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Authentication of Organic Feed by Near-Infrared Spectroscopy Combined with Chemometrics: A Feasibility Study

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Supporting Information

ABSTRACT: Organic products tend to retail at a higher price than their conventional counterparts, which makes them susceptible to fraud. In this study we evaluate the application of near-infrared spectroscopy (NIRS) as a rapid, cost-effective method to verify the organic identity of feed for laying hens. For this purpose a total of 36 organic and 60 conventional feed samples from The Netherlands were measured by NIRS. A binary classification model (organic vs conventional feed) was developed using partial least squares discriminant analysis. Models were developed using five different data preprocessing techniques, which were externally validated by a stratified random resampling strategy using 1000 realizations. Spectral regions related to the protein and fat content were among the most important ones for the classification model. The models based on data preprocessed using direct orthogonal signal correction (DOSC), standard normal variate (SNV), and first and second derivatives provided the most successful results in terms of median sensitivity (0.91 in external validation) and median specificity (1.00 for external validation of SNV models and 0.94 for DOSC and first and second derivative models). A previously developed model, which was based on fatty acid fingerprinting of the same set of feed samples, provided a higher sensitivity (1.00). This shows that the NIRS-based approach provides a rapid and low-cost screening tool, whereas the fatty acid fingerprinting model can be used for further confirmation of the organic identity of feed samples for laying hens. These methods provide additional assurance to the administrative controls currently conducted in the organic feed sector.

KEYWORDS: *authenticity, chemometrics, feed, fingerprint, NIRS, organic food*

INTRODUCTION

The organic food market has increased during the past decade, especially due to consumers' concerns on animal welfare and the environment and because of health and social status considerations, among other reasons.[1,2] However, the organic production system is more expensive than the conventional system, and therefore, organic products tend to retail at a higher price, which makes them susceptible to fraud. In the European Union, regulations related to organic animal products require that the animals are fed with organic feed (that is to say that at least 95% of its dry matter should come from ingredients of organic farming).[3]

Administrative controls and inspections are currently conducted to certify organic products and avoid frauds. The authenticity of organic feed needs to be verified to avoid intentional or accidental mislabeling, because fraud at this level of the food chain would affect the authenticity of all the products derived from these animals. Having an analytical tool to verify the organic identity of food and feed products would protect genuine organic food and feed producers, would reassure consumers, and would help the regulatory and inspection bodies.[2] Because of this, some tools have recently been developed to authenticate several organic food products.[4−7] Feed fatty acid fingerprinting has been successfully used to verify the organic identity of feed used for laying hens.[7] However, this determination is based on a multistep procedure (grinding of feed, fat extraction, derivatization, and gas chromatographic separation) which requires the use of chemicals and trained personnel and is relatively time-consuming.

The ultimate approach in organic product verification would be the development of methods based on rapid techniques that could be applied even during inspections on site. NIRS is a nondestructive, easily applicable, and fast technique that requires minimal or no sample preparation and permits the measurement of several components at once. By recording the response of certain molecular bonds (such as O−H, N−H, or C−H) to NIR radiation, NIR spectroscopy generates a spectrum that may be characteristic of a sample and may be considered its "fingerprint".[8] On the basis of previous results on the authentication of organic feed by fatty acid fingerprinting[7] and with regard to the particular advantages of NIR spectroscopy, NIRS might be a promising technique to authenticate organic feed. Indeed, NIRS is already being used in the feed sector to determine feed composition and its nutritive value, digestibility, and traceability.[9,10] It can even be implemented at the feed mill plant level.[11,12] Although extensive literature exists on the application of NIR spectroscopy to both qualitative and quantitative analysis of feed,[13−15] there are currently no studies on the authentication of the organic identity of feed by NIRS.

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The aim of this research was therefore to evaluate NIR spectroscopy for the authentication of organic feed used for laying hens in The Netherlands as a rapid alternative to the fatty acid fingerprinting approach.

