Stratfeed near infrared instrument network for detecting animal tissues in feedingstuffs

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Introduction

This work was undertaken within the framework of the EU STRATFEED Project (GRD1-2000-25002), titled *"Strategies and methods to detect and quantify mammalian tissues in feedingstuffs"*, in which the main objective is the development and validation of new methodologies (PCR, NIRS and NIR-Microscopy) for the rapid, precise and reliable detection of banned animal meal in feedingstuffs.

For that purpose, the near infrared (NIR) technique can allow a substantial increase of the number of controlled samples and provides an instantaneous response to detect adulterated specimens. The repeatability of NIR analysis in a single instrument is very good, but between different spectrophotometers can be poor, mainly due to the small differences in their optical parts[1]. Therefore, to can merge spectra collected in different NIR instruments, avoiding to have to transport specimens and scan these on each instrument, instruments must be standardised. The standardisation procedure involves making the spectra obtained in other instruments identical to the spectra from the master instrument. Moreover, when a network of cloned instruments is established, the models developed on one instrument (master) can be mounted on all the instruments in the network.

The goal of the present work is to create a European NIR spectrometer network that permits the merging of spectral libraries of meat and bone meal and feedingstuffs specimens into one large spectral library for the EU. This may allow harmonised evaluation of feed using shared common calibration models.

Materials and methods

Standardisation procedure

Five NIR instruments, spread over four European countries, have involved in the creation of this European NIR Spectrometer Network. A FOSS NIRSystems 6500 monochromator, equipped with

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spinning sample module and belonging to the University of Córdoba (UCO), was used as master. The four satellites instruments, that make up the NIR network, are described following:

- Satellite 1. A FOSS NIRSystems 6500, equipped with spinning module and belonging to the Agricultural Research Centre of Gembloux (CRAGx) in Belgique.
- Satellite 2. A FOSS NIRSystems 6500, equipped with transport module and belonging to the Official Agrofood Laboratory of Cabrils (LAGC) in Spain.
- Satellite 3. A FOSS NIRSystems 6500 equipped with spinning module and belonging to the Scottish Agricultural College of Aberdeen (SAC) in UK.
- Satellite 4. A FOSS NIRSystems 6500 equipped with transport module and belonging to Maasweide Laboratories of NUTRECO (NUT) in The Netherlands.

To achieve that the NIRS spectra and equations could be transferred between the NIRS instruments of the STRATFEED Project, the standardisation algorithm of Shenk and Westerhaus,[1,2] available in the WinISI software (ver. 1.5), was used.[3] A standardisation box, consisting of 30 sealed cups of different agricultural products, was analysed in duplicate in the master and in the four satellites instruments. Given that the standardisation of NIR instruments is a critical operation and requires very good data, a common standardisation protocol was designed. This protocol tries to control all the factors that can influence that procedure, as laboratory environment and instrument working conditions. With these data, standardisation matrixes (.STD files) were developed.

Evaluation samples

To show the performance of the network, a set of 9 feedingstuff samples were analysed in the five instruments described previously. These samples were commercial feeds, provided by the STRATFEED Sample Bank, and were scanned one time in NUTRECO instrument and by triplicate in UCO, SAC, LAGC and CRAGx instruments. Standard ring cups were used for analyse the samples, except in the LAGC instrument in which 1/4 rectangular cups were used and in SAC instrument that used 55 mm diameter round cups.

NIR equation

The spectra obtained were predicted with a NIR equation performed for quantifying the percentage of inclusion of meat and bone meal (MBM) in compound feedingstuffs (range = 0.00–31.90%; *SD* = 5.30%; *SECV*=0.84%; r^2 = 0.97). The equation was developed with a calibration set of 630 samples, scanned grounded in the UCO instrument. MPLS regression, first derivative math treatment and SNV and Detrend scatter correction were used to obtain this equation.

Results and discussion

The statistics obtained in the evaluation of the network performance are showed in Table 1. Before the standardisation, the standard errors of differences bias corrected or *SED*(*c*) and the bias values among instruments ranged from 0.41% to 0.86% and from –0.20% to 4.78%, respectively. After standardisation, it was observed a high reduction of the bias values, which now range from –0.27% to –1.53%.

