

Near infrared reflectance spectroscopy as an essential tool in food safety programmes: predicting ingredients in commercial compound feed

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Introduction

A number of EEC directives and decisions lay down rules governing the circulation of raw materials and feed; some of these stress the importance of a detailed statement of the products used in animal feed and set down rules for declaring the feed materials present in a compound product (77/101/EEC, 79/373/EEC, 90/44/EEC, 91/357/EEC, 98/87/EC).

The BSE and dioxin crises have highlighted the need for labelling to include a detailed quantitative statement of feed composition. On 10 January 2000 the Commission submitted a proposal to the Council for a Directive from the European Parliament and the Council, amending Directive 79/373/EEC. The purpose of the proposal is to render compulsory the statement of all the materials included in compound feedingstuffs for production animals, in terms of the percentage by weight, by descending order of weight.¹

Light microscopy is the method officially used to identify feed materials in mixtures and compound feed. However, this method is not without drawbacks.² There is, therefore, an urgent need for swift, economical and accurate analytical techniques that will ensure compliance with the rules mentioned earlier.

Various authors have demonstrated the ability of near infrared (NIR) technology, used either alone or in combination with other techniques (microscopy–NIR), to predict the percentage of different ingredients in mixtures.^{3–5} Previous studies have focused on ground samples and have mostly been what are known as viability studies, in that they used a small number of samples and only laboratory-prepared experimental mixtures.

The aim of the present study was to obtain NIR calibration equations for the instant and simultaneous prediction of both chemical composition and percentage of different ingredients in commercial feed analysed in their original form.

Material and methods

Samples

A total of 287 highly-varied compound feed samples were used (cattle, lamb, poultry, pig, ostrich, horse, rabbit, cat and dog feed), as supplied by a Spanish compound feed manufacturer (SAPROGAL SA).

Reference analyses

Samples were analysed using the methods proposed by the AOAC for moisture, H, crude protein, CP, crude fat, F, crude fibre, CF and ashes, ASH.⁶ Calibration equations for predicting ingredient proportions were obtained using the formula for each feed as supplied by the manufacturer as reference data.

Instrumentation and software

A Foss NIRSystems model 6500 scanning monochromator, (Foss NIRSystems, Silver Spring, MD, USA) equipped with a transport module, was used to measure reflectance spectra from 400 to 2498 nm every 2 nm. The analysis was carried out using the coarse rectangular transport cell with internal dimensions of 4.1 cm wide, 17.2 long and 1.4 cm deep. The 3.3 cm × 16.4 cm quartz viewing window allows 54.12 cm² of the sample surface area to be irradiated. The instrument was set up to read 12 reference scans of the ceramic reference material and the transport speed of the coarse transport cell was set to allow 32 complete wavelength range scans across the full length of the quartz window.⁷ Spectra were recorded with the ISI NIR 3 software ver.3.11 (Infrasoft International, Port Matilda, PA, USA).

Calibration development

Calibration development was performed using WINISI ver. 1.04 (Infrasoft International, Port Matilda, PA, USA). A modified partial least squares method was used to obtain regression equations for all analytical parameters. All equations were obtained using the standard normal variate and detrending method for scatter correction and different derivative mathematical treatments. The statistics used to select the best equations were the coefficient of determination (r^2), the standard error of cross-validation (*SECV*), the *RPD* and the *RER* values^{8,9}

Table 1. Calibration statistics for equations obtained for predicting the chemical composition of compound feed ($N = 287$).

	<i>N</i>	Mean	Range	<i>SD</i>	<i>SECV</i>	r^2	<i>RPD</i>	<i>RER</i>
<i>H</i>	263	10.664	6.7–13.6	1.324	0.54	0.84	2.45	12.78
CP	252	18.205	12.2–33.6	3.798	0.75	0.96	5.06	15.20
Fat	221	4.438	2.0–9.4	1.445	0.54	0.86	2.68	13.70
CF	248	6.756	2.0–18.6	3.903	0.63	0.97	6.20	26.35
ASH	245	8.401	3.9–16.7	2.196	0.83	0.86	2.65	15.42

Table 2. Calibration statistics for equations obtained for predicting feed ingredient percentages in compound feed ($N = 287$).

