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Effect of temperature and air velocity on drying kinetics, antioxidant capacity, total phenolic content, colour, texture and microstructure of apple (var. *Granny Smith*) slices

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ABSTRACT

The aim of this work was to study the effect of temperature and air velocity on the drying kinetics and quality attributes of apple (var. *Granny Smith*) slices during drying. Experiments were conducted at 40, 60 and 80 °C, as well as at air velocities of 0.5, 1.0 and 1.5 m s^{-1}. Effective moisture diffusivity increased with temperature and air velocity, reaching a value of 15.30×10^{-9} m^2 s^{-1} at maximum temperature and air velocity under study. The rehydration ratio changed with varying both air velocity and temperature indicating tissue damage due to processing. The colour difference, ΔE, showed the best results at 80 °C. The DPPH-radical scavenging activity at 40 °C and 0.5 m s^{-1} showed the highest antioxidant activity, closest to that of the fresh sample. Although ΔE decreased with temperature, antioxidant activity barely varied and even increased at high air velocities, revealing an antioxidant capacity of the browning products. The total phenolics decreased with temperature, but at high air velocity retardation of thermal degradation was observed. Firmness was also determined and explained using glass transition concept and microstructure analysis.

1. Introduction

In Chile, current apple production occupies 18% (39.68 ha) of the total land area in agricultural use in the country; 90% of this production is carried out in Chile's VI and VII regions (Odepa, 2011). The Granny Smith variety is typified by having a long growing period in relatively warm areas. Apples have been exported from Chile in ever increasing quantities over the last 20–30 years. More recently, dried apples and other dried fruit exports have become important, including raisins and prunes. The major markets for these products include the USA, the European community and Japan (Chilealimentos, 2011).

Drying of foods has been widely used throughout the history of man, allowing flexibility in availability of these products regardless of season. Today, the dehydrated food industry occupies an important place within general food industries throughout the world. Dehydrating apples is nowadays a frequent practise, since dried apples are part of numerous prepared foods including snack preparations, integral breakfast foods, as well as others (Akpinar, Bicer, & Yildiz, 2003). The advantages of dehydration are well known, as the reduction of moisture in the product greatly retards microbial and chemical deterioration and brings about a substantial volume reduction (Doymaz & Pala, 2003). However, food products are sensitive to drying conditions (temperature, air velocity and relative humidity), which can cause quality deterioration of products through oxidation, colour change, shrinkage or loss of texture and nutritional–functional properties (Vega-Gálvez et al., 2009). Apple is a frequently consumed fruit and constitutes one of the main sources of polyphenols in the western diet (Boyer & Liu, 2004). Polyphenols present in apples are important because of their contribution to the sensory quality of the fresh and processed fruits. They are also recognised for their health promoting antioxidant properties (Van der Sluis, Dekker, Skrede, & Jongen, 2002). Antioxidant activity of apple polyphenols is among the highest in fruits and vegetables commonly consumed (Lee, Kim, Kim, Lee, & Lee, 2003).

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Regardless of these benefits, dehydration causes evident changes on the microstructure of fruit and vegetable tissues and such changes can render the product unacceptable to consumers. Thus, the drying process requires microscopical analysis to address structural and microstructural changes produced at the cellular level and their potential influence on firmness (Nieto, Castro, & Alzamora, 2001). These changes are related to the loss of water from the inner parts towards the surface and the surrounding air, possibly causing stiffness, spoilage and disruption of the cell walls, or even a collapse of the cell tissues (Maltini, Torreggiani, Venir, & Bertolo, 2003). Several studies have tried to characterise these physical and structural changes in terms of parameters such as changes in volume, surface, size and shape (Krokida & Maroulis, 2001). Structural properties of the product depend on the type of drying, operative variables and food microstructure formed during the process (Aguilera, Chiralt, & Fito, 2003). Fruits and vegetables are generally difficult to dehydrate in hot air, owing to their high water content, which implies long drying times, leading subsequently to serious structural and colour changes (Krokida & Maroulis, 2001), and the inevitable loss of nutrients (Aguilera et al., 2003).

In low and intermediate-moisture food systems, water activity is traditionally used as a predictive tool for microbial, chemical and physical changes (Maltini et al., 2003). However, the state of water and solids in foods, kinetic changes and the physicochemical stability of intermediate moisture food polymers in a non-equilibrium state may be better explained with the glass transition concept (Roos, 1995).

Therefore, the aim of this work was to determine the effect of air-drying temperature and velocity on drying kinetics, rehydration ratio, water holding capacity, colour development, antioxidant capacity, firmness, glass transition and microstructure of dried–rehydrated apple var. *Granny Smith*.

2. Materials and methods

2.1. Raw material

Apples (var. *Granny Smith*) were purchased from a local market in the city of La Serena, Chile. Freshness, colour, size, state of ripeness and absence of any mechanical damage were used as the selection criteria. The fruits were then stored at 5 °C until analysis. For samples preparation, the apples were washed, peeled and the seeds removed. Slabs, 5.0 ± 0.2 mm thick, were cut perpendicular to the main axis of the apple. The initial moisture content was determined according to AOAC official method 934.06 (AOAC, 1990), using a vacuum drying oven (Gallenkamp, OVL570, Leicester, UK) and an analytical balance (CHYO, Jex120, Kyoto, Japan) with an accuracy of ±0.0001 g.

