Quantifying Surface Lipid Content of Milled Rice via Visible/Near-Infrared Spectroscopy[1]

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ABSTRACT

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Visible/near-infrared calibrations were developed and tested for surface lipid content (SLC) of milled long-grain rice. Three rice varieties were divided into two sample sets, with one containing two variables (degree of milling and variety) and another containing three variables (degree of milling, variety, and kernel thickness). The reflectance calibration equation from the set with three variables was much more accurate in predicting SLC than was the calibration from the two-variable set.

Optimal calibration and prediction were obtained by combining both visible and near-infrared wavelength ranges and using the modified partial least squares technique on spectra pretreated by standard normal variate and first derivative methods. The best calibration yielded a coefficient of determination (R^2) of 0.99 and a standard error of prediction of 0.04% SLC, which was approximately 1.5 times the standard error of calibration and also 1.5 times the SLC measurement error.

Degree of milling (DOM) is a measure of the extent to which rice bran has been removed from brown rice during milling. It is an attribute that is extremely important to both the rice industry and consumers. The amount of bran remaining affects the stability, quality, and value of the product, with respect to appearance and end-use functionality. Definitions for DOM can include lost bran mass, visual changes (i.e., color), and chemical assays (including surface lipid content [SLC], a measure of residual bran on the kernels). Each definition has advantages and disadvantages, depending on the specific needs of the processor and the end-user.

In terms of bran mass, DOM can be evaluated as a percent mass lost from brown rice during milling (Wadsworth et al 1991). However, this method does not lend itself to continuous on-line measurements. Additionally, the results can be influenced by the amount of broken rice (i.e., starchy mass) that exits the mill with the bran.

Visual examination is the current standard method used by the Federal Grain Inspection Service (USDA 1979) and is also a common practice in the milling industry. Even with standard line samples, this method is relatively subjective and personnel dependent. Color is important for whole grain products but can be influenced by factors other than residual bran.

In contrast to the above methods, chemical assays are repeatable and accurate means for evaluating DOM. Chemical analyses measure specific compositional factors (e.g., surface lipids, total lipids, thiamin, and phosphorus) associated with bran removal (Desikachar 1955, Hogan and Deobald 1961, Wadsworth et al 1991, Siebenmorgen and Sun 1994). Although relatively accurate and repeatable, chemical assays are costly and time-consuming for routine or on-line DOM measurements.

Optical or spectroscopic methods are likely to be better suited for rapid analysis of DOM (e.g., in terms of SLC). Optical measurement is based on the light intensity reflected from or transmitted through milled rice. Stermer (1968) found, in the wavelength range of 500 to 1,000 nm, that the greatest changes in transmittance due to milling occur at 660 nm (red color wavelength) and 850 nm (near-infrared). The ratio of light intensity transmitted at these two specified wavelengths was related to color and surface

lipid content, with correlation coefficients of 0.93 and 0.88, respectively. Because such an optical system is based merely on the information from one or two wavelengths, some relevant information hidden in other wavelengths is possibly neglected. It is expected that near-infrared (NIR) spectroscopy, which can use the information from multiple wavelengths or even a whole wavelength range, would provide a more accurate means for measuring DOM.

NIR spectroscopy has been extensively studied for applications in the grain industry during the last two or three decades. Calibrations for evaluating chemical composition (e.g., protein, moisture, starch, and amino acids) have been developed (Finney and Norris 1978; Williams et al 1983,1984; Kim and Williams 1990; Lamb and Hurburgh 1991; Villareal et al 1994; Delwiche 1995; Delwiche et al 1995,1996). Physical characteristics, such as grain hardness and sensory quality, have also been measured via spectroscopic analysis (Williams 1979, Delwiche 1993, Siska and Hurburgh 1993, Champagne et al 1996, Kawamura et al 1996). Additionally, calibrations have been developed for specific functional characteristics, such as viscosity, gross feed energy, and gelatinization temperature (Kim and Williams 1990, Delwiche et al 1996).

