Development and Validation of Prediction Models for Rice Surface Lipid Content and Color Parameters Using Near-Infrared Spectroscopy: A Basis for Predicting Rice Degree of Milling

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ABSTRACT

Degree of milling (DOM) of rice plays a key role in determining rice quality and value. Therefore, accurate, nondestructive, quick, and automated surface lipid content (SLC) measurement would be useful in a commercial milling environment. This study was undertaken to provide calibration models for commercial use to provide quick and accurate evaluation of milled rice SLC and Hunterlab color parameters *(L,a,b)* as indications of rice DOM. In all, 960 samples, including seven cultivars from seven southern United States locations, stored for 0, 1, 2, 3, and 6 months, were milled for four durations to obtain samples of varying DOM. The samples were used to develop calibration models of milled rice SLC and *L,a,b* values. Another sample set (n = 58) was commercially milled and used to validate the developed models. A DA 7200 diode array analyzer was used to scan milled rice samples in wavelength spectra of 950–1,650 nm. SLC and color parameters were measured using a Soxtec system and a HunterLab colorimeter, respectively. The partial least squares regression (PLS) method using the full near-infrared spectra was used to develop prediction models for rice SLC and color parameters. Milled rice SLC was well fitted with a correlation of determination of predicted and measured values of (R^2 = 0.934). Color parameters were also successfully fitted for L (R^2 = 0.943), a (R^2 = 0.870), and b (R^2 = 0.855). Performance of the developed models to predict rice DOM was superior in predicting SLC and *L,a,b* values with R^2 predicted and measured values of 0.958, 0.836, 0.924, and 0.661, respectively.

During the rice milling process, the outer layers, or the bran, of rice kernels are removed, yielding milled, white rice. The milling efficacy is measured by degree of milling (DOM). The amount of bran remaining on the rice kernel after milling affects rice quality, appearance, and texture and, thus, milled rice end-use preference of different consumers (Chen et al 1997; Perdon et al 2001; Saleh and Meullenet 2007). Various methods have been used to measure rice DOM, such as by visual examination (USDA 2005), measuring the mass lost or the percentage of bran removed during milling (Wadsworth et al 1991), and use of optical measurements (Stermer 1968; Wadsworth et al 1991; Siebenmorgen and Sun 1994; Delwiche et al 1996). Chemical composition analysis, including thiamin, phosphorus, and total and surface lipid content, has also been used to measure DOM (Chen et al 1997; Matsler and Siebenmorgen 2005; Wang et al 2006). However, these methods can be costly, requiring labor-intensive sample preparation and techniques. Moreover, chemical analyses, though presumably more accurate than optical measurements, require the use of chemicals and are difficult to use online in a production environment. Therefore, the search for nondestructive, rapid tools that require limited sample preparation has attracted researchers' attention (Delwiche et al 1995, 1996; Bergman et al 2004).

The use of near-infrared spectroscopy (NIRS) has generated accurate and consistent results in determining various characteristics of agricultural crops such as apparent amylose content (Delwiche et al 1996; Wu and Shi 2004), amino acids (Barton et al 2000; Wu et al 2002), lipids (Chen et al 1997; Wang et al 2006), moisture content (Natsuga and Kawamura 2006), and starch quality parameters (Bao et al 2001). Prediction of cooked rice sensory texture attributes and rice flour pasting properties was evaluated using NIRS (Delwiche et al 1996; Windham et al 1997; Meadows and Barton 2002; Meullenet et al 2002; Wu and Shi 2007). NIRS-measured samples including milled (Delwiche et al 1996; Windham et al 1997) and brown rice grain (Wang et al 2006), milled and brown rice flour (Delwiche et al 1995), and single rice grain (Wu and Shi 2004) were used to predict various rice characteristics.

