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## SHORT COMMUNICATION

## Prediction of chemical composition and peroxide value in unground pet foods by near-infrared spectroscopy

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### Summary

The massive development of the pet food industry in recent years has led to the formulation of hundreds of canine and feline complete extruded foods with the objective of meeting both the needs of the animals and numerous demands from pet owners. In the meantime, highly variable raw material compositions and the industry's new production techniques oblige manufacturers to monitor all phases of the extrusion process closely in order to ensure the targeted composition and quality of the products. This study aimed at evaluating the potential of infrared technology (visible and near-infrared spectrophotometer; 570–1842 nm) in predicting the chemical composition and peroxide value (PV) of unground commercial extruded dog foods. Six hundred and forty-nine commercial extruded dog foods were collected. For each product, an unground aliquot was analysed by infrared instrument while a second aliquot was sent to a laboratory for proximate analysis and PV quantification. The wide range of extruded dog food typologies included in the study was responsible for the wide variability observed within each nutritional trait, especially crude fibre and ash. The mean value of the 208 pet foods sampled for PV quantification was 17.49 mEq O<sub>2</sub>/kg fat (min 2.2 and max 94.10 mEq O<sub>2</sub>/kg fat). The coefficients of determination in cross-validation of NIRS prediction models were 0.77, 0.97, 0.83, 0.86, 0.78 and 0.94 for moisture, crude protein, crude fat, crude fibre, ash and nitrogen-free extract (NFE) respectively. PV prediction was less precise, as demonstrated by the coefficient of determination in cross-validation (0.66). The results demonstrated the potential of NIRS in predicting chemical composition in unground samples, with lower accuracy for moisture and ash, while PV prediction models suggest use for screening purposes only.

**Keywords** dog food, chemical composition, near-infrared reflectance spectroscopy, peroxide value

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### Introduction

The growing interest of people in the welfare of their pets has generated a dramatic development of the pet food industry, which now offers the market hundreds of canine and feline foods that meet the different needs of animals and the numerous demands of pet owners. Highly variable raw material compositions and the industry's new production techniques oblige manufacturers to monitor every phase of the pet food extrusion process closely in order to ensure the targeted composition and quality of the products. In this respect, NIRS is a potentially optimal control tool whose validity in estimating chemical composition and energy value of extruded pet foods has been

demonstrated (Castrillo et al., 2005; Hervera et al., 2008, 2012). The use of NIRS would therefore facilitate real-time monitoring of pet food composition and quality at all stages of production, but whether its application is also accurate in unground samples is not yet known.

The peroxide value (PV) quantify the amount of hydroperoxides in fats and oils, providing a measure of their oxidation level that negatively impact taste, flavour, odour, colour, texture and appearance (Frankel, 1988) and can be a valuable alternative to the quantification of antioxidant level of feed (Renzi et al., 2005). Therefore, the use of NIRS in predicting PV in dog food can be considered an important quality control analysis to be conducted by pet food manufacturers.

The aim of this work was to evaluate the potential of applying NIRS technology in predicting the chemical composition and PV of unground commercial extruded dog foods. The choice of using unground samples has the further advantage of reducing sample preparation time, an important aspect for pet food manufacturers needing a quick and reliable product quality control.

## Materials and Methods

Six hundred and forty-nine commercial extruded dog foods were collected from an Italian pet food company from August 2007 to August 2013. Dry dog food samples were randomly selected from all types of manufactured dog foods after extrusion and drying. Samples were collected following the internal manufacturer self-inspection procedures that were performed 2 days per week. A 100-g sample of kibbles of each food was collected and divided into two aliquots. The first aliquot was kept unground and immediately analysed in the industrial laboratory by infrared instrument, while the second was vacuum-packed in a black polyethylene multilayer bag, stored at 5 °C, and sent to a private laboratory for reference analyses within 48 h.