Specific to rice, a small amount of previous work has been published in the area of DOM measurement. Stermer et al (1977) used multiple linear regression methods to compare the reflectance data (800–1,800 nm) with surface lipids, total lipids, and bran removal. Though the correlations between NIR spectra and the three DOM references were not as strong as desired (i.e., <0.84), it was concluded that NIR spectroscopy was promising as a fast, objective method, particularly over the normal range of commercially milled samples. Further research was carried out in the wavelength ranges of 1,100–2,500 nm (Wadsworth et al 1991) and 450–1,048 nm (Delwiche et al 1996). By means of the partial least squares method, calibration equations were established with strong correlations (>0.9) for both the DOM expressed as percent bran removal (Wadsworth et al 1991) and the optical DOM measured by a Satake MM-1B milling meter (Delwiche et al 1996).

These previous calibrations were based on physical (i.e., mass removed) or optical definitions of DOM. In contrast, for further processed products, a chemical definition (i.e., SLC) is more important for product functionality and value. Siebenmorgen and Sun (1994) evaluated the Satake MM-1B milling meter, with respect to measurement of SLC. While strong correlations were reported, they were highly dependent on rice variety. Additionally, no reliable NIR calibration has yet been published specifically for rice surface lipid content. Consequently, the specific objectives of

this project were to evaluate the effect of sample set characteristics (DOM, variety, and kernel thickness) on calibration and prediction outcomes for SLC, and compare spectral pretreatment and regression techniques for optimal calibration and prediction of SLC in milled rice.

MATERIALS AND METHODS

Sample Preparation

Three long-grain rice varieties (Alan, Katy, and Newbonnet) were acquired from a commercial processor at approximately 11–13% moisture content (MC, wet basis). Prior to milling tests, each variety was cleaned in a dockage machine and separated randomly into two sets.

Set one was milled to 39, 35, and 38 different DOM levels for Alan, Katy, and Newbonnet, respectively. For Alan and Katy, rough rice was hulled in a commercial-scale Satake husker/paddy separator (model APS-30CX, Satake USA, Houston, TX). Brown

rice was then milled in a single pass through a commercial-scale Satake mill (model BA-7). The various DOMs were generated by adjusting the locations of the weight on the mill lever arm. For Newbonnet, 150-g rough rice samples were hulled in a McGill sample huller. Brown rice was milled in a McGill No.2 rice mill for durations ranging from 2.5 to 100 sec at intervals of 2.5 sec. A 1,500-g weight was fixed on the mill lever arm 15 cm from the centerline of the milling chamber. By both the commercial and laboratory milling systems, samples ranging from undermilled rice to well milled rice were generated.

Set two was hulled in the Satake husker/paddy APS-30CX separator, and each variety was milled to three DOM levels (low, medium, and high) in the Satake model BA-7 mill. Head rice was separated from brokens via a Satake test rice grader with a $\phi 5.2$ mm long-grain indented cylinder. Subsequently, using a Carter-Day precision sizer (style No. ABF2, Carter-Day Company, Minneapolis, MN), the head rice was separated into six thickness fractions for Alan and Newbonnet (<1.54, 1.54–1.59, 1.59–1.64,

TABLE I
Means, Standard Deviations (SDs), Minimum, and Maximum Values of Surface Lipid Content (SLC, %),
and Number of Samples for the Two Sample Sets

