NIRS technology transfer: experience of a livestock farmers cooperative

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Introduction

In 1996 an R & D Agreement was signed between the Animal Production Department (ETSIAM, University of Córdoba) and COVAP, that is one of the largest Spanish livestock farmer cooperative. COVAP spreads over three autonomous communities, and manufactures, processes and sells feeds, meat and dairy products. Moreover, it has feedlots for beef calf and lamb and one unifeed mixtures plant.

This R & D Agreement pursues to joint efforts to undertake effectively and appropriately activities of education, training and technology transfer in the field of animal nutrition. In this framework, the co-operation in R & D activities concerning NIRS Technology has been traditionally dealt.

Since 1998, COVAP started, as part of its quality policy, to implement Quality Assurance Programs of process and products. The slogan of the cooperative became to be "Quality from the Origin". In 1999, both parts agreed that the implementation of NIRS at COVAP laboratory for the routine analysis of their products may be essential to achieve the slogan goal. Since the acquisition of a NIR instrument by COVAP, the following co-operation activities have been carried out: 1.- Design of the work schedule for calibration development. 2.- NIRS training courses to cooperative specialists. 3- Calibration equations for the prediction of the chemical composition of unground compound feedingstuffs. 4.- Calibration equations for the prediction of the chemical composition of raw materials. 5.- Cloning with a master instrument placed at UCO, and equation transfer for the prediction of fatty acids in Iberian pig fats. The NIR predicted values of fatty acids are being used for the payment to Iberian pig producers that belong to the cooperative.

The objective of the present paper is to present results of some of the NIRS co-operation activities undertaken in the frame of the R & D agreement signed between COVAP and the University of Córdoba.

Material and methods

Samples and reference data

- Set I (compound feedingstuffs): consisting of 400 pelleted and 300 meals commercial compound feedingstuffs produced by COVAP. They were used in the development of calibrations for the prediction of chemical composition. Samples were analysed for moisture, crude protein, crude fibre, fat and ashes.

- Set II (compound feedingstuffs): consisting of 178 samples of commercial compound feedingstuffs produced by COVAP. They were used in the development of calibrations for the prediction of the percentage of ingredients. The reference data were obtained from the feed formulation.
- Set III (fat samples). One sample of Iberian pig fat (%C16:0=19.9%, %C18:0=10.0%, %C18:1=52.5%, %C18.2=11.2%) used as standardisation sample. Ten samples of fat taken from animals slaughtered in COVAP, used for validating the equation transfer. Fatty acid composition was obtained by gas chromatography (GC).

NIR instruments

Three monochromator instruments have been used in the present work :

- A Foss-NIRSystems 6500 monochromator with an attached spinning module and located in UCO (validation fat samples and fat standardisation sample). Fat samples were analysed after melting in a microwave oven. They were maintained at 35ºC in an oven to be scanned in liquid state.[1]
- A Foss-NIRSystems 6500 monochromator with an attached transport module and located in UCO (set II of compound feedingstuffs). Samples were scanned unground (Coarse Granular Cell) and ground (1/4 rectangular cell) after milling in a Cyclone mill (1mm).
- A Foss-NIRSystems 5000 composite with an attached transport module and located in COVAP (validation fat samples, fat standardisation sample and set I of compound feedingstuffs). All the samples were analysed, without previous milling, using the natural product cell.

Calibrations transfer

The standardisation sample was analysed by duplicate in the master (UCO) and satellite (COVAP) instruments using folded transmission and gold reflector surface cam-lock cups (0.1 mm pathlength). The 10 validations samples were analysed by duplicate in the COVAP instrument and also in the master (UCO). The standardisation algorithm included in the software WINISI version 1.05 (Infrasoft International, Port Matilda, PA, USA) was used.[2] The standardisation file produced was applied to the validation set scanned on the COVAP instrument, in order to make possible to use the equations previously developed in the UCO instrument.[3]

Results and discussion

Quality control of compound feedingstuffs

One of the first advisory activities carried out by UCO under the COVAP-UCO R & D agreement was concerned with the choice of the instrument and accessories to buy. COVAP agreed to acquire a FOSS NIRsystems 5000 equipped with transport module and provided of the natural product cup. That would allow COVAP to scan unground products, saving time and money, avoiding also the tedious milling task.