MATERIALS AND METHODS

Study Design. A total of 96 feed samples used for laying hens were collected during 2009 and 2010 from farms in The Netherlands. The set of samples consisted of 36 organic feed samples and 60 conventional feed samples (from 24 free-range farms, 24 barn farms, and 12 cage farms). The sampling was conducted in the framework of a larger project in which also methodology for authentication of organic eggs was developed.[5,6] The feed samples were collected from the same farms as used for the organic egg study, and they therefore represent feed that was used in practice in laying hen farms in The Netherlands in 2009 and 2010. Farms were selected with the help of the Dutch product board for poultry and eggs (CPE) and the Dutch organic produce certification body SKAL. The selection was balanced with regard to location (north, east, south, west) and farm size per production system (organic and conventional). The three farm size groups in each production system were defined taking into account the particular farm populations, as usually organic farms are smaller sized than conventional farms. Organic farm size groups consisted of farms with <5000, 5000−10000, and 10000−20000 hens; the conventional groups consisted of the categories 10000−20000, 20000−50000, and >50000 hens.[5] The feed samples were stored in the dark until analysis. Before being analyzed, they were ground to 0.5 mm particle size by using a ZM200 Retsch ultracentrifuge mill (Retsch Benelux, Nijkerk, The Netherlands).

Near-Infrared Spectroscopy of Feeds. NIR measurements were performed using a FOSS NIR 6500 SY-I system equipped with a spinning module, working in reflectance mode, in the spectral range of 1100−2498 nm, taking readings every 2 nm (FOSS NIRSystems, Inc., Laurel, MD). Measurements were taken using standard ring cups (diameter of 3.75 cm).

Statistical Analysis and Modeling. Principal component analysis (PCA) was performed with the 96 samples to screen the multivariate data for outliers and to explore the presence of any natural clustering in the data. PCA performs a reduction in the data dimensionality to facilitate the visualization of the multivariate data, retaining as much as possible the information present in the original data.[16]

Then partial least squares discriminant analysis (PLS-DA) was used to develop a classification model for organic feed vs conventional feed. PLS-DA is a supervised classification technique that is often used for highly dimensional data, especially when the amount of variables greatly exceeds the number of samples. It performs a variable reduction on the data set by calculating new variables (called latent components or factors) by combining the variables in the data set to find the maximum correlation between them and the class variable and, thus, the maximum separation among two classes (organic vs conventional). Then linear discriminant analysis is applied on the reduced variable set (the latent components) to provide the final classification model.

Since data preprocessing can have a profound effect on the model results, several methods of data preprocessing were evaluated: none (raw data), autoscaling (scaling to unit variance), first and second derivatives (gap 5), standard normal variate (SNV),[17] and direct orthogonal signal correction (DOSC).[18,19] In DOSC, the spectral information that is certainly not related to the response variable (or class membership in this case) is largely ignored. The optimal PLS-DA model was then determined using a stratified random resampling approach including internal and external model validation. It consists of the following steps:

(i) Randomly select 70% of the data set representative with respect to class membership and perform data preprocessing (training subset).

(ii) For each preprocessed training subset, find the optimal number of PLS components using the routine of Boulesteix,[20] a maximum of 8 components and 50 iterations, leaving 30% of the training set out as a pseudo test set.

(iii) Internally validate each preprocessed data subset using the optimal number of components as determined in step ii and calculate the sensitivity (i.e., number of organic feed samples correctly identified as organic divided by all organic feed samples included in the subset) and the specificity (i.e., number of conventional feed samples correctly identified as conventional divided by all conventional feed samples included in the subset).

(iv) Calculate the sensitivity and the specificity of for each data preprocessed subset using the remaining 30% of the data as an external validation subset. For autoscaling and DOSC, the parameters for preprocessing of the test set were based on the parameters as derived for the training set.

To study the robustness of these models, these four steps were repeated 1000 times. Thus, 1000 data subsets were created, and each of them was submitted to the five data preprocessing techniques. Then each of the 5000 models was internally and externally validated. The suitability of the preprocessing techniques applied was assessed by considering the median sensitivity and specificity during the external validation of the 1000 models conducted with each data preprocessing method.

To identify which variables (wavelengths) contributed most to the classification model, a variable selection approach[20] was followed. The procedure consisted of a random selection of 70% of the samples representative of the class membership. The variables were then ordered from the most to the least important with respect to the classification into organic and nonorganic feed according to the absolute value of the weight defining the first PLS latent component ("variable.selection" routine in the R package "pls.genomics").[20] A weight value was calculated for each variable by dividing 1 by the rank number. These three steps were repeated 1000 times, and the summed weight for each variable (wavelength) was calculated (and divided by 1000).

All calculations were performed in R version 2.12.2 (www.R-project.org)[21] using built-in functions and the package "pls.genomics" for various PLS-DA algorithms. For DOSC,[19] the Matlab code provided by the Biosystems Data Analysis Group of the University of Amsterdam (http://www.bdagroup.nl/) was rewritten to R code.

