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Accuracy of Near-infrared Transmission Spectroscopy for Determining Rice Constituent Contents and Improvement in the Accuracy

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Abstract. *The accuracy in determination of rice constituent contents using a commercial near-infrared transmission (NIRT) instrument was validated. And the accuracy of measurements using this instrument was improved by modifying light filters and calibration equations used in the instrument. In the determination of moisture content, the coefficient of determination (r^2) was 0.98, standard error of prediction (SEP) was 0.13%, and bias was -0.02%. In the determination of protein content, r^2 was 0.90, SEP was 0.17%, and bias was 0.01%. These results show that the newly developed instrument is sufficiently accurate to be used instead of reference analysis for measuring moisture content and protein content of brown rice. An automatic rice-quality inspection system was designed. The system consisted of a rice huller, a rice cleaner, an NIRT instrument and a visible light (VIS) segregator. Based on rice-quality information, this system enables rough rice transported to a rice grain elevator to be classified into six qualitative grades.*

Keywords. Near-infrared spectroscopy, Rice, Moisture content, Protein content, Automatic rice-quality inspection system, Grain elevator

Introduction

An instrument for determining rice constituent contents using near-infrared (NIR) spectroscopy, which was named a rice-taste meter, was developed in 1986 in Japan (Hosaka, 1987). It was an NIR reflectance (NIRR) instrument and was designed for analysis of ground white rice samples. Due to later demands for an instrument that can analyze brown rice as well as white rice and does not require the rice to be ground, an NIR transmission (NIRT) instrument was developed for analysis of whole grain brown rice samples in the mid 1990s.

Recent studies (Barton et al., 1998; Chen et al., 1997; Delwiche et al., 1995, 1996; Iwamoto et al., 1986; Kawamura et al., 1991, 1996, 1997, 1998, 1999; Li et al., 1997; Natsuga et al., 1992; Suzuki et al., 1996; Villareal et al., 1994) have demonstrated that NIR spectroscopy can be used to determine both chemical and physical compositions of rice and the taste of rice. Many studies have shown that the accurate determinations of moisture content and protein content are reliable alternatives to chemical analysis. In these studies they developed calibration equations with a sample set collected for the studies and validated the accuracy of the calibration equations with the sample set collected at the same time. In additions, they developed calibration equations with an NIR instrument and validated the equations with the identical instrument.

For routine analysis in practical use of NIR spectroscopy, however, an instrument, which is called a pre-calibrated instrument, is not the identical instrument used for developing calibration equations, which is called a master instrument. The sample set measured in routine analysis is different from the calibration sample set used for developing calibration equations. The accuracy of pre-calibrated instruments in the practical use for routine analysis, for instance the use in rice grain elevators and rice millers, may accordingly be worse than that of instruments used in the laboratory use.

The major constituent contents of rice are moisture, protein, starch (amylose and amylopectin) and fat. The protein content of rice is an important item for quality, especially in East Asian countries, where people eat Japonica type rice, because rice with low protein content is evaluated sticky and delicious for the people. After harvesting, rough rice is transported to a grain elevator. Upon arrival at the elevator, moisture content of the rice is usually checked using an electric resistance grain moisture meter. Recently, there has been a need in rice grain elevators in Japan for an accurate method to measure not only the moisture content but also the protein content of rice in order to grade the rice according to quality upon arrival at grain elevators.

The objectives of this study were to validate a pre-calibrated NIRT instrument for practical use in a grain elevator, to improve the accuracy of the instrument, and to develop an on-line automatic rice-quality inspection system that can be used in a rice grain elevator.

Materials and Methods

Rice Samples

Sample group A consisted of 36 brown rice samples collected from all over Japan, including 22 samples of Kirara397, a popular rice variety grown in Hokkaido, the northernmost island of Japan. Sample group B consisted of 48 brown rice samples, all Kirara397 rice samples.

