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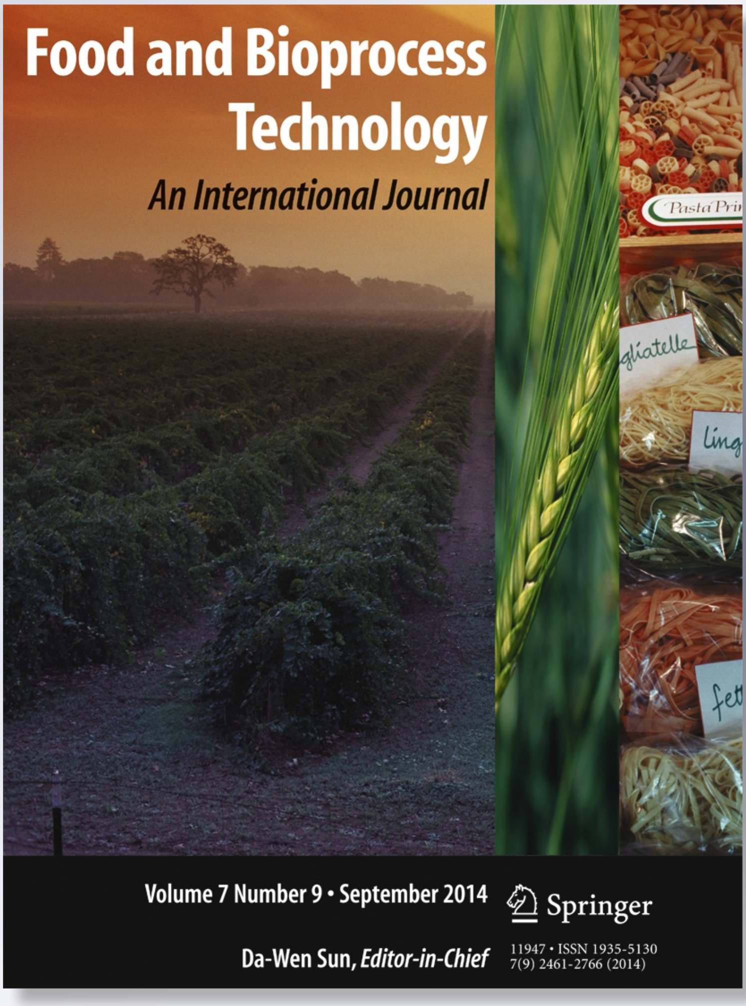

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ORIGINAL PAPER

Evaluation of Structural Shrinkage on Freeze-Dried Fruits by Image Analysis: Effect of Relative Humidity and Heat Treatment

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Abstract Stability of many dehydrated systems is related to the formation of a glass during drying. Highly porous materials, such as those prepared by freeze-drying are susceptible to shrinkage post-drying when stored at not optimal conditions. The aim of this study was to evaluate the occurrence of structural shrinkage on freeze-dried fruits through image analysis in relation to relative humidity (RH) and heat treatment. Freeze-dried fruits were humidified between 11 and 84 % RH at 25 °C for 14 days, and then stored at 45 °C. Sugar and water content, thermal transitions, and volume changes were determined. The shrinkage degree of fresh, lyophilized, humidified, and heat-treated samples was assessed by image analysis. The freeze-drying process did not produce important changes on fruits volume. At low RH values (11–22 % RH), the reduction of the cylinders volume was lower than 15 %. The maximum shrinkage values at each RH were attained in the initial period of humidification (first and second days), and then shrinkage rate decreased markedly, reaching a nearly constant value. The shrinkage degree increased with the increase in RH, reaching values up to 60 % volume reduction. During heat treatment, the volume reduction was less than 10 %. The shrinkage degree was limited by the structural characteristics of the fruits. The structural changes were strongly influenced by variables such as water content, glass transition temperatures, and storage time. In this work, the degree of shrinkage could be followed by image analysis and the use of a mechanical modeling software.

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Keywords Dehydrated fruits · Shrinkage · Freeze-dried · Glass transition

Abbreviations

RH	Relative humidity
CVS	Computer vision system
T_g	Glass transition
DSC	Differential scanning calorimetry.

Introduction

Changes that occur in dehydrated foods during processing, storage, and distribution affect their physical, chemical, and/or biological characteristics, thus influencing structure and appearance and consumer acceptance. Stability of many dehydrated systems is related to the formation of a glass during drying. The transition between the glassy and supercooled states occurs at the glass transition temperature (T_g) (Roos 1993). Most physical changes in amorphous foods result from the sharp decrease of viscosity and increase of molecular mobility in the supercooled state (Levine and Slade 1986; Roos and Karel 1991). Physical changes above T_g include stickiness, caking, porosity loss, and structural collapse, and are related to temperature, water content, and time (Slade and Levine 1991; Roos and Karel 1991). The "collapse" term refers to the loss of structure, reduced pore size, and volumetric shrinkage that occur in partially dry or wet materials. Highly porous materials, such as those prepared by lyophilization are susceptible to shrinkage post-drying when stored at not optimal conditions.

