Quality and SAfety of Feeds and Food for Europe

Identification of DDGs origin by ATR-FTIR spectroscopy after in situ fat extraction

Philippe Vermeulen*, Juan Antonio Fernández Pierna, Pierre Dardenne, Vincent Baeten

Walloon Agricultural Research Centre (CRA-W), Valorisation of Agricultural Products Department (D4), Food and Feed Quality Unit (U15), Chaussée de Namur, 24, B-5030 Gembloux (Belgium), *corresponding author: vermeulen@cra.wallonie.be

Introduction

The ban for using processed animal protein in the feeding stuffs is one of the reasons which led the feed sector to prospect other possible protein sources. Among the different possibilities and beside the soybean meal which is the main source of proteins for feed, Dried Distillers Grains with Solubles (DDGs) could be also considered as an important source to take into account. In USA, 30% of the corn is dedicated to ethanol production and the DDGS obtained as residue of the process are largely exported to Europe. The possible use of antibiotics or fermentation supplements to improve the ethanol production process involves some risks in the feed chain. The current work, performed in the framework of the QSAFFE project (http://www.qsaffe.eu), aims to identify the botanical and industrial process origin of DDGs using Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (ATR-FTIR).

Material and Methods

For this experiment, 71 samples of DDGs were collected and analysed. They are derived from corn, wheat or mixtures thereof, mainly from USA and China and produced in beverage or bio ethanol production. ATR-FTIR spectra of DDGS material were acquired using a Bruker Vertex 70 Fourier Transform spectrometer. Samples were spread and measured directly on the fat after extracting fat in situ using a paper filter on the crystal with press during 3 min. (**Figure 1**).

Figure 1: a) *Sample preparation on paper filter,* ***b)*** *Fat extraction using the Golden Gate ATR press during 3 min,* ***c)*** *FTIR Analysis of the fat on the diamond ATR (3 replicates)*

Reference values

All the samples were analysed using a FOSS XDS NIR spectrometer active in the 400-2500 nm range in order to predict moisture, fat, protein, fiber and ash content. Figure 2 shows the predicted values and the PCA on these predicted values according to the botanical or the industrial process origin.

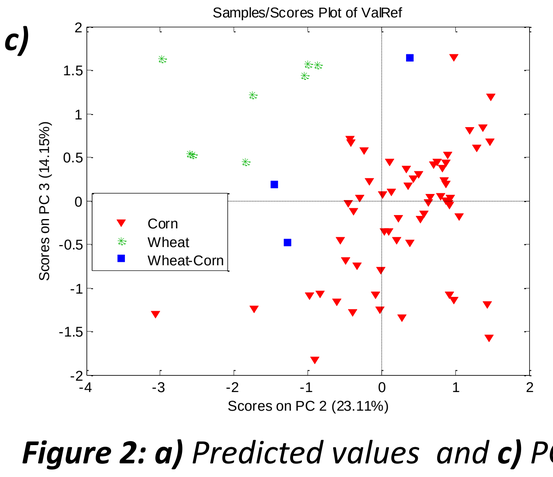
a)

Species	Moisture	Fat	Protein	Fibre	Ash
	1	2	3	4	5
Wheat	8,05	4,47	33,02	6,77	5,13
Corn	10,27	7,38	27,87	6,22	4,49
Wheat-Corn	9,42	5,52	30,69	6,80	4,82

b)

Process	Moisture	Fat	Protein	Fibre	Ash
	1	2	3	4	5
China-Zhongliang	11,21	3,29	29,81	6,39	4,12
China-Jilin	9,59	8,10	29,97	6,72	4,23
Provimi	9,54	10,23	27,04	5,93	4,43
USA-Thom	10,20	9,59	24,89	5,89	5,02

c)



d)

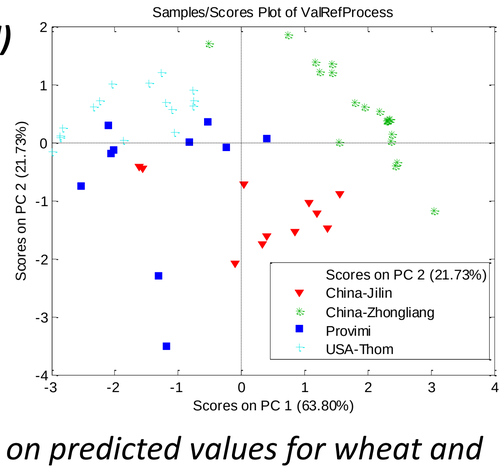
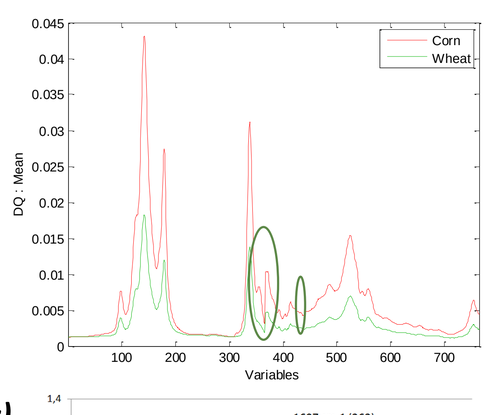


Figure 2: a) *Predicted values and* ***c)*** *PCA on predicted values for wheat and corn DDGs;* ***b)*** *Predicted values and* ***d)*** *PCA on predicted values for corn DDGs from 4 industrial processes.*

