

Optimal Geometries for the Development of Rice Quality Spectroscopic Chemometric Models

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ABSTRACT

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Three sample geometries, two different instrument types, and two spectral collection modes (reflectance and transmission) were used to assess rice quality and develop chemometric models for composition and sensory characteristics. Rice samples (120) including three cultivars, two growing locations, five drying treatments, two moisture levels, and two levels of milling were scanned in two locations. Data collected for modeling included amylose, protein, moisture, whiteness, transparency, and milling degree. Taste and texture were determined with the use of separate trained sensory panels. The NIR models show that composition is

best modeled in the 1,100–2,500 nm range, while the physical properties of whiteness, transparency and milling degree are best modeled in the 750–1,050 nm range. Additional models were developed using limited data subsets of the spectral data points. In some cases, adequate models were generated with as few as 20 wavelength data points. Results show that no one spectroscopic protocol is best for all analytes in rice and that for any complex food matrix more than one preprocessing or spectral range protocol is needed.

Rice is a staple part of the diet of over one-half the world's population (Juliano 1985, Rouhi 1996). The quality, perceived and real, of rice has become a focus of several studies (Champagne et al 1996, 1997, 1998; Delwiche et al 1996; Windham et al 1997) since the United States began exporting rice to Japan in 1994. Rice is produced principally in Asia, Australia, and the United States. Issues of quality have been raised because the cultural preferences of these societies are quite different. The Japanese prefer short- and medium-grain sticky rice served plain as a side dish. Americans prefer long-grain, nonsticky varieties, generally prepared with a sauce or gravy such that taste is not a factor. Objective determinations of quality and nutritive value are necessary as part of international commerce, and both parties must understand the definition of quality. The long-, medium-, and short-grain rices produced in the United States are, by compositional analysis, of very high nutritional quality (Webb 1985). However, minor flavor and textural properties expressed in cooked rice deem them less acceptable by the Japanese consumer. Wheat, corn, and soybeans are traded worldwide on the basis of near-infrared spectroscopy (NIRS) analysis. The same should be true for rice. Several studies have shown that NIRS could be used to measure compositional and quality factors in rice (Satake 1990; Villareal et al 1994; Delwiche, et al 1995, 1996). Villareal et al (1994) looked at rice amylose content. Delwiche et al (1995, 1996) developed reflectance models for rice protein, apparent amylose, milling properties, and rheological properties. Satake (1990) described an NIRS approach to measure taste sensory perception.

Several questions must be answered to develop quality standards for U.S. rice. First, how are rices evaluated in Japan as compared to the United States? Second, what are the taste and textural properties of rice, and can they be estimated by NIRS? The first two questions have been addressed (Champagne et al 1996, 1997, 1998; Windham et al 1997). Third, what is the best sample presentation mode for NIRS evaluation of rice quality? Fourth, what role does cooking play in affecting the NIRS analysis of rice?

This study was initiated to determine the instrumental requirements to measure "rice quality" by NIRS. Three things are required for any spectroscopic chemometric technique to be successful. First, the reference data must have a real relationship to the spectral data so that the appropriate statistical data treatment can be employed. Second, a well-behaved instrument (i.e., high signal-to-noise ratio [S/N] and wavelength precision) is required. Third, the spectra must be obtained in an optimal geometry to permit the first two criteria to achieve the best results.

Three instrumental sample geometries (large bulk sample cups, spinning cup whole kernel, spinning cup ground); two different instrument types (400–2,500 nm in an NIRSystems 6500 spectrometer [6500] and near NIR 850–1050 nm in a Tecator Infracore 1265 spectrometer [1265]); and two spectral collection modes (reflectance and transmission) were used to assess rice quality and develop chemometric models for composition and sensory characteristics. Furthermore, estimations of the minimal data required in terms of spectral data points for adequate property determination were made by limiting the spectral data used for model development. Spectroscopic comparisons were made on cooked vs. uncooked rice, to see the effect of water on the compositional characteristics of selected samples. Rice is essentially starch, protein, moisture, and a small amount of lipid in a complex matrix. It is the structure of the starch-protein complex as a matrix that gives the particular rice variety its unique characteristics. These attributes should be amenable to NIRS model development and assay.

