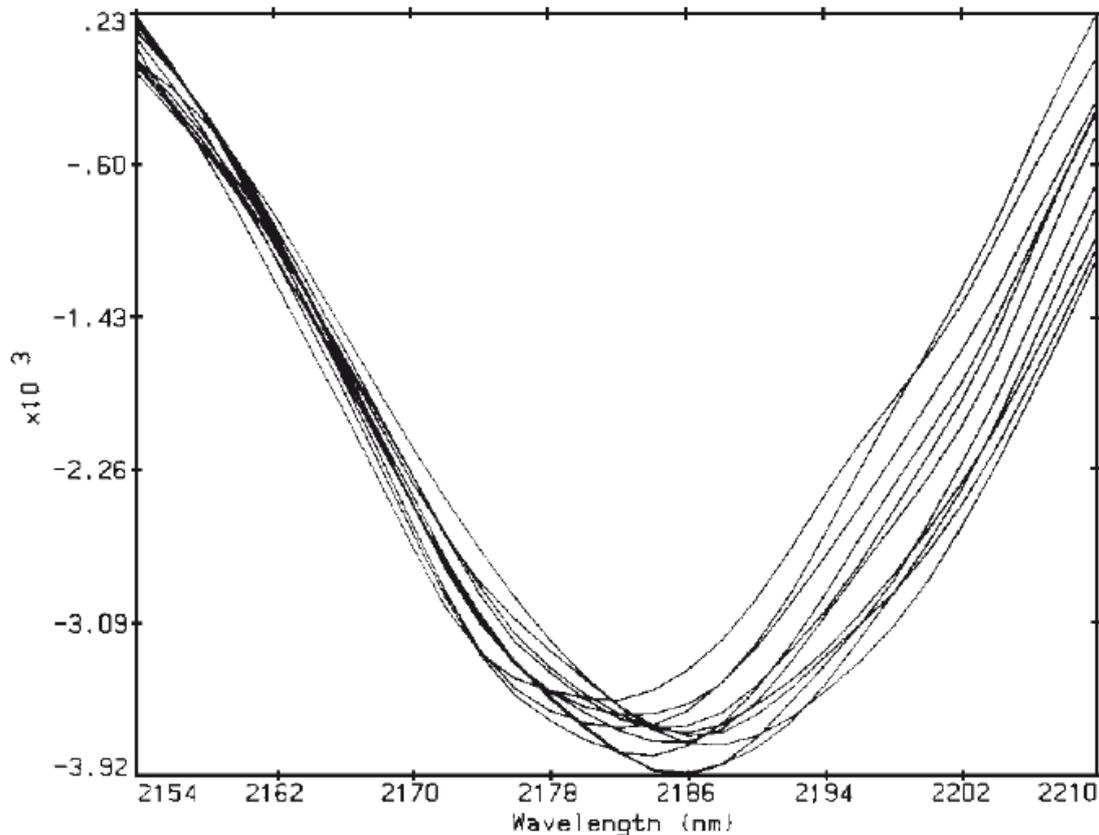# Analysis of lignin in wood pulp

This Application Note describes how NIR spectroscopy can be used to determine the residual lignin content in wood pulp. Using the major absorbance peaks of both lignin and cellulose in the second derivative spectra, a two-wavelength equation can be developed to monitor the residual lignin content in wood pulp during paper production.

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Method description

Introduction

The quality of paper produced by a paper mill is dependent upon numerous parameters: the type of stock used, the type of beaters, chemical treatments, the quality of the webbing, etc. Although it is impractical to measure all of the parameters to determine paper quality, it is possible to measure a few which would help to characterize the paper quality.

The amount of residual lignin is one such parameter which can be measured. Lignin has an influence on the texture and flexibility of the final product, so the lignin content of the pulp at any time is of great interest. Since the exact structure of this natural product is not clear (it is a mixture of natural polymers), the measurement is sometimes difficult. The wet chemical method is time consuming and can be erroneous if the moisture is not also taken into account. Therefore, two analytical measurements are needed under current methodology.

Using near-infrared (NIR) spectroscopy, a rapid, accurate method is developed. Using the major absorbance peaks of both lignin and cellulose, a two-wavelength equation was developed to follow the lignin concentration during a commercial run.

