Classification the geographical origin of corn distillers dried grains with solubles by near infrared reflectance spectroscopy combined with chemometrics: A feasibility study

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ABSTRACT

In this study, 137 corn distillers dried grains with solubles (DDGS) samples from a range of different geographical origins (Jilin Province of China, Heilongjiang Province of China, USA and Europe) were collected and analysed. Different near infrared spectrometers combined with different chemometric packages were used in two independent laboratories to investigate the feasibility of classifying geographical origin of DDGS. Base on the same dataset, one laboratory developed a partial least square discriminant analysis model and another laboratory developed an orthogonal partial least square discriminant analysis model. Results showed that both models could perfectly classify DDGS samples from different geographical origins. These promising results encourage the development of larger scale efforts to produce datasets which can be used to differentiate the geographical origin of DDGS and such efforts are required to provide higher level food security measures on a global scale.

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1. Introduction

Distillers dried grains with solubles (DDGS) is the co-product of bioethanol and alcoholic beverage production. It has high nutritional content, especially protein (Spiehs, Whitney, & Shurson, 2002), and studies have reported that using DDGS as a source of feed protein increases animal productivity (Masa'deh, Purdum, & Hanford, 2011; Whitney, Shurson, Johnston, Wulf, & Shanks, 2006). With the recent and dramatic increase in bioethanol production, millions of tonnes of DDGS are now produced each year. DDGS has quickly become a globally traded commodity playing an important role in the animal feed industry.

The quality and safety of animal feed has been problematic for many decades. Some major incidents, such as the bovine spongiform encephalopathy (BSE) crisis, the multiple supply chain dioxin contamination incidents and the melamine scandal, have shown how these events related to feed materials can cause great concern with regards to protecting the integrity of the feed-food supply chain (Banati, 2011; Haughey, Graham, Cancouet, & Elliott, 2013; Sharma & Paradakar, 2010). As a co-product of fermentation industry, the quality of DDGS produced may not be as highly considered as the quality of the ethanol produced. Several references have been reported that nutrient profile of DDGS can be influenced by factors including raw material, drying time, drying temperature, the ratio of distillers wet grains to condensed distiller solubles, etc. (Belyea et al., 2010; Liu, 2011; Nietner, Pfister, Glomb, & Fauhl-Hassek, 2013; Spiehs et al., 2002). The composition of raw material (corn) was related to the climate conditions and soil parameters of the geographic regions where it is grown. The fermentation process and the production technology particular to a geographic region mainly depends on its economic development level. Moreover, the adulteration or contamination of products is mainly from problematic raw materials or production procedure. Consequently, the quality of resulting DDGS may be largely determined by their geographical origins. In case of a crisis which may be related to DDGS contamination or adulteration, it is of extreme importance to know the geographical origin of the specific commodity immediately, thus preventing further spread of potentially harmful material. In this context, fast and reliable methods for

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determination of the geographical origin of DDGS are needed as a matter of some urgency.

Several techniques have been developed for authentication and traceability approaches for agricultural commodities, such as near infrared reflectance (NIR) spectroscopy, Raman spectroscopy, stable isotope ratio analysis and inductively coupled plasma mass spectrometry. (Geana et al., 2013; Gonzalvez, Armenta, & De La Guardia, 2009; Herrero Latorre, Pena Crecente, Garcia Martin, & Barciela Garcia, 2013; Sun, Guo, Wei, & Fan, 2012). Among these techniques, NIR spectroscopy technique is a non-invasive and rapid method which has the advantage of minimal sample preparation, no need of harmful chemical reagents, tedious chemical analysis and experienced laboratory staff. Several studies have shown the potential of NIR spectroscopy combined within different chemometric methods for classification geographical origin of food and feed but none to date have explored the methodology for DDGS (Casale, Casolino, Oliveri, & Forina, 2010; Chen et al., 2008; Herrero Latorre et al., 2013; Luo et al., 2011; Yu, Zhou, Fu, Xie, & Ying, 2007; Zhao, Guo, Wei, & Zhang, 2013).

