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Rapid, Multi-Component Analysis of Soybeans by FT-NIR Spectroscopy

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Key Words

- Antaris
- Linolenic Acid
- NIR
- Oilseeds
- Sample Cup Spinner
- Soybeans

Introduction

Near-infrared (NIR) spectroscopy has greatly simplified and improved the speed of analysis for quality testing of soybeans. The use of near-infrared spectroscopy has led to higher sample throughput by replacing multiple time consuming and complex wet chemistry techniques that often require significant sample preparation. The fact that near-infrared can accurately and non-destructively analyze inhomogeneous samples without processing makes it a suitable technique at all stages of production: grading, milling, oil extraction and final product quality verification. The reflection analysis technique that was used in this study is applicable for testing all types of oilseeds for important components such as moisture, oil, protein and fatty acid profile.

This application study will show how the Thermo Scientific Antaris Fourier transform near-infrared (FT-NIR) analyzer with the Sample Cup Spinner accessory (Figure 1) was successfully used to analyze whole soybeans. The calibration models developed during this study prove that rapid, non-destructive near-infrared technology can accurately predict important components in soybeans.

Figure 1: Antaris FT-NIR analyzer with Sample Cup Spinner accessory for analyzing large, unprocessed samples

Experiment

A set of whole soybean samples from several crop years were used as standards to develop a partial least squares (PLS) calibration model for moisture, oil, protein and linolenic acid. A total of 207 calibration and 50 validation samples were used to build and validate the calibration model. For this study, standard spectra were collected on an Antaris™ II FT-NIR analyzer with a 12 cm Sample Cup Spinner accessory. The spectra were collected at 8 cm^{-1} resolution with 64 co-averaged scans in 30 seconds. Representative spectra collected for this study are shown in Figure 2. The Sample Cup Spinner accessory combined with the integrating sphere module inside the Antaris is the optimum technique for collecting diffusely reflected light over a large sampling area on bulk samples that are inhomogeneous in nature. Since the chemical information used to predict component concentrations is in the diffusely reflected light coming from the soybean samples, being able to collect an average, representative spectra over the bulk sample is critical to the analysis of whole soybeans. The use of this 12 cm Sample Cup Spinner eliminated the need to process or grind the sample prior to analysis. By contrast, grinding of soybean samples prior to primary testing is a necessity for accurate determination of component concentrations.

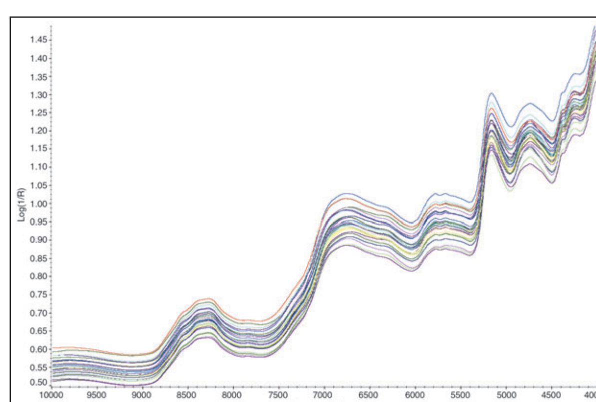

Figure 2: Representative standard spectra of whole soybeans used to develop PLS calibration models

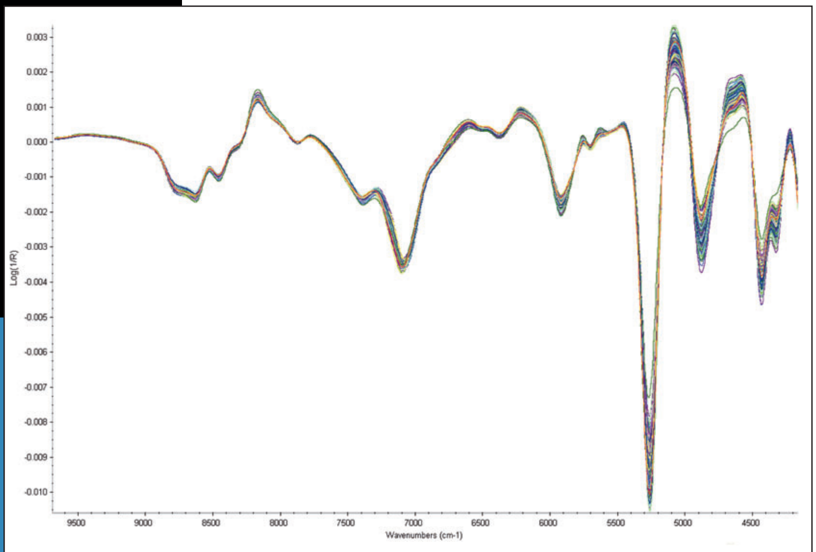

Figure 3: Representative 1st Derivative, Norris smoothed and SNV processed spectra used to develop and validate the calibration model

