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Near-infrared spectroscopic analysis of macronutrients and energy in homogenized meals

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Abstract

Near-infrared (NIR) reflectance spectroscopy was evaluated as a rapid and environmentally benign technique for the simultaneous determination of macronutrients and energy in commercially available, packaged meals. Reflectance spectra (400–2498 nm) of homogenized meals were obtained with a dispersive NIR spectrometer. Protein and moisture were measured by AOAC reference methods, total fat by a semi-automated acid hydrolysis, solvent extraction, gravimetric method and total carbohydrate calculated. Energy was calculated using Atwater factors. Using multivariate analysis software, PLS models ($n = 113$–115 products) were developed to relate NIR spectra of homogenized meals to the corresponding reference values. The models predicted components and energy in validation samples ($n = 37$–38 products), overall, with r^2 of above 0.96. Ratios of deviation to performance were between 3.6 and 6.6, and indicated adequacy of the models for screening, quality control, or process control. Performance of the models varied substantially when used to predict sub-groups of meals within the validation set.
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Keywords: High moisture; Fat; Protein; Carbohydrate; Convenience meals; Ready-to-eat; RTE; NIR

1. Introduction

Precooked and packaged meals sometimes called mixed meals, home meal replacements, ready-to-eat meals or ready-to-heat-and-eat meals are a significant portion of the retail grocery market in Europe and the USA and are gaining popularity worldwide. They are commercially available, packaged foods, typically already cooked, that contain two or more food groups, usually a protein (animal or plant) and staple (carbohydrate) or vegetable in single or multiple portion containers. As packaged products they are included under the US Nutrition Labeling and Education Act (Code of Federal Regulations, 2005). Component analysis of such foods is very time consuming partly because of the analysis time required but also because of the high moisture content and the need to dry samples prior to analysis. Near-infrared spectroscopic methods could provide an ideal method for rapid screening of such meals.

Near-infrared (NIR) spectroscopy is an analytical technique enabling several components to be determined simultaneously and rapidly without requiring extensive sample preparation (Norris, 1989). Since its first use for the analysis of protein and oil content in corn and

Abbreviations: ASTM, American Standards for Testing and Materials; HO, sample preparation homogenized only; HD, sample preparation homogenized and dried; HDF, sample preparation homogenized dried and defatted; *n*, number of samples; NIR, near infrared; PLS, partial least squares; R^2, multiple coefficient of determination; r^2, coefficient of determination; RPD, standard error of the reference data for the validation samples divided by the SEP; SD, standard deviation; SECV, standard error of cross-validation; SEL, standard error of the laboratory; SEP, standard error of performance.

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soybeans (Hymowitz, Dudley, Collins, & Brown, 1974), NIR spectroscopy has proved to be an excellent method for the nutrient analysis of foods, and has significant potential for the analysis and monitoring of foods for nutrition labeling (Kays & Barton, 2006). The technique is rapid, environmentally benign and has found widespread use in analyses for food components such as fat, protein, water, carbohydrate, and dietary fiber. Most prior studies on the prediction of food components have focused on developing specific NIR models for types of food, e.g., meat products (Ben-Gera & Norris, 1968), snack foods (Baker, 1985), dehydrated vegetables (Ito, Ippoushi, Azuma, & Higashio, 1999), oat bran products (Williams, Cordeiro, & Harnden, 1991), cereal products (Kays & Barton, 2006), dairy products (Giangiacomo & Cattaneo, 2007) and fish products (Adamopoulos & Goula, 2004; Uddin et al., 2006), and for animal diets (White & Rouvinen-Watt, 2004).

Commercially available homogenized dinners or meals are far more complex and varied, from one sample to another, than single food types or animal diets studied to date. The meals can include meat, seafood, fish, vegetables, fruits, dried fruits, nuts, grains, dairy, cereal products, food additives, dough conditioners, thickening agents, seasonings, spices and other ingredients in any number of combinations and proportions, with a wide range of cooking methods and ethnic cuisines. Their complexity makes them unique. Direct analysis of homogenized meals without the need for prior drying or drying and defatting would be advantageous; however, near infrared work on predicting the composition of human diets or meals is extremely limited. Almendingen, Meltzer, Pedersen, Nilsen, and Ellekjaer (2000) investigated the potential of NIR spectroscopy for prediction of fat and protein in homogenized daily diets of students; but, the diets were freeze-dried before obtaining NIR spectra. The only study that uses the spectra of human meals or diets that are homogenized only and not dried is our own work on prediction of total dietary fiber in commercially available packaged human meals (mixed meals) (Kim, Singh, & Kays, 2006). Results showed these samples also had to be dried to obtain useful correlations for dietary fiber. In the current paper we extend that study by developing useful models to predict protein, total fat, moisture, total carbohydrate and energy in dinners that are homogenized only. This addresses a deficit in the literature on the potential of NIR to predict composition of homogenized meals or dinners without prior drying.