The aim of the present study was to investigate the feasibility of using NIR spectroscopy combined with chemometrics as a rapid technique to classify the geographical origin of DDGS.

2. Materials and methods

2.1. Sampling

Within the European Union research project Quality and Safety of Feeds and Food for Europe (QSAFFE), a total of 137 corn DDGS samples from China (n = 48), USA (n = 54) and Europe (n = 35) were collected. More specifically, Chinese samples were from Jilin Province (n = 18) and Heilongjiang Province (n = 30). European samples were from Czech Republic (n = 21), Netherlands (n = 5), Poland (n = 3), Austria (n = 3), Hungary (n = 2) and Spain (n = 1), respectively. The samples from the same geographical origin, including different batches, were collected directly from production facilities at various times of the year from 2011 to 2013. Samples were

vacuum-packed and sent to Federal Institute for Risk Assessment (BfR) in Germany for grinding and homogenisation. DDGS samples were stored at 4 °C in the dark. Grinding of the samples was performed with a centrifugal mill (ZM 200, Retsch, Germany, mesh size 0.5 mm). Subsequently, the ground samples were homogenised in plastic containers (filling level ∼2/3) for 6 h using a drum hoop mixer (RRM 100, Engelsmann, Germany). Portions of the ground samples were distributed to laboratory of China Agricultural University (CAU) and Queen's University Belfast (QUB) in sealed containers which were stored at 4 °C in the dark before analysis.

2.2. Experimental design

Fig. 1 shows the flowchart of experimental design for this study. For chemical analysis, the content of moisture, crude protein, crude ash and crude fat were determined according to standard analytical method of ISO 6496 (1999), ISO 5983-2 (2009), ISO 5984 (2002) and ISO 11085 (2008), respectively. Each sample was analysed in duplicate. Between-group concentration of each composition was statistically compared using one way ANOVA by SPSS 17.0 software (SPSS Inc., Chicago, IL, USA). Differences were considered statistically significant at p-values $\leqslant 0.05$.

For NIR spectroscopy analysis, two laboratories (CAU and QUB) acquired spectral data of the DDGS samples using different spectrometers and processed the spectral data by different chemometrics software packages. At CAU, prior to NIR spectroscopy data collection, samples were kept in the room temperature (25 °C ± 1 °C) for 24 h with the temperature controlled by the air-conditioning. The spectra data were recorded using a Spectrum 400 instrument (PerkinElmer, Inc. Shelton, CT, USA) in reflectance mode at room temperature. Approximately 45 g of each sample were poured onto the standard quartz cup (9 cm in diameter and 1.5 cm high) spinner on the Integrating Sphere module of the instrument. The spectrum of each sample was recorded in triplicate by accumulating 32 scans at 8 cm^{-1} resolution between 10,000 and 4000 cm^{-1}. Each spectrum has 3001 variables. The
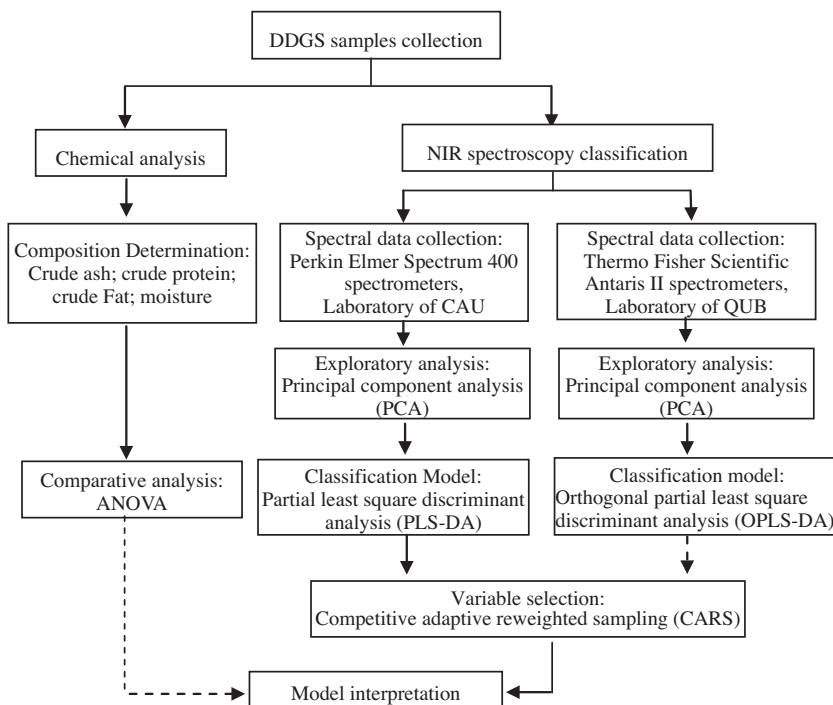