2.2. Drying process

The hot-air drying process was carried out in a convective dryer designed and built at the Department of Food Engineering of Universidad de La Serena, Chile (Vega-Gálvez et al., 2009; Vega-Gálvez, Miranda, Bilbao-Sáinz, Uribe, & Lemus-Mondaca, 2008). The inlet relative humidity was 72.0 ± 4.0 %, measured by an ambient digital hygro-thermometer (Extech Instrument Inc., 445703 MA, USA) and the load density was 4.4 ± 0.4 kg m^{-2}. All drying experiments were carried out in triplicate, using a mass of 100.0 ± 1.0 g. Samples were placed as a thin-layer in a stainless steel basket. This mass was measured on an analytical balance (Ohaus, SP402 NJ, USA) with a precision of ±0.01 g at defined time intervals, connected by an interface system (Ohaus, RS232 NJ, USA) to a PC, which recorded and stored data. Experiments were performed until an equilibrium condition was achieved and a constant weight of the samples was registered. The dried samples were kept in sealed polypropylene bags until further analysis.

2.3. Experimental design

The conditions applied in the experimental setups used for the drying of apples are based on a factorial design n^m, where, n is the number of levels and m is the number of factors. The air-drying temperature and velocity were the two factors under study ($m = 2$), each with three levels ($n = 3$). Thus, nine treatments were required (3^2). Table 1 shows the decodified levels of the variables used in the experimental design to represent the experiments. Each treatment combination in the experiment is denoted by (−1) level (40 °C and 0.5 m s^{-1}), (0) level (60 °C or 1.0 m s^{-1}) and (+1) level (80 °C and 1.5 m s^{-1}).

2.4. Determination of the effective moisture diffusivity (D_{eff})

Fick's second law of diffusion (Eq. (1)) was used to interpret the drying process since moisture diffusion is one of the main mass transport mechanisms that describe this process (Doymaz, 2007). In this model, the dependent variable is the moisture ratio (MR) which relates the gradient of the sample moisture content in real time to both initial and equilibrium moisture content (Eq. (2)).

$$\frac{\partial MR}{\partial t} = D_{eff}\frac{\partial^2 MR}{\partial Z^2} \quad (1)$$

$$MR = \frac{X_{wt} - X_{eq}}{X_{wo} - X_{eq}} \quad (2)$$

where: X_{wt} is the moisture content at time t (g water/g d.m.), X_{wo} is the initial moisture content (g water/g d.m.), X_{eq} is the equilibrium moisture content (g water/g d.m.) obtained from Eq. (1). D_{eff} is the effective moisture diffusivity ($m^2\ s^{-1}$), t is the drying time (s) and z is the spatial dimension (m). The mathematical solution of Fick's second law, when internal mass transfer is the controlling mechanism and one-dimensional transport in an infinite slab is assumed, is given by Eq. (3). For sufficiently long drying times, the first term in the series expansion gives a good estimate of the solution (Crank, 1975). In this case, a linear relationship between the logarithm of MR and time is obtained, which can be used to determine effective moisture diffusivity (D_{eff}) according to Eq. (4) (Simal, Femenia, Garau, & Rosello, 2005).

$$MR = \frac{8}{\pi^2}\sum_{j=0}^{\infty}\frac{1}{(2j+1)^2}\exp\left[\frac{-(2j+1)^2 D_{eff}\pi^2 t}{4L^2}\right] \quad (3)$$

$$MR = \frac{8}{\pi^2}\exp\left[\frac{-D_{eff}\pi^2 t}{4L^2}\right] \quad (4)$$

where: L is the half-thickness of the slab (m) and j is number of terms. The use of Eq. (4) is based on a constant D_{eff} assumption

Table 1
Matrix of the experimental design (3^2).

Treatments	Air velocity (m s^{-1})	Air temperature (°C)	Combination
T1	−1	−1	40 °C–0.5 m s^{-1}
T2	0	−1	40 °C–1.0 m s^{-1}
T3	+1	−1	40 °C–1.5 m s^{-1}
T4	−1	0	60 °C–0.5 m s^{-1}
T5	0	0	60 °C–1.0 m s^{-1}
T6	+1	0	60 °C–1.5 m s^{-1}
T7	−1	+1	80 °C–0.5 m s^{-1}
T8	0	+1	80 °C–1.0 m s^{-1}
T9	+1	+1	80 °C–1.5 m s^{-1}

for each drying experiment and a linear behaviour between the mentioned variables. This hypothesis of isothermia is only a simple assumption since drying is a complex process involving simultaneous heat and mass transfer, thus, all the complexity of the drying relies on D_{eff} (Doymaz, 2007).

3. Quality parameters

3.1. Rehydration ratio and water holding capacity

Before analysis of the quality attributes was carried out, the dried apple slices (10.0 ± 0.1 g) were put in distilled water at 20 °C for 14 h, using a solid to liquid ratio of 1:50. The samples were then removed, drained for 30 s, and weighed. This process was done in triplicate for each treatment. The rehydration ratio (RR) was calculated according to Eq. (5) (Vega-Gálvez et al., 2009). The water holding capacity (WHC) was determined by centrifuging the rehydrated samples at 4000×*g* for 10 min at 5 °C in tubes fitted with a centrally placed plastic mesh which allowed water to drain freely from the sample during centrifugation. WHC was calculated from the amount of water removed according to Eq. (6):

$$\mathrm{RR} = \frac{W_{rs} \cdot X_{rs} - W_{ds} \cdot X_{ds}}{W_{ds} \cdot (1 - X_{ds})} \quad (5)$$

$$\mathrm{WHC} = \frac{W_{rs} \cdot X_{rs} - W_{dt}}{W_{rs} \cdot X_{rs}} \times 100 \quad (6)$$

where: W_{rs} is weight of rehydrated sample (g), X_{rs} is moisture content of rehydrated sample (wet matter), W_{ds} is weight of dried sample (g), X_{ds} is moisture content of dried sample (wet matter) and W_{dl} is weight of dripped liquid after centrifugation.