Previous reports determined structural shrinkage in model systems of sugars and maltodextrins, and this phenomenon was analyzed as a function of temperature, through macroscopic and microscopic observations (To and Flink 1978; Gerschenson et al. 1981). Defined temperatures or shrinkage times were recorded indicating the beginning of structural modifications in the samples. The phenomenon was associated with the reduction in matrix viscosity. Later, Levine and Slade (1989, 1990) and Roos and Karel (1990, 1991) discussed the relationship between the glass transition and structural shrinkage.

There are different methods to determine the materials structural changes. Some researchers have expressed shrinkage as a function of the change of selected dimensions of the samples, measured with vernier or digital calipers (Guohong et al. 2012, Mayor and Sereno 2004). Another form is to express shrinkage in terms of the apparent volume. This volume can be measured by the Archimedes principle or by a number of displacement techniques (Yan et al. 2008, Kurozawa et al. 2012). In the last few years, emphasis has been placed on techniques applying computer vision systems (CVS) and image analysis for assessing different quality properties including the shrinkage degree (Fernández et al. 2005; Ören et al. 2006; Zheng et al. 2007; Kurozawa et al. 2012).

The aim of this study was to evaluate the occurrence of structural shrinkage on freeze-dried fruits through CVS and image analysis in relation to their physical properties, as a function of relative humidity (RH) and heat treatment.

Materials and Methods

Fruits

Fully ripe fresh apples (Granny Smith), melons (honeydew), pears (Packham's Triumph), and strawberries (Camarosa) were obtained from the local market and stored at 4 °C until the moment of the experiment. The fruits were washed, peeled (except for strawberry), and were cut in cylinders (1.5 cm diameter and 0.7 cm thickness). The cut material was immediately frozen with liquid nitrogen and stored at −20 °C.

Materials Preparation

Fruit cylinders were covered with liquid nitrogen before freeze-drying. A freeze dryer (ALPHA 1-4 LD2 Martin Christ Gefriertrocknungsanlagen GmbH, Germany) was employed. The freeze dryer was operated at −55 °C; at a chamber pressure of 4 Pa during 48 h. After freeze-drying, samples were distributed into vials for humidification and subsequent determination of water content and thermal transitions. Fruits cylinders were re-humidified over saturated salt solutions (in a range of 11–84 % RH) in vacuum desiccators for 14 days at 25 °C (Greenspan 1977). After humidification, the fruits cylinders used to determine shrinkage were placed inside glass vials hermetically sealed to avoid water loss. Then, they were placed in a forced air oven operated at 45±1 °C during 7 days. Six fruits cylinders for each relative humidity were removed every certain time to acquire images and placed back in the oven.

Sugar Content

Approximately 1 g of freeze-dried fruit was suspended in 10 mL of ethanol (80 %) by mechanical stirring. The resulting suspensions were maintained at 4 °C overnight. Sugar analysis was performed by HPLC (Kontron Eching Germany) isocratically, employing a Thermo Hypersil Amino column (5 μm, 250 mm×4.6 mm) and a refractive index detector. Acetonitrile/water mixture (70:30) was employed as the mobile phase. An average value of two replicates was reported along with the standard deviation.

Water Content

The water content was determined gravimetrically by difference in weight before and after vacuum drying over magnesium perchlorate at 80 °C for 48 h. An average value of at least two replicates was reported along with the standard deviation.

Mathematic Model

The mathematical form of the Guggenhein-Anderson-de Boer (GAB) is:

$$M = \frac{m_o * C * K * a_w}{(1 - K * a_w)(1 - K * a_w + C * K * a_w)}$$

Where m_o is the monolayer value (hydration limit) expressed in grams H_2O/100 g dry solids, C is the kinetic constant related to the sorption in the first layer, and K is the kinetic constant related to multilayer sorption. M is the water content in grams H_2O/100 g dry solids at each $a_{w.}$

Thermal Transitions

Glass transitions (T_g) were determined by differential scanning calorimetry (DSC) using a DSC 822e Mettler Toledo calorimeter (Schwerzenbach, Switzerland). The instrument was calibrated with pure water (0 °C), indium (156.6 °C), lead (327.5 °C), and zinc (419.6 °C). The samples were placed in the DSC oven at room temperature, they were cooled up to −100 °C, and then a heating program from −100 °C up to 100 °C at 10 °C/min was applied. All measurements were performed under a dry nitrogen gas flux of 200 mL min^{-1}. Hermetically sealed 40-μL medium pressure aluminum pans were used (an empty pan served as a reference). Thermograms were evaluated using Mettler STARe software. Glass transitions were recorded as the onset temperature of the discontinuities in the curves of heat flow versus temperature.

