

# Valorization of postharvest sweet cherry discard for the development of dehydrated fruit ingredients: compositional, physical, and mechanical properties

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## Abstract

**BACKGROUND:** Sweet cherries are an excellent source of phenolic compounds, which may contribute to a healthy diet. The objective of this work was to generate dehydrated ingredients from postharvest discard of sweet cherries.

**RESULTS:** Four dried ingredients were obtained from fresh sweet cherry discard (Lapins var.) using an osmotic dehydration pretreatment and freeze drying or air drying. The ingredients showed an important phenolic contribution (2.8–6.6 g gallic acid kg<sup>-1</sup> of product) and preserved the natural color of the fruit to a great extent. Freeze-dried ingredients were less hygroscopic than air-dried ones, and presented with a softer texture. All the ingredients were in a supercooled state at room temperature ( $T_g$  range: –23.0 to –18.8 °C). Sugar infusion pretreatment caused a decrease in water sorption capacity and molecular mobility; it also reduced the initial rehydration rate.

**CONCLUSION:** Relevant differences in nutritional and structural characteristics of the ingredients were observed depending on the processing method used. These ingredients could be incorporated into different processed foods, such as snacks, cereal mixtures, cereal bars, and bakery and confectionery products. Air-dried control ingredients presented better nutritional qualities and air-dried sweet cherries with sugar infusion pretreatment could be appropriate ingredients for applications where sweet flavor and slow rehydration rate are required.

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**Keywords:** sweet cherries; osmotic dehydration; air-drying; freeze-drying; physical and mechanical properties

## INTRODUCTION

Fruits are valuable foods that contribute to a balanced and healthy diet. Sweet cherry has become an important fruit worldwide due to its organoleptic characteristics such as color, sweetness, and sourness.<sup>1</sup> This fruit is also considered an excellent source of phenolic compounds. This is nutritionally relevant because of their health-promoting properties.<sup>2</sup> In particular, the high phenolic content of the Lapins cultivar is promising for the development of functional foods.<sup>3</sup> Traditionally, the largest volume of the sweet cherries that are produced are destined to be consumed fresh. However, the amount of discarded fruit is significant due to consumer demand. Only 60% of total cherry production meets market specifications and can be sold into the fresh market.<sup>4</sup> Indeed, any small defect presented by the fruit, such as small calibers, cracking, pitting, double fruits, peduncle loss, or peduncle browning causes its systematic rejection.<sup>5</sup> To take advantage of these discarded cherries it is therefore necessary to apply preservation methods to extend shelf-life and generate new high-quality products, emphasizing their nutritional value. Different dehydration methods could be employed here to obtain new dried products, at the same time adding value to fruit that would otherwise not

be marketable. Hot air-drying is the most commonly used dehydration method; however, heat exposure substantially affects fruit quality, causing damage to essential attributes like color, flavor, texture, nutrients, and rehydration capacity.<sup>6</sup> Water transport from the solid matrix causes changes in mechanical and structural properties, resulting in material with reduced molecular mobility due to low water content and a high concentration of solids. The phenomenon of volumetric shrinkage that occurs during drying also causes a change in food size and shape.<sup>7</sup> On the other hand, freeze drying usually offers higher quality food due to minimal shrinkage, high rehydration capacity, and nutrient retention in the dried product. However, in some cases, during the desorption stage

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of the freeze-drying process, structural collapse can also occur, causing the closure of pores and reducing rehydration capacity and swelling.<sup>8</sup> This phenomenon affects the rehydration capacity of dried products, the rate and extent of the rehydration being strictly related to the duration and severity of the previous dehydration process.<sup>9</sup> If the volumetric shrinkage is minimal, the presence of well defined intercellular holes may increase the rate of rehydration.<sup>7</sup> The application of osmotic dehydration as pretreatment before drying causes partial removal of water from the tissues together with the entry of solutes,<sup>10</sup> with a high impact on quality parameters, mainly in terms of color, volume, texture, and nutrients.

The objective of the present work was to analyze global quality, through evaluation of nutritional, physical, mechanical, rehydration and sorption properties, of four sweet cherry ingredients obtained by freeze drying or air drying, with or without the application of osmotic dehydration pretreatment.

## MATERIALS AND METHODS

### Fruit characterization

The postharvest discard of sweet cherries (*Prunus avium*, cv. Lapins) was purchased from 'Talzauber' Farm (Neuquén, Patagonia, Argentina). The fruit was characterized ( $n = 3$ ) according to AOAC methods<sup>11</sup> showing the following results: water content =  $785 \pm 8 \text{ g kg}^{-1}$ , total soluble solids =  $20.9 \pm 1.4$  Brix, pH =  $3.50 \pm 0.06$ , total acidity expressed as citric acid =  $7.86 \pm 0.12 \text{ g kg}^{-1}$  and ash =  $4.94 \pm 0.05 \text{ g kg}^{-1}$ .

### Ingredient preparation

To obtain dried ingredients, sweet cherries were conditioned (this entailed washing, removing peduncles and defective parts, and manual pitting), and were then cut into eight pieces. Immediately, one group was subjected to dry sugar infusion and another one without pretreatment was dried to obtain control samples (C). Dry sugar infusion (SI) pretreatment was carried out by immersing the fruits at 25 °C into a dry mixture of sucrose and preservatives. The amount of sucrose was calculated for 1 kg fruit to reduce  $a_w$  to 0.87 using the method described in Franceschinis *et al.*<sup>12</sup> The fruit/sugar ratio was 0.7 and potassium sorbate (100 mg/kg of food system) and sodium bisulfite (150 mg/kg of food system) were used as antimicrobial and anti-browning agents. The fruits and the syrup generated during the treatment were carefully mixed twice a day until the equilibration of food system components was reached after 15 days. Then, samples were taken out of the syrups and the residual syrup was removed from the surfaces. Reagents were all food grade (obtained from Saporiti S.A., Buenos Aires, Argentina). Different drying processes (air drying and freeze drying) were then applied to reach a final water activity of 0.33. For air drying (A), an air convection oven at  $60 \pm 1 \text{ °C}$ ,  $\cong 10\%$  relative humidity (RH), and air speed of  $1.5 \text{ m s}^{-1}$  was used for 24 h. Relative humidity was controlled with a Hygro Palm hygrometer (Rotronic Instruments, Crawley, West Sussex, UK). The freeze-drying (F) process lasted 48 h and was carried out in a freeze drier Alpha 1–4 LD/2–4 LD-2 (Martin Christ, Gefriertrocknungsanlagen GmbH, Osterode, Germany). It was operated at  $-84 \text{ °C}$  at a chamber pressure of 0.04 mbar. Previously, samples were quenched with liquid nitrogen and stored at  $-18 \text{ °C}$  for at least 48 h.

