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Contribution of vibrational spectroscopy to the characterisation and detection of insect meal in compound feed

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ABSTRACT

Insect-based products, such as insect meals, have experienced significant growth in recent years due to their composition, making them excellent substitutes for commonly used ingredients in feed. Currently, only eight insect species are authorised for use in feed, and their nutritional value varies depending on species, developmental stages, and diet. To ensure nutritional quality and compliance with current legislation, it is essential to develop tools to monitor insect-based products. In this context, two Near Infrared (NIR) spectroscopy techniques have been developed to determine the chemical composition of various insect meals and to distinguish and detect them in compound feeds. First, NIR Spectroscopy (NIRS) allowed for the assessment of protein, lipid, and moisture contents in several insect products, achieving coefficients of determination greater than 0.95 and low cross-validation errors. Secondly, NIR Microscopy (NIRM) enabled the differentiation of insect products from plant and animal products and was also able to detect the presence of insect meals at low inclusion rates in feed. These results suggest that NIR spectroscopy techniques can be effective, non-destructive tools for the characterisation, differentiation, and detection of insect-based products.

HIGHLIGHTS

- NIRS coupled with PLS regression can predict the protein, humidity and fat content of insect-based products.
- NIRM coupled with qualitative analysis can discriminate insects from other ingredients and detect insect meal in small quantities within a matrix.
- Results are of interest for the development of monitoring tools for insect-based products.

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Introduction

The search for new sources of animal proteins as ingredients for feed is a constant concern in the animal production sector. In this regard, insect meals are considered an interesting alternative, particularly in the current context of decreasing availability of certain protein sources and the search for ingredients that are more compatible with a more sustainable and environmentally friendly production. Insect meals are ingredients with high nutritional value, known for their effectiveness in animal production while being seen as having a low environmental footprint (van Huis and Oonincx 2017). Insect meals are a source of proteins containing essential amino acids in animal production, as well as lipids, vitamins, and minerals (Sánchez-Muros et al. 2014; De Marco et al. 2015; Veldkamp and Bosch 2015; Barragan-Fonseca et al. 2017; Kröncke and Benning 2022). Due to their composition, insect meals are excellent substitutes for ingredients such as soybean and fish meals in the breeding of pigs, poultry, and fish (Sánchez-Muros et al. 2014). Environmental impact-wise, compared to ingredients of similar nutritional interest, insect meals have a reduced impact because their production is not land-intensive, requires less water consumption, has low greenhouse gas emissions, and allows for the valorisation of recycled organic substrates or those not fit for food production (FAO 2013).

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Their composition varies according to the insect species, developmental stage, diet, and production type (Sánchez-Muros et al. 2014; Pinotti and Ottoboni 2021). Products derived from insects generally come in the form of powders with varying particle sizes obtained through technological processing involving cleaning, drying, and grinding steps (Oonincx and Finke 2021). Different insect species can be used for meal production. However, European legislation only permits eight species for feed and feed meal production: *Acheta domesticus* (Orthoptera, Gryllidae), *Alphitobius diaperinus* (Coleoptera, Tenebrionidae), *Bombyx mori* (Lepidoptera, Bombycidae), *Gryllodes sigillatus* (Orthoptera, Gryllidae), *Gryllus assimilis* (Orthoptera, Gryllidae), *Hermetia illucens* (Diptera, Stratiomyidae), *Musca domestica* (Diptera, Muscidae), and *Tenebrio molitor* (Coleoptera, Tenebrionidae) (European Commission 2017, 2021; Anselmo et al. 2023). In practice, black soldier fly larvae (*H. illucens*), house fly (*M. domestica*) and yellow mealworm (*T. molitor*) have received the most attention in the feed sector, thanks to their ability to feed on a wide range of substrates (Chia et al. 2019).

