Prediction of Total Dietary Fiber in Cereal Products Using Near-Infrared Reflectance Spectroscopy

Sandra E. Kays,* W. R. Windham, and Franklin E. Barton, II

Agricultural Research Service, Richard B. Russell Agricultural Research Center, U.S. Department of Agriculture, P.O. Box 5677, Athens, Georgia 30604-5677

The Nutritional Labeling and Education Act of 1990 has increased the need for more rapid, efficient, and nonpolluting techniques of analyzing the nutrients in foods, particularly dietary fiber. The use of near-infrared spectroscopy (NIRS) for the prediction of total dietary fiber content of cereal products was investigated in this study. Cereal and grain products, including breakfast cereals, flours, brans, crackers, and samples containing commercial oat and wheat fibers, were selected for analysis. Products ($n = 91$) were dry milled, and total dietary fiber was measured by the AOAC (991.43) enzymatic-gravimetric method. Total dietary fiber values ranged from <1 to 52% of dry weight. Milled cereal products ($n = 91$) were scanned from 1100 to 2498 nm with a NIRSystems 6500 monochromator. Using ISI software for scanning and data analysis, a dietary fiber calibration was obtained with partial least squares as the regression method. The standard error of cross validation and multiple coefficient of determination were 1.58% and 0.99, respectively. For equation validation, an independent group of cereal products ($n = 31$) was dry milled, and total dietary fiber was determined. The samples were scanned, and total dietary fiber was predicted by NIRS. Samples were predicted with a standard error of performance of 1.51%, coefficient of determination of 0.99, bias of −0.38, and slope of 1.06. This study shows that NIRS can be used to rapidly and accurately predict total dietary fiber content in a wide range of cereal products.

Keywords: *Dietary fiber; near-infrared spectroscopy; nutrition labeling*

INTRODUCTION

The health benefits of a high fiber diet have long been recognized by the medical and nutritional communities. For many years, Americans have been encouraged to increase their consumption of high fiber foods, such as vegetables, fruits, and whole grain cereal products (Position of the American Dietetic Association: health implications of dietary fiber, 1993). To help consumers make informed and healthful food choices, the Nutritional Labeling and Education Act (NLEA) was created in 1990. This legislation requires that the amount of total dietary fiber present in a product be included on the nutrition label (*Code of Federal Regulations*, 1995). As a result of this legislation rapid and accurate methods are required for the determination of dietary fiber in foods.

Dietary fiber is "the sum of lignin and polysaccharides that are not hydrolyzed by human alimentary enzymes" (Trowell et al., 1976; Lee and Prosky, 1995). The AOAC total dietary fiber method (AOAC, 1990b, 1992; Lee et al., 1992) is currently used in the United States, Canada, and many European countries for nutritional labeling. Nonstarch polysaccharides, resistant starch, and lignin remaining after α-amylase, protease, and amyloglucosidase digestion of food samples are measured with this method. Included in the analyte are other nonhydrolyzed materials, such as cutin and waxes, and remnants of plant components resistant to human alimentary enzymes, which are often also included as components of dietary fiber (Trowell et al., 1972; Lee and Prosky, 1995).

The AOAC method has been used with consistent results over time and on a wide variety of food products, grains, fresh fruits, and vegetables. However, it is very time consuming (taking 2 days to complete a determination), expensive, and labor intensive. A more rapid method that retains the same degree of accuracy would help industries in their compliance with the NLEA and could be used for efficient monitoring of compliance by regulatory agencies.

Near-infrared spectroscopy (NIRS) is a rapid and accurate method for measuring some constituents of materials without requiring extensive sample preparation, and NIRS usually does not lead to chemical waste production (Williams and Norris, 1987a; Marten et al., 1989). In the past, NIRS has been used successfully to determine fat, moisture, protein, acid detergent fiber, and neutral detergent fiber content of agricultural products (Norris et al. 1976; Williams and Norris, 1987b; Windham et al., 1988; Barton and Windham, 1988). Few studies have addressed the potential of NIRS to determine total dietary fiber in foods. Baker (1983) reported the successful prediction of neutral detergent fiber content of milled breakfast cereals by NIRS. Similarly, Williams et al. (1991) reported analysis of total, soluble, and insoluble dietary fiber in oat bran products by NIRS. The use of NIRS for the prediction of total dietary fiber content of a variety of cereal products has not been investigated.