2. Materials and methods

2.1. Samples and sample preparation

Samples were as described previously (Kim et al., 2006). Briefly, one hundred and fifty three packaged meals were selected from a cross section of retail stores to represent the types of meals commercially available. Ingredients included meat, fish, shell fish, eggs, dairy products, soybean products, vegetables, tomato products, sauces, cereal products, wine, oils, gums, starches, food coloring, and seasonings in varied combinations and with a wide range of cooking methods and ethnic cuisines. The meals contained two or more food groups, e.g., animal protein based (meat/fish) with vegetable (31% of the total samples purchased); plant protein based (legume) with vegetable or meat (29% of the samples purchased); or carbohydrate based (cereal) with vegetable or meat (40% of the samples purchased). The samples were homogenized, immediately after being removed from the commercial packaging, using a Robot Coupe homogenizer (Model RSI 10, Robot Coupe USA Inc., Joliet, IL) until a smooth and consistent texture was obtained. Frozen samples were homogenized while frozen. The homogenate of each sample was divided into sub-samples and placed in polyethylene freezer bags which were closed and allowed to equilibrate to room temperature. Immediately after equilibrating, one sub-sample (homogenized only, HO) was scanned to obtain NIR reflectance spectra; the other sub-sample was dried in a forced air oven at 105 °C for 16 h. The homogenized and dried (HD) samples were ground with an analytical mill (Model 4301-00, Cole-Parmer Instrument Co., Vernon Hills, IL) and divided into sub-samples; one was used to determine protein and total fat contents and a second was defatted using petroleum ether in a Soxhlet apparatus for 4 h to obtain the homogenized, dried, and defatted (HDF) samples. The HDF samples were used for ash determination.

2.2. Chemical analysis

All analyses were performed in duplicate. Moisture was determined by the AOAC air oven method 945.14 (AOAC, 1990) immediately after homogenizing the fresh samples. Total fat in HD samples was determined by the Foss Soxtec™ 2047 Soxcap™ and the Foss 2050 Soxtec Avanti Automatic System (FOSS North America, Inc., Eden Prairie, MN) for total fat, which involves acid hydrolysis of the sample followed by petroleum ether extraction and gravimetric measurement of the extracted lipid. Total fat differs from crude fat in that bound lipids are released by the hydrolysis step and included in the measurement. In this method, any non-lipid, petroleum ether-extractable substances are rinsed from the matrix after the acid hydrolysis step. Crude protein in HD samples was determined by AOAC method 990.03 (AOAC, 1997) using a LECO, FP-2000 Protein/Nitrogen Analyzer and LECO reagents (LECO Corporation, St. Joseph, MI). The nitrogen to protein conversion factor used was 6.25. Total fat and protein in HO samples were calculated based on moisture loss from HD samples. Ash in HDF samples was determined by AOAC Method 923.03 (AOAC, 2000) with a muffle furnace set at 550 °C. Ash content of HO samples was calculated based on moisture and fat loss from HDF samples. The standard error of the laboratory (SEL) was calculated for the reference methods for moisture, protein and fat (ASTM, 1995) based on HO samples. Although protein

and fat were measured in HD samples, values for each sample replicate were calculated based on water loss from HO samples before determining the SEL.

2.3. Calculation of total carbohydrate and energy

The US Code of Federal Regulations (2005) states that, for nutrition labeling purposes, "total carbohydrate content shall be calculated by subtraction of the sum of the crude protein, total fat, moisture and ash from the total weight of the food". Total carbohydrate in HO samples was, thus, calculated as the difference between 100 and the sum of the percentages of moisture, crude protein, total fat, and ash (Ferris, Flores, Shanklin, & Whitworth, 1995). The US Code of Federal Regulations (2005) states that, for nutrition labeling purposes, energy content of foods may be calculated by one of several methods that include "using the general factors of 4, 4, and 9 cal/g for protein, total carbohydrate, and total fat, respectively". Thus, energy in the current study was expressed as kilocalories per gram (kcal/g) in HO samples and was calculated from the percentage of crude protein, total carbohydrates and total fat. The conversion factors used were 4.0 kcal/g for protein and carbohydrates and 9.0 kcal/g for total fat (Ferris et al., 1995; Merrill & Watt, 1955). Pearson correlation analysis was performed on crude protein, total fat, total carbohydrate and energy using SAS version 9.1 (SAS Institute, Inc., Cary, NC).

2.4. Spectroscopic analysis

Samples were scanned in cylindrical cam-lock cells (internal diameter = 38 mm, depth = 9 mm) using the NIRSytems 6500 monochromator (Foss North America, Inc., Eden Prairie, MN), in reflectance mode, and ISI software (NIRS3 version 4.01, Foss North America, Inc., Eden Prairie, MN). Diffusely reflected radiation was detected from 400 to 2498 nm at 10 nm resolution with a data interval of 2 nm. Samples were scanned in triplicate portions to include intra-sample variation. The software was set so that each triplicate portion was scanned 16 times and the 16 scans were averaged. The data were transformed to log 1/*R* and scans from the triplicate portions of each sample were then averaged to give a single spectrum from which calibrations were developed.

