International Journal of Food Engineering

Some Quality Aspects of Persimmon Jam Manufactured by Osmotic Dehydration without Thermal Treatment

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Abstract

The traditional way of preparing persimmon jam produces astringent flavours due to the conversion of insoluble tannins into soluble tannins as a consequence of the high temperatures used in the process. For this reason, a new method for preparing this kind of product based on osmotic dehydration was tested, in order to obtain persimmon jam without thermal treatment. In this work, 'Rojo Brillante' persimmon slices were dehydrated to approximately 30 ºBrix in concentrated grape juice of 65.8 ºBrix at 30ºC, according to a previous kinetic study. Then, samples were mixed with concentrated grape juice, potassium sorbate, pectin and citric acid in order to reach a concentration of soluble solids of approximately 48 ºBrix. Samples were stored at room temperature, and at 4ºC. Determinations of ºBrix, humidity, astringency, water activity and analysis of optical and mechanical properties (consistency and extrusion) were performed after 1, 5, 7, 12 and 18 days of storage. Non-refrigerated jam spoiled after five days of storage, but the characteristics of the refrigerated samples barely changed, without the presence of astringency. In conclusion, this method of preparing jam from persimmons could increase the commercial possibilities of this fruit, using refrigerated storage.

KEYWORDS: persimmon, jam, osmotic dehydration, colour, consistency

INTRODUCTION

Persimmons are climacteric fruits whose ripening is regulated by ethylene (Wills et al., 1998). Once the climacteric phase begins, rapid softening occurs, resulting in fruit with jelly-like flesh which is difficult to transport (Harima et al., 2003). Some varieties of this fruit have the particularity of being astringent when they are in an immature state (Arnal and Del Río, 2005; Matsuo, 1998; Ragazzini, 1985). When astringent persimmon is eaten, the tannin cells in the flesh are crushed and soluble tannins are released, producing a strong astringent sensation (Taira, 1996). The astringency disappears when soluble tannins become insoluble (Taira et al., 1997; Matsuo, 1998).

The application of techniques to remove astringency, when the fruit still has a firm consistency and external orange colour, has increased the possibilities of commercializing this fruit and exporting it to distant markets. As a consequence, persimmon production (*Diospyros kaki* L.) in Spain has increased from 4000 tonnes in 1997 to 14000 tonnes in 2001 (Martin, 2005) and to 55600 tones in 2006 (GVA, 2008). In Valencia (Spain) the most important variety is the 'Rojo Brillante' persimmon, which is an astringent cultivar of large size and excellent sensory quality (Arnal & Del Río, 2003). This increase in persimmon production has resulted in overproduction (Escutia, 2000). In order to find a use for the excess fruit, there is a need to provide consumers with processed persimmon products such as: fresh cut persimmon (Albors et al., 2008), vacuum impregnated persimmon (Igual et al., 2008a), and snacks (Castelló et al., 2008; Igual et al., 2008b). Another easy way to consume persimmons would be as jam, since this product is highly stable.

Traditional manufacturing methods require concentration by heat treatments, which promote quality changes that affect sensory and nutritional properties. These effects have been studied in different fruits (strawberry, kiwi fruit, orange) by other authors such as Shi et al. (1996) and García-Martínez et al. (2002). Furthermore, the traditional way of preparing persimmon jam causes the development of an astringent flavour due to the conversion of insoluble tannins into soluble tannins as a consequence of the high temperatures used in the process. An alternative, to avoid thermal treatment could be osmotic dehydration. This technique allows us to obtain fruit products that have good flavour, aroma and nutritional content with minimal mineral and vitamin losses (Dixon & Jen, 1977; Lenart & Flink, 1984; Ponting, 1973). Osmotic dehydrated fruit is mixed with an osmotic solution in an adequate ratio to produce jam. As the osmotic process is carried out at lower temperatures the new jam product exhibits superior natural colour, good flavour and overall quality (Shi et al., 1996; García-Martínez et al. 2002).

The aim of this work was to obtain deastringented 'Rojo Brillante' persimmon jam by osmotic dehydration and to evaluate the physicochemical and quality parameters of jams, comparing them according to storage temperature.

