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Near-Infrared Spectroscopy for Measurement of Total Dietary Fiber in Homogenized Meals

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Near-infrared (NIR) reflectance spectroscopy was investigated as a method for prediction of total dietary fiber (TDF) in mixed meals. Meals were prepared for spectral analysis by homogenization only (HO), homogenization and drying (HD), and homogenization, drying, and defatting (HDF). The NIR spectra (400−2498 nm) were obtained with a dispersive NIR spectrometer. Total dietary fiber was determined in HDF samples by an enzymatic−gravimetric assay (AOAC 991.43), and values were calculated for HD and HO samples. Using multivariate analysis software and optimum processing, partial least squares models ($n = 114$) were developed to relate NIR spectra to the corresponding TDF values. The HO, HD, and HDF models predicted TDF in independent validation samples ($n = 37$) with a standard error of performance of 0.93% (range 0.7−8.4%), 1.90% (range 2.2−18.9%), and 1.45% (range 2.8−23.3%) and r^2 values of 0.89, 0.92, and 0.97, respectively. Compared with traditional analysis of TDF in mixed meals, which takes 4 days, NIR spectroscopy provides a faster method for screening samples for TDF. The accuracy of prediction was greatest for the HDF model followed by the HD model.

KEYWORDS: Near-infrared; composition; fiber; high moisture; homogenized meals; ready-to-eat; RTE

INTRODUCTION

Mixed meals are packaged foods that contain two or more food groups, usually a protein (animal or plant) and carbohydrate or vegetable in single or multiple portion containers. They have steadily gained popularity in the United States, partly because most are available to the consumer as ready-to-eat or ready-to-heat-and-eat meals; they are convenient, save time, and can be eaten at home (*1*).

U.S. nutrition labeling regulations require total dietary fiber (TDF) to be reported on the nutrition labels of frozen, packaged products including mixed meals (*2*). Total dietary fiber is defined (*3*) as the edible parts of plants or analogous carbohydrates that are resistant to digestion and absorption in the human small intestine with complete or partial fermentation in the large intestine. Dietary fiber includes nonstarch polysaccharides, resistant starch, oligosaccharides, lignin, and associated plant substances. Traditional TDF analysis for monitoring mixed meals includes homogenization, drying, and defatting of samples followed by an enzymatic−gravimetric (*4*) or enzymatic−chromatographic assay (*5*). This requires 4 days of processing time, is labor intensive, and generates chemical waste.

Near-infrared (NIR) spectroscopy presents a rapid, inexpensive, accurate, and environmentally benign method for the analysis of a variety of components in grains, feeds, cereal foods, oilseeds, dairy products, beverages, meats, and forages (*6*−*8*) and is used internationally for the evaluation of grain quality (*9*). The technique is based on the correlation between chemical properties of a sample, as determined by the traditional chemical methods of analysis, and the absorption of light at different wavelengths in the NIR region of the spectrum. The NIR region (700−2500 nm) is sensitive to molecules containing C−H, O−H, and N−H groups, which are the primary constituents in foods. NIR spectroscopy has been used for the analysis of TDF and other components in low moisture foods such as cereal foods and dehydrated vegetables (*10*, *11*). For example, TDF has been predicted using NIR for intact and ground, diverse cereal food products. Prediction of TDF in these studies was accurate enough for on-line screening and quality control (*12*−*14*). However, mixed meals pose special problems partly because of their high moisture and, often, high fat content and partly because of the diversity of materials present. Mixed meals can contain meat, seafood, fish, vegetable, cereal, soy, and dairy products in any number of combinations and proportions. Although nothing has been published on the NIR evaluation of TDF in mixed meals, Almendingen et al. developed NIR calibrations for the prediction of crude fat and protein in homogenized freeze-dried human diets (*15*). Büning-Pfaue et al. developed calibrations for dry matter crude fat, crude protein, and carbohydrate in homogenized, cafeteria style "consumable meals" (*16*), and White and Rouvinen-Watt developed NIR calibrations for dry matter, crude protein, and crude fat in wet homogenized mink diets (*17*).