Computer Vision System

Fruits volume changes were determined by image analysis. The CVS consisted of three elements: a lighting system, a digital camera, and a personal computer. The lighting system included a D65 lamp which corresponds to solar irradiation with a color temperature of 6,500 K (Lozano 1978) inside a gray chamber (N7 in the Munsell color space). A high-resolution (10.1 megapixel) digital camera, an EOS 40D (Canon Inc., Japan), was employed, with an EF-S 60 mm f2.8 macro lens (Canon Inc.). The digital camera was operated in manual mode, with the lens aperture at f=6.3 and speed 1/8 s (no zoom, no flash) to achieve high uniformity and repeatability. Images have a resolution of 3,888×2,592 pixels and were stored in JPEG format using Canon's Remote Capture program (EOS Utility, Canon Inc., USA). Samples were transferred from the forced air oven to the gray box, and images were acquired at different times during the whole storage period.

The photographs were taken after placing the sample at a distance of 24 cm to camera so as to achieve a 1:1 ratio by size.

Shrinkage Determination by Image Analysis

Volume changes of cylinders were analyzed from the photographs obtained with the computer vision system. Front and side views of the cylinders were taken, then images were processed and the volume was calculated through the mechanical modeling software 3D SolidWorks 2011 (SolidWorks Corp.) (Fig. 1). Shrinkage degree was determined by calculating cylinders volume after freeze-drying (V_0) and after different times of humidification or storage (V_t), and expressed as a percentage. The values reported correspond to the average of six determinations.

CVS Calibration

Two methods traditionally used for volume determination were employed to calibrate the CVS: (1) dimensions measurement (D: diameter and H: height) with a caliper and volume calculation ($\pi D2/4\times H$) and (2) volume determination using a cylindrical pycnometer for solid samples, based on the displacement of water volume by the sample at a constant temperature of 25 °C, employing a thermostatized water bath. The pycnometer was weighed before and after filling it with distilled water using an electronic balance, in order to register its whole volume. The samples were weighed precisely and transferred into the empty pycnometer. The pycnometer with the sample was then filled with water up to the top of its small hole, and weighed after drying the its outside. The displaced water mass was determined and its volume was calculated on the basis of the water density at 25 °C (Yan et al. 2008; Kurozawa et al. 2012).

The volume of 20 fresh fruit cylinders with 3 different diameters (0.6, 1.3, and 1.5 cm) was determined by the two methods mentioned above and by image analysis (as described in the previous section). A linear regression analysis minimizing the root of the mean squares deviation was employed, using the GraphPad Prism 5 software. The obtained correlation coefficients of the values from image analysis with dimension and pycnometry measurements were 0.9822 and 0.9481, respectively.

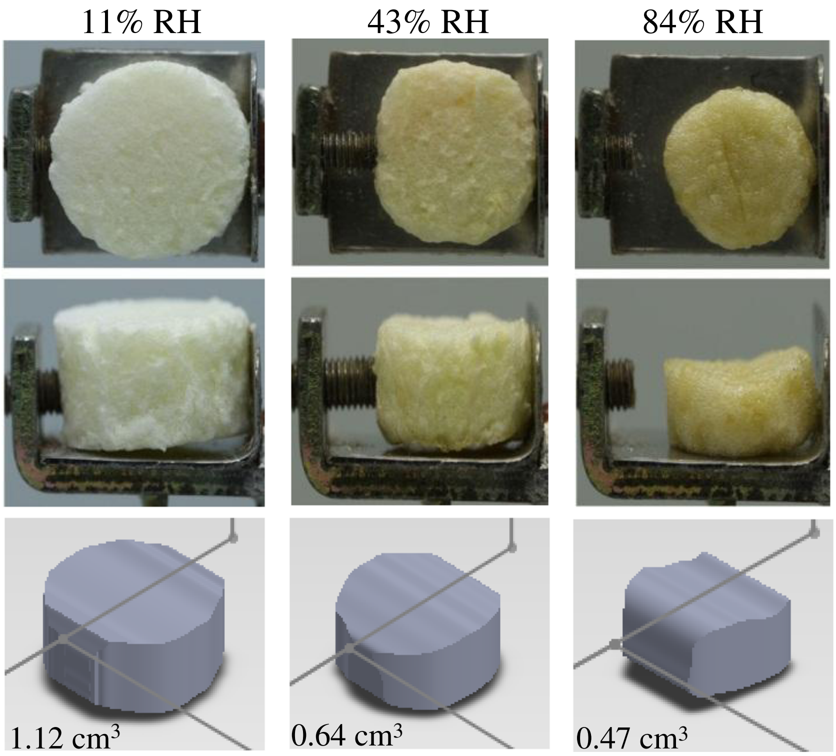
Since a linear correlation was confirmed between the image analysis and the two traditional methods employed to determine the volume, the shrinkage values reported in this work correspond to those obtained directly by image analysis.