### Water content and water activity

The water content (WC) was determined gravimetrically after vacuum drying at 60 °C in the presence of desiccant. The water

activity ( $a_w$ ) was determined at  $25 \pm 1 \text{ °C}$  by dew point using an Aqualab Series 3 TE (Decagon Devices, Pullman, WA, USA). Determinations were made in triplicate.

### Total sugar, bioactive compounds, and antioxidant capacity

Total sugar content (TS), total phenolic compound content (TPC) and monomeric anthocyanin content (ACY) were determined on ethanolic extracts (80%) using the method described in Franceschinis *et al.*<sup>12</sup> An anthrone/sulfuric acid procedure and glucose (GLU) as standard were used for TS. Folin–Ciocalteu reagent was used to estimate TPC, and gallic acid (GA) was employed as standard. Monomeric anthocyanin content was quantified using the pH-differential method and expressed as cyanidin-3-glucoside (MW: 445.2 and a molar extinction coefficient =  $29\,600 \text{ L cm}^{-1} \text{ mol}^{-1}$ ) per kg of ingredient. The antioxidant capacity was determined according to Sette *et al.*<sup>13</sup> by two different methods: antiradical power (ARP) by using the bleaching method of the radical 1,1-diphenyl-2-picrylhydrazyl (DPPH $\cdot$ ) and the ferric ion reducing ability (FRAP). Antiradical power was defined as the inverse of  $EC_{50}$  and corresponds to the concentration that scavenged 50% of the radicals. For the FRAP method, measurements were made at 30 min and a calibration curve with  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  was used. All determinations were done in triplicate.

### Superficial color

The superficial color was evaluated by photocolometry, in the CIELAB color space; with C illuminant and 2° observer using a Minolta photocolometer (model CR 400). Sweet cherry ingredients were placed on two petri dishes and measurements were done on eight different points of each dish ( $n = 16$ ). The  $L^*a^*b^*$  parameters were used to calculate the following color functions: 'chroma' ( $C^*_{ab}$ ), 'hue angle' ( $h_{ab}$ ) and 'global color change' ( $\Delta E^*_{ab}$ ).<sup>12</sup>

### Bulk density, shrinkage, and hygroscopicity

Due to the irregular form of the sweet cherry products, the bulk density and volume were determined by a toluene displacement technique using a pycnometer for solid samples.

The bulk density ( $\delta_b$ ), was calculated as:

$$\delta_b = \frac{W_s}{W_t + W_s - W_{s+t}} \times \delta_t \quad (1)$$

where  $\delta_t$  is toluene density, and  $W_t$ ,  $W_s$ ,  $W_{s+t}$  are the weights of the pycnometer filled with toluene, sample, and pycnometer with sample and toluene, respectively. Measurements were taken at room temperature using an analytical balance with an accuracy of  $10^{-4} \text{ g}$  (Ohaus Corporation, Parsippany, USA) and the density of toluene was corrected by temperature. Shrinkage (S) was evaluated as volume reduction percentage, and hygroscopicity (H) was analyzed by exposing the ingredients to a 75% RH. Both determinations were done in triplicate according to procedures described by Sette *et al.*<sup>7</sup>

### Water sorption isotherms

The water sorption isotherms of dried sweet cherries were determined by the static isopiestic method ( $n = 3$ ). The humidification of samples was performed at 25 °C. Approximately 2 g of ingredients were put into vacuum desiccators over saturated salt solutions from 11% to 90%.<sup>14</sup> 'Equilibrium' was considered at constant weight of samples. Three sorption isotherm equations were

used to fit experimental data: Guggenheim-Anderson-de Boer (GAB) (Eq. 2), Oswin (Eq. 3), and Halsey (Eq. 4) models. Model parameters were estimated using the non-linear regression procedure employing OriginPro 8 software.

$$X = X_0 \times \frac{(C \times K \times a_w)}{((1 - K \times a_w) \times (1 - K \times a_w + C \times K \times a_w))} \quad (2)$$

$$X = A \left( \frac{a_w}{1 - a_w} \right)^B \quad (3)$$

$$X = \left[ \frac{A}{\ln(1/a_w)} \right]^{\frac{1}{B}} \quad (4)$$

where  $X$  = water content (kg/kg db);  $X_0$  = monomolecular water content;  $a_w$  = water activity;  $C$  = Guggenheim constant related to the sorption heat of the first layer;  $K$  = is a factor related to the sorption heat of the multilayer;  $A$ ,  $B$  = model constants and characteristics of each food.

To select the best correlation and the goodness of fit of each sorption model to the experimental curve, the coefficient of determination ( $R^2$ ) and mean relative percentage deviation modulus ( $E\%$ ) were calculated. This modulus is defined as follows:

$$E\% = \frac{100}{N} \sum_{i=1}^N \frac{|X_{\text{exp}} - X_{\text{cal}}|}{X_{\text{exp}}} \quad (5)$$

where  $N$  is the number of experimental data,  $X_{\text{exp}}$  are water content experimental values, and  $X_{\text{cal}}$  are water content predicted values.