Fig. 1. Flowchart of experimental design.

repetitious spectra from a sample were averaged before analysis. At QUB, near-infrared spectra of the samples, obtained using reflectance mode, were recorded on an Antaris II FT-NIR (Thermo Fisher Scientific, Dublin, Ireland). The samples of DDGS (10 g) were poured onto the sample cup spinner (5 cm) on the Integrating Sphere module of the instrument. The spectrum of each sample was recorded in triplicate by accumulating 64 scans at 8 cm^{-1} resolution between 12,000 and 4000 cm^{-1} with the sample cup being refilled on each occasion. Instrument control and initial spectral manipulation were performed with Result Integration software.

To remove or minimise the noise and enhance the spectral features, SNV (standard normal variate) with 1st or 2nd derivative (15 points Savitzky–Golay filter and a second-order polynomial fit) (SNVd1 or SNVd2) pretreatment method was applied. Principal component analysis (PCA) was used for exploratory analysis. To compare and evaluate the model performance, Kennard and Stone algorithm (Daszykowski, Walczak, & Massart, 2002) was applied for dataset division base on the spectral data collected by CAU. The calibration set has 110 samples, including 15, 24, 28, and 43 samples from Jilin Province of China (JL China), Heilongjiang Province of China (HLJ China), Europe and USA, respectively. The external validation set has 27 samples, including 3, 6, 7 and 11 samples from JL China, HLJ China, Europe and USA, respectively. Based on the same dataset, partial least square discriminant analysis (PLS-DA) method (Barker & Rayens, 2003) by laboratory of CAU and orthogonal partial least square discriminant analysis (OPLS-DA) method (Bylesjo et al., 2006) by laboratory of QUB were used to develop calibration models, respectively. To interpret the classification model, the competitive adaptive reweighted sampling (CARS) method (Li, Liang, Xu, & Cao, 2009) was used to reduce irrelevant spectral variables and to select informative variables. To perform the CARS calculation, the number of Monte Carlo Sampling, the maximal number of partial least square components to extract, the number of folds for cross validation and the data pretreat method were set to 500, 7, 10 and 'autoscaling', respectively.

At CAU, spectral data pretreatment, PCA (Wold, Esbensen, & Geladi, 1987) and PLS-DA analysis were performed on MATLAB 2012a platform (The MathWorks, Inc. Natick, MA, USA) with PLS toolbox (version 6.71, Eigenvector Research, Inc, USA). The MAT-LAB code of CARS was downloaded at website: https://code.google.com/p/carspls/. At QUB, spectral data pretreatment, PCA and OPLS-DA analysis were performed by SIMCA 13 chemometric software package (version 13.0.3.0, Umetrics, Sweden).