3.2. Surface colour measurement

The surface colour of the apple slices was measured using a colorimeter (Minolta, CM-1000, Tokyo, Japan) based on the CIELab colour space, after calibration with the white and black glass standards. Three equidistant spots were examined on the major axis of each apple sample. Since the spot diameter of the instrument was 10 mm, the total area of the slab, from which information was taken, was 10 cm^2. The experiments were performed in triplicate. Colour changes were measured by colorimetric evaluation, which measures three parameters: Lightness (L^*), green–red hue (a^*) and blue–yellow hue (b^*). CIE L^*, a^* and b^* colour coordinates (considering standard illuminant D_{65} and observer 10°) were calculated (Vega-Gálvez et al., 2008). The colorimeter yielded L^*, a^* and b^* values for each spot, which were converted to total value of colour difference (ΔE) according to Eq. (7).

$$\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{0.5} \quad (7)$$

3.3. Determination of total phenolic content

Total phenolic content (TPC) was estimated as gallic acid equivalent (GAE) using the Folin–Ciocalteu (FC) method as modified by Vega-Gálvez et al. (2009). A 0.5 ml aliquot of the apple extract was transferred to a glass tube; 0.5 ml of FC reagent was added, vortex-mixed and left to stand for 5 min. Next, 2 ml of Na_2CO_3 (200 g l^{-1}) was introduced and after incubation of 15 min at ambient temperature, 10 ml of ultra-pure water was added and the formed precipitate was removed by centrifugation during 5 min at 4000×*g*. Finally, absorbance was measured at 725 nm with a spectrophotometer (Spectronic® 20 Genesys®, Illinois, USA) and compared to a gallic acid (GA) calibration curve. Results were expressed as mg GA/100 g dry matter. All reagents were purchased from Merck (Merck KGaA, Darmstadt, Germany), and all measurements were done in triplicate.

3.4. Determination of DPPH radical-scavenging activity

Free radical scavenging activity of the samples was determined using the 2,2,-diphenyl-2-picryl-hydrazyl (DPPH) method according to the work of Vega-Gálvez et al. (2009). Different dilutions of the extracts were prepared in triplicate. An aliquot of 2 ml of 0.15 mM DPPH radical in ethanol was added to a test tube with 1 ml of the sample extract. The reaction mixture was vortex-mixed for 30 s and left to stand at room temperature in the dark for 20 min. The absorbance was measured at 517 nm using a spectrophotometer (Spectronic® 20 Genesys®, Illinois, USA). Eighty percentage (v/v) ethanol was used to calibrate the spectrophotometer. The control sample was prepared without adding any extract. All solvents and reagents were purchased from Sigma (Sigma Chemical CO., St. Louis, MO, USA). Total antioxidant activity (TAA) can be expressed as the percentage inhibition of the DPPH radical and was determined by Eq. (8):

$$(\%)\mathrm{TAA} = \left[1 - \frac{\mathrm{Abs}_{sample}}{\mathrm{Abs}_{control}}\right] \times 100 \quad (8)$$

where: TAA is the total antioxidant activity and Abs is the absorbance. IC_{50}, which is defined as the concentration of substrate that brings about 50% loss of the DPPH, is typically employed to express the antioxidant activity and to compare the antioxidant capacity of various samples, since it is independent of the sample concentration. IC_{50} was determined from a graph of antioxidant capacity (%) versus extract concentration (μg ml^{-1} sample). By this means, it was possible to express antioxidant activity (μg mg^{-1}) in terms of a sample weight that has a standardised antioxidant activity.

3.5. Determination of firmness

Firmness of samples, as an indicator of texture, was defined as the maximum force applied to puncture the apple tissue. This physical property was measured using a Texture Analyzer (Texture Technologies Corp., TA XT2, Scardale, NY, USA). The probe had a puncture diameter of 2 mm, and was adjusted for a travel distance of 20 mm at a test speed of 1.7 mm s^{-1}. The maximum force was measured by making one puncture in each rehydrated apple sample, using 10 slices per treatment. The mean value of firmness for each treatment was then calculated and expressed in N mm^{-1}.

3.6. Glass transition measurements

The glass transition temperature (*Tg*) of dehydrated apple slices was determined by using a differential scanning calorimetry (DSC) (Model DSC823e, Mettler-Toledo, Schwerzenbach, Switzerland) equipped with DSC sensor HSS7. The instrument was calibrated by using an indium standard. A 10–15 mg sample was placed into a Mettler-Toledo DSC pan (ME-00026763), and hermetically sealed. An empty pan (air) was used as reference. The sample was first cooled to −50 °C at 10 K min^{-1}, then scanned from −50 to 80 °C at a rate of 10 K min^{-1} to determine its thermal behaviour. Before scanning the samples, a scan of two empty pans under the same test conditions was conducted to obtain baseline subtraction. *Tg* was recorded as the ASTM, IEC midpoint, that lies on the inflection tangent at half step height between Onset and Endpoint. STAR^e software version 9.1 (Mettler-Toledo) was used to determine midpoint temperatures for DSC glass transitions. Measurements of *Tg* were performed in triplicate and for each run water content of each sample was determined in duplicate, so that

through the Gordon–Taylor equation (Rahman, 1995) the glass transition temperature of dry fruit solids Tg_s could be calculated.

3.7. Cryo-SEM system

The microstructure of the cross-section of rehydrated apple slices (10 h at room temperature) was examined using Cryo-SEM (JSM-5410, JEOL, Kyoto, Japan). Samples were cryo-fixed in order to fix and stabilize the structure and composition of the biological system. Samples were placed in the SEM sample holder and plunged into subcooled nitrogen slush close to the freezing point of nitrogen (−210 °C). Slush nitrogen was used for its efficient cooling properties (Jeffree and Read, 1991). The frozen samples were freeze-fractured, etched at −90 °C, 10^{-5} Torr for 15 min, gold coated, and viewed on the SEM cold-stage. The fractured surface was viewed directly while it was maintained at −150 °C or lower. The micrographs were taken at 500× magnification to observe cell structure and its changes.

