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Rapid Prediction of Gross Energy and Utilizable Energy in Cereal Food Products Using Near-Infrared Reflectance Spectroscopy

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Near-infrared (NIR) spectroscopy has been used in foods for the rapid assessment of several macronutrients; however, little is known about its potential for the evaluation of the utilizable energy of foods. Using NIR reflectance spectra (1104−2494 nm) of ground cereal products ($n = 127$) and values for energy measured by bomb calorimetry, chemometric models were developed for the prediction of gross energy and available energy of diverse cereal food products. Standard errors of cross-validation for NIR prediction of gross energy (range = 4.05−5.49 kcal/g), energy of samples after adjustment for unutilized protein (range = 3.99−5.38 kcal/g), and energy of samples after adjustment for unutilized protein and insoluble dietary fiber (range = 2.42−5.35 kcal/g) were 0.053, 0.053, and 0.088 kcal/g, respectively, with multiple coefficients of determination of 0.96. Use of the models on independent validation samples ($n = 58$) gave energy values within the accuracy required for U.S. nutrition labeling legislation. NIR spectroscopy, thus, provides a rapid and accurate method for predicting the energy of diverse cereal foods.

KEYWORDS: Near-infrared spectroscopy; NIR; energy; calorie; kilocalorie; cereal; nutrition labeling

INTRODUCTION

The accurate evaluation of the energy content of foods and the inclusion of this information on the nutrition label are important for the selection of foods by consumers and health professionals. The U.S. Dietary Guidelines urge consumers to aim for a healthy body weight by "choosing a healthful assortment of foods"; "balancing energy intake and physical activity"; "choosing foods that are low in fat and added sugar most of the time"; and eating "sensible portion sizes" (*1*). The benefits of managing energy intake and avoiding obesity include a reduced risk for high blood pressure, heart disease, stroke, diabetes, and certain forms of cancer.

In the United States, statements of total energy per serving and energy derived from fat per serving (when fat is >0.5 g) are compulsory for nutrition labeling (*2*). Before determination of the energy content of foods, several physiological factors need to be considered. Although the gross energy content of a food is a useful figure in diet selection, the actual energy available to the individual will be less, primarily due to incomplete utilization of proteins and fiber in the human body. Whereas protein is oxidized completely in a calorimeter, which is used to determine gross energy value, it is incompletely oxidized in the body as nitrogen and some carbon and hydrogen are excreted, mainly in the form of urea. Fiber is also completely oxidized in a calorimeter but not digested at all by human alimentary enzymes. However, partial degradation of fiber occurs by colonic bacterial enzymes, which can metabolize some types of fiber, predominantly soluble fiber, to short-chain fatty acids. Short-chain fatty acids can be absorbed by the colon and become available for energy. Thus, adjustments in gross energy values for incomplete utilization of protein and indigestibility of fiber will more closely reflect food energy that can be utilized by humans.

In the current study energy is expressed as kilocalories (kcal) per gram of sample. A kilocalories (commonly called a Calorie) equals the amount of heat required to raise the temperature of 1 kg of water at 15 °C, 1 °C and is equivalent to 1000 "small" calories (*3*). Nutrition labels in the United States state the energy values of foods as kcal (Calories) per serving and as kcal (Calories) from fat per serving along with a statement of the serving size in grams and ounces (*2*). Several methods of measuring the energy value of foods are outlined in the U.S. Code of Federal Regulations (*2*). Among these, energy can be determined using bomb calorimetry data subtracting 1.25 kcal/g of protein to correct for unutilized protein. Alternatively, energy can be calculated using the general factors of four, four, and nine kcal/g for protein, total carbohydrate less the amount of insoluble dietary fiber, and total fat, respectively, taking into account both the incomplete utilization of protein and the nondigestibility of a portion of the fiber (the general factor for protein takes into account the incomplete utilization of protein

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by the human body). Other methods include using factors for the energy values of specific foods, ingredients, or components (*2*).