RESULTS AND DISCUSSION

Exploring NIR Data: PCA. As can been seen in Figure 1, the major features of the raw NIR spectra are quite similar for both organic and conventional feed. Mathematical treatment of the NIR spectra and chemometrics is usually required to exploit the information underlying these spectra and to reveal subtle differences that might exist between different types of samples that might not be directly evident.[22] PCA was conducted with the NIR spectral data (1100−2498 nm) of the whole sample set (96 samples) to reveal natural clustering of the samples and to detect outliers. Figure 1 shows a high variability among the absorbance values of different samples that might be due to some effects such as scattering. Therefore, preprocessing of raw data was necessary. Several data preprocessing methods were tried to reveal any natural clustering in the sample set and the presence of outliers. In the raw spectra, it is apparent that two samples seem to be quite different from the bulk of the data showing an increased baseline (Figure 1) at shorter wavelengths. Since the PCA scores plot (Figure 2) did not reveal any clear abnormalities from any of the preprocessing techniques applied in this study, there was no reason to exclude these samples for further modeling. While no differences between organic and conventional feed samples were evident in the raw data, the PCA scores plot of the

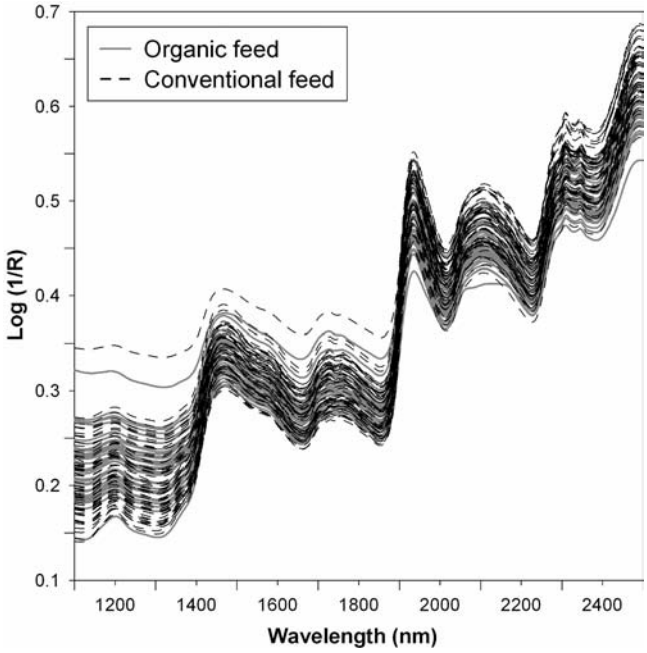

Figure 1. NIR spectra of organic and conventional feed.

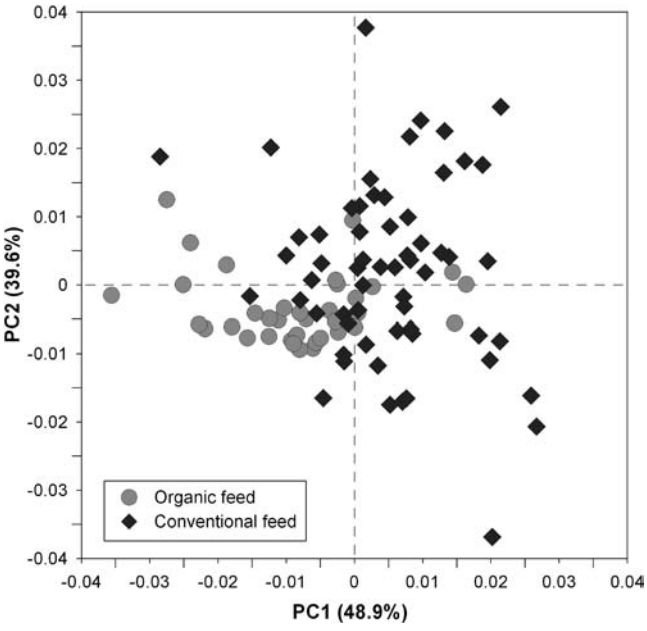

Figure 2. PCA scores plot of the NIR data (second derivative). Variance explained by each factor is provided in parentheses.

preprocessed data revealed a certain tendency of both feed types to cluster in two partly overlapping clusters (Figure 2).

Classification Model: Organic vs Conventional Feed. According to the European regulations, it is possible to use the same feed for the production of the three conventional egg categories (cage, barn, and free range).[3] Furthermore, in the PCA, the conventional feeds used for the production of free range, barn, and cage eggs did not show any tendency to cluster separately from each other (data not shown). Therefore, all conventional feeds were considered within one category for the development of a binary classification model (organic vs conventional feed).

