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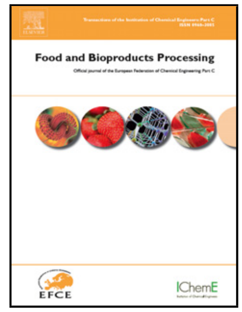

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PHYSICAL AND MECHANICAL PROPERTIES OF RASPBERRIES SUBJECTED TO OSMOTIC DEHYDRATION AND FURTHER DEHYDRATION BY AIR- AND FREEZE-DRYING

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ABSTRACT

The aim of this study was to analyze the effect of the application of dry and wet sucrose infusions, as pretreatments previous to air- and freeze-drying, on mechanical and physical properties of raspberries: water sorption, glass transition temperature (T_g), molecular mobility, texture and rehydration properties. Different dry and wet sugar infusions were prepared using combinations of additives: sodium bisulphite, citric acid, sodium bisulphite and citric acid, and no additives. These specific pretreatments are often used to obtain better sensorial characteristics of fruits upon further drying. After the dehydration step (air- or freeze-drying), all the samples were in the supercooled state. Pretreated samples presented lower T_g values and lower spin-spin relaxation times than control samples. Regarding texture, pretreated samples showed lower firmness than control samples. Also, freeze-dried pretreated samples showed higher firmness and lower deformability than air-dried pretreated ones. When considering the hygroscopicity, freeze-dried samples were more hygroscopic than air-dried ones. The fresh-like dried raspberries obtained could be directly consumed as snacks or incorporated in a composite food, such as a cereal mix. In this latter case, pretreated fruits would be more suitable, since their rehydration capacity at short times was relatively low.

Keywords: raspberries, osmotic dehydration, air-drying, freeze-drying, physical and mechanical properties.

1. INTRODUCTION

Raspberries are highly appreciated by consumers because of their aromatic flavor, in addition to providing essential nutrients for human health. However, this fruit is known for being very labile and having a short post-harvest life due to its high respiration rate, loss of firmness and freshness and susceptibility to browning (Duel and Plotto, 2004; Gómez Riera et al., 2014). Therefore, after harvest they must be consumed or processed in a few weeks in order to reduce economic losses. For these reasons, it is necessary to apply different methods of conservation that would generate long-life raspberry products with high quality and at the same time that are innovative for consumers (De Santana et al., 2014).

Dehydration has been one of the techniques more frequently used for preserving food, and a variety of methods have been studied, focusing on the quality of the obtained products (Barbosa-Canovas and Vega-Mercado, 2000). Air-drying is the most widely used method of dehydration, but the use of elevated drying temperatures implies a substantial degradation in quality attributes (Adiletta et al., 2015; Maskan, 2001; Moraga et al., 2006). On the other hand, freeze-drying is a technique used to obtain high quality dehydrated foods based on sublimation (Khalloufi and Ratti, 2003). Freeze-drying can produce porous, brittle, amorphous and hygroscopic structures (De Santana et al., 2014). A way of improving the quality of dehydrated products is the application of pretreatments. Sugar infusion, applied as a pretreatment, provokes the exchange of water and solutes, allowing a partial decrease of water activity prior to dehydration (Torreggiani and Bertolo, 2001). This process permits the formulation of products with intermediate moisture contents through dewatering and impregnation of desired solutes (Mauro et al., 2015).

In recent years, several nutritional studies recommend a higher consumption of fruits. To ensure these needs, the food companies have introduced new products. An example is the wide variety of breakfast cereals and granola bars and energy bars with dried fruit pieces that can be found in the market (Blessing and Ekwunife, 2015; Talens et al., 2012). The consumption of these types of snacks adds variety to the diet and allows the intake of dietary fiber, vitamins and minerals, while providing a substantial energy input (Demarchi et al., 2013).

The aim of this study was to analyze the effect of the application of different dehydration methods, with or without sugar infusion pretreatments on the physical properties (water sorption isotherms, thermal transitions, molecular mobility, shrinkage and hygroscopicity) and mechanical properties (texture) of dehydrated raspberries.

2. MATERIALS AND METHODS

2.1 Fruits

Frozen raspberries (cv. Autumn Bliss, reference sample) grown in Plottier (Neuquén province, Argentina) were used. After harvest, fruits were immediately individually quick frozen (IQF process) in an air blast tunnel (T = -48°C, air speed = 1.5 ms^{-1}) and then stored at -22°C until use. The characterization was carried out according to

AOAC methods (Sette et al., 2015): water content 85 ± 3 %, water activity (a_w) 0.97 ± 0.02, total soluble solids 8.8 ± 0.8 °Brix, pH 3.13 ± 0.02, total acidity 0.267 ± 0.004 % citric acid, ash 0.363 ± 0.012 %.

2.2 Pretreatments

Fruits were subjected to sugar infusion pretreatments performed at room temperature in glass vessels (8x16 cm). Different systems were prepared by immersing the frozen fruits into a mixture (dry or wet) of the humectant and the preservatives commonly used in the preparation of high- or intermediate-moisture fruits (Alzamora and Salvatori, 2006; Tapia de Daza et al., 1996). Potassium sorbate and sodium bisulphite are usually used as antimicrobial agents; sodium bisulphite also acts as an inhibitor of enzymatic and non-enzymatic browning. Citric acid was added in some conditions to achieve different pH levels. The final pH value of infused samples was 2.3 in wet infusions and 2.5 in dry infusions. Reagents were all food grade (Saporiti S.A., Argentina). The amount of sugars and chemical agents were determined according to the weight of the fruit (100 g) and the final levels required after equilibration of the components of the food system (a_w=0.85). Sucrose concentration in the mixture was calculated using the Ross equation (Tapia de Daza et al., 1996) to attain the a_w equilibration value desired between raspberries and the formed syrup. The selection of the additives was based in a previous work (Sette et al., 2015). Two different infusion treatments to reduce a_w to 0.85 were performed: dry infusion (DI) and wet infusion (WI). In DI, fruits were mixed directly with the humectant and the additives. In WI, fruits were immersed in an aqueous solution of the humectants and additives. The fruit/sugar ratio was 1.27 for dry infusions and 0.36 for wet infusions. Systems were prepared as follows:

Dry infusions: fruits and sucrose (the only additive) (DI), fruits and a dry mix of additives containing 95.8% sugar and 4.2% citric acid (DI-AC), fruits and a dry mix of additives containing sucrose and 250 ppm of sodium bisulphite (DI-B), fruits and a dry mix of additives containing 95.8% sugar, 4.2% citric acid and 250 ppm of sodium bisulphite (DI-BAC).

Wet infusions: fruits dipped in an aqueous solution of sucrose (61% w/w) (WI), fruits immersed in an aqueous solution of 59.4% sugar and 2.3% citric acid (WI-AC), fruits immersed in an aqueous solution of 61% sugar and 250 ppm of sodium bisulphite (WI-B), fruits immersed in an aqueous solution of 59.4% sugar, 2.3% citric acid, and 250 ppm of sodium bisulphite (WI-BAC).

Reference samples: frozen fruits were used as reference samples.

In all cases, 1000 ppm of potassium sorbate was added. The preparations were gently mixed twice daily and system a_w was controlled until equilibration was reached (fruit a_w = generated syrup a_w = 0.85). After that, the fruits were taken out of the generated syrup and drained on tissue paper to remove the residual syrup. Bioactive compounds and antioxidant capacity of both the osmosed raspberries and the different generated syrups were reported in a previous work (Sette et al., 2015).

2.3 Drying process

Raspberry samples with and without pretreatments were subjected to two different drying processes:

a) *Freeze-drying*: samples were quenched with liquid nitrogen, directly for control samples and after pretreatments for the rest of the samples. The freeze-drying process lasted 48 hours and was carried out in a freeze drier Alpha 1-4 LD/2-4 LD-2 (Martin Christ, Gefriertrocknungsaniagen GmbH, Osterode, Germany). It was operated at -55°C at a chamber pressure of 4 Pa.

b) *Air drying*: an air convection oven model Venticell 111- Standard (MMM Medcenter Einrichtungen GMBH, Munich, Germany) was used (air at 60 ± 1ºC, ≈10% relative humidity (RH) and speed = 1 - 1.5 m/s) with forced air and controlled temperature. RH was controlled with a Hygro Palm hygrometer (Rotronic Instruments, West Sussex, UK). The drying time required to achieve a_w close to 0.33 was 22 h for control samples and 24 h for dry and wet infusions samples.

Control samples (without pretreatment) and 8 different pretreated raspberries were obtained after each drying process.

2.4 Sample analysis

2.4.1 *Water Content (X) and Water Activity (a_w)*

The water content was determined gravimetrically according to AOAC methods (925.09, 1990). Results were expressed as g of water per 100 g of dry matter (d.w.). Water activity (a_w) was measured at 20ºC with a psycrometer model Series 3 (Aqua-Lab, Decagon Devices Inc., Pullman, Washington, USA), calibrated with saturated saline aqueous solutions.

2.4.2 *Total Sugar content (TS)*

The total sugar content was determined by an anthrone/sulfuric acid procedure (Southgate, 1976). A curve with glucose as standard was used for expressing results. Results were expressed as g of glucose per 100 g of dry matter (d.w.).

2.4.3 *Volumetric Shrinkage (Sh)*

The shrinkage caused by the different dehydration methods was evaluated through measurements of sample volume change. Volume (V) was estimated gravimetrically by displacement of toluene in a pycnometer. Shrinkage was expressed according to the following equation:

$$Sh = \left(\frac{V_0 - V}{V_0}\right) 100 \quad (1)$$

Where V_0 = initial average volume (taken from 10 reference raspberries) and V = volume of each raspberry after treatment. Volume displacement was determined in quintuplicate.

2.4.4 *Heywood shape factor (k)*

The Heywood shape factor is one of the parameters commonly used to assess the shape changes that occur in dehydrated products. It is calculated through the relationship between the real volume of the particle and its equivalent diameter, calculated in terms of the projected area of the particle in the most stable rest position and can be mathematically defined with the following equation (De Michelis et al., 2013):

$$k = \frac{V_p}{d_a^3} \quad (2)$$

Where k = Heywood shape factor; V_p = sample volume determined by pycnometry; d_a = equivalent diameter. The projected area of the particles was obtained by assigning the area of an equivalent circle with the same greater diameter of the fruit (De Michelis et al., 2008).