Results and Discussion

Figure 2 shows the water sorption isotherms at 25 °C and the glass transition temperatures (T_g) as a function of relative humidity for freeze-dried apple (a), melon (b), pear (c), and strawberry (d). The sorption isotherms presented a sigmoid shape, which is characteristic of most biological products and food components, and corresponds to a type II curve according to the classification of Brunauer and coworkers (1940). Water sorption experimental data were fitted to GAB model (Van den Berg and Bruin 1981) by nonlinear regression analysis, through a program (GraphPad Prism 5) which minimizes the square of the differences between experimental and theoretical values. The curves obtained in this work are typical of high sugar content products, which adsorb low water quantities at low relative humidity and show an increase in the amount of adsorbed water between 52 and 75 % RH. Similar water sorption isotherms were reported for apple (Moraga et al. 2011; del Valle et al. 1998; Acevedo et al. 2008; Venir et al. 2007), melon (Agudelo-Laverde et al. 2011), pear (Guiné and Castro 2002; Nguyen et al. 2004), and strawberry (Mosquera et al. 2012; Moraga et al. 2004).

The four freeze-dried fruits showed very clear glass transition temperatures (T_g), with high changes in heat capacity (delta C_p) values in a range between 0.6 and 1.2 J/g K, and the typical decrease of T_g values with RH increase (Fig. 2). The T_g data were similar to those reported for apple and pear (Khalloufi and Ratti 2003), melon (Agudelo-Laverde et al. 2011), and strawberry (Mosquera et al. 2012). It is to be noted that the dependence of T_g values for apple, pear, and strawberry on RH is similar, while melon presented higher T_g values at a given RH. Since the T_g values are highly dependent on the sugar content and composition (Roos 1993), the high T_g values of melon in comparison with the rest of the fruits can

Fig. 1 Front and side views of pear cylinders at 11, 43, and 84 % RH and the solid 3D images formed after modeling



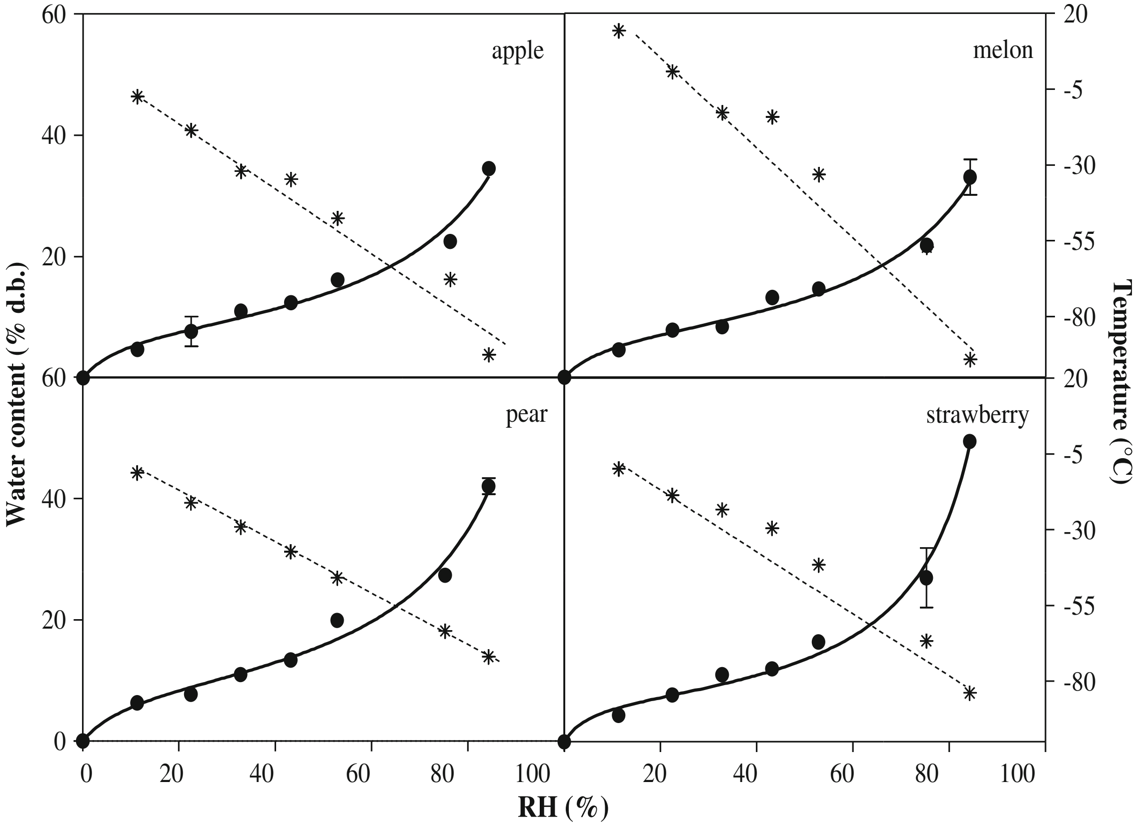


Fig. 2 Water sorption isotherms experimental points at 25 °C for apple, melon, pear, strawberry, and the corresponding GAB fitting (*solid line*); and glass transition temperatures (*stars and dotted line*) versus RH. The standard deviations are plotted in the figures

Table 1 Sugar composition of the studied fruits, expressed in grams/100 g dry solids

Sample	Glucose	Fructose	Sucrose
Apple	17.1±0.6	33.3±0.9	25.8±1.0
Melon	36.5±1.2	19.1±0.7	23.4±0.7
Pear	18.3±0.5	57.5±1.7	10.9±0.3
Strawberry	32.83±0.9	28.08±0.8	29.37±0.8

The average and the corresponding standard deviations are shown

be attributed to its lower proportion of fructose and higher proportion of glucose and sucrose (Table 1). However, it is to be noted that all the fruits were in rubbery state at 25 °C.

