



Spectral library validation to identify ingredients of compound feedingstuffs by near infrared reflectance microscopy

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ABSTRACT

To guarantee feed quality and safety the development and improvement of analytical methods for feed authentication and detection of contaminants is fundamental. Near infrared reflectance microscopy (NIRM) has been investigated as an alternative method to contribute to control systems for feed materials. The major task is the need to build NIRM reference spectral libraries that must represent the variability in feed ingredients. The aim of the present work was to evaluate the performance of a NIRM reference spectral library on animal feed, with external samples of animal feed ingredients and possible contaminants such as processed animal proteins, and in particular to assess its ability to identify ingredients in mixtures. Three external sample sets were used: (A) artificial mixtures, (B) synthetic mixtures and (C) synthetic binary mixtures. The prediction and repeatability results for set A, in which the spectra are from pure ingredients, were very good for both animal and vegetable ingredients and confirm that the spectral library is very good at identifying spectra from pure ingredients. For sets B and C, in which the spectra were measured on mixtures, the prediction results were very disappointing compared with the artificial samples. This means that a strategy that tries to match the spectra taken from a mixture with those of pure ingredients is unlikely to meet with much success. It is possible that an interpolation between pure ingredients for suitably chosen spectral ranges may provide a way to extend this system to mixtures, including mixtures of several ingredients.

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1. Introduction

Optimal nutrition implies that animal feeds must be considered not only in terms of production efficiency but also in the light of their ability to promote health and protect against disease [1]. The health of the animal is fundamental for determining the quality, safety and wholesomeness of foods of animal origin for human consumption [2]. These new nutritional concepts require the development and dissemination of improved and more readily available analytical methods for feed authentication and detection of contaminants, to support various aspects of feed quality and safety and contribute to the improvement of existing control systems across the world.

The official analytical method to obtain qualitative and quantitative information about the ingredients in mixtures and compound feedstuffs is classical microscopy. Optical microscopy is at the moment the only official method in the European Union for detection of constituents of animal origin in feeds [3].

A number of other methods, such as polymerase chain reaction, immunoassay, and macro near infrared spectroscopy have been

investigated for the analysis of ingredients in feed samples, including the detection of banned processed animal proteins [4–6]. The method studied here, near infrared reflectance microscopy (NIRM), allows the collection of hundreds or thousands of spectra from a feed sample and has been proposed as a new analytical approach for identifying ingredients in animal feeds [7,8] and detecting and quantifying undesirable substances such as meat and bone meal (MBM) in feedstuffs [9–12].

A major task in implementing this methodology is the need to build NIRM reference spectral libraries comprising thousands of spectra taken from samples from reliable sources in order to represent the natural variability in animal, vegetable and mineral feed ingredients. Once the library is built, spectra collected from animal feeds may be compared with the spectra in the library to identify the ingredients included in the feeds. As the number of possible ingredients becomes large, not only does it increase the effort involved in constructing the library and the computational effort involved in comparing an unknown with all the spectra in the library, but it becomes more difficult to discriminate between the ingredients. For example Gendrin et al. [13] reported that it becomes more difficult to find discriminating wavelengths when large numbers of ingredients are involved.

The optimization of conditions for sample presentation and spectral measurement and treatment, and the building of an NIRM

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Table 1

Ingredients, samples and total number of NIRM spectra included in the spectral library. Percentage ingredient contents for sample set A (artificial mixtures) and set B (synthetic mixtures).