2.4.5 *Mechanical Properties*

A shear Kramer test was performed using an Instron universal testing machine Model 3344 (Instron Corporation, Canton, MA, USA), connected by a computer to the *Instron Bluehill Material Testing Software*. For each test, a group of samples were weighed and placed inside the Kramer cell (55 x 50 x 60 mm^3) ensuring the formation of a 15 mm thick bed. The upper mobile part of the cell is made of 5 parallel 3 mm vertical metal blades, each one 3 mm distant from the other. The crosshead speed used in all experiments was 20 mm/min and the sensor scale was 0-5 kN for testing control samples and 0-1kN for pretreated samples. Force–distance curves were recorded during the test while the blades descended through the complete sample bed of raspberries. The peak force (F_{max}) reflected the mechanical resistance during compression. From the force-distance curves obtained other parameters were evaluated in order to describe the curves: distance corresponding to the maximum force (Δ_{Fmax}), maximum slope of the curve before peak (SL_{max}) and work (W) or energy at midpeak (area under the curve for d $\leq SL_{max}$ .The reported values parameters correspond to the average of individual measurements of ten samples for each type of dried raspberry.

2.4.6 *Water sorption isotherms*

Humidification

After drying, raspberries were put into vacuum desiccators over saturated salt solutions in the range between 11% and 90% RH. The salt solutions used were $LiCl_2$ (a_w=0.11), CH_3COOK (a_w=0.22), $MgCl_2$ (a_w=0.33), K_2CO_3 (a_w=0.43), $Mg(NO_3)_2$ (a_w=0.52), NaBr (a_w=0.58), NaCl (a_w=0.75), KBr (a_w=0.80), KCl (a_w=0.84) and $BaCl_2$ (a_w=0.90) (Greenspan, 1977). The samples were stored at 20°C until constant weight in order to achieve different moisture levels.

Water sorption modeling

The sorption isotherms represent the variation of the water content of the food with respect to the variation of water activity. The Guggenheim-Anderson-de Boer (GAB) (eq. 3) mathematical model was fitted to the water sorption data using the analysis software system ORIGIN PRO version 8.0 (OriginLab Corporation, Northampton, USA)

$$X = X_0 \frac{(C\,K\,a_w)}{\left((1 - K\,a_w)\,(1 - K\,a_w + C\,K\,a_w)\right)} \qquad (3)$$

Where X is the water content in dry weight (g H_2O / 100 g d.w.); X_o is the limit water content of hydration related with the kinetic adsorption in the first layer (g H_2O / 100 g d.w.); a_w is the water activity of the product expressed as HR/100; C is the Guggenheim constant, related to the heat of water sorption to the first layer of solid active sites; K is a factor which corrects the properties of the water molecules of the multilayer respect of free water.

2.4.7 *Thermal Transitions*

After humidification of the dried samples, the glass transitions were determined by differential scanning calorimetry (DSC; onset values) using a calorimeter model 822 (Mettler Toledo, Schwerzenbach, Switzerland). The instrument was calibrated with indium (156.6 °C). All measurements were performed over a temperature range from -130 to 70 °C, with a heating rate of 10 °C/min. Approximately 10 mg of each sample were placed in 40 µL aluminum pans, which in turn were hermetically sealed. An empty pan served as reference. Thermograms were evaluated using Stare software v. 3.1 (Mettler Thermal Analysis). An average value of at least two replicates was reported.

2.4.8 *Molecular Mobility*

A Bruker mq 20 Minispec pulsed nuclear magnetic resonance (NMR) instrument, with a 0.47 T magnetic field operating at resonance frequency of 20 MHz, was used. Measurements were performed at 20°C and 75% RH. The spin-spin relaxation time (t_2) associated to the fast relaxing protons (related to the solid matrix and to water interacting tightly with solids) was measured using a free induction decay analysis (FID) after a single 90º pulse. The decay envelopes were fitted to mono-exponential behavior with the following equation:

$$I = A \exp\left(-\frac{t}{t_2}\right) \quad (4)$$

Where I represents the protons signal intensity; t_2 corresponds to the relaxation time of protons in the polymeric chains of the material and of tightly bound water and A is a constant. Since no 180º refocus pulse was used in the experiments, the spin-spin relaxation time constants are apparent relaxation time constants, i.e. t_2^*. However, for solid materials (like ours), we can consider that the intrinsic t_2 is very close to the t_2^* as reported previously by Fullerton and Cameron (1988). Therefore, t_2 was used for convenience.

2.4.9 *Hygroscopicity*

The hygroscopicity of the dehydrated raspberries was evaluated by exposing the fruits to an atmosphere at 75% relative humidity at 20 °C until equilibrium was reached. A desiccator containing a saturated solution of NaCl was used to generate 75% RH. The samples were periodically removed and weighed and then returned to the desiccator. The weight gain as a function of time (water adsorption) was recorded in triplicate and the hygroscopicity % (Hi) was expressed as the average of ten consecutive records corresponding to the asymptotic zone of the curve (water content as a function of time).

2.4.10 *Rehydration*

The rehydration potential of dried raspberries was determined by combining approximately 1 g of berries with 20 ml of water in a 50 ml beaker. The samples were rehydrated over a specified period of time at 25 ± 1°C (room temperature) to evaluate the kinetics of water absorption and the dry matter of solids. At specified times, the samples were carefully removed from the beaker, blotted with paper towel to remove excess water, and weighted. Each rehydration experiment was performed in triplicate.

The coefficient of rehydration (RC) was calculated at 15 min. and at the end of the rehydration process with the following equation (Khraisheh et al., 2004):

$$RC = \frac{W_r(100\text{-}X_0)}{W_d(100\text{-}X_d)} \quad (5)$$

Where W_r is the mass of the rehydrated sample (g); W_d is the mass of the dehydrated sample (g); X_d is the water content of the dehydrated sample (%, w.b.) and X_0 is water content of the reference fruit (%, w.b.).

The RC coefficient represents the recovery degree of weight with respect to the reference fruit.

2.5 Statistical analysis

A completely randomized design was used. For all determinations, except for shrinkage, thermal transitions and mechanical properties, three replicates were measured. The results were expressed by the mean and standard deviation (SD). An analysis of variance was performed to establish the presence or absence of significant differences in parameters according to the factors ‘‘additive’’, ‘‘type of infusion’’ and “drying method”. Multiple comparisons were carried out by using the Tukey test and significance level was set at $p<0.05$. In the case of significant interactions between factors, the Tukey test was run for the interaction. For not significant interaction between factors, a Tukey test of main effects was performed. All of the measured variables used to characterize the raspberries under the different dehydration methods were descriptively compared with an analysis of principal components (PCA). All statistical analyses were carried out using the data analysis software system STATISTICA version 8.0 (StatSoft, Inc., Tulsa, OK, USA).

The GAB model parameters fitted to isotherms curves were determined by nonlinear least-squares regression analysis. In order to evaluate the quality of fit obtained, in addition to the determination coefficient (r^2), other statistical parameters such as the reduced chi-square ($\chi2$) and the root mean square error (RMSE) were considered:

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(X^*_{exp,i} - X^*_{pre,i}\right)\right]^{1/2} \quad (6)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(X^*_{exp,i} - X^*_{pre,i}\right)^2}{N - Z} \quad (7)$$

Where X^* is X/X_o; X^*_{exp} is the experimental water content; X^*_{pre} is the water content calculated for the GAB model; N is the number of experimental data points used for modeling.

3. RESULTS AND DISCUSSION

3.1 *Physical properties*

The water content decrease of dehydrated products induces supersaturation of their components, leading to an increase in the cohesive forces between the molecules of water and solutes, decreasing the molecular mobility. For this reason, the presence of water and its interactions with other components are important factors to consider not only in the process control, but also on dehydrated food stability and quality. In this work, different sugar infusion pretreatments and two dehydration methods (air- and freeze-drying) were applied to raspberries. After both drying processes, the final water activity of the products ranged between 0.30 and 0.34. **Table 1** shows some parameters that characterize the raspberries after the application of the different pretreatments and drying processes: water content, glass transition temperature (T_g) and t_2 relaxation time. The water content of raspberries decreased from 85% (wet basis) (reference fruit) to ≈51% (wet basis) after infusion pretreatment in all cases, with a final a_w of 0.85 (Sette et al., 2015). After further drying, raspberries experienced different changes in water content according to the type of applied pretreatment (**Table 1**). Water content (X) of samples without pretreatment (C) was reduced ≈97% after drying. Pretreated samples, partially dehydrated by osmosis during infusion, exhibited a slightly higher water loss in freeze-dried samples (≈90%) when compared to air-dried ones (≈86%).

The total sugar content (TS) of the reference sample was 49.4 ± 0.5% (d.w.) and increased till ≈70 % (d.w.) after dehydration (air or freeze-drying) and ≈100 % when sugar infusion pretreatment was applied.

All the analyzed samples presented clear glass transitions by DSC measurement (not shown). The glass transition temperature (T_g) values, obtained after the application of the drying processes (a_w ≈ 0.33), were low (**Table 1**), and all the samples were in the supercooled state at room temperature. Similar T_g values were reported for several dehydrated fruits: freeze-dried strawberry (Moraga et al., 2004; Roos, 1987), freeze-dried persimmon (Sobral et al., 2001), freeze- and air-dried apple (Sosa et al., 2012), freeze-dried grape (Fabra et al., 2009), and freeze-dried plum (Nicoletti et al., 2006). Control raspberry samples presented higher T_g values than pretreated fruits. In general, freeze-dried samples showed higher T_g values than air-dried ones. This fact could be attributed, at least in part, to the higher water contents present in air-dried fruits. Also, the heat treatment applied upon air-drying may cause compositional changes that affect the glass transition temperatures. The low T_g values observed in pretreated raspberries is not in accordance with the low water contents of these samples. This behavior could be related to sucrose crystallization caused by sugar concentration upon drying. Therefore, crystalline sucrose would not be contributing to increased T_g, and the available water would be plasticizing the remaining amorphous phase, leading to low T_g values. This behavior was also observed in several studies performed on fruits, showing that pretreated samples presented lower T_g values than control ones: air- and freeze-dried tomato (Telis and Sobral, 2001); air-dried apple (Del Valle et al., 1998; Sosa et al., 2012); freeze-dried apple (Sá et al., 1999; Sosa et al., 2012); and air-dried mango (Rosas-Mendoza et al., 2011).

^{1}H NMR relaxation times (t_2) were determined at 20 °C by a single 90° pulse, as an estimation of molecular mobility. This fast decay component (t_2) was attributed to solid protons, and water molecules that are strongly associated by hydrogen bonding to the solid matrix (Kalichevsky et al., 1992; Ruan et al., 1999). It can be observed that freeze-dried samples showed lower t_2 values when compared to air-dried fruits, which correlates with the higher water contents observed in air-dried samples. Also, although having a higher water content, the t_2 value corresponding to the control air-dried sample was lower than those for pretreated raspberries. This behavior is in accordance with the corresponding T_g values. In this case, it would also be expected that the available water which would be plastizicing the amorphous phase, would be confined to a particular zone with higher molecular mobility. On the other hand, the different additives did not significantly affect the molecular mobility. Up to now studies on the molecular mobility of fruits are scarce. They include apple (Agudelo-

Laverde et al., 2014; Hills and Remigereau, 1997; Mauro et al., 2015; Nieto et al., 2013; Sosa et al., 2012), and melon and pear (Agudelo-Laverde et al., 2014). Overall, the addition of sucrose caused a decrease in T_g values and an increase in molecular mobility, mainly in air-dried samples.