The water activity and the glass transition temperature are usually evaluated in the analysis of quality of dehydrated foods. The extent of shrinkage and the glass transition temperature are interrelated since significant changes in volume of the material can be noticed only if the process temperature exceeds T_g, at a particular water content. At temperatures higher than T_g, the viscosity is considerably reduced, facilitating the product deformation (Khalloufi and Ratti 2003). Upon water evaporation during the freeze-drying process, the product becomes porous, and the solid network should be able to hold this porous structure (Khalloufi et al. 2000). If the temperature of the dehydrating porous product is above the glass transition temperature, the viscosity of the solid material may not be enough to support the structure, and collapse or shrinkage occurs. This structural shrinkage leads to poor aroma retention, poor rehydration characteristics, and an uneven dryness. The volume reduction observed in our samples after freeze-drying was in the range of 5 and 7 %. These observed shrinkage degrees are in the range of those reported for freeze-dried strawberry (Shishehgarha et al. 2002; Khalloufi and Ratti 2003), apple, and pear (Khalloufi and Ratti 2003).

It is known that humidification impairs the structure of lyophilized materials (Krokida and Maroulis 2001; Levi and Karel 1995). Figure 3 shows the volume changes (as percent shrinkage) that occurred during humidification at different relative humidities at 25 °C for 14 days. At low RH values (11–22 % RH), the reduction of the cylinders volume was lower than 15 % at the end of storage. Higher volume reduction was detected at and above 33 % RH. Maximum shrinkage was reached at 84 % RH for apple, melon, and pear. In the case of strawberry, the maximum shrinkage was observed from 75 % RH. Probably at higher RH values, strawberry tissue may have swollen water, counteracting in part the effect of shrinkage. It is important to note that strawberry samples reached 50 % water content (d.b.) at 84 % RH, meanwhile the rest of the fruits showed water contents lower than 40 %.

Although melon showed the highest T_g values at each RH (Fig. 1), this was not reflected on a lower shrinkage degree.

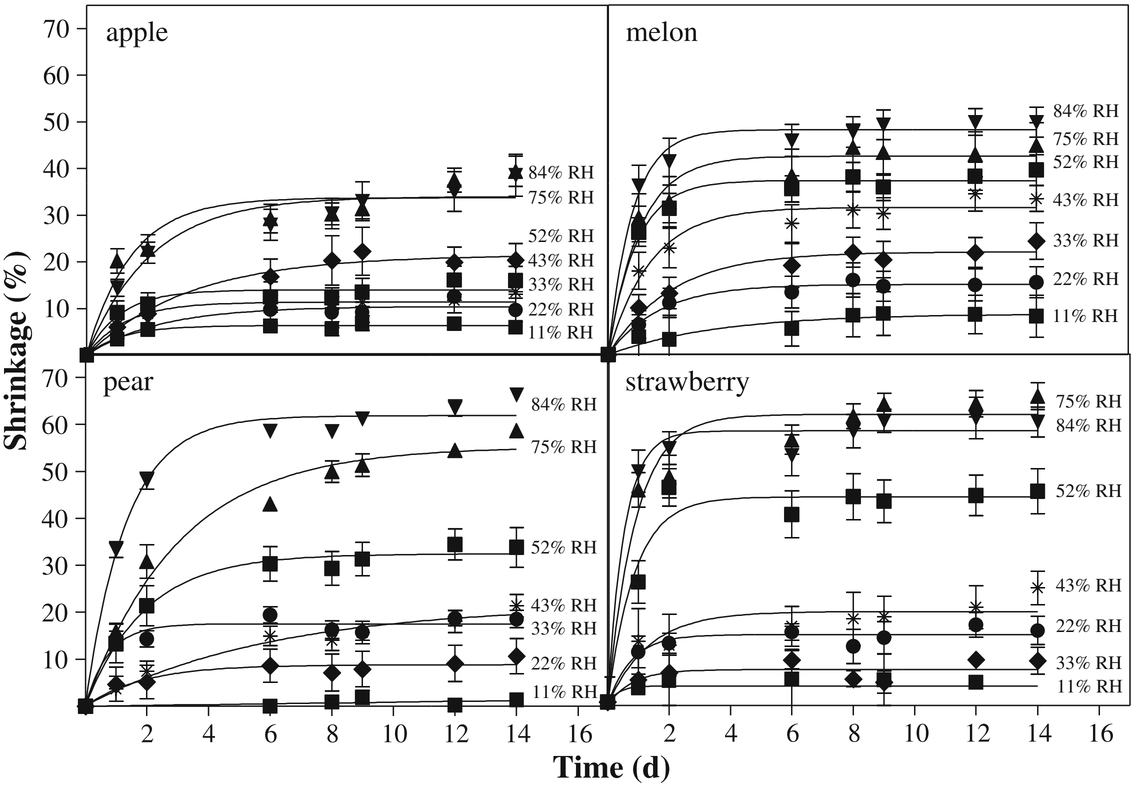


Fig. 3 Volume changes of apple, melon, pear, and strawberry as a function humidification time at 25 °C

