Chemometrics
Applications Overview

InfoMetrix

Assessing the Composition of Dairy Products and Grain by Near Infrared

Near infrared (NIR) spectroscopy can be used to replace wet chemistry in quantifying many compositional parameters of foods. Analysis of a sample using a traditional wet chemistry approach requires hours, but with the use of optical spectroscopy coupled with chemometrics data processing (using Pirouette® software), the time is cut to 60 seconds making the technique conducive to on-line or near-line process quality control.

The data outlined in this note demonstrates the use of NIR to assess the quality of cheese and wheat samples, but the technique has been proven effective for prediction of fat, moisture, protein and fiber in a diverse array of food products such as soy beans, fish meal, and ground meats. Using the NIR/PLS approach detailed in this application note, the test is non-destructive, quick, inexpensive, and there is very little difference in the results when different technicians are used.

Background

Wet chemistry for protein analysis is traditionally done by the Kjeldahl method and requires approximately 6 hours to perform a test. Moisture evaluation requires between 1 hour and 72 hours depending on the nature of the sample (for example, moisture in the cheese data presented here required a 15 hour test). Fat is the most involved and technically demanding of the tests with a professional chemist averaging between 8 and 12 samples per day using the modified Babcock procedure (although there is a less-accurate instrumented fat measurement where the throughput is more like 20 samples per day).

Because the quality analysis is a laboratory procedure, the sample throughput is constrained to a handful of analyses per day. When an upset occurs in the process line, the delay can often mean that there will be significant wasted product before the results are known and the process can be corrected. On the other hand, a typical NIR instrument requires 30 seconds to load and 30 seconds to measure and process the data, quickly alerting the operator to any process change. The basic issues to be addressed in this application note is whether the NIR instrument can match the accuracy of a wet chemical evaluation (as illustrated in Figure 1).

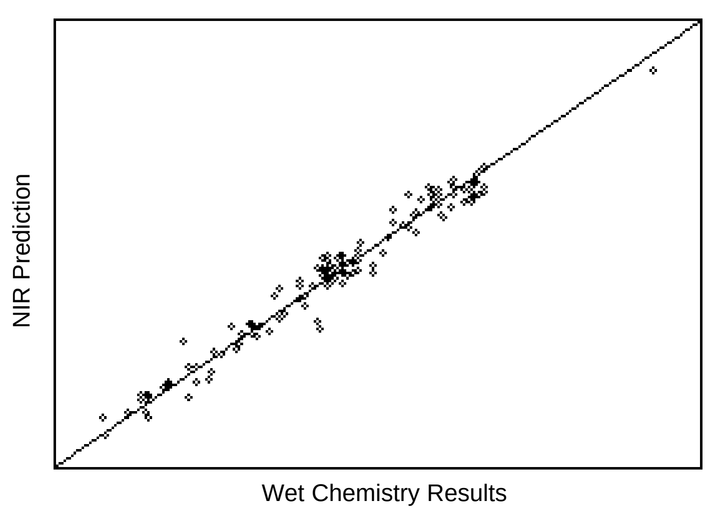
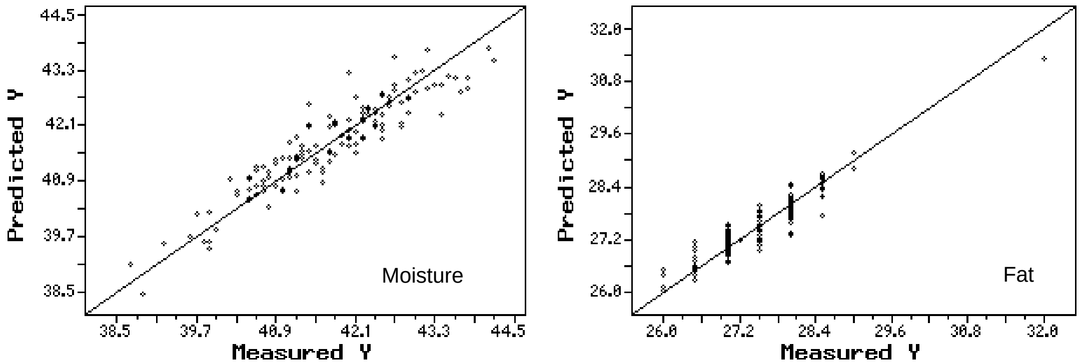


Figure 1. Comparison of NIR to Food Chemistry

Figure 2.
NIR Predictions of Moisture and Fat Compared to Wet Chemical Analysis



The near infrared instruments used in the analysis collected discrete absorbance values in the short-wave near infrared (918nm to 1050nm). Three of the four data sets were monitored with 12 wavelengths, one cheese data set was from a 7 wavelength system. Infometrix' Pirouette software was used to model the data using the Partial Least Squares (PLS) and Principal Component Regression (PCR) techniques.