### Glass transitions

Glass transitions were determined by differential scanning calorimetry (DSC; onset values) using a calorimeter model 822e (Mettler Toledo, Schwerzenbach, Switzerland) according to Sosa *et al.*<sup>15</sup>

### Molecular mobility

A pulsed nuclear magnetic resonance (1H NMR) Bruker Minispec instrument model mq 20 (Bruker Biospin GmbH, Rheinstetten, Germany) with a 0.47 T magnetic field operating at resonance frequency of 20 MHz was used for measurements as described by Sette *et al.*<sup>7</sup> Equilibrated samples ( $n = 3$ ) at 11, 22 and 33% RH were removed from the desiccators, placed into 10 mm diameter glass tubes (4 cm height), and returned to the desiccators for an additional time of 24 h before analysis. Before measurements, samples were tempered in a range of 20–35 °C ± 0.01 °C using a water thermostatic bath (Haake Phoenix II C35P, Darmstadt, Germany).

### Mechanical properties

Compression-shear tests ( $n = 10$ ) using a Kramer shear press with five blades were performed with a universal assay instrument model 3344 (Instron Corporation, Canton, MA, USA). A 50 kN load cell was used and the crosshead speed for all tests was 20 mm/min. Samples ( $4 \pm 0.1$  g) were randomly disposed into the Kramer shear press cavity (55 × mm) to obtain a single layer. Force–deformation curves were recorded using the Instron Bluehill Material Testing Software, and the following parameters were evaluated: maximum force ( $F_{\text{max}}$ , N), deformation or distance at maximum force ( $\Delta F_{\text{max}}$ , mm), maximum slope of peak before maximum force ( $SI_{\text{max}}$ , N/mm), and energy work or peak area up to the maximum force ( $W$ , J).

### Rehydration

Experiments ( $n = 2$ ) were determined by recording the weight evolution over time of approximately 5 g of ingredients immersed in 100 mL distilled water at 25 ± 0.1 °C. Superficial water excess was drained under vacuum for 1 min using a Büchner funnel connected to a Kitasato flask. The procedure was repeated until a plateau was reached and/or a decrease in weight gain occurred.

The rehydration capacity  $CR$  (% w/w) was defined as follows:<sup>16</sup>

$$CR = \frac{W_r}{W_d} \times 100 \quad (6)$$

where  $W_r$  and  $W_d$  were the weight of the rehydrated and dried sample, respectively.

The rehydration kinetics was modeled using the non-exponential equation proposed by Peleg:<sup>17</sup>

$$X = X_0 + \frac{t}{(k_1 + k_2 \times t)} \quad (7)$$

and, at  $t = 0$

$$\frac{dX}{dt} = \frac{1}{k_1} \quad (8)$$

at  $t \rightarrow \infty$

$$X_e = X_0 + \frac{1}{k_2} \quad (9)$$

where  $X$  is the water content on dry basis (db) at the different times of the process  $t$  (min);  $X_0$  is the initial water content in the sample,  $X_e$  is the water content at equilibrium,  $t$  is the time of the process,  $k_1$  is a kinetic parameter, and  $k_2$  is a parameter related to equilibrium moisture content.

### Statistical analysis

The experimental design was completely randomized. The results were expressed by the mean and standard deviation (SD). A factorial analysis of variance was performed considering two factors: 'pretreatment' and 'drying method.' Multiple comparisons were carried out using the Tukey test at  $P < 0.05$ , for the interaction of main effects according to significance. All statistical analyses were carried out using Statistica version 8.0 (StatSoft, Inc., Tulsa, OK, USA).

## RESULTS AND DISCUSSION

Table 1 shows the water content, total sugars, total phenolic compounds, monomeric anthocyanin content and antioxidant capacity of ingredients. The dehydration processes were performed in order to reach the same water activity ( $a_w = 0.33$ ). However, some differences in the final water content were observed depending on the processing method (there was a significant factor interaction): ingredient FC demonstrated higher water content than AC, and the SI pretreatment caused a reduction in water content. Giovanelli *et al.*<sup>18</sup> reported a similar behavior, observing lower residual moisture at the end of air drying for whole blueberries pretreated with an osmotic dehydration with sucrose, when compared to their respective control.

Upon dehydration, sugars, phenolic compounds, and monomeric anthocyanins were concentrated, showing much higher values than those for the fresh sweet cherry (TS: 130 ± 6 g GLU kg<sup>-1</sup>; TPC: 1.14 ± 0.13 g GA kg<sup>-1</sup>, ACY: 430 ± 5 mg Cyd-3-glu kg<sup>-1</sup>). The SI pretreatment caused changes in the fruit composition

**Table 1.** Water content (WC), total sugar content (TS), total phenolic compound content (TPC), monomeric anthocyanin content (ACY), antiradical power (ARP) and ferric ion reducing ability (FRAP) of dried sweet cherry ingredients

Ingredients	WC g H <sub>2</sub> O kg <sup>-1</sup>	TS g GLU kg <sup>-1</sup>	TPC g GA kg <sup>-1</sup>	ACY mg Cyd-3-glu kg <sup>-1</sup>	ARP 1/EC <sub>50</sub> kg <sup>-1</sup>	FRAP mM Fe <sup>2+</sup> kg <sup>-1</sup>
AC	86.4 ± 0.5 <sup>c</sup>	611 ± 15 <sup>A,a</sup>	6.6 ± 0.4 <sup>c</sup>	1152 ± 10 <sup>c</sup>	345 ± 11 <sup>c</sup>	47 ± 3 <sup>B,b</sup>
ASI	45.2 ± 0.3 <sup>a</sup>	778 ± 35 <sup>A,b</sup>	3.4 ± 0.03 <sup>a</sup>	222 ± 29 <sup>a</sup>	156 ± 4 <sup>a</sup>	27 ± 2 <sup>B,a</sup>
FC	93.4 ± 1.3 <sup>d</sup>	650 ± 20 <sup>A,a</sup>	5.12 ± 0.16 <sup>b</sup>	2033 ± 29 <sup>d</sup>	244 ± 19 <sup>b</sup>	38.6 ± 1.2 <sup>A,b</sup>
FSI	76.2 ± 0.5 <sup>b</sup>	852 ± 81 <sup>A,b</sup>	2.79 ± 0.09 <sup>a</sup>	773 ± 45 <sup>b</sup>	220 ± 13 <sup>b</sup>	23.3 ± 1.8 <sup>A,a</sup>
Pretreatment × drying*	S	NS	S	S	S	NS

Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.  
Means (*n* = 3) with the same letter superscript were not significantly different (*P* < 0.05). Uppercase letters and lowercase letters were used for main effect of factors: 'drying method' and 'pretreatment', respectively.  
\*Interaction factor: S (significant), NS (not significant).

because a pronounced increase in sugar content and a loss of phenolic compounds and anthocyanin pigments occurred. During SI, plant cellular structure acts as a semi-permeable membrane, and the components are transferred by a process usually considered as diffusion driven. The migration of water-soluble substances contained in the vacuoles towards the surrounding medium therefore takes place because the syrup formed during the SI consists in a hypertonic solution composed mainly by sucrose, which is diluted throughout time as a result of the sugar intake and the water loss exhibited by the fruits. This phenomenon was previously reported for sweet cherry pieces,<sup>12</sup> various berries,<sup>13,19–21</sup> and other fruits.<sup>22</sup> It is interesting to note that despite the decrease in bioactive compounds (phenolic and anthocyanin) from IS pretreatment, the phenolic contribution to the antioxidant capacity of the ingredients is still considerable. The higher retention of anthocyanin from ingredient FC did not result in a higher antioxidant capacity. Ingredient AC presented the highest antiradical capacity in agreement with the highest phenolic content. Air-dried ingredients showed a greater ability to reduce Fe<sup>2+</sup>, while a decrease was produced by IS pretreatment. Since better results in terms of phytochemical content and antioxidant capacity were obtained in ingredients subjected to air-drying without pretreatment (AC), the implementation of freeze-drying would not be convenient due to the high costs associated with the process.

The color change of a food product during dehydration is indicative of the severity of the drying conditions and may be related to the composition/concentration of pigments, as well as to their degradation under those conditions. Figure 1 shows photographs of the dried sweet cherry ingredients, which in general presented a similar appearance. Ingredients with SI pretreatment showed slightly higher L\* values (Table 2) and a more glossy appearance due to sugar incorporation and migration of pigments to the osmotic syrup. As for the chromatic coordinates, both chroma and hue angle showed main effects for the factors that were studied. Freeze-dried ingredients presented lower hue values, indicating a redder tone compared to air-dried cherries, which showed a more brownish appearance – probably due to the occurrence of non-enzymatic browning during the drying process at high temperature. On the other hand, SI pretreatment caused an increase in both chromatic values, showing a slight increase in hue angles towards the yellowish region of the color wheel (Table 2). All the ingredients presented very similar values of global color change, and only small differences due to drying method and SI pretreatment were detected. Taking into account that the unit of ΔE\*<sub>ab</sub> is normally accepted as the minimum noticeable difference,

Lozano<sup>23</sup> stated that a difference ranging from 2 to 5 units corresponds to a rigorous tolerance (for a specific color requirement), and from 5 to 10 units could be a normal difference in most manufacturing processes. Considering that all the products presented global color differences between 5.8 and 6.8 units with respect to the chopped fresh cherry, the ingredients obtained in this work preserved to a great extent the natural color of the fruit. This could be a highly valued attribute for the consumer.

The shrinkage suffered by the ingredients and the final bulk density are properties related to hygroscopicity, and reflect modifications to the tissue structure due to processing. Ingredient FC showed lower bulk density and shrinkage in comparison with AC (Table 3). It is widely known that freeze drying provides products with porous structure and little shrinkage.<sup>24</sup> In a comparative study of the effects of air drying and freeze drying on various berries, a minimum shrinkage was observed during the freeze-drying process (5–15%), whereas during convective air drying it was very significant (~80%).<sup>25</sup> Although freeze-dried cherry ingredients presented lower shrinkage values than air-dried ones, the observed tissue contraction was of considerable magnitude. Materials shrinkage during freeze-drying has been related to the collapse temperature of the concentrated amorphous solution.<sup>26</sup> If, during the primary drying stage, the product temperature is above the collapse temperature, viscous flow occurs and, consequently, there is loss in cake structure.<sup>27</sup> During structure collapse the capillaries seal, reducing the porous structure, which can make subsequent dehydration difficult.<sup>8</sup> In fact, ingredient FC presented slightly higher water content than ingredient AC (Table 1). It is also possible that other structural factors, such as cherry skin, may play a role in collapse. Ingredients with SI pretreatment showed slight but significant differences in bulk density values compared to the respective control; however, no significant differences were observed in shrinkage values. Several authors reported a decrease in shrinkage due to osmosis prior to convective drying processes in various fruit matrices.<sup>18,24,28</sup> On the other hand, there are few publications about combined processes of osmosis and freeze-drying. In the case of freeze-dried raspberries with SI pretreatment, the observed shrinkage only occurred during the osmotic dehydration process.<sup>7</sup>

Water adsorption from the environment was evaluated because it can affect the structural quality and compromise the stability of the product. Hygroscopicity (H) was associated with the weight gain reached at equilibrium when samples were exposed to a 75% RH atmosphere at 25 °C. Freeze-dried ingredients resulted less hygroscopic than air-dried ones. In general, freeze-dried





**Figure 1.** Photographs of dried sweet cherry ingredients obtained with different pretreatment-drying method combinations: (a) AC, (b) ASI, (c) FC, (d) FSI. Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.

