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Precision Solutions From Light

Quantification of Moisture Content in Starch Using NIR

Analytical Spectral Devices, Inc.

Objective: To show the feasibility of using NIR technology as an accurate means of predicting the moisture content in a corn starch food product.

The QualitySpec® Pro NIR analyzer scanning the 350–2500 nm spectral region manufactured by Analytical Spectral Devices, Inc. was the instrument used for the analysis. The QualitySpec® Pro was coupled to the ASD Bi-furcated reflectance fiber-optic cable via its standard SMA style 905 connectors.

Seventy different starch samples were obtained, and three spectral reflectance measurements were collected on each sample. A spectrum averaging of 25 was used to optimize the signal-to-noise ratio. The QualitySpec® Pro has a 100-ms scan time, thus each spectral acquisition was completed in 2.5 s. For a background subtraction, a reference spectrum was collected using a Spectralon reflectance standard.

Once spectral data collection was completed on the samples, they were then analyzed for moisture content via oven dry method. The oven dry results were then correlated to its relevant NIR spectrum generated from the QualitySpec® Pro. The Grams/AI™ chemometric software package with PLS/PlusIQ™ was used for the multivariate analysis and calibration model construction.

Chemometric Analysis & Results

The absorption coefficient of the O-H bond is extremely high in the NIR region (700–2500 nm). These major O-H bands associated with free water occur at approximately 970 nm, 1200 nm, 1420 nm, and 1900 nm. Figure 1

shows the Log 1/R spectral data set that was used for the analysis.

Because of the strong O-H absorption, the spectral region of the calibration data was truncated to the 900–2200 nm region for chemometric analysis. A Partial Least Squares 1 (PLS-1) calibration was applied to the data set, and was used to predict the concentrations in the calibration set. The “leave one out” cross validation was the method employed to vali-