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Table 1. Range, Mean, and Standard Deviation of TDF (Percent) in Mixed Meal Samples in the Calibration and Validation Data Sets

	calibration				validation			
	n[a]	range	mean	SD	*n*	range	mean	SD
homogenized meals	114	0.2–8.5	2.8	1.8	37	0.7–8.4	2.9	1.9
animal protein base[b]	42	0.2–3.7	1.4	0.7	12	0.7–2.9	1.4	0.7
vegetable protein base[c]	33	1.8–8.5	4.5	1.8	8	1.6–8.4	3.2	2.2
carbohydrate base[d]	39	0.6–7.8	2.8	1.4	17	1.1–7.3	3.8	1.7
HD meals	113	1.0–24.1	9.5	5.0	37	2.2–18.9	9.5	4.8
animal protein base	41	1.0–15.9	5.8	2.7	11	2.7–10.6	5.0	2.2
vegetable protein base	34	6.5–24.1	13.9	4.7	8	6.2–18.9	12.5	4.7
carbohydrate base	38	2.2–17.3	8.8	3.9	18	4.6–18.4	11.0	4.2
HDF meals	114	1.1–31.3	10.7	5.6	37	2.8–23.3	11.3	5.8
animal protein base	41	1.1–17.0	7.1	3.0	12	2.9–12.8	5.9	2.6
vegetable protein base	34	6.8–31.3	15.4	5.5	8	6.8–23.3	13.2	6.1
carbohydrate base	39	2.6–21.8	10.4	4.6	17	7.7–22.1	14.1	4.8

[a] Number of samples (*n*). [b] Meat or fish based with vegetable. [c] Bean based. [d] Flour or rice based with meat or vegetable.

On the basis of the success of NIR spectroscopy for rapid measurement of TDF in low moisture food products with no or little sample preparation (*13*, *14*), the current study was conducted to evaluate the feasibility of NIR spectroscopy for the prediction of TDF in mixed meals. Models were developed for a wide variety of meals using samples at three preparation levels: homogenized only (HO); homogenized and dried (HD); and homogenized, dried, and defatted (HDF).

MATERIALS AND METHODS

Samples. One hundred fifty-three mixed meal samples were obtained from retail stores and selected to represent the types available in the marketplace. Most samples were purchased frozen in retail boxes, seven were purchased in cans, and nine were purchased in pouches at room or refrigerator temperature. The selected samples were readily available entrées that required only heating and contained two or more food groups. One hundred fifty-one samples were used for the study, 114 for the calibration set and 37 for the independent validation set. Categories of samples are shown in **Table 1**. Samples encompassed a broad range in major constituents, i.e., dietary fiber (0−16%), carbohydrate (3−35%), fat (1−18%), and protein (2−37%), based on the product's nutrition label information, a wide range in moisture content (29−90%), and a diversity of additional ingredients such as salt, spices, and other seasonings. Soups were excluded from the sample set due to their extremely high moisture content and fluid consistency.

Sample Preparation. Frozen, packaged, or canned samples were removed from their containers and immediately homogenized using the Robot Coupe homogenizer (model RSI 10, Robot Coupe USA Inc., Joliet, IL) until a smooth and consistent texture was obtained. After homogenization, the samples (referred to as HO) were placed in individual polyethylene freezer bags and allowed to equilibrate to room temperature, and a subsample was scanned to obtain NIR spectra. A second subsample was weighed in an aluminum dish and dried in a forced air oven (105 °C for 16 h). The loss of weight was recorded. The dried samples (referred to as HD) were each divided into two subsamples. One HD subsample was ground using an analytical mill (model 4301-00, Cole Parmer Instrument Co., Vernon Hills, IL) and scanned immediately to obtain NIR spectra. The other HD subsample was fat extracted using petroleum ether and a Soxhlet apparatus with 4 h of extraction time (*18*). The loss of weight was recorded. The defatted samples (referred to as HDF) were ground in an analytical mill and divided into two subsamples. One HDF subsample was scanned to obtain NIR spectra, and the other was used for measuring TDF using AOAC method 991.43 (*4*).

Spectroscopic Analysis. The HO, HD, and HDF samples were scanned to obtain NIR reflectance spectra using a NIRSytems 6500 monochromator (Foss North America Inc., Eden Prairie, MN), described previously (*12*), with ISI40 software (NIRS3 version 4.01, Foss North America Inc.). Samples were scanned in triplicate, to include intrasample variation, in cylindrical cam-lock cells (internal diameter = 38 mm, depth = 9 mm). Diffusely reflected radiation from 400 to 2498 nm at 10 nm resolution and a data interval of 2 nm was collected and recorded. The data were transformed to log 1/*R*, and scans from the triplicate samples were averaged. Spectral data collected on different dates were standardized to spectra of a specific date using the WINISI monochromator instrument standardization software (Foss North America Inc.). The spectral data were converted into JCAMP format and imported into the Unscrambler software 9.0 (CAMO, Trondheim, Norway).