The range of total dietary fiber content declared on cereal product labels ranges from zero to ∼50%. Because the cereal and baking industries include products with a wide range of cereals, often with mixed grains in a single product, it is necessary for nutrition labeling purposes to be able to predict dietary fiber content in heterogeneous samples, as well as homogeneous groups of samples, with a wide range of dietary fiber values. Using the AOAC approved method as the reference, the current work assessed the potential of NIRS for the

* Author to whom correspondence should be addressed [telephone (706) 546-3338; fax (706) 546-3607].

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Table 1. Range, Mean, and Standard Deviation (SD) of Total Dietary Fiber (TDF) Percent for Cereal and Grain Products in the Calibration Data Set

product type	no. of products	range in TDF (%)	mean TDF (%)	SD TDF (%)
wheat	27	2.9−41.0	13.8	9.8
oats	6	8.1−19.0	12.7	4.4
corn	5	0.6−13.6	5.5	5.2
rice	10	1.0−4.3	2.3	1.4
rye	5	15.4−38.4	23.2	9.3
barley	2	12.4−19.4	15.9	
millet	2	3.0−3.6	3.3	
buckwheat	1	5.4		
multiple grains	19	5.9−47.6	19.9	12.7
commercial oat or wheat fiber and cereal product mixes	13	6.4−52.1	30.1	14.8

determination of total dietary fiber content in a wide variety of grain and cereal products. A calibration was obtained for the prediction of total dietary fiber, and the calibration was used to accurately predict the total dietary fiber content of an independent set of cereal samples.

MATERIALS AND METHODS

Instrumentation. The NIRSystems 6500 Monochromator (NIRSystems, Silver Spring, MD) is a visible/near-infrared scanning monochromator with a tungsten source and a holographically ruled grating. Diffusely reflected radiation is detected from 400 to 2500 nm in 2-nm intervals. The lower wavelength region (400−1098 nm) is observed by a pair of silicon detectors located 20 cm from the surface of the sample cell and at an angle of 45° to the incident beam. The upper wavelength region (1100−2500 nm, used for the present study) is observed by two pairs of lead sulfide detectors in the same orientation as the silicon detectors. Reference reflectance values are obtained with a ceramic block. Samples are presented in a sample cell that is placed on an oscillating shaft with an axis of rotation parallel to the incident radiation.

Reagents. Heat stable α-amylase, A 3306; protease, P3910; amyloglucosidase, A 9913; acid washed Celite, C 8656; total dietary fiber control kit, TDF-C10; 2-(*N*-morpholino)ethanesulfonic acid, M-8250 (MES); and tris(hydroxymethyl)aminomethane, T1503 (Tris) were purchased from Sigma Chemical Company, St. Louis, MO. Buffer (MES/Tris, 0.05 M) was prepared and adjusted to pH 8.2 at 24 °C. Adjustment of the buffer to pH 8.3 is required if the temperature is 20 °C, and to pH 8.1 if the temperature is 28 °C, with interpolation for intermediate temperatures.

Enzyme Purity and Activity. Purity and activity of α-amylase, protease, and amyloglucosidase used in the AOAC dietary fiber procedure were monitored with the Sigma Total Dietary Fiber Assay Control Kit. Briefly, α-amylase and amyloglucosidase activity were monitored by measuring the recovery of corn or wheat starch, and protease by measuring the recovery of casein in the AOAC total dietary fiber procedure. Contamination by β-glucanase, hemicellulase, and pectinase activity were monitored by measuring the recovery of β-glucan, arabinogalactan, and citrus pectin, respectively.

Samples and Sample Preparation. Cereal and grain products (n = 122), including breakfast cereals, crackers, brans, and flours, were selected from retailers. Samples were dry milled to <500 μm in a cyclone mill (Cyclotec 1093 Sample Mill, Perstorp Analytical, Silver Spring, MD). Because of limited numbers of cereal products with high total dietary fiber content, commercial oat and wheat fibers (range in total dietary fiber, 90−98%) were mixed with several processed cereals to provide 17 samples with high, medium, or low dietary fiber content. Thirteen samples in the calibration set and four samples in the validation set were prepared in this way (Tables 1 and 2). Commercial oat and wheat fibers were provided by Canadian Harvest USA L. P. (Cambridge, MN).

