Prediction of energy content in cereal food products by near infrared reflectance spectroscopy

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Introduction

Energy content is an important part of the evaluation and marketing of foods in many countries. United States dietary guidelines urge consumers to aim for a healthy body weight by choosing an assortment of foods that includes vegetables, fruits, grains, skimmed milk and fish, lean meat, poultry or beans.[1] The benefits of managing weight and avoiding obesity include a reduced risk for high blood pressure, heart disease, stroke, diabetes and certain forms of cancer.[1] Thus, knowledge of energy content of foods and food portions is one of many important criteria in the selection of foods by consumers.

Methods of measuring the energy value of foods are outlined in the US Code of Federal Regulations.[2] One method is measurement of gross calories by bomb calorimetry with an adjustment for unutilised protein; another is by calculation, using specific factors for the energy values of protein, carbohydrate less the amount of insoluble dietary fibre and total fat. Near infrared (NIR) spectroscopy has been used for the rapid and accurate prediction of nutrients in human foods[3] but very few studies have addressed the use of NIR for the prediction of energy.[4] Previous work has described the use of NIR for the prediction of energy content in feeds for ruminants and monogastric animals.[5,6] The current study explores the potential of NIR for the rapid and accurate determination of gross energy in cereal food products. Subsequent work will address the determination of physiologically available energy.

Materials and methods

Samples and sample preparation

Cereal food products included breakfast cereals, granolas, crackers, brans, flours, unprocessed grains and commercial oat and wheat fibres. The sample set had a wide range in grain types, multi-grain products, fibre, added fat and sugar and additives such as dried fruit, honey, herbs, nuts, cinnamon and cocoa. Samples were ground to < 500 µm in a cyclone mill (Cyclotec 1093, Perstorp Analytical, Silverspring, MD, USA) except for high-fat samples (> 10% fat) which were ground in a coffee mill (Model KSM-2, Braun Inc., Lynnfield, MA, USA).

Spectroscopic analysis

Near infrared reflectance spectra (400–2500 nm) of ground cereal samples were obtained in duplicate with an NIRystems 6500 monochromator (NIRystems, Silver Spring, MD, USA) using cylindri-

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cal sample cells (38 × 9 mm). Each sample was scanned 16 times, the data averaged and transformed to log 1/*R*. The duplicate scans of each sample were averaged.

Reference data

For the initial study, energy listed on the nutrition facts label of the products was used as the reference data. Total calories were converted to kcal g^{-1} using label information for serving size. Oxygen bomb calorimetry[7] was used to measure gross energy content of products at the University of Georgia, Poultry Nutrition Laboratory, Athens, GA, USA. Dry matter content of products was determined using a forced air oven at 105°C and gross energy expressed as kcal g^{-1} on a dry weight basis.

Multivariate calibrations

Multivariate analysis was performed using ISI software (NIR3 v. 4.01, ISI International, Port Matilda, PA, USA). For the initial study, with reference data based on nutrition label information, a selection algorithm (SELECT, NIR 3 v. 4.01) was used to select representative samples from the population of 116 spectra. Using a neighbourhood *H* value of 0.6 to define neighbourhoods and principal component analysis, 43 samples were selected and used to develop a modified PLS model for energy prediction. Log 1/*R* spectra were processed using normal multiplicative scatter correction and second derivative processing (gap = 16 nm, smoothing interval = 16 nm) prior to modified PLS. In addition, a modified PLS model was developed for the prediction of gross energy using calorimetry to determine gross energy reference values. One spectral outlier (Mahalanobis distance > 20) was discarded. Log 1/*R* spectra of 128 cereal food products were transformed with normal multiplicative scatter correction and second derivative processing (gap = 16 nm, smoothing interval = 16 nm), prior to modified partial least squares analysis. The number of modified PLS factors used for each model was determined by cross validation. The model for prediction of gross energy was tested using independent validation samples (n = 58). Validation samples consisted of additional cereal food products not included in the calibration data set and purchased and scanned at a different time.

Results

Reference analysis

The range in energy content, calculated from product nutrition label values was 1.70–5.00 kcal g^{-1}. After employing the selection algorithm to select representative samples the range was 2.0–4.8 kcal g^{-1} (n = 43). The range in gross energy of samples measured by calorimetry was 4.05–5.49 kcal g^{-1} with a method standard error of 0.035 kcal g^{-1}.