3.2 *Structural characteristics*

Physicochemical and structural changes that occur during dehydration processes directly affect the final product quality (Khalloufi and Ratti, 2003). One of the most important physical changes that the food suffers during drying is the reduction of volume. Loss of water and heating cause stresses in the cellular structure of the food leading to change in its dimensions (Kurozawa et al., 2012; Mayor and Sereno, 2004). In addition to the evaluation of particle shrinkage, shape changes have to be taken into account (De Michelis et al., 2013). Changes in shape, loss of volume and increased hardness cause, in most cases, a negative impression in the consumer (Mayor and Sereno, 2004). **Fig. 1** shows the volumetric shrinkage observed upon air- or freeze-drying with the application of different pretreatments. Although volume reduction is mainly associated with water loss during dehydration, it has been shown that changes in shape and dimensions of the products depend on the specific response of each material to the induced forces generated during dehydration, which also depend on the drying method applied and the operative parameters. When water is removed from the material, a pressure unbalance is produced between the internal and the external pressure, generating contracting stresses that lead to material shrinkage or collapse, changes in shape and occasionally cracking of the product. Drying under vacuum, as in freeze-drying, leads in general to much less shrinkage than air-drying (Mayor and Sereno, 2004). On the other hand, the dried products that exhibit a rigid surface are more difficult to deform during drying (Lewicki and Jakubczyk, 2004). Additionally, structural collapse would be expected to be reduced if fruits are impregnated with sugars and/or biopolymers prior to drying (Prothon at al., 2003).

Regarding control raspberries, a significant volumetric shrinkage (81 ± 3%) developed after air-drying, while freeze-dried samples showed only 11 ± 2% shrinkage. These results are in agreement with those reported by Ratti (2001) who made a comparative study of air-drying and freeze-drying effects in different berries (strawberry, raspberry and blueberry), noting that shrinkage during the freeze-drying is minimal (5 - 15%), while during air-drying it is excessive (≈80%). The same behavior was verified in studies performed with different varieties of vegetable matrices such as apple, potato, banana, carrot and pumpkin (Donsi et al., 1996; Krokida et al., 1998; Nawirska et al., 2009). Krokida et al. (1998) found that the freeze-drying process allows obtaining products with a low degree of shrinkage and a low bulk density, if the process temperature is maintained below the glass transition temperature of the material. On the other hand, the volume changes observed in samples subjected to air-drying may also have also occurred as a result of thermal degradation of structural components. According Prinzivalli et al. (2006) the solubilization of polymers from the cell wall can be a consequence of the high temperatures during air-drying, which would contribute to the disintegration of the cell walls, resulting in a considerable volume decrease. Femenia et al. (2007) studied the effect of dehydration at temperatures between 40 and 80°C on the cell walls of pineapple, and observed that fruits dried at higher temperature exhibited a higher solubilization/degradation of pectic substances, which could have a higher impact on the physical condition of the components and the texture of these dried samples. Other authors conducted comparative studies by SEM of the structure in apple tissue subjected to air-drying and freeze-drying. Sosa et al. (2012) observed a much more collapsed structure with folding of cell walls in air-dried apples when compared to freeze-dried ones. Lewicki and Pawlak (2003) observed the formation of large cavities in freeze-dried fruits without shrinkage; they also found that during air-drying shrinkage stresses caused numerous cell wall ruptures and the formation of many microcavities. The above results confirm that the structural rigidity of the frozen product during freeze-drying prevents the collapse of the solid matrix remaining after process and fruits tend to have a porous and non-shrunken structure.

In raspberries subjected to osmosis pretreatment, the behavior was different depending on the used combination of treatments (**Fig. 1**). In the case of freeze-dried samples, the application of pretreatments caused a volume reduction in comparison to the control fruit. Freeze-dried samples pretreated with wet infusions, showed volume changes significantly lower (shrinkage values for all WI combinations ≈25%), than freeze-dried DI samples that presented ≈60% shrinkage values. On the other hand, the infusion treatments caused a reduction of the observed shrinkage of air-dried fruits when comparing to the control fruit, being this fact attributed to a protective effect of sugars incorporated into the matrix during osmosis. A shrinkage decrease during the air-drying processes due to osmosis pretreatments has been reported by many authors in various vegetal matrices (Giovanelli et al., 2013; Koc et al., 2008; Mazza, 1983; Nieto et al., 1998; Sitkiewicz et al., 1996; Udomkun et al., 2015). There are few studies including the combination of osmotic dehydration and freeze-drying. Ciurzynska and Lenart (2010) observed by SEM that the osmotic dehydration of strawberries with concentrated sucrose and glucose solutions caused structure strengthening after freeze-drying as compared to freeze-dried fruits without pretreatment. As a consequence of tissue impregnation with sucrose, cellular walls became bulky, while cells closest to the dried surface of the material sustained substantial damage due to sugar crystallization; with glucose as humectant the fruits were uniformly impregnated and the superficial cells were deformed to a smaller degree. Sosa et al. (2012)

when comparing by SEM freeze and air-dried apples with previous infusion in sucrose solutions, confirmed tissue shrinkage in air-dried samples as well as the presence of sugars in several zones.

It is evident that the shrinkage study during drying is difficult, and the complexity is even greater when drying takes place throughout several stages of processing and with different operational variables. Some authors consider that the evaluation of shrinkage alone is not sufficient, since shape changes of the particles are not taken into account. Therefore they introduced the experimental treatment of data based on the so-called "Heywood factor" (De Michelis et al., 2008, 2013; Panyawong and Devahastin, 2007). This parameter gives a better idea of shape changes experienced by the material since shrinkage is neither symmetrical nor uniform in most food types. The reduction of the Heywood factor, results in a deformation of the particle. **Fig. 2** shows the Heywood factor (k) for dehydrated raspberries and the horizontal dashed line corresponds to the Heywood factor for the reference sample. It can be seen that all values, including the reference, are lower than the factor value corresponding to a sphere (0.523) (De Michelis et al., 2013). As expected, freeze-dried samples without pretreatments exhibited a shape factor similar to the reference sample. However, a certain deformation of the fruit was detected. In the case of air-dried fruits, the lowest k value was observed for the control sample, while pretreated samples showed values very close to the reference sample, indicating that not only the infusion pretreatment caused a reduction in the observed shrinkage but it also allowed conserving the fruit shape. In some cases, as in fruits with previous WI, Heywood shape factor varied towards the values for spherical geometry, which may be desirable in certain applications. Samples showing the larger deformation were those subjected to DI pretreatment and freeze-drying.

3.3 *Mechanical properties*

The ingredients added during pretreatments, such as sugar, can affect the structural organization of the product and therefore its interactions with water, which plays an important role in the mechanical properties of materials (Barrett et al., 1994; Roudaut et al., 2002; Udomkun et al., 2015). The concepts of water activity (a_w) and glass transition temperature (T_g) are useful tools to interpret the relationship between material properties and physical and chemical food changes (Venir et al., 2007), but they are not enough to interpret the texture changes. Regarding texture evaluation, the study of mechanical properties of solid bulk particles is important, since they are closely related to how they are broken during mastication (Sandoval et al., 2008). The loss of firmness or crunchiness in raspberries after air and freeze-drying was analyzed through the shear Kramer assay. This assay has been implemented in several kind of foods such as cornflakes (Chaunier et al., 2007), apple pieces (Harker et al., 2002), dried mushrooms (Jaworska et al., 2010), dehydrated tomatoes slices (Lee et al., 1999), dehydrated carrots (Rastogi et al., 2008), cranberries (Beaudry et al., 2004) and raspberries and blueberries (Sousa et al., 2007). **Fig. 3** shows that the Kramer assay applied on dried raspberries led to different experimental force-distance curves. As expected, important forces (> 1 kN) were required to cross over the layer of control fruits and the curves exhibited the typical shape of hard materials, with abrupt rupture peaks (**Fig. 3a**). However, the resistance of the material to the applied force (F_{max}) was lower in freeze-dried samples than in air-dried ones (**Table 2**). This behavior could be due to crust formation during air-drying, typical in food containing sugars and other solutes dissolved in high concentration such as fruits, as well as to the greater shrinkage and the lower water content exhibited by air-dried control fruits. Similar results were reported in apple (Sosa et al., 2012), cranberries (Beaudry et al., 2004) and pumpkin and green pepper (Guiné et al., 2012). However, a decrease in F_{max} value was evident in all pretreated fruits tissues (**Fig. 3b, Table 2**). The shape of the curves for these softer materials showed a gradual increase up to the maximum force, with more rounded peaks, indicating a loss of firmness. The zig-zag top portion observed was probably due to the high friction between the blades caused by adhering sticky pieces of fruits (**Fig. 3b**).

The significant firmness loss observed in pretreated fruits after both drying processes could be due to changes produced at the structural level during the osmotic dehydration stage. On one hand, a porosity decrease can take place by saturation of cell walls and intercellular spaces with sugar penetration. On the other one, during the osmosis step, degradation of polysaccharides can occur, as well as leaching of pectins and other wall-soluble components (Alzamora et al., 2000), thus making pretreated tissues softer and more plastic than control ones. According to Torreggiani and Bertolo (2001) the combination of osmotic dehydration and air dehydration can produce a softer product at low water activity, increasing the consistency with solid gain during osmotic pretreatment. However, Contreras et al. (2005) observed a higher firmness and resistance to deformation in dehydrated apples and strawberries with previous osmotic dehydration (vacuum impregnation with a commercial and isotonic apple juice, 50 mbar for 5 min) attributing this to the generation of a more compact cellular tissue during drying due to sugar content increase. Other authors such as Mandala et al. (2005) obtained different behavior with dried apples depending on the solute used as humectant during osmosis; with glucose they observed an increase in firmness but with sucrose the firmness decrease detected during drying was ascribed to the more severe shrinkage and the loosening of tissue structure. In general, F_{max} values of freeze-dried pretreated raspberries were higher than those for air-dried under the same experimental conditions. On the other hand, the acidified samples showed lower F_{max} values than samples without acid, which is consistent with the higher water content observed in these samples (**Table 1**).