Ingredients of library	ns	nsp	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6	
			A	B	A	B	A	B	A	B	A	B	A	B
Lucerne dehydrated	24	5109												
Maize silage	23	4836												
Fababean silage	2	450												
Grass silage	13	2713												
Grass hay	2	421												
Cereal straw	27	5644												
Beet pulp	14	3039											0.9	1.0
Cotton seed	7	1016												
DDGS barley	1	202												
Soybean meal	15	3330	50.0	50.0	40.2	40.0	32.5	32.3	25.2	25.0	25.2	25.0	25.2	25.0
Sunflower seed	7	1578												
Oats	4	847												
Wheat	15	3210							9.8	10.0	9.8	10.0	9.8	10.0
Barley	21	4449			19.7	20.0	32.5	32.3	35.0	35.0	35.0	35.0	34.6	35.0
Rye	5	1146												
Maize	21	4826	50.0	50.0	40.2	40.0	32.5	32.3	25.2	25.0	25.2	25.0	25.2	25.0
Bran	2	419												
Corn flakes	1	210												
Peas	2	427												
Palm seed	1	218												
Wheat middlings	1	210												
Mineral correctors	6	1222									1.7	2.0	2.1	2.0
By-pass fat	3	630												
Citrus pulp	3	663												
Milk powder	1	234												
Whey powder	1	210												
Blood meal	1	235												
Meat and bone meal	4	937					2.6	3.0	4.7	5.0	3.0	3.0	2.1	2.0
Haemoglobin	1	234												
Animal plasma	1	234												

ns: number of samples; nsp: number of spectra.

reference library have been reported previously [7,14]. An internal cross-validation of the use of the *K*-nearest neighbours algorithm [15] to classify spectra by comparison with the library showed some confusion between similar vegetal ingredients but good discrimination between ingredients of animal and vegetal origin [7].

The aim of present work was to evaluate the performance of this NIRM reference spectral library on animal feed, challenging it with external samples of animal feed ingredients and possible contaminants such as processed animal proteins, and in particular to assess its ability to identify ingredients in mixtures.

2. Materials and methods

2.1. Samples

The samples used to construct the spectral library, and the samples used for validation, were all provided by industrial sources in the north of Spain. They are samples of pure ingredients representing the natural variability encountered in the production process. The types and numbers of samples in each set are described in Sections 2.2 and 2.3.

Prior to analysis all samples were ground to a particle size of 1 mm. The ground sample was spread on a sample holder as a continuous film for spectroscopic measurement. This procedure has been described elsewhere in detail [7,14].

2.2. Spectral library

The construction of the spectral library has been described in a previous work [7]. The library includes a total of 48 456 spectra measured on 227 samples representing 30 different ingredients

corresponding to the detailed list in Table 1. It includes the most common feed ingredients of different origins, the first 26 in the list, together with the most important banned ingredients, the last four in the list.

2.3. Mixtures

To assess the ability of the NIRM reference spectral library to identify ingredients in mixtures three sample sets were used. In all cases the samples, though sourced in the same way as the library samples, were external to the library.

Sample set A, which will be called “artificial mixtures” was made not by measuring spectra on mixtures, but by measuring samples of pure ingredients and constructing the artificial mixtures by combining appropriate numbers of spectra randomly selected from those measured on the pure ingredients. This set contains six samples, with the percentage ingredient contents shown in Table 1.

Sample set B, which will be called “synthetic mixtures” was produced by physically mixing pure samples in the laboratory and measuring spectra on the mixtures. This set, also shown in Table 1, contains six samples with similar percentage ingredient contents to those of sample set A.

Sample set C, which will be called “synthetic binary mixtures” was produced in the same way as sample set B. Each of the three samples in the set is a 50/50 mixture of meat and bone meal with one other ingredient. This other ingredient is barley in sample 1, maize in sample 2, and soya meal in sample 3. Spectra were measured not only on the three mixtures, but also on the four pure ingredients used to construct them. Sample set C was constructed in an attempt to understand why the system does not work well when the spectra are measured on mixtures, especially for the identification of animal by-products.

Table 2
Percentage ingredient contents and predictions for sample set A, artificial mixtures.