Chemical analyses were performed as reference analyses and included proximate analysis – moisture, crude protein (CP); crude fat; crude fibre (CF); ash – and PV quantification. Each sample has been milled to pass a 1-mm screen prior to chemical analyses. Moisture content was determined by oven-drying at 105 °C for 24 h. CP content was determined using a Kjeldahl-Foss autoanalyser (Model 16210; Foss, Padova, Italy) (EC/152/2009 – Annex III Method C) and applying a nitrogen conversion factor of 6.25. Crude fat analysis was performed according to the Method H, procedure B reported in the Annex III of the EC/152/2009, by Soxhlet extraction using petroleum ether (Soxhlet, Foss Tecator, Hillerød, Denmark) after hydrolysis with hydrochloric acid. CF was obtained according to the same Regulation (EC/152/2009 – Annex III Method I) using a Fibertec 2010 analyser (FOSS). Weight loss after combustion was expressed as crude fibre. Ash content was measured gravimetrically after combustion in a muffle at 550 °C until white, light grey or reddish ash was obtained, and subsequent cooling to environmental temperature (EC/152/2009 – Annex III Method M). Nitrogen-free extract (NFE) was calculated by difference to 100 as follows:  $NFE = 100 - (\% CP + \% Crude Fat + \% CF + \% Moisture + \% Ash)$ . PV, expressed as mEq of O<sub>2</sub>/kg of fat, was determined according to the official

methods, described in the Commission Regulation (EEC/2568/91, Annex III), by dissolution in acetic acid/chloroform (3:2 v/v) with aqueous potassium iodide solution and then titration with sodium thio-sulfate using starch indicator according to the method MP-2097 rev 0 2013 (UNI EN 1528-2:1997, Food-stuffs). All chemical analyses were performed by duplicate.

Spectral data were collected on unground samples using a Vis-NIRS spectrophotometer (InfraXact; Foss Electric, Padova, Italy) equipped with a reflectance post-dispersive optical configuration and silicon detectors (Si 570–1100 nm, InGaAs: 1100–1850 nm). The spectrum was recorded over a wavelength range of 570–1842 nm at intervals of 2 nm with 32 scans obtained and averaged to provide the final spectrum.

Prediction models were calculated using WinISI II software (Infrasoft International, Port Matilda, PA, USA) and modified partial least squares (MPLS) as chemometric algorithm that differs from standard PLS regression as it involves the standardization of the residues after each iteration (Osborne *et al.*, 1993; Riovanto *et al.*, 2012). Absorbance data were stored as log (1/R). The visible region of the spectrum (from 570 to 1108 nm) was deleted prior to the analysis because the inclusion of this region did not improve the accuracy of the prediction models, according to De Marchi *et al.* (2012) that used vis-NIRS for the prediction of fatty acids in chicken breast. Before MPLS analysis, data were submitted to the detection of anomalous spectra using the Mahalanobis distance and to the identification of samples outliers using t-statistics, according to De Marchi *et al.* (2012). To improve MPLS accuracies, different combination of spectra pre-treatments and mathematical treatments were carried out; finally, the best combination used for all prediction models was the standard normal variance (SNV) and detrend (DT) for scatter correction of spectra followed by the 1,4,4,1 derivative, where the first digit refers to the number of the derivative, the second is the gap over which the derivative is calculated, the third is the first smoothing segment and the fourth is the second smoothing segment (Shenk *et al.*, 1989; De Marchi *et al.*, 2014).

Cross-validation was then performed by dividing the calibration dataset in four groups, using one to check the results (prediction) and the remaining three to construct the calibration model. The model was repeated as many times as there were groups available in such a way that all passed through the calibration and the prediction set. To evaluate the accuracy of prediction models, standard errors of calibration (SEC) and cross-validation (SECV), and the coefficients of

determination of calibration ( $R^2$ ) and cross-validation (1-VR = coefficient of determination of cross-validation), were calculated. Finally, the ratio to performance deviation (RPD that equals to  $SD/SECV$ ) was calculated to evaluate the practical utility of the calibration models; RPD values greater than 10 were considered equivalent to the reference method, whereas values above 2.5 were considered adequate for analytical purposes (Williams and Sobering, 1993; Sinnaeve et al., 1994).