**Table 2.** Luminosity ( $L^*$ ), chroma ( $C^*_{ab}$ ), hue angle ( $h_{ab}$ ) values and global color change ( $\Delta E^*_{ab}$ ) of dried sweet cherry ingredients

Ingredients	$L^*$	$C^*_{ab}$	$h_{ab}$	$\Delta E^*_{ab}$
ASI	$32.07 \pm 1.02^{B,b}$	$5.8 \pm 1.0^{B,b}$	$29 \pm 3^{B,b}$	$5.8 \pm 0.5^{A,a}$
FC	$29.1 \pm 0.2^{A,a}$	$2.0 \pm 0.2^{A,a}$	$13.4 \pm 1.4^{A,a}$	$6.8 \pm 0.2^{B,b}$
FSI	$31.4 \pm 0.7^{A,b}$	$4.2 \pm 1.0^{A,b}$	$19.1 \pm 2.0^{A,b}$	$6.1 \pm 0.5^{B,a}$
Pretreatment $\times$ drying*	NS	NS	NS	NS

Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C. Means ( $n = 16$ ) with the same letter superscript were not significantly different ( $P < 0.05$ ). Uppercase letters and lowercase letters were used for main effect of factors: 'drying method' and 'pretreatment', respectively.  
\*Interaction factor: NS (not significant).

**Table 3.** Shrinkage (S), bulk density ( $\delta_b$ ), and hygroscopicity (H) of dried sweet cherry products

Ingredients	S %	$\delta_b$ g mL <sup>-1</sup>	H %
AC	$83.8 \pm 0.9^b$	$1.51 \pm 0.02^c$	$26.7 \pm 0.4^b$
ASI	$81 \pm 3^{a,b}$	$1.41 \pm 0.03^b$	$28.8 \pm 0.6^c$
FC	$73 \pm 8^a$	$1.22 \pm 0.05^a$	$22.1 \pm 0.6^a$
FSI	$80 \pm 4^{a,b}$	$1.37 \pm 0.06^b$	$22.5 \pm 0.3^a$
Pretreatment $\times$ drying*	S	S	S

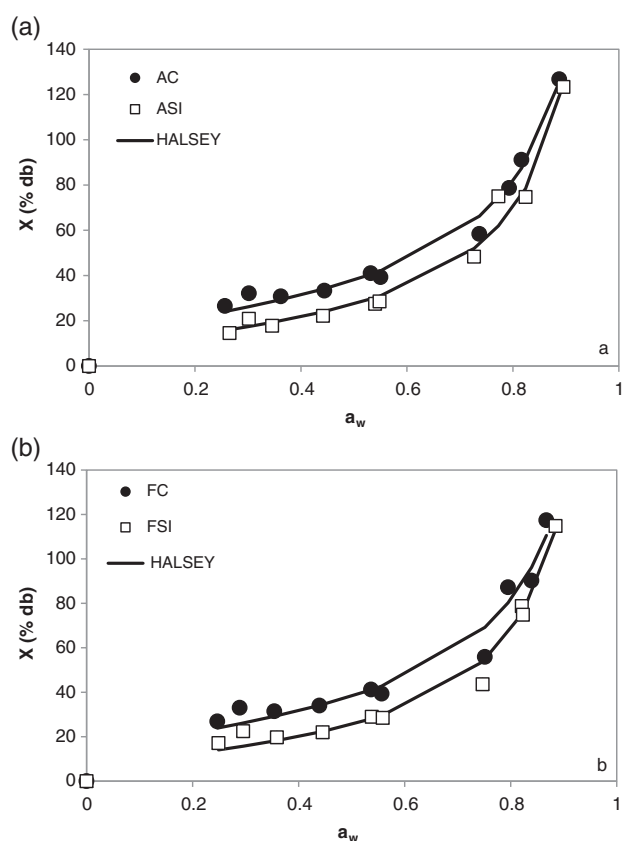
Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C. Means ( $n = 3$ ) with the same letter superscript were not significantly different ( $P < 0.05$ ).  
\*Interaction factor: S (significant).

samples have a porous and highly hygroscopic structure; however, freeze-dried ingredients showed a high degree of collapse, so the hygroscopic behavior is consistent with the structural characteristics of these samples. The ASI products also showed a slightly higher H than AC. This behavior is characteristic of products with high sugar content.<sup>29,30</sup> An interesting aspect to highlight is that no physical deterioration was visualized during the time necessary to reach the equilibrium state (150 days). These results would indicate that no special requirements for packaging would be needed.

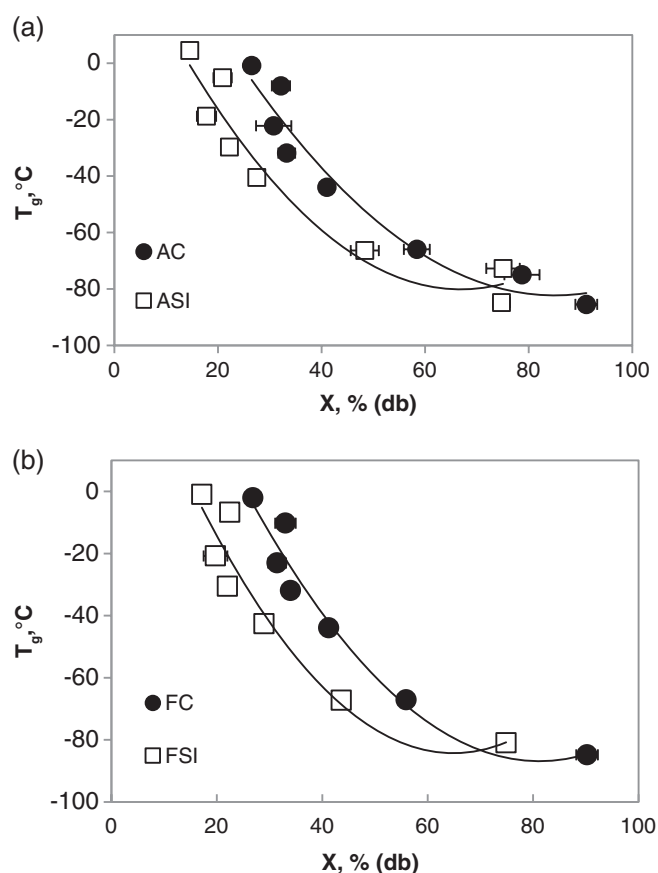
Figure 2 shows the water sorption isotherms of ingredients at 25 °C. The shape of the curves was typical of sugar-rich products that adsorb small amounts of water at low relative humidity (RH), and at high RH an increase in adsorbed water is usually observed.<sup>29</sup> The differences observed among the studied samples could be related to the divergence in chemical composition and structure, which in turn could be affected by both the pretreatment and the dehydration method. In this case, the dehydration method did