3.8. Statistical analysis

Bifactorial analysis of variance (ANOVA) was carried out to estimate least significant differences (LSD) among the media of the effective moisture diffusivities, at a confidence level of 95% ($p < 0.05$). Moreover, the multiple range test (MRT) was used to determine possible homogeneous groups existing among the diffusivities. The statistical estimation was done using the Statgraphics® Plus 5.1 software.

4. Results and discussion

4.1. Drying kinetics

Prior to approaching the study of drying of any food, it is necessary to evaluate its moisture sorption isotherms as these mathematically describe the relationship between water activity and equilibrium moisture content of the food product. They have a fundamental influence on many aspects of the dehydration process and the storage stability of the dried product. For food systems, these isotherms also give useful information about the sorption mechanism and the interaction of food biopolymers with water (Lemus, Lara, Betoret, & Vega, 2008). The experimental equilibrium moisture contents at 40, 60 and 80 °C for the complete range of a_w (0.10–0.96) obtained by Vega-Gálvez et al. (2008) were used in this study. Fig. 1 shows the drying curves for the three temperatures at air velocities of 0.5, 1.0 and 1.5 m s^{-1}. A clear effect of temperature on the drying behaviour of the apple slabs can be observed. An increase in drying temperature is accompanied by a decrease in drying time. The time needed to achieve equilibrium moisture content in all experiments was between 200 and 1000 min. In addition, Fig. 1 shows an extremely prolonged period of falling drying rate. Similar results were obtained by other authors working with other foods, as reported by Akgun and Doymaz (2005), Velić, Planinic, Tomas, and Bilic (2004) and Lemus et al. (2008). The drying time was longest at 40 °C and at an air velocity of 0.5 m s^{-1} (T1), while the shortest drying time occurred at 80 °C and at an air velocity of 1.5 m s^{-1} (T9).

4.2. Determination of effective moisture diffusivity

The effect of processing temperature on effective moisture diffusivities of the apple slabs can be seen in Fig. 2. At any given air velocity an increase in operative temperature led to an increase in effective moisture diffusivity. A bifactorial ANOVA on the media of D_{eff} of the apple slabs showed significant differences ($p < 0.05$)

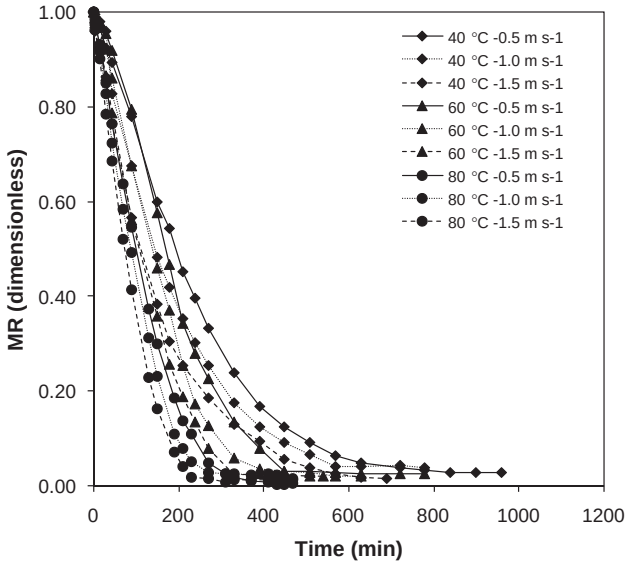

Fig. 1. Drying curves at air temperatures of 40, 60 and 80 °C for three different air velocities.

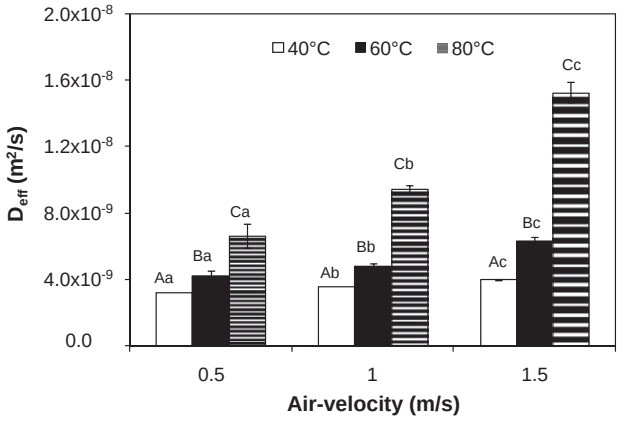

Fig. 2. Effective moisture diffusivity values for apple dried at different treatments.* Data are expressed as average ± standard deviation in three replicates. Values for the same air velocity having the same letter (A, B and C) for each parameter are not significantly different at a confidence level of 95%. Values for the same temperature having the same letter (a, b and c) for each parameter are not significantly different at a confidence level of 95%.

when observing the influence of temperature at a fixed air velocity. Similarly, observing the influence of air velocity at a constant drying temperature, bifactorial ANOVA on the media of D_{eff} of the apple slabs showed significant differences ($p < 0.05$). The highest value of D_{eff} ($15.30 \pm 0.60 \times 10^{-9}$ m^2 s^{-1}) was obtained in treatment T9 at the maximum experimental air temperature and velocity (80 °C and 1.5 m s^{-1} respectively), while the lowest value ($3.22 \pm 0.01 \times 10^{-9}$ m^2 s^{-1}) occurred at 40 °C and 0.5 m s^{-1} (T1). Several investigations, carried out on fruits and vegetables under similar temperature and velocity conditions showed D_{eff} values to lie between 5.59–6.51 $\times 10^{-9}$ m^2 s^{-1} for apricots (Togrul & Pehlivan, 2003), 3.00–17.21 $\times 10^{-10}$ m^2 s^{-1} for kiwi fruit (Simal et al., 2005), 1.7–4.4 $\times 10^{-10}$ m^2 s^{-1} for apples cv. *Jonagold* (Velić et al., 2004); 3.91–6.65 $\times 10^{-10}$ m^2 s^{-1} for tomato (Doymaz, 2007), 0.48–2.02 $\times 10^{-10}$ m^2 s^{-1} for apple cv. *Red Delicious* (Kaya, Aydin, & Demirtas, 2007) and 6.25–24.32 $\times 10^{-10}$ m^2 s^{-1} for Chilean papaya (Lemus et al., 2008).