Reference Analysis

Moisture content of brown rice was determined by the official method of the Japan Food Agency; about 5 g of ground rice was placed in a forced-air oven at 105°C for 5 h, and moisture was calculated on a wet basis. Protein content of brown rice was determined by the Kjeldahl method (N × 5.95) and calculated on a dry basis. Free fat acidity value of brown rice was determined by the rapid method of the American Association of Cereal Chemists, extracting free fat acid in benzene solution and titrating the extracted solution with potassium hydroxide solution. Amylose content (apparent amylose content) of white rice was measured using an autoanalyzer and calculated percentage of amylose in rice starch on dry basis. Each reference analysis was carried out two or three times for each sample.

Near-infrared Transmission Instrument

A near-infrared transmission (NIRT) instrument (model CTA 10A, Satake Engineering, Tokyo, Japan) was used in this study. This was called a prototype instrument in this study. After the experiment using the prototype instrument, we newly developed another NIRT instrument (model CTA 10B, Satake Engineering, Tokyo, Japan), which was called a modified instrument in this study. Each instrument consisted of a tungsten-halogen lamp, 10 discrete light filters and a silicon photocell. NIRT spectra could be obtained a wavelength range of 600 to 1100 nm using these instruments. Both of the instruments were commercially available pre-calibrated instruments. The calibration equations to determine moisture content, protein content, free fat acidity and amylose content were developed by the method of partial least squares using each master instrument of the two instruments used in this study. The calibration equations developed using the two master instruments were installed in the two instruments used in this study, respectively.

Measurements of sample group A were first carried out using the prototype instrument. Measurements of sample group B were then carried out using the modified instrument. The sample group A and B were different from the sample sets that were used to develop the calibration equations using each master instrument. The use of the two instruments in this study thus was considered to be in the same conditions as routine analysis in practical use of NIR instrument.

Results and Discussion

Reference Analysis

Table 1 shows results and precision of reference analysis. The standard deviation of difference among repetitions of measurement (SDD) of each reference analysis shows that the precision of the reference analysis was sufficient.

Accuracy of Prototype and Modified NIRT Instruments

The validation statistics of the two instruments in determination of rice constituent contents are shown in Table 2. The SDD of each instrument was almost same as that of the reference analysis. The SDD data indicated that the level of precision of each instrument among repetitions of measurement was sufficiently high.

Table 1. Results and precision of reference analysis.

	Sample group A						Sample group B					
Constituent content	n	Mean	Min	Max	SD	SDD	n	Mean	Min	Max	SD	SDD
Moisture content of brown rice (%)	33	15.0	14.3	15.8	0.27	0.02	48	17.0	15.0	18.2	0.83	0.02
Protein content of brown rice (%)	36	8.1	6.4	9.2	0.54	0.07	48	9.7	8.5	10.9	0.54	0.08
Free fat acidity of brown rice (mg)	36	16.9	11.2	36.4	5.84	0.40	48	14.8	10.6	21.7	2.85	0.47
Amylose content of white rice (%)	36	20.6	18.6	22.3	0.79	0.31	48	18.5	17.8	19.3	0.31	0.12

n: number of samples. SD: standard deviation. SDD: standard deviation of difference among repetitions of measurement.

Table 2. Validation statistics of near-infrared transmission instruments in determination of rice constituent contents.

Constituent	Instrument	n	SDD	r^2	SEP	Bias	Regression line
Moisture content of brown rice (%)	Prototype	33	0.04	0.64	0.22	-0.23	y= 0.6x +6.0
	Modified	48	0.06	0.98	0.13	-0.02	y= 1.0x -1.0
Protein content of brown rice (%)	Prototype	36	0.07	0.84	0.23	-0.49	y= 1.1x -0.6
	Modified	48	0.06	0.90	0.17	0.01	y= 1.0x -0.0
Free fat acidity of brown rice (mg)	Prototype	36	0.22	0.01	7.09	-1.97	y=-0.2x +19.9
	Modified	48	0.39	0.69	1.96	16.68	y= 0.7x -6.4
Amylose content of white rice (%)	Prototype	36	0.07	0.41	0.61	-0.68	y= 1.0x -0.7
	Modified	48	0.12	0.13	0.38	-0.23	y= 0.3x +12.8

n: number of samples. SDD: standard deviation of difference among repetitions of measurement. r^2: coefficient of determination. SEP: standard error of prediction. Regression line: regression line from predicted constituent content (x) to reference constituent content (y).