From the additional parameters used to characterize the bulk mechanical behavior of all dried samples (**Table 2**), a significant reduction was also observed in the maximum slope of the curve before the peak (SL_{max}) and the mechanical energy (W) of samples with previous infusion treatments when compared to control ones, which is related to a higher deformability of the matrices. Regarding the SL_{max} values, among the pretreated samples, the freeze-dried ones showed a higher slope than the air-dried ones. This is in accordance with the lower T_g values observed in the pretreated raspberries when comparing to control fruits, particularly for air-dried pretreated samples (**Table 1**). In general, no significant differences were observed between air-dried pretreated samples, whereas in the freeze-dried samples a higher slope was obtained in WI, WI-B and DI-B coinciding with their higher F_{max} values. Analyzing the energy values (W), no differences were observed between the air-dried and freeze-dried raspberries for the same pretreatment, except for DI-BAC samples. Regarding the effect of the additives added in the pretreatment, the samples with lower W values were the acidified ones, especially those treated with wet infusions, indicating that these conditions would lead to softer products because they need less energy to fracture.

From the observed mechanical behavior, it can be said that freeze-dried fruits with previous osmotic treatments could be generally characterized as products with higher firmness ($> F_{max}$) and lower deformability ($> SL_{max}$ and $< \Delta_{Fmax}$) with respect to air-dried samples. When compared to pretreated samples, control fruits exhibited a firmer and hard structure ($> F_{max}$, $> W$) and a more brittle or crispy tissue ($> SL_{max}$). The main reason for the loss of crispness in pretreated fruits is the increase of sugar content. The presence of a concentrated viscous liquid resulted in softer osmosed samples, which were more susceptible to deformation during drying and, after drying, presented a “gummy” rubbery texture.

3.4 *Water sorption isotherms*

The study of water sorption isotherms is relevant in the design of new processing methods and food formulation, the selection of adequate packaging materials, the prediction of optimal storage conditions and the determination of product stability (Arevalo-Pinedo et al., 2004; Vazquez et al., 2013). **Fig. 4** shows the water sorption isotherms at 20 °C for raspberries subjected to the different pretreatments and drying methods. The water sorption curves were typical of products with a high sugar content, in which little water adsorption was observed at low relative humidities and an increase in the amount of adsorbed water occurred at high RHs (Telis and

Sobral, 2001). Freeze-dried control samples showed higher water adsorption than air-dried control fruits, probably due to the higher porosity characteristic of samples subjected to freeze-drying (Delgado and Rubiolo, 2005). On the other hand, the development of a concentrated solids surface layer (crust), and the reduction in tissue porosity and/or shrinkage due to air-drying could be also responsible for the reduced water uptake observed in the air-dried fruits (Sosa et al., 2012).

Making a comparison between control and pretreated samples, there were differences in the shape of the isotherms. These differences could be attributed to the effect of sugar infusion on tissue structure, which may affect the interactions between solutes and water, as well as the composition, as the pretreated samples have high concentrations of sucrose and a lower proportion of fruit tissue in comparison with control samples. Moraga et al. (2004) studied the effect of sugar infusion pretreatment in water sorption isotherms of strawberries; they suggested that the amount of water retained in each sample will be different depending on the different changes that occur in each phase (solid and liquid) due to the pretreatments. The presence of a high sucrose proportion in pretreated samples may affect the water sorption behavior. Sucrose may be present in the raspberries mainly in the crystalline form, and a smaller amount in the amorphous state. According to several authors, the solubilization of crystalline sucrose takes place in the a_w range 0.84 – 0.86 (Lipasek et al., 2012; Penner, 2013). The solubilization is observed as an abrupt slope change in the water sorption isotherm from a_w 0.84 (Yu et al. 2008). This behavior was not observed in the raspberry isotherms. As there are many other components, besides sucrose, that also interact with water and may start solubilizing before, then the isotherm slope starts to increase at a_w values lower than 0.84 ($a_w \approx 0.7$). Similar results were reported for water sorption isotherms of fruits pretreated with osmotic dehydration: pear and apple (Mrad et al., 2013); strawberries (Ciurzynska and Lenart, 2010); papaya (Udomkun et al., 2015).

The pretreated raspberries showed different water sorption isotherm shapes. Samples containing acid (BAC and AC) showed higher water sorption than the other pretreated fruits. Moraga et al. (2004) suggested that pretreated samples having higher amounts of soluble compounds in the liquid phase would retain higher amounts of water. Bisulphite addition did not cause a particular effect on the water sorption behavior.

Water sorption experimental data were fitted to the GAB model (**Table 3**). The GAB equation has been widely used to describe the water sorption properties of foods, because the range of relative humidities where this model is valid is very wide (Timmermann, 2003). The hydration water content (X_0), which corresponds to the first sorption stage described by the GAB equation, corresponded in all the studied samples to a_w values between 0.2 and 0.3. The freeze-dried control sample presented the higher X_0 value, and the pretreated samples without additives (WI and DI) or with only bisulphite (B), recorded the lowest values of this parameter. The values obtained for the constant k_{GAB} were close to ≈1, as in many foods (Timmermann, 2003). This constant represents the difference between the standard chemical potential of molecules in the intermediate mobility zone ("multilayer" also called second sorption stage) and the pure water. The results obtained with the GAB model for raspberries are similar to those reported in other studies carried out with several fruits: strawberry (Janowicz et al., 2007); quince (Noshad et al., 2012); apple, pear and melon (Agudelo-Laverde, 2012); papaya (Udomkun et al., 2015).

The correlation coefficient (r^2) and the statistical parameters, such as the reduced chi square (χ^2) and the root mean square error (RMSE), were used to determine the quality of the fit (Adiletta et al., 2015; Vega-Gálvez et al., 2009). The GAB model showed a good fit to the experimental data since it presented a relatively high correlation coefficient (>0.98), and relatively low RMSE and χ^2 values.

3.5 *Hygroscopicity*

A knowledge of rehydration behavior (both water vapor adsorption and water recovery when the dried material is immersed in water) is essential to evaluate the possible applications of the dried products. Hygroscopicity is a relevant property in dehydrated materials; a highly hygroscopic dehydrated food may rehydrate rapidly and completely, which could be an advantage. However, in some cases, water sorption may affect the structural characteristics and compromise the stability of a dehydrated product (De Santana et al., 2014). According to Rhim et al. (2011), the analysis of water sorption kinetics can contribute to the selection of adequate packaging material and storage conditions. **Fig. 5** shows the kinetics of water vapor adsorption at 75 %RH at 20°C. The water adsorption curves exhibited an exponential growth and hygroscopicity (Hi) was associated with the humidity value reached at equilibrium (**Table 4**). In general, freeze-dried samples were more hygroscopic than air-dried ones. Also, air-dried pretreated samples showed lower water sorption than the control fruit. On the other hand, freeze-dried pretreated raspberries presented lower Hi than the control fruit up to water contents close to 18%. Above this point, pretreated samples shower higher water adsorption values (**Fig. 5**). Freeze-dried WI-BAC and DI-BAC samples showed lower water sorption than the control fruit over the whole range of variables studied.

No effect of the type of infusion was observed, both dry and wet infusion pretreatments showed similar hygroscopicity behavior. Another interesting aspect to analyze is the time required to reach equilibrium. In general, freeze-dried raspberries showed higher initial water sorption rates (Vi) than air-dried samples (**Table 4**).

In particular, the control freeze-dried fruit reached 34% of the total water gain after the first day of storage at 75 %RH, while the control air-dried sample only reached 16%; this behavior could be related to the higher porosity of the freeze-dried fruits. Also, the equilibrium values (Hi) were reached at relatively long storage times, approx. 40 and 70 days for air-dried and freeze-dried fruits, respectively. The longer times required for freeze-dried raspberries could be ascribed to the higher Hi showed by these samples. However, to reach the Hi value corresponding to the air-dried samples (11 – 18%), freeze-dried fruits required 30 days, instead of 40. On the other hand, all the samples reached 50% of their total water gain at approx. 10 days of storage at 75 %RH.

The exposure to a humid atmosphere caused an important T_g decrease (from values between -7 and 4°C at a_w 0.33, to values between -60°C and -72°C at 75 %RH). Therefore, the physical stability of the dried raspberries was noticeably altered; stickiness, softening and, to a certain degree, collapse were observed. The effect of glass transition temperature on physical changes of materials has been thoroughly studied; however, most of these studies were done on model systems containing sugars (De Santana et al., 2014; Levi and Karel, 1995; Roos and Karel, 1991; Vega-Galvez et al., 2014). In these cases, physical changes were detected when the difference between the storage temperature (T) and T_g, ($T-T_g$), was higher than 20°C. However, in vegetable matrices, that have biopolymers that limit the occurrence of structural changes, shrinkage was observed at ($T-T_g$) values higher than 50°C in freeze-dried apple, pear, melon and strawberry (Agudelo et al., 2014). In our raspberry samples, the time required to attain ($T-T_g$) = 50°C was approximately 12 days. These results suggest that if the raspberry products were exposed to a humid atmosphere for short periods of time, no deteriorative physical changes would occur.

3.6 *Rehydration behavior*

The behavior of food during rehydration is a measure of the structural damage suffered by the material during dehydration (Bilbao-Sáinz et al., 2005; Lewicki, 1998). The loss of tissue integrity and the reduction of the hydrophilic properties reduce the capacity of rehydration (Krokida and Maroulis, 2000; Marques et al., 2008). Many dehydrated fruits are rehydrated before use. Therefore, the knowledge of the rehydration behavior is necessary to evaluate the possible applications of the dried products. Rehydration is a complex process that involves the recovery of the properties of the fresh product when the dried material is immersed in water. During rehydration, water moves from the solution to the fruit, soluble solids move from the fruits to the solution, and swelling may also occur. These processes can be influenced by the application of pretreatments, the drying method applied and the drying and rehydration conditions (Moreira et al., 2008). Rehydration kinetics were evaluated by recording the water gain along with the rehydration time at 25°C. Rehydration coefficients (RC) were calculated at 15 min and at the final rehydration time (when the kinetic curves reached a plateau) (**Table 5**). Control samples showed higher RC values than pretreated ones. Control freeze-dried raspberries presented a particular behavior, showing an abrupt rise in the rehydration degree and then remaining stable over time. In contrast, although air-dried samples reached similar RC values to the freeze-dried ones at the end of rehydration process, in the early stages (15 min) they exhibited poor degrees of water recovery.

Osmosed raspberries showed variable behavior, reaching recoveries up to 30 % weight, compared to values close to 42% observed in control samples. Among the pretreated samples, WI and DI showed the higher rehydration coefficient. In general, those raspberries subjected to freeze-drying exhibited at 15 min rehydration more than 80% of the maximum capacity of weight recovery. This behavior can be associated with the changes occurred in chemical composition and distribution of components in the fruit tissue caused by osmotic dehydration.

Similar results were reported for freeze-dried strawberries (Ciurzynska and Lenart, 2010), and air-dried mangoes (Maldonado et al., 2010). According to Lazárides et al. (1999) impregnation with sugars causes a decrease in the water gain capacity. Prothon et al. (2001) and Venkatachalapathy and Raghavan (1998) suggested that the lower rehydration capacity observed in osmosed fruits is related to the lower porosity of these samples, due to the uptake of the osmotic solution by the intercellular spaces.