MATERIALS AND METHODS

Rice Samples

Four rice varieties from the 1994 season, including M401 and Koshihikari grown in Arkansas and California, Bengal grown in Arkansas, and Calrose grown in California, were harvested at 20% moisture and immediately dried (within 24 hr) by one of three techniques to 12 and 15% moisture levels: 1) air-drying at 18°C and 40% rh; 2) air-drying at ambient temperatures; and 3) continuous flow-drying with heated air. High (60°C), normal (50°C), and low (32°C) commercial drying temperatures were used. Following drying, the paddy (rough) rice was stored in closed containers for approximately two to three months at 18°C and 40% rh. One week before sensory testing, the samples were shelled using a Satake rice machine, model SB, (Satake Engineering Co. Tokyo, Japan) and then immediately milled. Regular (light) milling was accomplished using a laboratory Satake one-pass mill (pearler, model SKD). The first pass was with a 50-g weight in the 5th position; the second pass was with a 50-g weight

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in the 3rd position. Deep milling was performed on 250-g portions of the regular-milled rice using a laboratory Satake grain testing mill (model TM05). Milling conditions were 1 min at 1,250 rpm using a fine-mesh abrasive wheel. Broken rice was removed with appropriate laboratory-sizing devices using standard indented plates and cylinders as described in Champagne et al (1996). One sample of Calrose rice used as a control for sensory evaluation was used only once as a sample in the NIRS studies.

Reference Analyses of Uncooked Whole Grain Milled Rice

Protein ($N \times 5.95$) was determined by the method of combustion (AOAC 1990). Apparent amylose content was determined using a visible/near-infrared scanning monochromator (6500) with a calibration equation described by Delwiche et al (1996). Ordinarily, calibration with NIRS data would not be done, but since we are comparing optical geometries, we believe it is a valid approach. Values for whiteness, transparency, and milling degree were measured on a Satake model MM-1B milling meter in accordance with the manufacturer's instructions. Taste was measured by a Satake neuro fuzzy rice taster (Satake 1990).

Spectroscopic Analyses

Spectral geometries include a number of parameters that are associated with the instrument and the sample. In this study, two instruments that cover very different ranges are compared. One operates in transmission mode (i.e., the light travels through the sample to a detector), the other operates in reflectance mode (i.e., light is reflected off the sample to a detector). Two types of sample devices are employed: large bulk cells used with the NIRSystems transport device, which hold ≈ 100 – 150 g of rice and a smaller cup that holds 25–30 g. Finally, the sample is presented as whole kernels and as a ground flour. In this way, the models developed with different scanning segments, sample size, instrument optical mode (transmission vs. reflectance), and sample appearance (whole kernel vs. flour) can be compared.

Two visible/near-infrared scanning monochromators (6500 and 1265) were used to collect reflectance and transmittance readings

over a wavelength range of 400–2,498 nm and 850–1,050 nm, respectively. The geometries employed in reflectance mode were: the 6500 with whole kernel rice in a bulk half cell, 110- × 42-mm window (65RB); a 6500 spinning cup with whole kernel rice, 35-mm window (65SC); and a 6500 spinning cup with rice flour, 35-mm diameter window (65RF). The geometries employed in transmission mode were: a 6500 monochromator with whole kernel rice in a bulk half cell, with two 110- × 42-mm windows and single layer of rice pathlength (65TB); and the 1265 with whole kernel rice, 125-mm window, with an 11-mm pathlength. The instruments were operated by either the software package NIRS3 v.3.11 (Infrasoft International, Inc. Port Matilda, PA), which includes modules for acquisition and processing of spectra, or Tecator's data collection modules. Information was transferred to ISI format for processing. Whole-grain milled rice (100 g) was scanned in conjunction with sensory test sessions in a transport device reflectance bulk cell as described by Windham et al (1997). Whole-grain milled rice (150 g) was scanned in the Tecator cup. Whole-grain milled rice and rice ground to a flour (30 g) by a Satake cyclone-type mill with heater and sample vibrator as described in Satake (1990) were scanned in the 6500 in a spinning cup. Samples were scanned uncooked in conjunction with sensory tests, and portions of the cooked samples from sensory tests were subsequently scanned. In all cases, 32 scans were averaged for each sample.