The production and use of insect meals as ingredients in animal production require the development, validation, and implementation of analytical tools to ensure nutritional quality, compliance with regulatory requirements, and traceability. Therefore, it is important to have fast and reliable tools for monitoring insect meals at all stages of their production and use. Currently, analyses are primarily conducted using methods based on classical chemical techniques and molecular biology (European Commission 2013, 2022). However, various initiatives are being taken to develop rapid, multi-parameter analytical methods that allow for online analysis, have low operational costs, use little or no chemical reagents (green analytical techniques), and require minimal sample preparation. Among these, methods based on Near Infrared spectroscopy (NIRS) and multivariate analysis seem particularly suitable for ensuring the quality, safety, and traceability of insect meals. It should be noted that these methods require calibration and validation steps to build robust predictive models and a maintenance strategy for them.

This article aims to present recent developments in the implementation of a NIRS method for the characterisation of insect-based products, as well as a Near Infrared Microscopy (NIRM) method for the detection of different meal ingredients and the discrimination of types of insect meals.

Materials and methods

Near Infrared spectroscopy analyses

Spectra collection

Different kinds of insect-based samples namely dried and ground insects (larva or adult stages), and insect-based products (diverse processed products from both food and feed industry) were used to build an extensive database. *Tenebrio molitor* species was highly represented in different forms in the database, jointly with other kinds of species such as *Hermetia illucens*, *Acheta domesticus*, *Gryllodes sigillatus*, *Gryllus assimilis* and *Alphitobius diaperinus*. A total of 103 samples were studied. All samples were scanned using an XDS spectrophotometer (FOSS NIRSystems, Inc., USA) covering the VIS and NIR ranges from 408 nm to 2498 nm. All samples were scanned twice and the spectra were averaged for statistical analyses.

Reference analysis

The reference values (wet chemistry analyses) were realised to determine the dry matter, protein and fat contents of the sample. Dry matter was realised by oven drying at 103 °C for 4 h. Nitrogen was determined by the Kjeldahl method, with a correction 6.25 to express nitrogen as protein content. Finally, for fat content, gravimetric extraction was performed with Soxtec™ 2055 (FOSS Denmark), with petroleum ether.

Preprocessing and chemometric analyses

Spectra and reference values were used to calibrate spectroscopic models using WinISI software (Infrasoft International LLC, USA) using spectral range 1100–2498 nm. Pre-treatment (SNV Detrend) was applied to the raw spectra and Partial Least Squares (PLS) regression technique was used to build the calibration models.

To evaluate the predictive potential of NIR models, the database was split into two sets: a calibration set (to develop the calibration models) of 80 samples and a validation set (to test the performances of the models) containing 23 samples. The partition was realised according to the protein, fat and fibre contents to have two sets with the same variability. The accuracy of the calibration models was evaluated based on the coefficient of determination of calibration (R^2c), root mean square error of calibration (RMSEC), the coefficient of determination of cross-validation (R^2cv) and the root mean square error of cross-validation (RMSECV). For the validation step, the accuracy was evaluated based on the coefficient of determination of prediction (R^2p) and the root mean square error of

prediction (RMSEP). The samples included in the validation set are partially independent as they differ from the samples included in the calibration. However, some samples may come from the same batches or same providers. Ratios of performance to deviation (RPD) were also calculated for both calibration and validation steps, which is defined as the ratio between the standard deviation and the root mean square error of the calibration or validation set.

Near Infra-red Microscope analyses

NIR spectra were collected on a Near Infrared Microscope (NIRM) consisting of a Hyperion 3000 microscope connected to a Fourier Transform (FT) near infrared spectrometer (Bruker Belgium SA, Kontich, Belgium). The instrument was linked to a camera and used the OPUS 7.5 software (Bruker Belgium SA, Kontich, Belgium) to analyse and extract spectra.

For the analysis of the aluminium plate containing the different types of samples (Figure 3(A)), a 200×199 mapping with a 200 µm pitch was carried out to analyse the whole surface of the plate. Spectra were collected in the range 9000 cm^{-1} to 4000 cm^{-1} (1111 nm to 2500 nm) with a resolution of 8 cm^{-1} and 16 co-added scans by spectra. A total of 39,800 spectra were taken, covering the whole surface of the aluminium plate and including spectra associated with each sample analysed. The final spectra were the results of the ratio between the raw spectrum and the background consisting of aluminium.