Near-infrared (NIR) spectroscopy is a rapid method of measuring certain constituents of foods and agricultural commodities (*4*−*6*). The technology is applied widely throughout Europe and North America for the evaluation of protein and moisture in grains, and for the evaluation of animal feeds. NIR spectroscopy has been used for quality control in the pharmaceutical industry and the rapid evaluation of several macronutrients in foods (*6*). In diverse cereal food products, the potential of the technique has been demonstrated, in our laboratory and others, for the determination of many macronutrients included in U.S. nutrition labeling legislation. The nutrients include total dietary fiber (*7*, *8*), protein (*9*), and fat (*10*, *11*). NIR also provides a method for analysis of nutrients that may be listed on nutrition labels on a voluntary basis, such as insoluble dietary fiber (*12*, *13*). Very little has been reported on the use of NIR spectroscopy for the evaluation of the energy content of foods. Work on the use of NIR for the prediction of the energy content in ruminant feeds and feeds for monogastric animals has been reviewed (*14*−*16*). These reviews include studies on NIR prediction of energy that is actually metabolized and digested by ruminant and monogastric animals.

Work on the use of NIR spectroscopy for the determination of the energy available for humans is limited and includes a paper by Lanza (*17*) on the determination of calories in raw, emulsified, retail cuts of pork and beef using reflectance and transmittance (standard errors of performance ranged from 0.038 to 0.035 kcal/g) and a paper by Mitsumoto et al. (*18*) on raw beef cuts using NIR reflectance, transmittance, and fiber optic measurements (standard errors of calibration ranged from 0.12 to 0.20 kcal/g). In both cases, energy was determined by the procedure in which biological energy value of the meat is calculated by multiplying specific energy factors by protein and fat content.

The current study investigated the use of NIR reflectance spectroscopy, using a dispersive NIR instrument, for the prediction of the energy content of diverse cereal food products. Energy content was calculated from bomb calorimetry data and laboratory analysis of protein and insoluble dietary fiber. Thus, models were developed to predict the gross energy content of the cereal foods, the energy content adjusted for unutilized protein, and the energy content adjusted for unutilized protein and insoluble fiber. The models were tested using independent validation samples, and the adequacy of the predictions for quality control purposes is discussed.

MATERIALS AND METHODS

Cereal Food Products. Cereal food products used in the study were obtained from commercial retailers and included breakfast cereals, crackers, flours, brans, pastas, and unprocessed whole grains. The grains present in products in the calibration and validation data sets included wheat, oats, barley, rye, and millet. Some samples were multiple-grain products, and buckwheat and amaranth were also present in some multiple-grain products. Samples in the calibration and validation data sets contained a wide range of sugar, fat, dietary fiber, protein, and moisture and additives such as nuts, dried fruits (raisins, peaches, blueberries, and dates), honey, salt, cinnamon, cocoa, and herbs; these products were processed by a variety of methods. One hundred and twenty-eight samples were used in the calibration data set and 58 samples in the validation data set. Validation samples were purchased and scanned at a different time from the calibration samples.

Sample Preparation. Cereal food products were dry milled to <500 μm in a Cyclotec 1093 sample mill (Perstorp Analytical, Silver Spring, MD). Samples with >20% sugar content (based on the product's nutrition label value) were mixed with liquid nitrogen to aid grinding, and samples with >10% fat were ground with a coffee mill (model KSM-2, Braun Inc., Lynnfield, MA).