Artificial sample number	Ingredient	True composition (%)	Results	
			Average estimated composition (%)	SD
1	Maize	50.0	46.6	0.9
	Soybean	50.0	49.7	0.3
	Other	0.0	3.7	1.1
2	Maize	40.2	39.3	0.6
	Soybean	40.2	39.6	0.2
	Barley	19.7	20.0	0.5
	Other	0.0	1.1	0.5
3	Maize	32.5	31.6	1.2
	Soybean	32.5	32.0	0.4
	Barley	32.5	32.4	1.2
	Meat and bone meal	2.6	2.6	0.0
	Other	0.0	1.4	0.6
4	Maize	25.2	25.3	1.6
	Soybean	25.2	24.8	0.3
	Barley	35.0	35.5	1.5
	Wheat	9.8	7.3	0.6
	Meat and bone meal	4.7	4.7	0.0
	Other	0.0	2.4	0.5
5	Maize	25.2	25.6	1.2
	Soybean	25.2	23.6	1.1
	Barley	35.0	35.3	1.4
	Wheat	9.8	7.4	0.7
	Mineral correctors	1.7	1.6	0.2
	Meat and bone meal	3.0	3.0	0.0
	Other	0.0	3.6	1.3
6	Maize	25.2	25.5	1.6
	Soybean	25.2	23.2	1.1
	Barley	34.6	35.4	2.0
	Wheat	9.8	6.9	0.9
	Mineral correctors	2.1	2.0	0.2
	Beet pulp	0.9	0.9	0.3
	Meat and bone meal	2.1	2.1	0.0
	Other	0.0	4.0	1.4

2.4. Sample analysis and treatment of the NIRM spectra

Spectra were measured using an Auto Image Microscope connected to a Perkin-Elmer Spectrum One Fourier transform near infrared (FT-NIR) spectrometer in reflectance mode (1112–2500 nm), employing a resolution of 8 cm^{-1} . Spectra were obtained from the ratio between raw spectra and the background as measurement of the Spectralon plate and the spectral information was stored as $\log(1/R)$. This instrument is equipped with an InGaAs detector.

For each sample presented to the instrument, approximately 230 spectra (the number varies because of occasional bad spectra), each the average of 70 scans, were measured using fields of view of size $50 \mu\text{m} \times 50 \mu\text{m}$ arranged in a 13×18 grid over an area selected near the center of the sample. After referencing to a Spectralon standard and conversion to $\log(1/R)$ at 4 nm intervals, the spectra were transformed to first derivative using a Savitzky-Golay filter (The PLS Toolbox, version 3.5, Eigenvector Research, Manson, WA) with a window of 15 data points, and cut to a range of 1500–2448 nm. In the first derivative spectra of many of the ingredients there is a large peak centered on approximately 1900 nm. This peak is strongly influenced by the water band seen in the near infrared at around 1940 nm [16]. To reduce the influence of water content on the classification, the range from 1824 to 1976 nm was removed.

For mixture samples in the sets B and C 10 subsamples of each were measured as described above, to obtain information on the variability of the results. For the artificial samples in set A, 10 replicates were constructed by repeated random sampling of spectra from those measured on the pure samples, keeping the numbers of spectra from each ingredient fixed.

2.5. Data analysis

All three sets of mixture samples were analysed by comparing each spectrum in turn with all the spectra in the library, using a K -nearest neighbours (KNN) rule with $K = 10$ to classify the spectrum [7]. This simple algorithm finds the 10 spectra in the library that are closest to the unknown, and classifies according to the identity of these neighbours.

Each of the synthetic binary mixtures in set C was subjected to the following additional analysis. The first derivative spectra over the range 1500–2448 nm, without the removal of any peak, underwent an additional pre-treatment by SNV [17]. The combined set of spectra for the two pure ingredients and the mixture were subjected to a principal component analysis (PCA) and reduced to 20 scores. The scores for the two pure ingredients were then input to a linear discriminant analysis (LDA) and thus reduced to a single number for each spectrum, its score on the canonical variate from the LDA. Using the loadings, the canonical variate scores were calculated for the spectra from the mixture samples.