Table 2 shows the NIR predicted values of the nine feedingstuffs analysed in the master instrument (UCO) and in the four satellite instruments (CRAGx, NUT, LAGC and SAC).

It can be appreciated in Tables 1 and 2 that after the cloning procedure the agreement between predicted values in the master and in the satellites instruments is different for each instrument in the network. There is a decreasing of bias values in all the satellite instruments, except in the one belonging to CRAGx laboratory. This fact shows that CRAGx spectrophotometer is very similar to UCO one, and that in this case the standardisation has hardly influence in the results. After standardisation, the reduction in the bias values for NUT instrument was very marked. Thus, as can

be appreciated in Table 2, before standardisation in NUT instrument samples with MBM (i.e. Samples 112 and 113) were predicted as free of MBM; however, after standardisation they were correctly predicted, and these predictions were very similar to those reached in the master instrument for these samples.

Table 1. Statistics for evaluating the standardisation of five NIR instruments, using a validation set (*n* = 8, without sample 111) and an NIR equation for predicting the %MBM added to feedingstuffs.

	Master	Satellites							
	UCO	**Cragx^{before}**	**Cragx^{after}**	**NUT^{before}**	**NUT^{after}**	**LAGC^{before}**	**LAGC^{after}**	**SAC^{before}**	**SAC^{after}**
Mean	2.32	2.52	1.91	–2.46	1.88	3.20	2.59	4.81	3.85
SD	2.61	2.44	2.69	2.43	2.56	2.50	2.68	2.97	3.08
***SED*(*c*)**	**0.57**	**0.41**	**0.56**	**0.54**	**0.57**	**0.86**	**0.85**	**0.82**	**0.93**
Bias	**–0.27**	**–0.20**	**0.41**	**4.78**	**0.43**	**–0.88**	**–0.27**	**–2.49**	**–1.53**
r^2	0.97	0.98	0.96	0.96	0.95	0.89	0.90	0.93	0.92
Av. H	1.73	3.49*	2.27	6.51	1.89	4.76	1.83	3.31	2.27

[a]*SEP*(*c*) value

Table 2. NIR predicted values (% of MBM) of the validation set, before and after standardisation.

Sample	UCO master	Cragx^{before}	Cragx^{after}	Nut^{before}	Nut^{after}	LaGC^{before}	LaGC^{after}	SAC^{before}	SAC^{after}
102	1.3	1.6	0.7	–3.8	0.8	4.0	3.3	4.2	3.2
103	–0.1	–0.4	–1.7	–5.5	–1.5	0.7	–0.6	0.7	–0.5
111	3.1	—	—	—	—	4.6	4.2	4.3	3.3
112	3.5	4.3	3.8	–0.7	3.7	4.7	4.2	7.1	6.3
113	5.2	5.1	4.9	–0.2	4.3	5.8	5.5	8.5	7.7
114	2.9	2.7	2.0	–2.1	2.2	2.8	2.2	4.6	3.6
115	0.4	1.1	0.7	–3.4	0.9	1.4	0.9	3.0	2.1
116	–0.7	–0.3	–0.8	–5.3	–1.1	–0.4	–1.0	1.9	0.8
120	6.3	6.1	5.7	1.1	5.7	6.6	6.1	8.5	7.6

— error in spectral data

The results presented in Tables 1 and 2 indicate also that the SAC NIR instrument has a less matching with the master in comparison with the other three satellites. That may be caused by the different type of cups used in SAC laboratory to analyse the validation set of 9 feedingstuffs. While in the master (UCO) standard ring cups (3.75 mm diameter) were used, SAC laboratory used a 55 mm diameter round cup.

The preliminary equation used in this work is being expanded and new chemometric models are being evaluated. It is possible that the new equations may bring a further reduction in the SED(c) and bias values. Moreover, further works must be carried out to optimise the standardisation matrixes used to match these five instruments.

Conclusions

The results show that the STRATFEED NIR Network allows all instruments to produce harmonised results, which are of great importance to demonstrate that NIRS could be used as a standardised method for implementing the ban on the use of animal meal protein in compound feedingstuffs across Europe.

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