Binary classification models (organic vs conventional feed) were developed using PLS-DA. The general aim of these classification models is to use them to predict the (organic) identity of new unknown or suspicious feed samples. To obtain reliable results in the future, it is highly important to validate the models and to verify their robustness. Following the approach described in the section "Statistical Analysis and Modeling", the median sensitivity and specificity was determined for both internal and external validation using various data preprocessing techniques. As shown in Table 1, the

Table 1. Median Sensitivity and Specificity of PLS-DA Models during Internal and External Validation and the Median Optimal Number of PLS-DA Factors[a] Established from 1000 Realizations of the PLS-DA Model Using Several Methods of Data Preprocessing[b]

Internal Validation			
preprocessing method	sensitivity	specificity	no. of factors
none (raw data)	0.88 (0.72−1.00)	0.98 (0.88−1.00)	8 (2−8)
autoscaling	0.88 (0.76−1.00)	0.98 (0.88−1.00)	8 (6−8)
first derivative[c]	1.00 (0.80−1.00)	0.98 (0.95−1.00)	8 (4−8)
second derivative[c]	1.00 (0.88−1.00)	0.98 (0.93−1.00)	8 (5−8)
SNV	0.92 (0.80−1.00)	0.98 (0.95−1.00)	8 (7−8)
DOSC (one component)	1.00 (0.92−1.00)	1.00 (0.98−1.00)	2 (2−8)
External Validation			
preprocessing method	sensitivity	specificity	
none (raw data)	0.82 (0.27−1.00)	0.89 (0.50−1.00)	
autoscaling	0.82 (0.45−1.00)	0.94 (0.50−1.00)	
first derivative[c]	0.91 (0.45−1.00)	0.94 (0.72−1.00)	
second derivative[c]	0.91 (0.55−1.00)	0.94 (0.72−1.00)	
SNV	0.91 (0.45−1.00)	1.00 (0.72−1.00)	
DOSC (one component)	0.91 (0.55−1.00)	0.94 (0.56−1.00)	

[a]The maximum number of factors is 8. [b]Minimum and maximum values are provided in parentheses. [c]Gap 5.

model developed after the application of DOSC provided the highest sensitivity and specificity (median value 1.0) during internal validation. Models based on the first and second derivatives also reached a very high sensitivity (1.0) and specificity (0.98) during internal validation. SNV also performed quite well, but provided a slightly lower sensitivity.

The number of PLS-DA latent components of each model was optimized by evaluating the percentage of correct classifications (sensitivity and specificity) obtained during their internal validation. Of all the preprocessing techniques applied, the DOSC models required the lowest number of components (Table 1). Actually, DOSC was designed to reduce the effect of the spectral information not related to the class membership, which explains the small number of PLS-DA latent components required in the classification models.[18,19]

Once optimized, each of the 1000 models (for each preprocessing technique) was used to predict the identity of the 30% samples that had been excluded in the first step of the procedure (external validation). Median values of sensitivity and specificity decreased in comparison to the values found during internal validation, but they were still quite high, especially for the DOSC, SNV, and first and second derivative models (Table 1). The median sensitivity was slightly lower than the specificity, showing a wider range. The minimum sensitivity was close to 0.5 for all preprocessing techniques;

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however, these low values (<0.6) were only reached by a few data subsets (less than five) for the DOSC, SNV, and first and second derivative models (see box-plot graphics in the Supporting Information). Specificity values varied in narrower ranges for SNV- and first and second derivative-based models than for DOSC-based models (Table 1). Overall, these external validation results are quite successful, especially for DOSC, SNV, and first and second derivative models. Therefore, unknown organic feed samples would be, in most cases, correctly identified by applying NIR spectroscopy and PLS-DA classification.

Wavelength Contribution to the NIR Classification Model. The variables (wavelengths) that most contributed to the PLS-DA classification model were investigated according to an approach based on Boulesteix.[20] Figure 3 shows the