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Combinations of optical and near-infrared (NIR) techniques have also been used to predict rice quality characteristics, including DOM (Siebenmorgen and Sun 1994; Delwiche et al 1996; Chen et al 1997; Windham et al 1997; Barton et al 2000). Chen et al (1997), for instance, correlated NIRS results with DOM measured using a milling meter, which uses the amounts of reflected and permeated light to calculate DOM. The authors correlated this milling meter reading to milled rice surface fat concentration and reported a linear and inverse correlation of determination (R^2) > 88%. Delwiche et al (1996) also used a visible/NIR scanning monochromator to correlate milled rice whiteness and DOM as measured using a milling meter (r = 0.98). Gangidi et al (2002), in the same manner, used diffuse reflectance Fourier transform infrared (DFTIR) to develop models to predict milled rice surface lipid content (SLC).

Although rice characteristics were well predicted using these developed models, limited sample numbers and variability were used: n = 120 in Windham et al (1997); n = 61 in Natsuga and Kawamura (2006); n = 248 in Wang et al (2006). This restricted the suitability of these models to predict rice properties over cultivars, locations, and years. In addition, the various ranges of visible and NIR wavelength that were used in these studies could generate interference and potentially decrease the accuracy of prediction. Furthermore, no or limited NIRS equipment has been available commercially for use in rapid assessment of rice DOM.

Therefore, the objective of this study was to develop calibration models of milled rice SLC and Hunter color parameters (*L,a,b*) using NIRS that would allow the determination of rice DOM. More specifically, the objectives of this work were to 1) collect scans over a wide range of infrared wavelengths of a large variety of rice samples, including different cultivars and storage durations, milled for various durations to obtain various SLCs and color ranges; 2) develop calibration models for milled rice SLC and *L,a,b* color measurements using scans of milled rice and its correspondent SLC and color values; and, finally, 3) validate these calibration models using commercially milled rice samples.

MATERIALS AND METHODS

Sample Procurement and Pretreatment Conditions

Twenty-five rough rice lots consisting of seven cultivars and hybrids were harvested from four 2003 and five 2004 Arkansas or Missouri locations as part of a field-scale cultivar-testing program.

Table I shows a list for the experimental design. Harvest moisture contents (MC) were 18–20%. After harvest, samples were dried in a chamber maintained at 21°C and 62% rh, corresponding to a rough rice equilibrium MC ≈12.5%. Actual dried rough rice MC was 11.5–13.0%, determined as the average MC of 50 kernels measured with an individual kernel MC meter (CTR 800E; Shizuoka Seiki, Shizuoka, Japan). After drying, the samples were stored in plastic bags at 4°C until treatment.

Storage, Milling, and Head Rice Yield Determination

Samples were milled after 0, 1, 2, 3, and 6 months of storage. Before milling, samples from each of the rice lots were removed from storage and placed at room temperature for at least one day. After each storage period, duplicate rough rice samples (150 g) from each of the 25 sample lots were then dehulled in a laboratory sheller (Type THU, Satake, Tokyo) and subsequently milled for 10, 15, 20, and 40 sec in a laboratory mill (McGill #2; Rapsco, Brookshire, TX) to obtain samples of varying degrees of milling. The mill had a 1.5-kg weight on the lever arm situated 15 cm from the milling chamber. Excess bran and endosperm were removed from each milled sample duplicate using an aspirator (Grain Blower, Seedburo Equipment, Chicago, IL). Head rice was then separated from brokens with a sizing device (Seedburo) and placed in Ziplock bags at 4°C until SLC could be determined.

Surface Lipid Content Measurement

Each sample was removed from 4°C storage and, while in sealed bags, left to equilibrate to room temperature for at least 2 hr before SLC analyses. The SLC of duplicate head rice aliquots from each duplicate sample lot, storage duration, or milling duration was measured using a lipid extraction system (Soxtec Avanti 2055, Foss North America, Eden Prairie, MN) as in Matsler and Siebenmorgen (2005). SLC was expressed as the mass percentage of extracted lipid to the original head rice sample mass. Duplicate SLC measurements were averaged before data analysis.