For the main computations the spectral data were exported in ASCII format from the Perkin-Elmer Spectrum v. 5.0113 software into Matlab v7.4 (The Mathworks, Inc., Natick, MA, USA), and the analyses carried out with programs specially written by one of the authors (T.F.). Coding the KNN algorithm directly is a simple task. The principal component analysis applied to sample set C employed Matlab's singular value decomposition.

To evaluate the results, the composition of each replicate was estimated using the proportions of individual spectra classified to each ingredient. Spectra classified to an ingredient not present in the mixture concerned were counted as 'other' for presentation in

Table 3
Repeatability of predictions for one artificial mixture.

Ingredient	True composition (%)	Estimated composition for each replicate (%)										SD	Coef. Var.
		1	2	3	4	5	6	7	8	9	10		
Maize	25.2	22.7	24.4	27.4	26.1	26.5	24.4	26.1	24.8	24.8	27.8	1.6	6.1
Soybean	25.2	21.8	23.9	22.7	23.5	24.4	23.9	24.4	23.9	21.8	21.8	1.1	4.7
Barley	34.6	39.3	37.6	34.6	34.6	32.9	36.3	34.6	35.0	35.9	32.9	2.0	5.6
Wheat	9.8	5.6	5.6	6.0	7.3	7.3	7.3	8.1	7.3	6.8	7.7	0.9	12.7
Mineral correctors	2.1	2.1	2.1	1.7	2.1	2.1	2.1	2.1	1.7	1.7	1.7	0.2	10.6
Beet pulp	0.9	0.9	0.4	1.3	0.9	1.3	0.9	1.3	0.9	0.9	0.9	0.3	28.4
Meat and bone meal	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	0.0	0.0

Table 4
Percentage ingredient contents and predictions for sample set B, synthetic mixtures.

Synthetic sample number	Ingredient	True composition (%)	Results	
			Average estimated composition (%)	SD
1	Maize	50.0	25.8	7.2
	Soybean	50.0	40.6	9.1
	Other	0.0	33.6	5.7
2	Maize	40.0	30.7	4.3
	Soybean	40.0	16.5	3.8
	Barley	20.0	25.4	4.7
	Other	0.0	27.4	6.3
3	Maize	32.3	33.3	6.4
	Soybean	32.3	13.3	3.4
	Barley	32.3	19.9	3.1
	Meat and bone meal	3.0	0.0	0.0
	Other	0.0	33.5	6.7
4	Maize	25.0	35.9	6.7
	Soybean	25.0	7.7	2.9
	Barley	35.0	31.8	4.6
	Wheat	10.0	0.1	0.2
	Meat and bone meal	5.0	0.0	0.0
	Other	0.0	24.6	4.9
5	Maize	25.0	36.4	7.7
	Soybean	25.0	6.0	2.6
	Barley	35.0	27.2	9.7
	Wheat	10.0	0.0	0.0
	Mineral correctors	2.0	0.5	0.9
	Meat and bone meal	3.0	0.0	0.0
	Other	0.0	29.9	9.6
6	Maize	25.0	37.0	5.9
	Soybean	25.0	4.8	2.1
	Barley	35.0	36.4	6.7
	Wheat	10.0	0.1	0.2
	Mineral correctors	2.0	0.4	0.4
	Beet pulp	1.0	1.6	1.1
	Meat and bone meal	2.0	0.0	0.0
	Other	0.0	19.7	5.0

Table 5
Percentage ingredient contents and predictions for sample set C, synthetic binary mixtures.