3.7 *Analysis of Principal Component (PCA)*

PCA was applied to detect patterns between the variables and the analyzed samples. PCA incorporated the information on the studied parameters related to the physical and structural properties for sixteen different dehydration processes in two new, uncorrelated variables termed "principal components" (PC1 and PC2). PC1 explained 56% of the total variance of the data set while PC2 explained 20%. Several observations may be made from the sample score plot for PC1 vs. PC2 (**Fig. 6**). On one hand, samples subjected to freeze-drying are located on the right side of the graph, while those subjected to air-drying are located on the left side. Additionally, freeze dried samples with wet infusions are located in the top half, and freeze-dried samples with dry infusions were grouped within the lower half of the graph. This grouping between samples with different infusion pretreatments was not observed in the air-dried samples.

The mechanical behavior of the different pretreated raspberries was consistent with the observed behavior regarding glass transition temperatures and molecular mobility. **Fig. 6** shows that T_g, F_{max} and SL_{max} are in the opposite direction to the variable t_2, suggesting that samples with higher firmness ($>F_{max}$, $>SL_{max}$), also presented lower molecular mobility ($<t_2$).

The air-dried samples had lower firmness, higher deformability, a lower glass transition temperature (T_g) and higher molecular mobility (t_2) compared to the freeze-dried raspberries. Consistent with these differences, the higher deformability of the tissue in air-dried samples resulted in increased shrinkage (Sh) during that process. On the other hand the wet infused samples, both air-dried and freeze-dried, recorded the highest Heywood shape factor (k), which is grouped in the top half quadrant along with the wet infused samples.

It was also possible to verify that the acidified samples were characterized by a higher tendency to deformation and less firmness due primarily to the increased humidity of these samples, which leads to have a more ductile mechanical behavior (Dobraszczyk and Vincent, 1999).

4. CONCLUSIONS

Different dehydrated raspberry products were obtained. The low glass transition temperature values obtained for pretreated (-8.26 – 3.14°C) and control fruits (freeze-dried = 4.12°C; air-dried = 0.3°C) and the soft and gummy texture of the different raspberries suggest that they have appropriate characteristics to be incorporated as ingredients in complex food like cereal bars, cereal mix and cookies. Also, possible applications include the direct consumption of these fruits as snacks.

Regarding their application as ingredients in a cereal mix, pretreated fruits would be more appropriate, as their RC at short times is relatively low. Pretreated raspberries exhibited RC values lower than 0.19, while RC values for control fruits were higher (0.232 ± 0.002 for air-dried and 0.444 ± 0.014 for freeze-dried). Therefore, suggesting that if the pretreated products are mixed with a liquid such as milk or juice, they can preserve the characteristics of texture during the expected time of consumption of this type of food. On the other hand, the significant firmness loss observed in pretreated fruits (F_{max}<700 N) in comparison with control raspberries (F_{max}>1900 N) evidences that, depending on the dehydration process selected, the characteristics of the final product will be significantly different.

Concerning the physical stability of the dehydrated raspberries, the rate of water adsorption in a 75% RH atmosphere was not very high. It was lower than 1.4 %H_2O/day for all samples, except for the control freeze-fried raspberries (4.7 ± 0.7 %H_2O/day). This suggests that these products would be quite resistant to deteriorative physical changes if they would not be stored for long periods outside the packaging. Also, if the storage will take place in suitable packaging, the physical state of the dehydrated raspberries would not be altered for long periods. In this sense, air-dried pretreated fruits showed lower water sorption (Hi <17%) than control fruits (Hi values of 18.2 % and 21.4 % for air- and freeze-dried, respectively).

The results shown in this work are useful to select the most appropriate processing technology for obtaining high-quality processed raspberries for direct consumption or for incorporation in a composite food, as well as for determination of the better storage conditions.

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Nomenclature

DI	dry infusion
WI	wet infusion
AC	citric acid
B	sodium bisulphite
a_w	water activity
X	water content (g H_2O/100 g)
d.w.	dry matter
TS	total sugar content (g glucose/100 g)
Sh	volumetric shrinkage (%)
k	Heywood shape factor
F_{max}	peak force (N)
Δ_{Fmax}	distance corresponding to the maximum force (mm)
SL_{max}	maximum slope of the curve before peak (N/mm)
W	work or energy at midpeak (J)
C	Guggenheim constant
T_g	glass transition temperature (°C)
t_2	spin-spin relaxation time (μs)
Hi	hygroscopicity (%)
Vi	initial velocity of water vapor adsorption (% H_2O/day)
RC	coefficient of rehydration

FIGURE CAPTIONS

Fig. 1 Volumetric shrinkage (Sh) of raspberries obtained by freeze-drying or air-drying with and without pretreatments. Vertical bars represent standard deviation of the mean. Means with a different lowercase letter are significantly different ($p<0.05$). Lowercase letters were used for interaction between the three studied factors

Fig. 2 Heywood shape factor (k) of raspberries obtained by freeze-drying or air-drying with and without pretreatments. Vertical bars represent standard deviation of the mean. Means with a different lowercase letter are significantly different ($p<0.05$). (- - -) Heywood shape factor of reference raspberry. Lowercase letters were used for interaction between the three studied factors

Fig. 3 Typical force-distance curves of raspberries obtained without pretreatment **(a)**: air-dried (▬) and freeze-dried (---); and with pretreatment **(b)**: WI air-dried (– • • –), DI air-dried (▬), WI freeze-dried (---) and DI freeze-dried (▬)

Fig. 4 Water sorption isotherms at 20 °C of raspberries with the following treatments: C (●), WI/DI (■), AC (▲), B (▼) and BAC (◆). Air drying and WI (a), air drying and DI (b), freeze-drying and WI (c) and freeze-drying and DI (d). The symbols represent the average of the experimental values

Fig. 5 Kinetic of water vapor adsorption at 75 %RH at 20°C of raspberries subjected to the following treatments: C (●), WI/DI (■), AC (■), B (▲) y BAC (◆). Air drying and WI (a), air drying and DI (b), freeze-drying and WI (c) and freeze-drying and DI (d)

Fig. 6 PCA two-dimensional scatter plot based on the first two principal components (PC1 and PC2) generated for the studied dehydration processes and based on data of the analyzed variables. Dry infusions (open symbols): DI, DI-AC, DI-B, and DI-BAC. Wet infusions (grey symbols): WI, WI-AC, WI-B, WI-BAC. Air-drying (circles) and freeze-drying (diamonds). [····] Grouping of samples according to cluster of Euclidean distance

Table 1. Water content (X), glass transition temperatures (T_g) and relaxation time (t_2) of reference and dried raspberries obtained by freeze-drying or air-drying with and without pretreatments.

Drying method	Sample	X (%, d.w.)	T_g (°C)	t_2 (μs)
	Reference	567 ± 14	ND	ND
Air-drying	C	15.9 ± 0.5^{i}	0.83 ± 0.02^{j}	8.5 ± 0.2^{ab}
	WI	11.2 ± 0.3^{bcd}	-4.63 ± 0.08^{h}	9.9 ± 0.2^{bcde}
	WI-AC	16.3 ± 0.6^{ij}	-8.26 ± 0.05^{a}	10.9 ± 0.4^{e}
	WI-B	11.3 ± 0.9^{bcd}	-5.98 ± 0.08^{f}	10.6 ± 0.3^{e}
	WI-BAC	14.4 ± 1.5^{gh}	-6.86 ± 0.03^{e}	10.9 ± 0.3^{e}
	DI	12.7 ± 0.6^{defg}	-5.48 ± 0.04^{g}	11.6 ± 0.5^{e}
	DI-AC	13.7 ± 0.3^{fgh}	-7.39 ± 0.05^{c}	10.5 ± 0.3^{cde}
	DI-B	14.1 ± 0.5^{fgh}	-7.95 ± 0.03^{b}	10.6 ± 0.2^{de}
	DI-BAC	16.4 ± 0.4^{ij}	-7.11 ± 0.02^{d}	10.84 ± 0.15^{e}
Freeze-drying	C	17.7 ± 0.3^{j}	4.12 ± 0.04^{n}	7.2 ± 0.2^{a}
	WI	6.9 ± 0.3^{a}	2.98 ± 0.05^{m}	7.89 ± 0.09^{a}
	WI-AC	11.8 ± 0.5^{bcde}	3.05 ± 0.08^{m}	8.6 ± 0.1^{abc}
	WI-B	7.66 ± 0.03^{a}	2.32 ± 0.03^{l}	7.9 ± 0.3^{a}
	WI-BAC	12.3 ± 0.4^{cdef}	1.93 ± 0.05^{k}	8.62 ± 0.13^{abc}
	DI	10.9 ± 0.2^{bc}	0.67 ± 0.05^{j}	8.42 ± 0.02^{ab}
	DI-AC	14.7 ± 0.3^{hi}	0.12 ± 0.03^{i}	8.71 ± 0.14^{abcd}
	DI-B	10.3 ± 0.3^{b}	2.25 ± 0.14^{l}	8.2 ± 0.2^{ab}
	DI-BAC	13.3 ± 0.2^{efgh}	3.14 ± 0.06^{m}	8.43 ± 0.09^{ab}
Interaction[a]		**Drying*infusion*aditive**	**Drying*Infusion*aditive**	**Drying**

Means within columns with a different lowercase superscript letter are significantly different ($p<0.05$).
[a] Interaction factor obtained from ANOVA. ND: Not determined

Table 2. F_{max}, Δ_{Fmax}, SL_{max} and W of dried raspberries obtained by freeze-drying or air-drying with different pretreatments.

