), to minimize wavelength shifts of the -OH band. A calibration was developed at both 2080 nm (SEC of 0.4 OH number) and 1424 nm (SEC of 1 OH number) for OH number.

Results

The results indicate that NIR can be used to determine hydroxyl number in polyols. The precision of the NIR method has also been demonstrated. This study has also suggested that it would be possible to use either a cuvette method of analysis, or an interactance fiber optic bundle probe as the sampling method for the determination of hydroxyl number in polyols.

No. 3: Monitoring hydroxyl number in a polyol ranging from 8.3 to 9.95

Summary

This study presents a NIR application for monitoring hydroxyl number of polyols. Fourteen samples were provided for analysis, with OH number varying from 8.3 to 9.95.

System

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

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510

Sampling

The instrument was equipped with a fiber optic interactance immersion probe. The spectral range for the analysis was 1100 to 2500 nm. Two regions can be utilized for monitoring hydroxyl number: 1440, and 2080 nm. A least-squares regression was performed at 2092 nm (SEC of 0.4 OH number).

Results

The results indicate that NIR can be used to measure the hydroxyl number in polyols.

No. 4: Monitoring hydroxyl number and acid value in various polyol products

Summary

This application shows monitoring hydroxyl number and acid value in polyols using NIR spectroscopy. Twenty-three samples were analyzed with hydroxyl number ranging from 1 to 113 and acid value ranging from 0 to 55. Within this sample set existed smaller subsets of 10 to 15 different polymer products.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in transmission mode in the 1100 to 2500 nm region. All sample data was collected at a temperature of 45 °C, and allowed an equilibration time of 10 minutes. Due to the existence of various polyol products, a PLS calibration model was developed. Eight factors were necessary, yielding a SEC of 1.7 OH number. For acid value, an eight factor PLS calibration model yielded a SEC of 2.3.

Results

The results indicate that NIR can be used to monitor hydroxyl number and acid value in polyol samples consisting of various polyol products.

No. 5: Monitoring primary and secondary hydroxyl content in polyols

Summary

This study was aimed to monitor primary and secondary hydroxyl content in polyols. This was done using polyethylene glycol (PEG) to represent 100% primary O-H and polypropylene glycol (PPG) as 100% secondary O-H.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The spectra were collected in transmittance mode using a 1 mm cuvette. The spectral range was 1100–2500 nm. No calibrations were developed. Only the region assignable to O-H groups for model compounds whose hydroxyl groups are all primary or secondary was observed. The most significant difference was found for the O-H combination band, and a much smaller difference for the overtone of the O-H stretching vibration. It appears the greatest difference is due to the effect from the bending modes, which might be different due to a steric effect.

Results

The results indicate that NIR can be used to monitor the level of primary and secondary hydroxyl functional groups.

No. 6: Quantitatively determining the hydroxyl number in various solid and liquid polyols

Summary

The NIR spectroscopy was applied to monitor hydroxyl number in solid and liquid polyols. Approximately 40 samples were received which were divided into six categories: three groups had OH numbers ranging from 0 to 8.0, while the remaining groups had OH numbers ranging from 95–110, 218–238, and 205–225.

System

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

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The liquid samples were analyzed in transmission mode in the 1100–2500 nm region. An interactance immersion probe was used for analysis. The solid samples were also analyzed using a probe with a powder probe end. The liquid samples were heated to around 38 degree Celsius prior to analysis. For the liquid samples, a universal calibration with a large range (0–225 OH numbers) provided better results than individual calibrations. This was due to the small individual ranges. A calibration was developed at 1440 nm. For the solid samples, a calibration was performed at 1432 nm with a larger standard error.

Results

The results indicate that NIR can be used to monitor OH numbers to varying degrees of accuracy.

No. 7: Monitoring hydroxyl number (molecular weight) of polyols

Summary

This study shows the result of using NIR spectroscopy to determine hydroxyl number (indirectly monitoring molecular weight) of polyols. The samples provided ranged in hydroxyl number from 20 to 130.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

Samples were measured in transmission at a 4 mm pathlength. The melted samples were placed into quartz cuvettes for the NIR measurement, and the temperature was kept at 65 °C to insure the samples did not solidify. The 2042 nm band was used to monitor OH number (SEC of 2 OH number). To increase the accuracy, the samples were grouped by MW.

Results

The results indicate that NIR can be used to determine hydroxyl content. Determination can be directly performed on either the molten sample or on the room temperature solid. Molecular weight can be indirectly determined from the hydroxyl content at the same time.

No. 8: Monitoring the chain extender MOCA in polyols

Summary

This application was used to monitor the chain extender MOCA (methylenebis [2-chlorobenzenamine]) in polyols.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The spectral range was 1100 to 2500 nm. Depending on the material, either a transmission (liquid) or reflectance (solid) measurement was made. Spectral regions at 1980 and 2025 nm were identified as unique to MOCA in the polyol.

Results

This report demonstrates the feasibility of monitoring the MOCA chain extender content in a polyol. The fact that MOCA has unique absorption bands, and these bands are clearly seen in the polyol mixture but not in the pure polyol sample. It demonstrates that this analysis can be performed without interference from the other components in the samples.

No. 9: Detecting styrene-acetonitrile content in polyols

Summary

The objective of this study was to detect spectral differences between polyol samples differing in styrene-acetonitrile solids content (8, 25, and 45%).

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 to 2500 nm spectral region. Differences in the solids content are seen in the absorbance spectra. The baseline shifting occurs due to the difference in solids content. Also, absorptions associated with the acetonitrile are readily seen at 2150 nm.

Results

The results indicate that NIR can be used to detect differences in styrene-acetonitrile solids content of polyol samples.

No. 10: Qualitatively identifying poly-olefins

Summary

This study was aimed to evaluate the ability of NIR spectroscopy to identify polyolefins. Sixty nine samples (three each of twenty-three samples) were provided for the creation of the library.

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. The coarse sample cell was utilized for analysis. Each sample was reloaded and analyzed five times. Of the 345 NIR spectra which were obtained from the analysis of the calibration set, 230 spectra were used to create the NIR library, and the remaining 115 spectra were used to validate the library. The spectral regions, 1136–1220 nm, 1330–1444 nm, and 1618–1770 nm, were used for library development due to the spectral variations seen in these regions.

Results

The results indicate that NIR can be used to identify incoming raw material. Perfect matches were achieved for the four unknowns which were provided to test the use of NIR as an identification tool.

No. 11: Monitoring hydroxyl content in polyester

Summary

This NIR application was used to monitor hydroxyl content in polyester. The hydroxyl number ranged from 4.1 to 73.

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 polyester samples were crushed slightly so that the largest particles had a diameter of less than 1 cm. The samples were loaded into a coarse sample cell and analyzed in reflectance mode. The spectral range from 1100 to 2500 nm was used for this study. The hydroxyl changes were monitored at 1390 nm (SEC of 10 OH number). An outlier was removed which reduced the SEC to 9 OH number.

Results

The results indicate that NIR can be used to monitor hydroxyl content in polyester samples. While the samples measured with a large and wide variation in particle size showed a sizable sampling error, this can be drastically reduced by grinding the sample into a powder. By measuring the samples as a fine powder, the standard error of prediction for hydroxyl content should decrease even more. How the analysis is performed is dependent on two factors: the accuracy of the reference or lab method; and the precision necessary for the product.

No. 12: Monitoring hydroxyl value and acid value in esters

Summary

This study shows the NIR application for monitoring hydroxyl value and acid value in esters. Nineteen samples were provided with hydroxyl values ranging from 1.05 to 110.4 and acid values ranging from 0.052 to 71.9.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode using a fiber optic interactance immersion probe. A 1 mm pathlength was used for analysis. The hydroxyl region was monitored at 2010 nm. A least-squares regression was performed at this wavelength (SEC of 2 OH number). Due to the large range, the calibration set was divided into two groups, and then a calibration was developed for each (2008 nm, SEC of 1 OH number). For acid value, a calibration was developed at 1882 nm (SEC of 3).

Results

The results indicate that NIR can be used to monitor the hydroxyl value in the esters. It also appears that acid value can be monitored, however, the desired detection limit, and the accuracy of the primary method need to be clarified.

No. 13: Monitoring hydroxyl number in polyesters ranging from 184 to 192

Summary

The NIR application shows ability of NIR spectroscopy to monitor hydroxyl number in polyesters. Six samples with hydroxyl values ranging from 184 to 192 were analyzed. Five of these samples were used for calibration, while the remaining one was used for validation purposes.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100–2500 nm region using an interactance reflectance probe. Each sample was analyzed five times, repositioning the probe each time. The spectral band at 2057 nm is increasing in intensity with increasing hydroxyl value, indicating that a calibration model could be developed at this wavelength.

A calibration developed at 2057 nm yielded a SEC of 0.9 OH number. The error associated with the sampling method was 0.4 OH number, while the precision (error associated with the instrument) was 0.1 OH number. This indicates that some sample preparation may be necessary. This would involve simply assuring that the probe is placed on a flat surface for analysis.

Results

The results indicate that NIR can be used to monitor hydroxyl number in polyesters. The only requirement for the analysis is that a flat surface is present on the sample.

No. 14: Monitoring hydroxyl number in polyesters ranging from 23.2 to 129.4

Summary

This study shows the NIR application for monitoring hydroxyl number of polyesters in different ranges. Hydroxyl numbers ranged from 23.2 to 129.4. Six samples were provided, in addition to four unknown samples.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode, using a 10 mm cuvette. The only sample preparation involved warming the samples to 31 °C to minimize wavelength shifts of the OH band. A calibration was developed for OH number at 1458 nm (SEC of 3 OH number).

Results

The results indicate that NIR can be used to determine hydroxyl number in polyesters. This report has also demonstrated the precision obtainable with this particular method of analysis.

No. 15: Monitoring hydroxyl number and acid value in polyesters

Summary

This application shows the NIR application used to monitor hydroxyl number and acid value in polyester samples. Six samples were analyzed with hydroxyl number ranging from 39 to 93, and acid value from 8.25 to 9.21.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100–2500 nm region using an interactance reflectance probe. Each sample was analyzed five times, repositioning the probe between scans. A calibration for hydroxyl number was developed at 2057 nm yielding a SEC of 2 OH number. For acid value, a calibration was developed at 1952 nm (SEC of 0.1).

Results

The results indicate that NIR can be used to monitor hydroxyl number and acid value in polyesters.

No. 16: Monitoring vinyl acetate in polyethylene resin samples

Summary

This study was aimed to monitor vinyl acetate (VA) in polyethylene (PE) resin samples. Five pelleted samples were provided with VA concentration from 0.51 to 3.88%

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 to analyze the pellet samples. A calibration for VA was developed at 2146 nm (SEC of 0.2%). This is a region where PE does not interfere.

Results

The results indicate that NIR can be used to monitor VA in PE pelleted samples.

No. 17: Measuring the density of poly-ethylene powders

Summary

This study was aimed to evaluate ability of NIR spectroscopy to measure the density of polyethylene powders. Fifteen samples were analyzed for calibration development, ranging in density from 0.9376 to 0.9624. Five additional samples were analyzed as a validation set. Their densities ranged from 0.9589 to 0.9536.

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 to 2500 nm region in reflectance mode. Each sample was analyzed three times. Since density is affected by chain length, as well as cross linking, a multivariate calibration technique is necessary. A PLS calibration using five factors yielded a SEC of 0.002. Upon validation, it appears that this calibration model describes the density of the polyethylene.

Results

The results indicate that NIR can be used to monitor the density of polyethylene powders.