2.5. Calibration development

As samples were scanned over a period of months, all spectral data from different dates were standardized to spectra of one specific month using the WinISI monochromator instrument standardization software (Foss North America Inc., Eden Prairie, MN), then converted and imported into the Unscrambler software 9.0 (CAMO, Trondheim, Norway). Samples were divided into calibration and validation sets after sorting by ascending order for each parameter in 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) regression (Martens & Martens, 1986; Martens & Naes, 1989) was used to develop calibration models ($n = 113$–115) for each parameter. The wavelength range and preprocessing methods for each model were chosen for optimum performance with minimum error following full cross validation. The optimum number of PLS factors for calibration was that which gave a minimum cross validation error. For protein and moisture, the HO sample spectra were preprocessed with the Savitzky-Golay first derivative treatment (Savitzky & Golay, 1964) followed by a multiplicative scatter correction (MSC) (Martens, Jensen, & Geladi, 1983). For total carbohydrate, the spectra of the HO samples were preprocessed with the Savitzky-Golay first derivative. For the total fat and energy models, the spectra of HO samples were preprocessed with MSC followed by the Savitzky-Golay second derivative treatment.

2.6. Calibration performance

Calibration performance was first calculated as the multiple coefficient of determination (R^2) and standard error of cross-validation (SECV) (Martens & Naes, 1989). The models were then tested by predicting validation samples ($n = 37$–38) and sub-groups of validation samples. Performance was reported as the coefficient of determination (r^2), the standard error of performance (SEP), the bias, and the ratios of deviation to performance (RPD) (Hruschka, 1987; Martens & Naes, 1989; Williams, 2001). The RPD is the standard deviation of reference data for the validation samples divided by the SEP and provides a standardization of the SEP (Williams, 2001). In general, RPD values of 3.1–4.9 indicate the model is suitable for screening, values of 5.0–6.4 are considered to be adequate for quality control, values of 6.5–8.0 are adequate for process control, and values of >8.0 are considered adequate for any application (Williams, 2001).

3. Results

3.1. Components measured and calculated by the reference method

The range, mean and standard deviation of reference values of components for samples in the calibration and validation sets are presented in Table 1. Samples encompassed a broad overall range in moisture (45–90%), total fat (0–17%), crude protein (2–24%), total carbohydrate (3–36%) and energy (0.4–3.0 kcal/g). The range, mean, and standard deviation of components were similar for the calibration and validation sets. The SEL for the measured components, moisture, crude protein, and total fat in HO samples were 0.17, 0.21, and 0.16, respectively. Positive correlations (Pearson's correlation coefficient) were observed between energy and other components with total fat having the highest correlation with energy (0.80)

Table 1
Range, mean, and standard deviation of AOAC reference values for protein, moisture, total fat, total carbohydrate, and energy in homogenized meals in the calibration and validation data sets

	Calibration				Validation			
	n^a	Range	Mean	SD	n	Range	Mean	SD
Protein (%)	115	1.90–23.91	8.31	5.02	37	2.70–23.07	8.50	5.16
Moisture (%)	115	44.81–90.41	69.42	10.02	38	48.95–87.14	69.82	9.87
Total fat (%)	113	0.15–17.12	4.27	3.25	38	0.31–14.62	4.34	3.21
Total carbohydrate (%)	114	3.01–36.16	16.12	7.18	37	4.20–35.15	16.56	7.25
Energy (kcal/g)	115	0.40–3.00	1.38	0.53	38	0.55– 2.56	1.39	0.52

[a] Number of samples (n); standard deviation (SD).

followed by carbohydrate (0.65) and crude protein (0.48). Ash content in the entire data set ranged from 0.4% to 3.3%.

3.2. PLS models for components and energy in homogenized meals

The NIR spectra of homogenized meals showed distinct peaks that reflect composition, as described previously (Kim et al., 2006). The cross validation and validation statistics for the optimum PLS models to predict protein, moisture, total fat, total carbohydrate and energy are given in Table 2. The wavelength range of 1100–2498 nm was optimum for all models except total fat. Modeling of total fat using the 1100–1898 nm wavelength range resulted in similar accuracy to that using the 1100–2498 nm range (SECV = 0.93%; $R^2 = 0.96$), however, fewer factors were required (i.e., three rather than four) and the 1100–1898 nm range was used for the model. In contrast, models for protein, moisture and total carbohydrate when calculated without the 1900–2498 nm range, were lower in accuracy or used more factors than when the entire 1100–2498 nm range was utilized. Models for crude protein, total fat, and total carbohydrate calculated using wavelength ranges that exclude the main water peaks (1400–1460 nm and 1870–2026 nm) did not have improved performance compared with models calculated with the full wavelength range.