Drying method	Sample	F_{max} (N)	Δ_{Fmax} (mm)	SL_{max} (N/mm)	W (J)
Air-drying	WI	356 ± 14^{abcd}	21 ± 0.5^{cd}	51 ± 3^{ab}	1.9 ± 0.1^{bcd}
	WI-AC	312 ± 22^{ab}	21.7 ± 0.7^{cd}	49 ± 3^{ab}	1.3 ± 0.1^{a}
	WI-B	385 ± 23^{bcde}	22.0 ± 0.5^{d}	49 ± 4^{ab}	2.1 ± 0.1^{de}
	WI-BAC	274 ± 13^{a}	21.4 ± 0.6^{cd}	46 ± 2^{a}	1.3 ± 0.1^{a}
	DI	395 ± 28^{bcde}	20.6 ± 0.6^{bcd}	49 ± 4^{ab}	2.1 ± 0.2^{cde}
	DI-AC	323 ± 11^{abc}	22.1 ± 0.4^{d}	47 ± 3^{ab}	1.55 ± 0.07^{ab}
	DI-B	441 ± 25^{de}	20.3 ± 0.6^{abcd}	66 ± 3^{bc}	2.4 ± 0.1^{de}
	DI-BAC	392 ± 11^{bcde}	21.4 ± 0.5^{cd}	54 ± 3^{ab}	2.0 ± 0.1^{bcd}
Freeze-drying	WI	656 ± 8^{g}	17.8 ± 0.8^{ab}	150 ± 4^{ef}	2.19 ± 0.09^{de}
	WI-AC	358 ± 32^{abcde}	17.8 ± 0.8^{ab}	88 ± 8^{cd}	1.11 ± 0.10^{a}
	WI-B	677 ± 34^{g}	20.5 ± 0.9^{abcd}	161 ± 9^{f}	2.10 ± 0.09^{bcde}
	WI-BAC	425 ± 24^{cde}	17.2 ± 0.5^{a}	103 ± 4^{d}	1.22 ± 0.07^{a}
	DI	413 ± 9^{bcde}	17.8 ± 0.7^{ab}	94 ± 5^{d}	1.50 ± 0.05^{abc}
	DI-AC	464 ± 25^{def}	18.3 ± 0.5^{abc}	93 ± 7^{d}	2.0 ± 0.1^{bcde}
	DI-B	592 ± 20^{fg}	18.7 ± 0.7^{abc}	129 ± 6^{e}	2.24 ± 0.09^{de}
	DI-BAC	498 ± 19^{ef}	20.6 ± 1.2^{abcd}	86 ± 4^{cd}	2.7 ± 0.2^{e}
Control	Air-dried	2271 ± 52^{h}	23.4 ± 0.7^{d}	498 ± 9^{g}	8.4 ± 0.3^{f}
	Freeze-dried	1959 ± 77^{i}	26.6 ± 0.9^{e}	641 ± 20^{h}	5.8 ± 0.3^{g}
Interaction[a]	Drying*infusion*aditive		Infusion*aditive	Drying*infusion*aditive	Drying*infusion*aditive

Means within columns with a different lowercase superscript letter are significantly different ($p<0.05$).
[a] Interaction factor obtained from ANOVA.

Table 3. Parameters of the GAB equation for water sorption isotherms at 20 ° C and statistics used to evaluate the goodness of fit for each experimental condition.

Drying method	Sample	$X_0 \pm$ SD (g H_2O/100 g d.w.)	C ± SD	$k_{GAB} \pm$ SD	r^2	RMSE	χ_2
Air-drying	C	11.3 ± 1.9	10.2 ± 0.6	0.73 ± 0.06	0.98	1.08	1.45
	WI	8.78 ± 0.75^{Abc}	7.6 ± 2.6^{AB}	0.94 ± 0.03^{bc}	0.99	0.83	0.85
	WI-AC	13.14 ± 1.99^{Cc}	8.4 ± 2.4^{AB}	0.91 ± 0.04^{bc}	0.98	1.53	2.86
	WI-B	10.08 ± 0.95^{Abc}	5.6 ± 1.5^{A}	0.93 ± 0.02^{bc}	0.99	0.89	0.97
	WI-BAC	12.05 ± 0.92^{BCc}	10.5 ± 3.7^{B}	0.93 ± 0.02^{bc}	0.99	1.07	1.4
	DI	9.98 ± 0.92^{ABbc}	9.3 ± 2.9^{AB}	0.94 ± 0.03^{bc}	0.99	0.79	0.77
	DI-AC	9.45 ± 0.23^{ABc}	7.5 ± 1.9^{AB}	0.97 ± 0.02^{c}	0.99	2.32	6.53
	DI-B	11.03 ± 1.54^{ABbc}	5.8 ± 2.5^{A}	0.96 ± 0.02^{bc}	0.99	1.24	1.88
	DI-BAC	10.3 ± 0.6^{ABCc}	10.8 ± 3.4^{B}	0.97 ± 0.02^{bc}	0.99	2	4.89
Freeze-drying	C	13.6 ± 1.6	5.2 ± 0.6	0.78 ± 0.03	0.98	3.25	12.9
	WI	8.3 ± 1.2^{Aab}	5.2 ± 0.9^{AB}	0.7 ± 0.2^{a}	0.99	1.28	2.02
	WI-AC	11.15 ± 1.07^{Cbc}	4.6 ± 0.7^{AB}	0.96 ± 0.03^{bc}	0.99	1.18	1.7
	WI-B	6.8 ± 0.9^{Aa}	3.4 ± 0.6^{A}	1.03 ± 0.02^{c}	0.99	0.89	0.97
	WI-BAC	10.1 ± 1.2^{BCbc}	2.6 ± 0.7^{B}	0.79 ± 0.09^{ab}	0.98	1.72	3.62
	DI	9.2 ± 1.2^{ABab}	4.7 ± 1.7^{AB}	0.93 ± 0.04^{bc}	0.98	1.55	2.94
	DI-AC	8.8 ± 1.2^{ABbc}	4.1 ± 1.2^{AB}	1.05 ± 0.04^{c}	0.98	3.04	11.03
	DI-B	7.29 ± 0.06^{ABa}	4.4 ± 0.8^{A}	1.02 ± 0.03^{c}	0.99	0.99	1.22
	DI-BAC	10.4 ± 0.6^{ABCbc}	5.9 ± 0.8^{B}	0.97 ± 0.02^{bc}	0.99	0.84	0.86
Interaction[a]		**Drying*infusion/ Infusion*additive**	**Additive**	**Drying*additive**			

Means within columns with a different lowercase or capital superscript letter are significantly different ($p<0.05$).
[a] Interaction factor obtained from ANOVA.

Table 4. Hygroscopicity (Hi, %) and initial velocity of water vapor adsorption (Vi) of raspberries obtained by freeze-drying or air-drying with and without pretreatments.

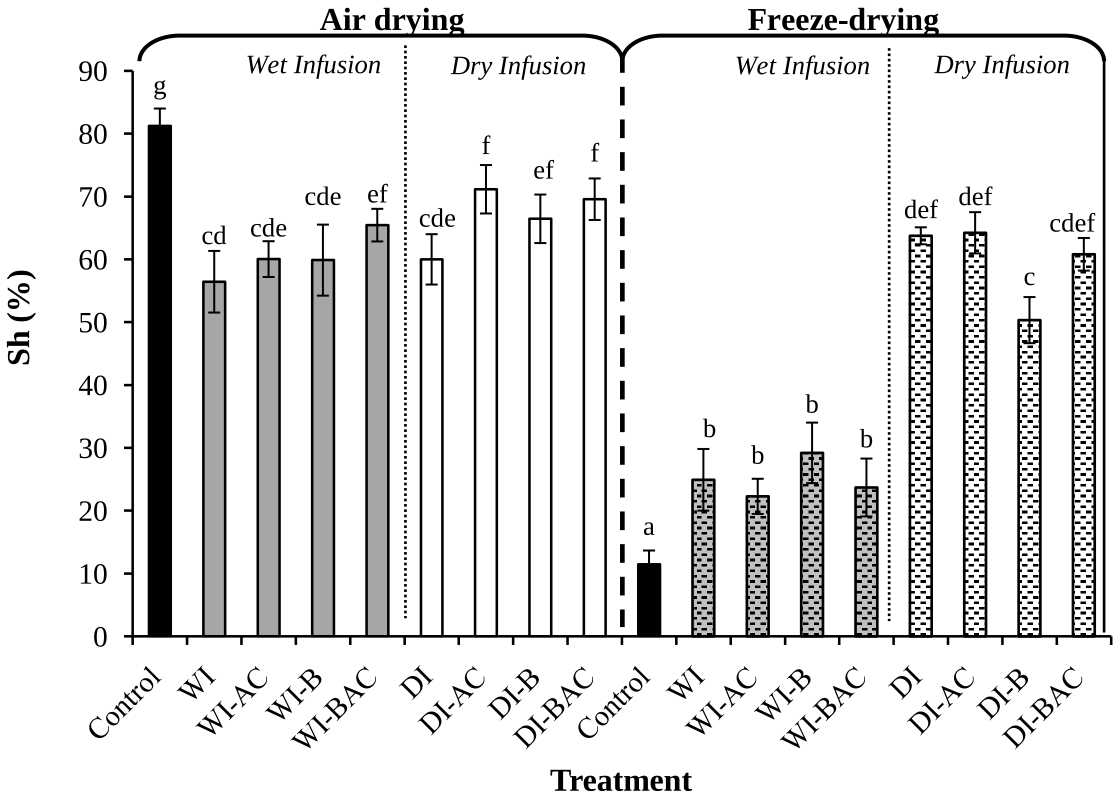
Drying method	Sample	Hi (%)	Vi (% H_2O/day)
Air-drying	C	18.2 ± 0.5^{d}	0.88 ± 0.05^{abc}
	WI	17.05 ± 0.34^{c}	0.69 ± 0.03^{abc}
	WI-AC	16.9 ± 0.5^{c}	0.67 ± 0.03^{ab}
	WI-B	11.5 ± 0.4^{a}	0.49 ± 0.03^{ab}
	WI-BAC	13.9 ± 0.6^{b}	0.46 ± 0.02^{ab}
	DI	14.2 ± 0.4^{b}	0.585 ± 0.007^{ab}
	DI-AC	14.13 ± 0.63^{b}	0.582 ± 0.014^{abc}
	DI-B	16.6 ± 0.5^{c}	0.79 ± 0.03^{abc}
	DI-BAC	14.8 ± 0.3^{b}	0.56 ± 0.03^{abc}
Freeze-drying	C	21.4 ± 0.5^{e}	4.7 ± 0.7^{d}
	WI	23.3 ± 0.8^{f}	0.943 ± 0.014^{abc}
	WI-AC	23.8 ± 0.7^{fg}	$0,672 \pm 0.014^{abc}$
	WI-B	23.8 ± 0.5^{fg}	0.765 ± 0.022^{abc}
	WI-BAC	18.8 ± 0.7^{d}	0.48 ± 0.03^{ab}
	DI	24.8 ± 0.7^{gh}	1.03 ± 0.02^{bc}
	DI-AC	23.2 ± 0.8^{f}	0.89 ± 0.02^{abc}
	DI-B	25.3 ± 0.4^{h}	1.35 ± 0.07^{c}
	DI-BAC	18.6 ± 0.9^{d}	0.48 ± 0.05^{ab}
	Interaction[a]	Drying*infusion*aditive	Drying*infusion*aditive

Means within columns with a different lowercase superscript letter are significantly different ($p<0.05$).
[a] Interaction factor obtained from ANOVA.

Table 5. The coefficient of rehydration (RC) at 15 min and at the end of the rehydration process at 25°C of raspberries obtained by freeze-drying or air-drying with and without pretreatments.