No. 18: Monitoring irganox 1010 in poly-ethylene pellets

Summary

This NIR application was used to quantify the amount of irganox 1010 in polyethylene pellets. Ten samples were provided for calibration development, along with the pure irganox 1010.

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 in reflectance mode. A coarse sample cell was used for analysis. A band unique to irganox 1010, with little interference from polyethylene, is found at 1686 nm. A least-squares regression was performed at 1688 nm.

Results

The results indicate that NIR can be used to determine the concentration of irganox 1010 in polyethylene pellets.

No. 19: Monitoring density and melt index in polyethylene pellets

Summary

This application was used to monitor density and melt index in polyethylene pellets. Twenty-two samples were analyzed with density ranging from 0.9173 to 0.9428 and melt index values ranging from 0.25 to 26.79.

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 to 2500 nm region. Each sample was analyzed two times, repacking the coarse cell between scans. Since density is affected by chain length, as well as cross-linking, it was necessary to utilize a more powerful regression technique than linear least-squares. A five factor PLS model was developed with a SEC of 0.0008. For melt index, a PLS model was again necessary. This nine factor model yielded a SEC of 2.2.

Results

The results indicate that NIR can be used to monitor density and melt index. Some questions remain concerning the obtainable accuracy for the melt index measurement.

No. 20: Monitoring the levels of isonox in polyethylene pellets

Summary

This study shows the result of NIR application for determining the level of Isonox in polyethylene pellets. Samples containing various levels were submitted for examination (hundreds of ppm level), as were samples of the Isonox, and two other additives which may pose an interference to analysis.

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 measured in reflectance mode using a coarse sample cell. The scan range was 1100 to 2500 nm. At 2138 nm, the polymer is essentially non-absorbing, and the Isonox shows an absorption. A calibration developed at this wavelength yielded a SEC of 55 ppm.

Results

The results indicate that NIR can be used to determine Isonox levels in polyethylene pellets.

No. 21: Monitoring the levels of vinyl acetate and three antioxidants in a low density polyethylene (LDPE) based polymer pellet

Summary

This application shows the use of NIR spectroscopy for monitoring the levels of vinyl acetate, and three antioxidants in a low density polyethylene based polymer pellet. Vinyl acetate (VA) concentration ranged from 0 to 2.7%. AO-1 concentration ranged from 15 to 945 ppm, AO-2 concentration ranged from 0 to 300 ppm, and AO-3 concentration ranged from 0 to 315 ppm.

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 NIR spectra of the polymer pellets were measured in reflectance mode in the spectral region from 1100 to 2500 nm. The pellets were analyzed using a coarse grain sample cell. A calibration for vinyl acetate was performed at 1670 nm (SEC of 0.3%). Calibrations for AO-1, AO-2, and AO-3 were developed at 2008 nm (SEC of 190 ppm), 1938 nm (SEC of 81 ppm), and 1634 nm (SEC of 47 ppm), respectively.

Results

The results indicate that NIR can be used to monitor not only vinyl acetate, but also antioxidants at the level of hundreds of ppm in a polyethylene based polymer.

No. 22: Monitoring a coating material on glass

Summary

This study shows NIR application for monitoring a coating material on glass. Four flat and four curved samples were provided. The samples were: the uncoated glass, glass coated with PE wax, glass coated with a new solution, and glass coated with PE wax and a new solution.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The spectral range was 1100–2500 nm. Although reflectance measurements were attempted, little energy was reflected (as would be expected); therefore, all results were based on transmission measurements. Two absorbances for the polyethylene wax can be seen at 1720 and 2320 nm. Furthermore, absorbances for the new treatment are seen at 1712, 1758, 2310, and 2250 nm. With the curved glass samples, differences were evident, however, the spectra were much noisier.

Results

The results indicate that NIR can be used to detect the presence of a new coating treatment for glass. The next step will be to determine whether quantitative determinations (whether actual or empirical) can be made for the coating treatment.

No. 23: Measuring ethylene, linear ethylene, and ethylene-propylene rubber content in polypropylene pellets

Summary

This application shows the use of NIR method to monitor the ethylene, linear ethylene and ethylene-propylene rubber content in polypropylene pellets. Fifteen samples were provided with total ethylene concentration ranging from 6.1 to 21.3%. Linear ethylene concentration ranged from 4.6 to 19.8%.

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

Samples were loaded into a coarse sample cell. The measurements were made in reflectance mode in the 1100–2500 nm region. Each sample was reloaded into the cell four times, with the resulting spectrum being an average of the four. Total ethylene was monitored at 2012 nm (SEC of 1). Linear ethylene monitored at 2012 nm (SEC of 0.8%). For EP rubber, no region of the spectrum is useful for calibration (vibrations for this constituent aren't separated enough from those of the comonomers to be useful for this determination).

Results

The results indicate that NIR can be used to determine ethylene content in polypropylene pellets. Linear ethylene also could be monitored, and seemed to be highly correlated to the total ethylene content.

No. 24: Qualitatively distinguishing between a series of polymer samples

Summary

This study was aimed to apply NIR analysis to qualitatively distinguish between a series of polymer samples. Nine samples were provided consisting of three virgin resin components (octene LLDPE, hexene LLDPE, and HMW-HDPE), two reprocessed plastic pellets (RPP), and four post-consumer materials (PCM).

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 400–2500 nm region using a coarse sample cell. Each sample was analyzed a total of five times. A spectral library was created using the regions: 624–1064, 1140–1400, and 1620–1778 nm. Match by distance revealed no errors in library development.

Results

The results indicate that NIR can be used to qualitatively distinguish between polymer samples.

No. 25: Qualitatively distinguishing between polypropylene pellets

Summary

This application was used to qualitatively distinguish between different polypropylene (PP) pellets.

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 to 2500 nm region in reflectance mode. It initially appeared as though the PP samples differed only in TiO2 content. However, after looking at the second derivative spectra, additional spectral absorptions were identified at 2060 and 2135 nm. These differences may be associated with antioxidants and/or UV stabilizers present in the pellets.

Results

The results indicate that NIR can be used to differentiate between samples of polypropylene.

No. 26: Monitoring calcium carbonate, calcium stearate, and talc in chlorinated polyethylene

Summary

The objective of this study was to monitor calcium carbonate, calcium stearate, and talc in chlorinated polyethylene (CPE). Sixty-seven samples were received with calcium stearate levels from 0.0 to 3.0%, talc levels from 0 to 7%, and calcium carbonate from 0 to 4%. These samples were provided at three levels of chlorination: 25, 35, and 42%.

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 samples were analyzed in a standard sample cup. Each sample was analyzed three times. PLS was used for calibration development to account for the complexity of the matrix. PLS was performed in the 1100–2500 nm region. For calcium stearate, the model yielded a SEC of 0.3%. For talc, the model yielded a SEC of 0.4%, and for calcium carbonate, the PLS model yielded a SEC of 0.2%. The PLS models required six factors, five factors, and five factors, respectively.

Results

The results indicate that NIR can be used to monitor calcium stearate, talc, and calcium carbonate in chlorinated polyethylene.

No. 27: Quantifying an additive in polypropylene pellets

Summary

This NIR application was used to quantify an additive in polypropylene (PP) pellets. Twenty samples were used for calibration development. Fifteen of these samples were prepared in a pilot plant, while the remaining five were actual process samples. A single calibration equation was desired. The samples ranged from 5 to 15% in sample concentration.

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 using a coarse sample cell. A least-squares regression was developed at 2156 nm (SEC of 0.5%). To compensate for multiplicative and additive scatter effects, a divisor wavelength at 1386 nm was added to the calibration model (2156/1386 nm, SEC of 0.3%).

Results

The results indicate that NIR can be used to develop a single calibration model for monitoring an additive in polypropylene pellets from two extruders.

No. 28: Monitoring additive levels (tinuvin 770 and irganox B225) in a copolymer blend of polypropylene and polyethylene

Summary

This study shows the use of NIR analysis to monitor Tinuvin 770 and Irganox B225 in a copolymer blend of polypropylene (PP) and polyethylene (PE). The ultimate goal was to make one calibration model for each additive to estimate the concentration in both the copolymer and the pure polymer, PP.

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 utilized for sample analysis. For the tinuvin in the copolymer, a calibration was developed at 1540 nm (SEC of 0.03%). For the irganox in the copolymer, a calibration was developed at 1412 nm (SEC of 0.07%). For the generic calibration in the copolymer + PP, a second wavelength had to be added to each calibration. For tinuvin, a calibration was developed at 1540/1490 nm (SEC of 0.04%). For irganox, a calibration was developed at 1412/1220 nm (SEC of 0.06%).

Results

The results indicate that NIR can be used to monitor Tinuvin 770 and Irganox B225 additive levels in polymer pellets.

No. 29: Predicting xylene soluble in polypropylene

Summary

This study was aimed to evaluate NIR's ability to predict xylene soluble ranging from 2.5–6% in polypropylene.

System

The NIRSystems instrument used to collect the spectra was a model 5000 Monochromator with a Multi-Mode Analyzer sampling system. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110

Sampling

42 samples of polypropylene containing xylene solubles covering the range of interest were placed in individual Standard Cup sample cells. Special attention was paid to avoid segregation of sample fines to the sample cup window. Samples were scanned in reflectance from 1100–2500 nm. A second derivative math pre-treatment using a 10 nm segment and a 0 gap, was applied to the raw spectra.

Results

A three term linear model was developed using wavelengths at 1700, 1734, and 1218 nm, producing a coefficient of determination (R^2) of 0.9868 and a Standard Error of Calibration (SEC) of 0.16%.

No. 30: Distinguishing between acceptable and contaminated polyethylene terephthalate (PET) samples

Summary

This NIR application was used to distinguish between acceptable and contaminated PET samples. Four sample sets were analyzed: PET pellets, PET flakes, PET strips, and PET sheets. Also provided were wash water samples.

System

Model 5000, reflectance detector module, sample transport module, transmission detector 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
NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100–2500 nm region. The pelleted and flake samples were analyzed in reflectance mode using a coarse sample cell. The strip samples were analyzed using an interactance reflectance probe, while the wash water samples were analyzed in transmission mode using a 1 mm pathlength cuvette. Libraries were created using the control samples. IQ2 was used to correctly identify all samples.

Results

The results indicate that NIR can be used to qualitatively distinguish between acceptable and contaminated PET samples.

No. 31: Monitoring the concentration of linear low density polyethylene (LLDPE) in polyethylene terephthalate (PET) sheets

Summary

This application shows determination of the concentration of linear low density polyethylene (LLDPE) in polyethylene terephthalate (PET) sheets using NIR spectroscopy.

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

A small piece, approximately 2 inches by 2 inches was cut from each sheet, and from the flat portion of the formed boats. These samples were placed into the sample transport mechanism. The sheets were analyzed in transmittance mode. The LLDPE beads were measured in reflectance mode using the standard sample cup. The 2310 nm band varies in intensity in the PET/LLDPE sheets, but is absent in the PET pure spectrum. Therefore, LLDPE can probably be monitored in this region. The 2260 nm band is due to PET in the sheets.

Results

The results indicate that NIR can be used to determine LLDPE levels in PET/LLDPE polymer sheets and boats. Since we have no data on the actual LLDPE levels of these polymer samples, it is impossible to assess the sensitivity or the precision of the NIR analysis.

No. 32: Monitoring the melt index (MI) of polypropylene pellets

Summary

This study shows the use of NIR spectroscopy to monitor the melt index (MI) of polypropylene pellets. Provided were samples with MI range from 0.8 to 36.55 and six unknown samples for prediction.

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

Samples were placed in a coarse sample cell and scanned in reflectance in the 1100–2500 nm region. Spectral differences can be seen in the 1700 to 1900 nm region. A calibration was developed at 1760 nm yielding a SEC of 4.