Table 1 shows calibration statistics for the prediction of compound feedingstuffs "as they are produced", that is, pelleted or meals. In a first step, it was decided to develop separated calibrations for the two sample presentation types. As can be seen, on Table 1, the accuracy of the equations developed for the prediction of the gross chemical composition is similar or even better that the obtained by other authors after fine milling.[4,5] The accuracy of the calibration for moisture should be improved by adding samples with NIRS and moisture analysis performed the same day or very close in time.

Table1. Calibration statistics for the prediction of the chemical composition in compound feedingstuffs.

	Constituent	Range	ETVC	r^2	RER	RPD
Pelleted	Moisture	8.69-14.95	0.56	0.73	11.1	1.9
	Fibre	2.47-14.59	0.38	0.98	31.5	6.5
	Ashes	4.94-13.01	0.42	0.90	19.1	3.1
	Fat	1.75-5.92	0.22	0.94	18.9	4.1
	Protein	12.8-19.62	0.35	0.94	19.1	4.1
Meals	Moisture	8.88-14.35	0.51	0.65	10.7	1.7
	Fibre	2.63-8.32	0.40	0.84	14.2	2.5
	Ashes	1.59-10.86	0.47	0.87	19.8	2.8
	Fat	2.12-7.77	0.25	0.96	22.3	4.1
	Protein	8.41-19.20	0.52	0.94	20.7	4.3

By the time that R & D Agreement was signed (2000), the European Parliament was having discussions for a new legislation concerning to the labelling of compound feedingstuffs (Proposal of an European Parliament and Council Directive 2000/0015 COD). UCO considered that the feed industry should cooperate in basic pre-normative research, in order to provide them with analytical methods that ensure the compliance with the coming legislation. For that reason, COVAP agreed to send samples to UCO to be included as part of a PhD Thesis work, aimed to demonstrate the ability of NIRS to predict the percentage of ingredients in compound feedingstuffs.[6]

Table 2. Preliminary statistics for the prediction of ingredients in compound feeds (n=178).

	UNGROUND			GROUND		
	Range	**ETVC**	**r^2**	**Rango**	**ETVC**	**r^2**
BARLEY	**7.4-42.9**	**3.81**	**0.86**	**7.4-40.0**	**4.11**	**0.83**
CORN	**0.0-49.6**	**3.04**	**0.95**	**0.0-45.0**	**1.77**	**0.98**
WHEAT	**0.0-32.0**	**3.84**	**0.80**	**0.0-32.0**	**3.78**	**0.79**
GLUTEN	**0.0-14.0**	**0.74**	**0.98**	**0.0-14.0**	**0.75**	**0.98**
SOYBEAN MEAL	**0.0-28.3**	**1.20**	**0.97**	**0.0-23.2**	**0.84**	**0.99**
BRAN	**0.0-10.0**	**1.28**	**0.80**	**0.0-12.0**	**1.47**	**0.77**
SUNFLOWER	**0.0-12.3**	**0.79**	**0.97**	**0.0-12.3**	**0.62**	**0.98**
ALFALFA	**0.0-15.0**	**0.52**	**0.99**	**0.0-15.0**	**0.95**	**0.96**
LUPIN SEEDS	**0.0-8.0**	**1.22**	**0.79**	**0.0-8.0**	**0.82**	**0.91**
BY-PASS FAT	**0.0-3.3**	**0.18**	**0.98**	**0.0-3.3**	**0.08**	**0.99**
CAL. CARBONATE	**0.6-2.59**	**0.18**	**0.82**	**0.6-2.59**	**0.17**	**0.84**
BIC. PHOSPHATE	**0.0-1.4**	**0.10**	**0.94**	**0.0-1.4**	**0.12**	**0.91**
SALT	**0.0-0.88**	**0.08**	**0.84**	**0.1-0.88**	**0.10**	**0.73**
LARD	**0.0-3.2**	**0.11**	**0.98**	**0.0-2.2**	**0.12**	**0.98**
MOLASSES	**0.0-4.0**	**0.24**	**0.98**	**0.0-4.0**	**0.22**	**0.98**
SOD. BICARBONATE	**0.0-0.84**	**0.07**	**0.96**	**0.0-0.84**	**0.05**	**0.98**
MV SUPPLEMENT	**0.0-0.7**	**0.13**	**0.51**	**0.0-0.6**	**0.08**	**0.77**
ANTIFUNGAL	**0.0-0.1**	**0.01**	**0.98**	**0.0-0.1**	**0.01**	**0.98**
ANTIOXIDANT	**0.0-0.01**	**0.002**	**0.85**	**0.0-0.01**	**0.001**	**0.87**

Table 2 shows preliminary results obtained for the equations developed for the prediction of different ingredients. Given the variability of ingredients used in the production of a specific compound feed and that the constituent to predict (% of ingredient) it is not a single chemical entity, it was decided to develop calibrations for both, ground and unground compound feedingstuffs. As can be seen on Table 2, with a few exceptions (ie. corn), the SECV values are very similar for both types of sample presentation and even some times (ie. alfalfa) the results are better for the unground form.