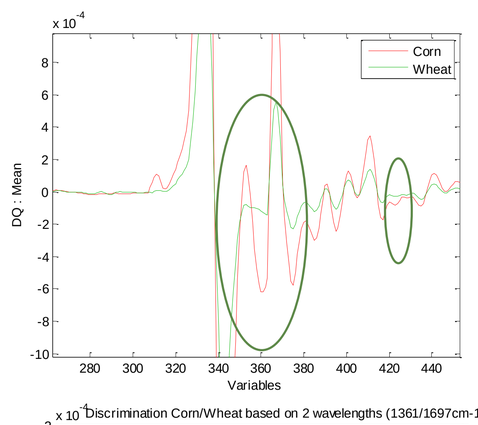
Results ATR-FTIR : classification based on 2 wavelengths

For this study, a calibration set and a validation set have been built from the database by selecting, for the validation set, samples from different sources than the calibration set. **Figure 3a** shows the mean raw spectra for corn and wheat. The data were preprocessed by 1st derivative Savitzky-Golay treatment 7,2,1 (**Figure 3b**). In order to discriminate between corn and wheat DDGs, the Fisher coefficient was used to select the wavelengths where the between-classes variation is higher than the within-classes variation. **Figure 3c** shows the Fisher coefficient calculated on preprocessed data for the wavelength range of the Bruker Vertex 70 FT spectrometer. Two wavelengths, 1361 cm-1 and 1697 cm-1 were selected, based on the fat specific spectral region of wheat/corn and on the Fisher coefficient value. **Figure 3d** shows the discrimination between corn and wheat DDGs using in X axis the preprocessed data near 1361cm-1 and in Y axis the preprocessed data near 1697 cm-1. The validation samples (empty circles for wheat DDGs and squares for corn DDGs) are included or very close to the ellipse corresponding to the 95% confidence limit. One corn DDGs was classified as wheat DDGs.

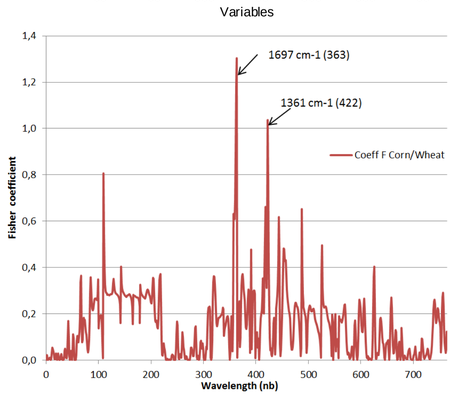
a)



b)



c)



d)

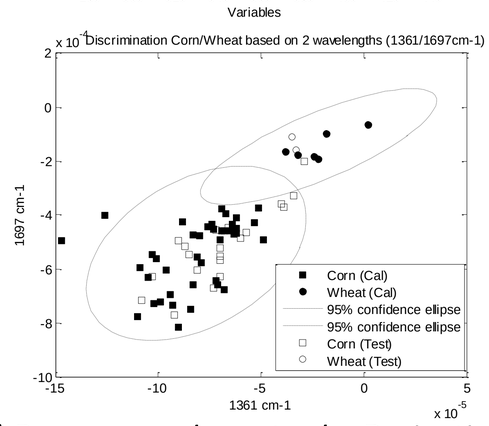
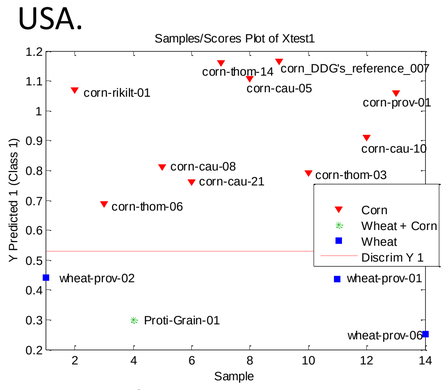


Figure 3: a) *ATR-FTIR mean spectra,* ***b)*** *Preprocessed spectra by Derivative (7,2,1) ,* ***c)*** *Fisher coefficient calculated from preprocessed spectra,* ***d)*** *Discrimination Corn/Wheat DDGs based on 2 wavelengths 1361cm-1 and 1697 cm-1*

Results ATR-FTIR : classification based on full spectra

PLSDA models were build in order to classify the DDGs according to botanical origin and industrial process origin. Figure 4a shows the correct classification of corn and wheat DDGs of the validation set and the classification of Protigrain (wheat 55%, corn 20%) as a wheat DDGs. Figure 4b shows that the corn DDGs provided by PROVIMI Company should be issue from a process similar to the corn DDGs provided by THOMPSON Company and issued from USA.

a)



b)

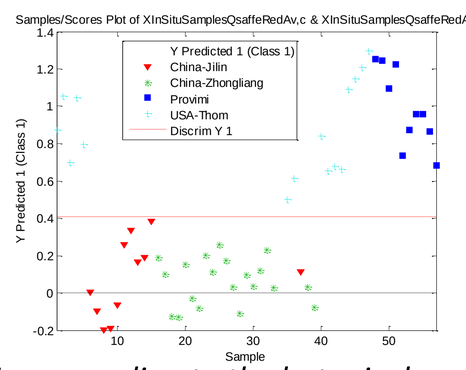


Figure 4: a) *PLSDA results for DDGs classification according to the botanical and the industrial process origin.*

Conclusion

Based on values predicted by NIRS, this study showed the variability between the DDGs from different botanical sources and issued from different industrial processes. This study showed also the potential of ATR-FTIR to discriminate the DDGs according to the botanical and the industrial origin . Additional samples from other sources and blind samples will be analysed to validate the models.

www.qsaffe.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 265702.