Table 2. Range, Mean, and Standard Deviation (SD) of Total Dietary Fiber (TDF) Percent Cereal and Grain Products in the Validation Data Set

product type	no. of products	range in TDF (%)	mean TDF (%)	SD TDF (%)
wheat	9	3.6−43.7	14.0	13.4
oats	2	9.9−15.4	12.6	
corn	1	9.4		
rice	3	1.2−2.2	1.5	0.6
rye	4	9.7−18.1	14.0	3.5
buckwheat	1	5.03		
multiple grains	8	2.0−42.2	20.3	14.8
commercial oat or wheat fiber and cereal product mixes	4	25.4−42.8	36.5	7.7

All samples for calibration and validation contained 10% or less fat and 20% or less sugar.

Reference Laboratory Method for Total Dietary Fiber. Total dietary fiber in all samples was measured in the laboratory by the AOAC approved method 991.43 (AOAC, 1992; Lee et al., 1992). Briefly, duplicate samples of milled cereal products were suspended in MES/TRIS buffer (0.05 M, pH 8.2 at 24 °C) and incubated sequentially with heat-stable α-amylase (95−100 °C, 30 min), protease (60 °C, 15 min), and amyloglucosidase (60 °C, 15 min, pH 4.5 ± 1) to digest starch and protein. The enzyme digestate was then treated with four volumes of 95% ethanol (1 h) to precipitate soluble fiber. The alcohol-treated digestate was filtered (Fibertec System, E 1023 Filtration Module, Tecator, Höganäs, Sweden) through borosilicate sintered glass crucibles (40−90 μm) that had previously been matted with celite, dried, and weighed. All crucibles containing celite, residue, or ash were weighed by the hot gravimetric technique (Windham, 1987). The total dietary fiber residue present in the crucible was washed with alcohol and acetone, dried overnight (105 °C), and weighed. One duplicate from each sample was used for ash determination (495 °C muffle furnace) and the other for protein determination. Protein was determined using the LECO, FP-2000 Protein/Nitrogen Analyzer (LECO Corporation, St. Joseph, MI) by the AOAC method 990.03 (AOAC, 1990c). Dry matter of milled cereal products was determined by the AOAC air oven method 945.14 (AOAC, 1990a). Samples were weighed by the hot gravimetric technique (Windham, 1987).

Total dietary fiber percent (TDF%) was calculated as

$$\text{TDF\%} = (100/\text{DM}) \times 100 \times \{[(R_1 + R_2)/2] - \text{protein} - \text{ash} - \text{blank}\}/[(S_1 + S_2)/2]$$

where DM is the percent dry matter, R_1 and R_2 are the residue weights for duplicate samples, and S_1 and S_2 are the sample weights.

Spectroscopic Analysis. Dry milled cereal samples were scanned with the NIRSystems 6500 monochromator (NIRSystems, Silver Spring, MD) to obtain reflectance spectra. Duplicates of each sample were presented in cylindrical sample cells (internal diameter, 38 mm; depth, 9 mm) with optical quartz surface and cardboard backing. Each sample was scanned 16 times, and the data were averaged and transformed to $\log_{10}(1/R)$. The duplicate scans of each sample were examined visually for consistency and averaged.

Multivariate Calibrations. Ninety-one cereal samples were scanned for the development of a calibration equation. The wavelength region used for analysis was 1100−2500 nm, with 2-nm intervals. A commercial spectral analysis program (NIRS3, Infrasoft International, Inc., Port Matilda, PA) was used to process the data and develop chemometric models, and partial least squares (PLS) regression was the method selected (Lindberg et al., 1983). The optimum number of PLS factors used for total dietary fiber prediction was determined by cross validation (Martens and Naes, 1989). During cross validation, one sixth of the calibration samples at a time was temporarily removed from the calibration set and used for prediction. Performance statistics were accumulated for each group of removed samples. The optimal number of factors for total

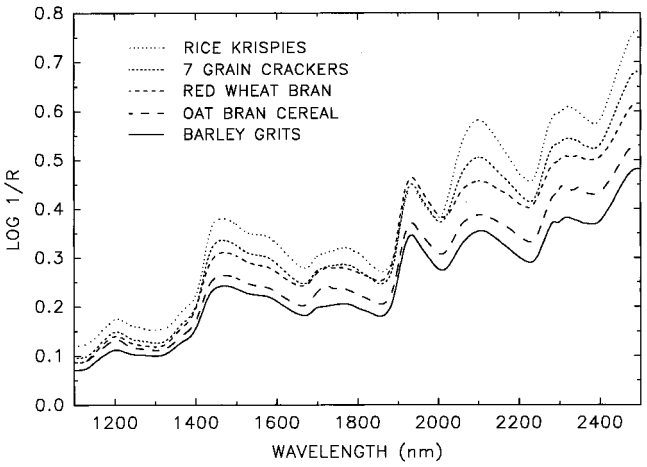

Figure 1. Typical NIR spectra of five cereal samples from the calibration data set.