Color Measurements

The *L* (white to black), *a* (red to green), and *b* (yellow to blue) color profiles of each sample lot, storage duration, and milling duration duplicate was measured using a colorimeter (Hunter Associates Laboratory, Reston, VA). Three readings were performed on each sample, which were averaged before further data analyses.

Spectroscopic Analyses

Milled rice samples were scanned using a diode array analyzer (DA 7200, Perten Instruments, Huddinge, Sweden). Each milled rice sample (≈60 g) was fitted in a 75-mm diameter cup that rotated during NIRS scanning. Absorbance readings at 5-nm wavelength increments were collected over an NIR wavelength range of 950–1,650 nm (141 data points were recorded). Three scans were conducted on each sample and averaged before data analyses.

Calibration Model Development and Data Analysis

For each of the sample lots at each storage duration and milling duration, SLC and *L,a,b* results were used to develop calibration models to predict these rice attributes using the NIRS scan data. Using multivariate regression software (Unscrambler, v.9.2, Camo, Oslo, Norway), partial least squares (PLS1) (where one variable is modeled) was performed to develop prediction models for milled rice SLC and PLS2 (where more than one variable is modeled) for milled rice *L,a,b*. Absorbance values were standardized by weighting each with the standard deviation so that all variables

TABLE II
Model Statistics for Prediction of Rice Surface Lipid Content (SLC) and Hunter Colorimeter Parameters (*L,a,b*) of Milled Rice[a]

Parameters[b]	SLC	*L*	*a*	*b*
Calculated				
Min value	0.20	61.0	–0.80	12.23
Max value	0.90	76.6	4.23	22.52
R_c	0.968	0.971	0.933	0.924
RMSEC	0.051	0.649	0.241	0.504
SEC	0.051	0.649	0.241	0.504
Bias	5.512e-07	–3.450e-06	8.522e-07	–1.380e-06
SD_c	0.203	2.752	0.683	1.344
Validated				
Min value	0.19	62.80	–0.810	13.39
Maxvalue	1.06	77.20	1.960	19.52
R_v	0.966	0.970	0.928	0.921
RMSEP	0.052	0.666	0.249	0.515
SEP	0.052	0.667	0.249	0.516
Bias	–6.439e-05	0.001470	–0.000422	4.154e-05
SD_v	0.197	2.647	0.624	1.225
RPD_c	3.90	4.13	2.74	2.61

[a] Using DA 7200 diode array analyzer (n = 960) (calibration sample set).
[b] R_c, calculated correlation; RMSEC, calculated root mean square error; SEC, standard error of calculation; SD_c, calculated standard deviation of each SLC and *L,a,b* parameters; R_v, validated correlation; RMSEP, predicted root mean square error; SEP, standard error of prediction; SD_v, validated standard deviation used to build calibration models to predict milled rice SLC and *L,a,b* color measurements; and RPD, relative ability of prediction, a discrimination index ($RPD = SD_c/SEP$).

TABLE I
Rice Cultivars or Hybrids, Harvest Season, and Harvest Location Summary of Rice Samples Used to Develop Surface Lipid Content (SLC) and *L,a,b* Hunter Color Calibration Models

2003		2004	
Cultivar	**Location**	**Cultivar**	**Location**
CF-XL8 (long-grain hybrid)	Davidson Essex Lodge Corner	XP723 (long-grain hybrid)	Essex Hazen Newport
Cocodrie (long-grain cultivar)	Davidson Riggs	Bengal (medium-grain cultivar)	Jonesboro Lodge Corner
Francis (long-grain cultivar)	Davidson Essex Lodge Corner	Cocodrie (long-grain cultivar)	Essex Hazen Newport
Wells (long-grain cultivar)	Davidson Essex Lodge Corner	Wells (long-grain cultivar)	Essex Hazen Newport
		XP716 (long-grain cultivar)	Jonesboro Lodge Corner

[a] Samples stored for 0, 1, 2, 3, and 6 months and subsequently milled for 10, 15, 20, and 40 sec in duplicate to achieve samples of different degrees of milling.

