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Short communication

Potential of fluorescence spectroscopy in detection of low-levels of gluten in flour: A preliminary study

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ABSTRACT

Celiac disease is caused due to the ingestion of gluten containing food products. Eating gluten free diet is only way to avoid its occurrence. Gluten free products may get contaminated during the handling and processing operation which leads to the adverse impact on the gluten intolerant patients. Three different gluten free mixes were adulterated with two different types of wheat flours to get the gluten concentration in the range of 0–5 % by making 94 sample combinations. The obtained fluorescence spectra from these samples were transformed using *multiplicative scatter correction (MSC)* pre-processing to develop *partial least square regression (PLSR)* model for the prediction of low-levels of gluten. The obtained results showed a high value of 0.90 correlation *coefficient* (R^2) with 0.46 % *root mean square error of cross-validation (RMSECV)*. The calibration and prediction models also showed the same R^2 with root mean square error of calibration (RMSEC) and prediction (RMSEP) of 0.41 % and 0.46 % respectively. Hence fluorescence spectroscopy can be used to estimate the level of adulteration which is helpful for developing a sensor for determination of low-levels of gluten as a rapid and non-invasive method in food applications.

1. Introduction

Celiac disease is an autoimmune disorder which occurs in genetically predisposed persons due to the ingestion of gluten containing foods that leads towards the inflammation of the small intestine and hinders the absorption of various essential nutrients like mineral and vitamins (Albanell, Miñarro, & Carrasco, 2012). The other symptoms include diarrhea, blistering of skin and various neurological problems (Haraszi, Chassaigne, Maquet, & Ulberth, 2011). Gluten free diet is only the therapy to prevent this problem.

However, the role of gluten cannot be overlooked as it is the main protein in wheat based products which develops after hydration of flour by the application of force. It gives visco-elasitic properties to the dough which is important for gas retention and high loaf volume (Masure, Fierens, & Delcour, 2015). Therefore, it is a big challenge to develop gluten free products with similar characteristics. As it is present in different types of grains like wheat, rye, oat and barley, however it can be used in various other products like beer and soy sauce and acts as stabilizing agent in ice cream and ketchup. During the handling and manufacturing the gluten free products, there is a risk of cross contamination and need to develop some methods which can determine the low-levels of gluten. The classical methods which are being used for the detection of low-levels of gluten are immunochemical analytical techniques like polymerase chain reactions (Albanell et al., 2012). These methods are laborious, time consuming and need competent personal to carry out. These methods also produce undesirable chemicals which are not environment friendly. So there is a need to develop some non-invasive and rapid methods for determination of low levels of gluten. The potentials of fluorescence spectroscopy cannot be overlooked and act as an alternative. Fluorescence spectroscopy is being used in various fields and proved its potential to monitor the process and ensures the quality of different food products due its sensitivity and specificity (Faassen & Hitzmann, 2015). It is being employed in dairy like thermal changes in milk (Kulmyrzaev, Levieux, & Dufour, 2005), cheese (Christensen, Povlsen, & Sørensen, 2003), storage stability of yogurt (Christensen, Becker, & Frederiksen, 2005) and ice cream formulations (Granger, Costa, Toutain, Barey, & Cansell, 2006). It is used in meat research to classify and determine the total plate count in chicken to ensure safe and health food for the consumers (Sahar, Boubellouta, & Dufour, 2011). It has been applied in cereal

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research to monitor the pH and degree of acidity during sourdough fermentation (Grote, Zense, & Hitzmann, 2014) and classification of various cereals and its products (Zeković, Lenhardt, Dramićanin, & Dramićanin, 2012). It has been employed to predict the analytical, rheological, baking and nutritional parameters of wheat flour by just taking the spectral signature (Ahmad, Nache, Hinrichs, & Hitzmann, 2016, 2016a). Furthermore, it is applied to differentiate the different phases of farinographic curve and classification of wheat flour on the basis of baking quality characteristics (Ahmad, Nache, Waffenschmidt, & Hitzmann, 2016b). It is also used for food authentication and detection of adulteration in various foods like in olive oil (Sayago, Morales, & Aparicio, 2004).

As fluorescence is widely employed in various applications in food process and quality assurance operations but nobody has applied this technique for detection of low-levels of gluten in wheat flours. So the propose of this study is to develop a rapid and no-invasive method for prediction of low-levels of gluten in wheat flours with linear regression method using fluorescence spectroscopy which may lead to develop a sensor based on these results.