dietary fiber was that which produced a minimum in overall error between modeled and reference values (standard error of cross validation). Prior to the PLS procedure, $\log_{10}(1/R)$ spectra were mean centered, transformed with standard normal variate and detrending procedures (Barnes et al., 1989), to remove multiplicative interferences of scatter, and then transformed with second-derivative processing (gap = 20 nm, smoothing interval = 10 nm). The transformations improved the standard error of cross validation compared with PLS analysis with untransformed data.

Upon completion of the calibration, the model was validated with an independent set of 31 cereal samples. Model performance was reported as the coefficient of determination (r^2), the standard error of performance, and the average difference between measured and modeled values (bias). Samples with a range in total dietary fiber content of 20 to 90% ($n = 4$), but containing the same matrix (Snowite Oat Fiber from Canadian Harvest USA L. P. and Common Sense Oat Bran from Kellogg Company), were scanned to observe systematic changes in $\log_{10}(1/R)$ with total dietary fiber content.

RESULTS

Total Dietary Fiber Measured by the Reference Method. The values for total dietary fiber in cereal samples determined by the AOAC enzymatic-gravimetric procedure ranged from <1 to 52% ($n = 122$). The standard error of laboratory determinations (Windham, et al., 1989) for the calibration data set was 0.69%. For the calibration and validation data sets, the distribution of samples of each grain type was similar (Tables 1 and 2). Likewise, the range, mean, and standard deviation of total dietary fiber percent, within grain types, were similar for each data set.

NIRS Calibration for Total Dietary Fiber. NIRS spectra obtained were typical of spectra for cereal samples (Figure 1; Murray and Williams, 1987). One sample was discarded, based on principal component analysis, as a spectral outlier (Mahalanobis distance, >3). An NIR calibration equation, using PLS, was obtained for the concentration of total dietary fiber in cereal products. The overall error between modeled and reference values (standard error of cross validation), using six cross validation groups, was 1.58%, with a multiple coefficient of determination (R^2) of 0.99 (Table 3). Linear regression of AOAC-determined dietary fiber against NIRS-predicted dietary fiber ($Y = -0.004 + 1.00X$) gave an intercept and slope not significantly different from 0.0 and 1.0, respectively ($p > 0.05$, Figure 2).

Equation Validation. Independent samples were predicted with the calibration equation. When NIR-predicted values for total dietary fiber were compared statistically with AOAC-determined values, the standard error of performance was 1.51% and the coefficient of determination (r^2) was 0.99 (Table 3). Linear regression of AOAC-determined dietary fiber against NIR-predicted dietary fiber ($Y = -1.46 + 1.06X$) gave an intercept and slope not significantly different from 0.0 and 1.0, respectively ($p > 0.05$, Figure 3). Two samples were identified as residual outliers (having a difference between AOAC-determined and AOAC-predicted values of 2.5 times, or greater, the standard error of the difference between the two values) and removed.

Loadings. Nine factors were employed in the calibration equation. The factors employed explained 98.9% of the spectral variation and 98.5% of the variation in total dietary fiber data. Sample scores with the highest correlation (calculated by Pearson Correlation Coefficient) with dietary fiber were for factors 2, 3, and 4, with correlation coefficients of −0.52, 0.72, and 0.31, respectively.