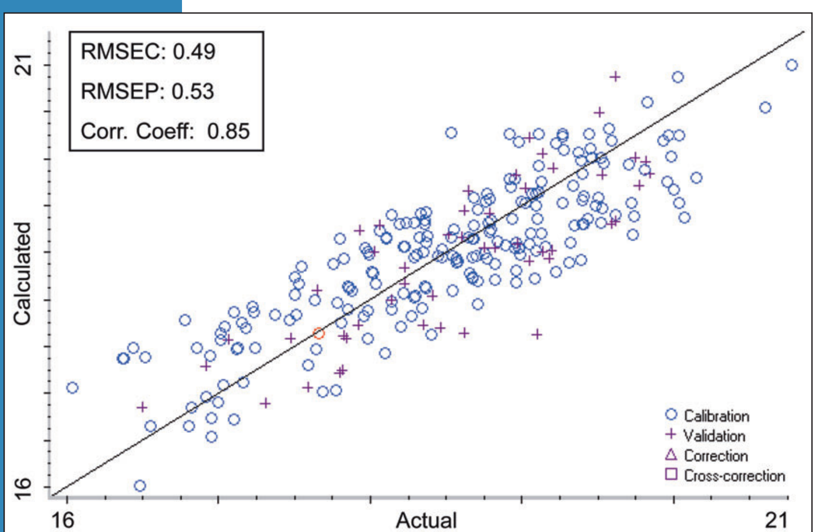

Figure 4: Oil calibration plot showing good correlation between Calculated (NIR) and Actual (Wet Chemistry) results

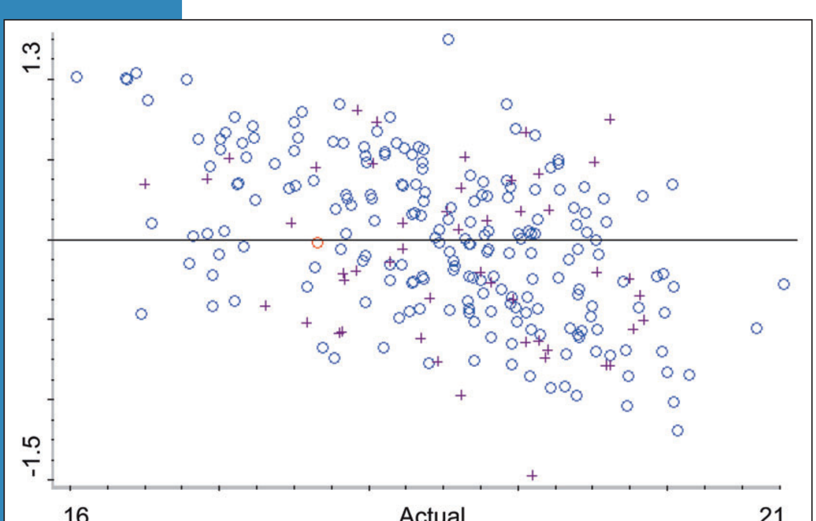

Figure 5: Oil residual (calculated value – actual value) vs. actual value plot showing similar errors in both calibration and validation samples across the whole component range

In order to enhance spectral features and lessen the baseline shifts seen in the raw spectra in Figure 2, 1st derivative with Norris smoothing and standard normal variate (SNV) processing were applied. The processing of the spectra prior to calibrating the method lessened the effect of the shifting and sloping baseline which is often seen in spectra from solid samples due to scattering of the light in the sample. Figure 3 shows the processed spectra over the spectral region used for developing the calibration model. It can be seen that the processed spectra reveal spectral differences in the sample set which were utilized for NIR calibration development. The spectral differences seen in the processed spectra were not apparent in the unprocessed spectra shown in Figure 2.