Reference Analysis. Gross energy (kcal/g) of cereal food samples and of Avicel PH-105 cellulose (FMC Corp., Philadelphia, PA) was determined at The University of Georgia, Poultry Nutrition Laboratory. Milled samples (0.7 g) were analyzed in duplicate by bomb calorimetry (Parr Instrument Co., Moline, IL) as described in Parr Manual 120 (*3*) using benzoic acid (6.318 kcal/g) as the standard. Protein was determined by combustion analysis (*19*) (AOAC Method 992.23) with the LECO FP2000 combustion analyzer. Insoluble dietary fiber was determined according to an enzymatic gravimetric method (*20*) (AOAC Method 991.43) and dry matter by the air oven method at 105 °C (*21*) (AOAC Method 945.15). Energy, protein, and insoluble dietary fiber were expressed on a dry weight basis for all calculations. Energy was reported as (1) gross energy, (2) energy adjusted for unutilized protein, and (3) energy adjusted for unutilized protein and insoluble dietary fiber. Gross energy values were adjusted for the amount of unutilized protein by subtracting 1.250 kcal/g of protein (*22*). The energy value of Avicel PH-105 cellulose was found to be 4.153 kcal/g on a dry weight basis. This value was used as the gross energy value for insoluble dietary fiber as cellulose is a typical component of insoluble dietary fiber in cereal products. Furthermore, the value is very close to the Atwater factor (4.2 kcal/g) for the energy content of carbohydrates in cereal foods (*22*). Gross energy values were also adjusted for both unutilized protein and insoluble dietary fiber by subtracting 1.250 kcal/g of protein and 4.153 kcal/g of insoluble fiber. For a number of samples (n = 142, limited by availability), the energy values adjusted for unutilized protein and insoluble dietary fiber were determined by an alternative method, that is, using the factors four, four, and nine kcal/g for protein, total carbohydrate less the amount of insoluble dietary fiber, and total fat, respectively. The fat content was determined according to AOAC petroleum ether extraction method 945.16 (*23*), and the amount of total carbohydrate was calculated by difference (100% − protein % − fat %). Linear regression was used to determine the agreement between the two methods of determination of energy adjusted for unutilized protein and insoluble dietary fiber.

Spectroscopic Analysis. Milled samples were scanned with the NIRSystems 6500 monochromator (NIRSystems, Silver Spring, MD). The instrument is a visible/near-infrared scanning monochromator with a tungsten source and a holographically ruled grating. Diffusely reflected radiation is detected from 400 to 2498 nm at 10 nm resolution and a data interval of 2 nm. Reference reflectance data are obtained with a ceramic block. Duplicates of each sample were scanned in cylindrical sample cells (internal diameter = 38 mm, depth = 9 mm). Each duplicate was scanned 16 times, the scans were averaged, and the data were transformed to log 1/R. Scans from the duplicate samples were then averaged to give one final spectrum.

Development of Multivariate Calibrations for Prediction of Energy as Kilocalories per Gram. The wavelength range used for analysis was 1104−2494 nm. Energy prediction models were developed with a commercial analysis program (NIR3, version 4.01, ISI International Inc., Port Matilda, PA). Models were developed using spectra of the 128 cereal food products and reference data for (1) gross energy, (2) energy adjusted for unutilized protein, and (3) energy adjusted for unutilized protein and insoluble dietary fiber. Log 1/R spectra were transformed with normal multiplicative scatter correction (*24*) and second-derivative processing (gap = 16 nm, smoothing interval = 16 nm) and centered using the CENTER program available in NIR3. The CENTER program uses partial least squares (25) (PLS) analysis, which allows centering of samples based on constituent values as well as spectral characteristics and allows identification of outliers. For calibration, modified PLS was the regression method used (*26*). The modification to PLS scaled the reference method data and reflectance data, at each wavelength, to have a standard deviation of 1.0 before each PLS term. Prior to modified PLS regression, log 1/R spectra were mean centered, transformed with multiplicative scatter correction to remove interferences due to particle size, and then transformed using second-derivative processing (gap = 16 nm, smoothing interval = 16 nm). The optimum number of modified PLS factors used for energy

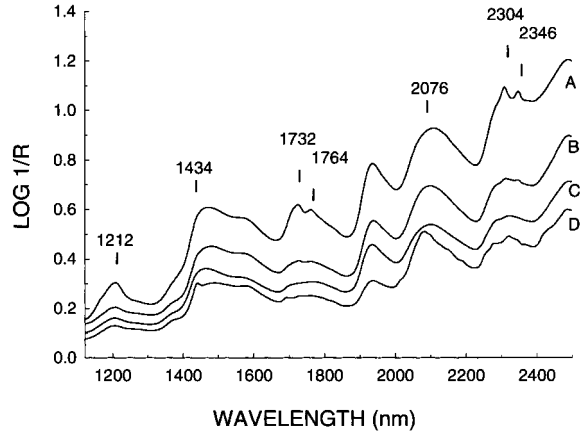

Figure 1. Spectra of selected representative cereal products: high-fat oat granola (A); multigrain, high-sugar, fruit- and fiber-containing breakfast cereal (B); rye crackers (C); sugar-coated, corn breakfast cereal (D).

