



# Assessing the structural stability of gluten-free snacks with different dietary fiber contents from adsorption isotherms



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## ABSTRACT

Gluten-free snacks (GFS) with high dietary fiber and relatively low-fat contents were made by combining unripe plantain:chickpea:maize flours in the following proportions 50:30:0; 10:60:30; and 33:33:34, respectively. The adsorption isotherms built from equilibrium moisture content data of the snacks exposed to water activity ( $a_w = 0.10$ – $0.90$ ) at different temperatures (20, 25, 30, 35, 40, or 45 °C) environments, using a dynamic vapor sorption analyzer, were compared to those of a commercial snack control with high-fat content (33.85 g/100 g). All the snacks reached equilibrium moisture content within 5 h for most values of  $a_w$ , and the experimental data were fitted with the Guggenheim-Anderson-de Boer model. The monolayer moisture content and the critical water activity (linked to glassy-to-rubbery phase transitions) of the GFS were significantly lower than that of the commercial snack, indicative that GFS underwent structural weakening under humidity stress. Also, the GFS showed a crossover in the isosteric heat of adsorption (estimated with Clausius-Clayperon equation) at approx. 11% relative moisture content that could be related to the formation of complex multilayered structures. The differences in the adsorption characteristics between GFS and commercial snack can be attributed to the chemical composition, mainly to the fat content.

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## 1. Introduction

An important amount of people (about 1:200 in Europe and 1:240 in USA) is affected by celiac disease (Nijhawan & Goyal, 2015), which is an inflammatory disease of the small intestine that is triggered and maintained by the intake of cereals containing gluten (e.g., barley, rye and wheat). It has been shown that gluten intake for long times can result in damage of small intestine villi (Holtmeier & Caspary, 2006; Kagnoff, 2007). Patients suffering celiac disease show impairments of nutritional status, involving nutritional deficiencies and decreased body weight (Matthias et al., 2011). The health problems related to gluten intake have focused on the development of gluten-free diets. Thus, the development of food products from non-gluten containing flours from botanical sources such rice (Torbica, Hadnadev, & Dapcevic, 2010; Torres, Fradinho, Raymundo, & Sousa, 2014), chestnut (Demirkessen,

Mert, Sumnu, & Sahin, 2010; Torres et al., 2014), soybean (Ribotta et al., 2004) have been considered. Results obtained with non-wheat flours are motivating, although some problems related to dough viscoelasticity and bread texture require further research (Berta, Gmoser, Krona, & Stading, 2015).

On the other hand, gluten-free products usually have low dietary fiber content, and their consumption can lead to overweight and obesity, and to deteriorating health risks (Hager, Axel, & Arendt, 2011). This situation has led to seek the development gluten-free products with high content of indigestible carbohydrates (Sabanis, Lebesi, & Tzia, 2009), including gluten-free bread added with resistant starch (Korus, Witczak, Ziobro, & Juszczak, 2009) and pasta enriched with fiber (Tudorica, Kuri, & Brennan, 2002). Pseudocereals (amaranth, quinoa and kañiwa) have shown to be suitable for combination with corn flour to produce stable snacks in the face of high relative humidity conditions (Diaz et al., 2013). Also, snacks based on non-conventional high dietary fiber containing flours such as unripe plantain, chickpea and maize have been developed, and reported as having low

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amounts of digestible carbohydrates and low glycemic index (Flores-Silva, Rodríguez-Ambríz, & Bello-Pérez, 2015). However, a neglected aspect in the development of dry snacks is the effect produced by relative moisture and temperature on their structural stability.

The structural stability of food matrices plays a central role in the product quality during storage, transportation and sale. Humidity and temperature changes can affect negatively the microstructure of food, and hence the underlying sensorial and nutritional properties. Microbial growth and texture modification might occur during humidity and temperature rise, having adverse effects in the acceptability by consumers. The components present in the product as lipids, proteins, hydrocolloids, carbohydrates (starch, non-starch polysaccharides and sugars) play an important role in the quality characteristics of the products when they are stored. It is well known that starch, sugars and proteins are hydrophilic molecules and they can join water molecules modifying the texture and structural stability of the products (Abdullah, Nawawi, & Othman, 2000). On the other hand, lipids are hydrophobic compounds that drive out water from the food matrix, producing phase separation (Jiménez, Fabra, Talens, & Chiralt, 2013).

Food matrices in glassy amorphous states can remain stable for long periods. The transition into the rubbery state accelerates the food matrix degradation, reflected as product quality and stability loss (Roos, 2007). A suitable approach for establishing the stability of food matrices is based on the thermodynamic properties of water vapor sorption as function of temperature. Sorption characteristics are increasingly used to determine storage stability of dehydrate products, as well as their effect on vapor water diffusion (Carter & Schmidt, 2012). The main concept behind the use of sorption kinetics is that phase transitions from glass to rubbery states can lead to drastic changes in molecular (mainly water) mobility in food matrices.