3. Results and discussion

3.1. Chemical analysis results of DDGS

The chemical analysis results for DDGS samples from different geographical regions are summarised in Table 1. There are no standards available for the quality of DDGS. In terms of crude ash content, samples from JL China, HLJ China and USA (4.3–4.8%) are in the range of those reported (3.7–8.1%) (Belyea, Rausch, & Tumbleson, 2004; Liu, 2008, 2012), except for samples from Europe (3.2%). The mean crude protein (26.9–30.3%) and moisture (8.7–
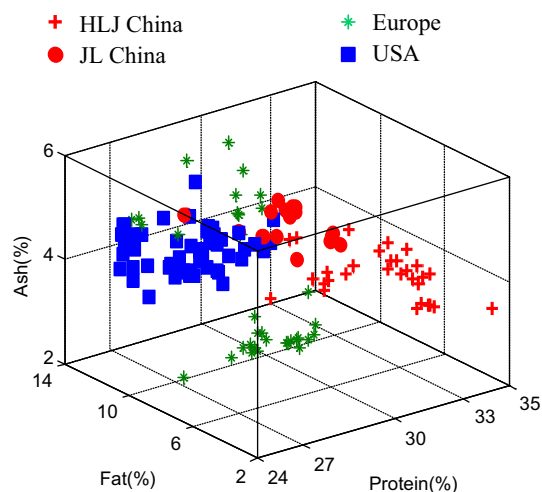
Table 1
Chemical analysis results for DDGS samples from different geographical origins.

	Crude ash (%)	Moisture (%)	Crude protein (%)	Crude fat (%)
JL China	4.82 ± 0.32[a]	9.02 ± 1.51[a,b]	29.52 ± 0.89[a]	8.40 ± 1.57[b]
HLJ China	4.54 ± 0.54[a,b]	9.06 ± 1.22[a,b]	30.28 ± 2.13[a]	3.29 ± 0.80[c]
Europe	3.19 ± 1.35[c]	8.78 ± 0.66[b]	29.86 ± 1.97[a]	11.00 ± 0.93[a]
USA	4.36 ± 0.34[b]	9.37 ± 0.70[a]	26.90 ± 1.49[b]	10.42 ± 1.59[a]

Note: [a–c]Means with different superscripts within a column differ ($p < 0.05$).

Fig. 2. Chemical analysis results for DDGS samples. Samples from different geographical origins are presented by different symbols.

9.4%) of DDGS are comparable to that reported results (26.0–31.7% and 7.3–12.9%, respectively) (Cromwell, Herkelman, & Stahly, 1993; Liu, 2011; Spiehs et al., 2002). With regards to the mean crude fat content, samples from JL China, USA and Europe (8.4–11.0%) are in the range of those reported previously (8.0–12.0%) (Liu, 2011; Salim, Kruk, & Lee, 2010), except for samples from HLJ China (3.3%). The extremely low crude fat content in samples from HLJ China may be due to bioethanol producers extracting the oil fraction from DDGS to produce biodiesel or other products for increasing the economic benefit in addition to bioethanol and the DDGS co-products (Ganesan, Rosentrater, & Muthukumarappan, 2009; Guney, Shim, Batal, Dale, & Pesti, 2013). Compared to the regular DDGS, the low oil DDGS has similar water activity, thermal properties and bulk density but better flow properties (Ganesan et al., 2009; Saunders & Rosentrater, 2009). Moreover, some recent studies showed that feeding broilers, cattle, lambs with 20% of low oil DDGS samples in diets did not affect their growth performances or carcass characteristics (Guney et al., 2013; He, Xu, Yang, Gibb, & McAllister, 2014; O'Hara et al., 2011).

DDGS samples from different geographical origins have distinctive chemical properties. One way ANOVA analysis showed that samples from Europe and USA have significant ($p < 0.05$) differences in their crude ash, moisture and protein content, except for crude fat content; samples from JL China and HLJ China have significant difference in their crude fat content ($p < 0.05$), but no significant difference ($p > 0.05$) was found in their crude ash, moisture and crude protein content; samples from JL China and HLJ China have significantly ($p < 0.05$) lower content of crude fat than samples from Europe and USA. However, by plotting the crude protein, crude fat and crude ash of all the samples in a 3-D figure (Fig. 2), it clearly shows that samples from the different regions were severely overlapped. As such, using these limited chemical results to classify the geographical origin of DDGS samples cannot be accomplished.

