Use of Near Infrared Analysis for the Evaluation of Rice Quality

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A. Introduction

Near-infrared analysis (NIR) has been well established as a rapid means of quantifying the protein and moisture content of wheat and other grain products for almost thirty years[1]. Surprisingly, the technique is just now finding widespread application in the analysis of rice. In Japan, NIR quickly gained acceptance as a premier analysis tool for rice. However, the United States has been slow to recognize the power of NIR for rice analysis.

This paper describes some recent NIR rice analysis work performed in Japan. Furthermore, it introduces several new products that facilitate easy application of NIR analysis of rice.

B. Background

B.1 Traditional Grain Testing

B.1.1 Kjeldall Protein Analysis - The official "gold standard" method for determining protein content in grains is known as the Kjeldall Process. This wet chemical digestion requires numerous reagents, a laboratory and a skilled technician. In addition, it is costly, time consuming (a minimum of 3 hours is required) and generates hazardous waste that must be disposed of properly. While the technique can be accurate, it is not feasible for on-site analysis. A trained technician following the standard procedures carefully can achieve accuracies of 0.2% with this method for such grains as wheat, barley and rice.[2]

B.1.2 Forced-Air Oven Moisture Analysis - The official method of determining the moisture content of grains is through a simple thermogravimetric analysis - a drydown. There are several USDA approved protocols, the simplest of which requires a single-stage, two-hour drying of the grains. Basically, the weight-loss on drying is used to determine the original moisture content of the grains. More typically, a complex two-stage drydown, which requires about 16 hours, is employed. Assuming all procedures are followed carefully, and all over and tray temperatures are accurately controlled, this methods is accurate to about 0.2% in such grains as wheat, barley and rice.[2]

One exciting new entry to the field of Rice Analysis is the ZX-800, from Zeltex, Inc. in Hagerstown, MD. The instrument is a near-infrared spectroscopic analyzer designed in Japan for the analysis of rice and other whole grains. Operating in the short-wavelength near-infrared, the analyzer accurately measures such constituents as moisture and protein on whole grains in less than 40 seconds. The technique requires no sample preparation and is completely non-destructive. [3]

The ZX-800 boasts the following features:

- Easy, one-button operations
- No sample preparation
- Analysis in about 40 seconds
- Can be calibrated for up to 20 different products
- Designed to meet current NTEP requirements
- Solid-state, robust optics (no moving parts)

B.3. The RN-500

The RN-500 Single Grain Rice Inspector is also now available from Zeltex, Inc. This new product individually inspects each grain of rice in a sample to determine percentages of even, cracked, immature, discolored and dead grain.

By employing a line image sensor, this instrument provides extremely reproducible results in a matter of seconds, and is insensitive to user error. A technician need only load the sample and press a single button. In addition, the instrument can be preset to match samples against different quality control levels.[4]

B.4. Near-infrared Analysis

NIR was developed in the early 1970s, when the need arose for rapid moisture and protein analysis in wheat and barley. Its first application was developed by a joint venture between the US Department of Agriculture and the Canadian Grain Commission. Since that time, the technique has been used for a host of agricultural applications, including constituent analysis in nearly every type of grain. In addition, the technique has found widespread application in process control in nearly every industry imaginable.

The near-infrared (NIR) spectral region is usually defined as that portion of the spectrum with wavelengths in the range 700 to 2500nm. In this spectral region, overtone frequencies of molecular vibrations absorb light quite readily. Because the overtone absorption bands are typically wide and overlapping, spectroscopists cannot merely measure peak heights to perform quantitative analysis. Instead, multivariate regression analyses are utilized t correlate spectral features with concentrations or physical properties of interest.

The ZX-800 operates in the short-wavelength NIR, from 900 to 1100nm wavelength. The instrument may be calibrated to analyze moisture, protein and other constituent contents from the absorption spectra of the grains being tested. This determination is accomplished through the use of multivariate regression equations of the form:

$$\text{Constituent Concentration} = K_0 + K_1 (OD_1) + K_2 (OD_2) \ldots$$

$$\ldots + K_{12} (OD_{12}) + K_{13} (T_a) + K_{14} (T_s)$$

where K_0 is a bias term, K_1 through K_{14} are slope coefficients, OD_1 through OD_{12} are the optical absorbances measured at each of 12 wavelengths, and T_a and T_s are the ambient and sample temperatures at the time of the test.

The instrument can store up to 20 calibration equations, which can be used for multiple constituents on multiple products.

C. Experimental Results

C.1. Use of the ZX-800 for Moisture, Protein, Amylose and Fatty Acids

The ZX-800 has been calibrated to measure such analytes as moisture, protein, amylose and fatty acids in rice. The calibration procedure requires that a finite number of samples (typically on the order of 100) be measured with the ZX-800, then measured by some official laboratory method for each of the analytes of interest. Following these physical measurements, the optical data collected with the ZX-800 are correlated to the analyte (constituent) data using multivariate regression statistics.

