



# Moisture sorption isotherms of high pressure treated fruit peels used as dietary fiber sources

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## ABSTRACT

High hydrostatic pressure (HHP) treatments can improve the potential of orange, mango, and prickly pear peels as food formulation fiber sources. Akaike Information Criteria differences identified Peleg and GAB as the best model alternatives to describe experimental moisture isotherms. HHP (600 MPa/10 min/22 and 55 °C) effects on moisture isotherms expressed as relative water sorption content change with respect to controls ( $RWSC_{aw}$ ) showed that in the 0.1–0.93  $a_w$  range, HHP improved the adsorption water retention of orange peels. The same was true for the desorption water retention for all HHP-treated fruit peels except for prickly pear HHP-treated at 22 °C and  $> 0.35 a_w$ . The area under the hysteresis curve ( $A_H$ ) in the 0.15–0.51  $a_w$  range showed that HHP increased hysteresis for all fruit peels tested. All this illustrates the HHP potential to modify the hygroscopic properties of fruit peels at lower temperature and in less processing time than conventional processes.

**Industrial relevance:** Orange, mango, and prickly pear peels are potential food fiber formulation sources with differentiated hygroscopic and functional properties. In this study, 600 MPa treatments at 22 and 55 °C for 10 min modified the adsorption and desorption moisture retention capacity of all fruit peels tested in this study. HHP technology can improve the potential of fruit peels as dietary fiber sources with the advantage of shorter processing times and lower temperatures than conventional technologies used to treat food fibers.

## 1. Introduction

In Latin America alone, the food industry generates ~56 million tons of waste from the production, processing, handling and storage of fruits and vegetables (Santivañez, 2014). These worldwide-generated waste materials, including peel, cores, pips, kernels, skins, and stems, are potential sources of functional ingredients such as dietary fiber (DF). By-products from apple, grape, lemon, mango, orange, and other fruits have been used as non-conventional DF sources (O'Shea, Arendt, & Gallagher, 2012; O'Shea et al., 2015; Tejada-Ortigoza, Garcia-Amezquita, Serna-Saldívar, & Welti-Chanes, 2015). However, thermal (Benítez et al., 2011), chemical (Šoronja-Simović et al., 2016), enzymatic (Laurikainen, Härkönen, Autio, & Poutanen, 1998), and other processes (Chau, Wang, & Wen, 2007; Chen, Gao, Yang, & Gao, 2013; Zhu, Du, Li, & Li, 2014) are required to extract DFs, modify their technological functionality and convert these residues into food ingredients with desirable properties. These treatments modify their properties including their total (TDF), soluble (SDF) and insoluble (IDF) dietary fiber content.

Treating onion bagasse at 115 °C for 31 min increased the SDF content from 8.8 to 10.5% (db) while the relationship SDF/TDF increased from 24.4 to 30.6% (Benítez et al., 2011). Chemical modification of sugar beet fibers dried at 55 °C using H<sub>2</sub>O<sub>2</sub> (30% v/v), NaOH (10 M) and HCl (35% v/v) for ~24 h increased the SDF content and the DF/TDF from 13.9 and 18.7% (db) for untreated to 17.6 to 23.9% (db) for treated samples, respectively (Šoronja-Simović et al., 2016). As previously reviewed (Tejada-Ortigoza et al., 2015), chemical modification methods involve the use of large amount of solvents, long processing time (0.5–30 h), and high temperature (60–100 °C). This has raised an interest in non-thermal technologies, including high hydrostatic pressure (HHP), as an alternative for DF modifications. For example, Tejada-Ortigoza, Garcia-Amezquita, Serna-Saldívar, & Welti-Chanes, (2017) showed that the DF content is affected by HHP treatments and that the modifications observed varied with the food matrix. Treating mango and orange peel samples for 10 min at 600 MPa and 55 °C increased the SDF content from 15.2 to 17.5% (db) and from 3.7 to 5.9% (db), respectively. In addition, the SDF/TDF ratio increased from 37.4 and 7.3% (db) for untreated mango and orange peels to 42.2