Results

The results indicate that NIR can be used to determine the melt index of polypropylene pellets.

No. 33: Monitoring melt flow rate (MFR) of polypropylene powders

Summary

The objective of this study was to monitor the melt flow rate (MFR) of polypropylene powders. Samples ranged from 3.0 to 19.5 MFR. Two groups of samples, A and B, were provided. The desired precision was +/- 5%. For A, the MFR ranged from 5.6 to 7.4, while for B, the MFR ranged from 3.0 to 19.5.

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. A standard sample cup was used. Two different processes and catalysts were used to develop the A and B groupings. When a single calibration was attempted for both groups, the best error obtained was 3.0 MFR. It suggested that two separate calibrations were necessary. PLS calibrations were used because MFR is a physical rather than a chemical characteristic. For A, a PLS model was developed using 4 factors (SEC of 0.3). For B, an 8 factor PLS model was developed (SEC of 2).

Results

The results indicate that NIR can be used to determine MFR of polypropylene samples from the A process. The B calibration set was essentially a two point calibration, not describing the entire B process. More samples are necessary to determine if MFR can be measured in the B process.

No. 34: Measuring ethylene, linear ethylene, and ethylene-propylene rubber content in polypropylene pellets

Summary

NIR spectroscopy was used to monitor the ethylene, linear ethylene and ethylene-propylene rubber content in polypropylene pellets. Fifteen samples were provided with total ethylene concentration ranging from 6.1 to 21.3%. Linear ethylene concentration ranged from 4.6 to 19.8%.

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

Samples were loaded into a coarse sample cell. The measurements were made in reflectance mode in the 1100–2500 nm region. Each sample was reloaded into the cell four times, with the resulting spectrum being an average of the four. Total ethylene was monitored at 2012 nm (SEC of 1). Linear ethylene monitored at 2012 nm (SEC of 0.8%). For EP rubber, no region of the spectrum is useful for calibration (vibrations for this constituent aren't separated enough from those of the comonomers to be useful for this determination).

Results

The results indicate that NIR can be used to determine ethylene content in polypropylene pellets. Linear ethylene also could be monitored, and seemed to be highly correlated to the total ethylene content.

No. 35: Monitoring the presence of an additive in polypropylene (PP) RCP base resin

Summary

This NIR application was used to monitor the presence of additive in a polypropylene (PP) RCP base resin. Five samples with additive concentrations ranging from 5 to 15% were provided for the feasibility study.

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 spectral region used for the study was 1100–2500 nm. Only the powdered samples were analyzed for the study, as the film samples would create difficulties due to differences in the pathlength. The powdered samples were analyzed in reflectance mode. The coarse sample cell was used to hold the samples. A calibration monitoring additive in PP was developed at 2170 nm (SEC of 0.36%).

Results

The results indicate that NIR can be used to monitor the presence of additive in polypropylene. Based on the results, it is likely that the required accuracy for the analysis is attainable.

No. 36: Qualitative comparison of polystyrene samples

Summary

This NIR application was used to qualitatively compare samples of high impact and general purpose polystyrene. Along with the high impact and the general purpose polystyrene, red color pellets, and two injection molded samples were received for analysis.

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. Each sample was scanned five times. Differences in the spectra were observed. Therefore, IQ2 could be used to statistically differentiate between these samples.

Results

The results indicate that NIR can be used to detect differences between the samples of polystyrene.

No. 37: Monitoring bromine content of polystyrene product

Summary

This NIR application was used to monitor the bromine content in a polystyrene product. Five standard samples of the blue polystyrene product of known bromine content (0, 0.3, 0.67, 1.06, and 2.13%) along with a small sample of the bromine containing additive, hexabromocyclododecane (HBCD) were provided.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100–2500 nm range. Stray light was excluded by means of an opaque skirt between the sample and probe. Eight spectra were taken in different positions on each sample (four positions on each side). Differences between the spectra of the samples with different bromine content were seen at 1143, 1681, 2164 nm. Although calibrations were developed using MLR, the most robust calibration was done using PLS. Three factors were used with a SEC of 0.09% (prediction SEC of 0.14%).

Results

The results indicate that NIR can be used to monitor bromine content (HBCD) in polystyrene product.

No. 38: Monitoring a coating material on polystyrene pellets

Summary

This study shows the use of NIR spectroscopy to monitor a coating on polystyrene pellets. Two sets of pellets were provided: T-series and B-series. These two sets are different products of distinct particle size. The concentration of the coating ranged from 99 to 922 ppm.

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. Absorptions for coating material can be seen without interference from the polystyrene at 1730, 1800, and 2060 nm. A separate calibration was developed for each product at 2062 nm. (No SEC reported). The low concentration samples could not be detected using NIR.

Results

The results indicate that NIR can be used to measure coating on B-series and T-series samples. The question remains concerning the desirable limit of detection.

No39: Monitoring polysulfone, glycerin, and moisture in polysulfone samples

Summary

This NIR application was used to monitor polysulfone (PSF), glycerin (GLY), and moisture in polysulfone fiber samples. Five sets of fiber samples were analyzed with constituent levels ranging from 16.64 to 31.52% PSF, 65.35 to 79.39% GLY, and 2.97 to 3.99% moisture.

System

Model 5000, rapid content analyzer was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110

Sampling

The samples were analyzed using a rapid content sampling system. Each fiber sample was simply laid on the window for analysis. The scan range was 1100 to 2500 nm. Each sample was analyzed five times. A strong absorption for PSF was identified at 1670 nm, while a GLY absorption was located at 2100 nm. Moisture has two distinct bands at 1450 and 1930 nm. A calibration for PSF was developed at 1670 nm (SEC of 1.2%). For GLY, a calibration was developed at 2114 nm (SEC of 1.3%). The moisture calibration developed at 1432 nm yielded at SEC of 0.2%.

Results

The results indicate that NIR can be used to monitor polysulfone, glycerin, and moisture in polysulfone fiber samples. By analyzing additional samples and including a denominator term in the calibrations to minimize pathlength variations, the SEC would be reduced.

No.40: The determination of sulfone in monochlorobenzene

Summary

This study was aimed to evaluate NIR's ability to quantitate sulfone in monochlorobenzene.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

Twelve samples were prepared gravimetrically using three production samples which had been quantified by GC and titration. The range of sulfone concentration in the 12 samples was 13–20%. Samples were placed in quartz cuvettes of 1 mm pathlength.

Results

A second derivative math pre-treatment (segment = 20 nm, gap = 0) was applied to the sample spectra. The application of a multiple linear regression (MLR) yielded a wavelength selection of 2204 nm producing an R^2 of 0.9973, SEC = 0.24, and SEP = 0.22.

No. 41: Monitoring a coating on expandable polystyrene beads

Summary

The NIR application was used to monitor a coating on expandable polystyrene beads. Samples were analyzed containing between 0.01 and 0.2% coating containing moisture (0.05 to 0.5%), pentane (5 to 6%), and anti-static agent (~0.005%). Raw expandable beads and the pure coating were also analyzed.

System

Process Analytics Model 5000 with remote 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

Each sample was placed in a sample cup which was then placed on the remote reflectance head. Absorptions due to the coating are found at 1720, 2010, and 2050 nm. Features due to the raw beads are found at 1700 and 2140 nm. Pentane has an absorption band at 1700 nm and water has distinct features at 1450 and 1900 nm. A PLS calibration for the coating was developed in the 1150–1650 nm, and 1716–2100 nm regions (SEC of 0.007%).

Results

The results indicate that NIR can be used to monitor a coating on expandable polystyrene beads. It was also shown that moisture and pentane show absorptions. Calibration for these two constituents would require sample sets covering the range of sample concentration. The anti-static agent is negligible due to the low concentration.

No. 42: The determination of chloro-benzene sulfonic acid in a polysulfone mixture

Summary

This study was aimed to evaluate NIR's ability to quantitate chlorobenzene sulfonic acid in a polysulfone mixture.

System

Spectra were collected from 400–2500 nm using a NIRSystems Model 6500 monochromator with a Liquid Sample module with the temperature set at 60 °C. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer   2.921.1410

Sampling

Eighteen samples were prepared gravimetrically using three production samples which had been quantified by GC and titration. The range of chlorobenzene sulfonic acid in the 18 samples was 17.4–78.8%. Samples were placed in quartz cuvettes of 1 mm pathlength.

Results

A second derivative math pre-treatment (segment = 20 nm, gap = 0) was applied to the sample spectra. The application of a multiple linear regression (MLR) yielded a wavelength selection of 1408 nm producing an R^2 of 0.9905, SEC = 1.8, and SEP = 3.4.

No. 43: The determination of sulfuric acid in a polysulfone mixture

Summary

This study was aimed to evaluate NIR's ability to quantitate sulfuric acid in a polysulfone mixture.

System

Spectra were collected from 400–2500 nm using a NIRSystems Model 6500 monochromator with a Liquid Sample module with the temperature set at 60 °C. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

Eighteen samples were prepared gravimetrically using three production samples which had been quantified by GC and titration. The range of sulfuric acid in the 18 samples was 0.26–4.08%. Samples were placed in quartz cuvettes of 1 mm pathlength.

Results

A second derivative math pre-treatment (segment = 20 nm, gap = 0) was applied to the sample spectra. The application of a multiple linear regression (MLR) yielded a wavelength selection of 1186 nm producing an R^2 of 0.9239, SEC = 0.33, and SEP = 0.31.

No. 44: Monitoring the reaction of the hydroxy group, polybutadiene (HTPB) and isopherone-di-isocyanate (IPDI) in forming a polyurethane

Summary

This NIR application was used to monitor levels of bisulfate, carbonate and oxone in crystalline toilet cleaner.

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

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were scanned in the 1100 to 2500 nm region. All spectra were collected in transmission mode, except the finished product which was collected in reflectance. Differences in the two starting materials are apparent throughout the spectrum. The starting materials can be seen in the first mixture, and the final product also. Polyurethane absorptions can also be seen in the final mixture.

Results

The results indicate that NIR can be used to monitor the presence of the two starting materials, along with the extent of reaction for the polyurethane. Furthermore, it appears that the presence of the aluminum filler will not significantly interfere with the NIR measurement; therefore, quantification of the various materials should be possible.

No. 45: Monitoring a polyurethane reaction

Summary

This NIR application was used to monitor a polyurethane reaction. The A polymer ranged from 1.96 to 9.81%, while the B polymer range was 2.05 to 9.80%.

System

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

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510

Sampling

The samples were analyzed from 1100–2500 nm using an interactance immersion probe with a 1 cm pathlength. The calibration for the A samples was used to monitor the reaction of NCO. The polyol, MDI and the fiber optic probe were placed in a 1 liter reactor with constant agitation. Upon completion of the initial reaction of MDI with the polyol, polyethylene was added as a chain extender. Samples were extracted and titrated every seven minutes throughout the reaction. These samples were used to correct the original equation for difference between static and flowing samples. The B polymer was monitored at 1950 (SEC of 0.2%) and 2224 nm (SEC of 0.2%). The A polymer was monitored at 1946 (SEC of 0.4%) and 2226 nm (SEC of 0.2%). Together, the A and B polymers were monitored at 2223+1626 nm (SEC of 0.1%) and 1938+1888 nm (SEC of 0.1%).

Results

The results indicate that NIR can be used to monitor reactions. Further expansion of these results could provide both MDI, polyol, PE and polyurethane results simultaneously every 30 seconds in this type of reaction.

No. 46: Monitoring the Curing of a Polyurethane Elastomer

Summary

This NIR application was used to monitor the curing of a polyurethane elastomer. One sample was provided for analysis. The sample was placed into an 80 °C oven to promote curing.