2. Materials and methods

2.1. Procurement of samples

Two different types of wheat flours 550 and 1050 were procured from local mill (Rettenmeier Mühle GmbH, Germany). Three different types of gluten free mixes (Schär) were purchased from the shelf of supermarket.

2.2. Preparations of samples

Gluten free (GF) mixes were adulterated with the two different types of wheat flours to produce different formulations with low gluten individually and in combinations. To produce more variability, all three gluten formulations were also mixed and contaminated with wheat flours to get combinations with different level of gluten. In this way 94 combinations were prepared with the variability ranged from 0 to 5 % (w/w) of wet gluten content as described in Fig. 1 the flow chart for the sample preparation. This sampling approach was adopted from the work of (Albanell et al., 2012) who used NIR spectroscopy for detection of low-levels of gluten in flour and dough preparations.

2.3. Determination of wet gluten

Wet gluten was determined by using the standard method given in ICC method 137. All measurements were done in duplicate (ICC, 1994).

2.4. Fluorescence spectroscopy

Spectra of each combination were taken using 2D-fluorescence spectroscopy BioView (Delta Light & Optics, Hørsholm, Denmark) sensor. It is equipped with a Xenon light source and 15 filter wheels and can measure the fluorescence spectra in the range between $\lambda_{Excitation} = 270-550$ nm and $\lambda_{Emission} = 310-590$ nm for excitation and emission respectively using different filter wheels due to the rotation for various wavelength combinations. The spectrum obtained comprised of 120 wavelength combinations having bandwidth in step of 20 nm. Each spectrum was taken in duplicate for every combination using fluorescence spectroscopy.

2.5. Data analysis

Data was composed of predictor (X) values which contain 188 spectra of 94 combinations whereas the predicted values (y) were respective low-level of gluten. The data set was exported to the MATLAB 2013b (The Mathwork TM, MA, United States) platform using the PLS tool box.

2.5.1. Pre-processing of data

After taking the spectra, it was pre-processed to remove the scatter and multiplicative effect due to the variation of the particle size. The data was pre-processed by using multiplicative scatter correction (MSC) transformation technique which was performed in two steps. In the first step, reference spectrum was calculated and subtracted from the individual spectrum, while intercept and slope were used according to the given Equation (1)

$$x_{corr,i} = \frac{(x_i - a)}{b} \quad (1)$$

2.5.2. PLSR modeling

Partial least square regression is a supervised technique which is

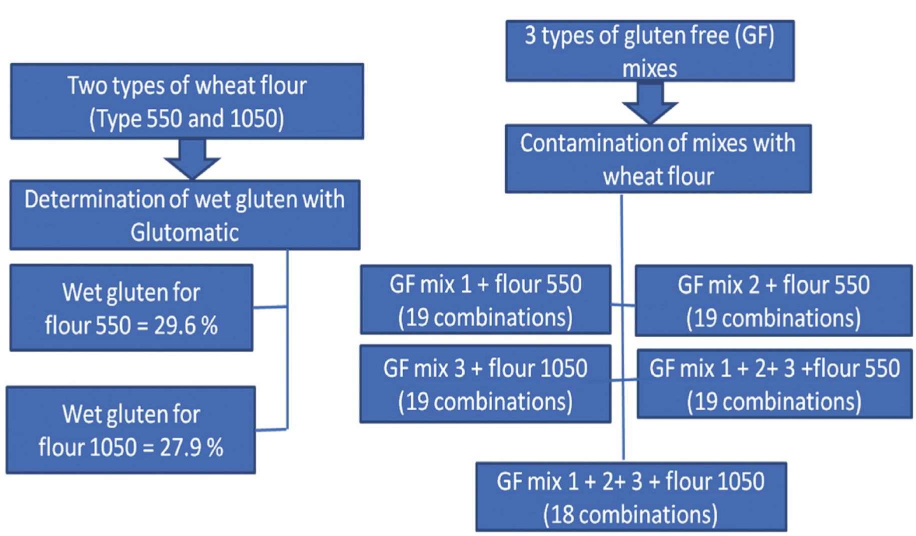

Fig. 1. Complete flow chart for sample preparation for low-levels of gluten combinations in flours.