were given equal influence on the predicted variables. Full cross-validation was employed to validate the predictive ability of the calibration models. In this approach, each sample was used to test the model derived from all other samples. The deviation from the expected value as a result of excluding each sample from the models was measured. This process was repeated so that each calibration value was excluded once, to test whether its removal had seriously affected the model. A root mean square error (RMSE) of cross-validation was then calculated. The uncertainty test was also performed during the full cross-validation computation to assess stability of the results. These procedures allowed for the removal of predicted variables that either did not influence the prediction or created interference in the model. This technique has also reduced the uncertainty in the prediction models and, in most cases, improved the validation statistics. The number of principle predictors used in the PLS calibration models were selected as suggested by the Unscrambler software. Calibrated and validated coefficient of determinations (R^2) and RMSE values were obtained to evaluate each calibration model. Meullenet et al (2002) provides more detail on model development and NIRS data treatments.

Validation of SLC and Color (*L,a,b*) Calibration Models

Another sample set of 58 commercially milled rice samples, collected from rice milling cooperatives and companies across the mid-South and California with a wide range of DOM, were used to validate the calibration models developed using the first sample set. These commercially milled samples were scanned using the same diode array analyzer, and the SLC and color measurements were measured as described previously. Milled rice SLC and *L,a,b* were predicted using the models developed earlier using the prediction option provided by the Unscrambler software.

Calculated and predicted root mean square error (RMSEC and RMSEP, respectively), standard error of prediction (SEP), R^2, bias (indicating systematic errors) of measured and predicted values, and the relative ability of prediction (RPD) (also known as the discrimination index, where RPD = SD_c/SEP) (SD_c is the standard deviation of each SLC and *L,a,b* parameter]) were used to assess the prediction accuracy of the calibration (Williams and Soebering 1993; Locher et al 2005).

RESULTS AND DISCUSSION

SLC and *L,a,b* Calibration Models

Scatter plots of measured and predicted parameter values of milled rice samples used to develop the NIRS calibration models are shown in Fig. 1, with the model statistics presented in Table II. SLC and *L,a,b* of milled rice samples were 0.20 to 0.90%, 61.0 to 76.6, –0.80 to 4.23, and 12.23 to 22.52, respectively. Moreover, results indicated that SLC had pleasingly high calibrated (R_c) and validated (R_v) correlations of 0.968 and 0.966 with a correspondent RMSEC and RMSEP of 0.051 and 0.052, respectively, indicating the fitness of the developed model in predicting milled rice SLC. Predicted and measured SLC had R^2 = 0.934 with RPD = 3.90 (Table II), indicating that the range of variation in SLC was significantly greater than the prediction error. Sitakalin and Meullenet (2000) reported that RPD values >2.0 are acceptable. The calibrated models had correlations of 0.971, 0.933, and 0.924 for *L, a*, and *b* with a corresponding RMSEC of 0.649, 0.241, and 0.504, respectively. Predicted and measured *L,a,b* had R^2 = 0.943, 0.870, and 0.855. RPD of the predicted values were 4.13, 2.74, and 2.61 for *L, a*, and *b*, respectively. These results are sufficiently high to predict milled rice color parameters.

Several factors affect accuracy and robustness of NIR calibration models. These factors were reviewed by Wu and Shi (2007) and include different sample geometries (whole kernel vs. ground sample), sample status, sample selection, and chemometric methods. Sample-affecting variables such as different cultivars and different storage and milling durations (resulting in a higher number of samples) were used in this study.

Model statistics of the PLS regression developed using NIR scans of milled rice samples are shown in Table I. These values are greater than those of Wang et al (2006), who reported validated

