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Energy evaluation of extruded compound foods for dogs by near-infrared spectroscopy

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Summary

Near-infrared reflectance spectroscopy (NIRS) was used to predict the chemical composition, apparent digestibility and digestible nutrients and energy content of commercial extruded compound foods for dogs. Fifty-six foods of known chemical composition and *in vivo* apparent digestibility were analysed overall and 51 foods were used to predict gross energy digestibility and digestible energy content. Modified partial least square calibration models were developed for organic matter (OM), crude protein (CP), ether extract (EE), crude fibre (CF), nitrogen free extracts (NFE) and gross energy (GE) content, the apparent digestibility (OMD, CPD, EED, NFED and GED) and the digestible nutrient and energy content (DOM, DCP, DEE, DNFE and DE) of foods. The calibration equations obtained were evaluated by the standard error and the determination coefficient of cross-validation. The cross-validation coefficients of determination (R_{cv}^2) were 0.61, 0.99, 0.91, 0.96, 0.94 and 0.92 for OM, CP, EE, CF, NFE and GE, the corresponding standard error of cross-validation (SECV) being 5.80, 3.51, 13.35, 3.64 and 16.95 g/kg dry matter (DM) and 0.29 MJ/kg DM respectively. The prediction of apparent digestibility was slightly less accurate, but NIRS prediction of digestible nutrient (g/kg DM) and DE (MJ/kg DM) gave satisfactory results, with high R_{cv}^2 (0.93, 0.97, 0.93, 0.83 and 0.93 for DOM, DCP, DEE, DNFE and DE respectively) and relatively low SECV (11.55, 6.85, 12.14 and 22.98 g/kg DM and 0.47 MJ/kg DM). It is concluded that the precision of NIRS in predicting the energy value of compound extruded foods for dogs is similar or better than by proximate analysis, as well as being faster and more accurate.

Introduction

The experimental determination of the digestible or metabolizable energy content of dog foods is expensive and time-consuming. The NRC (1985) proposes the estimation of the metabolizable energy content of dog foods from their proximate analysis using the Atwater approach, assuming

constant apparent digestibility coefficients from each analytical fraction.

Another more recent approach is to estimate the digestible energy content as the product of the gross energy content by the digestibility coefficient estimated from crude fibre or other indigestible carbohydrate fractions of foods (Kienzle et al., 1998; Castrillo et al., 2001). Near-infrared reflectance

spectroscopy (NIRS) has become one of the most powerful tools for rapid and non-destructible estimation of chemical composition and nutritive evaluation of feeds and mixed diets (Givens and Deaville, 1999) for ruminants (De Boever *et al.*, 1995; Aufrère *et al.*, 1996; De Boever *et al.*, 2003), pigs and poultry (Aufrère *et al.*, 1996; Van Barneveld *et al.*, 1999; Garnsworthy *et al.*, 2000) as well as rabbits (Xiccato *et al.*, 1999, 2003). The limitation of this approach is the difficulty to obtain enough samples with *in vivo* data for a robust calibration and we are not aware of any studies that have used NIRS to predict the energy value of compound feed for dogs.

The aim of this work was to evaluate the potential of NIRS to predict the chemical composition, apparent digestibility and content of digestible nutrients and energy of commercial extruded compound foods for dogs.

Materials and methods

Fifty-six commercial extruded compound foods of known chemical composition and *in vivo* apparent digestibility were used, except for the prediction of the gross energy digestibility and the digestible energy content, in which case only 51 foods were used. Air-dried samples milled through a 1-mm sieve were scanned twice in reflectance mode by a monochromator Foss NIRSystems 6500 (NIRSystems; Silver Spring, MD, USA) (every 2 nm from 1100 to 2500 nm). Spectra and reference data were recorded using the Infra-soft International (ISI) software NIRS2 v2.4 (NIRSystems). The standard normal variate and de-trend scatter correction procedure (Barnes *et al.*, 1989) was applied to the raw $\log 1/R$ data. Then the spectra were transformed using a mathematical first-order derivation. Modified partial least square calibration models were developed for organic matter (OM), crude protein (CP), ether extract (EE), crude fibre (CF), nitrogen free extracts (NFE) and gross energy (GE) content, apparent digestibility coefficients (OMD, CPD, EED, NFED and GED), and digestible nutrient and energy content (DOM, DCP, DEE, DNFE and DE) of foods. The statistics used to develop and evaluate the equation included the standard error of calibration and standard error of cross-validation (SECV), the coefficient of determination for the calibration (R_c^2) and for the cross-validation (R_{cv}^2) and the RER and RPD values. The RER value is defined as the ratio of the range in the reference data for the samples to the SECV. The RPD value is defined as the ratio of the standard deviation of the samples to the SECV (Williams and Sobering, 1996).

