Metrohm NIRSystems

Application Bulletin 412

Analysis of pulp and paper using near-infrared spectroscopy

Branch

Pulp and paper

Keywords

Near-infrared spectroscopy, paper, pulp, moisture determination, kappa number, resin content.

Summary

This Application Bulletin shows the examples of NIR applications which can be used to determine important parameters of pulp and paper quality. Each application briefly describes the measuring systems used in the studies as well as the recommended instruments and the test results.

Introduction

NIR analysis offers the time saved over the current wet method. The results can be obtained in more rapid turnaround of batches, more accurate determinations, and in time, a better product at a reduced cost. In the pulp and paper industry, NIR applications provide qualitative and quantitative information about incoming timber materials and lignin content. Discriminant NIR analysis can be used to determine species, hardwoods from softwoods, and sapwoods from heartwoods. Common paper and pulp attributes measured with NIR include: kappa number, lignin content, kraft pulp yield, tall oil, moisture, resin, brightness, wood species, hardwood/softwood ratio, coatings, and component analysis (clay, titanium dioxide, fillers, ash, etc.).

Contents

No. 1: Determining the percentage of softwood in pulp samples

Summary

This application shows that NIR spectroscopy can be applied to determine the percentage of softwood in pulp samples. Two sets of samples were provided and treated independently to provide calibrations for both lab (round) and production (square) samples.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in transmission mode using a 2 mm pathlength cuvette. The 1100 to 2500 nm range was scanned. A calibration for moisture was developed at 1960 nm (SEC 0.1%).

Results

The results indicate that NIR can be used to determine moisture levels in ethylene glycol to better than +/- 0.2%.

No. 2: Determining kappa number in blended wood pulp samples

Summary

This NIR application is used to determine kappa number in blended wood pulp samples. Along with the samples, pure lignin, cellulose, and six unknowns were provided. Twenty-seven spectra were used for the calibration set and nine for the validation set.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were placed in a standard sample cup and analyzed in reflectance mode. Each sample was divided into three pieces and labelled A, B, C. A regression was performed at 2180 nm (correlation to the phenolic hydroxyl group present in lignin). A standard error of 5.6 was obtained. A second wavelength, 1548 nm, was introduced into the calibration causing only a slight decrease in the standard error to 5.2.

Results

The results indicate that NIR can be used to monitor kappa number in pulp samples. The question remains whether the standard error is acceptable.

No. 3: Monitoring kappa numbers in pulp-cotton linters mixtures

Summary

NIR Spectroscopy is applied to monitor kappa numbers in pulp-cotton linter mixtures. Samples that ranged in the kappa number from 3.4 to 33.9 were provided.

The 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 analysis in the sample transport mechanism. A least-squares regression was performed at 1680 nm (SEC of 1.2 kappa numbers).

Results

The results indicate that NIR can be used to monitor the kappa number in both wet and dry wood pulp-cotton linter mixtures.

No. 4: Determining K No. (an estimation of kappa number) in pulp blowline samples

Summary

This study shows that NIR spectroscopy can be applied to monitor K no. in pulp blowline samples. K no. is an estimation of kappa number (logarithmically related to kappa number).

The 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 spinning sample module was used for analysis. Absorbance differences at approximately 1680 nm are due to variations in the lignin content. A least-squares regression was performed at 1672 nm (SEC of 1). However, the pathlength differences in the samples require a second wavelength to be included in the calibration. The 1436 nm wavelength was added as a divisor term (1672 nm/1436 nm, SEC of 0.5).

Results

The results indicate that NIR can be used to monitor K no. in pulp blowline samples. Further improvements are expected if a kappa number analysis was used as the reference method and the samples were pressed into sheets.

No. 5: Monitoring lignin in pressed wood pulp

Summary

This NIR application is used to monitor lignin in pressed wood pulp. The samples were separated into two groups, one group was used for calibration and the second group was used for validation. The lignin range was from 5.7 to 33.6%.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

All spectra were collected in the 1100–2500 nm region. All spectra were collected in reflectance mode using a standard sample cup. Samples were cut to size and inserted as a double layer to insure 'infinite' pathlength as reflectance requires. The cup was rotated between scans, and the five scans per sample were averaged. A two wavelength calibration equation was developed including absorption bands from both the lignin and the cellulose. The calibration was developed at 2172 + 1556 nm yielding a SEC of 1%.