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and 10.8% (db), and to 45.0 and 12.0% (db), when treated at 600 MPa at 22 and 55 °C, respectively. On the other hand, 10 min treatments at 600 MPa of prickly pear peel increased the IDF content from 32.6% (db) for untreated samples to 35.5 and 38.1% (db) for HHP treatments at 22 and 55 °C, respectively. In the case of prickly pear peel, TDF content increased up to 4.8 and 5.7% (db) after HHP treatments at 22 and 55 °C, respectively (Tejada-Ortigoza et al., 2017). Treatments at 200 and 400 MPa at 60 °C for 15 min increased okara's SDF content about 2.8 and 8.1 times, respectively (Mateos-Aparicio, Mateos-Peinado, & Rupérez, 2010). Under these conditions, the SDF/TDF ratio increased from 4.6% (db) for untreated okara to 11.1 and 37.2% (db), respectively. When aided by 0.025% of the food grade enzyme Ultraflo®, 30 min treatments at 600 MPa and 40 °C increased the okara total soluble carbohydrate content from 9 to 15.6% (db) (Pérez-López, Mateos-Aparicio, & Rupérez, 2016). Conversely, a 500 MPa treatment at 20 °C reduced the SDF content of untreated white cabbage from 7.6 to 5.9% (db) (Wennberg & Nyman, 2004).

The review of previous work shows that HHP treatments can change the content and properties of fruit peels DF and could widen their range of technological applications. Sorption isotherms are a potentially useful tool to analyze fruit peel modifications induced by HHP treatments since hygroscopic properties of food materials depend on their SDF and IDF content and SDF/TDF ratio. The relationship between equilibrium moisture content (EMC) and water activity ( $a_w$ ) at constant temperature and under equilibrium conditions is graphically expressed as moisture isotherm curves. Moisture sorption phenomena are not fully reversible and the EMC difference between adsorption and desorption values is known as hysteresis Al-Muhtaseb, McMinn, & Magee, 2004. Mathematical models used to describe and analyze water sorption properties of foods include those based on theoretical concepts such as the monolayer moisture value (BET, GAB), or they are semi-empirical (Iglesias and Chirife), and empirical expressions (Oswin, Kühn, Peleg). Standard statistical criteria such as  $R^2$  are not always able to determine the best model to describe experimental data. In such cases, statistic tools such as the Akaike Information Criteria (AIC) can be used to evaluate the goodness of fit while penalizing the use of an excessive number of parameters. AIC determines, among a set of models, which one is the best to use by supplying information on the strength of the evidence for each model and considering the information lost when the model is used to approximate experimental data (Posada & Buckley, 2004; Serment-Moreno, Fuentes, Barbosa-Cánovas, Torres, & Welti-Chanes, 2015).

A few studies have reported the effect of HHP treatments on the hygroscopic properties of treated materials. Increasing the pressure holding time at 20 °C of 300 MPa treatments of maize starch suspensions have been shown to shift isotherm curves to higher EMC values and increase the hysteresis effect at  $a_w > 0.44$  (Santos et al., 2014). Conversely, 15 min treatments at 300 and 500 MPa of a starch solution at 20 °C showed that increasing the pressure level decreased the hysteresis effect (Santos et al., 2014). In the case of brown rice, 300 MPa treatments had lower EMC values than samples treated at higher pressure (400, 500 MPa) (Yu et al., 2015). The authors stated that HHP treatments might have cracked the brown rice kernels resulting in the higher EMC value observed at higher pressure. In addition to HHP treatment effects, changes induced by homogenization, osmotic dehydration and convection drying in the EMC and GAB parameters of food materials have been evaluated too. Homogenization (8000 rpm, 3 min) affected the tissue structure of freeze dried strawberry reducing its EMC by 5 g water/100 g ds at  $a_w \sim 0.6$  (Moraga, & Martínez-Navarrete, N., & Chiralt, A., 2004). Osmotic dehydration in 25 g/L calcium lactate solution and convection drying (70 °C/10 g/kg dry air, 0.5 m/s) treatments lowered papaya  $M_0$  values from 37.5 to 13.1 and 11.5 g water/100 g ds, respectively. These reductions were attributed to changes in sugar content due to the osmotic dehydration and structural modifications caused by convection drying (Udomkun, Argyropoulos, Nagle, Mahayothee, & Müller, 2015). In general, structure and composition changes are responsible for the modifications of the moisture