In one recent series of experiments performed by a rice mill[5] in Japan, the ZX-800 was calibrated to measure the moisture content of brown and polished rice. Approximately 100 samples of each type of rice were used in the calibration. Regression analyses yielded excellent results, with standard errors of calibration of 0.149 for moisture in brown rice and 0.182 for moisture in polished rice. Figures 1 and 2 show scatterplots of these regressions.

This same mill performed calibrations of the ZX-800 for the measurement of protein in brown and polished rice samples.[5] Again, approximately 100 samples were used for each of the calibrations. Regression analyses yielded excellent results, with standard errors of calibration of 0.192 for protein in brown rice and 0.196 for protein in polished rice. Figures 3 and 4 show scatterplots of these regressions.

Calibrations for amylose and fatty acids were promising, but not as good as those for moisture and protein. Further work on these constituents is currently underway.

C.2. The ZX-800 and RN-500 in Practice

The Japan Rice Mill Industry Association, Inc. (JRMIA) publishes regular reports on the quality of product at various rice mills around Japan. These reports are generated from on- site inspections, and are distributed to the rice-production industry at large.

Rice mill inspection reports include many quality measures, such as rice fullness and percentages of unripe grain, split or sprouting grain and discolored grain. In addition, whiteness and moisture, protein, amylose and fatty acid contents are reported, as is the "fitness for process," a gauge of polishability.

In the most recent JRMIA report[6], data for two rice mills (Miyajaki in Hinami City, and Kagoshima in Ohsaki-Cho) were presented. Moisture, Protein, Amylose and Fatty Acid contents determined by the ZX-800 were reported for each mill. Grain Consistency, Whiteness, Water Content, Unripe Grain, Damaged Grain, Dead Rice, Foreign matter and Grain Composition as determined with the RN-500 were also reported for each mill. It is our understanding that the JRMIA was quite pleased with the ease-of-use of the ZX-800 and RN-500, and intends to utilize them for future rice mill evaluations.

C.3. A Field Portable Alternative

Kett Electric Laboratory, in Tokyo, measured sixty rice samples on the ZX100C Field-Portable Spectroscopic Analyzer, from Zeltex[7]. The samples were predivided into a calibration set (N=50) and a prediction set (N=10). Each samples consisted of approximately 25 grams of grain poured into a cuvette supplied with the ZX-100C. No sample preparation was performed.

Near-infrared spectra were acquired by simply placing the sample cup in the ZX-100C and pressing a single button. Following this measurement, the moisture content of each sample was determined using a forced-air oven according to the European Standard Method.

A calibration model for the near-infrared analyzer was developed using the optical and moisture data from the 50 calibration samples. The samples were varied in temperature from 1 to 30°C to facilitate a more robust calibration. After running a best-wavelength search routine, the optimal regression mode was developed for the prediction of moisture in the independent prediction samples. The final 9-term (7 wavelength and 2 temperature terms) regression model was constructed from 49 of the original 50 calibration samples, as one scan was identified as an outlier. The prediction results generated from this model were quite good, with prediction statistics as follows:

- Analytical Range = 11 to 15.5%
- Correlation (R) - 0.99
- Standard Error of Prediction (SEP) = 0.14%

D. Summary

The analysis of various grains using NIR continues to be high growth area. Traditionally, the technology had been applied to wheat, barley and corn samples. However, the recent application of NIR to rice analysis has been extremely successful in Japan. To date, over 250 ZX-800 instruments are being used in Japan for rice. This methodology is now gaining momentum in the United States' rice-production community.