The apparent digestibility of nutrients and energy was determined using six adult (2–6 years old) female Beagles of 12–15 kg live weight. Each trial consisted of a 10-day adaptation period followed by 7 days of total faeces collection. Dogs were kept in indoor-outdoor floor pens during the adaptation period and in metabolic crates (1.1 × 0.8 m² internal surface) during the collection period. Food was given at a fixed level of intake (350 g/day) which represented 1.2–1.8 times the energy requirements for maintenance (NRC, 1985). Food refusals and faeces were collected daily and dried at 105 °C for 24 and 48 h respectively. The ash, CP, EE (after acid hydrolysis) and CF (using the Fibertec system, Tecator 1020; Foss Tecator AB, Höganä, Sweden) of foods and faeces were analysed according to the AOAC (1990) methods and gross energy in an adiabatic calorimetric bomb (IKA.C-4000; Janke-Kunkel, Staufen, Germany).

Results and discussion

The chemical composition and gross energy content, apparent digestibility coefficients and digestible nutrients and energy content of compound foods are listed in Table 1.

The statistics of NIRS calibration and cross-validation for the studied parameters are given in Table 2.

Table 1 Chemical composition and gross energy content, apparent digestibility and digestible nutrients and energy content of compound foods

	<i>n</i>	Minimum	Maximum	Mean	SD
Chemical composition (g/kg DM) and gross energy content (MJ/kg DM)					
OM	56	902.2	949.3	929.2	9.26
CP	50	158.0	340.2	273.7	35.40
CF	55	6.1	94.0	22.6	17.63
EE	52	77.7	237.0	155.7	44.01
NFE	51	363.0	645.0	473.0	64.15
GE	55	19.7	24.0	21.7	1.03
Apparent digestibility (%)					
DMD	53	63.75	88.11	81.07	4.951
OMD	53	74.68	90.80	85.24	4.188
CPD	53	73.05	89.02	82.01	4.355
EED	56	79.98	96.06	89.87	4.180
NFED	51	80.90	93.68	89.37	3.214
GED	49	68.76	91.05	84.85	4.596
Digestible nutrients (g/kg DM) and energy content (MJ/kg DM)					
DOM	52	690.0	853.0	792.5	42.79
DCP	55	122.0	289.0	223.4	38.09
DEE	52	63.0	227.0	141.6	44.76
DNFE	54	323.0	580.0	418.1	52.17
DE	49	13.6	21.8	18.4	1.75

Table 2 Statistics of NIRS calibration and cross-validations to predict the chemical composition, apparent digestibility and digestible nutrients and energy content of dog extruded foods

	SEC	R_c^2	SECV	R_{cv}^2	RPD	RER
Chemical composition (g/kg DM) and gross energy content (MJ/kg DM)						
OM	4.35	0.779	5.80	0.613	1.6	8.1
CP	2.58	0.995	3.51	0.991	10.1	51.9
CF	2.70	0.977	3.64	0.958	4.8	24.1
EE	11.85	0.927	13.35	0.912	3.3	11.9
NFE	15.77	0.940	16.95	0.936	3.8	16.6
GE	0.23	0.952	0.29	0.923	3.6	14.6
Apparent digestibility (%)						
DMD	0.895	0.967	1.308	0.931	3.8	18.620
OMD	0.983	0.945	1.236	0.914	3.4	13.040
CPD	1.355	0.903	1.951	0.801	2.2	8.185
EED	1.633	0.847	1.780	0.824	2.348	9.030
NFED	0.751	0.945	1.046	0.895	3.073	12.218
GED	1.107	0.942	1.642	0.871	2.799	13.570
Digestible nutrients (g/kg DM) and energy content (MJ/kg DM)						
DOM	8.57	0.960	11.55	0.928	3.7	14.1
DCP	5.44	0.980	6.85	0.969	5.6	24.4
DEE	10.81	0.942	12.14	0.929	3.7	13.5
DNFE	14.54	0.922	22.98	0.823	2.3	11.2
DE	0.34	0.961	0.47	0.928	3.7	17.5

SEC, standard error of calibration; SECV, standard error of cross-validation; R_c^2 , coefficient of determination of calibration; R_{cv}^2 , coefficient of determination of cross-validation; RPD, ratio of the mean standard deviation to the SEC; RER, ratio of the range in the reference data to the SEC.