The number of PLS factors used for prediction of protein, moisture, total fat, total carbohydrate and energy were 5, 3, 3, 7 and 4, respectively. Performance statistics for the models ($n = 113$–115) to predict protein, moisture, total fat, total carbohydrate and energy are given in Figs. 1 and 2 and Table 2. One outlier was removed from the calibration sample set for the total carbohydrate model and two for the total fat model. One outlier was removed from the validation sample set for the protein and total carbohydrate models.

The RPD values for the overall models indicated the protein, moisture and energy models are suitable for process control and quality control, with RPDs of 6.6, 6.1 and 6.3, respectively. The total fat and total carbohydrate models are adequate for screening purposes with RPDs of 4.3 and 3.6, respectively.

3.3. Regression coefficients

Examination of the inflection points for the first derivative models revealed the moisture model had high variation around 1400 nm indicating absorption by the first overtone of O–H stretch in water (Murray & Williams, 1987; Osborne, Fearn, & Hindle, 1993). The first derivative crude protein model had main bands around 1692 and 2070–2306 nm indicating involvement due to absorption by amide N–H groups in protein. Regression coefficients for the first derivative total carbohydrate model had the highest variation around 1702 and 2000–2300 nm indicating possible involvement of O–H and C–H groups from starch and cellulose (data not shown).

The second derivative total fat model (Fig. 3a) had variation due to absorption by C–H stretch at 1728 and 1762 nm and additional peaks at 1212 and 1390 nm. When the total fat model was calculated with the full 1100–2498 nm range, the regression coefficients had poor correlations above 1900 nm.

Table 2
Cross validation and validation statistics for NIR prediction of protein, moisture, total fat, total carbohydrate, and energy in homogenized meals

	Cross validation						Validation						
	Wavelength (nm)	n^a	NIR mean	NIR SD	SECV	R^2	n	NIR mean	NIR SD	SEP	r^2	Bias	RPD
Protein (%)	1100–2498	115	8.27	4.99	1.07	0.98	37	8.33	5.01	0.78	0.99	0.07	6.6
Moisture (%)	1100–2498	115	69.60	9.67	1.93	0.98	38	69.69	9.37	1.63	0.99	−0.13	6.1
Total fat (%)	1100–1898	113	4.19	3.16	0.67	0.98	38	4.39	2.95	0.75	0.97	0.05	4.3
Total carbohydrate (%)	1100–2498	114	16.16	6.98	1.80	0.97	37	16.55	7.15	2.00	0.96	−0.01	3.6
Energy (kcal/g)	1100–2498	115	1.381	0.511	0.103	0.98	38	1.390	0.527	0.081	0.99	−0.00	6.3

[a] Number of samples (n); standard deviation (SD); standard error of cross-validation (SECV); multiple coefficient of determination (R^2); standard error of performance (SEP); coefficient of determination (r^2); ratio of the standard deviation of the AOAC values to the SEP (RPD).