A multiple lineal regression test carried out on effective moisture diffusivity for the apple slabs resulted in Eq. (9), which shows the interaction of moisture diffusivity with respect to air-drying temperature and velocity. According to this equation with a high coefficient of determination, $r^2 > 0.99$, the air-drying velocity (V) seems to be a more relevant factor compared to air-drying temperature (T). This showed that forced convection accelerated the drying process.

$$D_{\text{eff}} = -7.75 \times 10^{-9} + 1.71 \times 10^{-10} \cdot T(^{\circ}C) + 3.84 \times 10^{-9} \cdot V(ms^{-1}) \quad (9)$$

4.3. Rehydration index

Table 2 shows the results of rehydration ratio (RR) and water holding capacity (WHC) for each air-drying temperature and velocity studied. Experimental measures of RR in dried apples displayed a high variability, and ranged between 4.12 g absorbed water/g d.m. at T4 and 8.48 g absorbed water/g d.m. at T5. At constant air-drying velocity, RR increased as temperature increased, showing this pattern at 0.5 and 1.5 m s^{-1}. The maximum values of RR were observed at T7, T5 and T9, perhaps as a result of tissue collapse and cell damage produced by higher air temperatures ($p < 0.05$). In addition, the absorbed water decreased due to increasing air-drying velocity at constant air temperature ($p < 0.05$). We can infer that irreversible structural damages have taken place in the apple tissue during drying, resulting in a loss of rehydration ability (Kaymak-Ertekin, 2002).

It can be seen in Table 2 how WHC changed as air temperature increased at a constant air velocity ($p < 0.05$). At a constant air velocity of 1.5 m s^{-1} the difference in WHC obtained for the three temperatures is less pronounced. The maximum WHC was 56.30 ± 0.01 g retained water/100 g water at treatment T4, which implies that this drying temperature caused the greatest tissue structure damage; thus, apples dehydrated under this drying condition retain a great amount of water. On the other hand, samples dried at T1, T6 and T8 have reduced WHC, hence complete rehydration of the dried product is not achieved. A $p < 0.05$ for a confidence level of 95% was obtained from ANOVA on RR and WHC, suggesting that there is a significant difference among all the treatments.

4.4. Colour changes

Colour difference (ΔE) for all drying treatments with respect to the initial product is reported in Table 2. Chromatic coordinates for fresh apples were 82.9 ± 0.99, 0.64 ± 0.25 and 21.4 ± 0.58 for L, a and b, respectively. At constant air-drying velocity, ΔE presented a decreased with air-drying temperatures. Long drying times due to low process temperature could promote apple discoloration associated to formation of browning products. When analysing the trend of ΔE at constant temperature, an increase was observed at high air-drying velocity (1.5 m s^{-1}), however this increment was not significant ($p < 0.05$). It is also noticeable that treatments T7, T8 and T9 showed the lowest values of ΔE. The experimental result in Table 2 shows the effect of temperature and air velocity on colour change due to non-enzymatic browning. Changes in ΔE are brought about by simultaneous heat and mass transfer occurring at the surface of the apple samples and depended on drying time and temperature. Prolonged drying at higher temperature favoured browning reactions, that caused a decrease in lightness value (L^*). This is frequently reported to occur during thermal treatment, as in the case of sterilisation (Leadley, Tucker, & Fryer, 2008). However, at an air velocity of 1.5 m s^{-1} interfacial accumulation of heat and moisture is at its lowest, owing to high convective forces. Formation of browning products was less and depended only on the effect of a higher heat flow as the drying temperature increased. At an air velocity of 0.5 m s^{-1} stalling of heat and moisture at the air–solid interface favoured browning reactions. Only slight differences in ΔE were observed among the values obtained at the three different temperatures. At 40 °C a prolonged processing time was observed, in contrast to a higher heat flow at 80 °C. In both cases browning was favoured; at 60 °C a decrease in both drying time and heat energy resulted in a weaker colour change. Although at 60 °C the heat energy increased with respect to drying at 40 °C, the processing time diminished to such an extent that less browning took place. At an air velocity of 1.0 m s^{-1} the acting convective forces did not seem to be able to reduce the effect of heat energy

Table 2
Effect of drying temperature and air velocity on quality index of dried-rehydrated apple.

