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Detection of adulterations with different grains in wheat products based on the hyperspectral image technique: The specific cases of flour and bread

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ABSTRACT

The objective of this study was to test the capability of a SW-NIR hyperspectral image technique to detect adulterations in wheat flour and bread with cheap grains, such us sorghum, oats and corn, and to compare the hyperspectral information with the physicochemical alterations in the properties of products. Wheat flour was adulterated at four different degrees (2.5, 5, 7.5 and 10%) with sorghum, oat and corn flours. Flours were prepared and used to make bread. Flours and breads were characterized according to several physicochemical parameters (pasting properties, water activity, mass loss during the baking process and texture profile analysis). Crumbs were extracted from breads and conditioned. Hyperspectral image captures were taken of both flours and conditioned crumbs. The data analysis was based on multivariate statistical process control method (MSPC), where the differentiation of adulterated samples was observed in all cases for both flours and crumbs. Finally, in order to relate the image analysis results and the adulterated sample properties, a correlation significance map was created between the physicochemical properties of samples and the multivariate statistical parameters. The SW-NIR image technique was capable of detecting adulterations in each case and high correlation significances were observed ($\alpha = 0.01$) between wavelengths from specific spectra zones and the physicochemical properties of samples.

1. Introduction

Adulteration of food products (raw materials, intermediate products, authorized additives, end products, etc.) is of primary concern for consumers, food processors, regulatory agencies and industries. Adulteration typically involves replacing or diluting high-cost ingredients with cheap low-quality products (Kalivas et al., 2014). The history of food adulteration reports not only economic motives, but even criminal ones. Thus food frauds and adulterations may have economic implications for local authorities due to increased work load and costs to governments through loss of value-added taxes made from sales (Tähkäpää, Maijala, Korkeala, & Nevas, 2015). Therefore, rapid qualitative analyses are required to separate adulterated samples, followed by a quantitative analysis of this adulterant, if necessary.

The development of non-destructive rapid analysis methods is an area in which interest increased a few years ago. Within this area, numerous methods based on different chemical and physical principles have been proposed to carry out both qualitative and quantitative determinations in laboratories and process chains. The methods which have demonstrated high versatility and robustness are those based on the simultaneous analysis of the large number of wavelengths of electromagnetic spectra, and in all their modes (reflectance, absorbance, interactance, etc.) (Wu & Sun, 2013). Specifically, the study of infrared spectra (IR) has been used for a vast number of determinations from multiple food matrices and analytes.

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Two of the main techniques used for this purpose are IR spectroscopy and spectral imaging analysis. Several recent IR spectroscopy applications for detecting adulterations, fraud and contaminants have included the detection of pork adulteration in veal products based on FT-NIR spectroscopy (Schmutzler, Beganovic, Böhler, & Huck, 2015), minced lamb meat adulteration (Kamruzzaman, Sun, ElMasry, & Allen, 2013), minced beef adulteration with turkey (Alamprese, Casale, Sinelli, Lanteri, & Casiraghi, 2013), fraudulent adulteration of chili powders with Sudan dye (Haughey, Galvin-King, Ho, Bell, & Elliott, 2014), adulteration of oil used in animal feed production (Graham et al., 2012), etc. Concretely, into the area of grain product adulterations, (Cocchi et al., 2006), developed a method to quantify the degree of adulteration of durum wheat flour with common bread wheat flour based on near-infrared spectroscopy assisted by different multivariate calibration techniques. Although fewer works have been carried out using the spectral imaging analysis technique, it has also been used in a large number of applications to detect adulteration of beef and pork in raw meats (Ropodi, Pavlidis, Mohareb, Panagou, & Nychas, 2015), melamine in milk powders (Fu et al., 2014), gelatin adulteration in prawn (Wu, Shi, He, Yu, & Bao, 2013), and for the pattern recognition for the categorization and authentication of red meat (Kamruzzaman, Barbin, Elmasry, Sun, & Allen, 2012), etc.

The objective of this work was to study the feasibility of an SW-NIR hyperspectral image technique to detect adulterations in wheat flour and bread with other cheap grains, such as sorghum, oat and corn, and to compare the hyperspectral information with the adulteration repercussions for flour and bread properties.