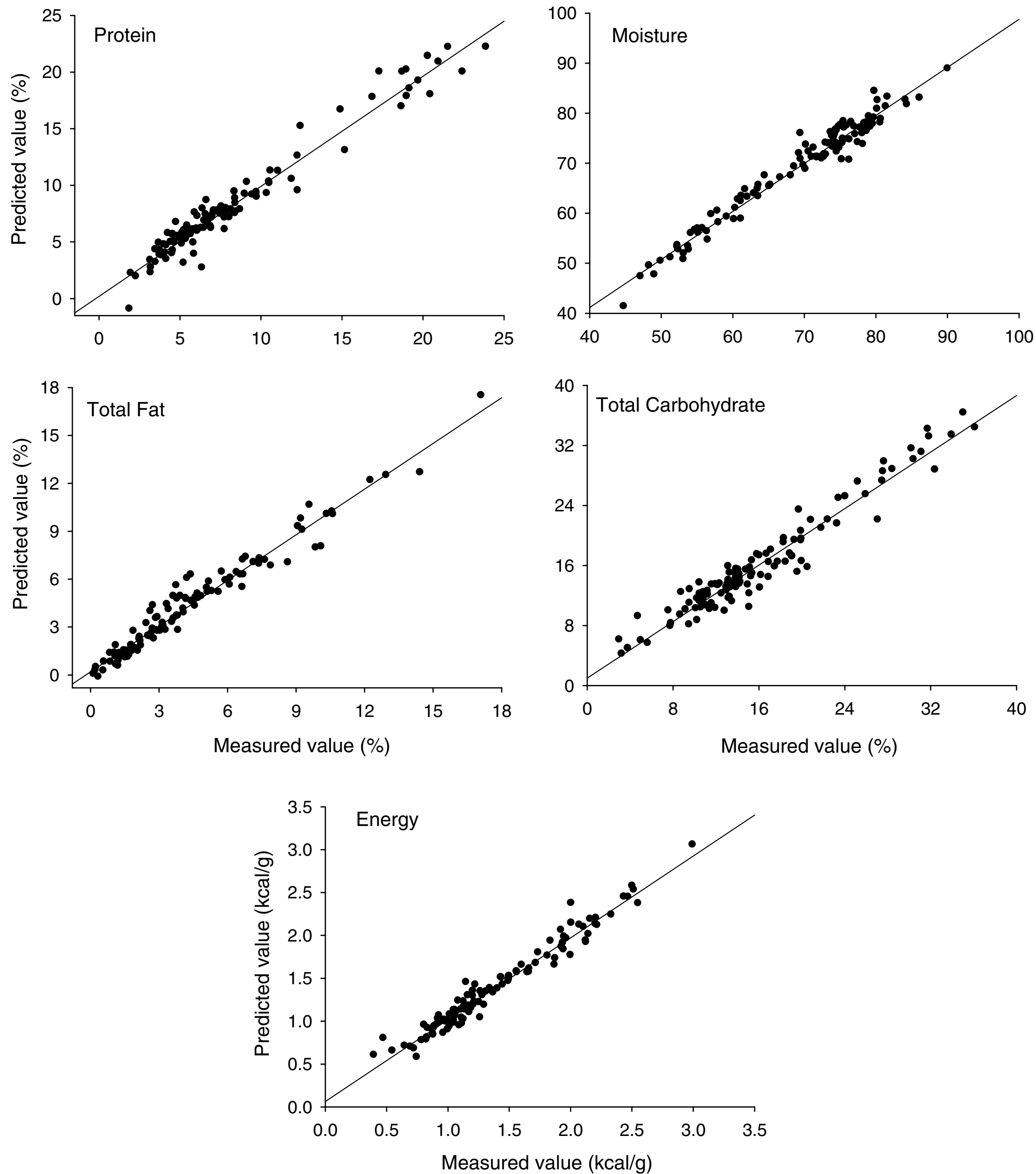

Fig. 1. Cross validation plots of reference method determined or calculated values versus NIR-predicted values for moisture, protein, total fat, total carbohydrate and energy in homogenized meals.

First and second derivative models for energy gave very similar performance. The second derivative regression coefficients (Fig. 3b) indicated that C–H groups in lipids at 1210, 1398, and 1726 nm were important in model development in addition to a band at 2282 nm indicating involvement of O–H and C–C stretch in starch (Murray & Williams, 1987; Osborne et al., 1993).

3.4. Performance of models when used to predict the subgroups of meals in the validation sample set

Based on RPD values, the components predicted with the highest accuracy were protein and moisture in the vegetable protein-based meals (10.1 and 9.9, respectively) followed by protein and moisture in the carbohydrate-based meals (5.7 and 8.3, respectively) (Table 3). Ratios of deviation to performance for the energy models were 13.4, 6.2 and 5.1 for animal protein-based, vegetable-based and carbohydrate-based meals, respectively.

4. Discussion

Conventional methods of food analysis are time consuming, especially when multiple components are analyzed. It was found that the major components and energy of homogenized meals could be determined by NIR reflec-