Synthetic sample number	Ingredient	True composition (%)	Results	
			Average estimated composition (%)	SD
1	Barley	50.0	4.4	2.2
	Meat and bone meal	50.0	0.0	0.0
	Other	0.0	95.6	11.4
2	Maize	50.0	67.6	4.8
	Meat and bone meal	50.0	0.3	0.4
	Other	0.0	32.0	5.9
3	Soybean meal	50.0	76.9	4.0
	Meat and bone meal	50.0	21.0	3.9
	Other	0.0	2.1	1.3

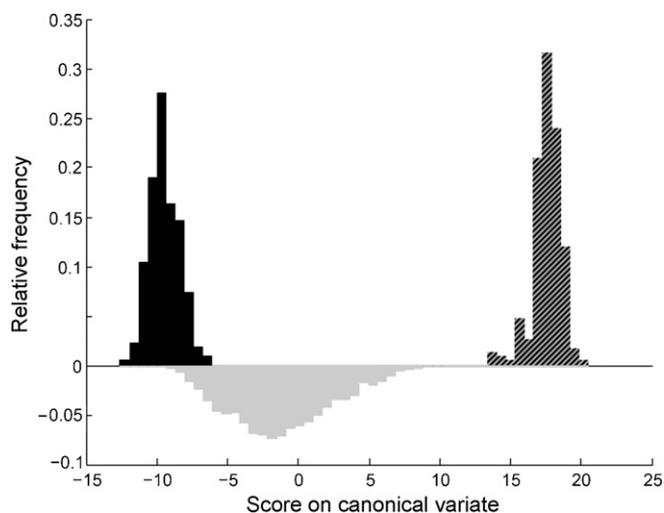


Fig. 1. Synthetic binary mixture of barley and MBM. Distributions of canonical scores of the spectra from the two pure ingredients (above the zero line, barley on the left, MBM on the right) and the spectra from the 50–50 mixture (below the line).

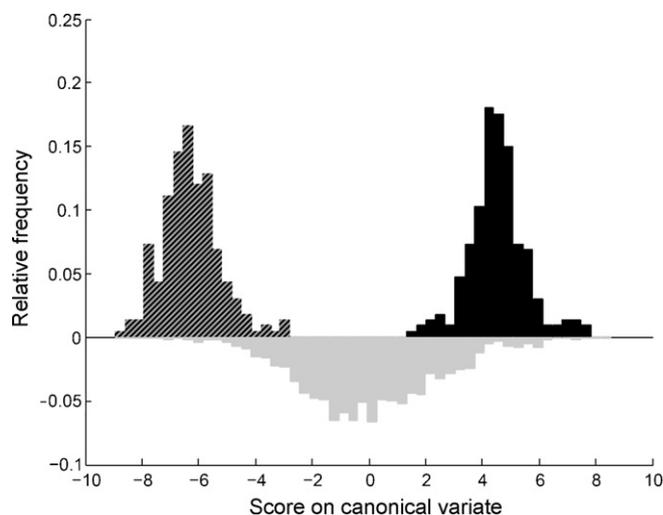


Fig. 2. Synthetic binary mixture of soya meal and MBM. Distributions of canonical scores of the spectra from the two pure ingredients (above the zero line, MBM on the left, soya on the right) and the spectra from the 50–50 mixture (below the line).

the tables of results. The mean and standard deviation over the 10 replicates were calculated for each ingredient and for the category ‘other’.

3. Results and discussion

3.1. Sample set A, artificial mixtures

Table 2 shows the true composition and the prediction results for the six artificial mixtures. The spectra of the animal ingredient, MBM, were always correctly classified, and there were no false positives. Even the cereal ingredients were generally correctly identified, despite the fact that there is some overlap between these ingredients in the library [7]. In Table 2, the classification ‘other’ corresponds to ingredients not present in the sample but included in the estimated composition. These were mainly confusions with other cereals. The standard deviations over the 10 replicates ranged from 0 to 2%. Table 3 shows an example of this repeatability in detail for the sixth artificial mixture. For the minor ingredients the fact that the result is a count of spectra is evident. There are exactly two

beet pulp spectra in each mixture, and the number found varies from one to three, hence the large coefficient of variation for this ingredient.

