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The use of infrared spectrometers to predict quality parameters of cornmeal (corn grits) and differentiate between organic and conventional practices

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ABSTRACT

Benchtop and handheld NIR and portable mid-infrared (MIR) spectrometers were evaluated as rapid methods for differentiating between organic and conventional cornmeal and to measure quality parameters of cornmeal used for production of snack foods. Twenty-seven conventional and eleven organic cornmeal samples were obtained from a local manufacturer of grain-based products. Reference quality parameters measured included moisture content, ash content, pasting properties and particle size. Soft independent modeling of class analogy (SIMCA) analysis accurately classified between organic and conventional cornmeal samples (interclass distance > 3.7) based on differences in the C=O signal associated with side chain vibrations of acidic amino acids. Residual predictive deviation (RPD) values for partial least squares regression (PLSR) models developed, ranged between 2.3 and 9.6. Overall, our data supports the capability of infrared systems to classify between organic and conventional cornmeal, and to predict important quality attributes of cornmeal for the snack food industry.

1. Introduction

With 7.4% growth rate in 2012, the sales of organic foods in the US were at $27 billion; up from $11 billion in 2004 (USDA, 2013). Organic products have also become more accessible to the general public because they are now sold in mainstream grocery stores instead of only specialty stores. Organic food products in the United States must be certified by the USDA National Organic Program following the U.S. Organic Standards. These standards were created in order to build consumer confidence that the products they were purchasing were indeed organic as well as to sustain and stimulate the growth of the organic industry (USDA, 2012). Premium prices can be charged for organic ingredients, which has led to the growth of certified organic farmland and overall market expansion (USDA, 2012). The regulatory and inspection system that is currently in place may not be sufficient enough to confirm the origin and authenticate organic products. Scientific findings and analytical tools will be needed to ensure the authentication of these organic products.

Cornmeal is a major ingredient to produce various extruded snack products. Along with the processing conditions, the characteristics of the cornmeal also have a great effect on the quality of the final extruded product (Guy, 1994). Properties such as particle size distribution are important features not only for the process but also to achieve the desired physical, mechanical and functional properties in the end product (Guy, 1994). Variations in the particle size of the sample will directly affect the uniformity of the moisture uptake (Onwulata and Konstance, 2006) and therefore the pasting properties of the extrude (Sahai et al., 2001). In applications, fine particle size flour is generally needed to make tortillas for the flexibility and cohesiveness, coarse particle size is required for corn

Abbreviations: ATR, attenuated total reflectance; BKD, breakdown; CV, cross validation; FV, final viscosity; ICD, interclass distance; MIR, mid-infrared; NIR, near infrared; PCA, principal component analysis; PV, peak viscosity; PLSR, partial least squares regression; RPD, residual predictive deviation; RVA, rapid visco analyzer; RVU, rapid visco units; SEP, standard error of prediction; SET, setback; SIMCA, soft independent modeling of class analogies; THR, trough.

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chips and tortilla chips to obtain crispiness after frying (Montemayor and Rubio, 1983).

Testing of incoming cornmeal for organic authentication is lacking. Currently, the cooking properties of starches and flours are often measured using a Rapid Visco-Analyzer (RVA) which constructs a viscoamylograph to assess properties such as final viscosity, peak viscosity, hot and cold paste viscosity or differential scanning calorimetry (DSC). Even though, RVA is a widely used reference method, it takes anywhere from 20 to 30 min to run each sample, making the RVA time-consuming as a quality control tool in real-time. Considering the need of testing many samples for accuracy, a faster method would be more practical to the food companies.

Infrared spectroscopy along with the chemometrics has already been in use for many quality control applications in the food industry, providing valuable information especially in the fingerprint region. Even though there are several studies available regarding the use of infrared spectroscopy (mainly NIR) and pasting characteristics of foods including, rice (Bao et al., 2001, 2007; Chueamchaitrakun et al., 2011; Delwiche et al., 1996) and barley (Cozzolino et al., 2013a,b), there has been no research conducted on the pasting properties of cornmeal. Additionally, the available studies on pasting properties of other grains utilized only benchtop NIR instruments (Bao et al., 2007; Chueamchaitrakun et al., 2011; Delwiche et al., 1996; Cozzolino et al., 2013b) except in one study, a benchtop MIR instrument was used (Cozzolino et al., 2013a). Optical technology is rapidly developing and instruments are now available commercially as portable, handheld and micro-devices, with performance similar to that of benchtop instruments used in laboratories, allowing these systems to be adapted for use in food quality control applications. These devices have been already employed in several research studies successfully for predicting quality parameters of foods such as sucrose levels in infant cereals (Lin et al., 2013).