Figure 1 shows the correlation between reference moisture content of brown rice and predicted moisture content using the prototype instrument. The coefficient of determination (r^2), standard error of prediction (SEP) and bias in measurement of moisture content were 0.64, 0.22% and -0.23%, respectively. Figure 2 shows the correlation between reference protein content of brown rice and predicted protein content using the prototype instrument. The r^2, SEP and bias in measurement of protein content were 0.84, 0.23% and -0.49%, respectively. The validation statistics of the prototype instrument indicated that the level of accuracy of the instrument was

not sufficient for determination of moisture content, protein content, free fat acidity and amylose content. We therefore modified the instrument as follows.

1. Modification in light filters: The prototype instrument had 10 light filters with mean half-width of 10±2 nm in each wavelength. The 10 light filters used in the modified instrument had the same light-pass wavelength and half-width as those used in the prototype instrument but had smaller wavelength drift by temperature than that of the light filters in the prototype instrument. The light filters in the modified instrument drifted 0.035 nm in wavelength per 1°C, whereas those in the prototype instrument drifted 0.1 nm per 1°C.

2. Modification in temperature control of light filters: The temperature of the light filters was controlled at 50°C in both instruments. The sensor used for monitoring and controlling the temperature in the filter chamber was moved closer to the filters in the modified instrument. Stirring air in the filter chamber of the modified instrument increased the uniformity of air temperature in the filter chamber. This modification resulted in a smaller range of fluctuations in the temperature of the filters in the modified instrument (50±0.5°C) than that in the prototype instrument (50±2°C).

3. Modification of local calibrations: The calibration equations for the prototype instrument were created using 200 samples collected from all over Japan, while those for the modified instrument were created using the same 200 samples as well as 60 samples of Hokkaido rice because the calibration equations were used for measurement of only local rice (Hokkaido rice).

4. Fine adjustment of calibration equations to the modified instrument: NIR spectra obtained by using a master NIR instrument and those obtained by using other pre-calibrated instruments are slightly different even if the instruments have the same specifications. To standardize the master instrument and the pre-calibrated instrument (the modified instrument in this study), we adjusted the calibration equations to the modified instrument. The adjustment was done using ten samples whose reference data were already known.

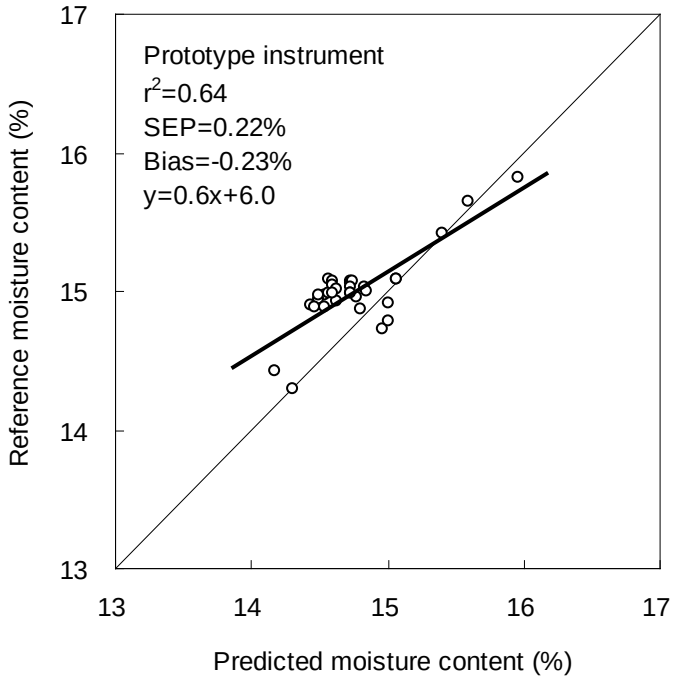

Figure 1. Correlation between reference moisture content of brown rice and predicted moisture content using prototype instrument.