Feed Ingredient	SECV	r^2	RPD	Feed Ingredient	SECV	r^2	RPD
% Wheat	8.20	0.43	1.32	% Meat & bone meal	1.65	0.61	1.59
% Barley	8.93	0.57	1.52	% Alfalfa	1.44	0.94	3.92
% Barley +wheat	8.36	0.81	2.28	% Molasses	1.17	0.76	2.06
% Corn	5.37	0.87	2.76	% Total meat meal	0.88	0.98	8.16
% Soybean 44	4.08	0.83	2.43	% Royal palm	0.75	0.95	4.41
% Bran	4.99	0.66	1.71	% CaCO ₃	0.70	0.67	1.74
% Manioc	4.01	0.51	1.43	% Animal fat	0.61	0.89	2.93
% Gluten meal	2.42	0.93	3.74	% Fish meal	0.35	0.68	1.77
% Lupin	2.13	0.77	2.06	% Sugar beet pulp	0.25	0.90	3.24
% Rice	1.95	0.53	1.47	% Whey	0.13	0.94	4.38
% Sunflower	1.60	0.95	4.67	% Salt	0.09	0.52	1.44
% Poultry meal	1.82	0.89	2.99	% NH ₄ Cl	0.04	0.78	2.50

Results and discussion

Calibrations for predicting CP and CF displayed (Table 1) excellently predictive ability, judging by the high values obtained for r^2 (0.96 and 0.97) and the low values recorded for SECV (0.75% and 0.63%). Calibrations for H, fat and ashes also display an adequate degree of precision, accounting for 84% of variation in moisture and 86% of variation in fat and ashes. Errors (SECV) in the calibrations obtained for the intact feeds analysed (Table 1) lie within the usual range of values for feed analysed in ground form.^{4,10,11} The fact that the calibrations obtained in the present study using unground feed were similar, in terms of both accuracy and precision, to those obtained by other authors using ground feed may be attributed to the greater product scanning surface, obtained by using the rectangular coarse cell which has a scanning area of 54.12 cm² instead of the 28.1 cm² of the small ring cup. This difference may be of major importance in NIR analysis of samples as heterogeneous as those analysed here.

Preliminary analysis of the data in Table 2 highlights the excellent predictive ability ($r^2 \geq 0.9$; $RPD \geq 3$) of the calibrations obtained for determining the proportions of alfalfa, sunflower, gluten, sugar beet, royal palm, poultry meal, total meat meal (% meat meal + % poultry meal) and whey. Other equations showing good predictive capacity ($r^2 \geq 0.7$) were those obtained for soybean, maize, molasses, fats and lupin. Calibrations obtained for predicting the proportions of barley, manioc, bran, rice, meat and bone meal, fish meal, calcium carbonate, ammonium chloride and salt displayed an acceptable predictive capacity ($0.5 \leq r^2 \leq 0.7$). Finally, the calibration for wheat at least enabled classification of samples into low, medium and high wheat content.

The equations obtained for predicting meat meal percentages require special comment. Since December 2000, use of these meals in production animal feeds has been banned throughout Europe. Although the equation for predicting meat and bone meal displays poor accuracy, the overall equation (total meat meal) affords excellent precision ($r^2 = 0.98$, $RPD = 0.88$). The SECV values should be interpreted on the basis of the reference method; in this case, the reference method was the percentage of

feed material shown in the manufacturer's formula, as obtained by industrial by-weight breakdown, with the attendant errors. The use of other reference methods is likely to enhance the accuracy of equations for predicting meat and bone meal. Indeed, in January 2001, a total of ten European R & D centres, universities, official laboratories and one industrial partner launched a 3-year period of collaboration on a European Project,¹² which has as one of its main objectives the development and validation of new methods (PCR, NIR and NIR reflectance spectro-microscopic techniques) for the rapid, precise and reliable detection and quantification of animal meal in feed.

The equations obtained are still of a preliminary nature and it is hoped that their accuracy and precision will be enhanced through the use of different optimisation strategies. These calibrations are currently being expanded with a larger number of samples ($N > 500$) and research is also being carried out into various aspects of their development, such as the design of the calibration log, with particular reference to the number of samples to be included for each interval of the range.

Conclusions

NIR technology enables swift and economic compliance with rules relating to the labelling of feed, avoiding both the need for reagents and the production of chemical residues. NIR must, therefore, be considered an essential tool in Food Safety programmes.

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