Since this analytical technique is simple, fast, highly specific and requires minimal or no sample preparation, it can be an ideal alternative for discriminating between organic and conventional cornmeal as well as assessing quality parameters including pasting properties, moisture content, ash content and particle size predictions from a single collected spectrum.

The objective of this study was to develop a simple and rapid method to differentiate between organic and conventional cornmeal and to measure the pasting parameters, moisture content, ash content and particle size of cornmeal using portable MIR, handheld and benchtop NIR spectrometers based on highly specific MIR/NIR spectroscopic signature profiles in combination with supervised pattern recognition techniques.

2. Materials and methods

Twenty-seven conventional and eleven organic cornmeal (corn grits) samples (total of thirty-eight different samples) were obtained from a local manufacturer of corn-based snack food products in Ohio, USA.

2.1. Reference methods

2.1.1. Moisture content

Moisture content of each cornmeal sample was determined according to AOAC Method (AOAC, 2000). Cornmeal ($\approx$2 g) was weighed into an aluminum weigh dish and placed in an Isotemp vacuum oven Model 282A (Fisher Scientific, Springfield, NJ, USA) at 105 °C and 100 mm Hg for 5 h. The samples were placed into a desiccator to cool to room temperature and then weighed to determine the percent moisture of the sample. The moisture content was calculated as percentage based on the loss in weight. The moisture content of each sample was done in duplicate.

2.1.2. Ash content

Ash content of each cornmeal sample was determined according to AOAC Method (AOAC, 2005). Approximately 1 g cornmeal sample was burned at 550 °C for 4 h using an Isotemp muffle furnace (Fisher Scientific, Dubuque, IA, USA). Samples were then cooled in a desiccator, and weighed soon after reaching room temperature. Ash content was calculated by dividing the ash weight to initial weight and then multiplying by 100. The measurements were done in duplicate.

2.1.3. RVA measurements

The pasting properties of each cornmeal sample were determined using a Rapid Visco-Analyzer (RVA, Model 4D, Newport Scientific, Jessup, MD, USA). A programmed heating and cooling cycle was used where the aqueous dispersions of cornmeal samples were prepared by combining 3.5 g cornmeal and 25 g distilled water. Amount of cornmeal and distilled water were corrected based on 14% moisture basis using the previously calculated moisture data for the samples. A combined 28 g cornmeal-water suspension was placed into the RVA and mixed at 960 rpm for 20 s to disperse the sample and to remove the lumps. The rest of the experiment was done at 160 rpm. Sample was initially equilibrated at 50 °C for 2 min, then heated to 95 °C in 4 min, held at 95 °C for 5 min then cooled back down to 50 °C in 5 min. Lastly, it was held at 50 °C for 4 min (total run time of 20 min). Peak time, peak viscosity, pasting temperature, final viscosity and setback values were calculated using the software TCW3 (Thermocline, Perten Instruments, NSW, Australia). Each sample was measured in duplicate.

2.1.4. Particle size

Particle size of cornmeal samples were measured in multi-disperse mode using a Malvern Mastersizer X equipped with a dry powder feeder (Malvern Instruments Ltd., Worcestershire, UK). Obscuration which is defined as percentages of the light intensity absorbed by the sample was maintained between 5 and 20% for all cornmeal samples as recommended by the manufacturer of the instrument for reliable results. Measurements were done in triplicate and particle sizes at volume 25 and 50 percentile were calculated and reported.

2.2. Infrared spectroscopy

2.2.1. Benchtop NIR system

An Excalibur 3500 Fourier-Transform IR spectrometer (Varian, Palo Alto, CA, USA) equipped with a NIR integrating sphere diffuse reflectance accessory (Integrat IR™, Pike Technologies, Madison, WI, USA) was used to collect the NIR spectra of the cornmeal samples. The instrument contained a quartz beam splitter and indium–gallium–arsenide (InGaAs) detector and it was continuously purged with CO_2 free dry air from a CO_2RP140 dryer (Domnick Hunter, Charlotte, NC, USA), since CO_2 has absorption in the IR region and it is important to standardize the CO_2 concentration during spectra collection. Approximately 2 g cornmeal sample was placed in a capped glass vial (Pike Technologies, Madison, WI, USA) and the spectra of samples were collected through the glass. The background spectrum data were collected before each sample with a highly reflective gold-coated reference material. For each cornmeal sample, three spectra were collected over a range of 10,000–4000 cm^{-1} by co-adding 64 scans at a resolution of 4 cm^{-1} and displayed in terms of absorbance. For each replication, fresh cornmeal was placed into the glass cuvette and spectrum was