MATERIALS AND METHODS

Jam preparation was carried out with persimmon fruit var. *'Rojo Brillante'* submitted to a deastringent treatment ($\approx$24 h at 20ºC, 95-98% CO_2) (Arnal et al., 2005). Fruit were then peeled and cut into slices 15 mm thick and 60 mm in diameter.

The first step was to perform a kinetic study to find out the time required for osmotic treatment in order to obtain dehydrated fruit to be used in the production of jam without heat treatment. To this end, slices were osmodehydrated in concentrated grape juice of 65.8 ºBrix at 30ºC. The ratio between concentrated grape juice and fruit was 15:1 (w/w). Soluble solid content (ºBrix,), water content (x_w), water activity (a_w) and the density of samples dehydrated at 15, 30, 60, 90, 120, 180, 240, 360 and 1350 minutes were analysed.

The kinetics of OD processes are usually evaluated in terms of water loss (ΔM_w), weight loss (ΔM_t) and solutes gain (ΔM_s), (Fito and Chiralt, 1997) and mainly depend on raw material characteristics (Raoult- Wack, 1994) and operational conditions, such as the concentration of the solution and temperature (Barat et al., 2001), exposure time (Escriche et al., 2000) or pressure (Barat et al., 2001; Fito and Pastor, 1994). For this reason changes in total, water and soluble solids mass were calculated in this kinetic study according to Equations 1, 2 and 3.

$$\Delta M_t = \frac{M_t - M_o}{M_o} \qquad (1)$$

$$\Delta M_w = \frac{M_t \cdot x_{wt} - M_0 \cdot x_{wo}}{M_o} \qquad (2)$$

$$\Delta M_s = \frac{M_t \cdot x_{st} - M_0 \cdot x_{so}}{M_o} \qquad (3)$$

Where: ΔM_t is the total mass variation, ΔM_w is the water mass variation and ΔM_s is the soluble solids gain. M is the mass and the mass fractions are represented by letter x. Subindex s means soluble solids and w means water. The subindex t means values registered at the considered time and 0 means initial values.

The effective diffusivity, D_e, ($m^2 s^{-1}$) of the osmotic dehydration of persimmon slices was determined using Fick's model considering an infinitive

slab (Equation 4) (Crank, 1975). The reduced driving force ($Y=Y_s=Y_w$) (Equation 5) was defined as a function of the liquid phase concentration of the fruit (Fito and Chiralt, 1997).

$$1 - Y = \left(\frac{4D_e t}{l^2 \pi} \right)^{1/2} \tag{4}$$

$$Y = \frac{(z_s^t - z_s^e)}{(z_s^0 - z_s^e)} = \frac{(z_w^t - z_w^e)}{(z_w^0 - z_w^e)} \tag{5}$$

Where: Y is the driving force (dimensionless), D_e (m^2/s) is the effective diffusivity, t (s) is the time of the osmotic treatment, l (m) is the semi-thickness of the slices, z_s^t, z_w^t are the mass fractions of soluble solids and water in the liquid phase respectively, at each time of the osmotic treatment, z_s^e, z_w^e are the mass fractions of soluble solids and water in the liquid phase, respectively, in equilibrium (in the concentrated grape juice), and z_s^0, z_w^0 are the initial mass fraction of soluble solids and water in the liquid phase respectively in the slices of fresh persimmon.

According to the kinetic study, slices of persimmon were osmodehydrated in concentrated grape juice of 65.8 ºBrix at 30 ºC for 3 h reaching ≈30ºBrix. After that, samples were ground in a shredder adding 50% of the concentrated grape juice with 1% of pectin as a gelling agent, 0.6 % of citric acid and 100 ppm of potassium sorbate in order to obtain a mix of around 48 ºBrix. Jams contained 61 % fresh fruit.

Finally, jams were left for 24 h before being analysed so that the pectins could work.

Storage conditions

The jams were stored in twist-off jars at 4ºC and at room temperature (≈25ºC) for 18 days, analysing ºBrix, pH, a_w, flow properties and colour after 1, 5, 7, 12 and 18 days of storage.