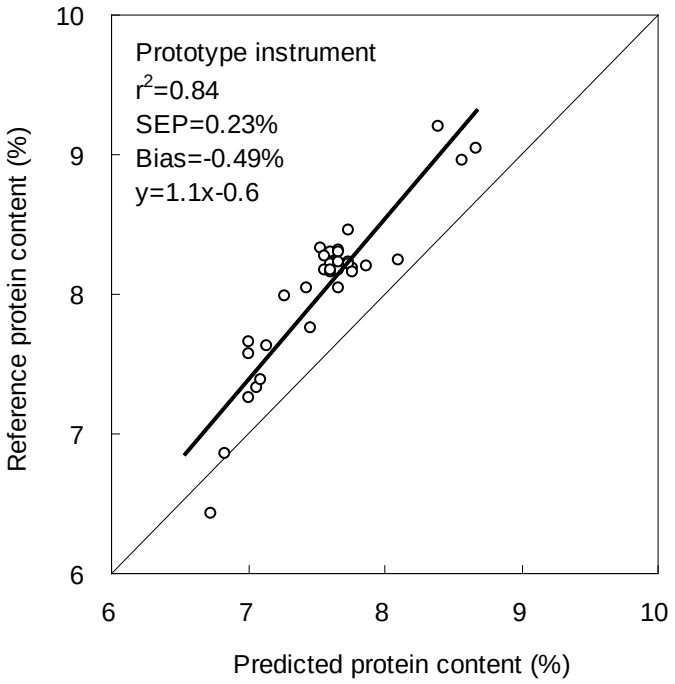

Figure 2. Correlation between reference protein content of brown rice and predicted protein content using prototype instrument.

The validation statistics of the modified instrument for determination of rice constituent contents are shown in Table 2. The correlations between reference moisture content and predicted moisture content, and between reference protein content and predicted protein content using the modified instrument are shown in Figures 3 and 4, respectively. The values of r^2, SEP and bias in measurement of moisture content were 0.98, 0.13% and -0.02%, and those in measurement of protein content were 0.90, 0.17% and 0.01%, respectively. These results show that the accuracy of the modified instrument was greater than that of the prototype instrument. The modified instrument could be used to determine moisture content and protein content instead of the reference analysis. On the other hand, r^2, SEP and bias in measurement of free fat acidity were 0.69, 1.96 mg and 16.68 mg, respectively, and those in measurement of amylose content were 0.13, 0.38% and -0.23%, respectively (Table 2). Measurements of free fat acidity and amylose content of rice using NIR spectroscopy are not sufficiently accurate.

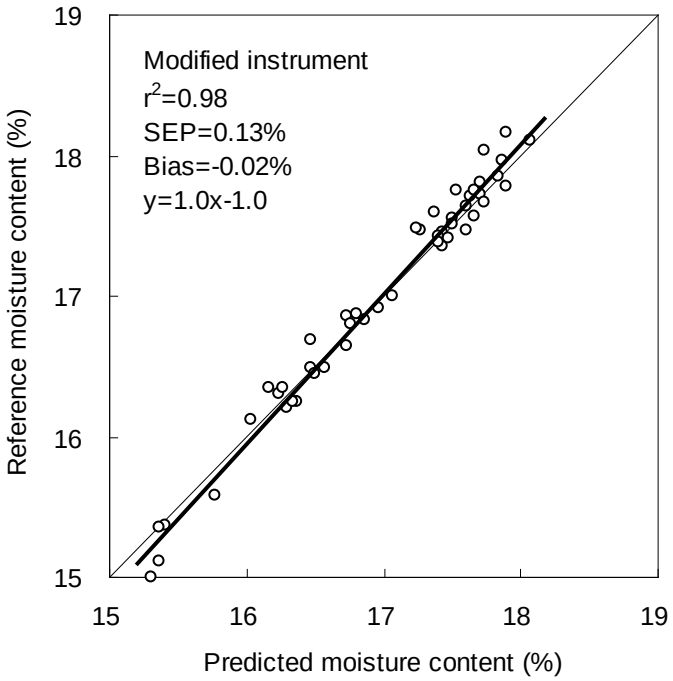

Figure 3. Correlation between reference moisture content of brown rice and predicted moisture content using modified instrument.