For adulterated samples, each pure sample, i.e. the ruminant feed and the *H. illucens* larvae meal, was placed in a well of a multi-well aluminium plate (Bruker Belgium SA, Kontich, Belgium) and analysed by 10×10 mapping with an automatic pitch. The adulterated samples were spread into three replicates on an aluminium slide, to obtain a thin layer facilitating detection of the adulterant product and analysed by 40×40 mapping with a 300 µm pitch. For this analysis, spectra were also collected in the range 9000 cm^{-1} to 4000 cm^{-1} (1111 nm to 2500 nm), with a resolution of 16 cm^{-1}, and 8 co-added scans by spectra. The final spectra were the result of the ratio between the raw spectrum and the background consisting of a gold well provided on the multi-well plate.

Preprocessing and chemometric analyses

Image treatment and analysis. Representative spectra of the different products of insects (*G. assimilis* and *H. illucens*), wheat bran, frass and bovine processed animal proteins (PAPs) were extracted from the image. The extracted spectra were used to calibrate a PLS-DA model discriminating each group, i.e. the insects, the wheat bran, the frass and the bovine PAP. Then, the whole image was predicted by applying the model on each individual pixel.

An alternative discrimination method was also tested by applying a decision rule based on the absorbance values at different wavelengths, i.e. 1944, 2060 and 2148 nm of the first and second derivative spectra (Baeten et al. 2005; von Holst et al. 2008). This equation is used to assess whether or not particles belong to the animal group. So, if Absorbance (1944) + Absorbance (2148)/2 > Absorbance (2060), the particle is classified as being of animal origin.

The spectral ratio was computed for each individual pixel of the image and the animal protein pixels were detected based on the threshold value.

Adulterated samples analysis. All NIRM spectra are pre-processed to remove the noise, the scattering effect and to facilitate the visualisation of the sample differences (Engel et al. 2013). Firstly, the Savitzky-Golay method with a first order derivative is applied to reduce baseline and background effect (Engel et al. 2013). Secondly, the standard normal variate (SNV) is used to remove variability in the reflectance spectra related to scattering effects (Engel et al. 2013).

All chemometric analyses are carried out using Solo 9.2.1 (2023) and the PLS toolbox plug-in included (Eigenvector Research, Inc., Manson, WA, USA 98831).

Since two levels of adulteration are studied, two separate chemometric analyses, based on the same approach, are carried out. To start with, 100 spectra of pure meal samples of *H. illucens* larvae meal and ruminant feed are measured per sample and used to create a calibration set. For the adulterated samples, 4800 spectra are collected.

Then, to assess the NIRM's ability to detect the presence of insect meals in a feed at a low level of adulteration, a supervised Partial Least Squares Discriminant Analysis (PLS-DA) (Barker and Rayens 2003) is performed using *k*-fold ($k = 10$) venetian blinds cross-validation (CV). During CV, the same number of latent variables (LVs) is used for the two single-class models (ruminant feed or *H. illucens* larvae meal) to only include the variability of interest for the prediction, and the selected number of LVs is the one that minimised the classification error for the whole – multi-class – approach. As two different levels of adulteration are tested, two separate PLS-DA models are built to obtain optimal LVs for each level of adulteration.

Discrimination results are assessed using sensitivity and specificity. The sensitivity refers to the ability of the model to correctly identify samples. It measures the ratio of true positive (TP) predictions to actual positive instances including false negative (FN) predictions (Westerhuis et al. 2008) (1).

$$\text{Sensitivity} = \text{TP}/(\text{TP} + \text{FN}) \quad (1)$$

Specificity refers to the ability of the model to correctly identify samples that are not of this class. It measures the ratio of true negative (TN) predictions to actual negative instances, including false positive (FP) predictions (Westerhuis et al. 2008) (2).

$$\text{Specificity} = \text{TN}/(\text{FP} + \text{TN}) \quad (2)$$

Sensitivity and specificity values vary between 0 and 1, with 1 being the optimum value for a prediction model. For each multi-class PLS-DA analysis, a confusion matrix is provided. It compares the actual class with the one predicted by the model.