Quality index	Air temperature (°C)	Air velocity (m s^{-1})		
		0.5	1.0	1.5
RR (g absorbed water g^{-1} d.m.)	40	4.62 ± 0.03^{Aa}	7.58 ± 0.17^{Ab}	4.28 ± 0.19^{Ac}
	60	4.12 ± 0.03^{Ba}	8.48 ± 0.16^{Bb}	4.81 ± 0.09^{Bc}
	80	5.31 ± 0.03^{Ca}	7.80 ± 0.01^{Cb}	4.87 ± 0.05^{Bc}
WHC (g retained water 100 g^{-1} water)	40	45.15 ± 0.43^{Aa}	51.67 ± 0.73^{Ab}	48.28 ± 0.21^{Ac}
	60	56.30 ± 0.01^{Ba}	53.74 ± 0.25^{Bb}	45.46 ± 0.47^{Bc}
	80	50.95 ± 0.10^{Ca}	44.12 ± 0.64^{Cb}	47.79 ± 0.01^{Cc}
ΔE (−)	40	35.70 ± 0.04^{Aa}	29.10 ± 0.02^{Ab}	37.17 ± 0.07^{Ac}
	60	28.15 ± 0.31^{Ba}	28.88 ± 0.25^{Bb}	33.88 ± 0.39^{Bc}
	80	18.75 ± 0.06^{Ca}	19.96 ± 0.41^{Cb}	27.04 ± 0.14^{Cc}
DPPH radical-scavenging activity (μg mg^{-1})	40	1787.99 ± 16.20^{Aa}	1994.57 ± 1.94^{Ab}	3201.90 ± 49.42^{Ac}
	60	3113.47 ± 14.09^{Ba}	3094.39 ± 13.32^{Aa}	1996.07 ± 37.75^{Bb}
	80	2142.96 ± 4.79^{Ca}	2495.34 ± 3.53^{Bb}	2308.81 ± 50.46^{Cc}
Total phenolic content (mg GA 100 g^{-1})	40	31.94 ± 0.35^{Aa}	40.15 ± 0.95^{Ab}	38.71 ± 1.39^{Ac}
	60	27.39 ± 0.05^{Ba}	39.31 ± 0.17^{Ab}	44.82 ± 0.69^{Bc}
	80	27.34 ± 0.72^{Ba}	33.62 ± 0.38^{Bb}	27.04 ± 0.14^{Cc}
Firmness (N mm^{-1})	40	1.03 ± 0.03^{Aa}	0.97 ± 0.01^{Ab}	1.23 ± 0.01^{Ac}
	60	1.09 ± 0.02^{Ba}	1.09 ± 0.02^{Ba}	1.09 ± 0.02^{Ba}
	80	1.28 ± 0.09^{Ca}	1.06 ± 0.03^{Cb}	1.22 ± 0.03^{Ac}

* Data are expressed as the average ± standard deviation for three replicates. Values in the same column having the same letter (A, B and C) for each parameter are not significantly different at a confidence level of 95%. Values in the same row having the same letter (a, b and c) for each parameter are not significantly different at a confidence level of 95%.

and moisture accumulation at the surface of the samples. The effect of drying time was enough to cause a substantial colour change at 40 and 60 °C; at 80 °C this effect was even enhanced by a higher heat flow.

4.5. Determination of DPPH radical-scavenging activity and total phenolic content

The radical scavenging activity and the TPC were investigated based on air-drying temperature ($p < 0.05$) as observed in Table 2. Initial DPPH radical-scavenging activity determined for fresh sample was 1063.52 ± 15.46 μg mg^{-1}. During drying process, this value reached 3201.90 ± 49.42 μg mg^{-1} for the samples dried at air velocity of 1.5 m s^{-1} and air temperature of 40 °C (T3). This showed that under the conditions of treatment T3, where the ΔE of apple samples was the least, the greatest loss of antioxidant capacity occurred. In treatment T1 where strong browning took place, the strongest antioxidant capacity was also observed. A comparison of the values of ΔE with those of DPPH radical-scavenging activity showed almost an opposite effect of the drying conditions on these parameters. As reported by some authors, long drying times associated to low process temperature may promote a decrease in antioxidant activity (Garau, Simal, Rosselló, & Femenia, 2007); but this was not evidenced in this study. The observed profile of DPPH seemed to be related to generation and accumulation of different antioxidant compounds having a varying degree of antioxidant activity developing antagonistic or synergistic effects with themselves or with other constituents of the apple extract (Zielisnki & Koslowska, 2000). Some authors also reported that processing caused no change to antioxidant potential of fruit and vegetables or enhanced it due to the improvement of antioxidant properties of naturally occurring compounds or formation of novel compounds such as Maillard reaction products having antioxidant activity (Manzocco, Calligaris, Mastrocola, Nicoli, & Lerici, 2001). Maillard-derived melanoidins, responsible for colour change during the drying process, may therefore be associated to enhanced quality of the dried apples.

The total phenolic content, as determined for the dehydrated apple samples of the nine treatments, can be seen in Table 2. The initial TPC of the fresh sample was 158.28 ± 0.65 mg GAE/100 g sample. It was observed that an increase in drying temperature caused a degradation of total phenolics with respect to corresponding content in fresh sample ($p < 0.05$). Prolonged drying time did not necessarily produce the strongest degradation (T1); a temperature rise is needed to cause a degradation of total phenolics. The experimental results showed that TPC decreased with increasing temperature at air drying velocities of 0.5 and 1.0 m s^{-1}, as observed in treatments T1–T4–T7 and T2–T5–T8. However, this tendency changed completely as the air-drying velocity was raised to 1.5 m s^{-1}. At 80 °C, the highest drying temperature, degradation of total phenolics is the least. This is probably due to high convective forces acting at the air–solid interface retarding heat diffusion into the solid apples. The glycosides of phenolics, being localised in hydrophilic regions of cell such as vacuoles and apoplasts, or as other soluble phenols in the cytoplasm and in the cell nuclei (Sakihama, Cohen, Grace, & Yamasaki, 2002), seemed to get a protective heat shield by material of the cell walls. Internal resistance to heat diffusion is therefore an important parameter to be considered when quality is at stake during heat treatment in the drying process of apples. Moreover, the TPC may be related to the amount of antioxidant capacity (DDPH-radical scavenging activity) since both act as scavengers of the free radicals produced during oxidation reactions (Di Scala et al., 2011). Increasing correlation between antioxidant activity and total phenolic content has been reported during food dehydration. However, data on the effects of drying on TPC and antioxidant activity of fruits are rather conflicting due to several factors, such as the drying method, the type of extraction solvent, the antioxidant assays and the interactions of several antioxidant reactions (Manzocco et al., 2001).