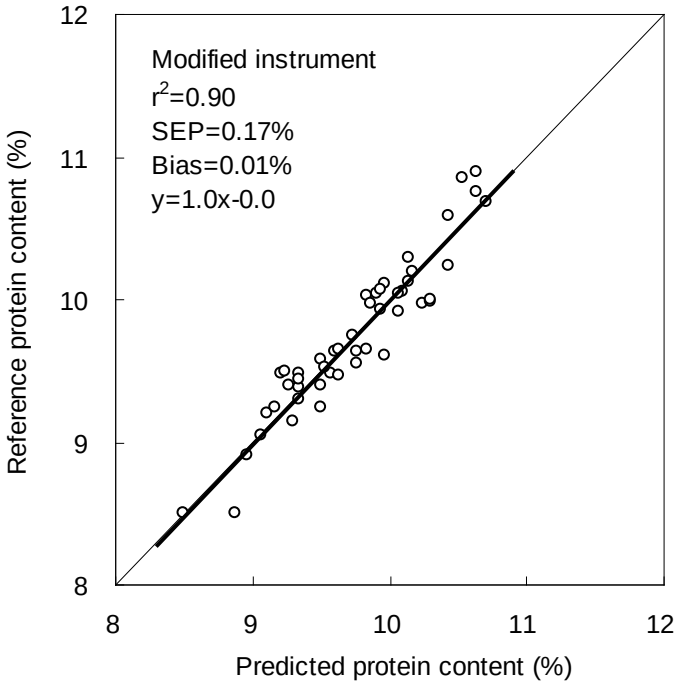

Figure 4. Correlation between reference protein content of brown rice and predicted protein content using modified instrument.

Development of an Automatic Rice-quality Inspection System

We designed an automatic rice-quality inspection system. As shown in Figure 5, the system consisted of a rice huller (an impeller-type huller), a rice cleaner (a thickness grader), an NIRT instrument and a visible light (VIS) segregator. Each rice sample was moved automatically from one apparatus to the next one through tubes by pneumatic conveyors, bucket elevators or by the force of gravity. In this system, rough rice samples should be hulled so as not to become stuck in the grain path and also to enable measurement of sound whole kernels of brown rice by the VIS segregator.

Based on information of quality items (protein content, moisture content and sound whole kernel ratio), rough rice transported to a rice grain elevator can be classified into six qualitative grades, i.e., three protein content levels times two sound whole kernel ratios, using the automatic rice-quality inspection system. About 200 automatic rice-quality inspection systems are currently being used at rice grain elevators in Hokkaido, Japan.

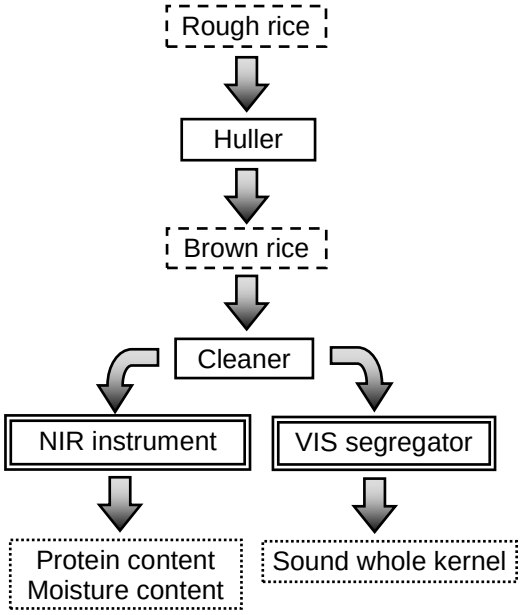

Figure 5. Flow chart of automatic rice-quality inspection system.

Conclusion

The precision among repetitions of NIRT measurement was sufficiently high. Modifications in light filters, in temperature control and of local calibrations, and fine adjustment of calibration equations resulted in significant improvements in the accuracy for determining protein content and moisture content of rice. An automatic rice-quality inspection system consisting of a rice huller, a rice cleaner, an NIRT instrument and a VIS segregator was developed. Based on information of quality items, the use of this system enables rough rice transported to a rice grain elevator to be classified into six qualitative grades. About 200 automatic rice-quality inspection systems are currently being used at rice grain elevators in Hokkaido, Japan.

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