The number of samples used in the calibration equations may be less than the total number of samples available, because outliers, identified by the two-tailed Student's *t*-test, were not used to develop the calibration equations.

The chemical composition of foods extended over a broad range that covered most commercial compound extruded foods usually fed to healthy dogs. The coefficients of determination were above 0.9 for all chemical constituents and gross energy in both calibration and cross-validation, except for OM content (Table 2), because of the small variation of this trait (CV = 1.0%) and because minerals do not absorb near infrared wavelengths (Shenk and West-erhaus, 1994). The standard errors associated with the cross-validation equations for CP, CF, EE, NFE and GE were also relatively low, especially for CP and GE (the coefficients of variation were 1.3%, 16.1%, 8.6%, 3.6% and 1.3% respectively). The RER and RPD ratios found in cross-validation equations for the chemical constituents and gross energy content were larger than the recommended minimum values (3 and 10 respectively; Williams and Sobering, 1996).

Variation in GE in compound foods is mainly determined by fat and protein content, which were well predicted by NIRS as found in rabbits (Xiccato *et al.*, 2003) or swine (Aufrère *et al.*, 1996). The SECV for GE [0.29 MJ/kg dry matter (DM)] was in the range of those found by Xiccato *et al.* (1999, 2003) in rabbits (0.37 and 0.25 MJ/kg DM respectively) or Aufrère *et al.* (1996) for swine (0.25 MJ/kg DM), even if the mean GE content of compound feeds used by these authors (18.3 and 18.4 MJ/kg DM respectively) was lower than in the present work (21.7 MJ/kg DM). Figures 1 and 2 show that

Fig. 1 Relationship between NIRS-predicted and measured gross energy content of compound dog foods (MJ/kg DM). **Fig. 2** Relationship between NRC-predicted and measured gross energy content of compound dog foods (MJ/kg DM).

GE prediction by NIRS ($R^2 = 0.92$, $SE = 0.29$ MJ/kg DM) was better than that obtained when GE was calculated from the CP, EE and carbohydrate (NFE + CF) contents of foods multiplied by its assumed gross energy content (23.6, 39.3 and 17.4 MJ/kg respectively) as proposed by the NRC (1985), ($R^2 = 0.853$, $SE = 0.40$ MJ/kg DM).

The calibration and cross-validation determination coefficients for the apparent digestibility were slightly lower than for the chemical constituents, although always above 0.80 (Table 2). The SECV associated with the GED prediction of studied compound foods (1.64%) was similar to that found by Xiccato et al. (1999, 2003) for rabbit compound feeds (2.0% and 1.9%) and Aufrère et al. (1996) for swine (1.7%) and cattle (2.1%) compound foods. The lower precision of digestibility estimations compared with chemical constituents and food gross energy is expected because digestibility depends on feed characteristics as well as animal response to feeding.

In any case, NIRS prediction of digestible nutrients and DE content of dog foods gave satisfactory results, with high R^2 and relatively low standard errors (Table 2). As with chemical composition, the RER and RPD ratios found in cross-validation equations for digestible nutrients and energy contents were higher than the minimum values recommended by Williams and Sobering (1996), except for the RPD of DNFE.

Predicted NIRS-DE fitted better with *in vivo* measured values ($R^2 = 0.928$, $SE = 0.47$ MJ/kg DM; Fig. 3) than DE estimated from chemical constituents, assuming a DE content of 18.9, 35.4 and

Fig. 3 Relationship between NIRS-predicted and *in vivo* values for digestible gross energy content of compound dog foods (MJ/kg DM).

Fig. 4 Relationship between NRC-predicted and *in vivo* values for digestible gross energy content of compound dog foods (MJ/kg DM).

Fig. 5 Relationship between fact-predicted [DE estimated as the product of GE content by the GED estimated from the equation: $GED (\%) = 94.00 - 4.04 \times CF (\% \text{ DM})$ from Castrillo et al. (2001)] and *in vivo* values for digestible gross energy content of compound dog foods (MJ/kg DM).