Results

The results indicate that NIR can be used to analyze lignin in pressed wood pulp. It is recommended that a coarse sample cell be used for any off-line work.

No. 6: Monitoring wax and a phenol formaldehyde resin in wood fiber

Summary

This feasibility study presents the result of using NIR to determine whether wax and a phenol formaldehyde resin could be detected in wood fiber. Four samples were provided for this study: wood fiber, pure wax, freeze-dried phenol formaldehyde resin, and wood fiber with wax and phenol formaldehyde resin.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The sample spectra were collected in reflectance mode scanning from 1100–2500 nm. A sample cup was used for the wax and resin, while a coarse sample cell was used for the wood fiber samples. It appears that either 2250 or 2310 nm can be used to monitor the wax content in the fibers. It also appears that 1725 nm can be used to monitor the wax content of the material. For resin content, the 1980 nm region may be used to monitor this constituent.

Results

The results indicate that NIR can be used to monitor both the wax and resin content in the wood fiber product.

No. 7: Monitoring wax and phenolic resin content in wood fiber

Summary

This NIR application is used to monitor wax and phenolic resin content in wood fiber. The samples provided covered a range of wax from 0.3 to 2.4% and resin levels from 1.3 to 4.3%.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were measured in reflectance in the 1100–2500 nm region. The samples were simply placed into a standard sample cup prior to the NIR measurement. A calibration was developed at 2158 nm (SEC of 0.4%) for the resin. Wax content was monitored at 1728 nm (SEC of 0.1%).

Results

The results indicate that NIR can be used to determine quantitatively wax and resin in wood fiber.

No. 8: Monitoring the amount of super absorbent polymer in fluff pulp

Summary

This application shows that NIR can be used to determine the amount of super absorbent polymer in fluff pulp. Six samples (two pads at each concentration) were provided to demonstrate feasibility, with the concentration of the polymer in the samples ranged from 0.0 to 44.9%.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The 1100 to 2500 nm spectral region was used for this study. Each pad was analyzed in reflectance mode using the remote reflectance sampling attachment. A calibration for polymer was developed at 1752 nm (SEC of 6%). It was also seen at this wavelength that the level of polymer changed as the remote reflectance head was moved across the fluff pulp (the highest polymer concentration was in the middle).

Results

The results indicate that NIR can be used to monitor the distribution of super absorbent polymer in fluff pulp.

No. 9: Monitoring resin levels in paper

Summary

This feasibility study shows that NIR can be used to determine the resin level in paper.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were cut into 7 by 1.5 inches strips, so as to fit into a coarse sample cell. The samples were analyzed in the 1100–2500 nm region in reflectance mode. For the resin, three bands were found without interference from the paper at 1688, 1766, and 2160 nm. A calibration was developed at 2160 nm (SEC of 0.7%).

Results

The results indicate that NIR can be used to determine the resin level in the paper with high sensitivity. Since the uncoated paper shows no significant spectral variation, there should be little interference from the paper in determining resin content.

No. 10: Determining moisture in paper and lacquer weight on foil-backed paper

Summary

This study shows the use of NIR spectroscopy for determination of moisture in paper and lacquer weight on foil-backed paper.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were cut into strips of about 2 x 7 inches to fit in a coarse sample cell. A piece of foam was placed behind the sample which pressed the paper against the cell window in a reproducible fashion when the cell was closed. The samples were analyzed in reflectance mode. The greatest amount of variation with lacquer weight was seen in the 2100–2300 nm region. A least-squares regression was performed at 2346 nm.

Results

The results indicate that NIR is sensitive to both moisture and lacquer weight for paper. In this study, sample variations were averaged out by scanning over a large sample area.

No. 11: The determination of calcium propionate in paper

Summary

The feasibility study shows the positive result of using NIR analysis for determination of calcium propionate when sprayed on paper.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

Four samples of the calcium propionate sprayed samples were obtained at levels of 0, 500, 900, and 2150 ppm propionate. Samples were placed directly on the analyzer window and rotated 4 times each for a total of 16 spectra that were used for calibration equation development.

Results

A second derivative math pretreatment (segment = 10, gap = 0) was applied to the spectra. A simple linear regression between the derivative spectra and the reference values provided a correlation of 0.98 at wavelength 1758 nm with a Standard Error of Calibration (SEC) of 112.5 ppm.

