Ad hoc labelling of compound feedingstuffs: the feed industry bets by near infrared spectroscopy

M[a] Dolores Pérez-Marín[a], Ana Garrido-Varo[a], José E. Guerrero[a], J. Zegers[b], A. Swinkels[b], L. Moya[c] and J. Lizaso[c]

[a] *Faculty of Agriculture and Forestry Engineering. ETSIAM. University of Córdoba. Apdo. 3048. 14080 Córdoba. Spain. pa2pemad@uco.es*

[b] *Maasweide Laboratory Services. NUTRECO. Boxmeer. The Netherlands.*

[c] *Nanta, S.A. NUTRECO. Ronda de Poniente, 9. 28760 Tres Cantos (Madrid). Spain.*

Introduction

The Directive 2002/2/EC of the European Parliament and of the Council[1] imposes a compulsory declaration for all the feed materials as well as their amount in compound feedingstuffs for production animals, introducing the so-called "open-declaration". All the feed materials used in the compound feedingstuffs must be listed in descending order, with their exact percentages by weight. As regards these percentages, a tolerance of +/- 15% of the declared value will be permitted. Members states shall apply these measures from 6 November 2003.

Nowadays, there are still technical problems regarding with the available analytical methods to control the quantitative information provided by the manufacturers and requested by the present Directive. Previous work[2], developed at the University of Córdoba (Spain), has proved that NIRS could predict an important number of ingredients and that could be an essential and useful analytical technology in the Safety Programs of feedcompounder plants. Nevertheless, this preliminary work has highlighted that calibration set characteristics, as set size, the presence of the different ingredient matrices and the variability of ingredients used to produce a given formula, could be determining factors for increasing the accuracy and precision of the equations developed to predict percentage of ingredients.

Recently, the University of Cordoba and NUTRECO have established a R & D co-operation agreement for demonstrating that NIRS technology, combined with optical microscopy, could be of great value as a screening technique for the quantitative declaration of ingredients in compound feedingstuffs. At present, NUTRECO feed plants laboratories use the official method (optical microscopy) for ingredients identification. Apart of the difficulty of this specialised technique for quantitative purposes, optical microscopy is a time consuming method which hampers the use of that technique for the routine control of the huge amount of compound feedingstuffs produced annually by NUTRECO.

This work tries to develop calibrations for the prediction of the inclusion percentage of each ingredient in feedingstuffs with large spectra libraries supplied by Maasweide Laboratory Services (NUTRECO-The Netherlands) and NANTA (NUTRECO-Spain).

Near Infrared Spectroscopy: Proceedings of the 11th International Conference.

Material and methods

Calibration Sets

Two large spectra libraries were used in this work in the development of NIR calibration to predict the inclusion percentage of each ingredient used in the production of feedingstuffs.

- **Calibration set 1,** supplied by Maasweide Laboratory Services (NUTRECO-The Netherlands). It has 531 unground feedingstuff samples destined to different animal species (poultry, cattle and pig). The samples were analysed in a FOSS NIRSystems 6500 monochromator, equipped with transport module, using the natural sample cell.
- **Calibration set 2,** supplied by NANTA (NUTRECO-Spain). It has 7598 ground feedingstuff samples destined to different animal species (poultry, cattle, pig, ovine and rabbits). Their NIR spectra were collected in a FOSS NIRSystems 5000 instrument, equipped with spinning module, that belongs to the NANTA NIR Network. Standard ring cups were use for the analysis.

The reference data (% of each ingredient) were obtained from the formulation declared by the feed company.

Validation Set

A set of 100 samples belonging to NANTA was used to validate the chemometric models performance with the calibration set 2, described previously.

Chemometric treatment of data

The software Win ISI ver. 1.05 was used in all cases to develop the different chemometric models.

Global equations

Global NIR calibrations for the prediction of in compound feeds were obtained for each calibration sets (1 from Maasweide and 2 from NANTA). The equations were developed using MPLS regression, and different signal pre-treatments (derivatives and scatter correction)[3].