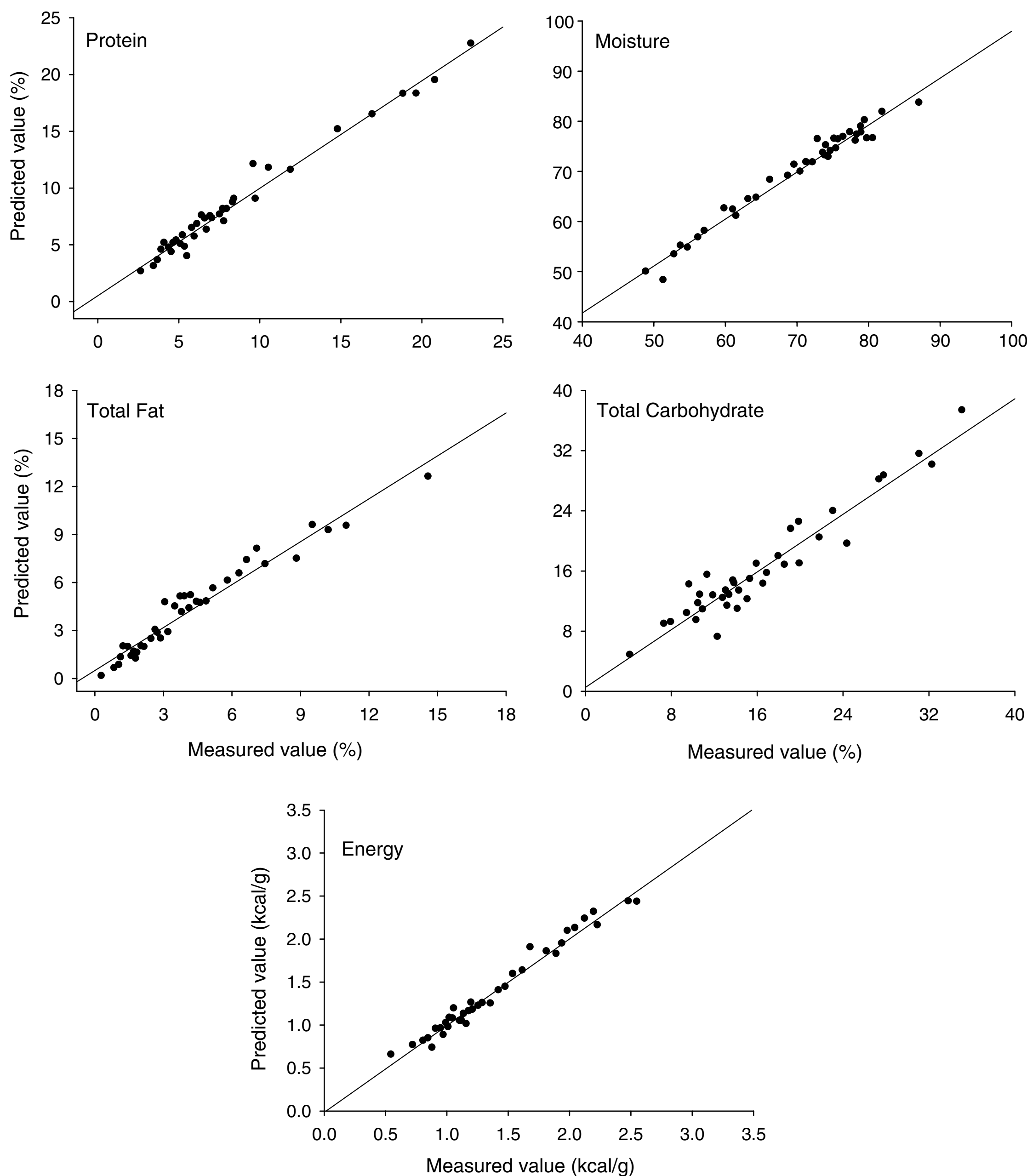

Fig. 2. Validation plots of reference method determined or calculated values versus NIR-predicted values for moisture, protein, total fat, total carbohydrate and energy in homogenized meals.

tance spectroscopy within the accuracy required for screening, quality control, or higher, depending on the parameter and the type of meal. The parameters can be determined concurrently by NIR spectroscopy in a wide variety of commercial packaged ready-to-eat meals after samples are homogenized only. This reduces analysis time substantially as conventional chemical analysis requires drying the samples after homogenization, thus, taking at least a day to perform. In contrast, the NIR method merely requires 10–15 min to load the homogenized meals into the sampling cells in triplicate and scan them.

The accuracy of NIR prediction of the components varied substantially based on the parameter and the type of meal. Over all, the prediction accuracy for validation samples was greatest for the crude protein model followed by moisture and energy, with accuracy estimated to be high enough for process control for protein and quality control for moisture and energy (Williams, 2001). Total fat and carbohydrate were predicted with the least accuracy over all but models were still sufficiently accurate for screening samples. When the samples were broken down into types of meals, substantial variation was observed in the prediction of protein and moisture. In contrast, energy was predicted well in all sub-groups and most accurately in the animal protein-based meals, possibly because of the higher fat content compared to the other sub-groups. From the

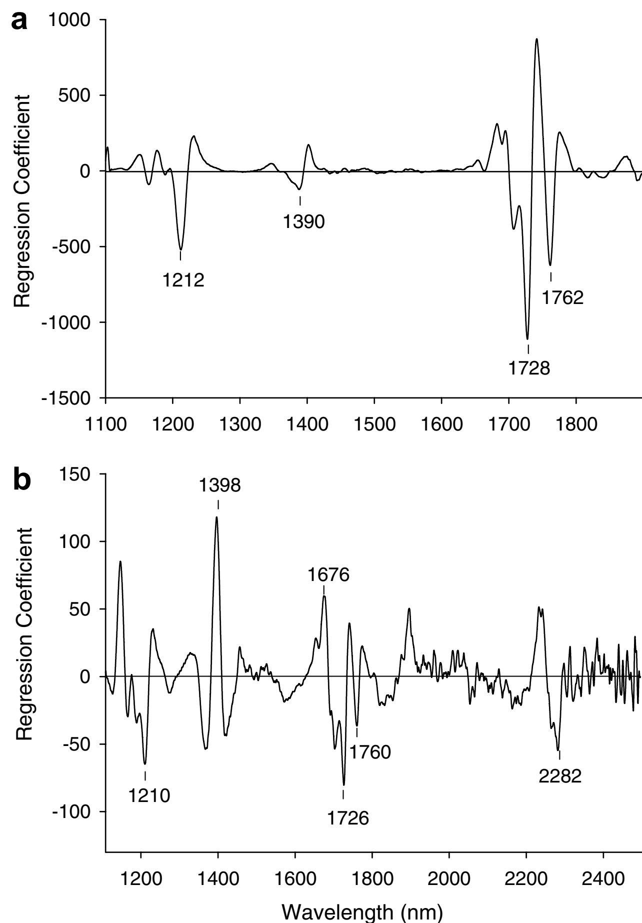

Fig. 3. Regression coefficients for the second derivative PLS models to predict total fat (a) and energy (b) in homogenized meals.

regression coefficients for the energy prediction model, fat appeared to be an important contributor to the energy model and has been shown to be a major factor in energy prediction models in other types of foods (Kays & Barton, 2002). In addition, Pearson correlation coefficients indicate that fat was the component most highly correlated with energy.