Initial model based on product nutrition label values

The initial model used 43 selected, representative samples with reference data for energy calculated from the product nutrition label information (Figure 1). The modified partial least squares model developed used five PLS factors and had a standard error of cross-validation (*SECV*) and multiple coefficient of determination (R^2) of 0.26 kcal g^{-1} and 0.84, respectively. These

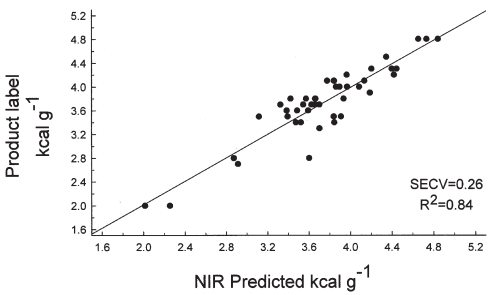

Figure 1. NIR predicted values for energy vs values for energy calculated from product nutrition label information for cereal food products in the calibration data set (n = 43 samples selected from a pool of 116 using a selection algorithm).

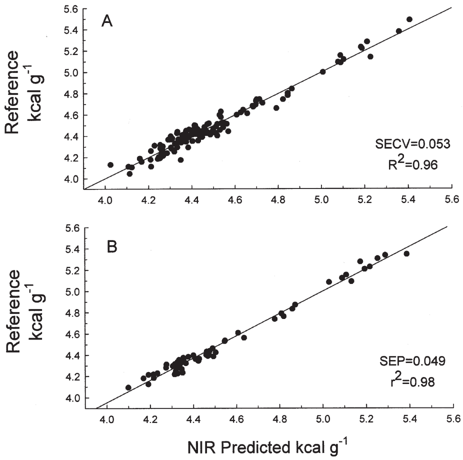

Figure 2. NIR predicted values for gross energy vs gross energy determined by bomb calorimetry for cereal products in the calibration (panel A, *n* = 127) and validation data sets (panel B, *n* = 58).

results indicated promise for NIR reflectance spectroscopy for prediction of energy in diverse cereal food products, therefore, the study was continued measuring the gross energy content using bomb calorimetry.

Calibration for gross energy

A modified PLS model was developed for the prediction of gross energy (Figure 2). The *SECV* was 0.053 kcal g^{-1} and R^2 was 0.96. Seven modified PLS factors were used in the model and described 97.1% of the spectral variation. Sample scores having the highest correlation with gross energy were for factor one and had a Pearson correlation coefficient of 0.92. The PLS loading for factor one had significant absorption peaks correlated to C–H stretch groups in lipids at 1212, 1722, 1764, 2304 and 2346 nm and O–H groups in carbohydrates at 1434 and 2076 nm. Thus, the model appeared to be predominantly influenced by lipids and carbohydrates. The model was used to predict the gross energy of independent validation samples (n = 58) with a standard error of performance of 0.049 kcal g^{-1}, coefficient of determination of 0.98, bias of – 0.020 kcal g^{-1} and slope of 1.05.

Discussion

The results indicate that gross energy of a diverse group of cereal products can be predicted accurately and rapidly with NIR reflectance spectroscopy using ground products. The model derived from calorimetry data is far more accurate than that derived from nutrition label values. This is not surprising as label values are not precise. The US Code of Federal Regulations[2] states that the number of calories per serving can be "expressed to the nearest five calorie increment up to and including 50 calories and ten calorie increment above 50 calories".

The gross energy of cereal products is a useful figure in diet selection, however, the actual energy physiologically available is generally less, primarily due to incomplete oxidation of proteins in the human body and indigestibility of fibre. Thus, adjustments in gross energy values for incomplete utilisation of protein and indigestibility of fibre will more accurately reflect food energy available to the body. These values for available energy may be calculated from the predicted values for gross energy, if the protein and insoluble dietary fibre composition of samples is known. Direct and accurate prediction of available energy by NIR spectroscopy will be the subject of subsequent work.

Acknowledgement

Calorimetry work was conducted at the University of Georgia, Poultry Nutrition Laboratory.

References

1. *Dietary Guidelines for Americans,* United States Department of Agriculture, United States Department of Health and Human Services, Home and Garden Bulletin No. 232 (5th Edn) (2000).

2. *Code of Federal Regulations*, FDA, HHS; 21, part 101.9 (2000).
3. B.G. Osborne, T. Fearn and P.H. Hindle, *Practical NIR Spectroscopy with Applications in Food and Beverage Analysis*; Longman Scientific and Technical, Harlow, UK (1993).
4. E. Lanza, *J. Food Sci.* **48,** 471 (1983).
5. D.L. Givens, J.L. De Boever and E.R. Deaville, *Nutrition Research Reviews* **10,** 83 (1997).
6. S. Leeson, E.V. Valdes and C.E.M. de Lange, in *Feed Evaluation: Principles and Practice*. Wageningen, Pers, Stichting, The Netherlands, p. 93 (2000).
7. *Parr Manual No. 120*, *Oxygen Bomb Calorimetry and Oxygen Bomb Combustion Methods*. Parr Instrument Company, 211 Fifty Third Street, Moline, Illinois, USA (1948).