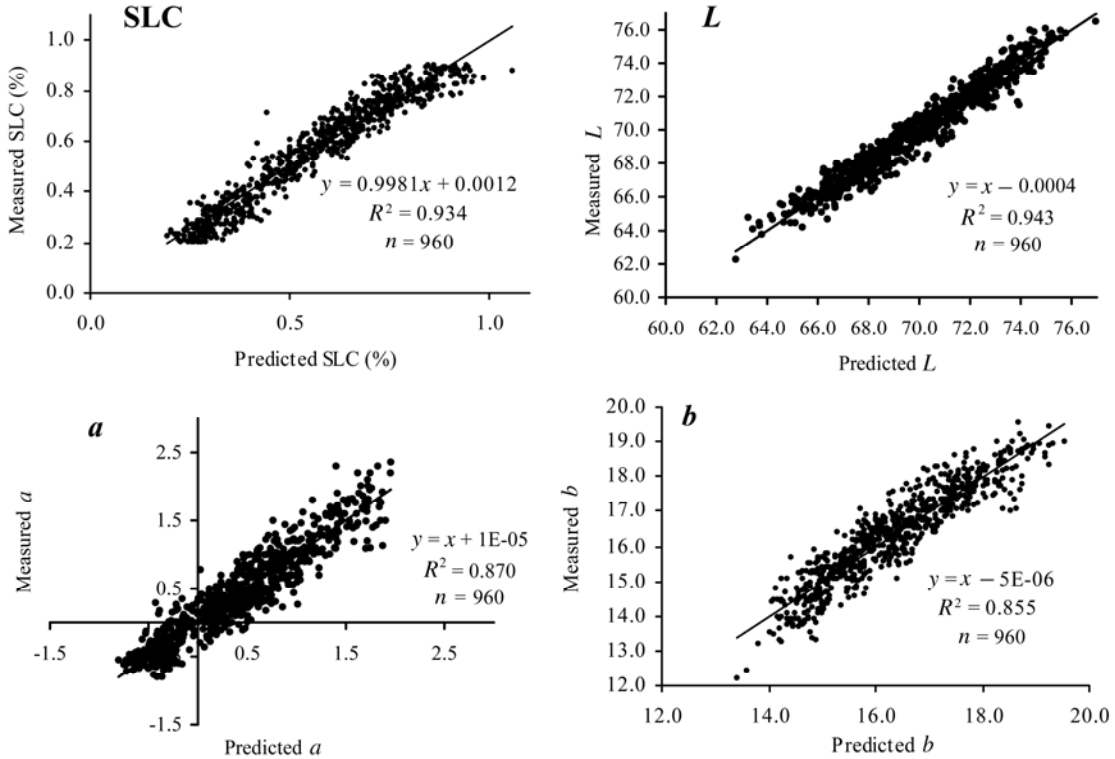

Fig. 1. Predicted versus measured surface lipid content (SLC) and *L,a,b* Hunter color measurements of milled rice samples (n = 960) used to build prediction models for milled rice SLC and *L, a,* and *b,* respectively.

correlation of determination of 0.81 (R_v = 0.90) for the determination of fat content in milled rice. Corresponding RMSEC and RMSEP values were 0.090 and 0.120, respectively, which are greater than what we are reporting here (0.051 and 0.052). These results could be due the smaller sample size (n = 201) and the use of short-, medium-, and long-grain rice that probably reduced the accuracy of the calibration model reported by Wang et al (2006). Moreover, Wang et al (2006) used a Fourier transform infrared spectrometer (model Vector/22N, Bruker, Berlin, Germany) to collect absorbance spectra over a wider wavelength range (400–1,200 nm), which probably resulted in interference in the spectra that was included in their model compared with the narrower wavelength range used here (950–1,650).

TABLE III
Model Statistics for Prediction of Rice Surface Lipid Content (SLC) and Hunter Colorimeter Parameters (*L,a,b*) of Commercially Milled Rice Samples[a]

Parameters[b]	SLC	*L*	*a*	*b*
Calculated				
Min value	0.22	67.74	–0.91	14.74
Max value	0.95	79.95	1.64	17.73
SD_c	0.161	1.942	0.502	0.768
Validated				
Min value	0.18	67.45	–0.41	14.01
Max value	0.78	75.57	2.76	17.05
R_v	0.979	0.915	0.961	0.813
RMSEP	0.0570	0.814	0.640	0.988
SEP	0.0389	0.809	0.199	0.449
Bias	–0.0421	–0.142	0.609	–0.882
SD_v	0.137	1.968	0.626	0.658

[a] Using DA 7200 diode array analyzer (n = 58) (validation sample set).
[b] SD_c, calculated standard deviation of each SLC and *L,a,b* parameter; R_v, validated correlation; RMSEP, predicted root mean square error; SEP, standard error of prediction; SD_v, validated standard deviation used to build calibration models to predict milled rice SLC and *L,a,b* color measurements.