## Results

The chemical composition (on as fed basis), the PV quantification (expressed as mEq/kg fat) of the dog foods analysed and the number of samples included for each chemical trait are shown in Table 1. The foods tested showed a variable moisture level concurring to the variability of the other parameters tested. The lowest variability was observed for NFE while the highest variability was found with regard to PV.

Crude fat and ash showed almost identical variability, and a wide range of variation was observed for CF.

The average raw absorbance ( $\log(1/R)$ ) and first-derivative mathematical pre-treatment spectra are depicted in Figure 1. Both the spectra plotted are composed only by near-infrared part because the visible one was deleted prior to the statistical analysis. On the raw spectrum, it is possible to observe peaks at various wavelengths: a first and defined peak was observed at 1210 nm, while a wider peak included wavelengths from 1400 to about 1500 nm; a further peak was found close to 1730 nm. The first-order derivate transformed the spectral shape and highlighted much more signals (1152, 1176, 1218, 1350, 1410, 1566, 1686, 1752 and 1788 nm).

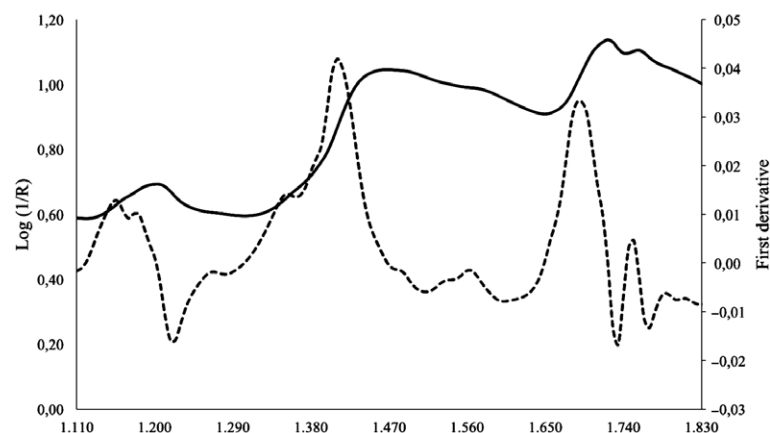
The statistics of NIRS prediction models in calibration and cross-validation for the traits studied are reported in Table 1. The prediction models can be evaluated based on the number of loading factor retained for each model; in this study, they ranged from 12 to 14. On average, the accuracies achieved in

**Table 1** Descriptive statistics and fitting statistics of prediction models for chemical composition (g/100 g as fed) and peroxide value (mEq O<sub>2</sub>/kg fat) in dry dog foods by NIR spectroscopy (1108–1842; 4 nm)

Trait	Mean (SD)	MIN	MAX	N	Terms	SEC	R	SECV	1-VR	RPD
Moisture	7.44 (1.49)	2.47	12.31	629	13	0.67	0.78	0.69	0.77	2.07
Crude protein	25.53 (4.09)	11.41	38.90	605	14	0.66	0.97	0.67	0.97	6.06
Crude fat	12.43 (2.56)	5.77	20.56	599	13	1.01	0.84	1.03	0.83	2.45
Crude fibre	2.39 (0.85)	0.85	8.55	595	14	0.29	0.87	0.30	0.86	2.70
Ash	6.94 (1.46)	1.48	13.87	598	14	0.66	0.80	0.69	0.78	2.13
NFE	45.27 (5.42)	39.54	61.42	580	14	1.23	0.95	1.26	0.94	4.22
Peroxide value	17.49 (14.41)	2.20	94.10	202	12	5.61	0.70	5.96	0.66	1.72

SD, standard deviation; NFE, nitrogen-free extract; N, number of samples considered to build the equations. Terms: number of modified PLS factors used in calibration. SEC, standard error of calibration;  $R^2$ , coefficient of determination of calibration; SECV, standard error of cross-validation; 1-VR, coefficient of determination of cross-validation; RPD, ratio performance deviation.

Nitrogen-free extract = 100 – (moisture + crude protein + crude fat + crude fibre + ash).