System

NIRS XDS Process Analyzer DirectLight/NonContact	2.928.0310

Sampling

The sample was analyzed in the 1100 to 2500 nm range using a Process Analytics Direct Light System. Initially, the sample was analyzed every few hours, then every couple of days. This was done until no change was detected in the spectra, indicating complete curing. Spectral differences were detected in the 1440 nm hydroxyl region. This band was seen to decrease with increasing curing time. Also detected were differences in the 1485 nm amine band. An increase in this band is detected with increasing curing time.

Results

The results indicate that NIR can be used to detect spectral changes during the curing of a polyurethane elastomer.

No. 47: Monitoring free isocyanate (NCO) content of a polyurethane reaction in-line

Summary

The NIR application was developed to determine the free isocyanate (NCO) content of a polyurethane reaction in-line. Over an eight hour period, spectral measurements were taken at five minute intervals and samples taken at ten minute intervals for reference assays.

System

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

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510

Sampling

The samples were analyzed in transmission mode using an interactance immersion fiber optic bundle probe. The scan range was 1100–2500 nm. The tip of the probe was placed in a circulation loop of the reactor. The optical pathlength was 1 cm (i.e. 5 mm between the probe tip and polished stainless steel reflector). For monitoring NCO, after glycidol/butanol addition, the 2116+1920 nm bands were used (SEC of 0.04% for 14 samples in range 0.66 to 2.02%). For monitoring NCO, after complete reaction, the 2112+1922 nm bands were used (SEC of 0.06% for 30 samples in range 0.66 to 3.38%). The reference method error is 0.03%.

Results

The results indicate that NIR can be used to determine the percentage of NCO in polyurethane polymerizations. The determination can be performed in-line enabling real time chemical process control to be performed.

No. 48: Determining percent linear expansion in polyurethane resins

Summary

The application shows the use of NIR method to determine the percent linear expansion in polyurethane resins. Six samples were received for analysis, each representing a specific value for percent linear expansion (LE) in the range of 110 to 128.9%. Linear expansion is related to hydroxyl value.

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. Two spectra were collected for each sample reloading the standard sample cup between scans. A general trend is observed in the 2080 nm region (characteristic hydroxyl region). A calibration was developed at 2112 nm (SEC of 5.01%). A denominator term, 2196 nm, was included in the calibration to correct for scattering differences caused by the irregularly shaped materials. This reduced the SEC to 2.08%.

Results

The results indicate that NIR can be used to monitor linear expansion in polyurethane resins.

No. 49: Monitoring a urethane prepolymer for viscosity and level of isocyanate

Summary

This study was aimed to evaluate NIR's ability for monitoring a urethane prepolymer for viscosity ranging from 1470–4680 cp and the level of isocyanate (NCO) ranging from 3.08 to 8.89%.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

| NIRS XDS RapidLiquid Analyzer | 2.921.1410 |

Sampling

The samples provided were either solid or liquid, all were liquefied at 60 °C. The samples poured into 4 mm cuvettes. A calibration for NCO was developed at 2124 nm (SEC of 0.3%). To compensate for differing molecular weights, a second wavelength, 1460 nm, (2124 + 1460 nm) was added to the above equation (SEC of 0.2%). A calibration of viscosity was developed at 1662 nm (SEC of 215). 1456 nm was added to this equation to compensate for MW differences (1662 + 1456 nm, SEC of 163).

Results

The results indicate that NIR can be used to monitor total isocyanate content, and viscosity for urethane prepolymer samples. After careful selection of a wavelength assignable to the source of isocyanate in this material, clustering into groups based on molecular weight difference was noted, and was corrected in the calibrations by addition of a term at a wavelength assignable to molecular weight differences. A similar determination was also performed for viscosity and good results were also obtained using this approach.

No. 50: Qualitative determination of good and bad samples of polyvinyl chloride (PVC)

Summary

This NIR application was used to qualitatively determine between good and bad samples of PVC. Eleven samples were received for analysis, including eight controls and three samples of unknown quality.

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 using a spinning sample module. IQ2 was used to distinguish between the good and bad samples. The different color of one sample, along with the larger particle size explained the failure in the quality category.

Results

The results indicate that NIR can be used to qualitatively distinguish between good and bad samples of PVC.

No. 51: Distinguishing among various PVC samples

Summary

This NIR application was used to distinguish between various PVC samples.

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 spectra were collected in reflectance mode in the 1100–2500 nm range. The PVA samples were analyzed using the coarse sample cell. Due to the small calibration range, and the standard error associated with the primary method, it is difficult to demonstrate feasibility based on a single wavelength. PLS was therefore utilized, in the 1600–1900 nm and 2000–2400 nm spectral regions (SEC not reported).

Results

The results indicate that NIR can be used to monitor the hydrolysis of polyvinyl alcohol. The results seem to indicate that accuracy similar to the primary method is achievable.

No. 52: Monitoring the level of an additive in PVC sidings

Summary

This NIR application was used to monitor the level of an additive in PVC sidings. Eight samples were provided with additive concentration ranging from 0.031 to 0.492%. Also provided were three unknowns.

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 strong absorption for the additive is seen at 2146 nm, calibration was developed at this wavelength, yielding a SEC of 0.03%.

Results

The results indicate that NIR can be used to monitor the presence of the additive in PVC sidings.

No. 53: Monitoring the hydrolysis reaction of polyvinyl alcohol (PVA)

Summary

The objective of this study was to monitor the hydrolysis reaction of polyvinyl alcohol (PVA). Thirteen samples were provided for the study with percent hydrolysis ranging from 95.59 to 98.52%.

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 spectra were collected in reflectance mode in the 1100–2500 nm range. The PVA samples were analyzed using the coarse sample cell. Due to the small calibration range, and the standard error associated with the primary method, it is difficult to demonstrate feasibility based on a single wavelength. PLS was therefore utilized, in the 1600–1900 nm and 2000–2400 nm spectral regions (SEC not reported).

Results

The results indicate that NIR can be used to monitor the hydrolysis of polyvinyl alcohol. The results seem to indicate that accuracy similar to the primary method is achievable.

No. 54: Monitoring hydroxyl concentration in terpolymer resins

Summary

NIR spectroscopy was applied to monitor hydroxyl concentration in terpolymer resins. The terpolymer consisted of polyvinyl chloride, vinyl acetate, and hydroxpropyl acrylate. Eighteen samples with hydroxyl concentration ranging from 1.37 to 1.89% were provided.

System

Model 5000, transmission detector module, fiber optic bundle 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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

All samples were analyzed in the 1100–2500 nm region in reflectance mode using a fiber optic bundle probe. A calibration was developed at 2086 nm, yielding a SEC of 0.04%.

Results

The results indicate that NIR can be used to determine hydroxyl concentration in a terpolymer of polyvinyl chloride, vinyl acetate, and hydroxpropyl acrylate.

No. 55: Monitoring hydroxyl percent in a terpolymer

Summary

This NIR application was used to monitor hydroxyl percent in a terpolymer of polyvinyl chloride, vinyl acetate, and hydroxpropyl acrylate. Eleven resin samples were provided with hydroxyl concentration ranging from 0.93 to 3.01%.

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 in reflectance mode. Each sample was scanned five times in a microsample cup. A calibration was developed at 2082 nm (SEC of 0.2 OH %). A second wavelength was added to the calibration to correct for differences in pathlength (a divisor wavelength at 1174 nm, 2082/1174: SEC of 0.03%).

Results

The results indicate that NIR can be used to measure hydroxyl concentration.

No. 56: Monitoring hydroxyl number in synthetic esters

Summary

This NIR application was used to monitor the hydroxyl number in synthetic esters. Nine samples were analyzed with hydroxyl number ranging from 1.13 to 6.00 mg KOH/g.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode. A 4 mm pathlength cuvette was utilized for analysis. Each sample was analyzed two times, at 25.5 °C. A calibration model for hydroxyl number was developed at 2046 nm (SEC of 0.4 KOH/gram). However, there were two distinct groupings of samples. By removing the lower samples, and regressing only on those samples with hydroxyl value between 3 and 6 mg KOH/g, a new calibration yielded a SEC of 0.07 KOH/g.

Results

The results indicate that NIR can be used to determine hydroxyl concentration in a terpolymer of polyvinyl chloride, vinyl acetate, and hydroxpropyl acrylate.

No. 57: Monitoring hydroxyl number in powdered resins

Summary

The NIR application was used to monitor hydroxyl number in powdered resins. The hydroxyl number ranged from 2.7 to 5.0.

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 to 2500 nm region. A coarse sample cell was utilized for analysis. A large hydroxyl absorption occurs at 2032 nm. A calibration was performed at 2032/1898 nm which yielded a SEC of 0.3 OH number. The divisor term was included to correct for pathlength variations.

Results

The results indicate that NIR can be used to monitor hydroxyl number in powdered resin samples.

No. 58: Monitoring percent methyl methacrylate in a styrene-maleic anhydride copolymer

Summary

NIR spectroscopy can be used for monitoring percent methyl methacrylate (MMA) in a styrene-maleic anhydride (SMA) copolymer. Thirteen samples were analyzed with MMA ranging from 1 to 15%.

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 to 2500 nm spectral region. A coarse sample cell placed into a sample transport mechanism was used for analysis. Each sample was analyzed three times. Several absorptions could be used to monitor MMA. A calibration was developed at 2252 nm (SEC of 0.2%).

Results

The results indicate that NIR can be used to monitor methyl methacrylate in styrene-maleic anhydride copolymer.

No. 59: Monitoring acetylables, primary, secondary, and tertiary amines in polyoxypropylene-diamine

Summary

This study was aimed to monitor acetylables, primary, secondary, and tertiary amines in polyoxypropylene-diamine. Thirty samples were provided for analysis. Acetylables ranged in concentration from 0.99 to 1.07%, primary amines ranged from 0.94 to 1.05%, and secondary and tertiary amines ranged from 0.03 to 0.09%.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in transmittance mode in the 1100–2500 nm region. A 1mm cuvette was used to determine the optimum spectral region, and then a 4 mm cuvette was used to improve sensitivity towards that spectral region. A calibration for acetylables was developed at 2138 nm (SEC of 0.01%). A calibration for primary amines was also developed at 2138 nm (SEC of 0.01%). For secondary plus tertiary amines, a calibration was developed at 1864 nm (SEC of 0.006%).

Results

The results indicate the feasibility and stability of the NIR technique. More detail on this system would aid in selection of analytical wavelengths for calibration development, rather than allowing the computer to generate the wavelengths.

No. 60: Monitoring pigment in polyvinyl alcohol in water

Summary

The objective of this study was to monitor pigment in polyvinyl alcohol in water.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

Samples were scanned in the 1100–2500 nm region, in transmittance mode (pathlength of 1 mm). Excellent results were obtained for the analysis of aqueous polyvinyl alcohol solutions. The 1700 nm region shows a change in intensity with change in concentration.

Results

The results indicate that NIR can be used for monitoring aqueous polyvinyl alcohol solutions.

No. 61: Monitoring the presence of the monomer vinyl pyrrolidone in polyvinyl pyrrolidone

Summary

This NIR application was used to monitor the presence of the monomer vinyl pyrrolidone (VP) in polyvinyl pyrrolidone (PVP). Seven samples were analyzed: five samples of known VP concentration ranging from 100 to 1600 ppm, one PVP sample containing no VP, and one unknown.

System

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

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100 to 2500 nm region in transmission mode. A 4 mm pathlength quartz cuvette was utilized for analysis. A sharp band due to the VP exists at 1620 nm, however, a slight interference is present due to the positive lobe of the 1688 nm band. A calibration developed at 1612 nm yielded a SEC of 79.2 ppm. Correcting for the interference by using a linear summation (1612+1688 nm) yielded a SEC of 10.5 ppm.