Analytical determinations

Moisture content was determined by drying to constant weight at 60ºC in a vacuum oven at 10 kPa for 72 h (adaptation of method 934.06 AOAC, 2000). Soluble solids were measured in previously homogenized samples with a refractometer (Zeiss, ATAGO model NAR-3T, Japan). Water activity (a_w) was measured with a hygrometer (Decagon CX-1, AQUA LAB, Washington, USA),

bulk density (ρ_b) was determined by the Mohsenin method (1983), real density (ρ_r) and porosity (ε) were calculated by Equation 6 and 7 respectively.

$$\rho_r = \frac{1}{\dfrac{1 - x_w}{1590} + \dfrac{x_w}{1000}} \tag{6}$$

$$\varepsilon = \frac{(\rho_r - \rho_b)}{\rho_r} \tag{7}$$

pH was measured with a CRISON pH-meter with penetration electrode.

Astringency

An adaptation of the Tannin Print Method (Taira, 1996) was used to evaluate the astringency. This method is based on the reaction of the fruit pulp with papers impregnated with 5% $FeCl_3$, since soluble tannins coming into contact with these papers stains them black. As the deastringent treatment changes the solubility of the tannins, the colour becomes lighter or the papers show no change.

Colour measurement

Colour values were acquired by measuring the reflection spectrum, CIE-L*a*b* uniform colour space, with a spectrocolorimeter (Minolta, CM 3600D, Tokyo, Japan), where L* indicates lightness, a* indicates chromaticity on a green (-) to red (+) axis, and b* chromaticity on a blue (-) to yellow (+) axis. Colour coordinates were obtained from a 10º observer and D65 illuminant. Numerical values of a* and b* were converted into hue angle (h*).

Flow properties

These were evaluated with two different analyses:
Back-extrusion test: The sample (at 20 ºC) was placed on a bakery glass (5 cm diameter), and back-extruded with a 4.5 cm diameter plunger at 1 mm/s deformation rate by using a Texture analyser TA/XT/PLUS. The parameters analyzed were: Force (N) and area of curve Force-distance (A_7) (Nmm) till 7 mm of plunger advance.
Consistency test: The flow distance of a controlled sample weight for a constant time was measured using a Bostwick consistometer. It consists of a level stainless-steel trough divided into two compartments. The first one initially

containing the sample (5x5x3.8 cm) is separated from the second by means of a spring-loaded gate. The second compartment is a trough 5 cm wide, 24 cm long and about 2.5 cm high, and has a series of parallel lines drawn across the floor at 0.5-cm intervals. Once the gate is opened, the distance the sample flows in 30 s is measured (Bourne, 1982). The parameter analyzed was the distance advanced by the samples in the consistometer related to the weight of the samples (mm/g).

Statistical analysis

ANOVA analysis with a confidence level of 95% ($p > 0.05$) using Statgraphics Plus 5.1 Software (Statistical Graphics Corporation, USA) was applied to evaluate differences between samples stored at different temperatures.

RESULTS AND DISCUSSION

Table 1 shows the mean values of water activity (a_w), soluble solids and water content (x_s, x_w) together with values of bulk and real density (ρ_b, ρ_r) and porosity (ε) of "Rojo Brillante" persimmon used as raw material. The values obtained in a previous work (Igual et al., 2008) were similar, indicating that few differences were found between harvests.

Table 1. Water activity (a_w), ºBrix, water and solid content [mass fraction of water (x_w), soluble solids (x_s)], bulk and real density (ρ_b, ρ_r) and porosity (ε) of persimmon.

|	
a_w	0.984 (0.001)
ºBrix	16.5 (0.1)
x_w	0.809 (0.001)
x_{ss}	0.160 (0.001)
ρ_b (kg/m^3)	1031 (4)
ρ_r (kg/m^3)	1076 (1)
ε	0.0419 (0.0009)

Osmotic dehydration kinetics

Figure 1 (A) shows all the values of ($\Delta M_s + \Delta M_w$) versus ΔM_t in order to check the mass variation balances. The slope of the straight line Equation was very close to 1, which means that there was a good balance with the results obtained in the kinetic study. A good fit with the experimental results ($R^2 = 0.9932$) was also seen. The evolution of total mass (ΔM_t), water mass (ΔM_w) and solute mass (ΔM_s) variation are shown in Figure 1 (B). It can be seen that there were two periods in