Multivariate Analysis

Multivariate analysis was performed with modules contained in the ISI software package. The multivariate method of partial least squares (PLS), as described by Martens and Naes (1989), was used to develop the chemometric models for protein, amylose, taste, whiteness, transparency, and milling degree. The preprocessing data technique of multiplicative scatter correction (Isaksson and

TABLE I
Summary of Chemical and Satake Milling Meter Constituents in Milled Rice^a

Constituent	Range	Mean	Standard Deviation
Amylose	16.40–23.50	19.42	1.51
Protein	3.90–8.30	5.98	0.88
Taste Value	50.00–85.00	73.93	11.09
Whiteness	32.30–52.00	43.93	5.59
Transparency	2.41–4.69	3.28	0.43
Milling degree	60.00–157.00	116.72	26.24

^a Percentage on a dry matter basis.

TABLE II
Standard Error of Cross Validation for Whole-Grain Milled Rice and Rice Flour for Different Sample Geometries and Software Systems^a

Constituent	6500 Reflectance (65RB)	6500 Spinning Cup (65SC)	6500 Spinning Cup Flour (65RF)	6500 RTCAL	1265 Transmission
Amylose	0.52	0.55	0.53	0.59	0.53
Protein	0.22	0.25	0.18	0.22	0.18
Taste value	3.22	3.85	3.88	4.15	4.32
Whiteness	0.65	0.99	1.51	0.59	1.01
Transparency	0.13	0.15	0.17	0.17	0.19
Milling degree	2.89	3.92	5.71	2.55	4.29

^a Two visible/near-infrared scanning monochromators (NIRSystems 6500 and Tecator Infracat 1265) were used to collect reflectance and transmittance readings. Full spectrum calibration models developed by multivariate method of partial least squares (PLS1), with cross validation used to determine standard error of performance. RTCAL = real-time calibration results.

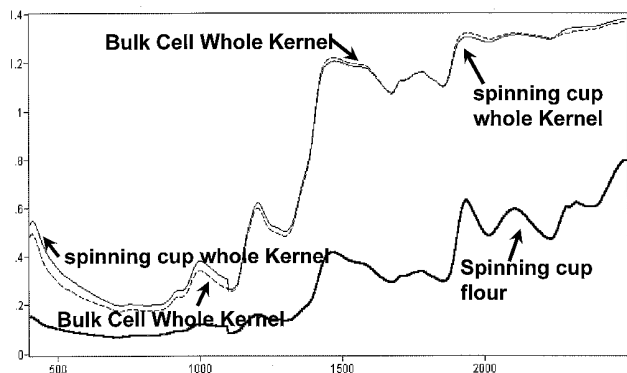


Fig. 1. Reflectance spectrum of rice from an NIRSystems 6500 spectrometer at 400–2,500 nm in three different spectral geometries: bulk whole kernel, spinning cup whole kernel, and spinning cup flour.

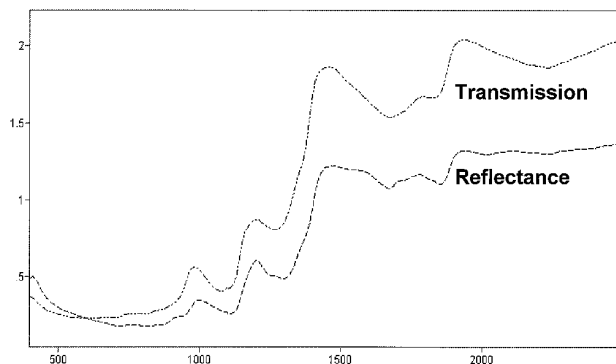


Fig. 2. Reflectance and transmission spectra of rice from an NIRSystems 6500 spectrometer at 400–2,500 nm in the bulk whole kernel geometry.