These samples are not a very stringent test of the system, being collections of spectra of pure ingredients rather than spectra measured on mixtures of ingredients. However their use confirms that the library is able to classify accurately such spectra, and enables an assessment of accuracy that is free of sampling variability. When a real mixture is measured, there is considerable uncertainty about the composition of the portion of the sample actually examined, especially for ingredients present in only small proportions in the bulk sample.

3.2. Sample set B, synthetic mixtures

The composition and prediction results for sample set B, the synthetic mixtures are displayed in Table 4. The results are very disappointing when compared with those for the artificial samples in Table 2. There are major confusions among the cereal ingredients, for example the wheat in samples 4–6 was almost undetected. The

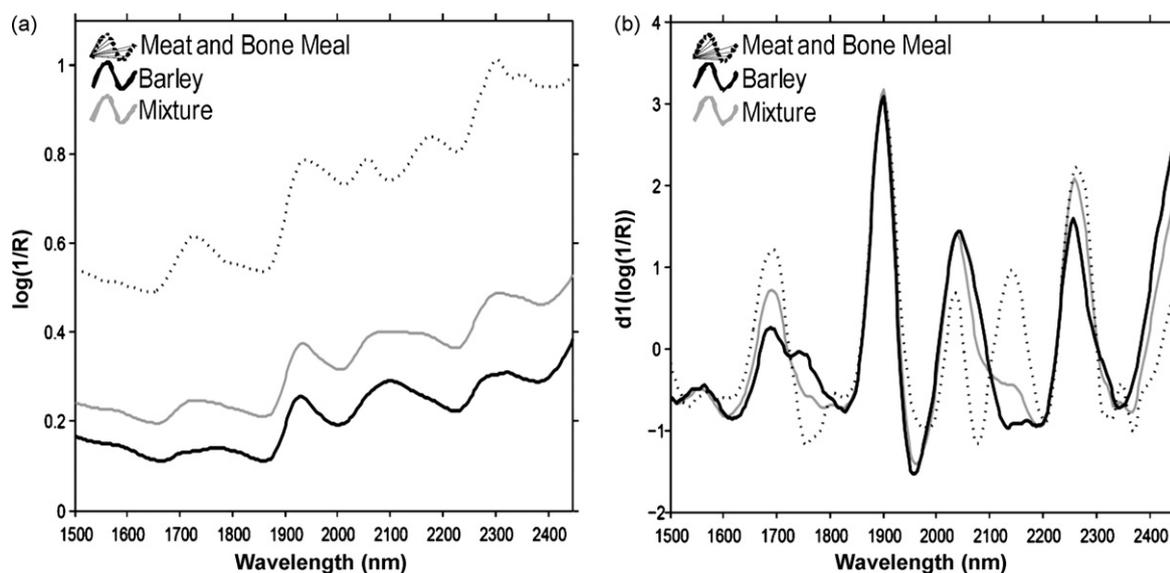


Fig. 3. Mean spectra for barley, MBM, and their mixture: (a) raw $\log(1/R)$ and (b) snv-treated first derivative.