Experimental

All NIR spectra (log 1/R) were obtained on a Foss NIRSystems Model 6500 spectrophotometer. Since this instrument is not available anymore, the NIRS XDS RapidContent Analyzer is recommended. Spectra were collected in the 1100 nm to 2500 nm spectral region. Pulp samples were pressed into cakes between two pieces of white filter paper before submission to the lab. No special care was taken to reproduce the water content or smooth the surface of the sample. Due to this inhomogeneous surface, each sample was analyzed five times, rotating the sample cup between scans. The spectra were then averaged to give a more representative picture of each sample.

Results and discussion

Figure 1
Averaged Samples
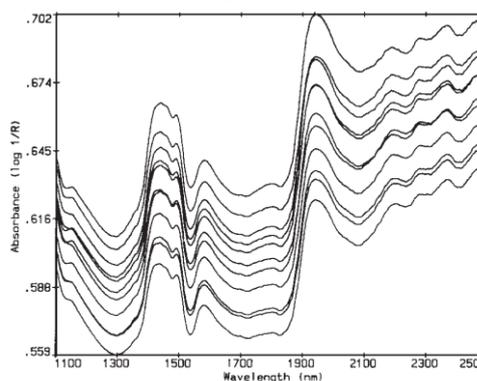
When reflectance measurements are taken, variability associated with background and matrix effects can create variations in the absorbance spectrum of a sample. Figure 1 shows the absorbance spectra of the samples.

The variations in the background suggest that averaging the five individual spectra would help to reduce the errors associated with the background variations.

The large baseline between samples indicates scattering differences which can be minimized by performing a second derivative calculation.

Figure 2
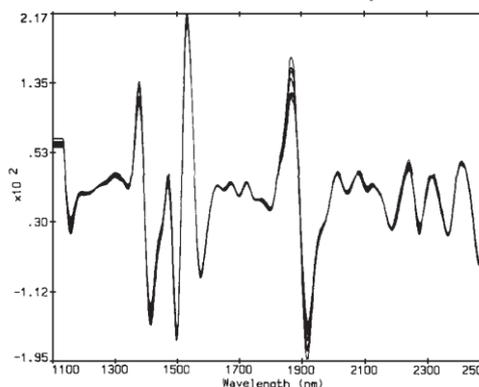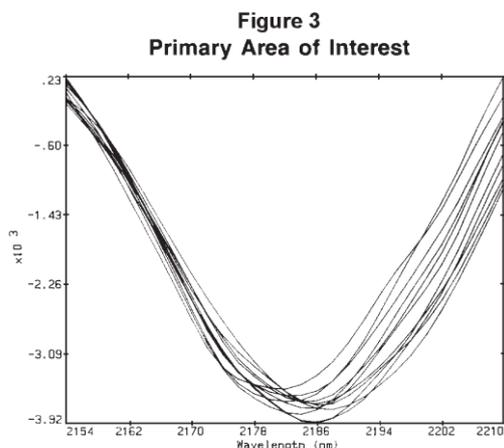**2nd Derivative of Samples**

Figure 2 shows the second derivative spectra. The chemical, not physical, differences are now the predominant features of the spectra. (Note: Absorbance peak maxima become 2nd derivative peak minima with positive lobes on either side.)

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Figure 3
Primary Area of Interest

While the exact structure of lignin is uncertain, there is known to be a phenolic hydroxyl group present. This function absorbs in the 2160-2180 nm region of the spectrum. An enlargement of this spectral region is shown in Figure 3.

A multilinear regression (MLR) was performed at 2172 and 1556 nm. The 1556 nm wavelength is attributed to cellulose. It is included in the model to reduce interferences due to the cellulose. A correlation (R) of 0.914 and a standard error of calibration (SEC) of 1.07% were obtained.

Conclusions

NIR spectrophotometry appears to be a sound method for the measurement of lignin in paper pulp. The time saved over the current wet method could result in more rapid turnaround of batches, more accurate determinations, and, in time, a better product at a reduced cost. One advantage of a rapid means of analysis is that realtime corrections may be made on the production line, avoiding over-reactions caused by the lag time between sampling and reporting of lab data.

Other applications in the paper industry which have been proven successful are hardwood/softwood ratios (see also AN-NIR-9), moisture, waxes, coatings, polymercellulose blends, and sizing to name a few.

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