LOCAL equations

LOCAL algorithm[4] were used to predict the inclusion percentage of two ingredients selected:

- Sunflower meal, that is one of the ingredients for which traditional calibration (GLOBAL) provided better results.
- Wheat, that is one of the ingredients with the poorest results in global calibration.

LOCAL equations were developed using the calibration set 2 as "Product Library", and the validation set described previously was predicted. The main parameters defined to perform LOCAL predictions were in both cases: 90 samples selected from the library; 20 as maximum number of PLS factors; and 4 as the PLS factors to be excluded from the final predictions. First derivative treatment was selected in the case of sunflower meal and second derivative treatment for the wheat.

Results and discussion

Table 1 show the calibration statistics for global equations obtained using the calibration set 1 (unground). In general, it can appreciate that the predictive ability of the equations are good or very good. Also, the results confirm that the prediction of different cereal percentages is more difficult that the prediction of other types of ingredients with more defined characteristics, as sunflower meal.[2,5,6]

Table 1. Calibration statistics for the prediction of ingredient inclusion percentage in compound feed, using the calibration set 1 (n=531) analysed unground.

Constituent	Mean	Range	SD	SECV	r^2	RPD
Animal Fat	0.37	0.0-1.5	0.42	0.26	0.61	1.61
Barley	2.19	0.0-39.5	6.18	2.43	0.85	2.54
Beetpulp	1.55	0.0-25.0	3.85	1.04	0.93	3.72
Bread bakery products	4.76	0.0-8.0	1.75	0.69	0.84	2.54
Citrus pulp	7.91	0.0-35.2	7.6	2.52	0.89	3.02
Coconut meal	6.15	0.0-15.1	4.26	2.53	0.65	1.68
Corn	12.13	0.0-60.0	14.26	2.74	0.96	5.21
Cornbyproduct	0.55	0.0-5.0	1.26	0.46	0.87	2.73
Cornglutenfeed	2.69	0.0-30.0	5.22	1.67	0.90	3.13
Fishmeal	0.44	0.0-7.3	1.22	0.72	0.66	1.70
Molasses	1.23	0.0-7.0	1.77	0.30	0.97	5.81
Palmkernelmeal	3.27	0.0-25.1	6.05	0.80	0.98	7.59
Rapeseed	4.72	0.0-9.0	2.00	1.33	0.57	1.50
Rapeseedmeal	2.30	0.0-30.7	3.38	1.16	0.88	2.93
Soybeanfullfat	1.28	0.0-30.0	2.52	0.55	0.95	4.59
Soybeanmeal	14.05	0.0-70.0	9.65	2.53	0.93	3.82
Sunflowermeal	2.90	0.0-17.7	3.86	0.76	0.96	5.09
Tapioca	2.48	0.0-40.5	6.22	2.51	0.84	2.48
Vinasses	5.65	0.0-9.0	0.75	0.36	0.77	2.08
Wheat	24.22	0.0-63.1	14.32	4.95	0.88	2.89
Wheatbran	1.85	0.0-14.4	2.89	2.75	0.11	1.05
Yeast	1.16	0.0-4.2	0.84	0.19	0.95	4.42

Table 2 show the calibration statistics obtained for global equations obtained using the calibration set 2 (ground). Xicatto et al.[5,6], working with ground samples of compound feeds for rabbits obtained all cases SECV values higher than the ones resulting in this work (ie. 9.5% for lucerne, 3.3% for beet pulp, 8.3% for barley, 8.2% for wheat, 4.2% for sunflower).

Table 2. Calibration statistics for the prediction of ingredient inclusion percentage in compound feed, using the calibration set 2 (n=7598) analysed ground.

