Osmotic dehydration of plums: Analysis of operation conditions and determination of effective diffusion coefficients

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Abstract: The objective of this work was to study osmotic dehydration kinetics of plums evaluating the influence of solution (type and concentration of solute, temperature, fruit/solution ratio) and process time on water loss, water content and solutes gain. Process analysis was performed experimentally by means of a set of 16 duplicate tests and numerically by mathematical modeling of the unsteady state mass transfer during osmotic dehydration. Aqueous solutions of glucose and sorbitol (40 and 60% w/w) were used for dehydrating plum pieces during 2 h at temperatures of 25 and 40ºC, and fruit/solution ratios of 1/4 and 1/10. For calculating effective diffusion coefficients the real shape of food pieces was considered using Finite Elements, working with the software COMSOL Multiphysics. Calculated diffusion coefficients are of the same order of those found in literature for regular shapes.

Keywords: Osmotic Dehydration, Numerical Solution, Diffusion Coefficients, Plums

INTRODUCTION

In the last decades, the rising demand of nutritious and natural foods such as fruits and vegetables has been remarkable, either as fresh or processed products or as ingredients to other final products (Forsido et al., 2013; Romojaro et al*.*, 2013). Owing to their high moisture content and water activity, they become highly perishable, and thus fresh fruits and vegetables have a short shelf life (Badwaik et al., 2014). In industrial terms, they require conservation techniques that minimize the changes in the characteristics of the original food.

Plum (Prunus domestica) is the most numerous and diverse group of fruit tree species (Živkovic et al., 2011). Preservation is necessary to give an added value to this agricultural product. Studies and developments of new preservation methods are continuously carried out in order to improve the quality of the final product.

During the last years numerous studies have been developed to osmotic dehydration of stone fruits, like plums (Tarhan 2007; Koocheki and Azarpazhooh, 2010; Rodríguez et al., 2010, 2013, 2014), cherries (De Michelis et al*.,* 2008; Konopackta et al., 2009), peaches (Riva et al., 2005, Ispir and Trogul, 2009; Germer et al., 2011), apricots (Khoyi and Hesari, 2007) and nectarines (Pavkov et al., 2011, Rodríguez et al., 2013) due to the nutritious properties of these fruits and to the increasing interest of obtaining extended high-quality shelf life. Osmotic dehydration (OD) pre-treatment with sugar solutions is a commonly used application in processing of fruits to improve the final product quality before final drying - by hot air, vacuum or microwaves - (Nieto et al., 2001).

Osmotic dehydration is a process of water removal by immersion of water-containing cellular solid in a concentrated aqueous solution (Ponting, 1973). The fundamental purpose of food dehydration is to lower the water content in order to minimize rates of chemical reactions and microbial growth to facilitate distribution and storage. In osmotic dehydration, foods are immersed or soaked in a sugar or saline or alcohol or combined solution. The driving force for dehydration is the difference in the osmotic pressure (in fact, chemical potentials of components) of solutions on both sides of the semi-permeable cell membranes. This results in three types of counter mass transfer phenomena (Ponting, 1973). First, water outflow from the food tissue to the osmotic solution, second, a solute transfer from the osmotic solution to the food tissue, third, a leaching out of the food tissue's own solutes (sugars, organic acids, minerals, vitamins) into the osmotic solution. The third transfer is quantitatively negligible compared with the first two types of transfer, but essential with regard to the composition of the product.

During OD, the rate of material fluxes between product and solution depends on the nature, shape, size of food product, type of osmotic agent (molecular weight and ionic strength) and its concentration, besides the process is influenced by the fruit/syrup ratio, solution temperature and agitation and process time (Shi *et al.*, 2009).

Mass transfer parameters, such as diffusivity and transfer coefficient, must be obtained for an efficient analysis of dehydration process (Rodriguez et al., 2013). For regular-shaped food pieces, the analytical solution of Fick's second law can be used – with good accuracy - for the determination of water (D_w) and solutes (D_s) effective diffusivities. This is the most frequent means to describe dehydration processes and as is known as "diffusive mechanism" (Farid, 2010). Most published research considers unidimensional diffusion in regular shapes, neglecting the contribution of other possible diffusion directions.

In this sense some recent research works have been reported. Abbasi Souraki et al. (2014) studied water and sucrose effective diffusivities of apple calculated using analytical solution for infinite slab shape samples during osmotic dehydration in sucrose solution. Sareban and Abbasi Souraki (2016) investigated osmotic dehydration of bulk of celery stalks in salt solution, in their research two different regular geometries (cylindrical and cubical) and anisotropic diffusion were considered to obtain the coefficients of the dehydration process using the analytical solution of Fick’s second law.

The analytical solution of Fick’s law is obtained with some restrictions in the formulation; those are not valid for irregular shape samples or finite systems due to the significant contribution of diffusion from peripheral regions. So, diffusion coefficients should be evaluated using the real shape of the food piece, usually making use of numerical techniques for the solution of the partial differential equations that describe the components diffusion (Ferrari et al., 2011).

In a previous research by these authors, the effective diffusion coefficients of water and solutes transfer of nectarines pieces, calculated by Fick’s law analytical solution and by computational tools - which considered the real shape of the fruits– were determined and their accuracy compared (Rodriguez et al., 2013).