the rate of the total and water mass loss. In the first one the speed was faster than in the second one since the driving force with time was reduced. Concretely, after 3 hours there was a total mass loss of around 18% and after ≈22.5 h around 45%, which means that almost half of the total mass loss carried out in this study, happened in the first period. In addition, the soluble solids flow was stable after 3 hours.
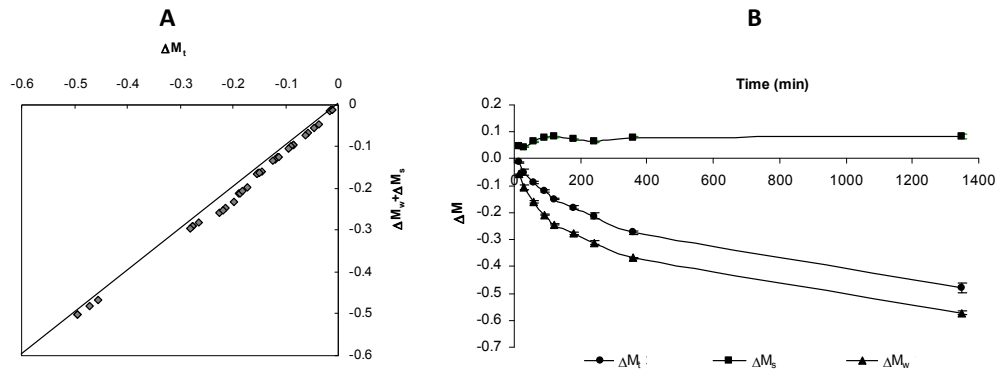
Figure 1. Mass balance: water loss and solute gain plotted against loss of mass (A). Evolution of total mass (ΔM_t), water mass (ΔM_w) and solutes mass (ΔM_s) variation during osmotic dehydration (B).

Figure 2 (A) shows the increase of soluble solids mass fraction in liquid phase (z_s) in persimmon samples according to the time of osmotic dehydration. Between 2 and 4 h the content of z_s was around 0.30 kg of soluble solids per kg of liquid phase. García-Martínez et al., 2002, osmotically pre-treated slices of fruit for 1 h, to prepare them for jam, to similar values of z_s (0.26 for kiwi and 0.23 for orange).
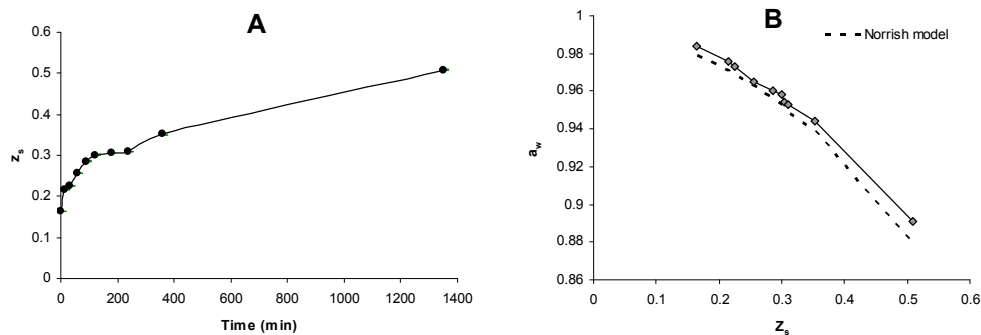
Figure 2. Evolution of concentration level of soluble solids in liquid phase (z_s) during osmotic dehydration (A). Relationship between the water activity (a_w) and the concentration level of soluble solids in liquid phase (z_s) (B).

For these reasons, the time chosen for the osmotic treatment was 3 h, since almost all of the main weight loss occurs in this period and the concentration of soluble solids is high enough to reduce the amount of concentrated grape juice to prepare the final jam without thermal treatment.

Figure 2 (B) shows the values of water activity analysed in samples according to the osmotic dehydration time and also the values predicted by Norrish's Equation (1966) versus the content of z_s (Equation 8). To apply Norrish's model it was considered that the soluble phase of samples was composed of the main sugars in concentrated grape juice with a ratio of 51.6 % glucose to 48.4% fructose (García-Pinchi, 1997). As was expected a_w was lower when z_s increased. Besides, Norrish's values fit the experimental result, which means that the composition of the osmotic solution determined the composition of the soluble solids in this fruit.