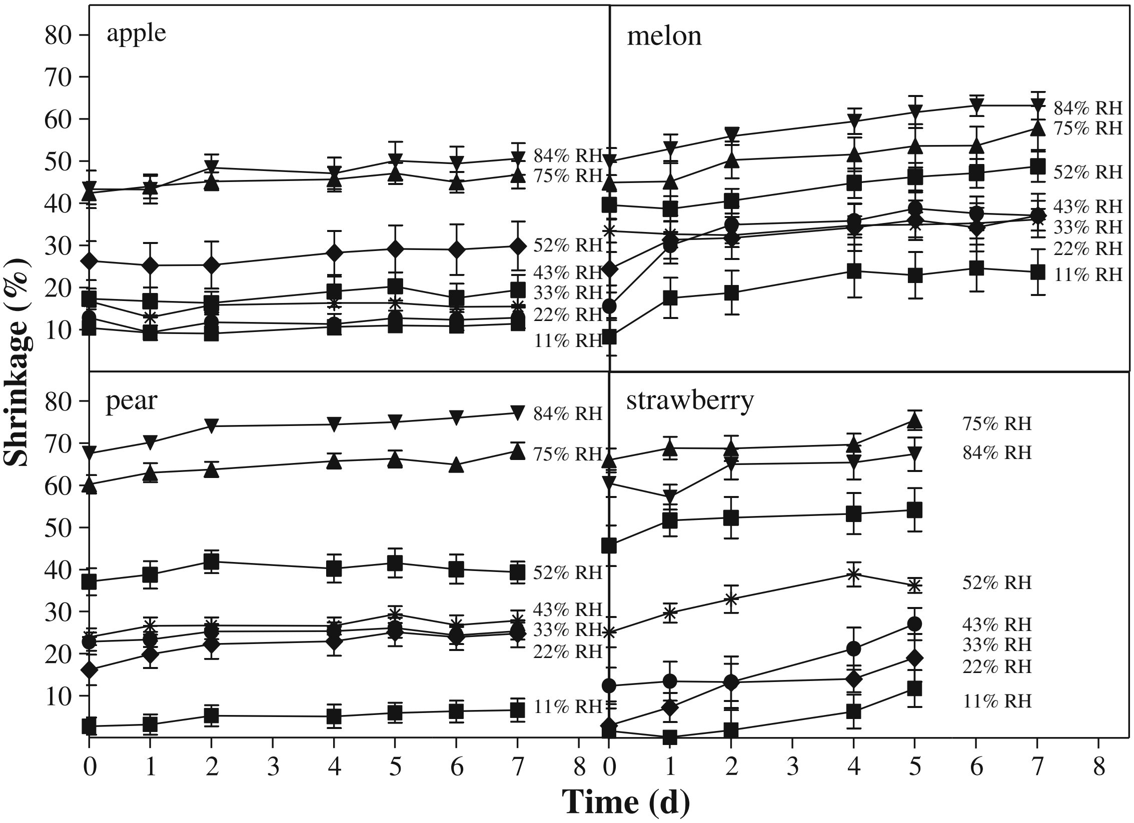


Fig. 4 Volume changes of apple, melon, pear, and strawberry as a function of storage time at 45 °C