3.2. NIR spectroscopy classification

3.2.1. PCA analysis

PCA was first applied to detect groups in the spectral data of all the DDGS samples. The spectral data was pretreated by SNVd1. The first and second principal component scores plots are shown in Fig. 3(a) and (b) for CAU and QUB. Based on this, it can be seen
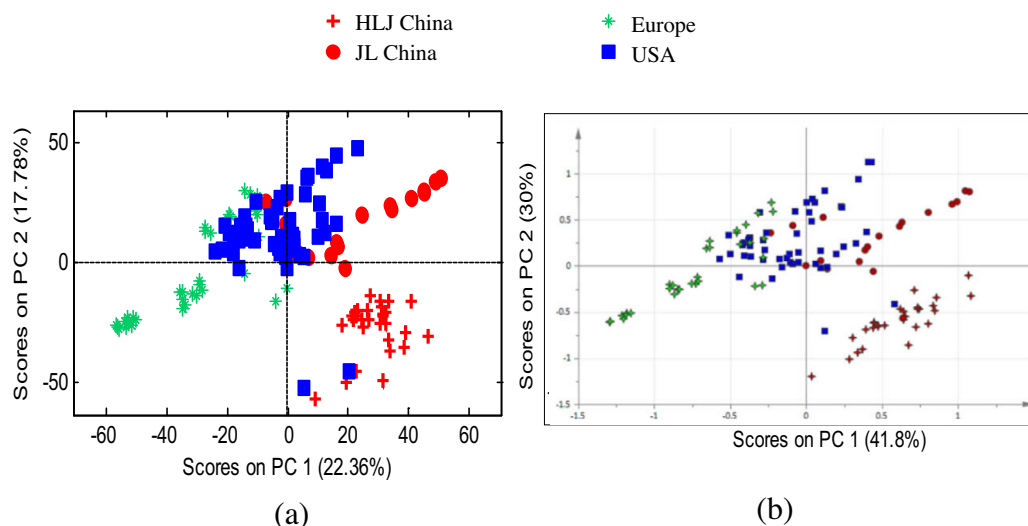
Fig. 3. PCA scores plot obtained by CAU (a) and QUB (b), respectively. Samples from different geographical origins are presented by different symbols.

in the figure that samples from HLJ China were located at the lower right corner of the scores plots. Samples from the USA, Europe and JL China origin had some overlap indicating that further classification analysis is needed.

3.2.2. PLS-DA and OPLS-DA models

PLS-DA is a binary discrimination method and therefore a two-step discrimination strategy was used as follows: (1) discrimination of samples from Chinese origin and other origins (USA and Europe); (2) discrimination of samples from two provinces of China as well as USA and Europe origin. The discrimination strategy and the model performances are shown in Table 2. Samples from different geographical origins were well discriminated by PLS-DA model.

At QUB, after removing six outliers based on PCA-Class analysis, the OPLS-DA model was constructed with 3 predictive components and 9 orthogonal components. The measure of fit (R^2X), i.e. how well the model fits the X data, was found to be 98.2%. Using cross validation, the predictability of the model (Q2) was found to be 87.9%. The OPLS-DA model rightly discriminated samples from each origin in the calibration set and correctly predicted all the samples in the external validation set. The 3-D scores plot for these

are shown in Fig. 4 where four distinct clusters are clearly observed within the scores plots for calibration and validation data demonstrating an exceptional level of separation.