Naes 1988) was applied to the spectra to remove interferences from scatter, and then transformed with a second derivative (gap = 1–12 nm) were tried. A gap of 4 nm was chosen and subsequently used for all comparisons to enhance absorption bands. The transmission spectra from the 6500 were truncated to 1,100–1,700 nm because of high absorptions at the longer wavelengths and excessive noise at shorter and longer wavelengths. The reduced spectral data sets were prepared by taking every *i*th data point, where *i* = 1–20. The specific number of data points selected in this manner will vary depending on the derivative gap (i.e., a larger gap will have fewer data points). Comparison of models was made on the basis of standard error of cross validation (SECV) as described by Martens and Naes (1989) and Shenk and Westerhaus (1991a,b). The RTCAL chemometric models were developed from a pooled library of rice samples as described by Naes et al (1990) and Naes and Isaksson (1992). Each sample's spectrum used the whole sample set's spectra as the library, and selected samples whose spectra were similar to the spectrum of the individual sample. A group of 40–60 sample spectra was the usual size. An individual PLS model was derived for each spectrum and the six parameters were determined. Again, SECV was used to compare the models developed.

RESULTS AND DISCUSSION

The compositional data for the rice samples are given in Table I. The same samples were used in the texture study reported by Windham et al (1997). While the variability of the samples in the data set is not large enough for establishing standards, it is sufficient for evaluating models and geometries. Each of the five specific instrument geometries will be discussed: (reflectance mode) 6500 bulk half cell (65RB), a 6500 spinning cup whole kernel (65SC), and a 6500 spinning cup flour (65RF); (transmission mode) a 6500 bulk half cell (65TB) and the 1265. In addition, the wavelength range of the two instruments differs considerably. The 6500 range is 400–2,500 nm, while the 1265 range is 850–1,050 nm.

Additionally, the models differences within these spectral ranges will be discussed, as will the use of limited portions of the spectral ranges. A further trial was made to find the minimal instrument solution. That is, for a given level of accuracy, how few data points are required. This could help in the development of smaller, less expensive instruments to measure rice quality. There has been no exhaustive effort to minimize model errors by pre-processing the data. Models were developed with default options, log 1/R (no smoothing) or second derivative, 4-nm gap, and 4-nm smoothing. SECV was used as the measure of model difference (Martens and Naes 1989). Since the reference data set is the same for all models, and the number of samples is the same, this is a simple and valid means of judging the models developed for the five geometries. In all cases, seven cross validation passes were made. This gives 103 samples in calibration and 17 samples in validation for each pass. The final model is developed from the number of factors chosen by cross validation applied to all 120 samples.

Reflectance Models

The spectra in Fig. 1 show the difference in the three reflectance geometries. There is very little difference between the 6500 spinning cup (65SC) and transport bulk (65RB) cells. The spinning cup absorbs more in the visible portion of the spectra, and both are close to totally absorbing at the end of the NIR region. The spinning cup flour (65RF) is lower absorbing and reflects more spectral character, as can be seen at 1,960 nm, (water O-H) and 2,310 nm (aliphatic C-H). While not resolved, the starch (2,104 nm, C-O-H) and protein (2,168 nm, C=O, N-H) band is narrower. The first three columns of data in Table II also depict the differences in SECV information for the three 6500 geometries. While reasonable SECV values can be seen for all spectral geometries, the 65RF has the lowest protein SECV and the highest SECV for whiteness. For the ground rice samples, there is much less difference in whiteness appearance between samples than for whole kernels, thus a higher SECV. The differences in all the models for 65RB

TABLE III
Standard Error of Cross Validation for Whole-Grain Milled Rice and Rice Flour for Different Sample Geometries and Software Systems in the 1,100–1,700 nm Range^a

Constituent	6500 Reflectance (65RB)	6500 Spinning Cup (65SC)	6500 Spinning Cup Flour (65RF)	6500 RTCAL	1265 Transmission	6500 Transmission
Amylose	0.37	0.43	0.53	0.59	0.53	0.55
Protein	1.12	0.22	0.15	0.22	0.18	0.21
Taste value	2.64	3.68	3.74	4.15	4.32	3.43
Whiteness	1.28	0.98	1.9	0.59	1.01	4.43
Transparency	0.19	0.14	0.2	0.17	0.19	0.21
Milling degree	6.3	4.2	8.2	2.55	4.29	9.9

^a Range 850–1,050 nm used for comparison. Two visible/near-infrared scanning monochromators (NIRSystems 6500 and Tecator Infracore 1265) were used to collect reflectance and transmittance readings. Full spectrum calibration models developed by multivariate method of partial least squares (PLS1), with cross validation used to determine standard error of performance. RTCAL = real-time calibration results.