	Set One ($n = 112$)			Set Two ($n = 51$)		
	Newbonnet	Alan	Katy	Newbonnet	Alan	Katy
Mean	0.47	0.66	0.56	0.78	0.75	0.65
SD	0.31	0.30	0.19	0.21	0.21	0.28
Min	0.15	0.14	0.28	0.45	0.42	0.32
Max	1.35	1.14	0.94	1.15	1.12	1.09
n	38	39	35	18	18	15
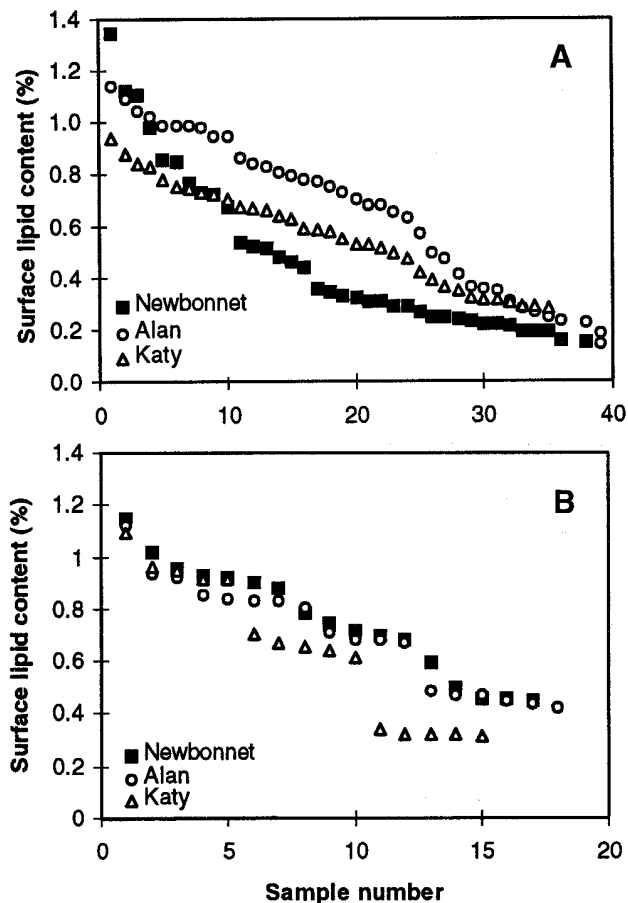
Fig. 1. Surface lipid content data for sample sets one (**A**) and two (**B**). Increasing sample number corresponds to increasing milling time on the laboratory mill or a more restricted flow rate in the commercial mill.
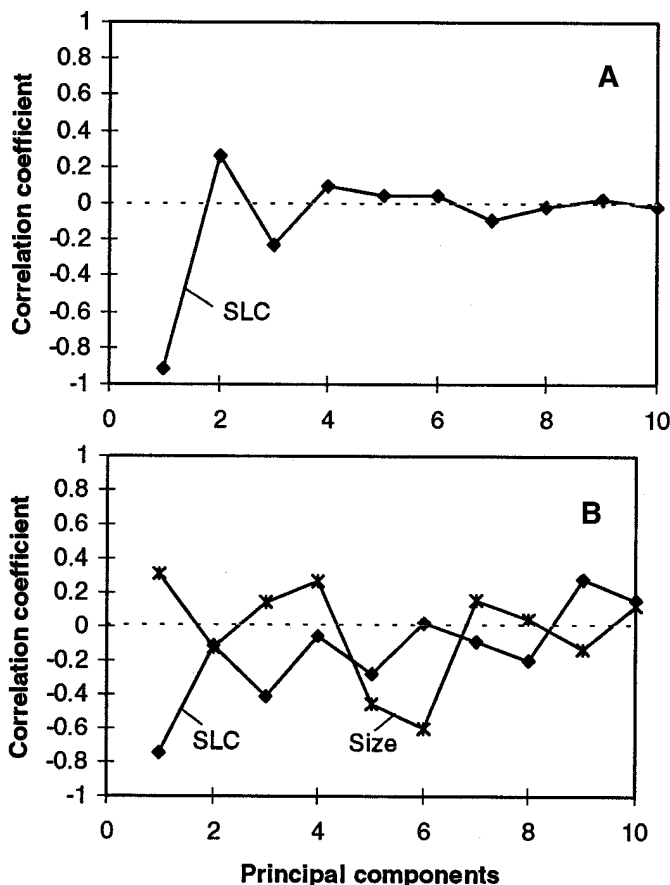
Fig. 2. Correlation coefficients of each principal component with surface lipid content (SLC) and kernel size. **A,** sample set one; **B,** sample set two.

1.64–1.69, 1.69–1.74, and > 1.74 mm) and five thickness fractions for Katy (<1.54, 1.54–1.59, 1.59–1.64, 1.64–1.69, and >1.69 mm).

Thus, set one contained samples at many DOM levels within a range, while set two contained samples with different size fractions at just three DOM levels. The former set involved two independent factors (DOM and variety), and the latter set involved three independent factors (DOM, variety, and kernel thickness).

Visible/Near-Infrared Measurement

After milling, samples were scanned in a scanning monochromator (NIRSystems 6500, formerly Perstorp Analytical, Silver Spring, MD). For each sample, approximately 100 g was poured into a rectangular cell, presented to the instrument in transport mode, and scanned (reflectance) from 400 to 2,500 nm at 2-nm intervals. Each sample was scanned 25 times without repacking the sample, with the entire cell moving past the light beam and detector at least once, and the average reflectance spectrum was stored for calibration development.