Drying method	Sample	RC 15 min	RC final
Air-drying	C	0.232 ± 0.002[g]	0.42 ± 0.02[j]
	WI	0.173 ± 0.003[abcde]	0.269 ± 0.003[h]
	WI-AC	0.1748 ± 0.0009[abcdef]	0.219 ± 0.004[de]
	WI-B	0.169 ± 0.005[abc]	0.252 ± 0.004[g]
	WI-BAC	0.183 ± 0.002[def]	0.221 ± 0.003[e]
	DI	0.171 ± 0.002[abc]	0.307 ± 0.003[i]
	DI-AC	0.1702 ± 0.0002[abcd]	0.199 ± 0.003[bc]
	DI-B	0.176 ± 0.002[abcdef]	0.229 ± 0.002[ef]
	DI-BAC	0.176 ± 0.003[bcdef]	0.242 ± 0.003[fg]
Freeze-drying	C	0.444 ± 0.014[h]	0.42 ± 0.03[j]
	WI	0.163 ± 0.002[a]	0.274 ± 0.005[h]
	WI-AC	0.1682 ± 0.0006[ab]	0.2074 ± 0.0007[cd]
	WI-B	0.187 ± 0.002[f]	0.197 ± 0.008[bc]
	WI-BAC	0.167 ± 0.006[ab]	0.201 ± 0.002[bc]
	DI	0.179 ± 0.006[cdef]	0.243 ± 0.002[g]
	DI-AC	0.184 ± 0.007[ef]	0.204 ± 0.002[bc]
	DI-B	0.177 ± 0.003[bcdef]	0.192 ± 0.003[ab]
	DI-BAC	0.1714 ± 0.0005[abcd]	0.1836 ± 0.0007[a]
Interaction[a]		Drying*infusion*aditive	Drying*infusion*aditive

Means within columns with a different lowercase superscript letter are significantly different ($p<0.05$).
[a] Interaction factor obtained from ANOVA.


Air drying
Freeze-drying
Wet Infusion
Dry Infusion
Wet Infusion
Dry Infusion
Sh (%)
90
80
70
60
50
40
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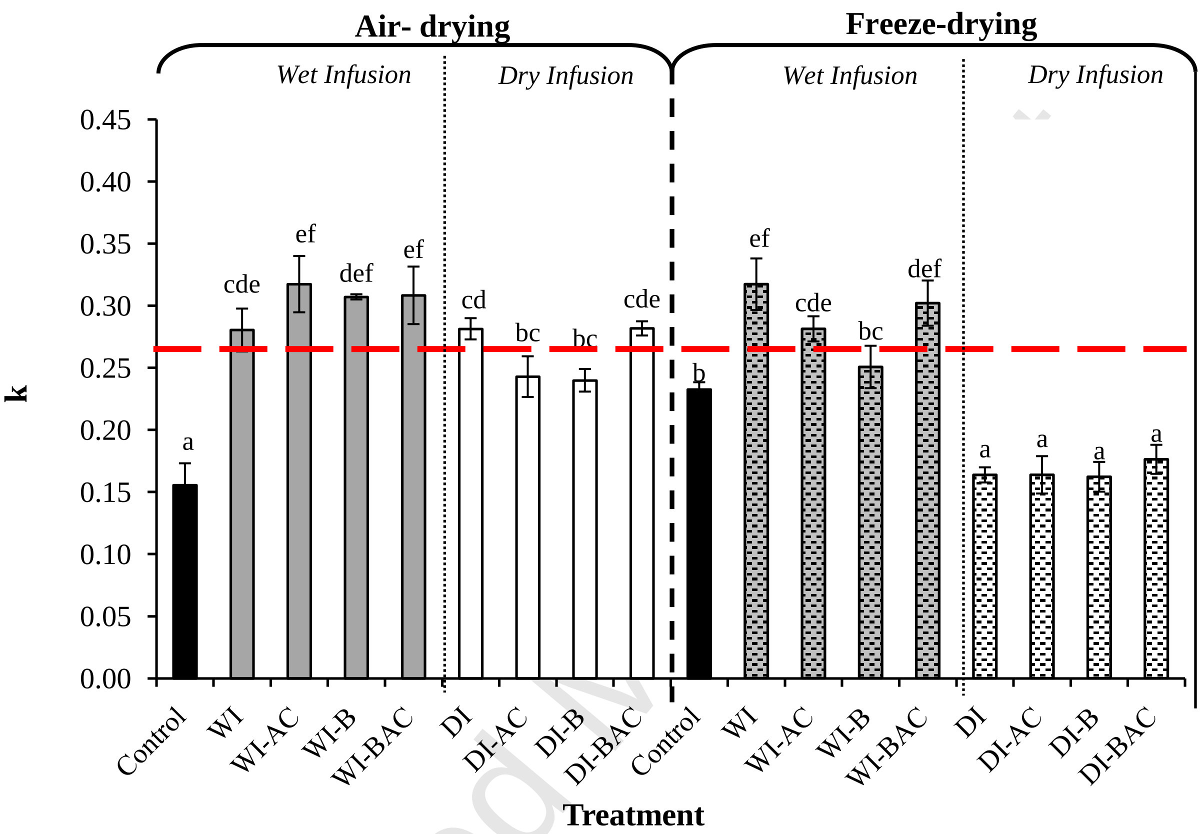
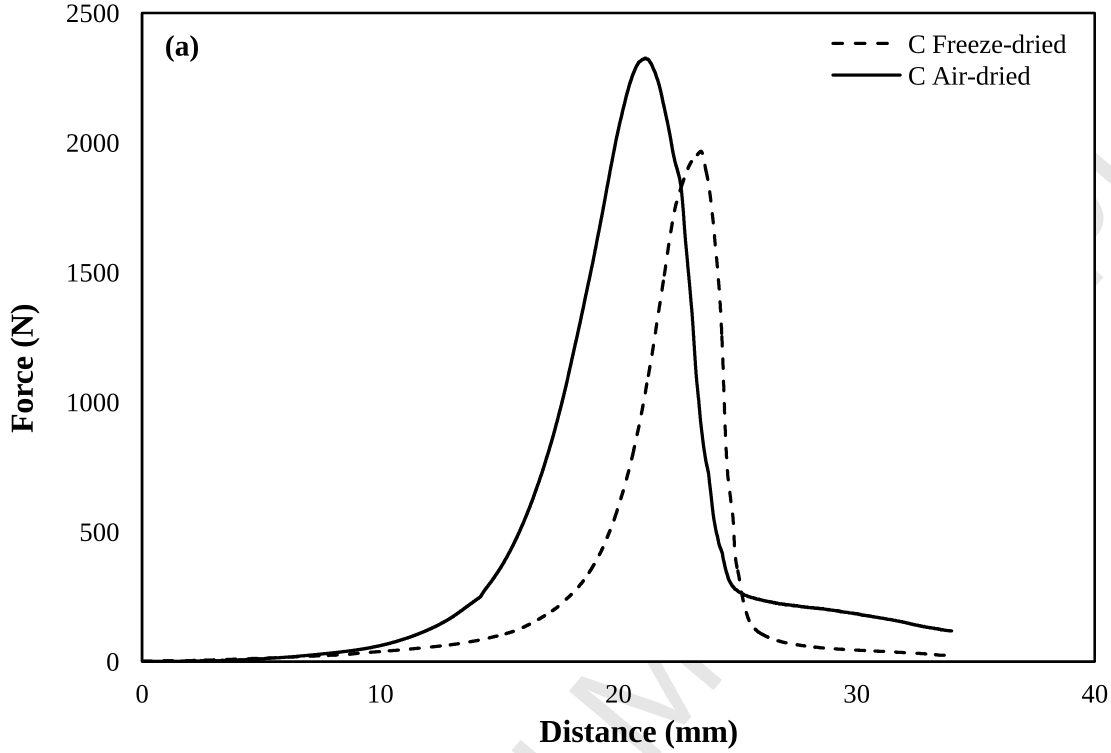
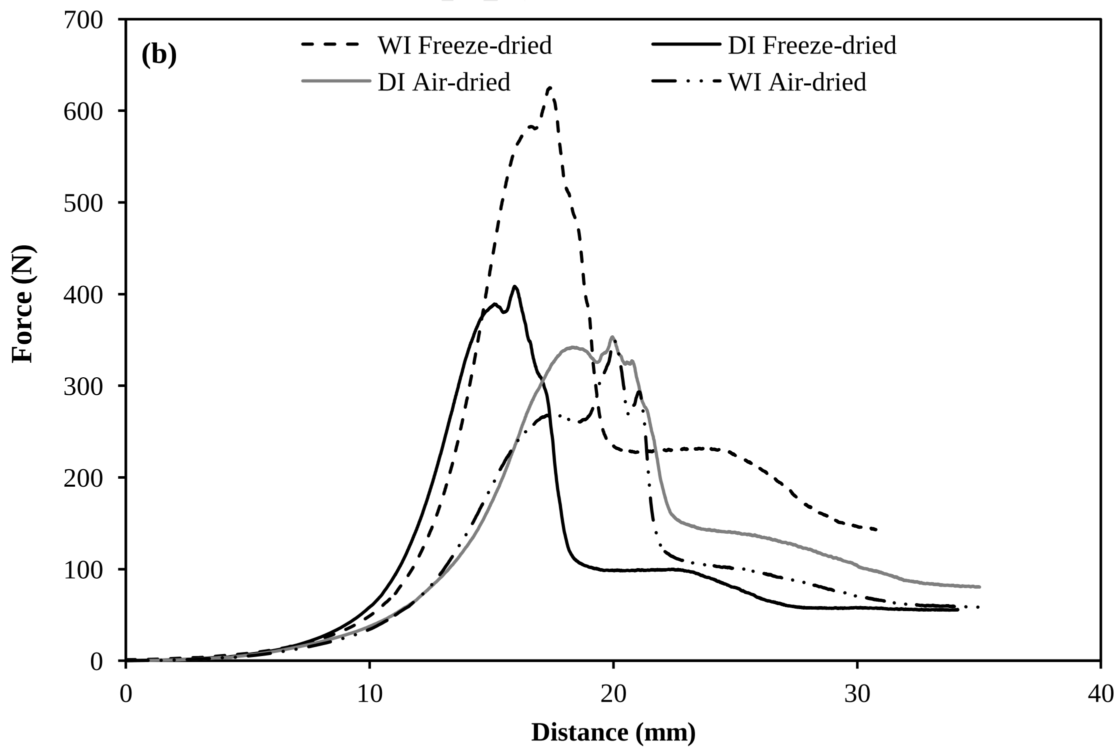
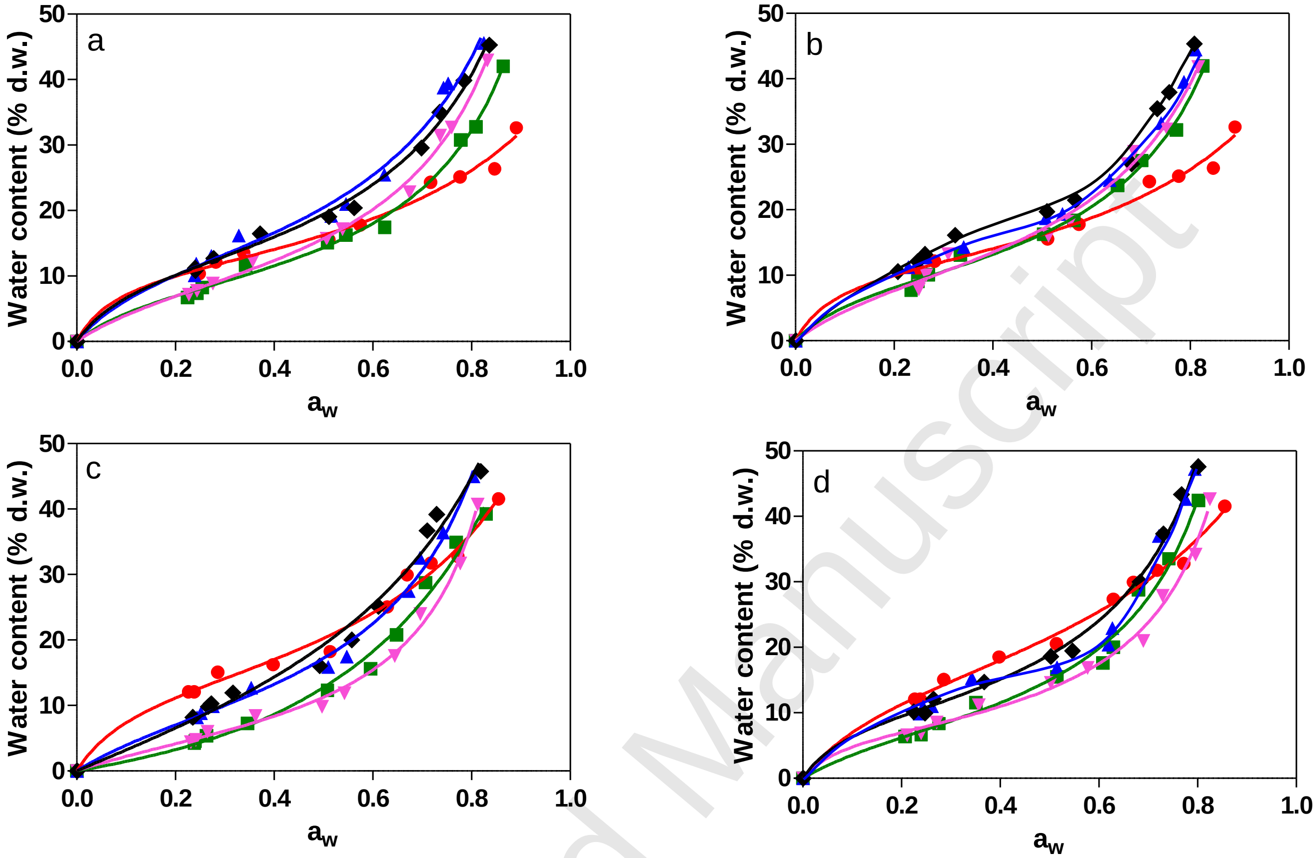
f
ef
f
a
b
b
b
b
def
def
c
cdef
Control
WI
WI-AC
WI-B
WI-BAC
DI
DI-AC
DI-B
DI-BAC
Control
WI
WI-AC
WI-B
WI-BAC
DI
DI-AC
DI-B
DI-BAC
Treatment

