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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 (Speihs, 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, & Fahl-Hassek, 2013; Speihs 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 $\sim 2/3$) 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 ≤ 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 ($25\text{ °C} \pm 1\text{ °C}$) 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

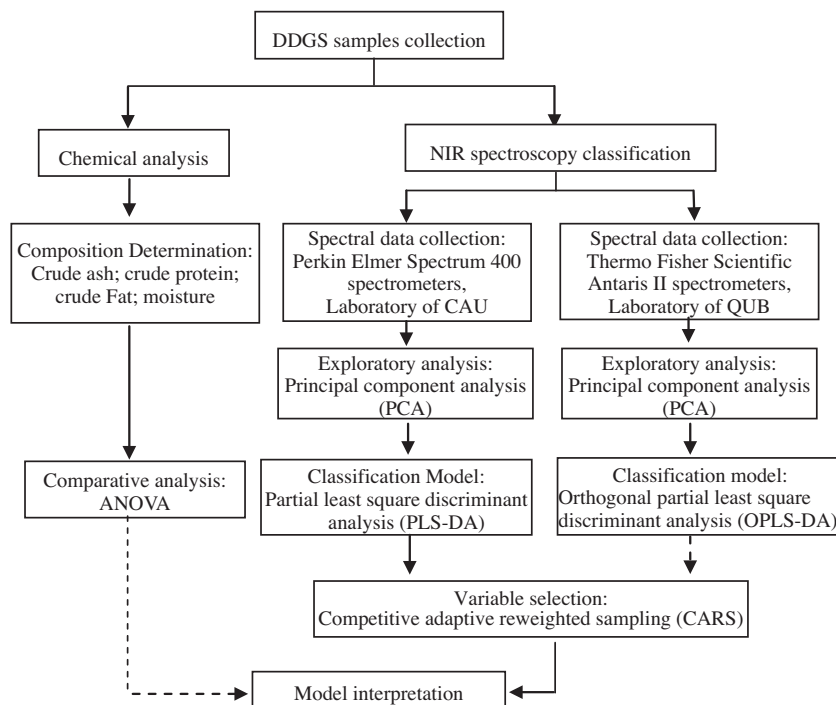


Fig. 1. Flowchart of experimental design.

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