Results

The results indicate that NIR can be used to monitor the monomer vinyl pyrrolidone in polyvinyl pyrrolidone.

No. 62: Monitoring the hydrolysis of polyvinyl acetate (PVAC) to polyvinyl alcohol (PVOH)

Summary

This study was aimed to monitor the presence of ammonia in vinyl pyrrolidone (VP). Eight VP samples were analyzed with ammonia content ranging from 0 to 100 ppm dissolved ammonia.

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	2.921.1110

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm range. Each sample was placed into a standard sample cup and analyzed. Percent hydrolysis was monitored at 2148 nm (SEC of 1%).

Results

The results indicate that NIR can be used to monitor the degree of hydrolysis of PVAC to PVOH. The material presented contained a wide particle size distribution, which affected the precision of the results obtained by NIR. Grinding the sample reduced the particle size distribution, and resulted in a lower error for each individual sample scan. The error can be reduced further by averaging spectra from the individual scans.

No. 63: Monitoring of carboxyl end groups in polybutylene terephthalate pellets

Summary

The levels of carboxyl groups in polybutylene terephthalate pellets can be monitored by NIR spectroscopy. Five samples were provided with carboxyl end groups ranging from 15.1 to 71.5.

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 using a coarse sample cell in the 1100 to 2500 nm range. Carboxyl end groups were monitored at 2030 nm (SEC of 5).

Results

The results indicate that NIR can be used to monitor the level of carboxyl end groups in polybutylene terephthalate pellets. The results were obtained using only five samples in the calibration sample set, and will improve as the number of samples used in the calibration are increased.

No. 64: Monitoring of carboxyl end group levels in polybutylene terephthalate pellets

Summary

This NIR application was used to monitor carboxyl end group levels in polybutylene terephthalate pellets. The samples for this study covered the range from 26 to 62 carboxyl numbers.

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 using a coarse sample cell. The spectral range was 1100 to 2500 nm. All samples were analyzed in reflectance mode. The low viscosity (MW) samples were monitored at 2030 nm (SEC of 2 was obtained for thirteen samples in the 26 to 60 range). The high viscosity (MW) samples were monitored at 2032 nm (SEC of 2 was obtained for nine samples in the 30 to 44.7 range).

Results

The results indicate that NIR can be used to measure carboxyl end group levels in polybutylene terephthalate. The quantitative determinations were actually performed on an absorption region due to the hydroxyl group (hydroxyl group level decreases with increasing carboxyl group level). Large changes in sample viscosity appeared to affect NIR results for carboxyl group level. This problem can be minimized by separating the samples into different viscosity ranges.

No. 65: Monitoring of carboxyl numbers in polybutylene terephthalate pellets

Summary

This NIR application was used to monitor carboxyl numbers in polybutylene terephthalate pellets. Seventeen samples were provided with carboxyl numbers ranging from 36.8 to 57.9.

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 using a coarse sample cell in reflectance mode. The spectral range was 1100 to 2500 nm. Two separate calibrations were developed. A calibration was developed at 2054 nm (SEC of 1 for 11 samples in range 36.8 to 41.8). A calibration was also developed for the remaining six samples at 2058 nm (SEC of 2 for 53.3 to 57.9 range). On older samples, a strong moisture absorption was found at 1910 nm. After heating for an hour, changes in this peak were seen (the moisture was picked up over time, but could be driven off by heating).

Results

The results indicate that NIR can be used to monitor carboxyl number in PBT pellets. Also, the amount of moisture picked up by this polymer can be monitored by NIR. These measurements can be performed simultaneously from one NIR measurement.

No. 66: Monitoring the levels of acrylate comonomer in a copolymer resin

Summary

This NIR application was used to monitor the levels of acrylate comonomer in an acrylate polyethylene resin.

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 spectral region was 1100–2500 nm. The pellets were analyzed in reflectance mode using a coarse sample cell. The 1942 nm band showed the highest correlation to the comonomer. A calibration was developed at this wavelength, however, the SEC was not reported.

Results

The results indicate that NIR can be used to detect various levels of the acrylate in an acrylate polyethylene copolymer.

No. 67: Monitoring blend composition in butadiene-styrene-acrylonitrile polymer resins

Summary

This application shows the use of NIR to monitor blend composition in butadiene-styrene-acrylonitrile polymer resins. Three samples were provided with butadiene level at 15, 35, and 50%.

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 powdered samples (polystyrene and polyacrylonitrile) were analyzed in reflectance mode in a standard sample cup, while the polybutadiene liquid was analyzed in transmittance. The scan range was 1100 to 2500 nm. A unique absorption for polybutadiene was found at 1720 nm. A calibration was developed on the three samples at 1722 nm (SEC of 0.2%).

Results

The results indicate that NIR can be used to find spectral regions unique to each component, although only three samples were provided. Therefore, it appears feasible to quantitatively measure the blend composition in butadiene-styrene-acrylonitrile resins.

No. 68: Monitoring viscosity during a phenolformaldehyde resin reaction

Summary

This NIR analysis was performed to monitor viscosity during a phenolformaldehyde resin reaction.

System

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

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510

Sampling

The samples were analyzed in the 1100–2500 nm region. The instrument was configured with a fiber optic bundle module and interactance probe (0.5 mm gap) attached to simulate on-line sampling procedures. A total of 25 samples from 3 batches were provided for calibration. The samples were maintained at a constant temperature of 5 °C in order to minimize further polymerization of the sample. The 1680 nm spectral region was used to monitor the phenol-formaldehyde reaction. A calibration was developed (SEC of 35 for range of 33 to 262). Through Step 13 of the reaction, SEC was reduced to 9 for range of 33 to 136.

Results

The results indicate that NIR can be used to monitor this particular phenol-formadehyde reaction. Due to the exponential characteristics of the reaction, it may be necessary to perform the analysis based on the natural logarithmic value of centistoke, rather than the actual centistoke value. If the process only requires monitoring through Step 13, then it appears to be possible to work with only the centistoke values.

No. 69: Monitoring the degree of cure of partially cured epoxy resins on woven glass (prepegs)

Summary

The objective of this study was to monitor the degree of cure of partially cured epoxy resins on woven glass (prepregs). The samples provided for this analysis were the untreated glass, the uncured resin, the totally cured laminate, and several prepregs partially cured to various degrees (30 to 120 for four calibration samples).

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 from 1100–2500 nm. The samples were simply cut into 1 inch x 7 inches sections, and placed into a coarse sample cell. A calibration was developed at 2210 nm (SEC of 6), a band unique to the resin, and absent in the totally cured material.

Results

The results indicate that NIR can be used to monitor the degree of cure in lightweight prepregs.

No. 70: Qualitatively distinguishing between good and bad lots of photosensitive diazo resin

Summary

This NIR application was used to qualitatively distinguish between good and bad lots of photosensitive diazo resin. Two batches were received for analysis.

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 samples were analyzed in a dark room to avoid damage to the light-sensitive resins. The two batches can be distinguished from one another, mostly in the water regions. There appears to be a particle size difference between the two batches.

Results

The results indicate that NIR can be used to qualitatively distinguish between the diazo resin samples. The next step involves the development of a library using representative samples from the target population.

No. 71: Determining the relative amounts of water in an acrylic resin throughout a three step drying process

Summary

This NIR application shows the use of NIR spectroscopy for monitoring the degree of cure of partially cured epoxy resins on woven glass (prepregs). The samples provided for this analysis were the untreated glass, the uncured resin, the totally cured laminate, and several prepregs partially cured to various degrees (30 to 120 for four calibration samples).

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 Process Analyzer MicroBundle SinglePoint	2.928.0110

Sampling

The samples were analyzed in reflectance mode from 1100–2500 nm. The samples were simply cut into 1 inch x 7 inches sections, and placed into a coarse sample cell. A calibration was developed at 2210 nm (SEC of 6), a band unique to the resin, and absent in the totally cured material.

Results

The results indicate that NIR can be used to monitor the degree of cure in lightweight prepregs.

No. 72: Monitoring hydroxyl groups in alkyd resin

Summary

This NIR application was used to monitor hydroxyl groups in alkyd resin samples. Ten samples were provided with hydroxyl values ranging from 7.81 to 38.34.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

All samples were analyzed in transmittance mode in the 1100 to 2500 nm region. The samples were placed in 4 mm pathlength cuvettes. A calibration for OH groups was developed at 2084 nm (SEC of 0.4 OH number).

Results

The results indicate that NIR can be used to monitor hydroxyl concentration in alkyd resins.

No. 73: Quantitative determination of a copolymer resin (mixture of octylacrylamide, acrylates, and butyl-aminoethylmethacrylate copolymer) in various types of hairspray

Summary

NIR spectroscopy was used for determining the amount of copolymer resin (mixture of octylacrylamide, acrylates, and butylaminoethylmethacrylate copolymer) in hairspray. The samples ranged from 0.9 to 7.54% copolymer resin in ethanol and water (these samples were made in the lab).

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100–2500 nm range in transmission mode using a 2 mm cuvette. A calibration was developed at 2242 nm where water exhibits no spectral features, but the resin has an absorbance band.

Results

The results indicate that NIR can be used to monitor the copolymer resin.

No. 74: Monitoring percent silicone in polycarbonate

Summary

This NIR application was used to monitor percent silicone in polycarbonate samples. Six samples were analyzed with silicone content ranging from 0 to 10%.

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 to 2500 nm spectral range using the coarse sample cell. The samples were analyzed in reflectance mode. Although the pure silicone component was not analyzed, two spectral bands were identified, 1740 and 1850 nm, where the polycarbonate did not pose an interference. A calibration developed at 1742 nm yielded a SEC of 0.2%.

Results

The results indicate that NIR can be used to measure silicone content in polycarbonate samples.

No. 75: Monitoring the adhesion properties of adhesives on silicone-coated liners

Summary

This NIR application was used for monitoring the adhesion properties of adhesives on silicone-coated liners.

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 NIR spectra were measured in reflectance in the 1100–2500 nm region. The top release liner was removed from the samples, and then placed into a coarse sample cell, adhesive side forward. The first group of samples was analyzed for the effect of residual solvent in the adhesive (by varying the drying times, and temperatures of drying). The second group of samples was studied to monitor the degree of cross-linking. A calibration was developed at 2302 nm (SEC of 0.1% and range of 1.99 to 3.88%).

Results

The results indicate that NIR can be used to monitor the amount of residual solvent, and the degree of cross-linking induced by temperature in adhesives. In addition, the ability to monitor adhesion values, as affected by the extent of cross-linking was demonstrated quantitatively. It should be possible to monitor the amount of residual solvent, and the degree of cross-linking simultaneously, using different regions of the NIR spectrum, so long as the thickness remains constant.

No. 76: Qualitatively distinguishing between various polymers

Summary

The study was aimed to use NIR spectroscopy to distinguish between ABS, Acrylic, PC, Styrene, Nylon, PVC, Polypropylene, Random, Acetal, Polyester, and HDPE. Several different samples of the eleven products were provided.

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 three times, reloading between scans. Only ten of the eleven samples provided were used for creation of the library (acetal was omitted because there was insufficient variation between the sample scans). Black samples were also omitted due to the lack of a spectrum (there were a total of eleven black samples). The library was created in the 1100–2200 nm region.

Results

The results indicate that NIR can be used to match all samples by distance (162 correct). NIR can be used to qualitatively distinguish between polymers.