Constituent	Mean	Range	SD	SECV	r^2	RPD	CV
Bakery by-products	0.75	0.0-10.0	1.64	**0.55**	0.89	2.96	73.72
Barley	13.75	0.0-52.2	14.89	**3.67**	0.94	4.05	26.72
Beet pulp	0.22	0.0-7.0	1.18	**0.66**	0.69	1.80	305.43
Bicalcium Phosphate	0.75	0.0-2.3	0.57	**0.15**	0.93	3.77	19.91
Cacium Carbonate	1.01	0.0-16.7	1.28	**0.25**	0.96	5.03	25.16
Cassava	0.74	0.0-15.0	1.49	**0.30**	0.96	4.96	40.77

Corn	3.85	0.0-60.0	7.41	**2.94**	0.84	2.52	76.40
Corn Bagasse	1.60	0.0-15.0	2.87	**0.98**	0.88	2.92	61.67
Corn Germ20	0.29	0.0-8.0	1.16	**0.60**	0.73	1.91	205.82
Corn GlutenMeal	0.06	0.0-2.8	0.22	**0.15**	0.55	1.48	243.20
Corn Pulp	2.22	0.0-10.0	3.35	**1.09**	0.89	3.07	49.01
Fat	0.77	0.0-8.0	1.25	**0.29**	0.94	4.25	38.28
Full Fat Soy	0.68	0.0-19.2	1.99	**1.48**	0.45	1.35	217.86
Lard	0.02	0.0-5.5	0.15	**0.07**	0.78	2.12	297.05
Lucerne	7.35	0.0-40.4	13.12	**1.12**	0.99	11.67	15.31
Lysine	0.09	0.0-1.0	0.13	**0.06**	0.77	2.08	69.58
Metionine	0.04	0.0-0.3	0.06	**0.01**	0.94	4.16	36.21
Red complement	0.00	0.0-1.0	0.03	**0.01**	0.92	3.65	238.24
Rice bran	0.66	0.0-7.0	1.57	**0.70**	0.80	2.26	105.85
Salt	0.33	0.04-1.6	0.14	**0.05**	0.85	2.55	16.20
Sepiolite	0.09	0.004-2.0	0.28	**0.04**	0.98	6.41	51.12
Sodium Bicarbonate	0.08	0.0-1.25	0.14	**0.04**	0.90	3.11	52.09
Soy Oil	0.28	0.0-5.5	0.61	**0.36**	0.66	1.72	126.60
Soymeal 44+47	13.34	0.0-55.6	10.54	**1.70**	0.97	6.20	12.74
Soymeal44	9.84	0.0-55.6	8.64	**2.76**	0.90	3.14	27.99
Soymeal47	0.33	0.0-37.2	2.66	**1.29**	0.76	2.06	396.46
Straw	0.33	0.0-19.8	1.15	**0.16**	0.98	7.08	48.56
Sugarcane Molasses	1.76	0.0-4.0	1.48	**0.35**	0.94	4.25	19.85
Sunflower Meal	5.84	0.0-15.1	5.44	**0.77**	0.98	7.09	13.14
Sunflower Seed	0.86	0.0-6.9	1.36	**0.14**	0.99	9.98	15.91
Wheat	14.41	0.0-67.5	15.77	**3.30**	0.96	4.77	22.93
Wheat bran	9.39	0.0-35.0	12.84	**2.01**	0.98	6.39	21.39
Zootecnic Meal	4.71	0.0-30.0	8.24	**2.42**	0.91	3.41	51.34

Figures 1 and 2 show the results obtained in the prediction of the percentage of sunflower and wheat for the 100 compound feeds belonging to the validation set, using the equations developed with GLOBAL and LOCAL algorithms with the calibration set 2 (n= 7598). It can be observed that for both ingredients the predictive ability of LOCAL equations is better. Particularly, for the ingredient "wheat", for which the SEP value obtained with LOCAL algorithm (2.09%) was less than the half of the SEP value reached with GLOBAL (5.33%). This decrease can be appreciated also when the bias values are compared (0.01 for LOCAL *vs* –0.32 for GLOBAL). In the case of the ingredient "sunflower", for which the results obtained with GLOBAL are quite good, there is a slightly improvement in the prediction statistics when using LOCAL.