4.6. Firmness of dried-rehydrated apple slices

Firmness is a very important textural property of fruits as it gives information on the storability and resistance to injury of the products during handling and processing. Moreover, this quality attribute is used to describe the mechanical properties of fruit tissues (Vega-Gálvez et al., 2009). The firmness of rehydrated apple samples was evaluated by a puncture test, and the results can be seen in Table 2. The final moisture content of the rehydrated apples used for the firmness analysis ranged from 12% to 20% of dry basis. The fresh apple presented a firmness of 3.21 ± 0.83 N mm^{-1}, which is high compared to the firmness values of all treatments. Dry air would cause a substantial decrease in firmness of the apple slabs. In treatments T1, T2, T4, T5, T6 and T8 firmness decreased at an average of 67% with respect to the fresh sample; however, in treatments T3, T7, and T9 it felt only at an average of 61%. These results indicate that in treatments T1, T2, T4, T5, T6 and T8, higher tissue softening occurred, which is in accordance with the reduction of the strength of cellular adhesion and the increase of the intercellular spaces observed by Cryo-SEM (Fig. 3). At higher temperatures the samples achieved relatively higher firmness (T7 and T9); although in treatment T6 an equivalent firmness was obtained. This may be due to some extent to the effect of air velocity of 1.5 m s^{-1}. ANOVA and LSD tests showed statistical differences between firmness values of the different treatments (fresh, T1, T2, T3, T4, T5, T6, T7, T8 and T9; $p < 0.05$). However, according to the result of the simple range tests carried out for the firmness values, four homogeneous groups were identified. The first group includes only the fresh product; the six treatments T1, T2, T4, T5, T6 and T8 formed the second group; T2, T5, T7, T8 and T9 built the third group of five treatments, while the fourth group of four treatments consisted of T3, T5, T7 and T9.

4.7. Glass transition measurements

In fruits, glass transitions are detected due to the presence of sugars, including glucose, fructose and sucrose and biopolymers. Multiple glass transition temperatures have been observed in many food systems. For example, in dried banana, two glass transition temperatures were detected at 46 ± 1 °C and at 0 ± 1 °C (Katekawa & Silva, 2007). For rice kernels at specific water contents, three apparent glass transition temperatures have been observed and are attributed to the structural characteristics of heterogeneous starch and the coexistence of the crystalline/semicrystalline and amorphous regions of starch (Perdon, Siebenmorgen, & Mauromoustakos, 2000). Apples are known to contain high molecular weight biopolymers, such that appearance of two glass transition temperature ranges, as found in this work at −39.4 ± 0.3 °C–−11.0 ± 0.3 °C and at 34.9 ± 0.7 °C –43.9 ± 2.5 °C is not surprising. In both cases the lowest *Tg* was observed for air-drying at 40 °C and air velocity of 1.5 m s^{-1} (T3) and the highest *Tg* for air-drying at 80 °C and air velocity of 0.5 m s^{-1} (T7). This occurrence is probably due to heterogeneity of a complex food system, which often leads to the detection of more than one glass transition (Matveev, Grinberg, & Tolstoguzov, 2000). In hot air drying applications, the glass transition at lower temperatures will not be significant. The first *Tg* is at most around or below 0 °C for very low moisture contents. Since hot air drying is conducted at temperatures higher than the first *Tg*, this transition will not take place and the solid matrix will always be in the rubbery state. This glass transition will only be of importance in drying at lower temperatures, such as freeze-drying applications. For the hot air-drying process considered in this article,

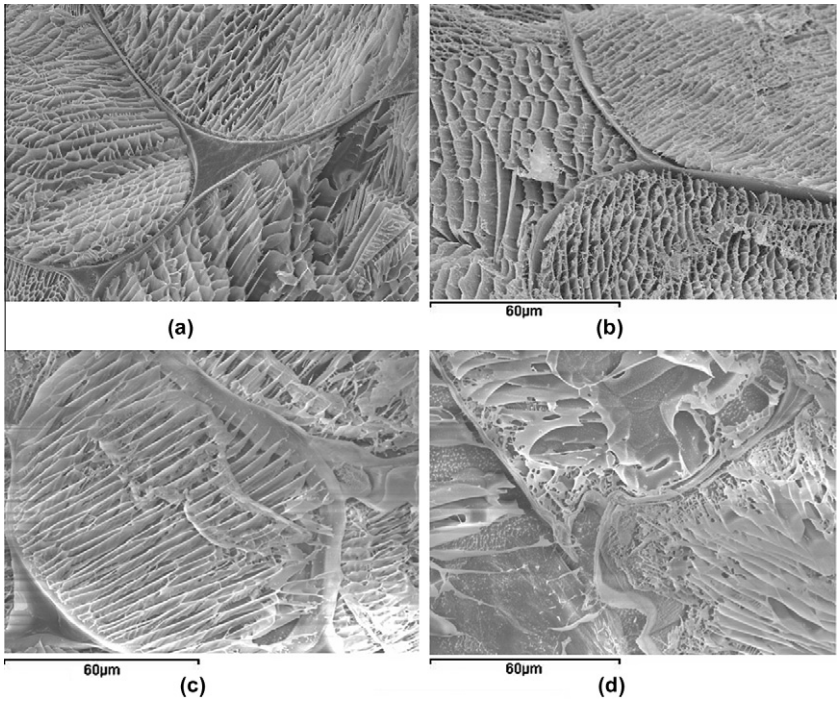

Fig. 3. Cryo-SEM images 500×: (a) fresh, (b) T1, (c) T6 and (d) T9.

only the higher value of glass transition temperature is significant. For air-dried apples at 30 °C and at 60 °C, reported transition temperature Tg_s for dry fruit solids were −8.7 °C and −1.9 °C, respectively (Welti-Chanes et al., 1999). For freeze dried apples and freeze dried apple juice, as reported by the same authors, Tg_s was still lower, at −13.3 °C and −16.3 °C respectively. The *Tg* values of osmoconcentrated air-dried apples as reported by Deng and Zhao (2008) range from 2.19 °C to 5.12 °C at moisture content of 5.72 ± 0.51 and 4.77 ± 0.40 g/100 g d.m., respectively. This is far less than those reported by Contreras, Martín, Martínez-Navarrete, and Chiralt (2005), in which air-dried apple cylinders, pre-treated in apple juice for 5 min vacuum and 10 min atmospheric restoration, had *Tg* values of 40.3 °C and 39.5 °C with moisture content of 4.4 and 7.9 g/100 g d.m. respectively. This difference was attributed to more uptakes of low molecular weight compounds such as fructose and glucose in the osmoconcentrated apples under the different pretreatment conditions, resulting in decreased *Tg* values (Deng & Zhao, 2008) (Table 3).