prediction was determined by cross-validation (*27*). During cross-validation, one-sixth of the calibration samples was temporarily removed from the calibration set at a time and used for prediction. Performance statistics were accumulated for each group of removed samples. The optimum number of factors for energy prediction was that which produced a minimum overall error between modeled and reference values [standard error of cross-validation (SECV)]. Preprocessing transformations were the optimum required to improve the SECV compared to modified PLS values with untransformed data.

Validation of Models. Energy models developed were tested using an independent set of cereal food products ($n = 58$). The cereal food products used for validation were purchased, prepared, scanned for NIR spectra, and analyzed for reference data with the same methods used for the calibration samples but at a different time. Performance of the models was reported as the standard error of performance (SEP), coefficient of determination (r^2), slope, bias (*28*), RPD (*29*), and coefficient of variation. The RPD is the ratio of the SEP to the standard deviation of the reference values. The statistic provides a standardization of the SEP, and values of 5−10 are considered to be adequate for quality control applications.

RESULTS

Spectral Characteristics of Samples. Selected typical spectra of the cereal food products are shown in **Figure 1**. Predominant differences between spectra of samples were due to the quantities of fat and quantities and types of sugar. Samples with high fat content, such as many granolas and crackers, had sharper peaks at 1212, 1732, 1764, 2304, and 2346 nm, compared to low-fat samples, due to absorption by C−H stretch groups in lipids (*30*, *31*). Samples with high sucrose content, such as sugar-coated cereals, had sharp peaks at 1434 and 2076 nm, compared to low-sugar samples, due to absorption by O−H groups in carbohydrate.

Energy Measured by the Reference Method. The distribution of energy values in the calibration and validation data sets for the three models is shown in **Figure 2**. The overall range in gross energy of samples measured by calorimetry was 4.05−5.49 kcal/g with a standard error of the method (*32*) of 0.035 kcal/g. The range in energy of the samples after adjustment for unutilized protein was 3.99−5.38 kcal/g and was 2.42−5.35 kcal/g after adjustment for unutilized protein and insoluble dietary fiber. The standard errors of the reference methods for protein and insoluble dietary fiber were 0.21% (range of values = 4.04−20.14%) and 0.41% (range of values = 0−43.92%), respectively.

Linear regression of energy values adjusted for unutilized protein and insoluble dietary fiber derived from calorimetry

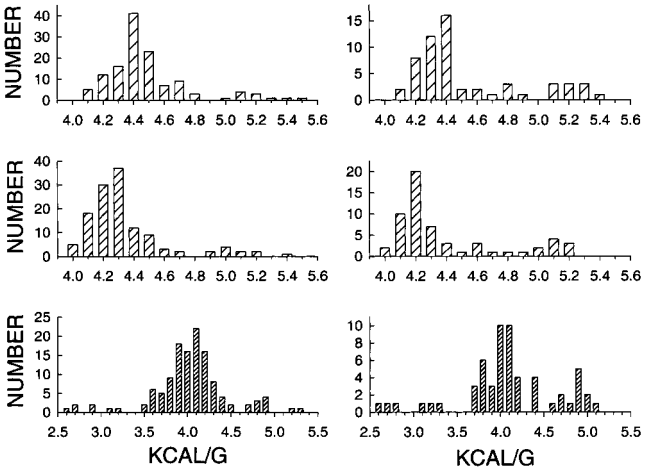

Figure 2. Distribution of energy content values in cereal sample data sets used for the calibration (left panels) and validation (right panels) of models for prediction of gross energy (upper panels), energy adjusted for unutilized protein (middle panels), and energy adjusted for unutilized protein and insoluble dietary fiber (lower panels).