Results and discussion

Near Infra-red spectroscopy analyses

NIR spectra

A large spectral variability was observed due to the high diversity between insect-based products used in this study as shown in Figure 1. Variability can be observed in different NIR regions: between 1150 and 1250 nm (second overtone region), between 1650 and 1800 nm (first overtone region), between 1900 nm and 2400 nm (combination regions). As the database contains several samples of dried and ground *Tenebrio molitor* larvae, their spectra were averaged to better highlight the NIR bands of this species (Figure 2). So, seven bands can be identified at wavelengths about 1204, 1724, 1760, 2058, 2174, 2306 and 2348 nm. Bands about 1204, 1724, 1760 nm are related to fat content. Kröncke and Benning (2022) also highlighted bands corresponding to lipids at 1200, 1726 and 1785 nm. The slight band at 1940 nm is related to moisture content. Kröncke and Benning (2022) showed major bands at 1450 and 1950 nm corresponding to water bands, which were not so visible in our study as dried and ground larvae were scanned here. The absorption bands at 2058 nm and at 2174 nm correspond to combination bands and are probably related to proteins since according to Bertrand and Dufour (2006), bands around 2050 nm and 2180 nm characterised peptide liaison. Bands at 2306 and 2348 nm are in a region where C-H combination bands are observed. In particular, bands at 2304 and 2348 have been related to specific combination bands of lipids (Bertrand and Dufour 2006).

Calibration results

The data were split into a calibration set of 80 samples and a validation set of 23 samples. As not all samples were characterised for fat content, only 59 samples and 17 samples constituted the calibration and validation set respectively for this parameter. The sets were established to have as much as possible the same variability. However, the calibration samples were chosen to contain the maximum variability (extreme reference values). The number of samples, mean and standard deviation of both sets are shown in Table 1.

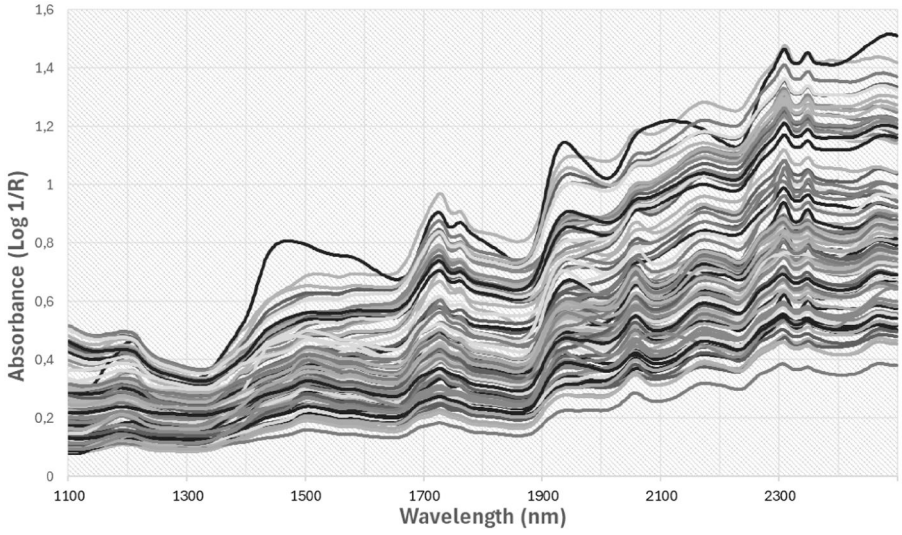

Figure 1. NIR raw spectra of the insect-based products contained in the database.