Surface Lipid Extraction

Surface lipids were extracted in a Soxtec System HT, which consisted of an extraction unit (model 1043) and a service unit (model 1044). Prior to extraction, a cellulose extraction thimble (26 mm diameter, 60 mm length) was filled with 5 g of head rice, covered with a thin layer of cotton wool, and dried in a convection oven at 100°C for 1 hr for the purpose of controlling sample moisture content. The thimble and sample were then immersed into 50 ml of petroleum ether (boiling point 35–60°C) in an extraction cup for 30 min to extract most of the lipids on the kernel surfaces. The sample was then raised above the solvent surface and washed with solvent for another 30 min to rinse the lipids remaining on the kernel surfaces. After rinsing, excess solvent from the thimble was collected into the extraction cup for 15 min.

All of the extracted surface lipids were thereby retained inside the extraction cup. The content of the cup was dried at 100°C for 30 min to drive off the petroleum ether. The SLC was computed as the mass of the dry extract expressed as a percentage of the original sample weight (5 g). This analysis was duplicated for each sample, and results are reported as means of duplicates.

Statistical Analyses

The two sample sets were combined in three ways for calibration and prediction. In combination I, set one (112 samples) was used as the calibration set, and set two (51 samples) was used as the prediction set. Combination II was the inverse operation; set two was used for calibration, and set one was used for prediction. Combination III was performed by mixing the two sample sets into one (163 samples in total) and then randomly separating it into two sets, with 100 samples for calibration and 63 samples for prediction.

Commercial spectral analysis software (NIRS 2, ISI International Co., Port Matilda, PA) was used to process spectral and SLC data for calibration and prediction. To develop calibration equations, the spectra of the calibration set were first pretreated. Common pretreatment techniques include scatter correction and spectral derivative (ISI International 1995). Three scatter correction methods, referred to as standard normal variate (SNV), detrend, and multiplicative scatter correction (MSC), were compared for optimal calibration. First and second derivative treatments (gap = 8 nm) were also evaluated.

Multilinear regression techniques were performed on the pretreated spectra and associated SLC to develop calibration equations. Four multilinear regression methods, referred to as stepwise,
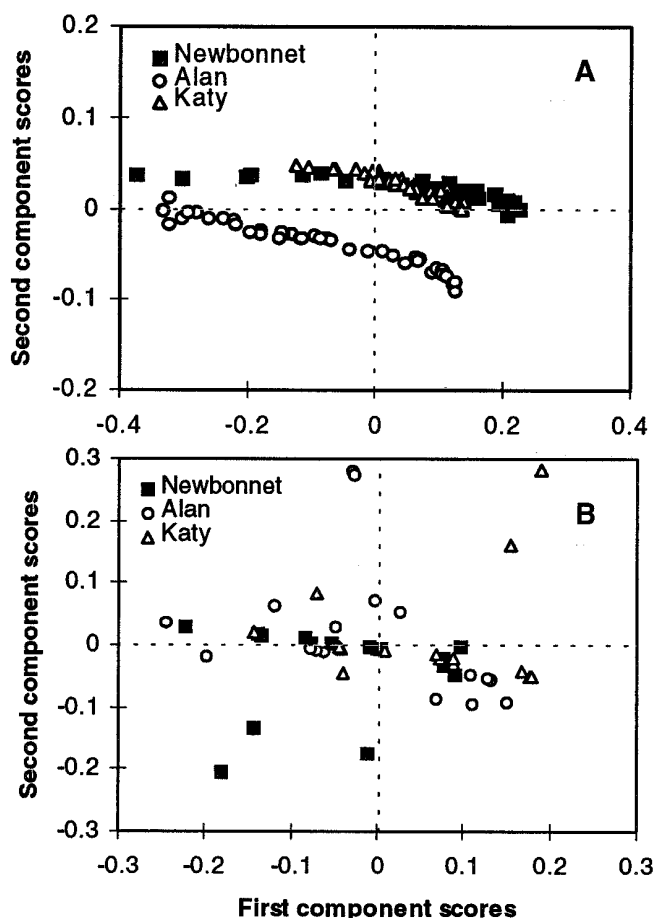
TABLE II
Standard Error of Calibration (SEC), Coefficient of Determination (R^2, for Calibration), and Standard Error of Prediction (SEP), as Affected by Different Combinations of the Two Sample Sets[a]