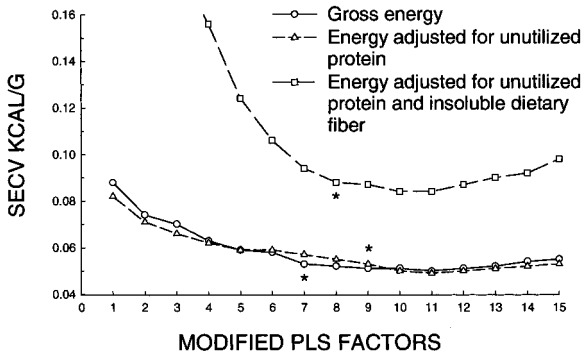

Figure 3. Number of modified PLS factors vs SECV for three energy models. Asterisks denote the number of factors used for each model.

versus values derived from factors for protein, carbohydrate less the amount of insoluble fiber, and fat gave a coefficient of determination of 0.94 and a standard error of the estimate of 0.13 kcal/g (range of values = 2.42−5.35 kcal/g derived from calorimetry and 2.66−5.24 kcal/g derived from factors).

All Calibrations for Energy. One sample was a spectral outlier and was removed from the calibration data set (Mahalanobis distance = 10.0−21.1).

Calibration for Gross Energy. For the prediction of gross energy, seven modified PLS factors were used in the model (**Figure 3**) and described 97.1% of the spectral variation. The overall error between modeled and reference values (SECV), using six cross-validation groups, was 0.053 kcal/g with a multiple coefficient of determination (R^2) of 0.96 (**Table 1**; **Figure 4**). The model was tested with the independent validation samples, and NIR predicted values for gross energy were compared with values determined by bomb calorimetry, using linear regression. The standard error of performance (SEP) was 0.049 kcal/g, the coefficient of determination (r^2) was 0.98, the slope was 1.05, the bias was −0.02 kcal/g, and the RPD was 7.35 (**Table 1**; **Figure 5**). The coefficient of variation for prediction of gross energy was 1.08%.

Sample scores having the highest correlation with gross energy were for factor one, which had a Pearson correlation coefficient of 0.92. Loadings are the regression coefficients of each variable (wavelength) for each factor and indicate which wavelengths are predominantly influencing the model. The

Table 1. Calibration and Validation Statistics for Prediction of Energy (Kilocalories per Gram) of Cereal Food Products[a]

		calibration						validation								
	method	*n*	range	mean	SD	SECV	R^2	*n*	range	mean	SD	SEP	r^2	bias	slope	RPD
gross calories	AOAC	127	4.05–5.49	4.49	0.27			58	4.09–5.35	4.53	0.36					
	NIR	127	4.02–5.41	4.49	0.27	0.053	0.96	58	4.10–5.39	4.55	0.34	0.049	0.99	–0.020	1.05	7.35
energy adjusted for unutilized protein	AOAC	127	3.99–5.38	4.34	0.27			58	4.00–5.22	4.39	0.36					
	NIR	127	3.95–5.31	4.34	0.26	0.053	0.96	58	4.08–5.26	4.41	0.33	0.053	0.99	–0.023	1.06	6.79
energy adjusted for unutilized protein and insoluble fiber	AOAC	127	2.42–5.35	4.01	0.45			58	2.74–5.18	4.09	0.56					
	NIR	127	2.52–5.28	4.02	0.44	0.088	0.96	58	2.64–5.19	4.14	0.54	0.085	0.99	–0.048	1.04	6.59

[a] Mean, standard deviation (SD), standard error of cross-validation (SECV), and multiple coefficient of determination (R^2) for calibration. Mean, standard deviation, standard error of performance (SEP), coefficient of determination (r^2), bias, slope, and RPD for validation using independent validation samples.