TABLE IV
Standard Error of Cross Validation for Whole-Grain Milled Rice and Rice Flour for Different Sample Geometries and Software Systems^a with Reduced Spectral Data Sets^b

Constituent	6500 Reflectance (65RB)	6500 Spinning Cup (65SC)	6500 Spinning Cup Flour (65RF)	1265 Transmission	6500 Transmission	6500 Reflectance (700)
Amylose	0.108 (53)	0.542 (15)	0.567 (208)	0.338 (18)	0.479 (20)	0.55
Protein	0.096 (53)	0.191 (15)	0.203 (15)	0.141 (23)	0.202 (20)	0.21
Taste value	3.471 (33)	5.487 (15)	2.298 (15)	2.616 (89)	3.906 (20)	3.43
Whiteness	0.130 (208)	2.878 (170)	1.769 (34)	0.978 (18)	2.225 (25)	4.43
Transparency	0.010 (208)	0.168 (170)	0.412 (68)	0.135 (44)	0.092 (25)	0.21
Milling degree	0.078 (208)	12.35 (170)	8.410 (34)	3.617 (19)	10.59 (25)	9.9

^a Two visible/near-infrared scanning monochromators (NIRSystems 6500 and Tecator Infracore 1265) were used to collect reflectance and transmittance readings. Full spectrum calibration models developed by multivariate method of partial least squares (PLS1), with cross validation used to determine standard error of cross validation.

^b Numbers in parens indicate the number of spectral data points used in the model. Data points were selected by the use of every *i*th data point, where *i* = 2,4,8,16, etc.

and 65SC can be explained by sampling. The 65RB scans a 120-g sample, while the 65SC sees only ≈ 20 g, thus all the SECV values for the 65SC are somewhat larger. One anomaly is the milling degree model for 65RF. Since milling degree is closely associated with lipids, we would expect the geometry whose spectra most clearly depicts the aliphatic C-H stretch to be superior (Wadsworth 1993). However, the best model was the 65RB, and the 65RF SECV was almost twice as large.

Transmission Models

The spectra labeled in Fig. 2 depict the differences between the reflectance (dashed) and transmission (dash-dotted) geometries for the 6500. The transmission spectra are higher absorbing even though it was taken through a monolayer of grain. The transmission curve does not show it, but the spectra are quite noisy beyond 2,000 nm. The main advantage in theory of a transmission mode is that it eliminates the scatter phenomena associated with reflectance spectra and, thus, will have simpler spectral preprocessing for chemometrics. The data in column 5 of Tables II and III and column 6 of Table III allow for the comparison of transmission models among instruments (65TBand 1265) and comparison to reflectance models among geometries. The 6500 data $>1,700$ nm were discarded because of excess noise. Both instruments in transmission mode gave reasonable and similar SECV values for amylose (0.53 and 0.55) and protein (0.18 and 0.21). In Table II, compared to the reflectance geometries, the amylose and protein values were ≈ 0.53 and 0.20, respectively. Taste value, whiteness, and milling degree were poor for both instruments when compared to the reflectance SECV values of Table II. Sample size was not an issue here, as the 6500 sample was only a few grams, while the 1265 sample was 150 g.