Analysis of TDF. The TDF was measured in HDF samples by AOAC method 991.43 (*4*), as previously described (*12*), using the Megazyme TDF assay kit (Megazyme International Ireland Ltd., Bray, Ireland) and calculated as follows:

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

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 (*12*). The dry matter of HDF samples was determined by the AOAC air oven method 945.14 (*19*). The TDF for HDF samples was expressed on a dry weight basis. The TDF was calculated for HD samples based on fat loss. The TDF for HO samples was calculated based on fat and moisture loss and expressed on an as-is basis.

Calibration Development. Samples (n =153) were divided into calibration and validation sets after sorting by ascending TDF reference data values for HO samples. The first three samples were assigned to the calibration set, and the fourth to the validation set and so on. Partial least squares (PLS) (*20*, *21*) was the regression method used to develop the models for each of the three sample preparation levels. The wavelength range and preprocessing methods used for each sample set were selected based on the options that gave optimum performance, i.e., minimum error with full cross-validation (*21*). The optimum wavelength ranges used to calculate models with the HO, HD, and HDF data sets were 700−2498, 1100−2498, and 1100−2498 nm, respectively. The spectra of the HO samples were preprocessed using Savitzky−Golay first derivative (*22*, *23*) (second order polynomial, seven point convolution interval); the spectra of the HD samples were preprocessed with the multiplicative scatter correction (MSC) (*24*) followed by the Savitzky−Golay first derivative treatment (second order polynomial, seven point convolution interval); and the spectra of the HDF samples were preprocessed using MSC followed by the Savitzky−Golay first derivative treatment (second order polynomial, nine point convolution interval). The optimum number of PLS factors for calibration was also determined by cross-validation.

Calibration Performance. Statistics used to assess the PLS models were the multiple coefficients of determination (R^2) and standard errors of cross-validation (SECV) (*21*, *25*). In addition, the models were tested with independent validation samples (n = 38), and performance was reported as the coefficient of determination (r^2), standard error of performance (SEP), root-mean-square standard error of performance

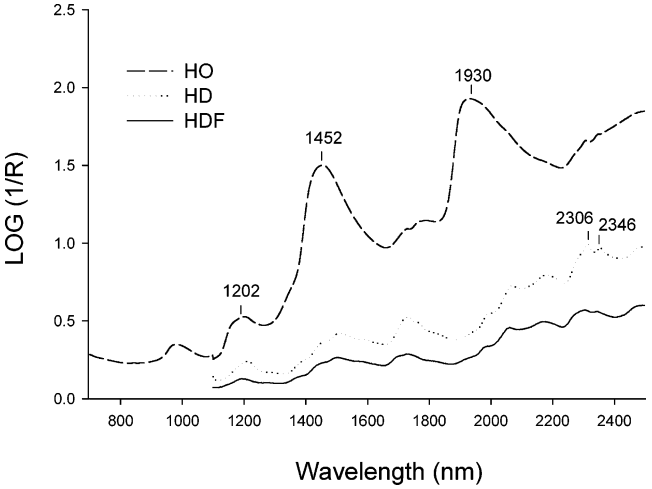

Figure 1. NIR (log 1/*R*) spectra of a representative mixed meal sample at three sample preparation levels: HO, HD, and HDF. Respectively, TDF, moisture, and fat contents in HO samples were 3.5, 62, and 6.7%; in HD samples, they were 9.2, 1.8, and 19%; and in HDF samples, they were 11, 1.9, and 1.0%.

(RMSEP), which is corrected for bias, and RPD (*21*, *25*, *26*). The RPD (*26*) is the ratio of the standard deviation of the reference values to the SEP and provides a standardization of the SEP (values of 3.1−4.9 generally indicate that the model is suitable for screening, and values of 2.4−3.0 indicate that the model is suitable for rough screening).