Loadings are the regression coefficients of each variable (wavelength) for each factor and indicate which variables (wavelengths) are dominantly influencing the model. The loading plots for factors 2, 3, and 4 are shown in Figure 4. Examination of Figure 4 suggests that certain peaks are common among the loadings (i.e., peaks at 1416, 2262, 2304, and 2328 nm). Sample scores from factor 3 had the highest correlation with dietary fiber and a loading (Figure 4B), with large intensities related to O−H absorption in the water band at 1416 nm and the carbohydrate band at 2082 nm (Murray and Williams, 1987; Williams and Norris, 1987b). Loading 3 also had large intensities related to C−H absorption in the carbohydrate bands at 2262 and 2328 nm and the aliphatic C−H bands at 1722 and 2304 nm. Factor 2 had the second highest correlation with dietary fiber and its loading (Figure 4A) was dominated by O-H absorption in the water bands at 1413 and 1920 nm. Loading 2 also had significant intensity related to C−H absorption in the carbohydrate band at 2262 nm and the aliphatic C-H band at 2304 nm, and due to C═O absorption in the protein band at 2052 nm. Factor 4 had the third highest correlation with dietary fiber with intensities (Figure 4C) due to C−H absorption in the carbohydrate bands at 2262 and 2328 nm and the aliphatic C−H band at 2310 nm. Overall, for the three loadings, absorbance was predominantly influenced by effects due to O−H and C−H groups in the water and carbohydrate bands.

Factor 1 was not correlated with total dietary fiber content ($r = 0.08$). However, factor 1 was highly correlated with sample moisture ($r = 0.96$). The loading for factor 1 contained O−H absorption in the water bands at 1428 and 1932 nm. The range in moisture content of calibration samples was 3.0−12.5% with mean and standard deviation of 7.8 and 2.5%, respectively. For the validation samples the range in moisture content was 3.6−12.2% with mean and standard deviation of 7.6 and 2.3%, respectively.

Variation in Dietary Fiber Concentration. The spectra of samples containing 20, 40, 60, and 90% total dietary fiber are shown in Figure 5. Systematic changes in absorbance with varying total dietary fiber concentrations occurred in the C−H and O−H absorption regions. Systematic change in peak intensity with increased total dietary fiber concentration corresponded to the greatest influence in loadings 2, 3, and 4 (i.e., at

Table 3. Calibration and Validation Statistics for Dietary Fiber Prediction by NIRS[a]

	calibration ($n = 90$)				validation ($n = 29$)				
method	mean	SD	SECV	R^2	mean	SD	Bias	SEP	r^2
AOAC	16.54	13.36			17.43	13.77			
NIRS	16.54	13.32	1.58	0.99	17.81	12.89	−0.38	1.51	0.99

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

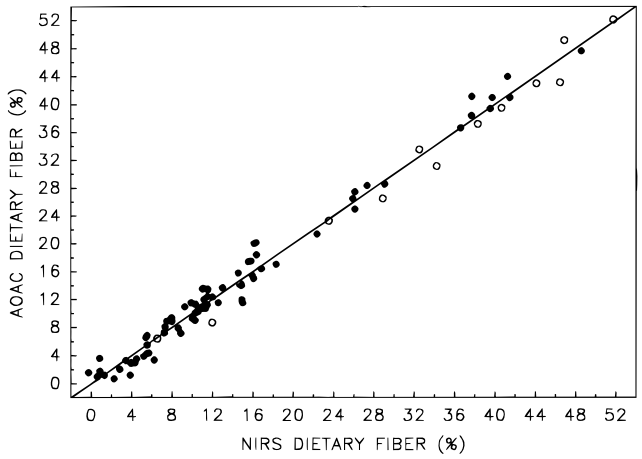

Figure 2. AOAC-determined total dietary fiber versus NIR-predicted total dietary fiber for cereal products in the calibration data set ($n = 90$). Open circles indicate commercial oat or wheat fiber and cereal product mixtures.

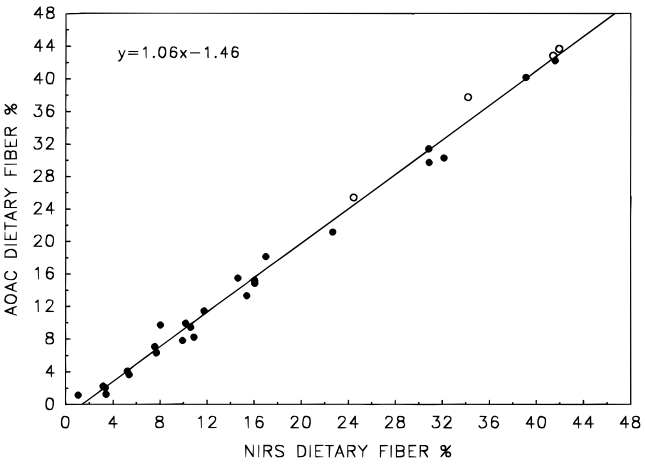

Figure 3. AOAC-determined total dietary fiber versus NIR-predicted total dietary fiber for cereal products in the independent validation data set ($n = 29$). Open circles indicate commercial oat or wheat fiber and cereal product mixtures.

wavelengths 1922 and 2326 nm), with a shift in the peak at 2060 nm.