not affect the water sorption behavior; however, the sugar infusion pretreatment caused a 32% decrease in the water sorption capacity compared to the control sample, particularly in the range of 25 to 84%RH. The sugar uptake might have affected the tissue structure, the water-solute interactions, and the composition, given that a ~26% increase in total sugars was observed upon sugar infusion (Table 1). Ciuzyńska and Lenart<sup>31</sup> also observed a similar behavior when studying the effect of osmotic dehydration as a pretreatment in freeze-drying of strawberries. They suggested that the lower water sorption of the pretreated samples could be related to the saturation of intracellular spaces and cell walls with sugar, causing a porosity decrease.

To select a suitable mathematical model for the description of the water sorption isotherms of ingredients, the GAB, Oswin and Halsey models were fitted to experimental data (Table 4). In general, good fits to the three mathematical models were obtained in all cases, giving  $R^2 \sim 95$  or higher. The Halsey model was found to be the best equation taking into account the higher  $R^2$  value, and it could also be used with predictive criteria in these products because in all cases (E%) were lower than 10%.



**Figure 2.** Water sorption isotherms of air-dried (a) and freeze-dried (b) sweet cherry ingredients ( $n = 3$ ). Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.



**Figure 3.** Glass transition temperatures ( $T_g$ ) of air-dried (a) and freeze-dried (b) sweet cherry ingredients ( $n = 3$ ). Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.

**Table 4.** Parameters estimated from Eqns (2)–(4) (GAB, Oswin, and Halsey models) and statistics used to evaluate the goodness of fit for each experimental condition

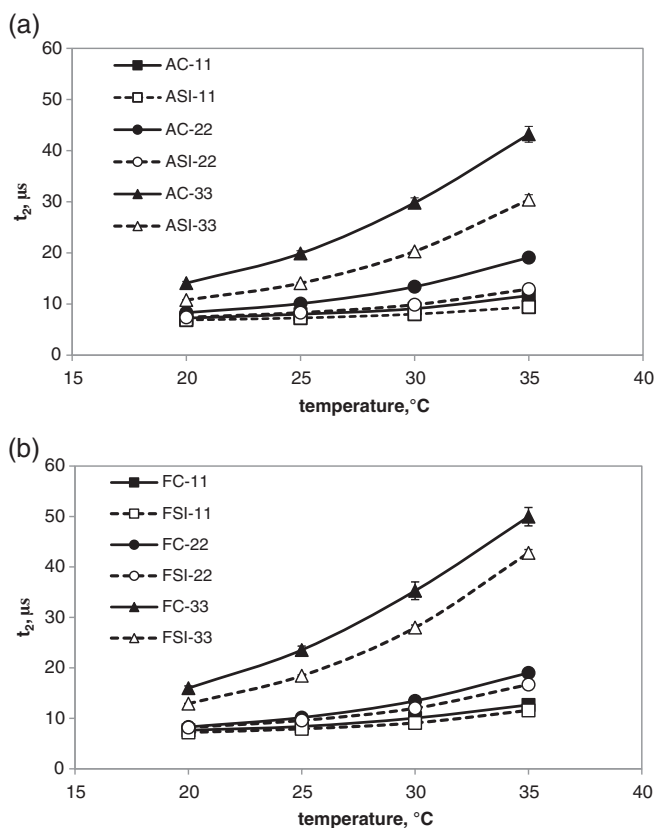
Model	Parameter	AC	ASI	FC	FSI
GAB	$X_0$	22.17	16.96	21.99	23.03
	C	15.6	5.21	24	1.69
	K	0.93	0.97	0.92	0.92
	$R^2$	98.34	97.87	95.56	95.29
OSWIN	E%	6.8	8.24	9.09	16.62
	A	38.39	27.13	39.09	25.46
	B	0.56	0.7	0.54	0.72
	$R^2$	97.81	98.03	94.97	97.21
HALSEY	E%	9.35	9.65	12.18	13.39
	A	147.89	38.42	157.82	30.11
	B	1.47	1.21	1.49	1.16
	$R^2$	98.86	98.04	96.33	98.31
	E%	6.33	9.16	9.38	9.51

Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C. ( $n = 3$ ).

Figure 3 shows the glass transition temperatures ( $T_g$ ) for air-dried (Fig. 3(a)) and freeze-dried (Fig. 3(b)) ingredients. All the samples studied were in a supercooled state at room temperature. No differences were observed due to the drying method, which is in accordance with the similar behavior of the water sorption isotherms (Fig. 2). Although SI samples presented lower water

contents than control samples for the same RH, control cherries showed higher  $T_g$  values than pretreated ones for water contents lower than 60%. This behavior could be related to the presence of monosaccharides generated due to the hydrolysis of part of the sucrose incorporated during osmosis pretreatment. It should be considered that sucrose has a relatively high glass transition temperature compared to the  $T_g$  values of monosaccharides (glucose and fructose). Similar behavior was reported by Riva *et al.*<sup>32</sup> for osmodehydrated and air-dried apricots, suggesting that sucrose hydrolysis might have happened due to the natural acidity of the fruit. On the other hand, part of the sucrose incorporated in pretreated samples could have crystallized due to its concentration upon dehydration. The crystalline sucrose therefore could not contribute to increasing the  $T_g$ , and the available water would be plasticizing the remaining amorphous part of the sample, thereby giving lower  $T_g$  values. Sette *et al.*<sup>7</sup> also showed a decrease in  $T_g$  values for dehydrated raspberries pretreated with sugar infusions when compared to their respective control samples.