$$a_w = X_w \cdot \exp[-(\sum k \cdot X_i)] \tag{8}$$

Where X_w: is the molar fraction of water, k is the constant for each solute and X_i is the molar fraction of each solute (i).
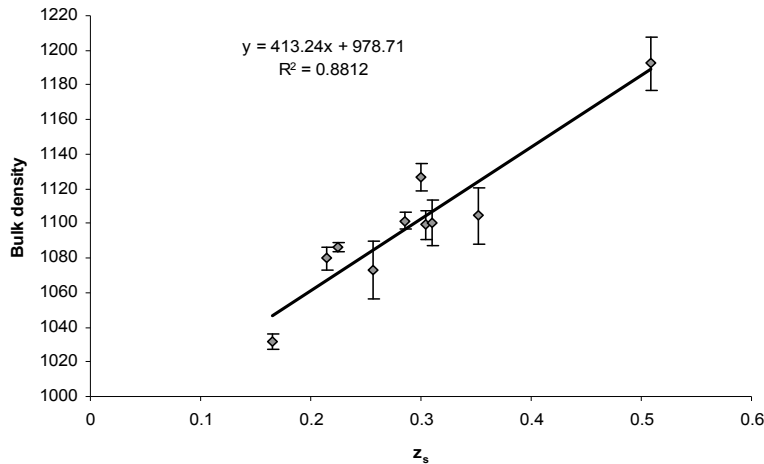
Figure 3. Bulk density during osmotic dehydration versus concentration level of soluble solids in liquid phase (z_s).

After applying Fick's model, as has been commented before in the material and methods section, the value of effective diffusivity obtained in osmotically pre-treated persimmon slices was 2.99×10^{-10} m^{2}s^{-1}. It is remarkable that effective diffusivity is quite high in comparison with the results obtained for other fruit (Moraga et al., 2008; Panadés et al., 2008, Ceballos, 2006, Giraldo et

al., 2003).This result could be due to the differences in the fruit tissue which has different parts, offering less resistance to the osmotic stress.

With regards to the changes of density according to the content of z_s (Figure 3) it was seen that there was an increase in density directly proportional to z_s and the Equation of the fitting straight line allows us to predict the density by knowing values of z_s and vice versa.

Quality parameters in jam

Figure 4 shows that ºBrix remained constant in jam stored at 4ºC while decreasing in jam stored at room temperature, probably due to the fermentation of sugars as could be seen from the development of gases inside these jars after 5 days of storage. With respect to pH, at room temperature, this parameter was lower, showing a greater acidity of jams kept at 25ºC than at 4ºC. Water activity decreased in time after 5 d of storage in both cases, without significant differences between them, maybe because of the gellification of pectins that interact with water in the samples, reducing the available water. No appearance of astringency was observed in any case which supports this methodology for developing a new persimmon jam avoiding consumer rejection.
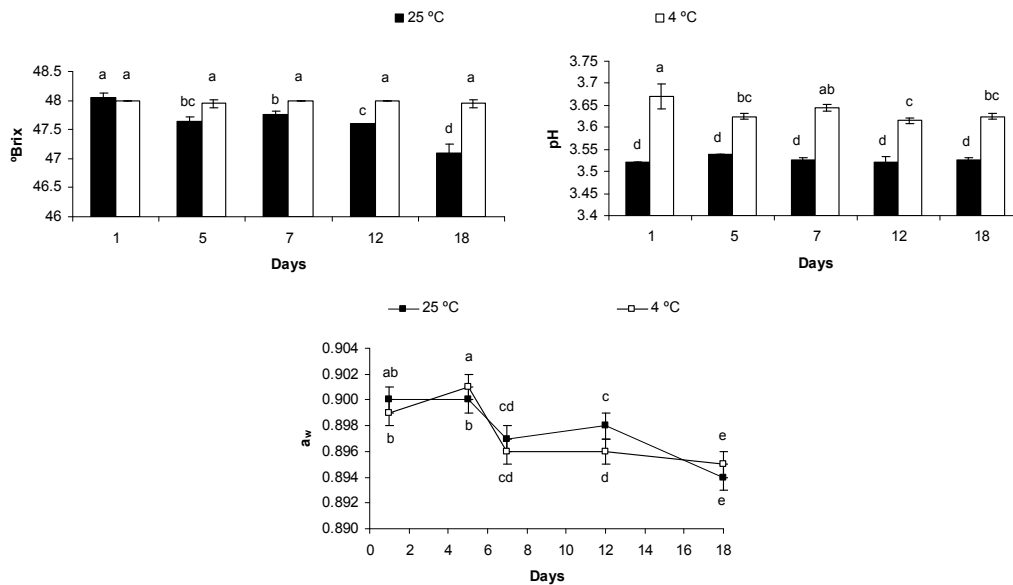

Figure 4. Evolution of ºBrix, pH, and water activity (a_w) for persimmon jam at 25 and 4ºC during 18 days of storage. Letters (a-d) indicate homogeneous groups established by the ANOVA ($P<0.05$).

