Analysis of nutritional components in cereal foods with near infrared reflectance spectroscopy

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Introduction

Simultaneous determination of constituents, using near infrared (NIR) reflectance spectroscopy, could have a significant impact on the evaluation of nutrients in foods as regulated by the U.S. Nutrition Labeling and Education Act.[1] Previous work has described the use of NIR reflectance spectroscopy for the rapid determination of total dietary fibre and insoluble dietary fibre in a broad range of cereal and grain products with a wide range of fibre values. The initial models did not include products with > 10% fat or > 20% sugar.[2,3] However, the models were subsequently expanded to include high fat products (for example, crackers and granola type breakfast cereals) and high sugar products (for example, frosted breakfast cereals).[4] Near infrared methods reduced the time of assay for dietary fibre from two to three days by the AOAC enzymatic–gravimetric method (AOAC Method 991.43)[5] to a few minutes. Determination of additional nutritional components, such as protein and fat, at the same time as dietary fibre, would increase both the speed and efficiency, while substantially reducing the cost of nutrient analysis. Near infrared spectroscopy has been used extensively to measure protein and fat in specific grains and oil seeds in the ground state or whole kernel forms.[6–9] However, little has been reported on the potential of NIR spectroscopy to predict protein and fat in diverse cereal products. This paper summarises the data from the work already completed[2–4] and presents the current results for prediction of nutritional components in cereal food products by NIR reflectance spectroscopy.

Materials and methods

Samples and sample preparation

Cereal products in the calibration and validation data sets included breakfast cereals, crackers, brans, flours and unprocessed grains as described previously.[2,4] Grains represented in the sample set were wheat, oats, rice, rye, corn, millet, amaranth, buckwheat and triticale. Samples were dry-milled to < 500 μm in a cyclone mill (Cyclotec1093, Perstorp Analytical, Silver Spring, MD, USA). High fat samples were ground in a coffee mill (model KSM-2, Braun Inc., Lynnfield, MA, USA). The initial calibration data set (n = 90) and validation data set (n = 29) for total and insoluble dietary fibre contained products with < 10% fat and <20% sugar, as high fat and high sugar interfered with the AOAC assays for dietary fibre.[5] The expanded calibration and validation data sets used for total dietary fibre, insoluble dietary fibre, protein and fat included products with high fat (> 10%) and high sugar (> 20%). Fat and sugar were extracted from these samples before the assays for total and insoluble dietary fibre were performed.[4]

Near Infrared Spectroscopy: Proceedings of the 9th International Conference

Reference laboratory methods

Dry milled cereal samples were assayed for: total and insoluble dietary fibre by an enzymatic–gravimetric method (AOAC Method 991.43);[5] protein by combustion analysis (AOAC Method 992.23);[10] and total fat using the Soxtec 1040 Extraction System (Perstorp Analytical) with petroleum ether as the solvent (AOAC Method 945.16).[11] Laboratory results were expressed as percent of dry weight, following dry matter determination by the air-oven method (AOAC 945.15).[12]

Spectroscopic analysis

All dry-milled cereal samples were scanned with the NIRSystems 6500 monochromator (NIRSystems, Silver Spring, MD, USA) to obtain reflectance spectra. Duplicates of each sample were scanned in a cylindrical sample cell (internal diameter 38 mm, depth 9 mm) with quartz window. Samples were scanned 16 times, the data averaged and transformed to $\log_{10}(1/R)$.

The duplicate scans of each sample were averaged.