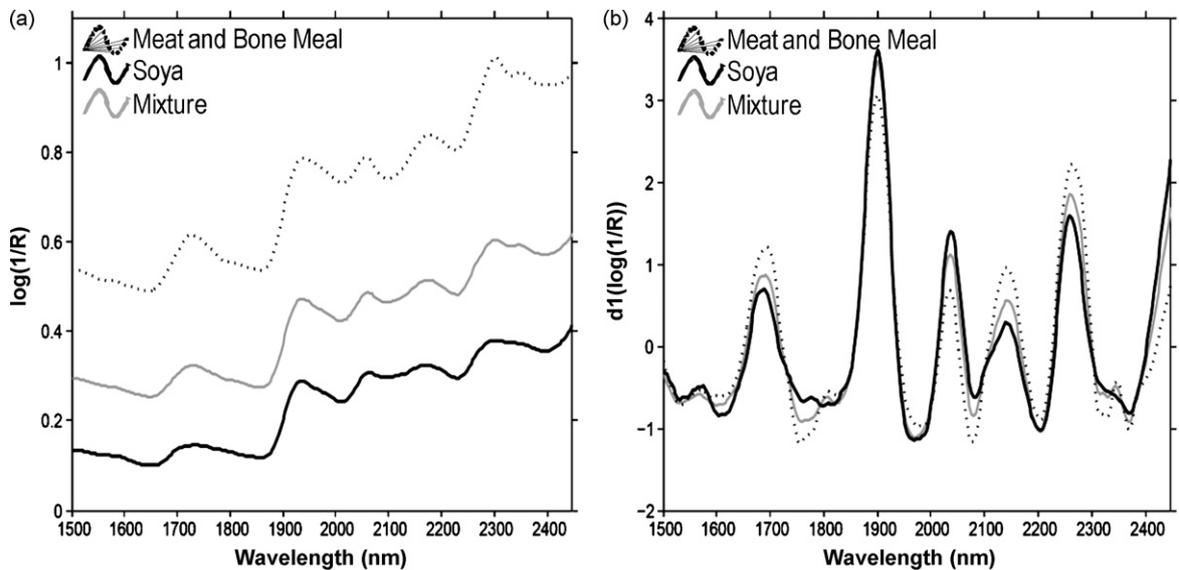


Fig. 4. Mean spectra for soya, MBM, and their mixture: (a) raw $\log(1/R)$ and (b) snv-treated first derivative.

main contributors to the 'other' category were oats and bran, in all the samples, and barley in sample 1. None of the four banned animal ingredients appeared in this category. Nevertheless, these confusions between cereal grains are not particularly important. In fact, the mandatory labelling for feed materials according to Article 16 (1) of Proposal for regulation of the European Parliament (2008/005 COD) says that the label shall include the name of the feed material corresponding to the respective category. Cereal grains are the category that includes oats, wheat and barley. Worse, though, is the failure to detect the MBM in any of samples 3–6, even though it is present at 5% in sample 4.

3.3. Sample set C, synthetic binary mixtures

The results for the three binary mixtures of sample set C are shown in Table 5. They are no better than those for sample set B, with the MBM being misclassified completely in sample 1, and almost completely in sample 2. Only in sample 3, a mixture of soya and MBM, does comparison with the library detect the correct two ingredients, and even in this case the estimated proportions are incorrect. The main contributors to the 'other' category are maize, soya and DDGS barley in sample 1; and soya in sample 2.

Some understanding of the spectral behaviour of the mixtures can be obtained by comparing the spectra from the mixtures with the spectra of the pure ingredients they contain. Figs. 1–4 show such a comparison for samples 1 and 3, the barley–MBM and soya–MBM mixtures. The case of sample 2, the maize–MBM mixture is very similar to that of sample 1, and will not be discussed in detail.

Figs. 1 and 2 show scores on the canonical variates for binary discrimination in the case of samples 1 and 3 respectively. The canonical variate is the direction in spectral space that best discriminates between the two ingredients. These scores are shown as relative frequency histograms for each of the three sets of spectra. The histogram for the mixture points downwards merely so that it can be distinguished from the other two where it overlaps.

The most important conclusion that can be drawn from these two figures is that in the mixture, despite the very small sample area that an individual spectrum represents, most if not all of the individual spectra are spectra of mixtures, of varying proportions. Some spectra may resemble those of the pure ingredients, and thus be correctly classified, but the majority will not, and their correct classification using the KNN rule will be a matter of chance. This

explains the poor results obtained when classifying the real mixtures.