Sample Set Combination	Description	SEC (%SLC)	R^2	SEP (%SLC)
I	Set one (112 sample) for calibration Set two (51 samples) for prediction	0.026	0.99	0.11
II	Set two (51 samples) for calibration Set one (112 samples) for prediction	0.027	0.99	0.06
III	Mixing two sets, 100 random samples for calibration and remaining 63 samples for prediction	0.024	0.99	0.04

[a] Pretreatment: standard normal variate (SNV) and first derivative; regression: modified partial least squares (MPLS); wavelength ranges: 400–700 and 1,500–2,500 nm; number of PCs: seven.

TABLE III
Standard Error of Calibration (SEC), Coefficient of Determination (R^2, for Calibration), and Standard Error of Prediction (SEP) from Different Wavelength Ranges[a]

Wavelength Range, nm	SEC (%SLC)	R^2	SEP (%SLC)
400–700	0.04	0.97	0.07
1,500–2,500	0.03	0.99	0.05
400–700, 1,500–2,500	0.024	0.99	0.04
400–2,500	0.03	0.99	0.04

[a] Sample set: combination III; pretreatment: standard normal variate (SNV) and first derivative; regression: modified partial least squares (MPLS); number of PCs: seven.

Fig. 3. Second principal component versus first principal component. **A,** sample set one; **B,** sample set two.

principal component, partial least squares (PLS), and modified partial least squares (MPLS) (ISI International 1995), were used to develop calibration equations. Calibration accuracy was evaluated by the coefficient of determination (R^2) and the standard error of calibration (SEC) (Marks and Workman 1991). Once developed, the calibration equations were applied to the independent prediction set for validation. Prediction accuracy was evaluated in terms of the standard error of prediction (SEP) (Marks and Workman 1991).

RESULTS AND DISCUSSION

Degree of Milling of the Two Sample Sets

Table I lists the means, standard deviations, minimum and maximum values of SLC, and number of samples for each variety in sets one and two. Figure 1 shows the distribution of SLC among samples, with each data point representing an average of two duplicated SLC measurements. A standard error of 0.027% SLC in surface lipid extraction was calculated from the duplicated data.

In set one (Fig. 1A), with an increase in sample number , SLC decreased exponentially for Newbonnet and almost linearly for Alan and Katy. Consequently, the latter two varieties had more uniform sample distribution over the whole SLC range than did Newbonnet. The difference in the sample distribution can be attributed to the different milling methods used to derive the various DOM levels. In set two (Fig. 1B), each sample represented a single size fraction in one of the three DOM levels (low, medium, and high). With the change of sample number, there existed obvious discontinuities in the SLC for Katy, representing the three different DOMs achieved in the commercial mill. The discontinuity was less distinct for Newbonnet and Alan. In general for set two, Newbonnet and Alan had more uniform sample distribution across SLC than did Katy.

Compared with set two, set one had a wider range of SLC and more uniform and continuous sample distribution across SLC. However, set one represented only two independently controlled factors: variety and DOM. In set two, kernel thickness was added as a third independent factor.

Spectral Properties of the Two Sample Sets

A principal component analysis method was used to reduce spectral data (400–700 nm, and 1,500–2,500 nm) for each sample to several principal components (PC). For the two sample sets, correlation coefficients of the first 10 PCs with SLC were calculated (Fig. 2). For sample set two, the correlation of each PC to kernel thickness was also computed (Fig. 2B). For both sample sets, the first PC had the strongest correlation with SLC. These correlation coefficients were –0.88 for set one and –0.74 for set two. The negative values indicated that the first PC was inversely related to SLC. As another independent factor in set two, kernel thickness was most highly correlated with the sixth PC (correlation coefficient –0.6).

Figure 3 shows the second PC versus the first PC for the two sample sets (400–700 nm and 1,500–2,500 nm). In set one (Fig. 3 A), Alan was discriminated clearly from the other two varieties by the second PC. In other words, variety was a significant factor affecting visible/NIR spectroscopy in sample set one. In set two (Fig. 3B), the three varieties could not be discriminated from each other by either the first PC or second PC. Variety had much less effect on visible/NIR spectroscopy in set two than it did in set one.