The data produced has shown that two laboratories using different near infrared spectrometers combined with different chemometric software packages achieved similar results. The result also indicated that samples from different geographical origins have inherent differences and classification of samples by NIR spectroscopy combined with suitable chemometrics was feasible. However, due to many thousands of variables that were used to construct the discrimination models, interpretation of the above classification results proved to be difficult.

3.2.3. Variable selection and model interpretation

In order to determine which variables (wavelengths) contributed most to the separation of samples from different origins and to interpret the classification models, the CARS calculation was applied. As shown in Table 2, within limited selected variables, the corresponding PLS-DA models were simplified and their discrimination abilities were equivalent to those PLS-DA models which were developed with 3001 variables (full spectrum). These

Table 2
Discrimination strategy and partial least square discriminant analysis model performances based on full spectrum and competitive adaptive reweighted sampling selected variables. The spectral data was pretreated by standard normal variate (SNV) with 1st derivative. Latent variables (LV) are determined by full cross validation when minimum cross validation classification errors were obtained.

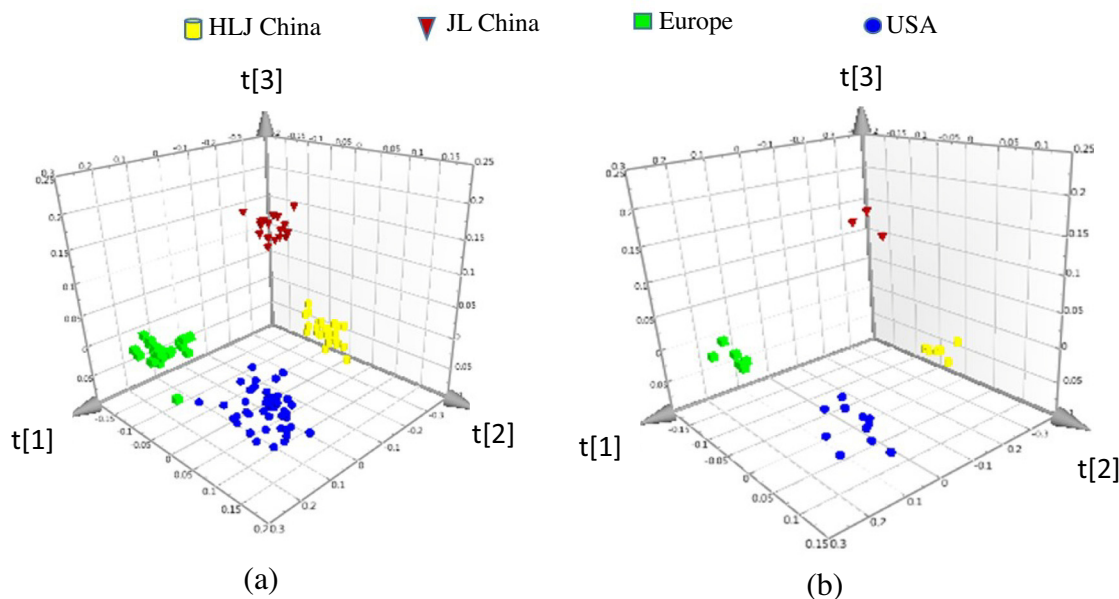| Discrimination strategy		Step 1 China origin vs. other origins	Step 2.1 Europe origin vs. USA origin	Step 2.2 JL China origin vs. HLJ China origin
Model parameters		3 LV; 3001 variables	6 LV; 3001 variables	1 LV; 3001 variables
Calibration	Sensitivity	100%	100%	100%
	Specificity	100%	100%	100%
Full cross validation	Sensitivity	100%	89.29%	100%
	Specificity	98.59%	97.67%	100%
External validation	Sensitivity	100%	100%	100%
	Specificity	100%	100%	100%
		2 LV; 13 variables	2 LV; 10 variables	1 LV; 2 variables
Calibration	Sensitivity	100%	100%	100%
	Specificity	100%	100%	100%
Full cross validation	Sensitivity	100%	100%	100%
	Specificity	100%	100%	100%
External validation	Sensitivity	100%	100%	100%
	Specificity	100%	100%	100%