In neither case is the system quite linear, though it is much closer to being so for the soya–MBM mixture than for the barley–MBM mixture. In Fig. 1, the scores from the mixture have a distribution much closer to those of the barley than those of the MBM, despite the equal proportions in the mixture. In Fig. 2, although the separation between the two ingredients is much less, the mixture is much more symmetrically placed between them, with a slight bias in the direction of the soya, on the right of the figure. This fits with the failure to identify any MBM in sample 1 and with the too-high proportion of soya predicted for sample 3.

The source of the nonlinearity is in the spectra. Figs. 3 and 4 show the mean raw and SNV-treated first derivative spectra for the pure ingredients and the mixture in each of the two cases. In both panels of Fig. 3 it can be seen that the mean spectrum from the mixture, although between those of the two pure ingredients, is generally closer to that of the barley. This is especially true in the region around 2000–2200 nm. In Fig. 4, the mean mixture spectrum is roughly mid-way between the other two for most of the

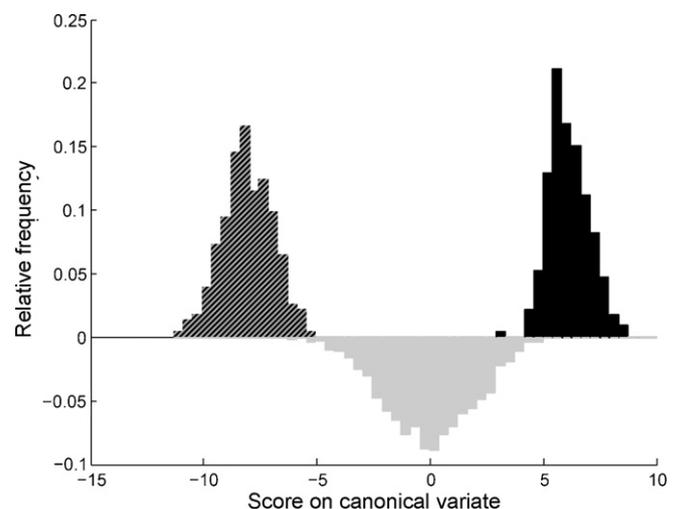


Fig. 5. Synthetic binary mixture of barley and MBM. Distributions of canonical scores of the spectra from the two pure ingredients (above the zero line, barley on the left, MBM on the right) and the spectra from the 50–50 mixture (below the line), with the spectral range restricted to 1600–1800 nm.

spectral range, especially for the derivative spectra. The positions of the mean mixture spectra explain the behaviour seen in Figs. 1 and 2, and suggest a way of making the barley–MBM system more linear. Fig. 5 is the result of carrying out the same analysis that gave rise to Fig. 1, but restricting the spectral range to 1600–1800 nm. In this region the mean mixture spectrum in Fig. 3 does resemble an average of the pure ingredient spectra, and as expected the scores for the mixture spectra have a much more symmetric and centrally situated distribution in Fig. 5.

Given the problems that arise when mixtures are measured with the methodology used here, it would be tempting to conclude that only the measurement of individual particles offers a way forward. However the sample presentation and measurement procedures used in this work offer advantages of simplicity and speed, and the results with the binary mixtures of set C are encouraging enough to suggest that further work to develop chemometric strategies to deal with the problem is a worthwhile prospect.

4. Conclusions

The artificial samples of set A confirm that the spectral library is very good at identifying spectra taken from pure ingredients. However, these excellent results were not repeated for sets B and C, in which the spectra were measured on mixtures. The explanation for this is clear from the investigation of sample set C. Even though the sample area from which the spectra are taken was small, the samples are so well mixed that few if any of the resulting spectra correspond to pure ingredients. This means that a strategy that tries to match the spectra taken from the mixture with those of pure ingredients is unlikely to meet with much success. The evidence from sample set C is that interpolation between pure ingredients is possible, and that the choice of spectral ranges is likely to be important in achieving this. Much work remains to be done to extend this system to mixtures of larger numbers of ingredients, and hence solve the general problem.

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