Comparing Two Sample Sets for SLC Calibration and Prediction

Table II lists SEC, R^2 (from calibration), and SEP for SLC from three combinations of the two sample sets. The calibration equations were developed with standard normal variate (SNV) and first derivative pretreatment, modified partial least squares (MPLS)

regression, seven PCs, and combined wavelength ranges of 400–700 nm and 1,500–2,500 nm. Predicted SLC versus measured SLC is shown in Fig. 4.

All three sample set combinations gave high coefficients of determination ($R^2 = 0.99$). The SECs from these combinations were 0.026, 0.027, and 0.024% SLC, respectively. As mentioned earlier, SLC measurements had an error of 0.027% SLC. Consequently, these data combinations and calibrations resulted in SECs at the same level as the SLC measurement error.
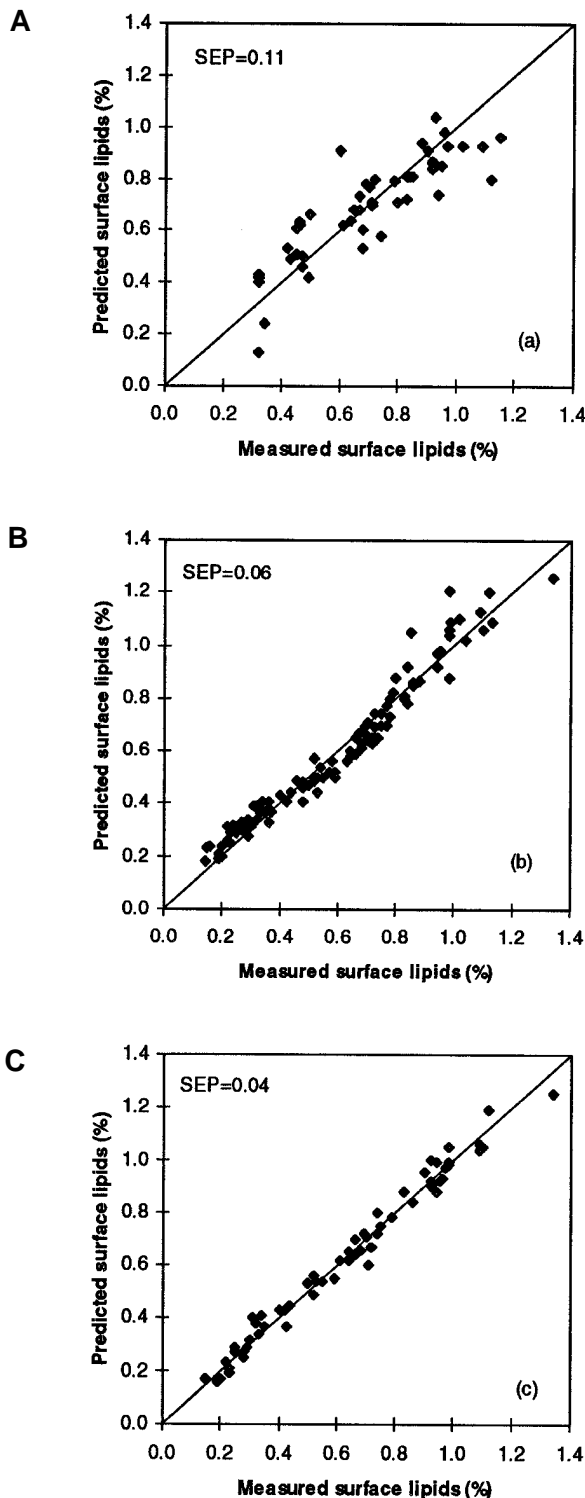

Fig. 4. Predicted surface lipid content versus measured surface lipid content via petroleum ether extraction. **A,** set one for calibration, set two for validation; **B,** set two for calibration, set one for validation; **C,** 100 random samples for calibration, 63 random samples for validation.

Combination I yielded an SEP of 0.11% SLC, which was approximately four times the SLC measurement error (Fig. 4A). Combination II reduced the SEP to 0.06% SLC (2.2 times the SLC measurement error) (Fig. 4B). The best prediction was from combination III (Fig. 4C). In this case, SEP was reduced to 0.04% SLC (1.5 times the SEC and the SLC measurement error).