Figure 5 shows the back-extrusion curves for persimmon jam at 25 and 4°C initially (A) and after 18 days of storage (B). The results of texture analysis indicated that temperature hardly affected the behaviour of force-distance curves. However, after storage it was seen that refrigerated jam was firmer than that kept at room temperature. This behaviour could be related to the fact that pectinesterase enzyme is more active at room than at refrigerated temperature, causing consistency loss, as was observed by other authors (Umme et al., 1999; Umme et al., 2001).
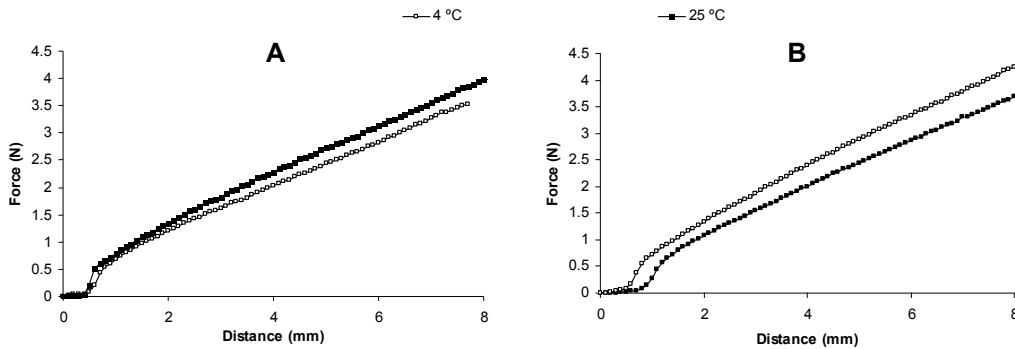
Figure 5. Back-extrusion curves for persimmon jam at 25 and 4°C initially (A) and after 18 days of storage (B).
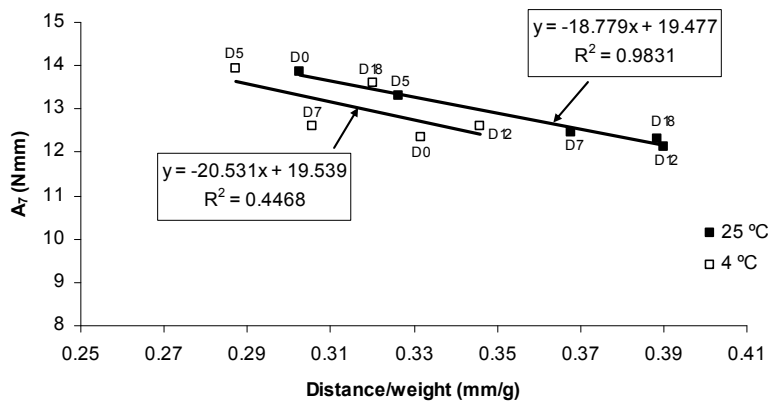

Figure 6. Relationship between the back-extrusion parameter A_7 (area below the force-distance curve till 7 mm extrusion distance) and Bostwich consistency for persimmon jam at 25°C and 4°C considering days of storage (D0, D5, D7, D12 and D18).

According to Figure 6, only in the case of jam kept at room temperature was there a good correlation between A_7 and the distance/weight relationship

during storage time, jam being less consistent with the increase in storage time. In general the mechanical behaviour of jam kept at 4 ºC was similar to that kept at 25 ºC (lines are parallel), although the first ones had greater consistency as a consequence of the reduction in the speed at which changes in the mechanical behaviour took place at lower temperature. Nevertheless, storage time had no effect on consistency at 4ºC.