Models Validation Using Commercially Milled Rice Samples

Model statistics of SLC and *L,a,b* of commercially milled samples as predicted using previously developed models are shown in Table III. Measured SLC and *L,a,b* of this sample set were 0.22 to 0.95%, 67.74 to 75.95, –0.91 to 1.64, and 14.74 to 17.73, respectively. SLC was well predicted using the developed model with a validated correlation (R_v) of 0.979 and corresponding RMSEP and SEP values of 0.0570 and 0.0389, respectively. The validated correlations of *L,a,b* were 0.915, 0.961, and 0.813, with corresponding RMSEP of 0.814, 0.640, and 0.988, respectively.

A scatter plot of measured SLC and *L,a,b* color and predicted values using our developed calibration models is shown in Fig. 2. Predicted and measured values were highly correlated, with correlations of determination of 0.958, 0.836, 0.924, and 0.661, respectively, for SLC, *L,a,b*. These results indicate the appropriateness of the developed models in providing accurate prediction of milled rice SLC and color measurements (hence DOM) in a very short time (i.e., time required to scan a milled rice sample ≈ 6 sec).

Although Wang et al (2006) reported calibration models for fat content in rice, the validation of these models was not reported on any sample sets other than those used to develop the calibration models. Natsuga and Kawamura (2006) also developed prediction models used to measure physicochemical properties of Japanese short-grain nonwaxy rice. The authors reported acceptable predictions of milled rice color parameters (*L,a,b*). However, they used a visible wavelength range of 400–798 nm. Use of longer wavelength ranges (800–1,098 or 1,100–2,498) significantly lowered color parameter prediction. In addition, they reported validated correlations (R_v) of 0.88, 0.74, and 0.91 and RPD values of 2.1, 1.5, and 2.5 for *L,a,b*, respectively, which were lower than those achieved in this study: R_v = 0.970, 0.928, and 0.921; RPD = 4.126, 2.743, and 2.605, respectively). The lower validation correlation reported by Natsuga and Kawamura (2006) was probably due to smaller sample size (n = 61), type of rice (short-grain and waxy rice), and the wide wavelength range compared with a larger sample size and wider sample variability used in this study.