The equations produced have still a very low number of samples, but clearly help to demonstrate that NIRS may predict ingredients in a complex matrix, as a compound feedinsgtuff.[7]

Quality control of Iberian pig products

Several years of cooperation among the Iberian pig sector and the Animal Production Department of the University of Córdoba resulted in the clear demonstration of the ability of NIRS Technology for the analysis of fatty acids, with an accuracy similar or better than the obtained with the reference analysis (Gas Chromatography).[8]

Traditionally, COVAP make payments to the Iberian pig producers according to the type of feeding of the animal, controlled by “on farm inspectors”, and to the quality of the pig fat determined by GC. Both payment systems are expensive in terms of personnel and analytical costs. That is why COVAP decided to implement NIRS for producer payments.

Equations were developed on the UCO instrument and equations with a high precision and accuracy were obtained. The r^2 values were higher than 0.98 for C16:0, C18:0, C18:1 and C18:2 and the SECV values were 0.28, 0.27, 0.20 and 0.16, respectively. The methodology used for equation development and in particular the need of a repeatability file has been previously reported.[3]

The equations were further transferred to the COVAP instrument. Firstly, the instrument should be optically matched. According to previous results,[2] the master and satellite instruments were cloned using one single fat sample with a composition in fatty acids similar to the mean values of the calibration set. To validate the cloning procedure, ten samples of fats from producers were scanned in the satellite instrument. Table 3 shows the fatty acid predictions for the 10 validation samples before and after standardisation. When the predicted data before standardisation are compared to the reference data, it can be observed that there are important biases (1 to 3 units of difference) depending of the fatty acid. However, after standardisation the NIRS predicted values by the COVAP instrument are very close to the GC data.

The success of the calibration transfer was crucial for implementing NIRS for the quality control of the animals entering to the COVAP slaughterhouse plant. NIRS would allow to trace each individual Iberian ham, one of the most expensive and luxurious Spanish food products.

Table 3. Fatty acids GC data and NIRS predicted data for 10 samples analysed in COVAP instrument before and after standardisation.

	Reference data (GC)				Predicted before standard.				Predicted after standard.			
	C16:0	C18:0	C18:1	C18:2	C16:0	C18:0	C18:1	C18:2	C16:0	C18:0	C18:1	C18:2
1	23.42	12.06	51.07	7.84	25.73	12.61	48.62	9.45	22.99	11.94	51.56	8.33
2	20.65	9.40	54.49	9.84	23.32	9.73	51.58	11.47	20.76	9.34	54.30	10.11
3	21.81	9.78	51.90	10.53	24.60	10.34	48.80	11.99	21.91	9.89	51.80	10.59
4	23.20	11.46	50.46	9.32	25.68	12.13	47.62	10.79	22.88	11.49	50.74	9.49
5	21.39	9.87	53.24	9.77	24.32	10.36	50.09	11.36	21.63	9.90	52.98	10.01
6	25.23	13.60	47.44	8.15	28.00	14.41	44.45	9.70	24.98	13.55	47.89	8.52
7	22.06	10.85	51.68	9.98	24.75	11.34	48.91	11.36	22.04	10.78	51.91	10.01
8	22.25	10.49	51.76	9.69	25.01	11.29	48.54	11.41	22.26	10.73	51.58	10.05
9	22.74	10.94	50.99	9.59	25.54	11.69	47.76	11.29	22.73	11.09	50.89	9.94
10	24.08	12.35	49.04	9.15	26.67	13.03	46.15	10.75	23.77	12.22	49.42	9.46

Conclusions

The implementation of NIRS Technology at COVAP cooperative represents a significant step forward to ensure that the cooperative slogan "Quality from the Origin" can be dealt effectively at minimal analytical costs. The cooperation COVAP-UCO is expected to produce a marked influence in the way on which NIRS may be inserted into traceability schemes ("in plant" and "at farm" levels).

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