In a prior study from this laboratory (Kim et al., 2006), it was found that the prediction of total dietary fiber (TDF) in homogenized meals was limited partly because of the broad water peak in the 1850–2300 nm range and also because of the low range in dietary fiber content in the meals. Drying or drying and defatting of samples were required for adequate model performance in screening samples for TDF content. In contrast, in the current study it was shown that other major constituents and energy could be predicted adequately for screening without drying and defatting, suggesting that more information was available in the spectra for prediction of protein, total fat and carbohydrate and that important bands for these constituents occurred in areas of the spectra outside the broad water peak. In addition, in general, the ranges in quantities of the components in the current study were wider than those for total dietary fiber.

Further testing of the calibrations could include validation samples that are completely independent, i.e., tested at a different point in time than the calibration samples. Furthermore, larger numbers of samples in each sub-group would provide more conclusive information on the ability to predict parameters in specific types of meals.

Table 3
Statistics for NIR prediction of protein, moisture, total fat, total carbohydrate, and energy in the sub-groups of homogenized meals in the validation sample set

Components	Sub-group[a]	Validation				
		n[b]	Reference range	SEP	r^2	RPD
Protein (%)	Animal protein based	11	3.48–10.57	0.77	0.96	2.5
	Vegetable protein based	11	2.70–23.07	0.77	1.00	10.1
	Carbohydrate based	15	3.72–18.87	0.73	0.98	5.7
Moisture (%)	Animal protein based	16	59.92–87.14	1.95	0.97	3.6
	Vegetable protein based	10	53.81–79.00	1.00	1.00	9.9
	Carbohydrate based	12	48.95–75.81	1.19	0.99	8.3
Total fat (%)	Animal protein based	14	0.87–14.62	1.14	0.96	3.6
	Vegetable protein based	9	0.31–8.85	0.69	0.96	3.6
	Carbohydrate based	15	1.16–11.04	1.17	0.95	2.5
Total carbohydrate (%)	Animal protein based	12	4.20–32.34	1.63	0.98	4.6
	Vegetable protein based	13	8.00–31.15	1.98	0.96	3.7
	Carbohydrate based	12	9.72–35.15	2.34	0.95	3.2
Energy (kcal/g)	Animal protein based	8	0.727–2.484	0.045	1.00	13.4
	Vegetable protein based	12	0.550–2.232	0.073	0.99	6.2
	Carbohydrate based	18	0.884–2.555	0.100	0.99	5.1

[a] Animal protein based group (meat or fish based with vegetable); vegetable protein based group (bean based); carbohydrate based group (flour or rice based with meat or vegetable).

[b] Number of samples (n); standard error of performance (SEP); coefficient of determination (r^2); ratio of the standard deviation of the AOAC values to the SEP (RPD).

5. Conclusions

Major components and energy were predicted by NIR spectroscopy in validation samples of homogenized commercially available dinners within the accuracy required for screening samples and, in some cases, within the accuracy required for quality control or process control. These products with high moisture, a wide range in moisture and extremely diverse ingredients, can be screened simultaneously for constituents and energy in less than 15 min for each sample in contrast to the conventional methods of analysis which take at least 24 h.

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References

Adamopoulos, K. G., & Goula, A. M. (2004). Application of near-infrared reflectance spectroscopy in the determination of major components in taramosalata. *Journal of Food Engineering, 63*, 199–207.

Almendingen, K., Meltzer, H. M., Pedersen, J. I., Nilsen, B. N., & Ellekjaer, M. (2000). Near infrared spectroscopy – a potentially useful method for rapid determination of fat and protein content in homogenized diets. *European Journal of Clinical Nutrition, 54*, 20–23.

Association of Official Analytical Chemists (AOAC) (1990). Official Methods of Analysis, Method 945.14, Moisture in cereal adjuncts: air oven method (103–104 °C) (15th ed.). Arlington, VA.

Association of Official Analytical Chemists (AOAC) (1997). Official Methods of Analysis, Method 990.03, Protein (crude) in animal feeds: combustion method (16th ed.). Arlington, VA.

Association of Official Analytical Chemists (AOAC) (2000). Official Methods of Analysis, Method 923.03, Ash of flour: direct method (17th ed.). Arlington, VA.

American Society for Testing and Materials (ASTM) (1995). Standard practices for infrared multivariate, qualitative analysis. In *Annual book of ASTM standards* (p. 24). West Conshohocken, PA: ASTM.

Baker, D. (1985). The determination of fiber, starch, and total carbohydrate in snack foods by near-infrared reflectance spectroscopy. *Cereal Foods World, 30*, 389–392.