Fig. 4. OPLS-DA score plot showing clear separation of the four geographical origin groups for samples in (a) calibration set and (b) external validation set. The spectral data is pretreated by SNVd2. Model was constructed with 3 predictive components and 9 orthogonal components.

results indicate that CARS was an effective method in selecting informative variables and reducing high co-linearity variables. Moreover, the interpretation of classification models was achieved. Thirteen out of 3001 variables were selected by CARS which mainly contributed to the separation of samples of Chinese origin compared to other origins. According to the reference (Workman & Weyer, 2007), selected variables including 9874 cm^{-1}, 6738 cm^{-1}, 6618 cm^{-1}, 6526 cm^{-1}, 5088 cm^{-1}, 4788 cm^{-1} and 4166 cm^{-1} are mainly associated with N—H vibrations of amine; selected variables including 8664 cm^{-1}, 7384 cm^{-1}, 7316 cm^{-1}, 7290 cm^{-1} and 7074 cm^{-1} are associated with C=O or C—H vibrations of hydrocarbons; selected variables 5508 cm^{-1} is attributed to combinations of C—H stretching and O—H stretching of fibre. These findings indicated the influences of the protein, fat and fibre content differences to the discrimination model. Ten out of 3001 variables were selected by CARS for discriminating samples from USA and European origin. These variables are 8474 cm^{-1}, 5794 cm^{-1}, 5792 cm^{-1}, 5582 cm^{-1}, 4608 cm^{-1}, 4522 cm^{-1}, 4494 cm^{-1}, 4380 cm^{-1}, 4342 cm^{-1} and 4340 cm^{-1}. Selected variables including 5794 cm^{-1}, 5792 cm^{-1} and 4608 cm^{-1} are associated with C—H vibrations of hydrocarbons. Variables including 4522 cm^{-1}, 4494 cm^{-1}, 4342 cm^{-1} and 4340 cm^{-1} are associated with combinations of N—H, C—N and C=O vibrations or C—H vibration of amine. The selected variable 5582 cm^{-1} is associated with O—H vibrations of moisture. The selected variable 4380 is attributed to C—H vibration of starch. These results indicated the influences of the protein, fat, moisture and starch content differences obtained in the discriminatory models. Two out of 3001 variables were selected by CARS for discriminating samples from JL China and HLJ China. These two variables (5738 cm^{-1} and 4102 cm^{-1}) are associated with C–H vibrations indicating the influence of the fat content on the differences between them.

A previous study showed that >80% correct classification rate can be achieved when using Fourier transform infrared spectroscopy to discriminant corn DDGS from China and USA (Nietner et al., 2013). Another interesting study reported again a >80% correct classification rate obtained when classifying DDGS from USA, Europe and China by isotope ratio mass spectrometry (Nietner, Haughey, Ogle, Fauhl-Hassek, & Elliott, 2014). In this study, NIR spectroscopy combined with chemometrics achieved better classification results (100% correct classification rate). These promising results encourage the development of larger scale efforts to produce datasets which can be used to differentiate the geographical origin of DDGS and such efforts are required to provide higher level food security measures on a global scale.

4. Conclusion

The results of this study have shown that samples from China, Europe and USA have distinctive chemical properties. NIR spectroscopy combined with suitable chemometric methods has been shown to be a promising method for classifying the geographical origin of DDGS. Based on the same DDGS sample dataset, two independently laboratories (CAU and QUB) acquired spectral data by different spectrometers and processed them using different chemometrics software packages but achieved very similar results. Corn DDGS samples from the JL China, the HLJ China, the USA and Europe can be completely determined by PLS-DA or OPLS-DA models. CARS selected variables which effectively simplified the classification model and well interpreted the classification results. Further development is needed to fully validate the effectiveness of the method using more samples from the existing and new geographical origins.

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