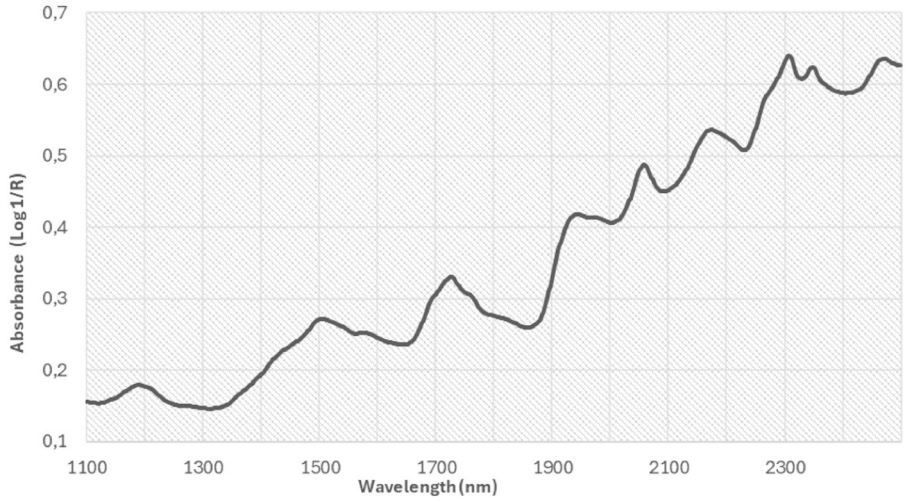

Figure 2. NIR raw spectra of *Tenebrio molitor* larvae.

Calibration set was used to develop models using PLS regression on pre-treated spectra. Performances of the calibration models are presented in Table 2. For all parameters, some outliers (based on GH distances and *t* criteria) were removed during the development of the equations, so that the number of samples in the calibration models differed from the calibration set one. Coefficients of determination superior to 0.95 and relatively low errors of cross-validation (SECV) were observed for all parameters. High values of RPD (higher than 3) were obtained indicating good performances of the models.

To evaluate their performances, the samples of the validation set were predicted using the calibration models. After the removal of one outlier (based on *t* criteria), validation results showed similar values of coefficient of determination compared to the calibration set (Table 3). The Standard Errors of Prediction (SEP) were similar, or slightly higher, to the errors of Cross-Validation (SECV). High values of RPD (higher than 3) were also obtained for the validation. Therefore, results suggested that NIR spectroscopy can be used to determine accurately these chemical compounds in insect-based products.

These results may be compared to the study of Kröncke and Benning (2022). They analysed different preprocessings of the spectra and showed that the best calibration models were obtained using the 1st derivative spectra of mealworm larvae. They attained very low errors of prediction for protein content (RMSEP = 0.51) and for moisture content (RMSEP = 0.46). Compared to our study, the error of prediction for protein was three times lower. Regarding the fat content prediction, a very low error or prediction (RMSEP = 0.28) was attained by Kröncke et al. (2023) which is again several times lower than the error obtained in this study. The differences of accuracy may be explained by the different set up of both studies and the very different material investigated. Indeed Kröncke and Benning (2022) focused on larvae of *T. molitor* grown in specific controlled conditions whereas this study aimed to build a very large database of diversified insect-based products.

In this study, calibration models were built for the total protein content. However, it should be noted that the protein content was estimated based on the nitrogen content combined with a conversion factor (6.25). It is likely that the estimated protein content is biassed or misestimated by the chitin contained in the cuticula of the insects. Indeed, chitin is composed of nitrogen which has also been converted into protein in this case. Further studies should be investigated to determine the chitin content chemically and to investigate the potential of NIR to determine this compound specifically.

Distinction of multiple ingredients by NIRM

The image obtained following the NIRM analysis is a 'pseudo-image'. The NIRM does not provide an RGB image composed of pixels, but creates a 'false' image from points, corresponding to the location of the 39,800 spectra taken during the analysis. The OPUS 7.5 software automatically calculates the total area under the spectra and assigns a colour according to its value. The more information contained in the spectrum, the redder the assigned colour (Figure 3(B)). This pseudo-image was converted into pixels to perform the different chemometric analysis.