No. 77: Monitoring plasticizer in a polymer film

Summary

This NIR application was used to monitor the amount of plasticizer in a polymer film. Three samples with known concentrations of plasticizer were used to establish a calibration. The plasticizer ranged in concentration from 34.4 to 41.2%. An additional sample, whose plasticizer concentration was unknown, was used to test the calibration.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode. A calibration for plasticizer concentration was performed at 2096 nm yielding a SEC of 0.09%.

Results

The results indicate that NIR can be used to monitor plasticizer concentration in a polymer film. More samples should be analyzed to generate a robust calibration.

No. 78: Monitoring the monomer content on a polymer film

Summary

This study shows ability of NIR spectroscopy for monitoring the monomer content on a polymer film. Fourteen samples were analyzed in the range of 20% to 55% cured (monomer content).

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The film samples were analyzed in reflectance mode using a remote reflectance modular attachment in the 1100–2500 nm region. Each sample was analyzed four times, turning between scans. The calibration film samples were also analyzed in transmission mode using a fiber optic transmission pair module. Each sample was analyzed twice, again turning between scans. Using the remote reflectance, a calibration for monomer content was developed at 1656/1618 nm (SEC of 2%). The second wavelength was included to correct for variations in sample uniformity and thickness. With the transmission pair, a calibration was developed at 1660/1612 nm (SEC of 2%).

Results

The results indicate that NIR can be used to monitor the degree of cure (monomer content) in film samples. Either a remote reflectance unit or a transmission pair unit can be utilized to make the measurements.

No. 79: Monitoring degree of cure (monomer content) on a polymer film

Summary

NIR spectroscopy was applied for monitoring degree of cure (monomer content) on a polymer film. Powder and film samples were provided. 100% monomer, 100% polymer, and 40/60 monomer/polymer samples were also provided.

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 using the remote reflectance probe. The primary wavelength associated with the monomer was 2458 nm. A calibration developed at this wavelength yielded a SEC of 14.7% (range was not reported). To correct for film thickness, a second denominator wavelength was included in the model (1436 nm) which reduced the SEC to 6.5%.

Results

The results indicate that NIR can be used to monitor monomer content or polymer content in film samples.

No. 80: Monitoring protein within polymer membranes

Summary

NIR spectroscopy was used in this study to determine protein within polymer membranes.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

All spectra were recorded in the transmission mode in the 1100 to 2500 nm region. The NH region, 1980 to 2180 nm, was examined; however, the spectral differences are most likely associated with thickness rather than NH intensity. No calibrations were developed.

Results

The evident spectral differences are probably attributable to varying polymer thicknesses rather than N-H band intensity.

No. 81: Detecting erucamide on polymer plaques

Summary

This study shows that NIR spectroscopy can be used to detect erucamide on polymer plaques. Twelve samples were provided with erucamide levels ranging from 0.1 to 2%.

System

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

The equivalent and recommended instrument:

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The spectra were obtained in transmission mode, using the 1100–2500 nm region. A calibration equation was developed at 1996 nm (SEC of 0.2%) for erucamide on plaques. Due to variations in the thicknesses of the plaques, and therefore changes in pathlength, division by a wavelength was necessary. The divisor wavelength was 1336 nm (1996/1336, SEC of 0.08%).

Results

The results indicate that NIR can be used to determine erucamide on a polymer plaque. Also, the sensitivity of NIR to small changes in erucamide concentration was illustrated.

No. 82: Monitoring the level of PIB in various packaging materials

Summary

This NIR application was used to monitor the level of PIB in various packaging materials. The samples provided consisted of three different types of packaging material with levels of PIB ranging from 0% to 6.2%.

System

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

The equivalent and recommended instrument:

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

Single sheets of the packaging materials were placed into a coarse sample cell with additional glass windows to hold the material in place. All spectra were obtained in transmission mode using the spectral range from 1100 to 2500 nm. With only three samples, it is difficult to say that variations at 1730 nm are due to PIB or sample thickness.

Results

The results suggest that NIR can detect a trend in the spectra of the packaging materials. With more samples, it should be possible to monitor the presence of PIB.

No. 83: Monitoring butadiene, polycarbonate and butyl acrylate in polymer pellets

Summary

NIR spectroscopy was used in this study for monitoring butadiene (BD), polycarbonate (PC), and butyl acrylate (BA) in polymer pellets. Five samples were provided with BD concentration ranging from 0 to 16%, and BA concentration from 0% to 16%.

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

All samples were analyzed in reflectance mode in the 1100–2500 nm region. A coarse sample cell was utilized for analysis. A least-squares regression was performed at 2048 nm for BD (SEC of 1%). For PC, spectral differences can be seen in the 1660 nm region, but no regression was performed due to the small number of samples (3). A regression for BA was performed at 1606 nm (SEC of 0.2%).

Results

The results indicate that NIR can be used to measure the concentration of BD, PC, and BA in polymer pellets. Although a 'formal' feasibility study was not performed, the spectral changes seen with the changing constituent values indicate that little difficulty should be encountered when developing a calibration.

No. 84: Measuring antioxidant levels in polymer pellets

Summary

NIR spectroscopy was used in this study to monitor additive levels in polymer pellets. The three antioxidants were AO-1 (0.12–0.72%), AO-2 (0.24–0.72%), and AO-3 (0.534–0.734%). Also provided were the pure components of interest.

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 using a coarse sample cell. Each sample was analyzed a total of five times. An absorption due to AO-1 is evident at 2132 nm, with little interference from the other constituents. Calibration at this wavelength yielded a SEC of 0.04%. AO-2 displays an absorption at 2090 nm again without interference from the others. Calibration at 2090 nm yielded a SEC of 0.04%. Absorptions for AO-3 appear at 2156 and 2245 nm. A calibration was developed at 2156 nm for this constituent yielding a SEC of 0.03%.

Results

The results indicate that NIR can be used to monitor each of these constituents. More samples should be analyzed, covering the full range of concentrations, in order to determine the accuracy of this method.

No. 85: Monitoring styrene content in styrene/butadiene copolymer pellets

Summary

NIR spectroscopy was used to monitor styrene content in styrene/butadiene copolymer pellets. Twelve different samples were provided, of which six were approximately 44% styrene, and the rest were approximately 78% styrene. Also provided were pure polystyrene beads, while pure polybutadiene was available in the laboratory.

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 spectrum of polystyrene was collected in reflectance, in a coarse sample cell, and polybutadiene in transmission. The samples were scanned in the 1100 to 2500 nm region. Styrene absorption appears in the 1800–1850 nm region where butadiene has no significant absorptions. The 2050 nm band is attributable to butadiene without interference from styrene. A calibration was developed at 2042 nm.

Results

The results indicate that NIR can be used to determine styrene content in styrene/butadiene copolymer pellets.

No. 86: Distinguishing between good and bad polymer beads

Summary

This study shows the use of NIR spectroscopy to distinguish between good and bad polymer beads. Sixteen samples were analyzed: eight good beads and eight bad beads.

System

Model 5000, rapid content analyzer 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 to 2500 nm region. Spectral differences, as well as baseline variations were observed between the good and bad samples. Significant spectral differences were observed in the 1670, 1950, 2100, and 2180 nm regions.

Results

The results indicate that NIR can be used to distinguish between good and bad polymer beads.

No. 87: Monitoring vinyl acetate content in ethylene/vinyl acetate polymer pellets

Summary

This study shows a NIR application for monitoring vinyl acetate content in ethylene/vinyl acetate (EVA) polymer pellets. Twenty-two samples were provided ranging in VA concentration from 4.2 to 39.9%.

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 pellets were analyzed in a coarse sample cell in reflectance mode. The scan range was 1100–2500 nm. The VA concentration was monitored at 1670 nm (SEC of 3%). The relatively high SEC is due to the two different types of pellets in the calibration set, one group cylindrical, and the other oval. Separating the groups and developing calibrations reduced the SEC to 0.7%.

Results

The results indicate that NIR can be used to monitor vinyl acetate content in ethylene/vinyl acetate copolymers.

No. 88: Determining differences between good and bad nylon 66 polymer pellets

Summary

NIR spectroscopy was applied in this study to determine whether spectral differences exist between the good and bad batches of Nylon 66 polymer pellets. Nine samples were provided for analysis, and the identity of the good and bad batches were not revealed to us.

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 using a coarse sample cell in reflectance mode in the 1100–2500 nm region. Variations can be seen throughout the 1100–2200 nm region.

Results

The results indicate that NIR can be used to detect spectral differences among Nylon 66 pellet samples. The black samples displayed weak spectral features which may make detection between good and bad batches difficult.

No. 89: Monitoring dye ability in nylon tow

Summary

This study was aimed to evaluate NIR's ability to detect the dye ability in nylon tow.

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. Each sample was cut to about a 7 inch length, and placed in a coarse sample cell. The fibers were aligned in order to mimic the process where the online measurement would be made. Two scans of each were taken, and then averaged. The greatest variation in the nylon spectra occur in the 1980–2020 nm and 2330–2360 nm regions. The band near 2010 nm is assignable to carboxyl groups, which seem to decrease as nylon becomes more dyeable. The band near 2350 nm, which is a region near amine group absorptions, increases in intensity as the dye ability increases.

Results

The moiety responsible for dye ability must be present at fairly low levels since the spectra are quite similar, yet NIR seems to be sensitive enough to differentiate between low and high dye ability in nylon tow. The above two spectral regions should be useful in determining the dye ability in the on-line system.

No. 90: Monitoring hydroxyl level of polymer in an ethanol/water solution

Summary

This NIR application was used to determine the hydroxyl level of a PVA based polymer in an ethanol-water solution. Four solutions, at a concentration of 8% solids w/v, containing levels of (residual) polyvinyl alcohol from 18 to 20.5% were prepared and used in this study. These solutions were prepared from dry material.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in transmission mode in the 1100–2500 nm region. The pathlength was 4 mm. For PVA, a calibration was developed at 1764 nm (SEC of 0.3%).

Results

The results indicate that NIR can be used to monitor the level of residual OH in the PVA-based polymer in solution.

No. 91: Monitoring moisture and lubricant on filter tow

Summary

This NIR application was used to monitor moisture and lubricant on filter tow. The moisture ranged from 3.45 to 9.70%, while the lubricant ranged from 0.465 to 1.495%.

System

Process Analytics Model 5000 with interactance fibers and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle SinglePoint	2.928.0110

Sampling

The samples were analyzed using a process analytics instrument in the 1100–2500 nm region. The moisture region was monitored in the 1930 and 1420 nm regions (calibrations were developed). Calibrations for moisture were developed at 1930 and 1420 nm (each calibration yielded a SEC of 0.7%). PLS was used to monitor the lubricant on the filter tow (2 PLS factors, with a SEC of 0.2%).

Results

The results indicate that NIR can be used to monitor moisture and lubricant levels on filter tow.

No. 92: Monitoring moisture in nylon

Summary

This study shows a NIR application for monitoring moisture concentration in nylon. Thirty samples of nylon exhibiting a moisture concentration range of approximately 5–90% were provided for this study.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

NIR reflectance spectra of all samples were recorded from 400–1100 nm. Sample presentation consisted of placing the nylon sample into a 500 cm^3 jar and then inserting the probe into the jar so that it contacted the nylon. Each sample spectrum used in the linear regression analysis is actually the average of four spectra recorded at different sample positions. This was done to reduce sampling error which is usually the largest error component in the NIR analytical method. A calibration for moisture was developed at 976 nm (SEC of 0.7%).

Results

The results indicate that NIR can be used as an automated on-line NIR method for determination of moisture in nylon.