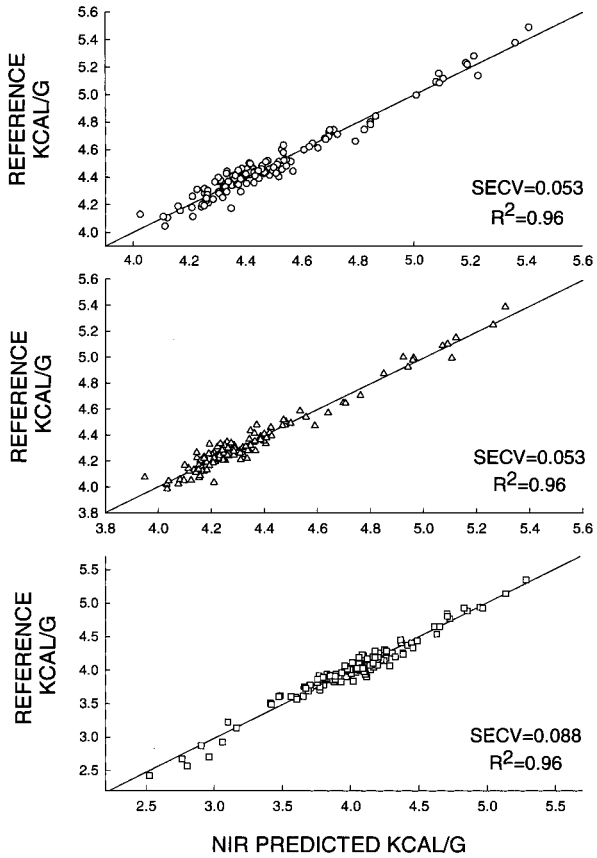

Figure 4. Calibration plots of calorimetry determined energy vs NIR predicted energy in cereal food products ($n = 127$) for three energy models: model for gross energy (upper panel); model for energy adjusted for unutilized protein (middle panel); model for energy adjusted for unutilized protein and insoluble dietary fiber (lower panel).

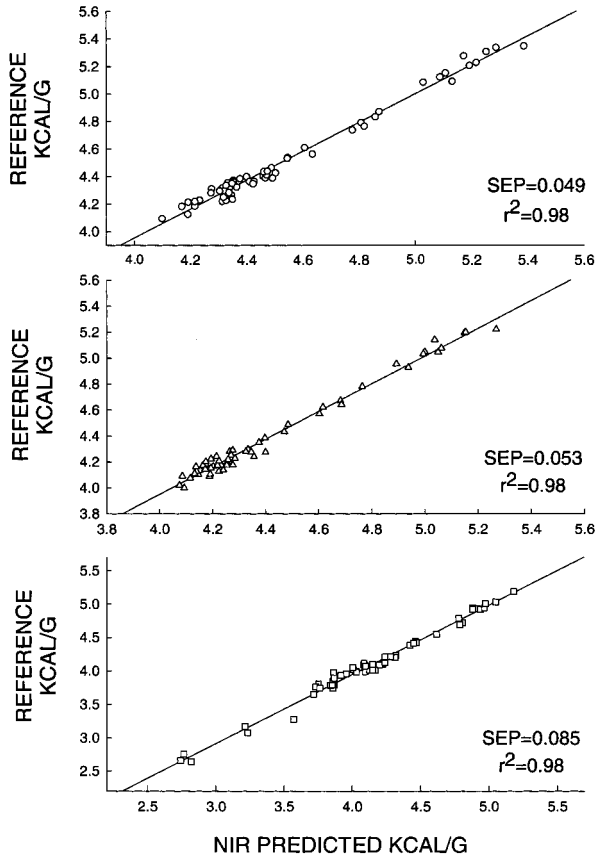

Figure 5. Validation plots of calorimetry determined energy vs NIR predicted energy in independent validation samples of cereal food products ($n = 58$) for three energy models: model for gross energy (upper panel); model for energy adjusted for unutilized protein (middle panel); model for energy adjusted for unutilized protein and insoluble dietary fiber (lower panel).

modified PLS loading for factor one (**Figure 6**) had large intensities related to C−H stretch absorption in lipid at 1212, 1722, 1764, 2304, and 2346 nm and O−H absorption in carbohydrate at 1434 and 2076 nm (*30*, *31*).

Calibration for Energy Adjusted for Unutilized Protein. For the prediction of energy adjusted for unutilized protein, nine modified PLS factors were used in the model (**Figure 3**) and described 97.5% of the spectral variation. The SECV, using six cross-validation groups, was 0.053 kcal/g, and the R^2 was 0.98 (**Table 1**; **Figure 4**). The model was tested with the independent validation samples, and NIR-predicted values for energy adjusted for unutilized protein were compared using linear regression with values determined by the reference method. The SEP, r^2, slope, bias, and RPD were 0.053 kcal/g, 0.98, 1.06, −0.023, and 6.70, respectively (**Table 1**; **Figure 5**). The coefficient of variation for the prediction of energy adjusted for unutilized protein was 1.20%.