RESULTS

Spectral Characteristics. Principal differences in the bands of the spectra for the three sample preparation methods are due to the quantities of moisture and fat present. Log (1/*R*) spectra of the HO samples have two dominant and broad peaks at 1452 and 1930 nm for water, as shown in the spectrum of a representative sample in **Figure 1**. The two peaks involve the first overtone of the O−H stretching bands (1452 nm) and the combination of the O−H stretching and the O−H bending bands (1930 nm) (*7*, *27*−*29*). The O−H stretching and combination bands are also observed as the largest peaks in the second derivative spectrum of the HO sample as shown in **Figure 2**. In addition, smaller peaks are observed at or near 1726, 1760, 2306, and 2346 nm due to absorption by C−H stretch groups in lipids. For the HD sample (**Figure 1)**, the log(1/*R*) peak in the 1400−1452 nm range is less pronounced and the peak around 1930 nm is not evident, as expected since the majority of the water has been removed. The second derivative spectrum of the HD sample (**Figure 2**) has a less pronounced peak at 1406 nm relative to other peaks in the HD sample. Lipid-related bands at 1726, 1760, 2306, and 2346 nm were sharper and were the most dominant peaks for the sample. However, significant peaks were also evident at or near 1976 and 2056 nm due to NH stretch and amide in protein. In the HDF samples, second derivative peaks at or near 1976, 2056, 2268, 2306, and 2346 nm are the most dominant with an additional peak at 1680 nm that may be due to vibration of aromatic C−H from lignin (a component of TDF). Peaks around major lipid-absorbing areas are still evident but much smoother (the sample contained 1.0% residual fat).

TDF Measured by the Reference Method. The overall ranges for TDF in HO, HD, and HDF samples, using AOAC method 991.43, were 0.2−8.5, 1.0−24.1, and 1.1−31.3%, respectively (**Table 1**). Removing moisture and fat extended the range and changed the distribution of values for TDF in the resulting data sets. Among different meal types, vegetable

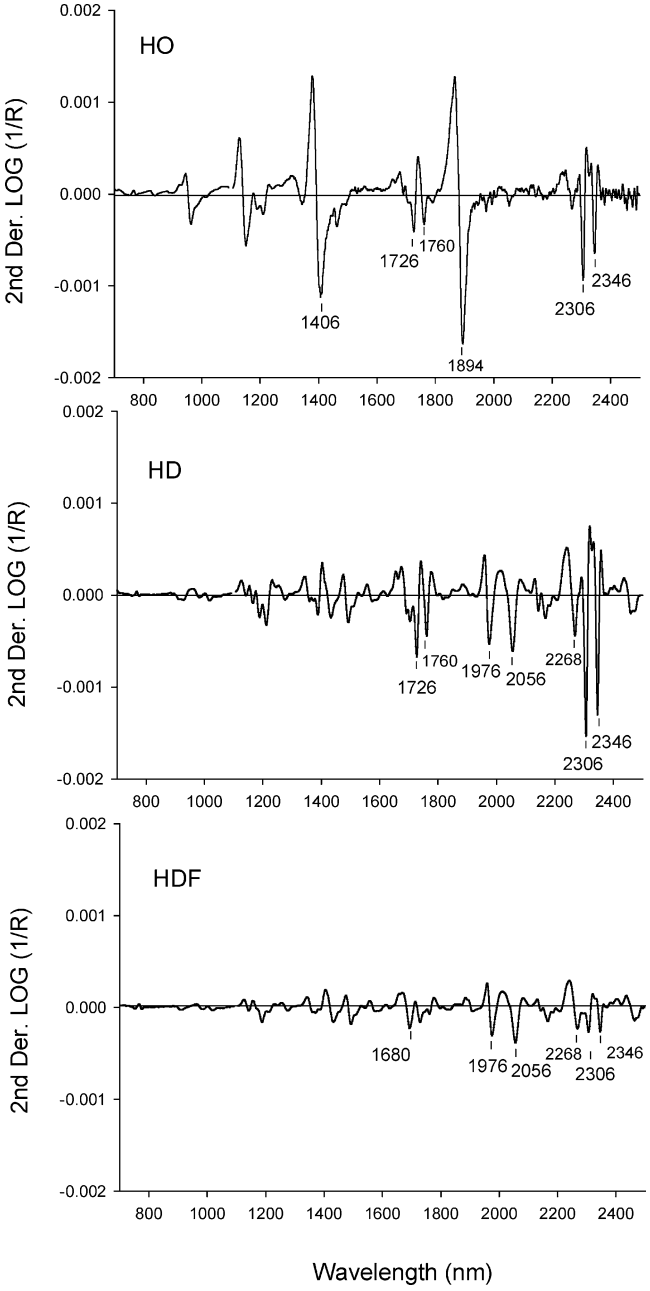

Figure 2. NIR second derivative spectra of the mixed meal sample from **Figure 1** at three sample preparation levels: HO, HD, and HDF.

protein-based meals and carbohydrate-based meals had wider ranges in TDF for both the calibration and the validation sets than animal protein-based meals. The range, mean, and standard deviation of TDF were similar within calibration and validation sets. **Table 1** shows the distribution of TDF values in HO, HD, and HDF samples used for the calibration and validation sets. The distribution was positively skewed in all sets. Ideally, sample sets for the calibration and validation should be assembled with a uniform distribution of composition across the anticipated range. However, mixed meals in the marketplace are predominantly lower than 5.5% in TDF content. As many high fiber meals, i.e., greater than 5.5% TDF, as possible were included in the data sets. Calibration and validation sets have similar distribution patterns for the HO samples, whereas the HD and HDF validation data sets did not contain samples in the highest part of the TDF range as compared to the calibration samples.