No. 93: Monitoring extractables in nylon 6 fibers

Summary

NIR spectroscopy was applied to monitor extractable in Nylon 6 fibers. Three extractable samples (2.15, 2.61, and 3.11%) were 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

All spectra were collected in reflectance mode in the 1100–2500 nm spectral region. Samples were placed into a standard sample cup in a random fashion and the spectra were averaged over four different orientations. The 1380 nm region shows changes in absorptions as extractables concentration increase. However, more samples are needed to assess this region.

Results

The results indicate that NIR can be used to provide a wealth of spectral information on such polymers. What is evident from the spectra is the complexity of the product and the need for a more detailed analysis, with more information on the chemical nature of the product being provided.

No. 94: Measuring solvent levels in a polymer/solvent mixture

Summary

This NIR application was used to measure solvent levels in a polymer/solvent mixture. Provided were the raw materials to prepare two sets of seven samples containing various levels of polymer, acetonitrile, methanol, and water.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. Four spectra were collected for each sample, and then the spectra were averaged. At 1708 nm, an increase in acetonitrile concentration is seen with increasing band intensity. At 2060 nm, methanol increases with increasing band intensity. Water can be monitored at 1456 nm.

Results

The results indicate that NIR can be used to distinguish and measure the solvent levels. The accuracy of the method will have to be determined with actual production samples analyzed by referee lab method.

No. 95: Monitoring percent resin and percent volatiles in staging materials

Summary

This application was aimed to use NIR process analyzer for monitoring percent resin and percent volatiles in staging materials. A total of 21 samples were collected for calibration, while an additional two samples were collected for validation. Resin concentration ranged from 51.3 to 54%, while volatiles ranged from 4.5 to 9.2%.

System

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

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle SinglePoint	2.928.0110

Sampling

The samples were analyzed in the 1100–2500 nm region using a process analytics remote reflectance system. A calibration for percent resin was developed using PLS in the 1600–2200 nm region. Three factors were found optimal to describe the system (SEC of 0.35%). A calibration model for percent volatiles was developed using PLS in the 1600–2200 nm region. Again, three factors were optimal to describe the system (SEC of 0.25%).

Results

The results indicate that NIR can be used to monitor percent resin and percent volatiles in the staging process.

No. 96: Monitoring polymer content in liquid samples

Summary

NIR spectroscopy was easily applied for monitoring the polymer content (measured as percent solids) in liquid samples. Eight samples were analyzed with polymer content ranging from 3 to 40% solids. Also analyzed was the pure monomer, methyl methacrylate.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and immersion probe was used for this application.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed using an interactance immersion probe set to a 4 mm pathlength. The entire spectral range, 400 to 2500 nm, was scanned. The 1170 and 1680 nm spectral regions were directly related to the percent solids in the samples. A calibration for monitoring percent solids was developed at 1678 nm (SEC of 0.4%).

Results

The results indicate that NIR can be used to monitor polymer content in liquid samples.

No. 97: Monitoring cis 1, 4 butadiene in rubber

Summary

This NIR application was used to monitor cis 1,4 butadiene in rubber samples. Ten samples were analyzed with cis values of 92.02 to 93.31%.

System

Model 6500, 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 400 to 2500 nm spectral range using a remote reflectance module. For analysis, each sample was placed on a sheet of teflon and the remote reflectance head was placed directly on the rubber. Each sample was analyzed five times. A PLS calibration was developed in the 700–1058 mn, 1150–1370 nm, 1440–1660 nm, 1755–2220 nm regions. Four factors were necessary yielding a SEC of 0.2%.

Results

The results indicate that NIR can be used to monitor cis 1,4 polybutadiene in rubber.

No. 98: Monitoring oil content in styrene butadiene rubber

Summary

This study was aimed to monitor oil content in styrene butadiene rubber samples using NIRSystems analyzer. Eight samples were analyzed with oil content ranging from 16.9 to 31.0%. The pure rubber and the pure oil were also analyzed.

System

Model 6500, remote reflectance module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Smart Probe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 400 to 2500 nm spectral range using a remote reflectance module. For analysis, each sample was placed on a sheet of teflon and the remote reflectance head was placed directly on the rubber. Each sample was analyzed five times. A PLS calibration was developed in the 700–1058 mn, 1150–1370 nm, 1440–1660 nm, 1755–2220 nm regions. Four factors were necessary yielding a SEC of 0.2%.

Results

The results indicate that NIR can be used to monitor cis 1,4 polybutadiene in rubber.

No. 99: Monitoring the level of water in blends

Summary

This NIR application was used to measure the level of water in blends. Five samples were provided with water levels from 0.32 to 5.99%

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The blends were placed into a 1 mm cuvette and were measured in transmittance in the 1100 to 2500 nm spectral region. Water was monitored at 1926 nm (SEC of 0.3%).

Results

The results indicate that NIR can be used to monitor water in blend samples.

No. 100: Monitoring irganox 1010 and irgafos 168 in a blend

Summary

This feasibility study was aimed to use NIR application for monitoring a blend of Irganox 1010 and Irgafos 168. Eleven samples were used for calibration purposes with the additives ranging from 0 to 100%.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100 to 2500 nm region. Each sample was analyzed two times using the interactance reflectance probe. A unique band due to Irgafos 168 was located at 1782 nm. A calibration at this wavelength yielded a SEC of 3%. Although this band could also be used to monitor Irganox 1010 content, a calibration was developed at 2114 nm (a band unique to Irganox 1010). A SEC of 3% was obtained for this calibration model.

Results

The results indicate that NIR can be used to monitor blend composition. The blend was composed of Irganox 1010 and Irgafos 168.

No. 101: Monitoring vinyl acetate content in an ethylene-vinyl acetate copolymer

Summary

This NIR application was used for determination of vinyl acetate (VA) content (0–40%) in an ethylene-vinyl acetate (EVA) copolymer. Twenty-five samples were provided.

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

All spectra were collected in reflectance mode in the 1100–2500 nm region. Samples were analyzed in a coarse sample cell. Each sample was reloaded and scanned four times. A calibration was performed at 2146 nm with a SEC of 0.7%.

Results

The results indicate that NIR can be used to measure vinyl acetate content in an ethylene-vinyl acetate copolymer.

No. 102: Monitoring five components simultaneously in a polymer blend

Summary

The objective of this feasibility study was to monitor polyvinyl chloride (PVC), diisononyl phthalate plasticizer (DINP), epoxidized soybean oil plasticizer (ESO), a barium/cadmium stabilizer, and stearic acid in polymer blend. Five samples were provided for analysis, along with the pure components. PVC concentration ranged from 82.3 to 94.1%, DINP ranged from 40.9 to 51%, ESO ranged from 5.5 to 7.7%, and the stabilizer ranged from 4.6 to 12.2%.

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 using a micro sample cup. Two calibration for PVC and DINP were developed at 1786 nm (SEC of 1%) and at 1956 nm (SEC of 1%) respectively. 1232 nm was added as a divisor wavelength for pathlength correction (SEC of 0.4%). Three more calibrations for ESO, the stabilizer and stearic acid were performed at 1908 nm (SEC 0.2%) and at 1768 nm (SEC of 0.4%) at 1540 nm (SEC of 0.4%) respectively.

Results

The results indicate that NIR can be used to measure the five constituents of interest. The problem arose from the small differences in constituent concentration in these samples. The smallest difference is 0.01%. NIR cannot detect this small difference.

No. 103: Quantitatively determining the amount of polyamine in adhesive coating

Summary

This study used NIR technology to determine whether the particle size of the polyamine has an effect on its quantitative determination. Seven samples were received: one sample of the pure base resin, and two samples each of 2, 4, and 6% polyamine with different particle sizes.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. A fiber optic probe was used for analysis. A calibration was developed at 1984 nm. A SEC of 0.1% was obtained.

Results

The results indicate that NIR can be used to monitor different particle sized polyamine in adhesives.

No. 104: Determining the hydroxyl numbers of ethoxylated lauryl alcohols

Summary

This study was aimed to show ability of NIR spectroscopy to determine if NIR could be used to determine the hydroxyl numbers of ethoxylated lauryl alcohols. The hydroxyl numbers (OH number) ranged from 172.3 to 233.9. Nine samples were provided for this study, in addition to four unknowns.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

This study was performed in transmission mode in the 1100 to 2500 nm region. The only sample preparation involved warming the samples to 30 °Celsius to insure complete dissolution of the lauryl alcohols. A calibration model was developed at 2080 nm. A standard error of 1 OH number was obtained.

Results

The results indicate that NIR can be used to determine hydroxyl numbers in ethoxylated lauryl alcohols. This report has also demonstrated the precision obtainable with this particular method of analysis.

No. 105: Distinguishing glass bottles, identifying plastic bottles, and identifying plastic films and their thicknesses

Summary

There are three parts to this study: study of two types of glass bottles to distinguish between them, the study of plastic bottles to identify the base polymer as high density polyethylene, and the study of plastic films to identify each film and the thickness of the film.

System

Model 5000, transmission detector module, fiber optic bundle 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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed using an interactance reflectance fiber optic probe in the 1100–2500 nm region. All of the samples were analyzed five time each, moving the probe between scans. Little signal was obtained from the glass samples. The plastic bottles were compared to the already existing polymer library. Different thicknesses in the plastic films are evident in the form of intensity differences around 1650 nm.

Results

Due to little signal from the glass bottles, it is unlikely that NIR can be used to distinguish between the two types. However, it appears that NIR can be used for both polymer applications. The different thicknesses of the plastic film samples can be seen through the peak intensity differences in the NIR spectrum, and identification of the polymer is also feasible.

No. 106: Monitoring acid value and hydroxyl number in the process

Summary

This study used NIR technology to monitor acid value (AV) and hydroxyl number (OH number) in the process environment. Two batch reactor processes were studied. Sixty-five samples were collected for AV calibration, while 37 were used for OH number calibration. The AV range was 34 to 0.7, while the OH number range was 60 to 10.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission. A 12 inch interactance immersion probe set to a 6 mm pathlength was used for analysis. The temperature of the reactor was approximately 220 °C. For AV, a MLR calibration was performed at 1898+1444 nm (SEC of 0.3). The 1444 nm wavelength was introduced into the calibration to correct for interference from moisture and ROH. For OH number, a calibration was developed at 1420 nm (SEC of 1).

Results

The results indicate that NIR can be used to monitor acid value and hydroxyl number in the process environment. It was also demonstrated that a calibration could be developed which was independent of batch reactor for both AV and OH number.

No. 107: Monitoring the quantity of copolymer present in maleic anhydride activated well plates

Summary

This study shows the NIR application used to monitor the quantity of copolymer present in maleic anhydride activated well plates. Provided was a solution (10 mg/ml) of the polymer in DMSO (dimethyl sulfoxide), along with some well plates.

System

Model 5000, transmission fibers and probe pair was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

All were recorded between 1100 to 2500 nm using a fiber optic transmission pair. The gap between source and detector probes was 1 inch. A least-squares regression was performed at 1870 nm (SEC of 0.4% five samples in the range of 10 to 30%).

Results

The results indicate that NIR can be used to measure the quantity of polymer present in treated well plates.

No. 108: Distinguishing the different degrees of cure for two different epoxy/glass prepeg samples

Summary

NIR spectroscopy was applied to distinguish the different degrees of cure for two different epoxy/glass prepeg samples. Three samples were provided: the two different cure samples and one sample of the pure glass fabric.

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 reflectance mode in the 1100–2500 nm region. The spectral band at 2015 nm shows a shift with the degree of cure. No calibrations were developed.

Results

The results indicate that NIR can be used to locate spectral differences between epoxy/glass samples which differ only in the degree of cure. It is likely that a calibration equation could be developed which correlates the primary analysis data with the spectral differences.