The relationship between *Tg* and textural attributes may depend on the specific texture parameters analysed. In the glassy state, characteristics exhibited by materials are those of brittleness, high strength, clarity and ultimately low molecular mobility (Rahman, 1995). According to Konopacka and Plocharski (2001), hardness and crispness are the most considered texture attributes of dried apples by consumers. Hardness or crispness of six dried apple samples as measured by Deng and Zhao (2008) showed no clear relationships with the corresponding *Tg* values. The same was reported by Contreras et al. (2005) for dried apples. Since *Tg* is a characteristic of the water soluble phase, other phases (non-soluble compounds of cell matrix) in dried product contribute mainly to its mechanical behaviours. In this study too, no direct relationship between firmness and *Tg* values was found. However, the *Tg* values obtained can served to explain effect of drying temperature and the similarity in firmness values for the different drying treatments. Applying the Gordon–Taylor equation on the *Tg* values obtained, dry fruit solids transition temperature, Tg_s, was calculated and used to understand the drying process. An increase in the first Tg_s as drying temperature increased was observed; this was probably caused by the destruction of low molecular weight sugars at higher temperature. On the other hand, the second Tg_s observed were not significantly different from one another, indicating that the higher molecular weight biopolymers would not be destroyed within the drying temperature range investigated. Since these biopolymers contribute to rigidity and cross-linkages in the solid structure, it was evident that no significant difference in firmness of the samples would be observed.

Table 3
Glass transition temperature of dehydrated apple samples obtained during drying at three air temperatures and three air velocities.

Glass transition temperature	Air temperature (°C)	Air velocity (m s^{-1})		
		0.5	1.0	1.5
1st *Tg*	40	-18.72 ± 4.77^{Aa}	-16.00 ± 1.03^{Aa}	-10.99 ± 0.29^{Ab}
	60	-17.51 ± 2.10^{Aa}	-17.24 ± 1.25^{Aa}	-30.38 ± 4.39^{Bb}
	80	-22.43 ± 1.23^{Aa}	-24.53 ± 1.25^{Ba}	-36.39 ± 0.33^{Cb}
2nd *Tg*	40	42.35 ± 2.50^{Aa}	40.89 ± 1.54^{Ab}	43.87 ± 2.47^{Ac}
	60	40.46 ± 1.16^{Aa}	42.16 ± 1.31^{Bb}	37.31 ± 3.30^{Bc}
	80	40.54 ± 4.87^{Aa}	39.20 ± 1.57^{Cb}	34.87 ± 0.68^{Cc}

* Data are expressed as the average ± standard deviation for three replicates. Values in the same column having the same letter (A, B and C) for each parameter are not significantly different at a confidence level of 95%. Values in the same row having the same letter (a, b and c) for each parameter are not significantly different at a confidence level of 95%.

4.8. Microstructure analysis

In Cryo-SEM micrographs, bright regions correspond to areas where cell walls and cellular membranes are present. Darker regions indicate presence of ice microcrystals (Fig. 3). Differences in intensity come from the differences in height between the areas where ice microcrystals occur and the areas where insoluble tissue structures are present. The porous structure is usually formed as a result of sublimation of the ice microcrystals (Gerschenson, Rojas, & Marangoni, 2001). Structural changes of fresh and dried-rehydrated apple were observed by Cryo-SEM and shown in Fig. 3. When comparing these figures with the tissue of raw apple (Fig. 3a), a clear cell breakage is observed, indicating loss of turgor and loss of cell content from the breakage zone. This probable turgor loss presents degradation and causes a possible shrinkage in the contours of the cell wall. In general, air drying of vegetable tissue is characterised by an extensive shrinkage and microstructural changes (Aguilera et al., 2003). The cellular shrinkage during drying operations has been observed during osmotic dehydration of apples (Lewicki & Porzecka-Palak, 2005). A cell damage is least at 40 °C (Fig. 3b) and occurred mainly because of prolonged drying time, while at higher temperature (80 °C) thermal destruction predominates and weakened the cell tissue (T6 and T9 in Fig. 3c and d). At higher air velocity this would enable the stronger convective forces acting over the product to cause a more separated and ruptured structure (T9).

5. Conclusions

Air drying is a process where heat and mass transfer occur simultaneously. The experimental results of this study showed that dehydration were faster when air temperature and air velocity increased, which is reflected in the values obtained for effective moisture diffusivity. Other quality attributes like colour difference (ΔE) and DPPH-radical scavenging activity decreased at higher temperature. RR and WHC showed differences among treatments indicating tissue damage due to processing. At 80 °C antioxidant activity values did not differ much from that measured at 40 °C. This can be explained by the development of Maillard browning reactions occurring concomitantly with other events, contributing to generation and accumulation of different antioxidant compounds having a varying degree of antioxidant activity. Total phenolic content (TPC) on the other hand showed unexpectedly least destruction at 80 °C and 1.5 m s^{-1} (T9). This was probably due to short drying time and therefore less exposure of the phenolics to thermal effect. Determination of glass transition temperature of dry apple solids, Tg_s, also revealed that during the hot air drying process, destruction of low molecular weight sugars occurred in the dried apples. The long chain biopolymers were more resistant to heat treatment and being decisive for firmness of the dried apple samples, this property had its highest value at 80 °C. Microstructure analysis also showed that cell disruption occurred at high temperature, and increased at high air velocity.

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