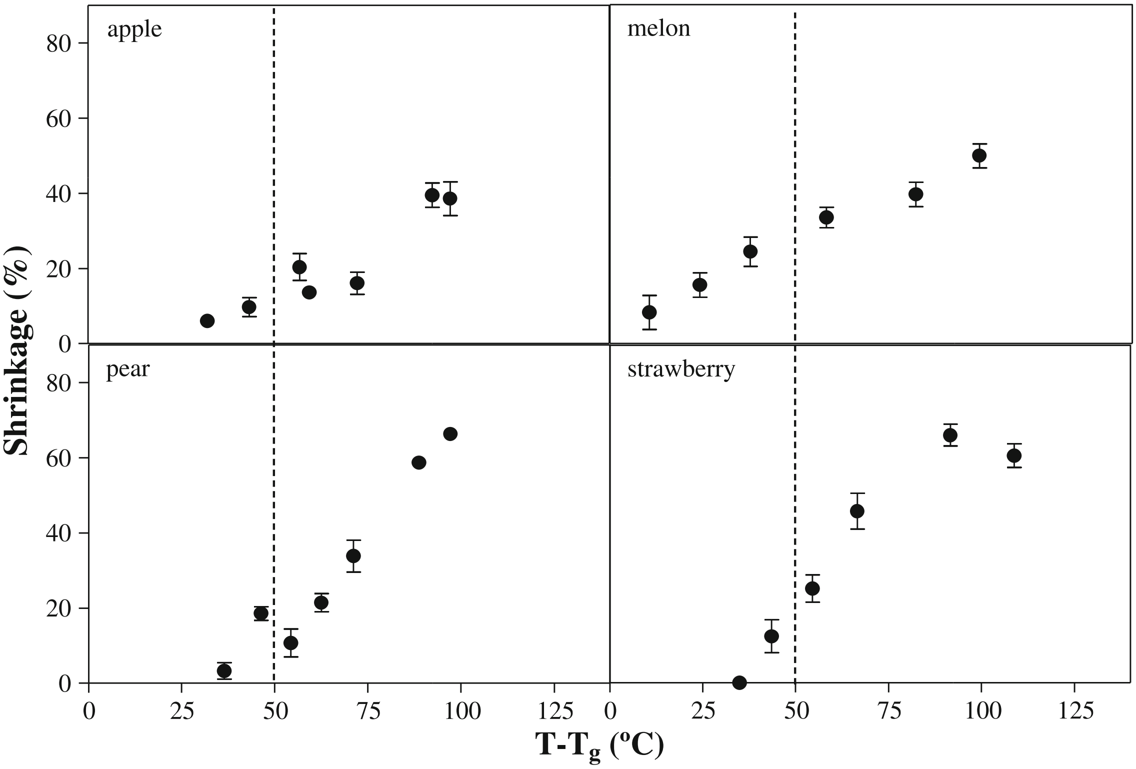


Fig. 5 Volume reduction of apple, melon, pear, and strawberry as a function of ($T-T_g$). T is the storage temperature (25 °C) and T_g is the glass transition temperature

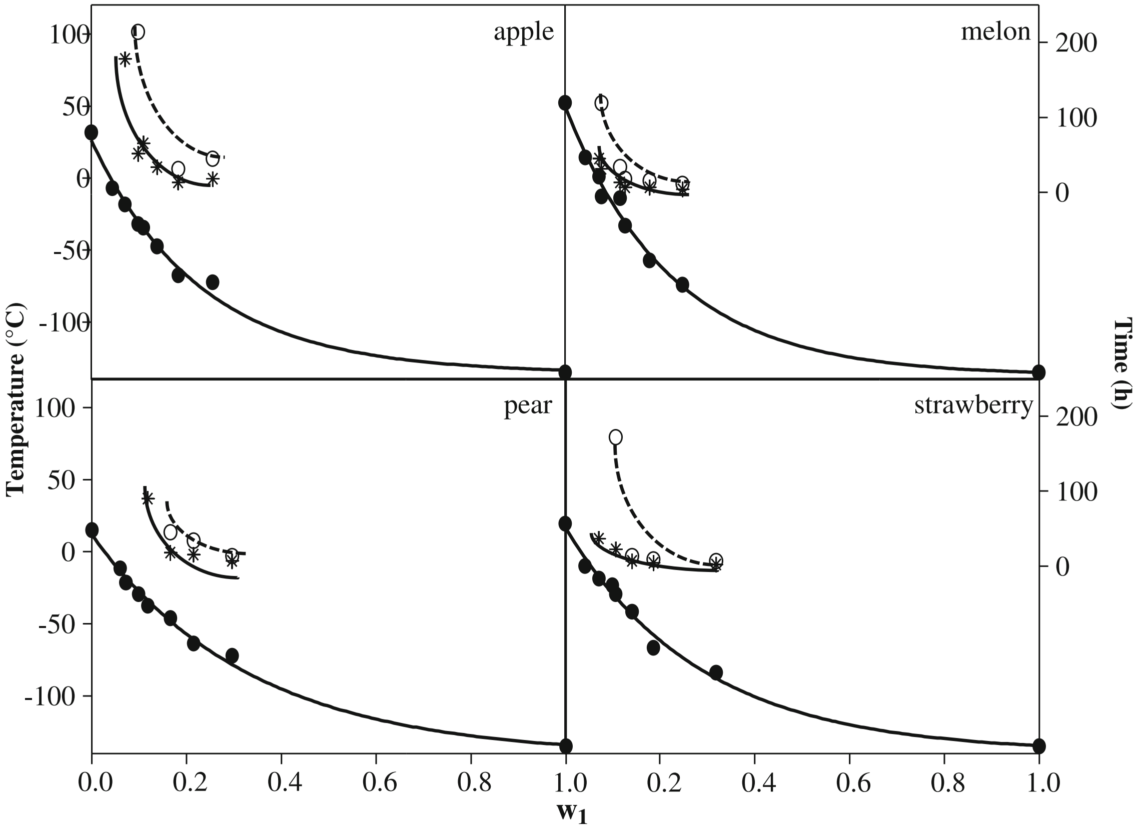


Fig. 6 Glass transition temperature (*black circles and solid line*) and the time in which the material reached 10 % (*stars and solid line*) and 20 % (*open circles and dashed line*) shrinkage as a function of mass fraction of water (w_1, the ratio between mass of water and total mass) of apple, melon, pear, and strawberry. T_g value at w_1=1 from Corti et al. 2010