Results and Discussion

Near-infrared can be applied in multiple stages of the soybean milling process including: grading of incoming soybeans based on moisture and linolenic acid; monitoring oil content to determine extraction efficiency and ensuring the correct protein content of final products such as soybean meal or soy protein concentrate. This study showed that calibrations with good correlations and low errors were successfully achieved. Table 1 summarizes the Root Mean Square Error of Calibration (RMSEC), Root Mean Square Error of Prediction (RMSEP) and correlation coefficient for each component in the calibration model. For all four components, we see that the independent validation sample set error (RMSEP) is very close to the calibration sample set error (RMSEC). The very close agreement between calibration and validation errors, combined with the good correlation coefficients for all four components, indicates that the Antaris FT-NIR analyzer replicated the accuracy of the primary wet chemistry techniques very well across the entire range in component values shown in Table 1. It is worth noting that the calibration was able to accurately predict the linolenic acid percentage in soybeans. The application of the Antaris FT-NIR analyzer would allow production and storage facilities to grade and segregate based on low and high linolenic acid hybrid soybeans. Since low linolenic acid soybeans produce oil that is trans fat free, due to the fact that hydrogenation is not required, there is great value in determining linolenic acid content of soybeans prior to soybean oil production.

	Moisture	Protein	Oil	Linolenic Acid
RMSEC	0.19	0.67	0.49	0.84
RMSEP	0.23	0.73	0.53	1.11
Corr. Coefficient	0.98	0.87	0.85	0.94
Range	8.9–14.6	32.5–39.6	16.0–20.8	1.0–9.5

Table 1: Summary of soybean calibration model errors and correlations

Moisture, protein, oil and linolenic acid components all exhibited, good performance and high correlation across the whole range in component values. The oil calibration and validation plots highlight the excellent accuracy and correlation seen across all four components in the model. The oil calibration plot (Figure 4) shows that the validation (+) samples have predicted (calculated) values that agree well with the actual (wet chemistry) values. The residual plot (Figure 5) illustrates that the errors in the validation samples are equivalent to the errors in the calibration samples across the entire oil component range. The excellent calibration and validation results for this 207 sample calibration set were also attained with relatively few PLS factors. The Predicted Residual Error Sum of Squares (PRESS) plot in Figure 6 shows that a minimum Root Mean Square Error of Cross Validation (RMSECV) was achieved with only nine factors. This combined with the fact that each of the nine factors contributed to lowering the RMSECV error demonstrates the robustness of the calibration model.

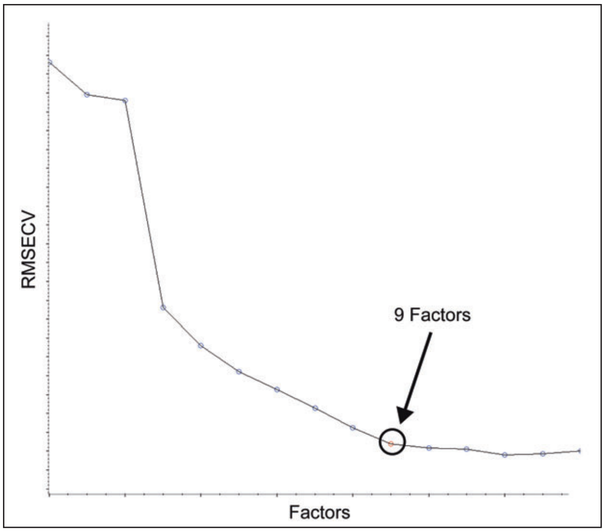

Figure 6: Oil PRESS plot showing minimum error achieved with nine factors used in the PLS calibration

Conclusion

Near-infrared has many advantages over primary wet chemistry techniques for analyzing whole oilseed samples including no sample preparation, significantly shorter analysis time and the ability to perform multi-component analysis on a single instrument. These advantages lead to higher sample throughput and cost savings from fewer consumables and reduced equipment maintenance costs. The Antaris FT-NIR is a robust, simple to operate, fit-for-use analyzer designed for use in dusty production environments with large vibration and temperature variations, similar to the conditions often encountered in oilseed processing facilities. Thermo Scientific RESULT software, which controls the Antaris, simplifies implementation of near-infrared testing as it was designed for high throughput testing and can be operated by non-technical personnel with minimal training. Antaris analyzers also exhibit excellent calibration model transfer and are well suited for inline analysis of production processes through the use of fiber optic cables with probes. This study demonstrates that the Antaris FT-NIR analyzer can quickly and accurately quantify multiple critical components in soybeans.

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