Figure 4 shows the  $^1\text{H}$  NMR relaxation times ( $T_2$ ) determined by a single  $90^\circ$  pulse as a function of temperature, at different relative humidities, for air-dried (Fig. 4(a)) and freeze-dried (Fig. 4(b)) sweet cherries. The fast decay component ( $T_2$ ) can be attributed to solid polysaccharide protons, and water molecules that are strongly associated by hydrogen bonding to the solid matrix.<sup>33</sup> The NMR analysis was performed at RH and temperature ranges that the sweet cherry products could undergo during storage, alone or

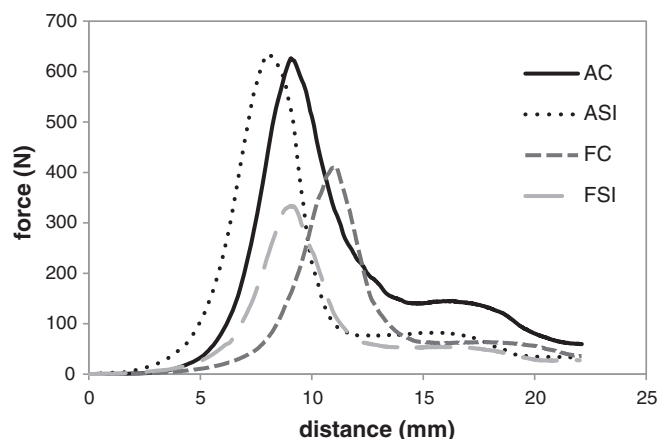


**Figure 4.** Relaxation times ( $t_2$ ) of air-dried (a) and freeze-dried (b) sweet cherry ingredients, equilibrated at 11, 22 and 33% RH ( $n = 3$ ). Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.

incorporated in a mixture with other low  $a_w$  ingredients. In general, an increase in the  $T_2$  values was observed while increasing RH and temperature. Freeze-dried cherries showed slightly higher  $T_2$  values compared to air-dried ones, although no important differences were observed in  $T_g$  values (Fig. 3) and water sorption behavior (Fig. 2). The application of the SI pretreatment caused an important reduction in the molecular mobility, which was mainly observed at 33% RH. This effect could be attributed to the lower water contents observed in the pretreated cherries at all the RHs that were analyzed (Fig. 1). Similar results were reported by Sosa et al.<sup>15</sup> for freeze-dried and air-dried apple discs with a sugar infusion pretreatment.

Mechanical behavior of cherry pieces subjected to air drying and freeze drying with or without SI pretreatment is shown in Fig. 5 and Table 5. Force-distance curves obtained during the Kramer assay (Fig. 5) showed a similar general behavior. The initial stage corresponds to the reordering of particles during the first moments of the assay leading to a low resistance to shear. Then, the force increased until the maximum packing of the particles occurred, and the blades cut the sample completely reaching the maximum force ( $F_{max}$ ). Then, resistance decays symmetrically up to values close to one-third of  $F_{max}$ ; after that, symmetry is lost, probably due to adhesion of the fruit to the blades. Freeze-dried cherries showed a longer reordering stage than air-dried ones. The SI pretreatment caused, for both drying methods, a shorter reordering stage and lower adherence to the blades in the last stage.

Air drying led to harder samples ( $>F_{max}$ ) and higher  $SI_{max}$  values (Table 5), probably due in part to the formation of a crust



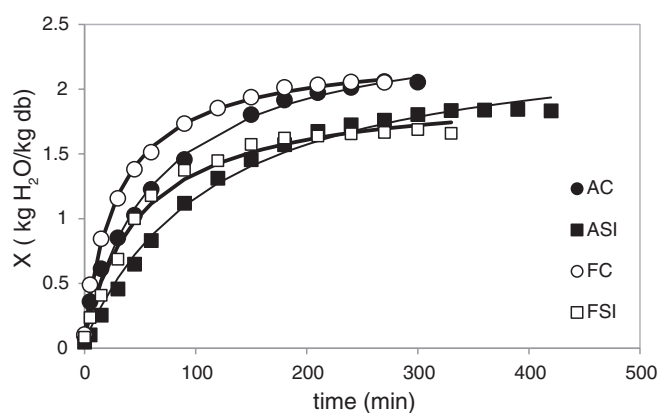
**Figure 5.** Force-distance curves obtained during the compression of dried sweet cherry products using a Kramer cell ( $n = 10$ ). Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.

**Table 5.** Variables obtained from the force-distance curves of dried sweet cherry products: maximum force ( $F_{max}$ ), distance at maximum force ( $\Delta F_{max}$ ), maximum slope of peak before maximum force ( $SI_{max}$ ), and peak area up to the maximum force ( $W$ )

Ingredients	$F_{max}$ N	$\Delta F_{max}$ mm	$SI_{max}$ N/mm	WJ
AC	$627 \pm 19^c$	$9.3 \pm 0.2^a$	$238 \pm 8^{Bb}$	$1.22 \pm 0.06^b$
ASI	$633 \pm 18^c$	$8.9 \pm 0.3^a$	$224 \pm 11^{Ba}$	$1.30 \pm 0.05^b$
FC	$419 \pm 9^b$	$10.7 \pm 0.3^b$	$161 \pm 7^{Ab}$	$0.78 \pm 0.03^a$
FSI	$337 \pm 13^a$	$8.4 \pm 0.2^a$	$122 \pm 5^{Aa}$	$0.64 \pm 0.04^a$
Pretreatment $\times$ drying*	S	S	NS	S

Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C. Means ( $n = 10$ ) with the same letter superscript were not significantly different ( $P < 0.05$ ). Uppercase letters and lowercase letters were used for main effect of factors: 'drying method' and 'pretreatment', respectively.  
\*Interaction factor: S (significant), NS (not significant).

caused by migration of sugars to the tissue surface during the drying process.<sup>34</sup> The SI pretreatment caused a decrease in the  $F_{max}$  values of freeze-dried cherries; however, it did not affect the corresponding values for air-dried samples. The work or energy required for sample deformation was affected by the applied dehydration method, and the observed differences were probably due to different structural characteristics of air- and freeze-dried cherries. The lower  $F_{max}$  and lower energy observed in the freeze-dried samples could be related to the damage suffered by the tissue during the freezing step, causing internal cracks and big pores, when compared to the air-dried cherries, having a compact and harder structure. In general, the mechanical behavior of cherries seemed to be more related to the structural changes caused during the sugar infusion and the drying process than to the glass transition temperatures, given that, at the analyzed RH, all the samples showed similar  $T_g$  values, close to  $-20^\circ\text{C}$ . According to Peleg,<sup>35</sup> in complex food materials like dehydrated tissues, which consist of several components and more than one phase, their properties may not change at the same time as that predicted by the glass transition theory.