Multivariate Analysis of Sample Subset. A model was developed using a subset ($n = 42$) from the calibration data set. The subset consisted of cereal samples containing wheat and oat grains only. The model utilized four factors, the overall standard error of cross validation was 1.74%, and $R^2 = 0.98$. Independent samples containing oat and wheat only ($n = 11$) were predicted with the subset calibration model. When NIRS predicted values for total dietary fiber were compared with AOAC-determined values, the standard error of performance was 1.75%, $r^2 = 0.99$, bias = −0.13, and slope = 1.06.

DISCUSSION

The potential of using NIRS for the prediction of dietary fiber in cereal products was investigated. A calibration was obtained using NIRS for the prediction of dietary fiber in a wide range of cereal products, and the calibration was found to accurately predict the total dietary fiber content of similar cereal samples. The standard error of performance, coefficient of determination, bias, and slope observed indicated a high degree of accuracy and reliability in determining total dietary fiber by NIRS.

Examination of the individual loadings in the calibration equation indicates that effects related to O−H and

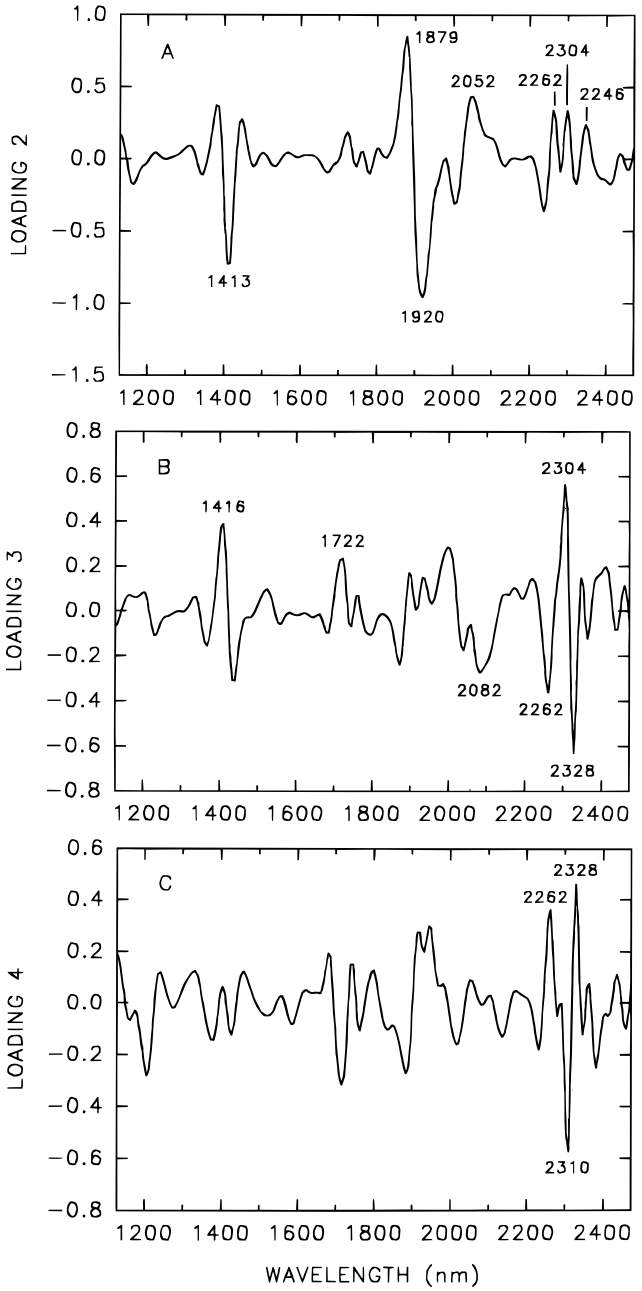

Figure 4. PLS loading spectra for total dietary fiber in cereal products. Panels A, B, and C represent loadings for factors 2, 3, and 4, respectively.