Apple tissue showed the least shrinkage of all the analyzed fruits (with a maximum value of 40 %), although its T_g values were similar to those for pear and strawberry, which reached volume reduction values higher than 50 %. These observations indicate that, while the T_g values in freeze-dried fruits are related to the sugar composition, as has been reported (Telis and Sobral 2001; Khalloufi and Ratti 2003; Venir et al. 2007), the shrinkage behavior of the different fruits can be mainly attributed to the biopolymers composition showing both the abilities of maintaining the structure (as in the case of apple), and of swelling excess of water (as in the case of strawberry). Dry fruit tissues are complex materials, consisting of many components and more than a single phase, with properties that may not change at the same time as predicted by the glass transition theory (Peleg 1999). Then, the information of the glass transition alone might not be enough to understand texture changes. Mechanical properties are strongly related with the microstructure developed as a result of deformations (shrinkage/swelling) in cells and intercellular spaces and of rupture of cellular bonds taking place throughout drying process (Contreras et al. 2005; Deng and Zhao 2008).

The maximum shrinkage values at each RH were attained in the initial period of humidification (first and second days), and then shrinkage rate decreased markedly, reaching a nearly constant value (plateau). Due to fast shrinkage development observed during humidification at 25 °C it was not possible to determine the shrinkage kinetics.

During heat treatment of the humidified samples at 45 °C, less than additional 10 % volume reduction was observed, as shown in Fig. 4, indicating that the shrinkage occurred by humidification at 25 °C increased slightly by 20 °C increase of temperature. Pear and strawberry samples showed the highest shrinkage degrees during storage at 45 °C, meanwhile apple showed the lowest volumetric reduction.

Previous works reported the dependence of shrinkage on the difference between the storage temperature (T) and the glass transition temperature (T_g) values, and on the storage time for amorphous materials (Levi and Karel 1995; Bonelli et al. 1997). Figure 5 shows the percentage of volume reduction as a function of ($T-T_g$) values for the humidified samples, where T was 25 °C and T_g was the corresponding glass transition temperature at each RH. While all the samples were in the supercooled state at 25 °C, shrinkage values above 20 % were observed at ($T-T_g$) values higher than 50 °C, which corresponds to samples humidified at and above 33 % RH. These properties have been mainly studied in sugar matrices (Roos and Karel 1991; Levi and Karel 1995; Bonelli et al.

1997), where the biopolymers absence allows much higher structural shrinkage values than those observed in more complex matrices as fruit tissues. In sugar systems, the shrinkage is observable from ($T-T_g$) values above 20 °C and the shrinkage development as a function of $T-T_g$ showed an exponential correlation or Williams, Landel, and Ferry (WLF) type behavior (Williams et al. 1955). In this work, the studied materials fell away from this behavior, obtaining an almost linear dependence of shrinkage and ($T-T_g$), except for strawberry samples, which after reaching a maximum shrinkage degree, it decreased at 84 % RH.

Volume changes were visually detected at about 10 % shrinkage. According to Shishehgarha et al. (2002), when the shrinkage percentage exceeds 20 %, it can be considered a high loss in the uniformity of the final products' quality. Figure 6 shows the glass transition temperature and the time at which the material reached 10 and 20 % of shrinkage degree as a function of the mass fraction of water (w_1). Some authors (Levine and Slade 1989, 1990; Roos and Karel 1990, 1991) discussed the relationship between the glass transition and the structural shrinkage, and represented hypothetically different shrinkage degrees in parallel to the glass transition temperature decrease as a function of the water content. As shown in Fig. 6, for all the fruits studied in this work, the postulated parallel behavior was verified in a narrow range of mass fraction of water. The shrinkage degree achieved by a material is limited by the structural characteristics of its components. Parallelism loss can be observed in Fig. 6, in which the time to reach a certain shrinkage degree decreases exponentially with the increase in water content, and then, approaches a constant value.

Conclusions

Structural changes are strongly influenced by variables such as water content, glass transition temperatures, and storage time. Furthermore, in comparison with pure sugar systems, the biopolymers presence affects the tissue structure. The fruits studied at relative humidities below 22 % resulted in stable materials, having ($T-T_g$) values lower than 25 °C, and shrinkage degrees lower than 20 %. Sugar composition affected T_g values at a given RH but this was not reflected on the shrinkage degree, which could be more related to biopolymers as structural components.

These results allowed integrating the information provided by all experiments and demonstrate the importance of the structure and the physical characteristics of the materials in stability and appearance of dried fruit.

Image analysis by computer vision system with the adequate software constitutes a useful tool to study the structural characteristics of food products. This could have more generalized application related with process stages and allow practical, cheap, and nondestructive quality analysis.

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