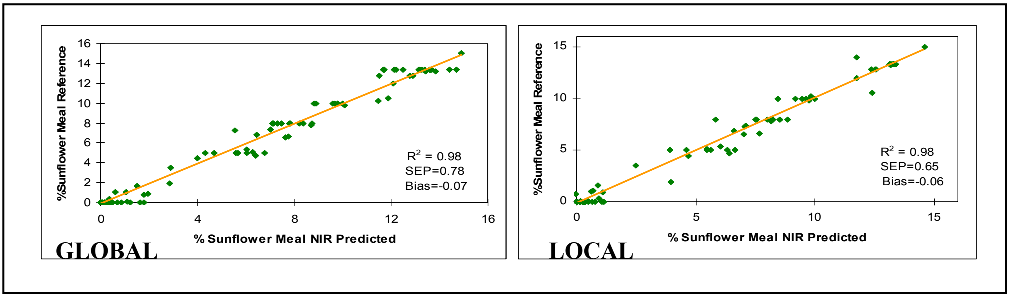

Figure 1. Global *vs* LOCAL predictions for sunflower meal percentage.

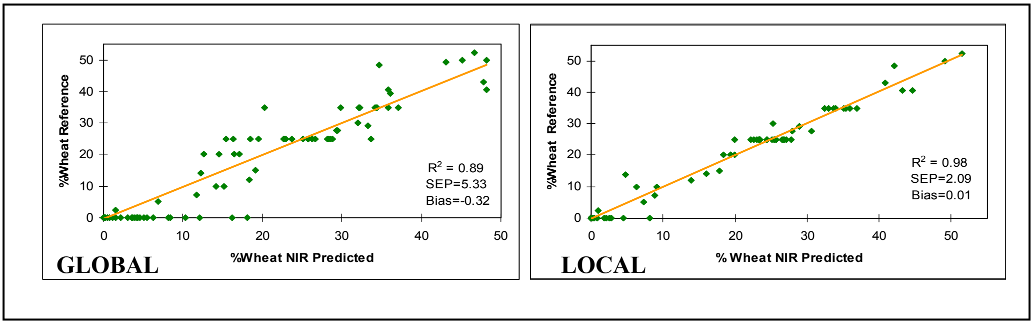

Figure 2. Global vs LOCAL predictions for wheat percentage.

Conclusions

The results show that NIRS could provide the industry and inspection bodies with a fast screening procedure for implementing the Open-Declaration regulation in compound feedingstuffs

References

1. Directive 2002/2/EC of the European Parliament and of the Council of 28 January 2002. OJ of the European Communities No L63, 6.3.2002, p.23 (2002).
2. A. Garrido-Varo, M.D. Pérez-Marín, A. Gómez-Cabrera, J.E. Guerrero, F. De Paz and N. Delgado, in *Near Infrared Spectroscopy: Proceedings of the 10th International Conference*, Ed by A.M.C. Davies and R.K. Cho. NIR Publications, Chichester, UK, p. 145 (2002).
3. ISI, The complete software solution using a single screen for routine analysis, robust calibrations, and networking. Manual. FOSS NIRSystems/TECATOR. Infrasoft International, LLC. Sylver Spring MD, USA (2000).
4. J.S. Shenk, M.O. Westerhaus and P. Berzaghi, *J. Near Infrared Spectrosc.* **5**, 223 (1997).
5. G. Xicatto, A. Trocino, A. Carazzolo, M. Meurens, L. Maertens and R. Carabaño. *Anim. Feed Sci. Technol.* **77,** 201 (1999).
6. G. Xicatto, A. Trocino, J.L: De Boever, L. Maertens, R. Carabaño, J.J. Pascual, J.M. Perez, T. Gidenne and L. Falcao-E-Cunha. *Anim. Feed Sci. Technol.* **104,** 153 (2003).