First, a PLS-DA analysis was applied on the spectra. The results of this analysis are given in pixel form in Figure 3(C) and in numerical form in the confusion

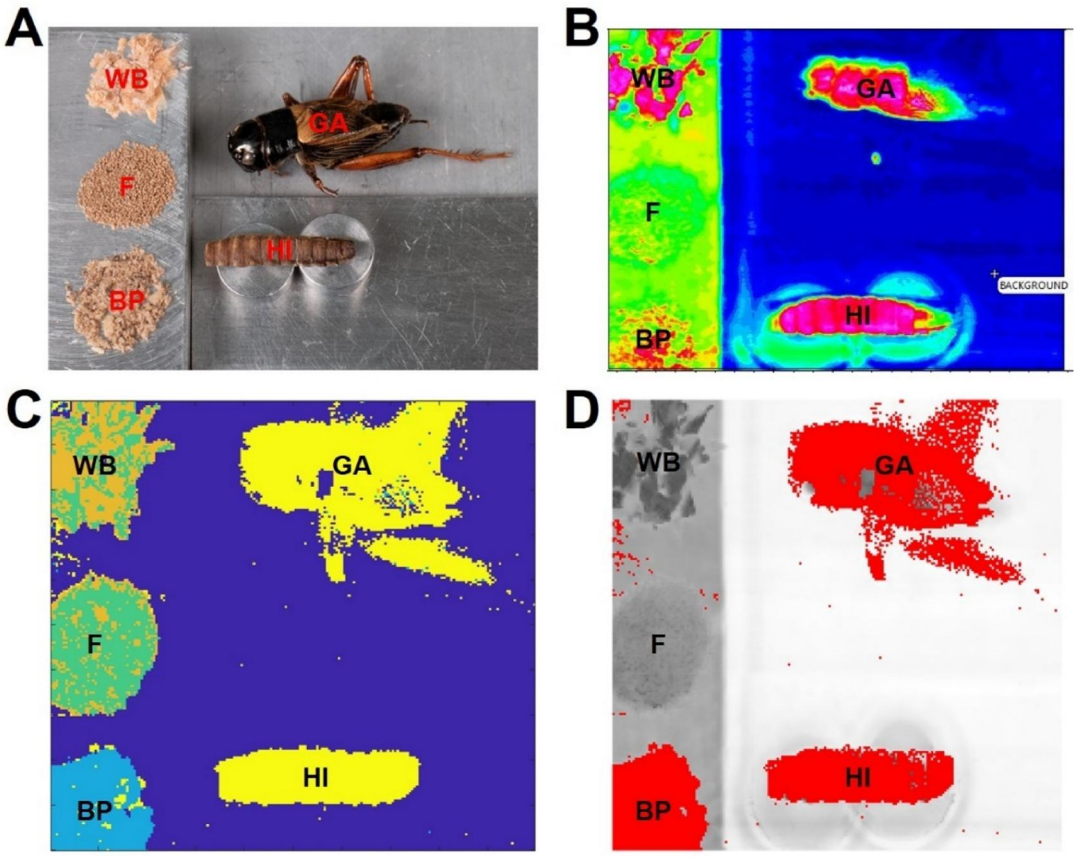

Figure 3. Photograph (a), spectral image (B), PLS-DA image processing (C) and absorbance equation image processing (D) of different samples analysed by NIRM. WB: Wheat bran; F: Frass; BP: Bovine PAP; GA: *Gryllus assimilis*; HI: *Hermetia illucens* larvae.

matrix presented in Table 4. In view of the confusion matrix, it appears that insects and bovine PAP are the classes for which the spectra were most accurately predicted, with very few confusions between these ingredients and the others. On the other hand, there was a significant amount of confusion between frass and wheat bran. This result is not really surprising since frass is defined as a mixture of insect excrement, food substrate, insect parts and dead eggs (European Commission 2021). Therefore, it is quite logical to find traces of plants in frass, and consequently particles predicted as such. Furthermore, this result could also be due to confusion between the two types of matrices. Given that frass is ultimately a by-product of the substrate and therefore of wheat bran, it is highly probable that their chemical composition is similar, leading to confusion between frass and wheat bran. However, 278 spectra were still predicted as frass, which could also mean that frass may have its own spectral signature.