It can be concluded that the SEC was not significantly influenced by the sample set combination but that the SEP was significantly affected. For reliable prediction of SLC, calibration equations should be developed on the basis of a mixed sample set, where independent factors such as variety, kernel thickness, moisture content, etc. are fully represented. Further experiments remain to expand our current sample sets with more factors and more uniform sample distribution for each factor.

Optimal Calibration and Prediction

In Table III, four wavelength ranges were compared for the optimal calibration and prediction. The calibration equations were developed with sample set combination III, SNV and first derivative pretreatment, MPLS regression, and seven PCs. Of the four wavelength ranges, the visible wavelength range (400–700 nm) resulted in the highest SEC (0.04% SLC) and SEP (0.07). The NIR range (1,500–2,500 nm) reduced the SEC and SEP to 0.03 and 0.05% SLC, respectively. The best calibration and prediction were obtained by the combined visible/NIR wavelength range

(400–700 nm, and 1,500–2,500 nm). Extending the combined wavelength range to the entire visible/NIR spectrum (400–2,500 nm) did not improve the SEC and SEP.

Scatter correction and derivative pretreatment methods were also evaluated, with respect to calibration and prediction. The calibration equations were developed with sample set combination III, MPLS regression, seven PCs, and combined wavelength ranges of 400–700 nm and 1,500–2,500 nm. Table IV shows SEC, R^2 (for calibration), and SEP before and after three scatter correction methods (SNV, Detrend, and MSC) were applied to the first derivative of the spectral data. Without scatter correction, SEC and SEP were 0.026 and 0.05% SLC, respectively. All three scatter correction methods slightly reduced the SEC. SNV and MSC reduced the SEP to 0.04% SLC, and Detrend actually increased the SEP to 0.06% SLC. SNV and MSC were therefore selected. Table V shows the SEC, R^2 (for calibration), and SEP before and after the first and second derivative were applied to the spectral data ($\log[1/R]$) pretreated with SNV. Compared to the SEC and SEP without derivative treatment, the first derivative reduced SEC and SEP, while the second derivative did not. The first derivative therefore improved both calibration and prediction for SLC.

In addition to spectral pretreatment, spectral regression techniques were also evaluated. The calibration equations were developed with sample set combination III, SNV and first derivative pretreatment, and combined wavelength ranges of 400–700 nm and 1,500–2,500 nm. Table VI shows SEC and R^2 of the calibration equations developed by means of four multilinear regression methods and the optimal numbers of terms or PCs as derived by means of cross-validation (ISI International 1995) in calibration development. The corresponding SEPs from these equations are also listed in Table VI. PCR with nine PCs yielded the worst calibration (SEC = 0.06% SLC) and prediction (SEP = 0.1% SLC). By means of the stepwise regression technique, a calibration equation was built on the basis of eight wavelengths (436, 620, 1,712, 1,724, 1,800, 2,024, 2,088, and 2,240 nm). This equation had an SEC and SEP of 0.03 and 0.06% SLC, respectively, which were approximately equal to the calibration results for PLS with five PCs. The best calibration and prediction were obtained by the MPLS regression technique with seven PCs. The resulting SEP (0.04% SLC) was 67% of the SEP resulting from stepwise and PLS regression, and 40% of the SEP resulting from the PCR method.

In developing calibration equations by means of PCR, PLS, or MPLS techniques, the number of PCs is an important factor affecting SEP. As an example, Fig. 5 shows this effect on the SEP when MPLS was performed to develop the calibration equation.

TABLE IV
Standard Error of Calibration (SEC), Coefficient of Determination (R^2, for Calibration), and Standard Error of Prediction (SEP), as Affected by Different Scatter Correction Methods[a]

Scatter Correction Methods[b]	SEC (%SLC)	R^2	SEP (%SLC)
None	0.026	0.99	0.05
SNV	0.024	0.99	0.04
Detrend	0.025	0.99	0.06
MSC	0.023	0.99	0.04

[a] Sample set: combination III; pretreatment: first derivative; regression: modified partial least squares (MPLS); wavelength ranges: 400–700 and 1,500–2,500 nm; number of PCs: seven.
[b] SNV = standard normal variate, MSC = multiplicative scatter correction.