TABLE V
Standard Error of Cross Validation for Cooked Rice^a

Constituent	1265 Transmission	6500 Reflectance (65RB)	6500 Reflectance (uncooked)
Amylose	0.470 (50)	0.362 (47)	0.52
Protein	0.115 (15)	0.197 (525)	0.22
Taste value	3.320 (50)	4.218 (108)	3.22
Whiteness	2.418 (20)	2.509 (103)	0.65
Transparency	0.131 (50)	0.257 (15)	0.13
Milling degree	11.47 (20)	9.287 (23)	2.89

^a Two visible/near-infrared scanning monochromators (NIRSystems 6500 and Tecator Infracat 1265) were used to collect reflectance and transmittance readings. Full spectrum calibration models developed by multivariate method of partial least squares (PLS1), with cross validation used to determine standard error of performance.

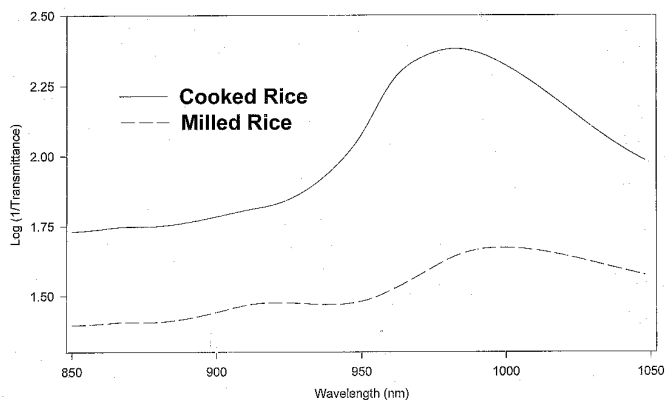


Fig. 3. Transmission spectra of milled and cooked rice from a Tecator Infracat 1265 spectrometer at 850–1,050 nm in bulk whole kernel geometry.

RTCAL

The real-time calibration results in Tables II and III compare favorably to the models obtained in the usual fashion. The RTCAL modeling approach has several advantages. First, each sample is matched to a subset and a specific calibration developed for it. Thus, in theory the most precise determination will be made. Second, outliers are reduced and detection enhanced, because if an insufficient subset is chosen, no result will occur. Third, since the library is a single file, transferring it from one instrument to another is easily facilitated. Perhaps the best advantage is that it gives solutions equivalent to a neural network, and the results are easily understood. That is, you can determine that the samples chosen were appropriate and that the model is performing properly. This technique is further explained by Isaksson and Naes (1988), Naes et al (1990), Naes and Isaksson (1992), and Shenk and Westerhaus (1991a,b).

Reduced Data Sets

The reduction of data or truncation of the spectral files to eliminate the noisy portion of the transmission spectra for the 6500 prompted us to try the other geometries over the same spectral region (1,100–1,700 nm). The results in Table III are quite mixed. The 65RB gave a slightly better SECV for amylose, protein was a quite a bit higher for the bulk cell reflectance geometry, a little higher for the other reflectance geometries, and higher for both transmission instruments. Of the milling properties, only the SECV for transparency was adequate, the other models were much worse. Since the visible portion and the aliphatic C-H region were not represented in the 1,100–1,700 nm range this is quite understandable.

Table IV shows the data for the systematic elimination of data from the spectral set. The 6500 collects 1,150 data points from 400–2,500 nm. The 1265 collects 100 data points from 850–1,050 nm.

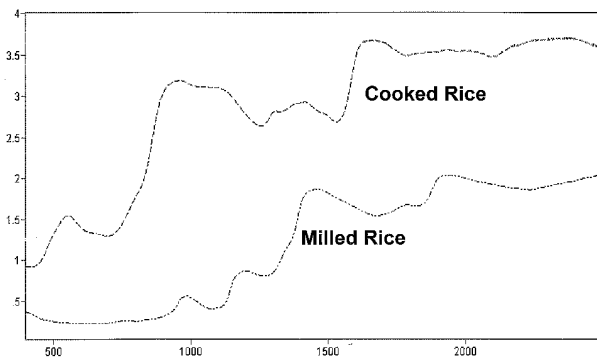


Fig. 4. Reflectance spectra of milled and cooked rice from an NIRSystems 6500 spectrometer at 400–2,500 nm in the bulk whole kernel geometry.