2. Material and methods

2.1. Flour types and raw materials

The commercial wheat flour (WF) used was obtained from a local producer (Molí de Picó-Harinas Segura S.L. Valencia, Spain), whose chemical composition was: 12.7 ± 0.6% of proteins, 1.0 ± 0.03% of fat, 13.09 ± 0.5% of moisture, and 0.32 ± 0.1% of ash (w.b). The alveographic parameters were also facilitated by the company, which were P = 94 ± 2 (maximum pressure (mm)), L = 128 ± 5 (extensibility (mm)), W = 392 ± 11 (strength (J-4)) and P/L = 0.73. Oat and corn flours were obtained from a local supermarket (La Carabasseta, Valencia, Spain). Their composition was 11.3 ± 0.1% of proteins, 8.0 ± 0.1% of fat, 12.6 ± 0.6% of moisture and 0.92 ± 0.1% of ash (w.b), and 8.3 ± 0.1% of proteins, 2.8 ± 0.1% of fat, 12.89 ± 0.6% of moisture and 0.38 ± 0.1% of ash (w.b), respectively. Sorghum flour was obtained from a commercial bakery (Integral Food S.A. Barcelona, Spain), whose composition was 10 ± 0.1% of proteins, 1.7 ± 0.5% of fat, 12.6 ± 0.6% of moisture and 1.74 ± 0.1% of ash (w.b). In order to maintain particle size homogeneity, grain flours were analyzed and remilled in a stainless steel grinder, whenever necessary (Retsch GmbH, ZM 200, Haan, Germany), until particle size distribution with no significant differences was achieved with the wheat flour used. The particle size of flours was measured 6 times by laser scattering in a Mastersizer 2000 (Malvern, Instruments, UK), equipped with a Scirocco dry powder unit. The results were expressed as a maximum size in μm at 10%, 50% and 90% (d (0.1), d (0.5) and d (0.9), respectively) of the total volume of the analyzed particles as their averages (D [4, 3]). The average results were d (0.1) = 25.5 ± 1.1, d (0.5) = 92.0 ± 0.6, d (0.9) = 180.6 ± 0.8 and D [4, 3] = 99.4 ± 1.2. Having ensured homogeneous particle size, in order to simulate possible adulterations, binary mixes were made by adding different percentages of sorghum, oat or corn to wheat flour. Specifically, adulterations were 2.5, 5, 7.5 and 10% (w/w) of wheat flour with all the different flour grain types.

The other ingredients to make bread were sunflower oil (maximum acidity 0.2° Koipesol Semillas, S.L., Spain), pressed yeast (*Saccharomyces cerevisiae*, Lesafre Ibérica, S.A., Spain), white sugar (≥99.8% of saccharose, Azucarera Ebro, S.L., Spain) and salt (refined marine salt ≥ 97% NaCl Salinera Española S.A., Spain), which were purchased in local stores.

2.2. Bread-making process

The formulation used to prepare bread dough was based on previous works (Verdú, Vásquez, et al., 2015) and was as follows: 56% flour (pure wheat flour or the adulterated versions), 2% refined sunflower oil, 2% commercial pressed yeast, 4% white sugar 1.5% salt and 34.5% water. The process was carried out by mixing all the ingredients in a food mixer (Thermomix® TM31, Vorwerk, Germany) according to the following method: in the first phase, liquid components (water and oil), sugar and salt were mixed for 4 min at 37 °C. Pressed yeast was added in the next phase to be mixed at the same temperature for 30 s. Finally, flour was added and mixed with the other ingredients according to a default bread dough mixing program, which makes homogeneous dough. The program system centers on mixing ingredients with random turns of the mixer helix in both directions (550 revolutions/minute) to obtain homogeneous dough. This process was applied for 4.5 min at 37 °C. Then 450 g of dough were placed in the metal mold (8 × 8 × 30 cm) for fermentation. Height was approximately 1 cm.

Dough fermentation was carried out in a chamber with controlled humidity and temperature (KBF720, Binder, Tuttlingen, Germany). The fermentation process conditions were 37 °C and 90% relative humidity (RH). Samples were fermented for 1 h. The baking process was carried out at the end of fermentation. Metal molds were placed in the middle of the oven (530 × 450 × 340, grill power 1200 W, internal volume 32 L, Rotisserie, DeLonghi, Italy) plate, which was preheated to 180 °C. Baking time was 35 min.

2.3. Crumb conditioning

Having baked the breads, crumbs were extracted and processed to be analyzed as so: crumbs from central bread zones (the middle third of total bread length) were removed from the crust and dried in a food dryer (Excalibur 3900B Deluxe Dehydrator) at 50 °C to obtain the same moisture of raw flours (approx. 13% of moisture). Dried crumbs were milled in a stainless steel grinder (Retsch GmbH, ZM 200, Haan, Germany) until particle size distribution with no significant differences was achieved with the flours used.

2.4. SW-NIR data acquisition and processing

Images of both groups of samples (flours and crumbs from pure wheat and adulterated versions) were taken with a Photonfocus CMOS camera, model MV1-D1312 40gb 12 (Photonfocus AG, Lachen, Switzerland), and using a SpecimImSpector V10 1/2″ filter (Specim Spectral Imaging, LTD., Oulu, Finland), which works as a linear hyperspectral camera. The illuminant was an ASD illuminator reflectance lamp (ASD Inc, Boulder, USA), which produces stable illumination over the full working spectral range. Fifteen grams of sample were placed into a glass Petri dish (10 cm diameter) and a homogeneous surface and height were maintained (approx. 0.75 cm). Spectra were collected directly at room temperature. Four samples of each flour, mix and crumb flours were prepared. Five images of each were acquired by rotating 1.04 radians each time around its normal axis. Twenty image acquisitions of each case were obtained. The position of the illuminant and camera in relation to the sample was always constant in order to control lighting conditions and to obtain a con-