Table 2. Cross-Validation and Independent Validation Statistics for NIR Prediction of TDF in Mixed Meal Samples at Different Sample Preparation Levels

	cross-validation				independent validation					
model[a,b]	*n*[c]	NIR range	SECV	R^2	*n*	NIR range	SEP	RMSEP	r^2	RPD
HO	114	0.2–6.8	0.99	0.84	37	0.1–8.5	0.93	0.92	0.89	2.1
HD	113	0.5–22.6	1.83	0.93	37	3.7–20.2	1.90	1.95	0.92	2.5
HDF	114	2.2–29.1	1.54	0.96	37	3.1–23.4	1.45	1.44	0.97	4.1

[a] Model sample preparation level: HO, HD, and HDF. [b] Model wavelength range: HO, 700–2498 nm; HD and HDF, 1100–2498 nm. [c] Number of samples, *n*.

NIR Model for TDF. Individual NIR models were developed for the prediction of TDF using PLS regression (*20*, *21*). Spectral outliers were identified and removed from the HO and HD calibration sample sets (one outlier in the HO set, two outliers in the HD set) and HO, HD, and HDF validation sample sets (one outlier in each set). Six PLS factors were used for the HO model, which had an error between predicted and reference values (SECV) of 0.99% TDF (range 0.2−8.5%) and R^2 of 0.84 (**Table 2** and **Figure 3**). Ten factors were used for the HD model, which had a SECV of 1.83% TDF (range 1.0−24.1%) with an R^2 of 0.93. Nine factors were used for the HDF model, which had a SECV of 1.54% TDF (range 1.1−31.3%) and R^2 of 0.96. When independent validation samples were predicted with the HO, HD, and HDF models, the SEP was 0.93% TDF (range 0.7−8.4%) with an r^2 of 0.89 for the HO model, 1.90% TDF (range 2.2−18.9%) with an r^2 of 0.92 for the HD model, and 1.45% TDF (range 2.8−23.3%) with an r^2 of 0.97 for the HDF model (**Table 2** and **Figure 4**). RPD values were 2.1, 2.5, and 4.1 for HO, HD, and HDF models, respectively, indicating model suitability for screening purposes in the case of the HDF model and rough screening in the case of the HD model.

Modeling of HO samples using the 700−1800 nm wavelength range (RMSEP = 0.98% TDF, r^2 = 0.86) resulted in model performance very similar to that using 700−2498 nm. In contrast, HD and HDF models calculated without the 1800−2498 nm range were substantially lower in accuracy (RMSEPs were 2.3−2.6% TDF) than when the range was included.

The distribution of TDF values was positively skewed in all sample data sets (**Table 1**). However, reduction of the number of samples below 3% TDF in the HO data set using a selection algorithm (WinISI, Foss North America Inc.) did not appreciably affect performance of the HO model (data not shown).

Performance of the models was not appreciably different when used to predict the subgroups of meals in the independent validation samples, i.e., meals that were animal protein-based, vegetable protein-based, or carbohydrate-based.

Regression Coefficients for TDF PLS Models. Regression coefficients for the model constructed with the HO samples have high variation at 1700−1750 nm (**Figure 5**), indicating possible involvement of C−H stretch groups in lipids in development of this model (*7*, *30*). Regression coefficients for the models developed with the HD and HDF samples have high variation at 2070 nm and the 2200−2498 nm regions of the NIR spectrum indicating that absorption related to O−H, C−H, and C−C groups in carbohydrate are the major factors in model development. The intercept at approximately 1690 nm indicates possible involvement of aromatic C−H from lignin (*7*).