No. 12: Monitoring coating levels on four different paper products

Summary

This NIR application is used to monitor coating levels on four different paper products.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were scanned in the 1100–2500 nm spectral region. The paper samples were analyzed using a remote reflectance accessory, which utilizes fiber optics to direct the NIR beam to the sample surface. The spectra of the slurry coatings were obtained using a transport mechanism. The spectra were collected in reflectance in a 20 mm pathlength cuvette. For monitoring the coating on the donor sheet, a calibration was developed at 2316 nm (SEC of 0.1% and range from 0 to 12%). The receiver sheet was also analyzed. The inorganic layer present in this sheet may be interference. Coatings on mylar film could be done at 2300 or 2350 nm.

Results

The results indicate that NIR can be used to monitor at least three of the applications shown in this study, concerning coatings on paper and Mylar. The experimental paper was not of infinite thickness for a reflectance measurement. Normally to correct for variable path lengths, division by a wavelength is done. In this case, the sample set was very small, therefore making this correction hard to do.

No. 13: Monitoring triacetin and moisture in cellulose acetate fiber rods

Summary

The study shows the result of monitoring triacetin and moisture in cellulose acetate fiber rods using NIR method. Samples included pure cellulose acetate and triacetin, along with samples of filter rods containing triacetin levels from 3.7 to 15.9%.

The 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 rods were simply placed into an elongated sample cell, which held about 30 rods when aligned in one direction. Two regions of interest for monitoring the triacetin appear at approximately 2360, and 1995 nm.

Results

The results indicate that NIR can be used to monitor triacetin and moisture content in cellulose acetate fiber rods.

No. 14: Monitoring levels of nitrocellulose in cellulose/nitrocellulose casings

Summary

NIR analysis is used to measure the levels of nitrocellulose in cellulose/nitrocellulose casing. Seven samples were provided in the 59.4 to 70.8 range.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

Spectra of the pure casing components were measured in reflectance from 1100 to 2500 nm. The pure component samples were in the form of a sheet which was placed in a circular sample cup for measurement. For the calibration, the casing samples were in the form of 1.5inches squares and were analyzed using a coarse sample cell. A calibration for nitrocellulose was developed at 2346 nm (SEC of 1). For samples with marbon resin, a calibration was developed at 2240 nm (SEC of 0.3).

Results

The results indicate that NIR can be used to quantitatively determine nitrocellulose in nitrocellulose/cellulose casings with and without added resin. This report also shows that NIR is also sensitive to the presence of cellulose and marbon resin, and quantitative determinations of these materials should also be possible.

No. 15: Monitoring percent asphalt in wet expansion joint mats

Summary

This NIR application is used to monitor the percent asphalt in wet expansion joint mats. Twenty samples ranging from 30.9 to 35.6% asphalt concentration were used for calibration. Ten additional unknown concentration samples were also provided.

The recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120

Sampling

The samples were analyzed in the 1100–2500 nm region in reflectance mode with a coarse sample cell. Each sample was analyzed four times, twice on each side of the sample. Since asphalt has no assignable spectral band, it is impractical to perform a single wavelength least-squares regression. Therefore, partial least-squares were utilized. The spectral regions 1150–1375, 1500–1875, and 2100–2300 nm were used for calibration development (to avoid the influence of moisture). Five PLS factors were used with a SEC of 0.9%.

Results

The results indicate that NIR can be used to perform quantitative analysis on expansion joint mats for percent asphalt.

No. 16: Monitoring moisture in paper coating mixtures

Summary

This NIR application is used to monitor moisture in polymers used in paper coating mixtures.

The recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. A coarse sample cell was utilized for analysis in the sample transport mechanism. A least-squares regression was performed at 1680nm (SEC of 1.2 kappa numbers).

Results

The results indicate that NIR can be used to monitor the kappa number in both wet and dry wood pulp-cotton linter mixtures.

No.17: Monitoring silicone level on tissue paper

Summary

The objective of this study was to determine whether the level of silicone on tissue paper could be monitored. A total of eight samples were provided with silicone concentration ranging from 0.69 to 5.67%.

The recommended instrument

NIRS XDS Process Analyzer DirectLight/NonContact	2.928.0310

Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. A coarse sample cell was utilized for analysis in the sample transport mechanism. A least-squares regression was performed at 1680 nm (SEC of 1.2 kappa numbers).

Results

The results indicate that NIR can be used to monitor the kappa number in both wet and dry wood pulp-cotton linter mixtures.