14.7 MJ/kg for CP, EE and NFE, respectively (NRC, 1985) ($R^2 = 0.859$, $SE = 0.67$ MJ/kg DM, Fig. 4), or calculated by multiplying the measured gross energy of foods by their digestibility estimated from CF content using the equation $DE (\%) = 94.0 - 4.04 \times CF (\%)$ proposed by Castrillo et al. (2001) [$R^2 = 0.886$, $SE = 0.60$ MJ/kg DM, Fig. 5].

Conclusions

The precision of using NIRS to predict the energy value of compound extruded foods for dogs is similar

or better than by proximate chemical analysis. The analysis is rapid and accurate, although the accuracy of the prediction could be improved by including more data covering the complete range of commercial foods.

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References

- AOAC, 1990: *Official Methods of Analysis*, 15th edn. Association of Official Analytical Chemists, Arlington, VA.
- Aufrère, J.; Graviou, D.; Demarquilly, C.; Perez, J., 1996: Near infrared reflectance spectroscopy to predict energy value of compound feeds for swine and ruminants. *Animal Feed Science and Technology* **62**, 77–90.
- Barnes, R.J.; Dhanoa, M.S.; Lister, S.J., 1989: Standard normal variate transformation and de-trending of near-infrared diffuse reflectance spectra. *Applied Spectroscopy* **43**, 772–777.
- Castrillo, C.; Vicente, F.; Guada, J.A., 2001: The effect of crude fibre on apparent digestibility and digestible energy content of extruded dog foods. *Journal of Animal Physiology and Animal Nutrition* **85**, 231–236.
- De Boever, J.L.; Cottyn, B.G.; Vanacker, J.M.; Boucqué, Ch., 1995: The use of NIRS to predict the chemical composition and the energy value of compound feeds for cattle. *Animal Feed Science and Technology* **51**, 243–253.
- De Boever, J.L.; Vanacker, J.M.; De Brabander, D.L., 2003: Rumen degradation characteristics of nutrients in compound feeds and the evaluation of tables, laboratory methods and NIRS as predictors. *Animal Feed Science and Technology* **107**, 29–43.
- Garnsworthy, P.C.; Wiseman, J.; Feregos, K., 2000: Prediction of chemical, nutritive and agronomic characteristics of wheat by near infrared spectroscopy. *Journal of Agricultural Science* **135**, 409–417.
- Givens, D.I.; Deaville, E.R., 1999: The current and future role of near infrared reflectance spectroscopy in animal nutrition: a review. *Australian Journal of Agricultural Research* **50**, 1131–1145.
- Kienzle, E.; Opitz, B.; Earle, K.E.; Smith, P.M.; Maskell, I.E., 1998: The influence of dietary fibre components on the apparent digestibility of organic matter and energy in prepared dog and cat foods. *Journal of Animal Physiology and Animal Nutrition* **79**, 46–56.
- NRC, 1985: *Nutrient Requirements for Dogs*. National Research Council of the USA. National Academy Press, Washington, DC.
- Shenk, J.S.; Westerhaus, M.O., 1994: The application of near reflectance spectroscopy (NIRS) to forage analysis. In: G.C. Fahey, M. Collins, D.R. Mertens, L.E. Moser (eds), *Forage Quality, Evaluation and Utilization*, Crop Science Society of America, Lincoln, NE, pp. 406–449.
- Van Barneveld, R.J.; Nuttal, J.D.; Flinn, P.C.; Osborne, B.G., 1999: Near infrared reflectance measurements of the digestible energy content of cereals for growing pigs. *Journal of Near Infrared Spectroscopy* **7**, 1–7.
- Williams, Ph.; Sobering, D.C., 1996: How do we do it: a brief summary of the methods we use in developing near infrared calibrations. In: A.M.C. Davies, P. Williams (eds), *Near Infrared Spectroscopy: The Future Waves*. NIR Publications, Chichester, UK, pp. 185–188.
- Xiccato, G.; Trocino, A.; Carazzolo, A.; Meurens, M.; Maertens, L.; Carabaño, R., 1999: Nutritive and ingredient prediction of compound feeds for rabbits by near-infrared reflectance spectroscopy (NIRS). *Animal Feed Science and Technology* **77**, 201–212.
- Xiccato, G.; Trocino, A.; De Boever, J.L.; Maertens, L.; Carabaño, R.; Pascual, J.J.; Perez, J.M.; Gidenne, T.; Falcao, L.; Cunha, E., 2003: Prediction of chemical composition, nutritive value and ingredient composition of European compound feeds for rabbits by near infrared reflectance spectroscopy (NIRS). *Animal Feed Science and Technology* **104**, 153–168.

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