Secondly, instead of a classical PLS-DA analysis, the equation described by Baeten et al. (2005) was applied to the spectra generated by the NIRM and a new image was created. The result is shown in Figure 3(D). Most of the spectra identified as animal are from the two insect species and the bovine PAP. This result is in line with what would be expected from the use of this equation, since it is used to classify the spectra according to whether they belong to the animal group or not. In fact, in the spectral image, only the *G. assimilis*, the *H. illucens* larvae and the bovine PAP are animal samples.

Once again, using this equation, it appears that there are no spectra classified as animal in the frass sample, despite it being derived from insect rearing. It is therefore quite probable that this type of sample is sufficiently degraded by insects that, for example, no potential traces of moulting remain.

Detection of low levels of insect meal in a ruminant feed using NIRM

The detection of low levels of insect meal in a ruminant feed was carried out using 4800 spectra obtained by NIRM analysis. The PLS-DA model to analyse the ruminant feed adulterated with 1% of *H. illucens* larvae meal included 8 latent variables. Three ruminant feed spectra are considered outliers as they did not fit the

Table 1. Humidity (HUM), protein (MPT), and fat (FAT) content (expressed in %) for calibration and validation sets.

	Calibration set			Validation set		
	HUM (% as_is)	MPT (% as_is)	FAT (% as_is)	HUM (% as_is)	MPT (% as_is)	FAT (% as_is)
N	80	80	58	23	23	17
Mean	4.66	60.98	14.32	4.87	60.27	15.94
Minimum	1.5	16.59	1.56	1.5	18.81	4.56
Maximum	10.18	75.80	40.96	8.21	73.60	32.78
SD	1.94	12.13	7.71	1.76	13.48	8.00

N: Number of samples; *SD*: Standard Deviation.

Table 2. Calibration models for humidity (HUM), protein (MPT), fat (FAT), content (expressed in %) obtained using PLS regression.

Constituent	*N*	Mean	*SD*	SEC	R^2c	SECV	R^2cv	RPD
HUM (% as_is)	74	4.48	1.76	0.24	0.98	0.32	0.96	5.5
MPT (% as_is)	71	62.75	9.65	0.95	0.99	1.52	0.97	6.3
FAT (% as_is)	51	13.67	6.07	0.49	0.99	0.97	0.97	6.2

N: Number of samples in the calibration; *SD*: Standard Deviation; SEC: Standard Error of Calibration; R^2c: Coefficient of Determination of the Calibration; SECV: Standard Error of Cross-Validation; R^2cv: Coefficient of Determination of Cross-Validation; RPD: Ratio of Performance Deviation = *SD*/SECV.

Table 3. Validation results for the humidity (HUM), protein (MPT), fat (FAT), cellulose (CELL) and chitin (ADF-ADL) content (expressed in %) obtained using PLS regression.

	N Val	SEP	R^2p	RPD
HUM (% as_is)	22	0.44	0.94	4.1
MPT (% as_is)	22	1.79	0.97	5.7
FAT (% as_is)	16	1.64	0.96	5.1

N Val: Number of samples in the validation; SEP: Standard Error of Prediction; R^2p: Coefficient of Determination of Prediction; RPD: Ratio of Performance Deviation = *SD*/SEP.

Table 4. Confusion matrix of results obtained by PLS-DA for the analysis of the image by NIRM: Near Infrared Microscopy.

	Actual class			
Predicted class	Bovine PAP	Frass	Wheat bran	Insects
Bovine PAP	182	0	0	7
Frass	0	278	134	0
Wheat bran	0	47	238	2
Insects	7	0	0	1035

model and are therefore removed. These outliers are mainly generated by the fact that the spectrum taken is located at the edge of the well and therefore included part of the background.