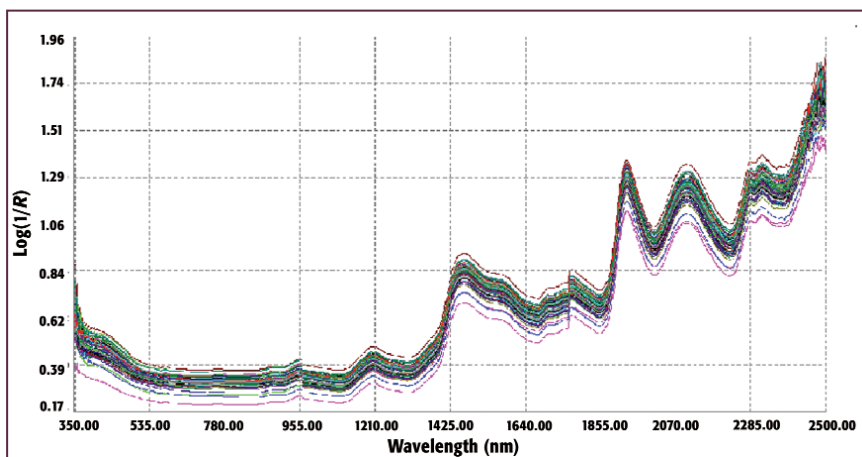


Figure 1

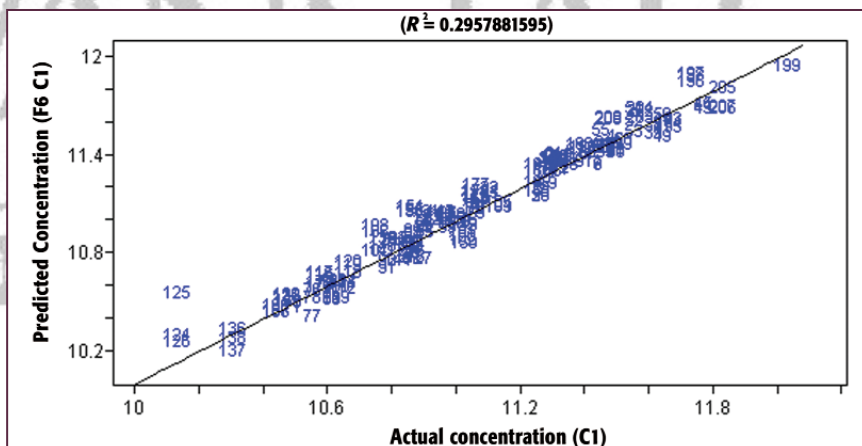


Figure 2

date the calibration set. A 1st Derivative function with a gap of 17 points was applied to the spectra to eliminate any baseline variation between samples. Figure 2 shows the actual tested moisture concentration versus predicted moisture concentration from the calibration set.

The correlation coefficient (R^2) was calculated at 0.958 and the standard error of cross validation (SECV) was calculated to be only 0.081% at 6 recommended factors or principle components. Figure 3 shows the SECV results.

Figure 4 shows the "Scores Plot" which helps to indicate any potential outliers in the calibration set. Each number in the cluster represents a single spectrum in the calibration set. As seen on the right, there are a few spectra that fall out of the main cluster. The spectra outside the data set suggest a potential outlier, or less

PLSplus/IQ Prediction Report			
Calibration File: starch.cal			
Constituent: % Moisture			
Sample	Concentration	Actual	M Distance
Starch predict #1	11.544151	11.82444	1.0711781
Starch predict #2	10.96072	11.13825	0.9836445
Starch predict #3	10.817815	11.28427	2.1448264
Starch predict #4	11.599906	11.84201	0.907799
Starch predict #5	11.173616	11.42434	0.9411015
Starch predict #6	11.322384	11.45472	0.9758999
Starch predict #7	11.443747	11.63574	0.9894161
Starch predict #8	11.430831	11.56328	0.8972861
Starch predict #9	11.506459	11.69405	0.8702141
Starch predict #10	11.032349	11.25522	1.2730673

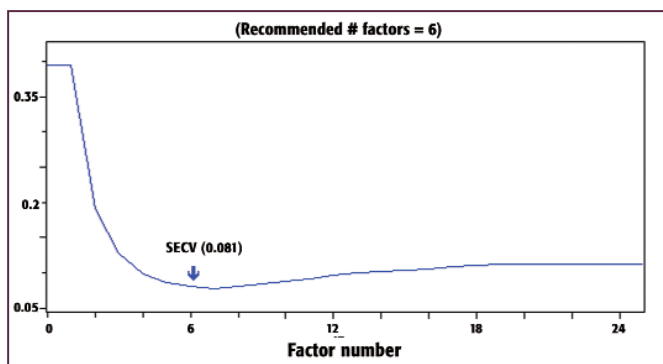


Figure 3

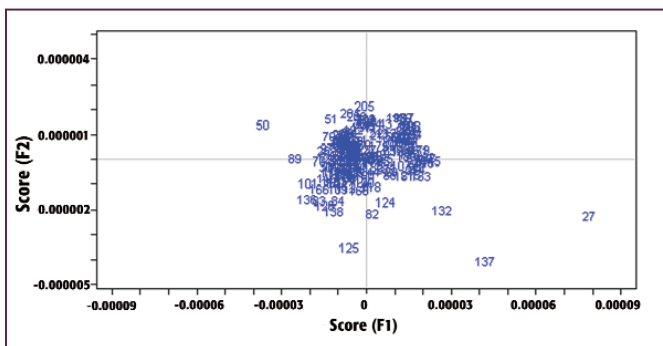


Figure 4

than ideal sample. Oftentimes these outliers may be attributed to a sample not filling the field of view of the fiber-optic probe.

A calibration model was created, and predictions were performed on an unknown data set of 10 starch samples. The prediction report is shown on the left.

Conclusion

This paper demonstrates the feasibility of using the QualitySpec® Pro NIR analyzer to accurately predict the moisture content in corn starch. This technology will allow answers in seconds versus minutes, thus providing answers when and where they are needed most.

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