The glass transition temperature is one of the key physico-chemical parameters for characterizing phase transition from glassy to rubbery states. Indeed, the concept of glass transition temperature has been useful for understanding the processing, quality and stability of low-moisture foods (Yuan, Carter, & Schmidt, 2011). Valuable efforts have been carried out to link departures from the glass transition temperature to events of stability loss of amorphous food matrices (Roos & Drusch, 2015). On the other hand, most approaches for estimating the glass transition temperature are based on detecting changes in mechanical, dielectric and thermodynamic properties while scanning temperature at constant moisture content. However, temperature is rarely the central variable to be considered for dehydrated food storage. Given that moisture is the main food plasticizer, it has been argued that increased moisture content is the variable that can lead to glass transition events even while temperature is held constant (Roos & Drusch, 2015). In this way, a critical water activity leading to glass transition is a more suitable parameter for characterization of amorphous food matrices. This parameter can be estimated by scanning water activity under constant temperature conditions. More detailed information (e.g., multilayer number) can be obtained by fitting experimental data with Guggenheim-Anderson-De Boer (GAB) model.

The aim of this study was to: (i) produce gluten-free snacks with high dietary fiber contents; (ii) obtain the water adsorption isotherms over a wide water activity range at different temperatures; (iii) determine the critical water activity as an indicative of the phase transition from glassy to rubbery states at a given temperature, and of the isosteric heat as indicative of the binding strength of water molecules on the snack surface; (iv) compare the results with those obtained for a commercial snack.

## 2. Materials and methods

### 2.1. Sample preparation

Traditional commercial snack (CS; Fritos® Chorizo y Chipotle, Sabritas, Mexico City, Mexico) was purchased in a local supermarket. The CS is made of a combination of nixtamalized maize flour, maltodextrin and wheat flour, and was chosen as the control. The fabrication of this snack is carried out under a three step process: extrusion, drying and deep-frying. This three step process was used in the fabrication of the experimental snacks studied in this work. Three gluten-free snacks with high dietary fiber and relatively low-fat (GFS) contents were made by combining unripe plantain:chickpea:maize flours in the following proportions 50:30:20; 10:60:30; and 33:33:34, and the formulations were coded as Snack A, Snack B, and Snack C, respectively. Starch was dispersed in water (200 g/1000 g) by gentle stirring, and letting to stand for 24 h for achieving full hydration. To this end, a single screw laboratory extruder (Beutelspacher, S.A. de C.V., Mexico City, Mexico) was used at constant screw speed of 75 rpm. The temperature in the feed zone was of  $75 \pm 2$  °C,  $114 \pm 4$  °C in the blending zone, and of  $100 \pm 2$  °C at the end zone. The resulting pellets were oven dried (Biotechnica del Bajío, Celaya, State of Guanajuato, Mexico) at 45 °C for 3 h, cooled down to room temperature, and stored in sealed plastic containers. Prior to each determination, the pellets were subjected to deep-frying on a fryer (T-43 Deep Fryer, Moulinex) during 10 s at 180 °C.

### 2.2. Chemical composition of snacks

The GFS and CS were ground in a household coffee grinder (Krups F203, Groupe Seb Mexico, Mexico City, Mexico). The approved methods of analysis of the AACC (2000) were used for determining moisture (44–15.02), ash (08–01.01), protein (46–13.03), fat (30–25.01), dietary fiber (32–05.01), and total starch (76–13.01) contents. All analyses were carried out in triplicate.

### 2.3. Adsorption isotherms

Moisture adsorption isotherms of GFS and CS were determined with a dynamic vapor sorption (DVS) automated gravimetric sorption system (model DVS-1, Quantachrome Instruments, FL, USA). The instrument is equipped controlled atmosphere ultra-sensitive microbalance, which can detect changes in the sample mass during adsorption-desorption phenomena. The required relative humidity is generated by mixing dry nitrogen and distilled water in the corresponding proportions using mass flow controllers and a calibrated humidity probe.  $25 \pm 1$  mg of ground sample was weighed and placed in the microbalance and dried to a zero relative humidity ambient by purging with ultra high purity nitrogen. The change in sample weight was continuously monitored in real time with help of the Aquawin 1.11 software, when exposed to 0.10 to 0.90 of relative activity at 20, 25, 30, 35, 40 or 45 °C environments. The equilibrium moisture content (EMC) values used to build the isotherm were recorded. Equilibrium was considered to have been reached when the change in the sample mass was less than  $0.001 \text{ mg min}^{-1}$ .

### 2.4. Guggenheim, Anderson and de Boer (GAB) model

Adsorption isotherms for moisture content are commonly described by the GAB model, which accounts for multilayer formation on homogeneous surfaces. The GAB equation is an extension of the well-known two-parameter BET model for adsorption

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