The sample scores having the highest correlation with energy adjusted for unutilized protein were for factor one, which had a Pearson correlation coefficient of 0.92. Similarly to the model for gross energy, the modified PLS loading for factor one (**Figure 6**) showed high variation in the regions related to C−H stretch absorption in lipid at 1212, 1722, 1764, 2304, and 2346 nm and O−H absorption in carbohydrate (*30*, *31*). In addition, variation related to O−H absorption in water was observed at 1914 nm.

Calibration for Energy Adjusted for Unutilized Protein and Insoluble Dietary Fiber. For prediction of energy adjusted for unutilized protein and insoluble dietary fiber, eight modified PLS factors were used in the model (**Figure 3**) and described 96.4% of the spectral variation. The SECV, using six cross-validation groups, was 0.088 kcal/g, and the R^2 was 0.96 (**Table 1**; **Figure 4**). The model was tested with independent validation

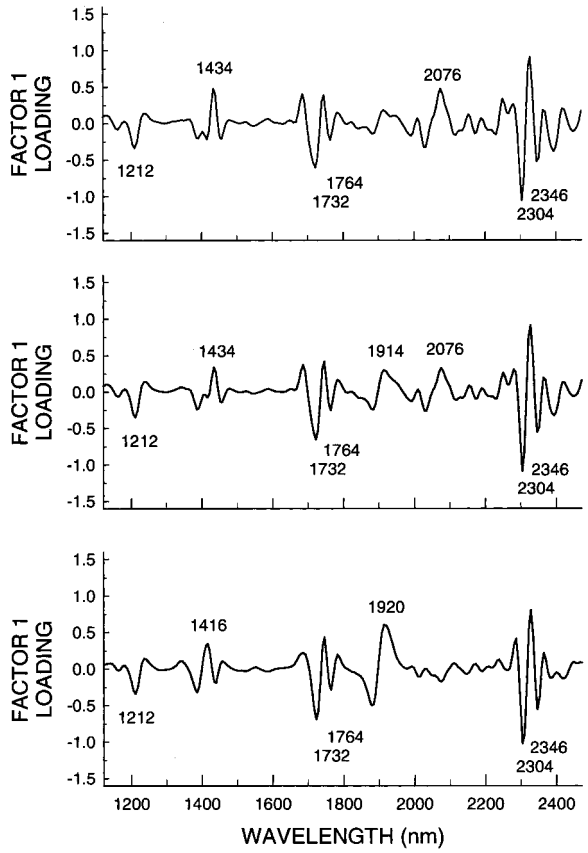

Figure 6. Loading spectra for the first modified PLS factor of each of three models used to predict energy in cereal food products: model for gross energy (upper panel, Pearson correlation coefficient = 0.92); model for energy adjusted for unutilized protein (middle panel, Pearson correlation coefficient = 0.92); model for energy adjusted for unutilized protein and insoluble dietary fiber (lower panel, Pearson correlation coefficient = 0.66).

samples. When NIR-predicted values for energy adjusted for unutilized protein and insoluble fiber were compared using linear regression with values determined by the reference method, the SEP, r^2, slope, bias, and RPD were 0.085 kcal/g, 0.99, 1.04, −0.048 kcal/g and 6.59, respectively (**Table 1**; **Figure 5**). The coefficient of variation was 2.08%.

Factors one, three, and two had sample scores with the highest correlation to energy values adjusted for unutilized protein and insoluble fiber, with Pearson correlation coefficients of 0.66, 0.42, and 0.36, respectively. The modified PLS loading for factor one (**Figure 6**) had significant variation related to C−H stretch groups in lipid at 1212, 1722, 1764, 2304, and 2346 nm and O−H in water at 1416 and 1920 nm (*30*, *31*). Factor three had significant variation related to O−H in carbohydrate at 1434 and 2076 nm, C−H groups in carbohydrate at 2200−2400 nm, and O−H in water at 1914 nm. Factor two had significant variation related to O−H and C−H in carbohydrate at 2076 and 2250 nm, respectively, and water at 1410 and 1926 nm.