**Fig. 1** Average Vis–NIR absorbance spectrum (1100–1850 nm) of dry dog foods (continuous line) and average NIR absorbance spectrum (1100–1850 nm) of dry dog foods subjected to a first derivative as mathematical treatment (dotted line).

calibration were better than cross-validation, and the 1-VR values were greater than 0.90 for CP and NFE, while they ranged from 0.77 to 0.86 for the other traits, with the exception of PV, which showed a 1-VR of 0.66.

## Discussion

The present work was aimed to evaluate the potential of applying NIRS technology in predicting the chemical composition and PV of commercial extruded dog foods in unground form. The chemical analysis conducted on the samples showed a wide variability within each nutritional trait, especially crude fibre and ash. This wide variation can be explained by the fact that the samples included in the study represented the broad range of extruded dog food formulations available in the market. Dog foods vary widely in composition because they are formulated to individuals at different stages of life with different nutritional and energy requirements. Meat and bone meal, which are common components of canine and feline foods, make particularly high contributions to this variation due to their own wide range in crude protein, fat and ash contents (Parsons *et al.*, 1997; Shirley and Parsons, 2001). The level of CF in the diet can be raised by increasing the amount of specific ingredients in the formula, such as beet pulp, cellulose, bran and hulls, and this leads to a reduction in the diet's energy value. As previously reported by Alomar *et al.* (2006) in a study aimed at establishing reliable NIRS calibration models, a wide variability in the chemical composition of the samples included is highly recommended.

The oxidation of fats and oils is an important deteriorative reaction with significant commercial implications in terms of product value. The initial oxidation products that accumulate in triacylglycerol are hydroperoxides, which may subsequently break down to form compounds of lower molecular weight, such as alcohols, aldehydes, free fatty acids and ketones, and ultimately lead to a rancid product (Li *et al.*, 2000). It is well known that lipid oxidation causes a decrease in the nutritional value of the foods and can induce food poisoning (Gotoh and Wada, 2006). Despite this knowledge, the literature available on PV quantification in pet food is very limited, and no reference values on the maximum PV allowed in canine or feline foods are given by the National Research Council (NRC), the Association of American Feed Control Officials (AAFCO) or the European Pet Food Industry Federation (FEDIAF). Neither is any information available on the level of PV above which a commercial pet food is considered rancid or non-edible for a dog

or cat. In a study by Osawa *et al.* (2008), the PV of 18 expired pet foods was measured using both a modified official AOCS method and a commercially available kit (Saf Test PeroxiSafe MSA kit; MP Biomedicals, Santa Ana, CA), and the maximum quantified level was 6.8 and 2.9 mEq O<sub>2</sub>/kg of fat, respectively, which are much lower than the mean value (17.49 mEq O<sub>2</sub>/kg of fat) recorded in the samples collected in our study. It is well known that extrusion processing and storage are the main conditions that cause fat and oil oxidation in pet foods (Lin *et al.*, 1998). As reviewed by Singh *et al.* (2007), no oxidation occurs during extrusion due to the very short treatment time, but rancidity is a problem for stored extruded products because traces of metal left by the wear of the screw can act as pro-oxidants, and the larger surface area created by the air cells in highly expanded extrudates can favour oxidation. Moreover, extrusion has been shown to exert a direct detrimental impact on antioxidant vitamins, such as vitamin C (Andersson *et al.*, 1981; Riaz *et al.*, 2009), and on vitamin A and E levels both during treatment (Riaz *et al.*, 2009) and subsequent preservation (Mooney, 2014), making the latter absolutely critical. For this reason, extruded dog and cat diets are usually enriched with antioxidants that help in preventing rancidity and maintain the nutritive value of the food over the 12- to 18-month shelf-life period. In our study, storage was not likely responsible for any oxidation reaction as all samples were collected immediately after extrusion and before packaging. However, whether the average value of 17.49 mEq O<sub>2</sub>/kg fat and the maximum level of 94.10 mEq O<sub>2</sub>/kg fat observed in our pet food samples were indicative of a certain level of lipid oxidation remains unknown. Although this was not the aim of the present study, the authors believe that further research is needed to investigate whether PV can be used to predict pet food quality and acceptability and to set an admissible PV range for canine and feline foods after production and storage.