TABLE V
Standard Error of Calibration (SEC), Coefficient of Determination (R², for Calibration), and Standard Error of Prediction (SEP), as Affected by Spectral Derivative Treatments[a]

Derivative	SEC (%SLC)	R^2	SEP (%SLC)
None	0.032	0.98	0.05
First	0.024	0.99	0.04
Second	0.031	0.99	0.05

[a] Sample set: combination III; pretreatment: standard normal variate (SNV); regression: modified partial least squares (MPLS); wavelength ranges: 400–700 and 1,500–2,500 nm; number of PCs: seven.

TABLE VI
Standard Error of Calibration (SEC), Coefficient of Determination (R², for Calibration), and Standard Error of Prediction (SEP) from Different Multiple Linear Regression Methods[a]

Regression Methods[b]	Number of Terms or PCs	SEC (%SLC)	R^2	SEP (%SLC)
Stepwise	8	0.03	0.98	0.06
PCR	9	0.06	0.95	0.10
PLS	5	0.04	0.97	0.06
MPLS	7	0.024	0.99	0.04

[a] Sample set: combination III; pretreatment: standard normal variate and first derivative; wavelength ranges: 400–700 nm and 1,500–2,500 nm. Optimal numbers of terms or PCs are based on cross-validation procedures.
[b] PCA = principal component analysis, PLS = partial least squares, MPLS = modified partial least squares.
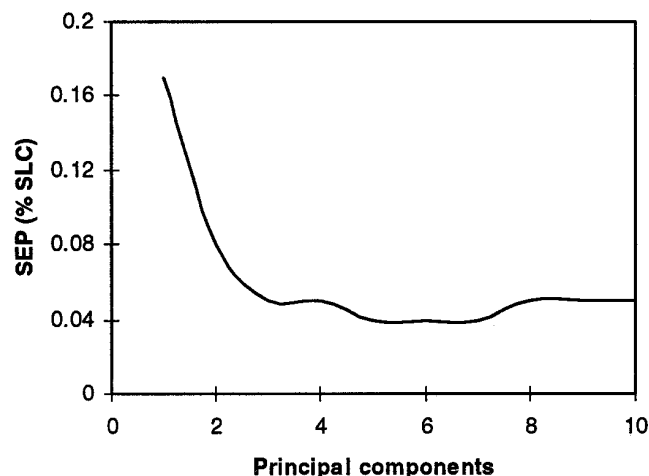

Fig. 5. Effect of number of principal components on standard error of prediction (SEP), for modified partial least squares. SLC = surface lipid content.

Using one PC resulted in an equation with a very large SEP (0.17% SLC). The SEP decreased dramatically when the number of PCs was increased to three, and reached the lowest (0.04% SLC) at five to seven PCs. The SEP tended to increase again as the number of PCs was increased past seven, due to data overfitting. In the reported MPLS calibrations, seven PCs were included in the calibration equation.

CONCLUSIONS

Two sample sets were prepared, with one containing two independent factors (variety and DOM) and another containing three independent factors (variety, DOM, and kernel size). The calibration equations developed from both sample sets had similar SECs. However, the calibration equation from the set with three factors yielded much better accuracy in predicting SLC than did that from the set with two factors. Combining both sets yielded even better accuracy in prediction.

The motivation for these analyses (i.e., using two different sample sets) lies in the novelty of SLC as an economically important but non-natural variable. Past research by others, previously mentioned, focused on correlating factors such as chemical composition and physical properties to reflectance or transmittance. In these studies, the calibration and validation sample sets included a naturally occurring range of the factors of interest. In contrast, variability in SLC is caused primarily by changes in the process that created the samples (i.e., milling). Consequently, it is especially important to ensure that any calibrations for this type of factor (i.e., non-natural) are developed from an appropriate sample set, in order to maximize calibration robustness.

With respect to calibration optimization, the combination of visible and NIR ranges yielded the best calibration and prediction. Given these spectral data, SNV and multiplicative scatter correction were recommended as scatter correction methods for optimal calibration and prediction. Additionally, the first derivative spectra improved calibration and prediction accuracy. MPLS, with 5–7 PCs, yielded the best calibration and prediction among several regression methods. The resulting SEC (0.024% SLC) was at the same level as the SLC reference measurement error, and SEP (0.04% SLC) was $\approx 1.5 \times$ reference measurement error.

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