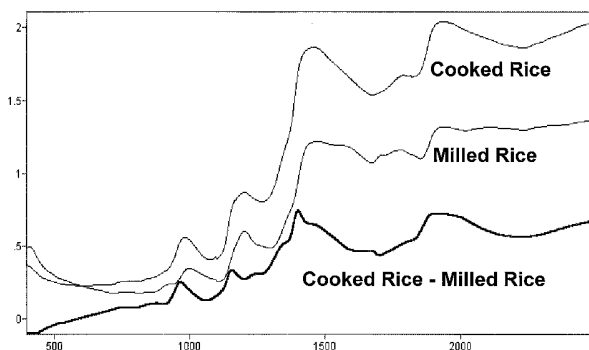


Fig. 5. Reflectance spectra of milled and cooked rice from an NIRSystems 6500 spectrometer at 400–2,500 nm in the bulk whole kernel geometry and the difference spectrum (bold line).

By picking every i th data point with $i = 1, 2, 3, \dots, n$, it is possible to reduce the data set to a small number of wavelengths spaced evenly and distributed across the spectrum. In the case of the 6500, the bandpass is 10 nm and data is taken every 2 nm, so the spectrum is oversampled by a factor of 5. The 1265 has an 8-nm bandpass and takes data every 2 nm, which is a spectral oversampling by a factor of 4. The number of data points in each spectral set was systematically reduced until the SECV began to increase. The values in Table IV show the results of minimizing the number of data points. In theory, if signal-to-noise is sufficient, only 20–25% of the data will give as good a result as 100% of the data. This appears to be the case for some components such as amylose and protein with the 65RB (0.108 and 0.096, respectively, with 53 data points; $i = 20$). The fact that taste value could be modeled for all but the 1265 with 15–33 data points, indicates that the data requirement, for “taste” as such may not be very large. The determination of milling properties with the various geometries and limited data sets was quite varied, particularly milling degree. More data points were required for milling degree and still some values for SECV were considerably higher. The implication of this is that much simpler instruments could be used to determine some of the quality properties of milled rice.

Cooked Rice

The results in Table V for cooked rice indicate that adequate models can be developed on cooked rice for compositional properties, for transparency, but not for whiteness, and milling degree. Since cooking properties are altered by milling, it is logical that the milling properties cannot be determined with cooked rice (Perez et al 1993, 1996). Figure 3 depicts the differences between the cooked and milled rice in transmission mode for the 1265. The spectra are almost featureless, except for the bands for water. There is not sufficient structure to the spectra to say anything other than there is additional water. The milled rice spectrum shows some structure at ≈ 930 nm, which is probably associated with lipid. Figure 4 from the 6500 shows more spectral detail. Here, it can be seen at $\approx 1,400$ and $1,900$ – $2,000$ nm that there is a difference in water content, but also that the water is bound to the rice in multiple ways. That is, water bound to protein absorbs at a longer wavelength than water bound to starch, and that the bands are somewhat resolved at 1,430 and 1,445 nm. The apparent lipid in the milled rice is also obvious at 930 and 1,730 nm. If the difference spectra (i.e., spectrum of cooked rice minus the spectrum of milled rice) is overplotted as in Fig. 5, these differences are enhanced and the hydrogen bonding bands are quite pronounced at 960, 1,200, 1,430, and 1,920 nm.

CONCLUSIONS

All of the instruments, even the 1265, which is optimized for meat, not grains, did remarkably well. No one instrument geometry was best for all analyses. The best instrument-geometry performance was the 6500 in reflectance mode with the bulk transport half cell. It appears that the chemical composition of rice is best measured in the 1,100–2,500 nm range, and the physical milling properties best measured in the 850–1,050 nm range. The most important conclusion is that the reduced spectral regions and small sets of data points spread across the spectrum give equivalent and, in some cases, improved results. Thus, simple sensors are quite feasible for specific analyses.

For the next study to try to expand the models to establish spectroscopic standards, the 65RB geometry and the 65RF geometry will be used as the base methods. A new instrument from Tecator (Infracore 1229) specifically used for grains will also be employed. Thus, the models developed will be compared for instruments currently used in the regulatory process. The reduced data approach

will be further investigated to determine the minimum instrumental requirements for the analysis of rice.

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