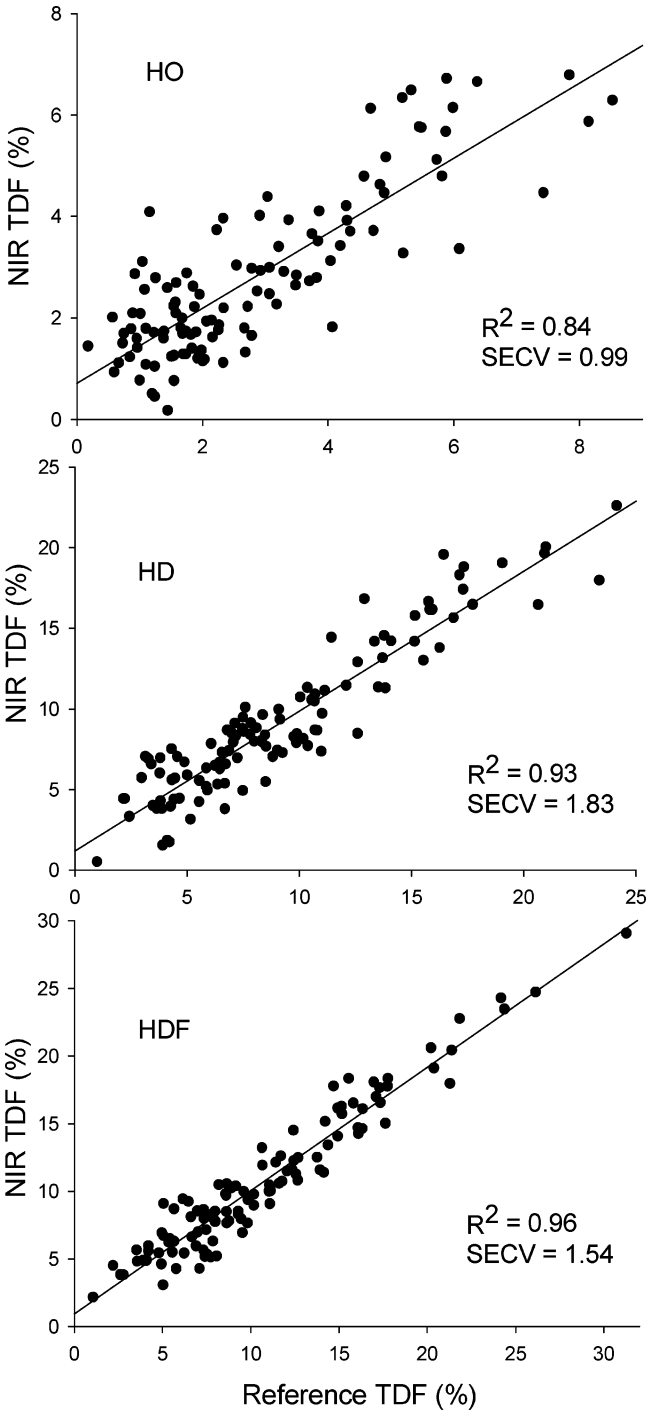

Figure 3. Calibration plots of AOAC-determined TDF vs NIR-predicted TDF for three models to predict TDF in mixed meals. Models were developed with HO, HD, or HDF mixed meal samples.

DISCUSSION

Total dietary fiber analysis by traditional methods is very time-consuming, taking 4 days to complete, partly due to the extensive sample preparation required (2 days) but also due to the TDF assay itself (2 days). To develop a rapid method for the determination of TDF, the current study investigated the use of NIR reflectance spectroscopy. In an attempt to reduce