Ben-Gera, I., & Norris, K. H. (1968). Direct spectrophotometric determination of fat and moisture in meat products. *Journal of Food Science, 33*, 64–67.

Code of Federal Regulations (2005). FDA, HHS, 21, part 101.9.

Ferris, D. A., Flores, R. A., Shanklin, C. W., & Whitworth, M. K. (1995). Proximate analysis of food service wastes. *Applied Engineering in Agriculture, 11*, 567–572.

Giangiacomo, R., & Cattaneo, T. M. P. (2007). Applications to foodstuffs: Dairy products. In Y. Ozaki, W. F. McClure, & A. A. Christy (Eds.), *Near-infrared spectroscopy in food science and technology* (pp. 323–340). Hoboken, NJ: John Wiley & Sons, Inc.

Hruschka, W. R. (1987). Data analysis: Wavelength selection methods. In P. Williams & K. Norris (Eds.), *Near-infrared technology in the agricultural and food industries* (pp. 35–55). St. Paul, MN: American Association of Cereal Chemists.

Hymowitz, T., Dudley, J. W., Collins, F. I., & Brown, C. M. (1974). Estimations of protein and oil concentration in corn, soybean, and oat seed by near-infrared light reflectance. *Crop Science, 14*, 713–715.

Ito, H., Ippoushi, K., Azuma, K., & Higashio, H. (1999). Potential of near-infrared reflectance spectroscopy for estimating total dietary fiber content of dehydrated vegetables. *Acta Horticulturae, 483*, 265–274.

Kays, S. E., & Barton, F. E. II, (2002). Rapid prediction of gross energy and utilizable energy in cereal food products using near-infrared reflectance spectroscopy. *Journal of Agricultural and Food Chemistry, 50*, 1284–1289.

Kays, S. E., & Barton, F. E. II, (2006). Applications to foodstuffs: Cereal foods. In Y. Ozaki, W. F. McClure, & A. A. Christy (Eds.), *Near-infrared spectroscopy in food science and technology* (pp. 297–310). Hoboken, NJ: John Wiley & Sons, Inc.

Kim, Y., Singh, M., & Kays, S. E. (2006). Near-infrared spectroscopy for measurement of total dietary fiber in homogenized meals. *Journal of Agricultural and Food chemistry, 54*, 292–298.

Martens, H., Jensen, S. A., & Geladi, P. (1983). Multivariate linearity transformation for near-infrared reflectance spectrometry. In *Proceedings of the Nordic symposium on applied statistics* (pp. 205–234). Stavanger, Norway: Stokkand Forlag.

Martens, M., & Martens, H. (1986). Partial least squares regression. In J. R. Piggott (Ed.), *Statistical procedures in food research* (pp. 293–359). London, UK: Elsevier Applied Science.

Martens, H., & Naes, T. (1989). Assessment, validation and choice of calibration method. In *Multivariate calibration* (pp. 237–266). New York: John Wiley & Sons.

Merrill, A. L., & Watt, B. K. (1955). Energy value of foods-basis and derivation. In *U.S. Department of Agriculture Handbook No. 74* (pp. 1–5). Washington, DC: U.S. Department of Agriculture.

Murray, I., & Williams, P. C. (1987). Chemical principles of near-infrared technology. In P. Williams & K. Norris (Eds.), *Near-infrared technology in the agricultural and food industries* (pp. 17–34). St. Paul, MN: American Association of Cereal Chemists.

Norris, K. H. (1989). Definition of NIRS analysis. In G.C. Marten, J. S. Shenk, & F. E. Barton II. (Eds.), *Near infrared reflectance spectroscopy (NIRS): Analysis of forage quality, USDA-ARS Agriculture Handbook no. 643* (p. 6). Washington, DC: USDA.

Osborne, B. G., Fearn, T., & Hindle, P. H. (1993). *Practical NIR spectroscopy with applications in food and beverage analysis* (2nd ed.). Harlow, UK: Longman Scientific and Technical.

Savitzky, A., & Golay, M. J. E. (1964). Smoothing and differentiation of data by simplified least squares procedures. *Analytical Chemistry, 36*, 1627–1639.

Uddin, M., Okazaki, E., Fukushima, H., Turza, S., Yumiko, Y., & Fukuda, Y. (2006). Non destructive determination of water and protein in surimi by near-infrared spectroscopy. *Food Chemistry, 96*, 491–495.

White, M., & Rouvinen-Watt, K. (2004). Near-infrared evaluation of wet mink diets. *Animal Feed Science and Technology, 111*, 239–246.

Williams, P. C. (2001). Implementation of near-infrared technology. In P. Williams & K. Norris (Eds.), *Near-infrared technology in the agricultural and food industries* (2nd ed., pp. 145–169). St. Paul, MN: American Association of Cereal Chemists.

Williams, P. C., Cordeiro, H. M., & Harnden, M. F. T. (1991). Analysis of oat bran products by near-infrared reflectance spectroscopy. *Cereal Foods World, 36*, 571–574.