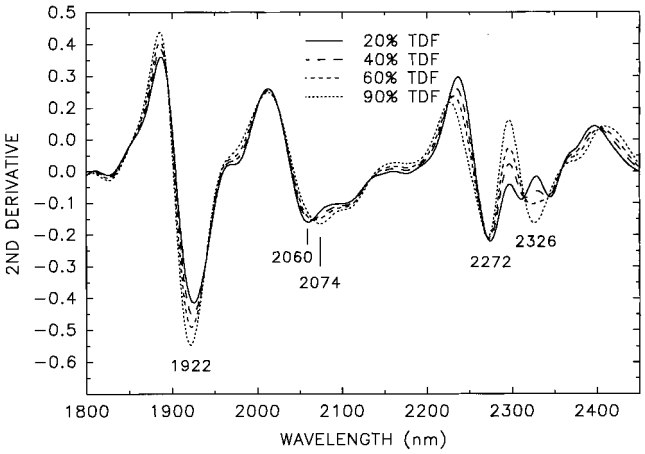

Figure 5. Second-derivative NIR spectra of cereal samples that have a range in total dietary fiber (TDF) content and contain the same matrix.

C−H groups in the water and carbohydrate bands are most important in the model. For this data set the three factors with the highest correlation to dietary fiber have loadings with significant intensity in these regions of the spectrum. It is not known whether the influence of O−H absorption is due to residual water or carbohydrate. Other constituents, such as lipid and protein, appear to be contributing to the model as shown by the intensity of loadings associated with regions of the spectrum typically associated with aliphatic C−H and protein absorbance (e.g., 2304 and 2052 nm, respectively). These results are similar to observations by Williams et al. (1991) in oat bran products. They observed influences in the carbohydrate, water, lipid, and protein absorption regions of the spectrum in the important loadings for total dietary fiber, and observed influences from lipid and protein absorption in the loadings for soluble dietary fiber and neutral detergent fiber.

The calibration model developed in the current study utilized a very wide range of cereal types and dietary fiber values (Table 1). Included in the set were wheat, oats, rye, barley, amaranth, corn, rice, and wild rice (both processed and unprocessed). Furthermore, many samples contained several types of grain in a single product. The model was used to predict total dietary fiber in an independent set of samples that had a similar diversity of products and range, mean, and standard deviation of dietary fiber values. The diversity of the data set is reflected in the number of factors in the model. The calibration reported here employs nine factors, with 88% of the variability for total dietary fiber modeling being explained by factors 2, 3, and 4. This type of equation has broad utility with manufacturers using multiple grains and multiple grain products and for regulatory agencies monitoring products from many sources.

Many manufacturers process products that contain one or two types of grains. For these industries, a simpler model may suffice. When samples containing oat and wheat only are utilized from the present data set, a new calibration is obtained with four factors, and similar standard error of cross validation, multiple correlation coefficient, and accuracy of prediction as the model derived from the original complete data set. Thus, calibration development and sample prediction can be successfully achieved for determination of dietary fiber in either homogeneous or heterogeneous groups of products.

Two categories of cereals not included in the calibration model were cereal products containing >10% fat or >20% sugar. Cereals in these categories were, understandably, not predicted well by the model. Preliminary data has shown that the prediction of dietary fiber in high fat/sugar samples can be accurately achieved by scanning the sample after extraction of the fat or sugar rather than scanning the original sample. It would be advantageous to be able to predict the dietary fiber in high fat/sugar samples without the need for solvent extraction; therefore, expansion of the calibration to include high fat and high sugar samples is the subject of ongoing research.

The accuracy of predicting total dietary fiber by NIRS is within the precision required by the Food and Drug Administration. FDA regulations state that "The nutrient content of the composite is at least equal to 80% of the value for that nutrient declared on the label" (*Code of Federal Regulations*, 1995). This level of accuracy can be easily achieved using NIRS for samples of moderate to high fiber levels. Although, below ≈3% fiber precision tends to decline, the FDA further states "no regulatory action will be based on a determination of a nutrient value that falls below this level by a factor less than the variability generally recognized for the analytical method". Hence, NIRS provides an acceptable method for measurement of total dietary fiber in a wide cross section of samples with a wide range of fiber values.

CONCLUSIONS

Near-infrared reflectance spectroscopy can be used to rapidly and accurately predict, the total dietary fiber content of a wide range of cereal products. Visual assessment of the spectral loadings suggests that analytically useful absorbance is dominated by effects related to O-H and C-H groups in the water and carbohydrate regions of the spectrum.

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