**Figure 6.** Rehydration kinetic at 25 °C of dried sweet cherry products ( $n = 2$ ). Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C.

**Table 6.** Model parameters estimated from Eqn (7) ( $k_1$ ,  $k_2$ ), rehydration capacity at 5 min ( $CR_{5min}$ ) and at the end ( $CR_f$ ) of the rehydration process at 25 °C of dried sweet cherry products

Ingredients	$k_1$	$k_2$	$CR_{5min}$	$CR_f$
	min (kg H <sub>2</sub> O/kg db) <sup>-1</sup>	(kg H <sub>2</sub> O/Kg db) <sup>-1</sup>		
AC	26 ± 2 <sup>B,a</sup>	0.402 ± 0.009 <sup>A,a</sup>	27 ± 3 <sup>A,b</sup>	287 ± 10 <sup>A,b</sup>
ASI	52 ± 6 <sup>B,b</sup>	0.408 ± 0.010 <sup>A,a</sup>	5.2 ± 0.3 <sup>A,a</sup>	270 ± 3 <sup>A,a</sup>
FC	11 ± 4 <sup>A,a</sup>	0.49 ± 0.05 <sup>B,a</sup>	43 ± 11 <sup>B,b</sup>	284 ± 11 <sup>A,b</sup>
FSI	21 ± 8 <sup>A,b</sup>	0.58 ± 0.08 <sup>B,a</sup>	14.7 ± 1.0 <sup>B,a</sup>	239 ± 13 <sup>A,a</sup>
Pretreatment × drying*	NS	NS	NS	NS

Air-dried: A; freeze-dried: F; sugar infusion pretreatment: SI; control: C. Means ( $n = 2$ ) with the same letter superscript were not significantly different ( $P < 0.05$ ). Uppercase letters and lowercase letters were used for main effect of factors: 'drying method' and 'pretreatment', respectively.  
\*Interaction factor: NS (not significant).

Rehydration capacity and rate are quality attributes in relation to drying. Rehydration behavior has been considered as a measure of the induced damage in the material during drying,<sup>36</sup> such as integrity loss and reduction of hydrophilic properties, which decrease the rehydration ability. The rehydration properties are also important characteristics of many products, related to their later preparation for consumption.<sup>37</sup> Figure 6 shows the rehydration curves at 25 °C. All the curves showed typical rehydration behavior, with a high water uptake rate at the beginning of the process, followed by a decrease in the absorption rate until a plateau was reached. Table 6 shows the parameters obtained from the application of the Peleg model to the experimental rehydration curves ( $k_1$  and  $k_2$ ), and the rehydration capacity (CR) at 5 min and at the end of the rehydration at 25 °C. For all the variables studied, interaction factors were not significant, so the main effect of the factors could be explained. The dehydration method affected the rehydration behavior. Freeze-dried cherries showed a higher initial rehydration rate ( $< k_1$ ) and higher  $CR_{5min}$  than air-dried ones. Similar results were observed by Cierzyńska and Lenart<sup>31</sup> when studying the rehydration capacity of dried strawberries. In the first hydration stage, water occupies the spaces

filled with air.<sup>38</sup> The collapse of capillaries occurred due to high temperatures during air drying, which might have reduced the rehydration rate.<sup>36,39</sup> It is interesting to note that, although the water uptake was faster in the freeze-dried sweet cherries, the final water recovery was lower ( $> k_2$ ) than that observed for air-dried samples, probably due to structural damage produced during freezing. The application of the sugar infusion pretreatment reduced the initial rehydration rate and  $CR_{5min}$  when compared with the respective control samples. This effect was more marked for the air-dried cherries. In this case, the incorporated sugar saturated the intercellular spaces and the cell walls, contributing to the reduction in porosity and the rehydration capacity of the fruit.<sup>31,40</sup>

The final water content of the rehydration process was lower than the water content of the fresh cherry pieces ( $4.65 \pm 0.18$  kg H<sub>2</sub>O kg<sup>-1</sup> db), reaching  $59 \pm 5\%$ ,  $67.9 \pm 1.2\%$ ,  $74 \pm 4\%$ , and  $79 \pm 3\%$  for FSI, ASI, FC and AC, respectively. The damage caused to the cell membranes by the combination of pretreatment and the drying process directly affects the pectic substances, cellulose, and hemicelluloses.<sup>41</sup> Then, the structural damage and cell reduction that occur during the drying process result in the irreversible loss of the rehydration capacity.<sup>42</sup>

## CONCLUSIONS

Relevant differences between cherry ingredients were observed according to the processing method used. Freeze drying was not particularly appropriate for this fruit, probably because of tissue damage during the freezing step together with the tensions caused by the skin. However, these ingredients offered an attractive red hue and a good source of bioactive compounds.

Although the use of SI pretreatment caused a marked reduction in bioactive compounds, it also allowed increased product stability. Air-dried fruits with previous SI presented a low rehydration rate and a sweet taste. If more healthy products without added sugar were desired, air-dried ingredients without pretreatment could be selected, giving the highest antioxidant capacity and a harder structure. These samples also exhibited greater rehydration capacity.

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