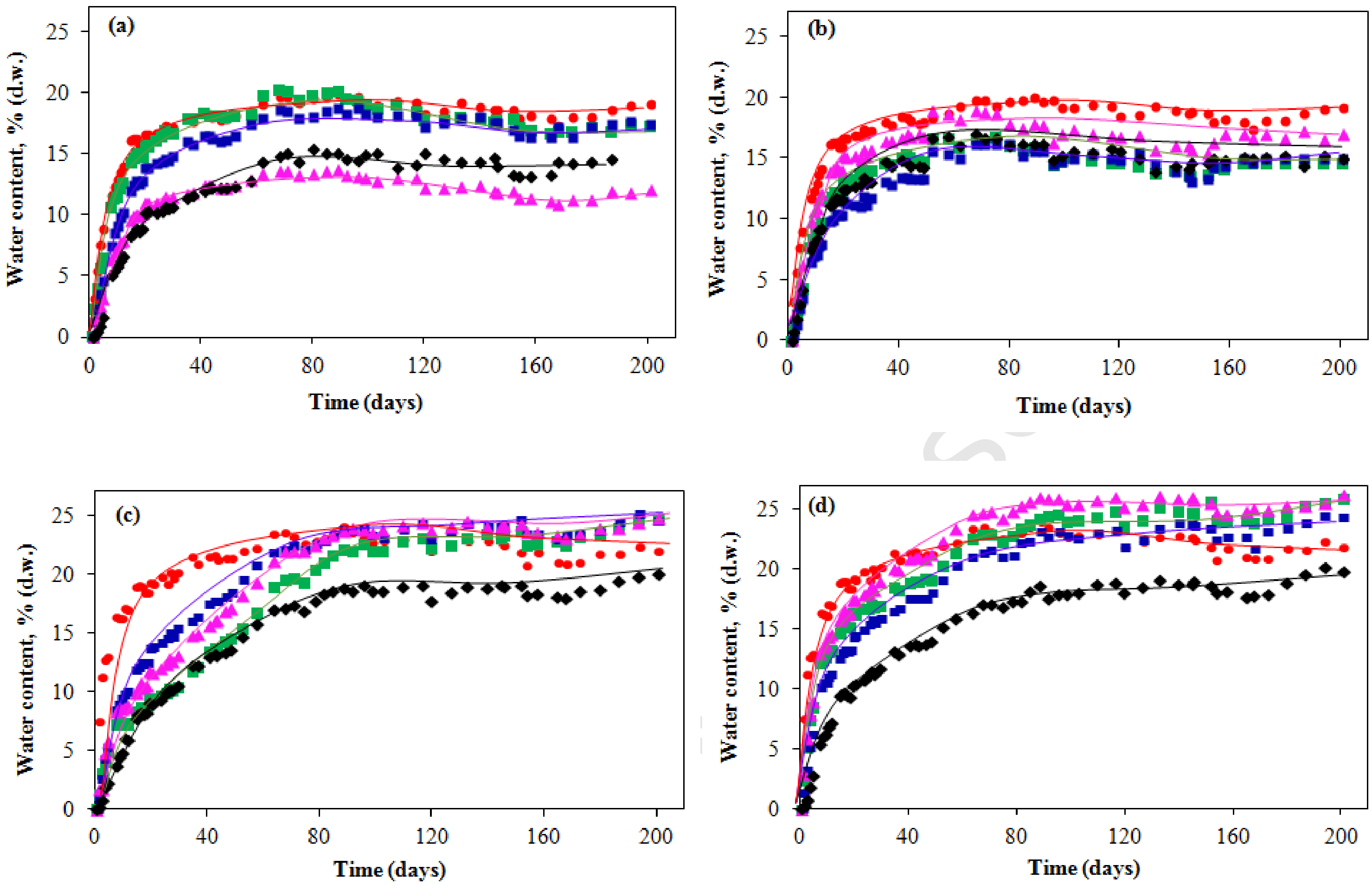


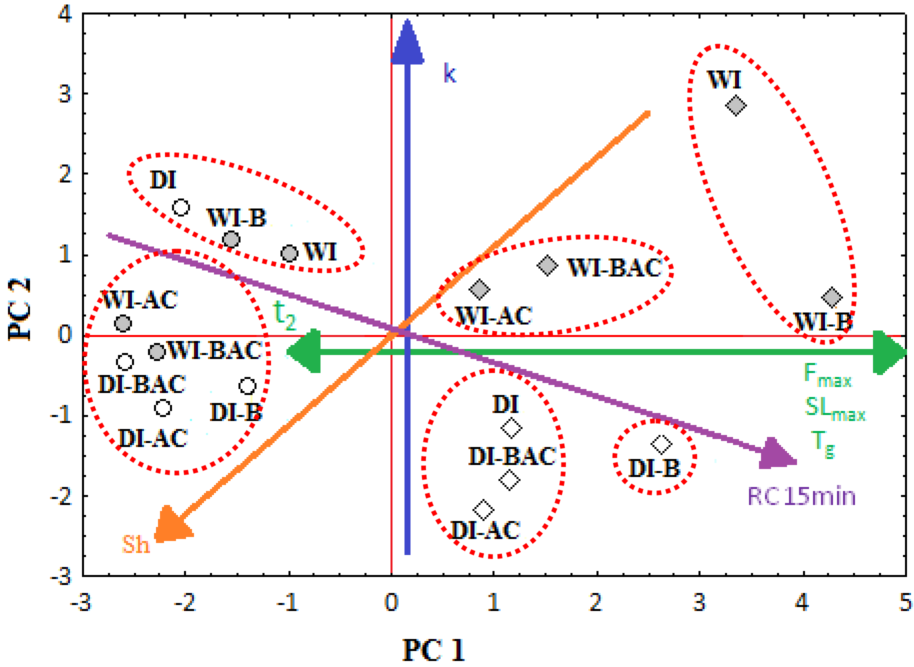
Air- drying
Freeze-drying
Wet Infusion
Dry Infusion
Wet Infusion
Dry Infusion
k
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
a
cde
ef
def
ef
cd
bc
bc
cde
b
ef
cde
bc
def
a
a
a
a
Control
WI
WI-AC
WI-B
WI-BAC
DI
DI-AC
DI-B
DI-BAC
Control
WI
WI-AC
WI-B
WI-BAC
DI
DI-AC
DI-B
DI-BAC
Treatment


(a)
C Freeze-dried
C Air-dried
Force (N)
2500
2000
1500
1000
500
0
0
10
20
30
40
Distance (mm)


(b)
WI Freeze-dried
DI Freeze-dried
DI Air-dried
WI Air-dried
Force (N)
700
600
500
400
300
200
100
0
0
10
20
30
40
Distance (mm)


a
b
c
d
Water content (% d.w.)
a$_w$
0.0
0.2
0.4
0.6
0.8
1.0
0
10
20
30
40
50


(a)
(b)
(c)
(d)
Water content, % (d.w.)
Time (days)
0
5
10
15
20
25
0
40
80
120
160
200



k
WI
DI
WI-B
WI
WI-BAC
WI-AC
WI-B
WI-AC
t2
WI-BAC
DI-BAC
DI-B
DI-AC
DI
DI-BAC
DI-B
DI-AC
Fmax
SLmax
Tg
RC15min
Sh
PC 2
PC 1

Highlights

- “Fresh-like raspberry snacks developed by sugar infusion and further drying”
- "Freeze-dried pre-treated raspberries showed higher firmness than air-dried ones."
- “Physically stable dried raspberries due to low hygroscopicity.”