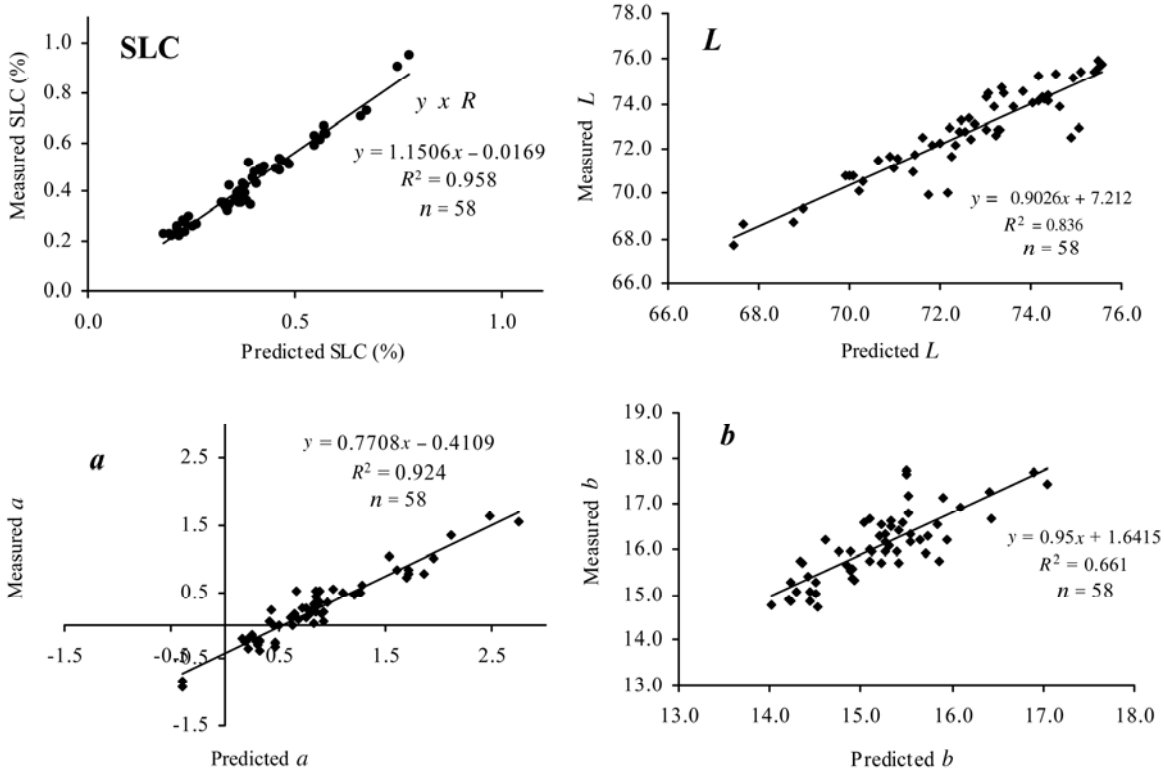

Fig. 2. Predicted versus measured surface lipid content (SLC) and *L, a,* and *b* Hunter color measurements of commercially milled rice samples (n = 58) predicted using calibration models generated using scanned absorbance 960 milled rice samples.

Delwiche et al (1996) reported high validated prediction of milled rice color measured in term of R^2 (0.966 and 0.927) for milled rice whiteness and transparency, respectively. Values for whiteness, transparency, and milling degree were measured on a milling meter (MM-1B, Satake), where a shorter wavelength range (450–1,048 nm) was used. Delwiche et al (1996) indicated an improvement of the prediction models when using a wavelength range of 450–1,048 nm. The improvement was probably due to wavelength overlapping when the meter was used to measure milled rice whiteness, which is perceived in the visible light range (400–700 nm), rather than in the longer nonvisible wavelength range (1,100–1,800 nm). However, a smaller sample number (n = 196) was used to generate these prediction models with no indications of *L,a,b* color parameters.

CONCLUSIONS

Previous NIRS calibration research that focused on a prediction of rice functional properties had small sample sets with limited variability or used a wider range of NIR wavelength that, in many cases, created interference that affected the prediction models. In this study, a large sample number with a wider milling range and a narrower NIR wavelength range was used to maximize calibration robustness of the developed models. The use of NIRS (DA 7200) provided a superior prediction model of milled rice SLC and *L,a,b* color parameters, indications of rice DOM. This is probably the first report on the availability of calibration models to predict milled rice DOM for commercial use. The use of the NIRS method for predicting milled rice SLC and color parameters would provide a fast and accurate tool to monitor rice DOM and, thus, head rice yield (HRY), the amount of kernels ≥75% of the original length (USDA 2005). For instance, Cooper and Siebenmorgen (2006) related changes in rice SLC to HRY. Cooper and Siebenmorgen (2006) indicated that HRY of a rice lot can be adjusted by a factor of 9.4 for every percentage point difference between rice SLC and a target one. Therefore, the use of NIR calibration models would provide significant computing of rice HRY in a very short time. Rice DOM also have effects on rice end-use properties. For example, Saleh and Meullenet (2007) and Kim et al (2001) demonstrated the relationship between rice DOM and cooked rice texture properties. A decrease in cooked rice firmness with increased milling duration was reported. The availability of accurate calibration models to predict rice DOM will provide significant tools to mill rice to target functional properties.

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