DISCUSSION

In the current study, near-infrared reflectance spectroscopy was shown to provide a rapid and accurate method for the determination of energy in diverse cereal food products. The standard error of performance, coefficient of determination, bias, slope, and RPD indicated a high degree of accuracy and reliability in the determination of gross energy by NIR. In addition, coefficients of variation for the method were very low. Compensations are made for unutilized protein or unutilized protein and insoluble dietary fiber. An exact value for the available energy in a food would be difficult to obtain due to differences in digestibility of proteins, physiological conditions, and lower intestinal factors that affect the bacterial degradation of soluble and insoluble dietary fiber. Thus, the methods provide estimates of the physiologically available energy in foods.

On the basis of one of the allowed methods of determining energy, an NIR model was developed to predict the energy value of cereal products taking into account incomplete utilization of protein. The standard error of performance, coefficient of determination, bias, slope, RPD, and coefficient of variation indicated the usefulness of this approach for quality control in cereal food products. The U.S. Code of Federal Regulations (*2*) states that "A food with a label declaration of calories...shall be deemed to be misbranded...if the nutrient content of the composite is greater than 20% in excess of the value for that nutrient declared on the label." Predictions of energy adjusted for unutilized protein were well within the accuracy required for U.S. food labeling purposes for all of the independent validation samples predicted. Similarly, the model developed to predict energy adjusted for unutilized protein and insoluble dietary fiber was acceptable for quality control, with 100% of the samples in the independent validation data set being predicted with sufficient accuracy for nutrition labeling. For NIR prediction of total dietary fiber (*8*) and protein (*9*) 96% (S. E. Kays, unpublished results) and 100% of diverse cereal food samples, respectively, were predicted within the accuracy required by nutrition labeling legislation. The application of NIR spectroscopy to the analysis of utilizable energy has, thus, expanded the potential of NIR for the rapid evaluation of most macronutrients required for nutrition labeling.

The method for determination of energy adjusted for unutilized protein and insoluble dietary fiber described in the U.S. Code of Federal Regulations (*2*) involves use of the factors four, four, and nine kcal/g for protein, total carbohydrates less the amount of insoluble dietary fiber, and total fat, respectively, to calculate the energy content. The values obtained in the current study, using calorimetry and laboratory assays to calculate sample energy adjusted for unutilized protein and insoluble dietary fiber, were highly correlated to values for energy calculated with the above factors.

The modified PLS loadings for energy models were dominated by variations due to C−H groups in lipids. This may be due to lipids contributing ∼9 kcal/g to the energy value of cereal foods, significantly more than protein or carbohydrate, which each contribute ∼4 kcal/g. In addition, 17 of the 127 products used in the calibration contained >10% fat. Other influences were from O−H groups in carbohydrates in the models for gross energy and energy adjusted for unutilized protein and O−H groups in water in the model adjusted for unutilized protein and insoluble dietary fiber. Protein, although contributing to caloric value, does not appear to be an important influence in the energy models.

Once a NIR calibration for prediction of energy of cereal food products is developed, the determination of energy by NIR for new cereal samples involves merely grinding the sample and using a suitable sample cell for the collection of spectra with the monochromator. The process, involving loading duplicate sample cells and scanning each of the duplicates 16 times, takes ≤5 min. In contrast, bomb calorimetry takes ∼45 min for each sample replicate and involves weighing the sample, making a sample pellet, and performing the calorimetry. Thus, near-infrared spectroscopy provides a rapid method of evaluating both the gross energy and utilizable energy content of diverse

cereal food products within the precision that is required for U.S. nutrition labeling and quality control.

ABBREVIATIONS USED

NIR, near-infrared; kcal, kilocalorie; PLS, partial least squares; SECV, standard error of cross-validation; SEP, standard error of performance; R^2, multiple coefficient of determination; r^2, coefficient of determination.

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