The use of near infrared spectroscopy to measure total dietary fiber content of cereal products

Sandra E. Kays, W.R. Windham and F.E. Barton, II

USDA-Agricultural Research Service, Richard B. Russel Agricultural Research Center, PO Box 5677, Athens, GA 30613, USA.

Introduction

The importance of dietary fiber to human health has long been recognized. To aid consumers in selecting a healthy diet, the United States Nutritional Labeling and Education Act (NLEA) requires that the amount of total dietary fiber be included on food labels. The method currently used for dietary fiber analysis in the USA is time consuming and personnel intensive. Thus, a rapid and accurate method of dietary fiber determination would help industries in compliance with the NLEA and could be essential for efficient monitoring of compliance.

Near infrared (NIR) reflectance spectroscopy represents a very rapid and accurate method of measuring constituents of materials without requiring extensive sample preparation, nor creating chemical waste.[1,2] In the past, NIR reflectance spectroscopy has been used successfully to determine fat, moisture and protein content of agricultural products.[1] Very little is known about the potential of NIRS to determine total dietary fiber in foods. In 1983, Baker[3] reported the successful determination of neutral detergent fiber content of breakfast cereals by NIR reflectance spectroscopy. Similarly, Williams *et al*.[4] reported analysis of total, soluble and insoluble dietary fiber in oat bran products using NIR reflectance spectroscopy. The current work was conducted to assess the potential of NIR reflectance spectroscopy for the determination of total dietary fiber content in a wide variety of grain and cereal products.

Materials and methods

Samples and sample preparation

Cereal products were selected from grocery stores or kindly provided by Canadian Harvest USA LP (Cambridge, MN). Products included flours, brans, breakfast cereals, crackers and commercial oat fibers. Samples were dry milled using the Cyclotec 1093 sample mill (Perstorp, Sweden) to pass through a 0.5 mm screen. All samples contained less than 10% fat or sugar.

NIR spectroscopic analysis

Cereal samples were scanned using an NIRSystems 6500 monochromator (NIRSystems, Silver Spring, MD). Two independent sets of data, the calibration set (n = 67) and the validation set (n = 25) were scanned. Reflectance measurements were recorded at 2 nm intervals from 1100 to 2500 nm, and averaged over 16 scans. The data were analysed using ISI software (Infrasoft International Inc., State College, PA). De-trending and standard normal variate transformation were used to remove particle size effects. The math treatment was a second derivative with a gap

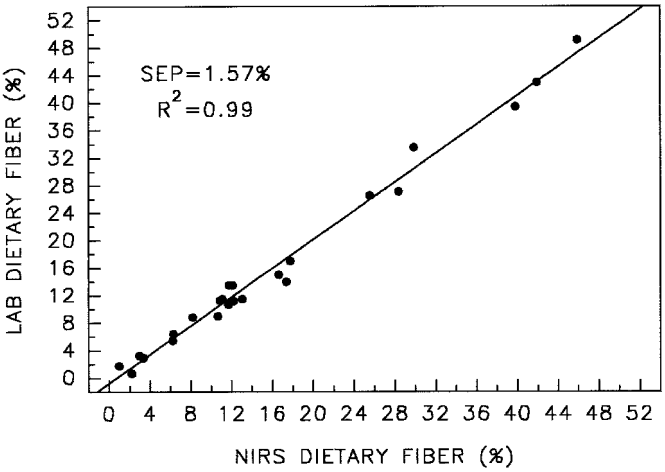

Figure 1. NIR spectroscopic predicted values versus laboratory reference method values for total dietary fiber. Bias = –0.03%, *n* = 24.

of 10 and a smoothing interval of five data points. Principal component analysis (PCA) and partial least squares (PLS) calculations were made on the second derivative spectra. A third data set, containing samples of the same matrix (Snowite Oat Fiber from Canadian Harvest USA LP and Common Sense Oat Bran from Kellog Company) with a range in total dietary fiber content (20–90%), was scanned for examination of the spectra only.

Laboratory reference method

AOAC method 985.29,[5] with modifications,[6] was used as the reference laboratory method to determine total dietary fiber content of the cereal products.

Results

Calibration

An NIR spectroscopic calibration was obtained for the concentration of total dietary fiber in cereal products. The standard error of cross-validation was 1.67% and R^2 = 0.98 (n = 66). One sample was discarded by the PLS program as a spectral outlier. The standard error of triplicate laboratory determinations was 0.63% and the range in fiber values <1–52%.

Validation

NIR predicted values versus laboratory values of 25 independent cereal samples used for equation validation resulted in a standard error of performance (*SEP*) of 1.57%, R^2 = 0.99, and bias of –0.03% (n = 24, Figure 1). One sample was discarded as a residual outlier.

Loadings

Ten terms were used in the calibration. The three loadings having the highest correlation with total dietary fiber were 2, 3 and 5 with correlation coefficients of –0.54, –0.64 and –0.29, respectively. Visual assessment of spectral loadings suggested that absorbance was dominated by effects related to water and carbohydrate. The weights have a large intensity in the $–CH_2–$ and

CH_3 absorption region (2200–2400 nm) and therefore probably describe the variation in total dietary fiber.

Variation in dietary fiber concentration

Systematic changes in absorbance were observed in the 2300–2400 nm absorption region in spectra of samples containing 20, 40, 60 and 90% total dietary fiber (data not shown).

Conclusions

While the ability of NIR reflectance spectroscopy to determine total dietary fiber in all cereal products is an ongoing investigation, NIR appears to have significant potential for industries needing continual monitoring of total dietary fiber. The *SEP*, R^2 and bias observed in the current study indicate a high degree of precision in determining total dietary fiber by NIR spectroscopy. Preliminary examination (data not presented) indicates that cereal products containing more than 10% fat or sugar, without extraction, are not adequately modeled. These products are the object of ongoing research.

References

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