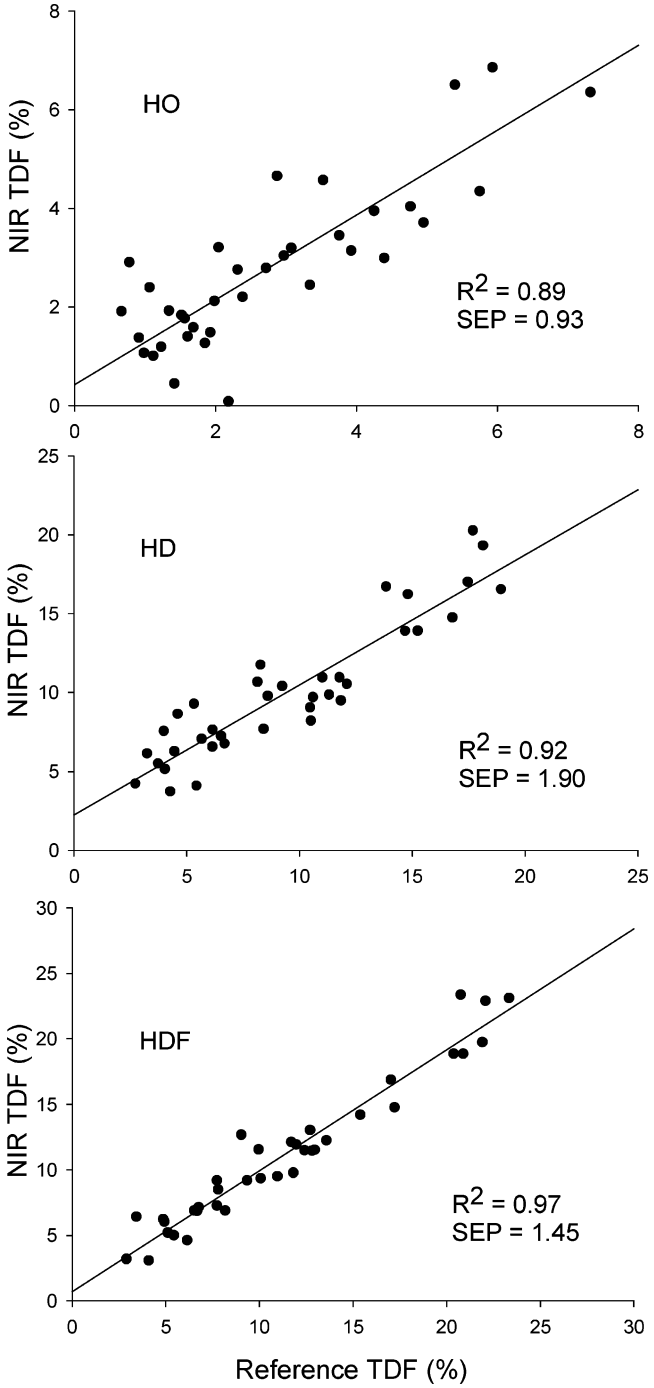

Figure 4. Validation plots of AOAC-determined TDF vs NIR-predicted TDF for three models to predict TDF in mixed meals. Models were developed with HO, HD, and HDF mixed meal samples.

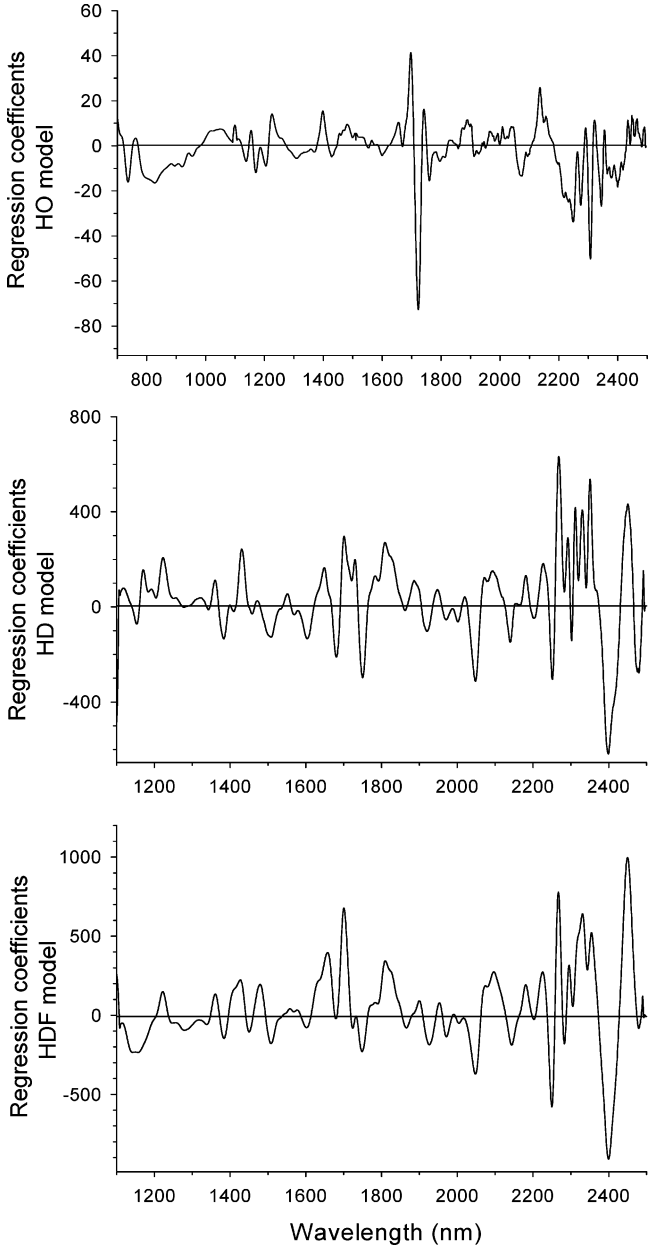

Figure 5. Regression coefficients for three PLS models to predict TDF in mixed meals. Models were developed with HO, HD, or HDF mixed meal samples.

sample preparation and analysis time, three different sample preparations were used for NIR spectral analysis; samples were homogenized only (HO) to reduce total analysis time per sample to less than 1 h; homogenized and dried (HD) to reduce total analysis time to 24 h; and homogenized, dried, and defatted (HDF) to shorten total analysis time to 2 days.