Multivariate calibrations

A spectral analysis program (NIRS3 V. 4.01, Infrasoft International Inc., Port Matilda, PA, USA) was used for recording spectral data and for multivariate data analysis. The initial models for total and insoluble dietary fibre contained 90 samples. The expanded model ($n = 117$) for total dietary fibre used 77 initial samples plus an additional 40 high fat and high sugar samples selected from a pool of 57 high fat or high sugar samples using an algorithm called SELECT,[13] as previously described.[3] Likewise, 36 high fat and high sugar samples were selected via PLS from a pool of 57 to add to the initial insoluble dietary fibre calibration data set ($n = 90$) to form the expanded insoluble dietary fibre calibration ($n = 126$). The expanded model for total fat was developed by selecting 90 samples, using the SELECT algorithm and PCA, from a pool of 147 cereal samples. Due to a preponderance of samples below 3% total fat (61 samples out of 90), 16 samples with 0–3% total fat were selected for the calibration using the SELECT algorithm and PLS. The result was 45 samples in the total fat calibration. For each calibration data set, $\log_{10}(1/R)$ spectra were mean centred and transformed with standard normal variate and de-trending procedures followed by first derivative (protein) or second derivative (total dietary fibre , insoluble dietary fibre and fat) processing. Partial least squares was the regression method used for all models and the optimum number of factors for each model determined by cross-validation. During cross-validation, a sixth of the samples at a time was temporarily removed from the calibration set, to be used for prediction. The number of PLS factors determined for each model was eight or nine, with the exception of fat, where the number of PLS factors was four. Performance statistics were accumulated for each group of removed samples. Performance was reported as standard error of cross validation (*SECV*) and multiple coefficient of determination (R^2). The performance of each model was also evaluated using an independent set of cereal samples, purchased and scanned at a different time from the calibration samples and model performance reported as standard error of performance (*SEP*), coefficient of determination (r^2), slope and bias.

Results and discussion

Parameters measured by the reference method

The range in total dietary fibre measured by the AOAC, enzymatic–gravimetric method was from 0.3 to 52.1% for both models and the standard error of the laboratory determinations[14] was 0.69% for the initial model and 0.73% for the expanded model. For insoluble dietary fibre, the range was from 0 to 48% for both models and the standard error of the laboratory determinations was 0.46% for the initial model and 0.49% for the expanded model. For protein the range was 2.56 to 20.7% and the stan-

Table 1. Calibration and validation statistics for prediction of nutrients in cereals by near infrared reflectance spectroscopy.

		Calibration data set					Validation data set						
Model	Method	*n*	Mean	*SD*	*SECV*	R^2	*n*	Mean	*SD*	*SEP*	r^2	Bias	Slope
TDF 1	AOAC	90	16.54	13.36	—	—	29	17.43	13.77	—	—	—	—
	NIR	90	16.54	13.32	1.58	0.99	29	17.81	12.89	1.51	0.99	–0.38	1.05
TDF 2	AOAC	117	12.59	12.13	—	—	72	10.98	11.48	—	—	—	—
	NIR	117	12.63	11.91	1.73	0.98	72	10.74	11.37	1.33	0.99	–0.58	1.00
IDF 1	AOAC	90	13.32	12.44	—	—	32	14.56	12.56	—	—	—	—
	NIR	90	13.34	12.43	1.34	0.99	32	14.63	12.44	1.13	0.99	-0.07	1.01
IDF 2	AOAC	126	10.44	11.24	—	—	62	9.57	10.71	—	—	—	—
	NIR	126	10.49	11.13	1.65	0.98	62	9.2	10.64	1.56	0.98	0.37	1.00
Protein	AOAC	147	11.99	3.40	—	—	72	11.29	3.38	—	—	—	—
	NIR	147	12.00	3.36	0.56	0.97	72	11.1	3.19	0.49	0.98	0.03	1.05
Total fat	AOAC	45	8.22	7.48	—	—	72	5.44	6.61	—	—	—	—
	NIR	45	8.26	7.42	1.16	0.98	72	5.54	6.71	0.96	0.98	–0.11	0.98

n = number of samples
SD = standard deviation
SECV = standard error of cross-validation
R^2 = multiple coefficient of determination
SEP = standard error of performance
r^2 = coefficient of determination
AOAC = Association of Official Analytical Chemists
NIR = near infrared
TDF 1 = total dietary fibre—initial model
TDF 2 = total dietary fibre—expanded model
IDF 1 = insoluble dietary fibre—initial model
IDF 2 = insoluble dietary fibre—expanded model

dard error of the laboratory determinations was 0.21%. For total fat the range was 0.02 to 25.6% and the standard error of the laboratory determinations was 0.18%.

NIR calibrations

Results for NIR reflectance models are presented in Table 1. Samples in the independent validation data sets were predicted with sufficient accuracy for nutrition labelling purposes, based on the US Food and Drug Administration regulations,[1] for total dietary fibre and crude protein using the initial and expanded models and for insoluble dietary fibre using the initial model. However, improvements are required in the expanded models for prediction of insoluble dietary fibre and total fat.

Conclusions

The potential of NIR reflectance spectroscopy for the evaluation of nutrients in cereal products is an on-going investigation. NIR spectroscopy appears to have significant potential for nutrition labelling and monitoring, especially for the evaluation of dietary fibre and protein.

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