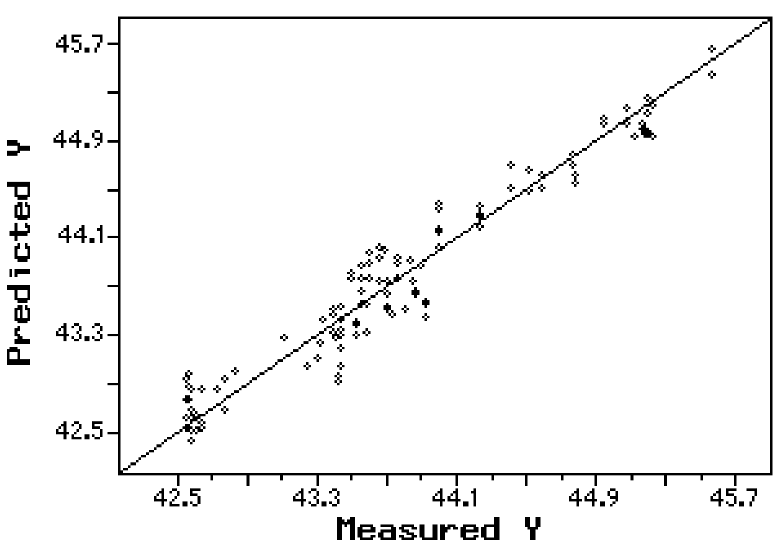
Analysis of Cheese

The quality of cheese is monitored by measuring the fat and moisture contained in the product. In essence, the fat content is a measure of the richness of flavor in the product, moisture relates to texture (and profitability). It is desirable to test as much of the product as possible in order to control the quality closely, but cost is the trade-off.

To test the effectiveness of near infrared results for evaluating moisture and fat content, a succession of brick cheese batches were analyzed by both instrumental and wet chemical techniques. In this example, 12 absorbances in the shortwave near infrared plus two process temperatures were measured for 140 random samples of the product. The resulting NIR spectral data was assembled into a file. Moisture and fat content (the latter by modified Babcock) were measured to supply reference values.

A PLS model for moisture and fat content of the cheeses was created using the NIR results. The resulting comparisons of wet chemistry and instrument results are shown in Figure 2. The validity of the instrument model was tested (using a leave-one-out cross validation technique) and was found to be comparable to the wet chemistry assessment.

Figure 3.
NIR Predictions of Moisture for Mozerella Cheese



There is some scatter in the comparisons of Figure 2, with the data points distributed evenly about the prediction line. The variation is caused by errors in the wet chemistry as well as errors in the NIR spectral measurement. In addition, a resolution effect is seen in the fat measurements, where, because the modified Babcock only measures fat to the nearest 0.5%, the NIR method cannot be assessed below that level of error.

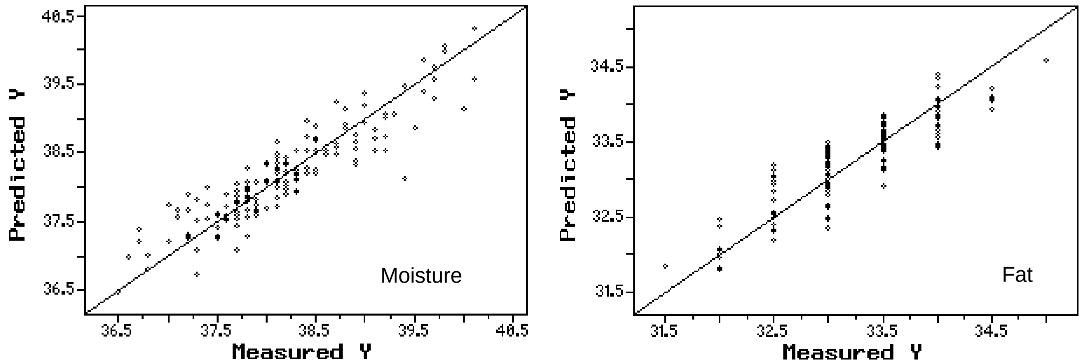


Figure 4.
NIR Predictions of Moisture and Fat for Cheddar Cheese

Note that one of the brick cheese samples displays an unusual fat content that is higher than the typical range for this type of cheese. The NIR technique can clearly identify this sample as out of standard range, but the prediction of fat content will be subject to increased errors unless additional data points are collected in this region to give a more detailed calibration.