E. References

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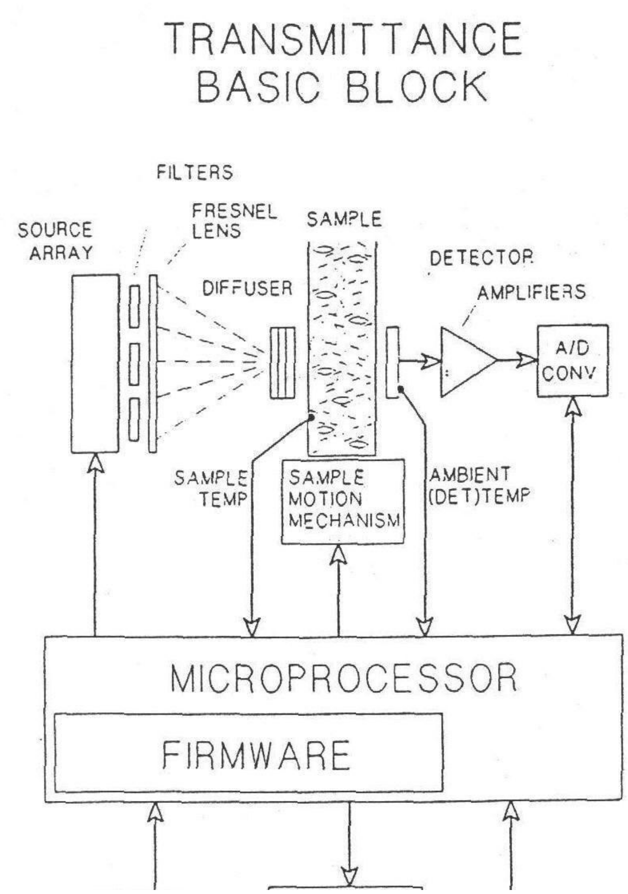
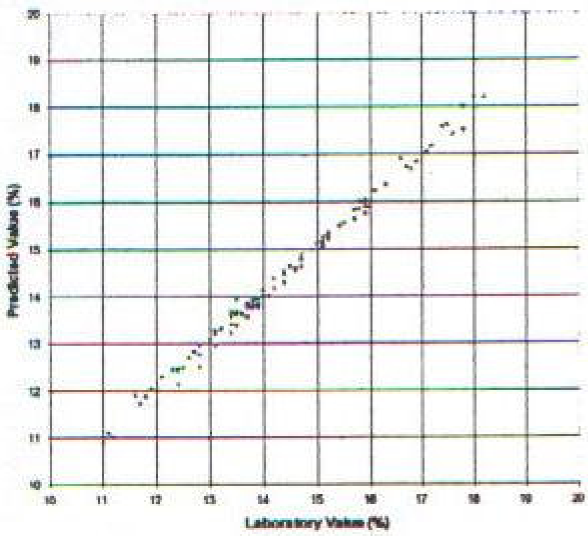


Figure 1. Prediction of Water Content in Brown Rice



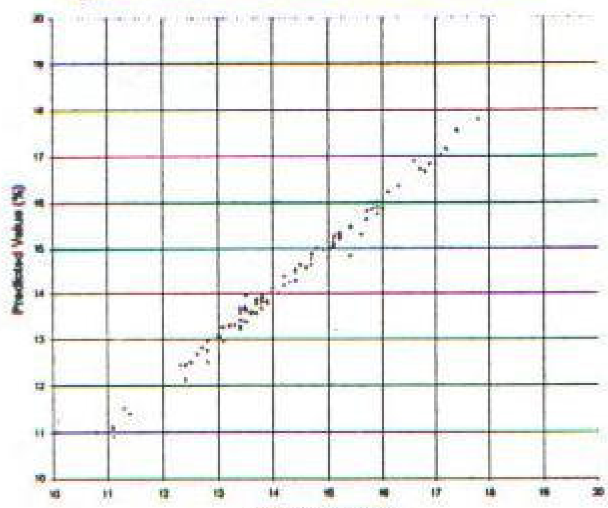
Prediction Results

N = 97

Correlation = 0.996

SEC = 0.149

Figure 2. Prediction of Water Content in Polished Rice



Prediction Results

N = 99

Correlation = 0.994

SEC = 0.182

Figure 3. Prediction of Protein Content in Brown Rice

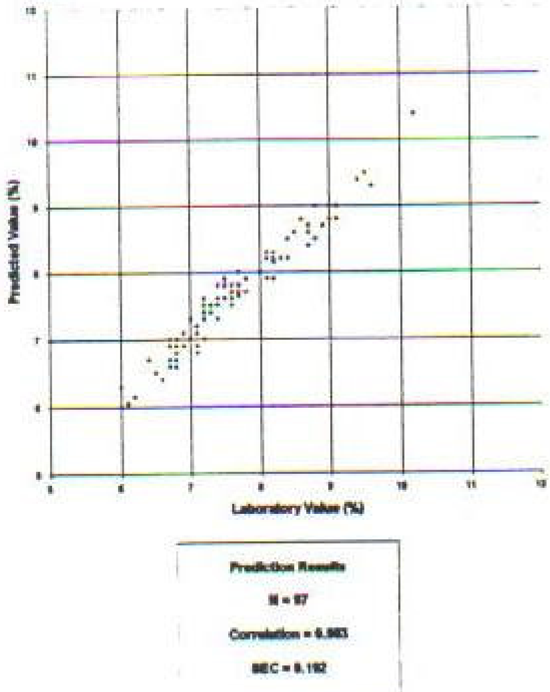


Figure 4. Prediction of Protein Content in Polished Rice