The present results indicate that NIR spectroscopy is able to determine the TDF content in a wide variety of mixed meals. In PLS models, the RPD and coefficients of determination of the cross-validation and the independent validation improved with increased sample preparation. The model developed with samples that were homogenized and dried was intermediate among treatments in accuracy for prediction of TDF in mixed meals. Use of this NIR model reduces the TDF total analysis time from 4 days to 24 h. Removing fat as well as moisture from the samples significantly improved the modeling of TDF by NIR spectroscopy. The RPDs indicate that the HDF model is the most precise of the models and is adequate for screening TDF in mixed meals. Performing spectroscopic analysis with the dried and defatted samples reduced the TDF analysis time from 4 days for conventional analysis to 2 days.

From the regression coefficients, it appears that the HDF model was predominantly influenced by the wavelength region from 2200 to 2498 nm, with absorbance related to O−H, C−H, and C−O groups in carbohydrates. Previously reported NIR models developed to predict TDF in cereal products were also influenced by groups in the 2200−2400 nm range and by O−H groups in water (*12*, *13*, *31*, *32*). However, most of the water had been extracted from the HDF samples and water was not a major influence in the HDF model. Aromatic C−H at 1690 nm,

associated with aromatic groups in lignin, appears to be an additional influence in the HD model. The regression coefficients for the HD and HDF models reflect the composition of TDF in the samples.

The predominant influence observed in the regression coefficients for the HO model appeared to be from lipid with little influence from carbohydrate. The regression coefficients for the HO model were observed to have poor correlation above 1800 nm. This could be because the TDF content of many of the HO samples is low (most are below 5%); thus, information for TDF in sample spectra might have been obscured by the broad water peak in the 1850−2300 nm area. The absence of useful information from this region could have substantially limited TDF model development and explain the lower accuracy of the HO model. The narrow range of TDF in mixed meals, especially in the HO model, might also be limiting accuracy. Model performance could possibly be improved for all models by expanding the TDF range by artificially fortifying some samples with commercially available fiber.

The HD model could be limited in accuracy by the high fat (>10%) nature of most of the samples at this level of sample preparation. The range in fat content of the HD data set was 0.2−41.5% (Kim, unpublished data). High fat samples are a problem as they are difficult to grind and often have a "pastelike" consistency (*26*). This can lead to in-homogeneity of the sample and problems in obtaining representative NIR spectra and, thus, reduced model accuracy. The HDF samples, in contrast, were relatively dry and fat free, did not pose problems in grinding, and were likely to be more homogeneous than HO or HD samples. Because the TDF assay was performed on the HDF samples and TDF values were then calculated for HD and HO samples using fat and water extraction factors, the accuracy of the reference data was likely to be greater for HDF samples and their corresponding spectra.

Prediction of TDF in HDF mixed meals is less accurate than prediction of TDF in ground cereal products (*12*, *13*, *32*) possibly because of the more complex nature of the meals. Mixed meals can contain animal material, fish or seafood, as well as plant material (grains and vegetables) in infinite combinations and proportions. This is in contrast to cereal products, where the predominant ingredient is cereal grains. Thus, the complex combination of materials in mixed meals together with the wide range in moisture content may significantly reduce the accuracy of NIR calibrations, as compared to calibrations for pure ingredients and less complex foods.

In summary, NIR spectroscopy can provide a rapid method for determination of TDF in mixed meals with substantial savings in time if samples are dried or dried and defatted prior to obtaining NIR spectra. The accuracy of NIR prediction of TDF increases with drying and further by drying and defatting from RPDs of 2.1 and 2.5 for HO and HD samples, respectively, to an RPD of 4.1 for HDF samples. Development of the NIR models for TDF in dried or dried and defatted mixed meals appears to be dependent on absorbance by C−H, O−H, and C−C groups in carbohydrates and aromatic C−H groups in lignin.

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