The plots of predicted versus actual in Figure 2 show a clear correlation between the modeled values of fat and moisture and the wet chemistry results. The standard error for the NIR results using PLS was computed to be 0.44% for the moisture results and 0.32% for fat. In comparison, errors in a model created by an alternate technique (PCR) were larger, displaying standard errors of 0.47% and 0.37% for moisture and fat, respectively. All values were within the expected variation for the wet chemistry of approximately 0.5% for both measurements within the range of values seen in the brick cheese data.

To confirm the validity and robustness of a NIR/PLS approach to assessing moisture and fat in cheese, two additional data sets were analyzed. One set contained the moisture results for 110 samples of mozzarella cheese (as assessed by 7-wavelengths in the NIR), the other 150 samples of cheddar cheese (using 12 wavelengths). Results for the confirmation tests were similar to the first data set.

Figure 3 shows the moisture predictions for the mozzarella evaluations. These results display the same trends as were seen in the original brick cheeses. The NIR predictions were always within 0.5% of the measurements derived from drying and show a standard error that is somewhat better than the results for brick cheese at 0.23%.

Results from the cheddar cheese batches is given in Figure 4 and show typically higher fat content and lower moisture content than the brick or the mozzarella analyses. Comparison of NIR predictions to the results of wet chemistry are much the same, however. The standard error of prediction for the analysis was 0.33% for the assessment of moisture and 0.31% for fat.

In both the brick and cheddar examples of Figures 2 and 4, the fat content, as measured by the modified Babcock procedure, which yields values in 0.5% increments. This resolution makes the measured values appear to be discrete rather than continuous measures of fat content. The error value for fat should be considered as an upper limit for the technique inasmuch as the resolution on the wet chemistry is only 0.5%, thus affecting the ability of the NIR model to perform to a level better than the resolution allows.

Table 1.
Comparison of Standard Errors for Cheese

	NIR/PLS				Laboratory
	Brick	Mozerella	Cheddar	Average	Range
Moisture Error	0.44	0.23	0.33	0.33	0.2 to 0.5
Fat Error	0.32	--	0.31	0.32	0.3 to 0.5

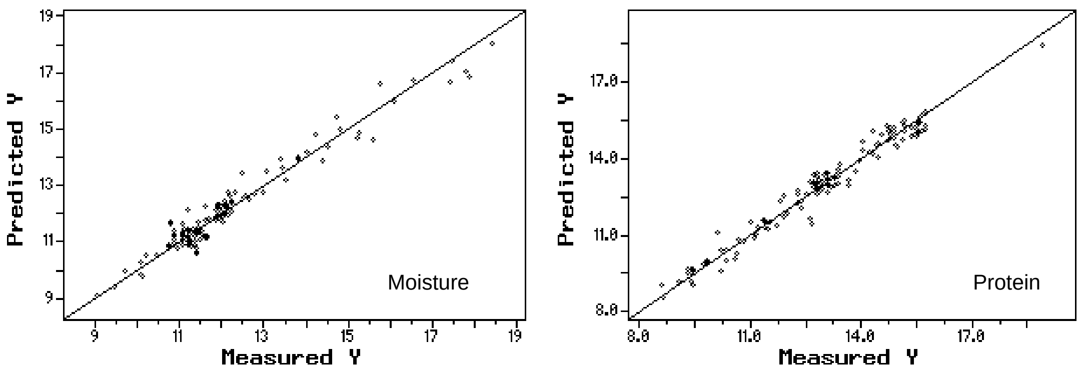


Figure 5.
NIR Predictions of Moisture and Protein for Wheat Samples

Analysis of Wheat

An evaluation similar to the cheese data was applied to wheat samples and NIR was used to evaluate the moisture and protein content of 135 samples. The NIR model is based on data that include 12 wavelengths and 2 temperatures. As with the analyses of the three types of cheese in the previous section, the purpose of this study is to see how closely the near infrared technique can match the precision of wet chemistry methods.

The NIR evaluation versus chemical analysis is shown in Figure 5. The results were comparable with standard errors for moisture of 0.42% and for protein of 0.40%. Again, the results compare favorably with the laboratory method and fall within the guidelines set by the U.S.D.A.

Summary

We have demonstrated the effective use of a simple near infrared spectrometer to quantify the composition of wheat and cheese products. The technique requires that a model be built calibrating wet chemistry results to the instrument data using chemometric data processing techniques (in this case Partial Least Squares modeling). After the calibration step, the spectrometer can be used to augment or replace the wet chemical techniques for routine quality control.

The standard errors of NIR instrumented assessment of moisture, fat and protein match those of the wet chemistry techniques. The advantage of the NIR approach is that it is requires less operator time, is more independent of the operator technique and is less expensive to run and maintain the equipment. In addition, the NIR approach does not require the same level of laboratory support and is conducive to on-line or near-line evaluation of a variety of food products.

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