No. 109: Monitoring the levels of polylactic acid (PLA) and lactic acid (LA) in reaction mixtures

Summary

This application shows a NIR application for determining the levels of (poly) lactic acid (PLA) and the starting compound (LA) in reaction mixtures. Twelve samples were provided with PLA concentration ranging from 71.5 to 96.

System

Process Analytics Model 5000, interactance fibers with immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle SinglePoint	2.928.0110

Sampling

A one-liter, three-holed round bottom flask was used for the controlled reaction. The LA was first melted before scans were taken. The reaction was monitored using a fiber optic (bundle) probe with a nominal pathlength of 4 mm. The samples were scanned in the 1100–2500 nm region in transmission mode. Numerous differences are apparent around 1700, 1900, and 2120 nm. A calibration was developed for PLA at 1684 nm (SEC of 2 for 12 samples in the 71.5 to 96 range).

Results

The results of this reaction study seem to indicate that it is feasible to monitor the reaction.

No. 110: Monitoring the degree of cure on prepeg samples

Summary

This NIR application was used for measuring the degree of cure on prepregs. Also, the optimum sample presentation mode (reflectance or transmission) was determined.

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

Scans of prepregs with several different gel times were scanned in the 1100–2500 nm region. The samples were placed into a coarse sample cell, either taped into position for transmission measurements, or backed with an aluminum sheet for reflectance measurements. A calibration was developed at 2210 nm (SEC of 1 for five samples) for gel time in thinner prepegs. For the thicker prepegs, the 1650 nm region gives the best correlation. A calibration was developed at 1652 nm (SEC of 8). A PLS equation was developed containing both the thin and thick prepegs.

Results

The results indicate that NIR can be used to monitor the degree of cure (determined as gel time). The results show that the measurement is preferably made in reflectance, using a reflective backing to obtain the optimum signal and better precision than obtained by transmission. The results from the multilinear calibrations suggest that the changes due to cure are too subtle to be adequately modeled by linear calibration methods. A multilinear regression was used to better characterize the subtle spectral variations due to curing, and improved the results.

No. 111: Monitoring the concentration of a polymer intermediate and moisture in a feed reactor

Summary

NIR spectroscopy was used in this study to monitor the concentration of a polymer intermediate and moisture in a feed reactor. Actual production samples were used for this study with a concentration range of 2.64 to 4.29% for the polymer intermediate and 0.95 to 1.35% for moisture.

System

Process Analytics Model 5000, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle SinglePoint	2.928.0110

Sampling

The samples were analyzed using a process analytics instrument in the 1100–2500 nm region. An interactance immersion probe was used for analysis. The polymer intermediate exhibits an absorption at 2060 nm with no interference from the other constituents. A calibration was developed at 2058 nm (SEC of 0.2%). For moisture, a calibration model was developed at 1924 nm (SEC of 0.04%).

Results

The results indicate that NIR can be used to monitor a polymer intermediate and moisture in a feed reactor.

No. 112: Monitoring plasticizer in a polymer film

Summary

This NIR application was used to monitor the amount of plasticizer in a polymer film. Three samples with known concentrations of plasticizer were used to establish a calibration. The plasticizer ranged in concentration from 34.4 to 41.2%. An additional sample, whose plasticizer concentration was unknown, was used to test the calibration.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode. A calibration for plasticizer concentration was performed at 2096 nm yielding a SEC of 0.09%.

Results

The results indicate that NIR can be used to monitor plasticizer concentration in a polymer film. More samples should be analyzed to generate a robust calibration.

No. 113: Monitoring a melamine reaction and determining moisture in a melamine mix

Summary

NIR spectroscopy was applied for monitoring the endpoint of a melamine reaction. Also, to detect moisture in a melamine mix.

System

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

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510

Sampling

The melamine reaction was analyzed using an interactance immersion fiber optic probe, while the melamine mix samples were analyzed using an interactance reflectance probe. All samples were analyzed in the 1100 to 2500 nm spectral range. For the melamine reaction, spectral changes were observed as the reaction proceeded. IQ2 was then used to identify when the specific endpoint of the reaction was reached. Moisture in the melamine mixes were easily identified in the 1400 and 1900 nm spectral regions.

Results

The results indicate that NIR can be used to monitor a melamine reaction as well as detect moisture in melamine mixes.

No. 114: Monitoring the thickness of the silicone layer on silicon solar cells

Summary

This NIR application was used to monitor the silicone layer of silicon solar cells. Thirty-six samples were analyzed with thickness of the silicone layer ranging from 0.3 to 2.0 mil.

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 SmartProbe Analyzer 2m Fiber	2.921.1610

Sampling

The samples were analyzed in the 1100 to 2500 nm region using an interactance reflectance sampling probe.

The absorption around 1696 nm was used to monitor the silicone thickness. A calibration developed at this wavelength yielded a SEC of 0.3 mil.

Results

The results indicate that NIR can be used to monitor the thickness of the silicone layer on silicon solar cells.

No. 115: Monitoring phenolic resin in wood fiber board

Summary

This study shows a NIR application for monitoring phenolic resin in wood fiber board samples. Eight solid wood fiber board samples were analyzed with phenolic resin content ranging from 0 to 10%. Also analyzed was the pure phenolic resin.

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	2.921.1110

Sampling

The samples were analyzed in the 1100 to 2500 nm spectral region in reflectance mode. A remote reflectance sampling module was used for analysis. Each sample was analyzed four times, repositioning the probe between scans. Unique spectral bands attributable to the phenolic resin were identified at 1690, 1758, and 2164 nm. A calibration was developed at 1690 nm (SEC of 0.4%).

Results

The results indicate that NIR can be used to monitor phenolic resin content in wood fiber boards.

No. 116: Monitoring the degree of cure of a binder on a fiberglass mat

Summary

NIR spectroscopy was applied to monitor the curing of a binder on a Fiberglass mat. The samples sent in for evaluation were Fiberglass mats that had been cured at different temperatures for the same length of time for five samples in the range of 325 to 475.

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 cut into 1 inch by 7 inches strips to fit into a coarse sample cell. The samples were analyzed in reflectance mode in the 1100 to 2500 nm range. The 2028 nm band was used to develop a calibration model (SEC of 14). This band was seen to decrease in intensity with cure temperature.

Results

The results indicate that NIR can be used to monitor the degree of cure of a binder directly on a Fiberglass mat. The wavelengths used for this calibration were chosen to allow the use of fiber optics.

No. 117: Determining cure times in fiberglass samples

Summary

This NIR application was used to determine cure times for five fiberglass samples.

System

Model 5000, reflectance detector module, sample transport mechanism, transmission detector 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

All NIR absorbance spectra were recorded from 1100–2500 nm in both transmittance and reflectance mode. Samples were placed in the coarse sample cell. A thorough evaluation of both modes revealed that the transmittance spectra represented the total composition of each sample whereas, the reflectance spectra showed large spectral differences due to the physical differences of each side. In the 2180 to 2200 nm region, a correlation between the NIR spectra and cure time could be seen.

Results

The results indicate that NIR can be used to differentiate between various cure times. What is evident from the spectra is the complexity of the product and the need for a more detailed analysis, with more information on the chemical nature of the products being provided.

No. 118: Monitoring the degree of cure of resin-coated fiberglass

Summary

NIR spectroscopy was used to monitor the degree of cure of resin-coated fiberglass. The samples sent for this analysis were resin treated fiberglass mats which were heated for a set period of time at different temperatures, ranging from 126 °C to 226 °C. Two different sets of samples were evaluated, one with an added dye and one with no dye added. In addition, uncured samples were also supplied.

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 cut into 7 inches by 1.5 inch strips to fit in the coarse sample cell. The analysis was performed in reflectance in the scan range of 1100–2500nm. For the dyed samples, a calibration was developed at 2362 nm (SEC of -9 °C). For the undyed samples a calibration was also developed at 2362 nm (SEC of -11 °C). The major differences seen at this wavelength are attributable to changes that occur in the resin upon curing.

Results

The results indicate that NIR can be used to identify spectral differences that appear upon curing resin on fiberglass. In addition, the degree of curing, measured as the temperature at which the materials were cured, can be quantitatively determined by NIR.

No. 119: Monitoring amide levels in erucamide

Summary

This NIR application was used to monitor amide levels in erucamide.

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 measured as is using a standard sample cup. The samples were measured six times, with one reload and three rotations for each loading. The samples were analyzed in reflectance mode in the 1100 to 2500 nm region. A calibration for amide was developed at 2030 nm yielding a SEC of 0.9% (range not reported). Another calibration was developed at 1562 nm (SEC of 1%). This calibration can be used for the on-line analysis of erucamide.

Results

The results indicate that NIR can be used to monitor amide percent in erucamide samples.

No. 120: Determining the thickness of each layer of nylon and polyethylene terephthalate (PET) in a multi-layer bottle

Summary

This study was aimed to evaluate ability of NIR spectroscopy to determine the thickness of each layer in a multi-layer bottle. Two bottles, one comprised of a thin layer of nylon sandwiched by two thicker layers of polyethylene terephthalate (PET) and another with an extra layer of PET, were obtained for this study.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The spectra were analyzed in transmission mode in the 1100 to 2500 nm region. Qualitative determination of the absence of any individual layer can be easily accomplished by means of a spectral matching algorithm. The spectra of PET and nylon are quite distinct from one another, especially in the spectral regions near 2050, 2170, and 2290 nm.

Results

The results indicate that NIR can be used to perform qualitative and quantitative analysis. Qualitative analysis for the absence of any individual layer can easily be accomplished using the spectral matching algorithm.

No. 121: Determination of polymer coating levels on glass

Summary

This NIR application was used for quantitative analysis of the amount of a coating, polyethylene wax, on glass. Five glass samples were received for analysis. The samples ranged from zero coatings to four coatings.

System

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

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

The glass samples were analyzed in transmission mode in the 1100–2500 nm region. A calibration was developed at 2346 nm for the polyethylene wax. This wavelength is free from interference from the glass.

Results

The results indicate that NIR can be used to quantitate the amount of coating on glass.

No. 122: Detection of the presence of coating treatment on glass samples

Summary

NIR spectroscopy was applied in this study to detect the presence of coating treatment on glass samples. Class 1 and class 2 bottles were provided in both flint and amber colors.

System

Model 6500, transmission detector module, transmission fibers and probe pairs was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520

Sampling

Samples were analyzed in transmission mode in the 1100–2500 nm region. A pair of transmission probes was used for analysis. A calibration for the six flint glass bottles (class 1) was developed at 2356 nm (SEC of 0.2 for range of 0 to 4). For both the class 1 and class 2 bottles, a calibration was developed at 1762 nm. Finally, a calibration was developed for both the flint and amber bottles at 2300/1408 nm. The denominator term accounts for pathlength variations caused by bottle size and wall thickness.

Results

The results indicate that NIR can be used to quantitatively determine the number of coats applied to whole glass bottles. The accuracy of the calibration depends upon whether the equation is set up to determine a specific set of bottles (a SEC of 0.168 is then attainable) or if the equation is set up to determine the number of coatings on all bottles of various sizes, colors, and wall thicknesses (a SEC of 0.200 is attainable).

No. 123: Qualitative comparison of nonwoven polymer cloths

Summary

This study shows the NIR application to perform qualitative comparisons of nonwoven polymer cloths.

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 cloth samples were cut into strips of approximately 70 cm^2 in area and placed in a coarse sample cell. The samples were analyzed in reflectance mode. It is apparent that the cloth samples are quite different. A spectral matching algorithm was used to compare the samples.

Results

The results indicate that NIR can be used to differentiate between different nonwoven cloth materials. Differences in the binder also can be detected, as well as difference in minor components within a single type of cloth.