Figure 7 shows the results of b* versus a* chromatic diagram of fresh persimmon, initial jam and jam stored at 4 and 25ºC. It can be seen that initially there were no changes in colour between fresh persimmon and jam, which supports the fact that this methodology does not affect the colour of fresh fruit. However, after 18 days of storage refrigerated jam lost chrome and hue, because of the decrease of coordinate b*, meanwhile in samples kept at room temperature time had no influence on b* values, but there was a slight reduction in a*.
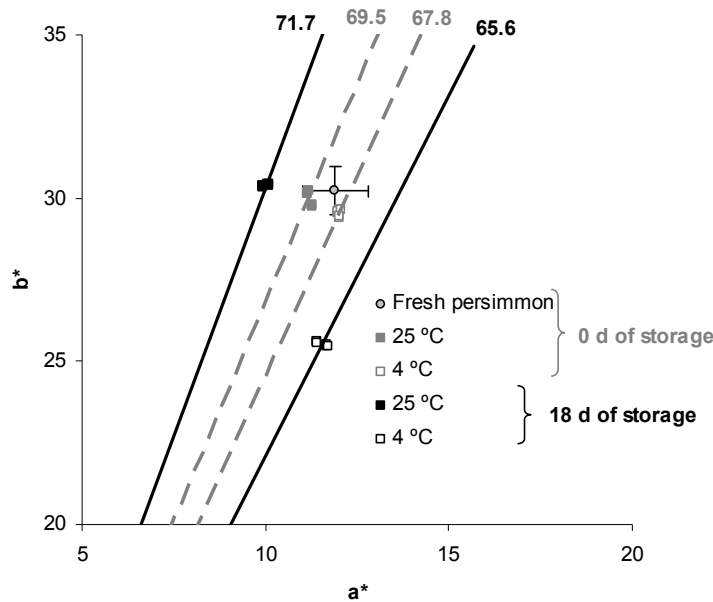
Figure 7. Position of colour parameters (a* and b*) of persimmon jam at 25 and 4 ºC in the chromatic diagram, at 0 and 18 days of storage. Numbers along the straight line indicate the hue values.

Figure 8 shows the evolution of luminosity (L*) in refrigerated and non-refrigerated persimmon jam, during storage time. Jam kept at 25ºC showed an increase in luminosity during storage time and the opposite behaviour was observed in jam at 4ºC. The greater increase in L* in jam at 25ºC could be associated with fermentation reactions, since there was a coincidence with the

decrease in the level of ºBrix in this case. Therefore, the development of fermentation metabolites could be responsible of this increase.
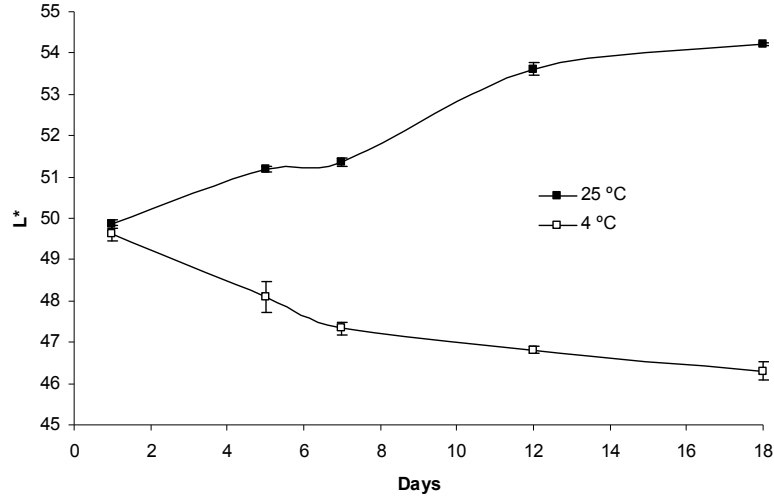
Figure 8. Evolution of parameter L* for persimmon jam at 25 ºC and 4 ºC with storage time.

CONCLUSION

According to the kinetic study 3 hours of osmotic pre-treatment is recommended in order to reach 30ºBrix. The production of persimmon jam employed 61% fresh fruit, which means high fruit content in the final product. Storage temperatures above 4ºC must be avoided for this type of product in order to prevent undesirable changes, especially those associated with fermentation reactions and also because texture was maintained better at low temperatures. Finally, astringency did not appear in any case, which implies that this process provides a new way to prepare spreadable persimmon fruit avoiding the thermal treatments that usually promote the solubilisation of tannins and the astringent flavour of this product.

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