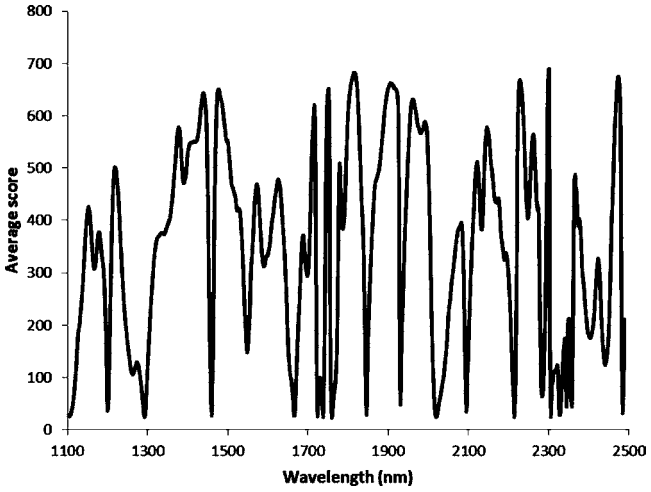

Figure 3. Importance of variables for classification using PLS-DA (organic vs conventional feed, based on first derivative).

importance of each variable (wavelength) for the PLS-DA classification model based on the first derivative data. The regions with the highest importance correspond to the maxima and minima of the spectra after the first derivative is taken.

The region located around 1460−1590 nm has been attributed to the N−H stretch and has been used to estimate the crude protein content.[23,24] Also some wavelengths in the 2020−2124 nm region have been used to predict the crude protein content in cereals.[24] Both regions are shown to be among the most important for the classification model (Figure 3). This could indicate that the protein content and/or composition might differ between organic and conventional feeds. Differences in the ingredients selected to formulate organic and conventional feeds might explain these protein differences.

Water content has been related to the 1450 and 1930 nm wavelengths.[12,14] However, both regions did not contribute to a great extent to the classification model, indicating that water content might not be important to discriminate organic and conventional feeds.

Wavelengths located within the 1150−1170 region and around 1218 nm have been related to CH_3- and $-CH_2-$ groups of fatty acids since this region corresponds to the second overtone of the C−H stretching vibration.[22,25] Indeed, fats with different unsaturation degrees have been discriminated using some wavelengths in this region.[25] Other important spectral regions for the classification into organic and conventional feeds have also been related to the fat content and fat composition of several food products. For instance, the 2100−2200 nm region or the 2230−2400 nm region has been related to differences in the unsaturation degree of oils and other products.[8,23,25,26] These regions showed several wavelengths recognized as important for the classification model (Figure 3). Also, the region 1350−1430 nm, attributed to the C−H combination,[8,25] has been attributed to the spectra of fats and oils. Overall, this indicates that organic and conventional feeds might present differences in the unsaturation degree of their lipid fraction. This is according to previous results where the fatty acid composition of feeds was used to verify the organic identity of these feeds.[7] Moreover, the study[7] showed that some polyunsaturated (C18:2n−6, C18:3n−3) and monounsaturated (C16:1n−9) fatty acids had a high contribution to the classification model.

NIR Model vs Fatty Acid Fingerprinting Model. In a previous part of this study, using the same set of samples as those used here, it was shown that it is also possible to verify the identity of organic feed by means of fatty acid fingerprinting.[7] Indeed, better sensitivity values (1.0) were reached than with the NIRS-based method. However, to obtain the feed fatty acid fingerprint, the analytical procedure requires the extraction of the fat from feed, the derivatization of its fatty acids into fatty acid methyl esters, and its determination by gas chromatography. This implies that the feed fatty acid fingerprinting approach is a relatively time-consuming method that does not allow an immediate answer on the real identity of samples and requires the use of chemicals, expensive equipment, and trained personnel. NIR spectroscopy is a more rapid, cost-effective method that does not require the use of highly qualified personnel, might even be applied in situ, and allows a massive number of analyses of samples per day. Both authentication models could be applied to verify the organic identity of unknown or suspicious organic feed samples used for laying hens, being complementary to the administrative controls and inspections usually conducted in the organic food sector. Due to the above-described practical aspects, together with the higher sensitivity of the fatty acid fingerprint model, the NIRS-based method would be more suitable as a rapid screening tool. The fatty acid fingerprinting method would then provide further confirmation in the case of any suspected feed sample labeled as organic. The fatty acid fingerprinting method would be required only for those feed samples labeled as organic, but identified as conventional by the NIRS model. The feed fatty acid fingerprint model would then further show if the feed sample is truly organic (thus, it would have been a false negative in the NIRS method) or conventional (detection of a fraud).

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Box-plot graphics for the sensitivity and specificity values obtained during both internal and external validation of the 1000 models developed after the 5 different preprocessing techniques. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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■ ABBREVIATIONS

NIRS, near-infrared spectroscopy; PLS-DA, partial least squares discriminant analysis; DOSC, direct orthogonal signal correction; SNV, standard normal variate; PCA, principal component analysis

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