sorption capability of DF sources. Changes in the hygroscopic properties of HHP-treated fruit peels would allow explain their functionality as food ingredients and their role in digestive processes and other health benefits of DFs (Gurak, De Bona, Tessaro, & Marczak, 2014). The analysis of hygroscopic properties based on sorption isotherms is needed to evaluate modifications of the water adsorption or desorption capacity of fruit peels treated by HHP. Therefore, this study focuses on evaluating the effect of HHP treatments at room and moderate temperature on the moisture isotherms of mango, orange, and prickly pear peels.

## 2. Material and methods

### 2.1. Fruit peel samples

Mango (*Mangifera indica* L. cv Ataulfo), orange (*Citrus sinensis* L.) and prickly pear (*Opuntia ficus-indica* cv Verde Villanueva) purchased at a local supermarket (Monterrey, N.L., Mexico) were immersed 10 min in chlorinated water (15 ppm). Fruit peel was then manually removed, ground (VM0103, Vitamix, Cleveland, OH), vacuum packed (model EVD 4; Torrey, Monterrey, N.L., Mexico) in 12 × 15 cm polyethylene bags (Filmpack SA de CV, Guadalupe, N.L., Mexico) and stored at 4 °C until use within 2 h.

### 2.2. High hydrostatic pressure (HHP) processing of fruit peel

Polyethylene pouches (~11 × 7 × 1 cm) with ~50 g peel samples were treated in duplicate runs at 600 MPa for 10 min (2 L Welch food processor, Avure Technologies, Middletown, OH) using water at 22 or 55 °C as the pressurizing fluid and reaching during pressure holding an average temperature of 30.5 ± 1.4 and 65.9 ± 2.5 °C, respectively. A 430 MPa min<sup>-1</sup> compression rate yielded a 1.4 min come-up time (CUT) to 600 MPa while decompression was almost instantaneous (< 5 s). Samples were stored at -80 °C immediately after processing and used within 8 weeks.

### 2.3. Moisture sorption isotherms

Untreated (control) and HHP-treated peel samples were freeze-dried at -50 °C and 2.0 mbar (Labconco, Kansas City, MO), hand-milled, sieved through a mesh number 40 (425 µm), and stored in desiccators containing P<sub>2</sub>O<sub>5</sub> (25 °C) at least for 5 days before analysis. Initial moisture content was determined as specified by AOAC Official Method 920.151. Duplicate adsorption and desorption moisture isotherms in the ~0.10 to 0.93  $a_w$  range were determined at 30 °C with ± 0.0001  $a_w$  resolution following the device manufacturer's instructions (Aqasorp Isotherm Generator, Decagon Devices Inc., Pullman, WA).

### 2.4. Isotherm modelling

The GAB, Iglesias and Chirife, Oswin, Kühn and Peleg models (Eqs. 1–5, Table 1) previously reported for similar materials were used to describe moisture sorption isotherm data (Caballero-Cerón, Guerrero-Beltrán, Mújica-Paz, Torres, & Welti-Chanes, 2015). Parameters for each model were estimated using the least-square method and the Microsoft Excel solver function. Model fit was evaluated based on values for the coefficient of determination ( $R^2$ , Eq. (6)) and residual sums of squares (SSE, Eq. (7)), where  $y_i$  and  $\hat{y}_i$  are the moisture experimental observation and estimated value, respectively.

$$R^2 = 1 - \frac{\sum (y_i - \hat{y}_i)^2}{\sum (y_i - \bar{y}_i)^2} \quad (6)$$

$$SSE = \sum (y_i - \hat{y}_i)^2 \quad (7)$$

Model selection was performed with the AIC (Eq. (8)), which evaluates the goodness of fit (first term) and penalizes the excessive use of

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