Across the spectrum, several peaks underlined wavelengths related to specific chemical bonds. Peaks at 1210 and 1726 nm have been demonstrated to be related to the absorption of O-H and C-H bonds, respectively, and mainly to carbohydrates and lipid compounds respectively (Miller, 2001). Peaks closer to 1450 nm are related to starch content (Osborne *et al.*, 1993). Similar spectral variation was depicted by Vis-NIRS spectra also in meat products; De Marchi *et al.* (2012, 2011) reported some broad peaks at wavelengths of around 1130 and 1400–1450 nm that may be related to functional groups such as O-H and C-H respectively. Peaks closer to 1600–1680 are related to C=C.



The best mathematical treatment used to build the prediction models was the SND+DT followed by the D 1,4,4,1 in agreement with mathematical treatment applied by Castrillo et al. (2005) to raw log (1/R) spectra in a study aimed at predicting chemical composition, digestibility and energy content of commercial extruded compound dog foods. Moreover, as expected, the first-derivative pre-treatment confirmed to enhance the spectrum resolution.

Overall, the statistics of NIRS prediction models in calibration and cross-validation results showed the potential of NIRS in predicting several traits of dry dog unground foods, and this was confirmed by RPD values that were always greater than 2. Lower NIRS predictive potential was only observed with PV; the RPD value was 1.72, in fact, indicating that NIRS can be suggested only for the screening of this trait at the moment. As shown in the scatter plots of predicted and measured traits, PV showed a very high degree of scatter according to the great standard error of cross-validation (5.96 mEq/kg of fat).

The overall results of the present study were slightly worse than those reported by Castrillo et al. (2005) and Alomar et al. (2006) for dry dog foods. These authors reported 1-VR and SECV values of 0.99 and 0.99 and 0.351 and 0.5 g/100 g DM, respectively, for protein; 1-VR and SECV values of 0.91 and 0.89 and 1.335 and 1.240 g/100 g DM, respectively, for fat; and 1-VR and SECV values of 0.96 and 0.91 and 0.364 and 0.310 g/100 g DM, respectively, for CF. Concerning NFE, the results of our study were equal to those obtained by Castrillo et al. (2005) (1-VR and SECV of 0.94 and 1.695 g/100 g DM respectively). The slightly lower accuracy of the prediction model reported in our study is probably related to the quality of Vis-NIR spectra recorded, which may have been affected by the fact that samples were kept unground. The previous studies were carried out under experimental conditions using air-dried ground samples, while the data collected in the present study came from an analysis conducted in field conditions. It is well known that

sample treatments such as air-drying and/or grinding improve the accuracy of the infrared prediction models, and similar effects were reported by several studies carried out on different matrices (Prieto et al., 2009). Nevertheless, infrared technology is used to greatest advantage under field conditions, and therefore, the use of NIR spectroscopy on unprocessed samples offers pet food manufacturers an extremely quick and cheap analysis tool.

Concerning pet food peroxide content, Vis-NIRS demonstrated potential in discriminating PV, even if a higher number of samples and a wider variability in calibration data would be suggested, together with more accurate reference methods for PV quantification to improve the accuracy of this model. Some studies in human foods and beverages have used NIRS (Yildiz et al., 2001; Huang et al., 2008) and Fourier transform near-infrared spectroscopy (Li et al., 2000) to monitor oxidation levels and PV. To our knowledge, the present work represents the first study to investigate the application of a rapid and low-cost tool in determining this crucial trait in pet foods.

## Conclusions

Near-infrared spectroscopy is proposed as an innovative technique to predict several quality traits in unground commercial extruded dog foods. Results showed the potential of NIRS in predicting chemical compositions in unground samples with a lower accuracy for moisture and ash, while the prediction model for PV suggests its use for screening purposes only. More research on this topic is needed because rapid analytical techniques and at-line applications are highly required by dog food manufacturers for the detection and quantification of chemical or technological traits. Moreover, another need is shortening the pre-processing part of NIR analysis that is generally considered the most time-consuming.

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