Classification results are given in Table 5. Concerning the calibration set, the classification is free of errors. Following application of the model to the sample adulterated with 1% *H. illucens* larvae meal, 12 spectra are predicted as potentially *H. illucens* larvae meal and 4788 as ruminant feed. We can deduce that 0.25% of our adulterated sample is classified as *H. illucens* larvae meal, which is lower than the percentage added. This may be related to the composition of our sample and the possibility that the 1% of added insects may be partially covered by other particles. In addition, these data remain predictions, and it is therefore possible that in the 4788 spectra identified as ruminant feed there are spectra corresponding in reality to insects.

For the ruminant feed adulterated with 0.5% of *H. illucens* larvae meal, The PLS-DA model included 7 latent variables. For the same reasons as mentioned above, two *H. illucens* larvae meal spectra are considered outliers and are removed.

Classification results are given in Table 6. Concerning the calibration set, the classification is also free of errors. Regarding the application of the model on the ruminant feed adulterated with 0.5% of *H. illucens* larvae meal, only 3 are predicted as *H. illucens* larvae meal and 4797 are predicted as ruminant feed. In this case, we can deduce that only 0.06% of the spectra of the adulterated sample are classified as *H. illucens* larvae meal. This result can be explained in the same way as for the result obtained with 1% of insect meal.

The results obtained both with ruminant feed adulterated with 1% *H. illucens* larvae meal and with that adulterated with 0.5% tend to demonstrate that the NIRM is capable of detecting adulteration at low concentrations. It is also found that when the adulteration rate is reduced, the number of spectra detected as being *H. illucens* larvae meal decreased, which is perfectly logical. In fact, the rate of adulteration between the two samples is divided by two but the number of spectra analysed remained the same. To detect more spectra of the adulterant, the total number of spectra analysed would have had to be increased.

These results are also in line with those already demonstrated in the literature using other NIR techniques. As an example, the article by Alagappan et al. (2024) demonstrated both the ability of vibrational spectroscopy techniques to detect adulteration of

Table 5. Confusion matrix of calibration set and performance parameters obtained by PLS-DA for the ruminant feed adulterated with 1% of *H. illucens* larvae meal analysed by NIRM.

	Actual class	
Predicted class	*H. illucens* larvae meal	Ruminant feed
Calibration		
H. illucens larvae meal	100	0
Ruminant feed	0	97
Sensitivity	1	1
Specificity	1	1

Table 6. Confusion matrix of calibration set and performance parameters obtained by PLS-DA for the ruminant feed adulterated with 0.5% of *H. illucens* larvae meal analysed by NIRM.

Predicted class	Actual class	
	H. illucens larvae meal	Ruminant feed
Calibration		
H. illucens larvae meal	98	0
Ruminant feed	0	100
Sensitivity	1	1
Specificity	1	1

ingredients with insects, and also the matrix effect on NIR analyses.

In the case of contamination, increasing the number of spectra analysed will be crucial. In fact, although in this study NIRM analysis enables adulterants to be detected at low concentrations, these concentrations could potentially not fully reflect the levels of contamination that can be found on the market. A complementary study needs to be carried out using even lower levels of adulteration to make NIRM a reliable method for detecting contaminants in feed.

Conclusions

These studies on the use of NIR spectroscopy to evaluate the chemical composition of insect-based samples shows promising results. Indeed, the results suggest that NIRS coupled with simple PLS regression constitutes a good tool for the prediction of humidity, protein and fat content with a relatively low error of prediction. It should be noted that NIRS models were developed using relatively small data sets, as the available data (spectra and reference values of insect-based samples) were quite limited. In the future, the database should be fed with new samples to increase its variability and improve the predictive performances and develop models to determine the chitin content.

On the other hand, the results obtained by NIRM analysis have demonstrated the ability of this technique to distinguish between several types of products and to detect the presence of ingredients in low concentrations in a feedstuff. Further analysis is still required to determine the extent to which this technique can differentiate between different product categories, and to detect even lower levels of adulteration in other types of matrices.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

Data availability statement

Due to the nature of the research and the commercial supporting, data are not available.

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