In the present work, the objective is to study osmotic dehydration of plums as a function of process conditions and to determine the effective diffusion coefficients for water and solutes transfer through the use of computational tools that allow consider the real shape of food pieces.

MATERIALS AND METHODS

Preparation and characterization of samples

Plums of the variety D'ente (*Prunus doméstica* L.) harvested from the Chacra Experimental at the Facultad de Agronomía of UNCPBA located in the city of Azul, Buenos Aires (Argentina) were used. Initial moisture of the fresh fruit was 4.205±1.218 g water g/dry solid (84.43% a 74.92%, w.b.) (AOAC, 1980a) and the initial content of soluble solids was 18.75±1.48%, determined by Abbe refractometer (accuracy±0.01) (AOAC, 1980b). Water activity was determined through the equipment Aqualab (model 3TE, Pullman, WA), initial value was 0.966±0.002. The fruits were kept refrigerated at 5ºC before the tests. Samples, selected by size and quality, were washed and dried with absorbent paper, then the stones were removed and they were manually cut into pieces of one-eighth (average weight 2.4 g) (Figure 1a).

Osmotic Dehydration

OD was carried out for 2 h– initial period of high water removal (Barbosa Cánovas and Vega Mercado, 2000) – by immersing of samples in solutions of glucose ($C_6H_{12}O_6$) or sorbitol ($C_6H_{14}O_6$), prepared at two concentrations: 40 or 60% (w/w) in distilled water. Samples were kept immersed in the solutions using a stainless steel mesh to prevent flotation; two fruit:solution ratios 1/4 or 1/10 were employed. The experiments were carried out at two temperatures: 25 and 40ºC (Rodríguez et al., 2010). At regular intervals, the weight of samples was measured (analytical scale, METTLER AE240, precision ±0.0001 g), together with their water and soluble solids content. Samples were taken at 15, 30 45 60, 90 and 120 min of dehydration. All the experiences were performed in duplicate.

To determine the water loss (*WL_t*), solids gain (*SG_t*) and weight reduction (*WR_t*) as a function of time *t*, the following equations were used, respectively (Rodriguez et. al., 2013):

$$WL_t(\%) = \left[\left(\frac{1-TS_0}{100}\right) - \left(\frac{1-TS_t}{100}\right)\left(\frac{1-WR_t}{100}\right)\right]100 \tag{1}$$

$$SG_t(\%) = \left[\left(\frac{1-WR_t}{100}\right)\frac{TS_t}{100} - \frac{TS_0}{100}\right]100 \tag{2}$$

$$WR_t(\%) = \left(\frac{W_o - W_t}{W_o}\right)100 \tag{3}$$

where TS_o is the initial total solids of sample; TS_t is the total solids present in sample at time *t*; W_o is the initial mass of sample; W_t is the mass of sample at time *t*.

Determination of water and solids diffusion coefficients

To describe mass transfer during OD, the following microscopic mass balances are valid for both water and solids, respectively (García Noguera et al., 2010):

$$\frac{\partial C_w}{\partial t} = \nabla(D_w \nabla C_w) \tag{4}$$

$$\frac{\partial C_s}{\partial t} = \nabla(D_s \nabla C_s) \tag{5}$$

where C is concentration in the food (kg m^{-3}), t is time and D is apparent diffusion coefficient. Subscripts w and s refer to water and soluble solids, respectively.

a) Assumption of regular geometry:

These expressions may be analytically solved considering constant properties, uniform initial conditions and constant concentration of water and soluble solids at boundary (surface). In this way, they may be analytically solved for regular semi-infinite media, such as infinite slabs, infinite cylinders and spheres (Crank, 1975). In this work, the analytical solution of the equations was obtained considering each piece as a slab shape.

The following assumptions were done for the analytical solution: *i)* mass transfer is unidirectional; *ii)* solution concentration is constant in time; *iii)* diffusive mechanism of water removal is considered as valid; *iv)* fluxes interaction is not considered; *v)* shrinkage and external resistance to mass transfer are dismissed; *vi)* a slab equivalent to 6.25 mm of half-thickness is assumed.

Crank's mathematical solution for average concentration in semi-infinite slabs is presented below:

$$\frac{(C_{wt}-C_{w\infty})}{(C_{wo}-C_{w\infty})}=\sum_{n=0}^{\infty}\frac{8}{(2n+1)^2.\pi^2}.\exp\left(-(2n+1)^2\frac{\pi^2 D_w}{4l^2}t\right) \qquad (6)$$

where C_{wt} is the water concentration at time t; C_{wo} is the initial water concentration; l is the half-thickness of the sample, and $C_{w\infty}$ is the equilibrium concentration value. This equilibrium value can be obtained from Azuara's empirical model (Azuara et al., 1992). Azuara model establish a relation between kinetic variables such as water loss, solids gain and process time to obtain significant coefficients or parameters for the physical process interpretation (Arballo et al., 2012).

$$WL_t = \frac{S_1 t\,(WL_\infty)}{1 + S_1 t} \tag{7}$$

where WL_∞ is the corresponding value at equilibrium and S_1 is the constant related to the outward water diffusion rate in the food. Eq. (7) can be expressed in linear form as:

$$\frac{t}{WL_t} = \frac{1}{S_1\ WL_\infty} + \frac{t}{WL_\infty} \tag{8}$$

The water loss at equilibrium (WL_∞) and the constant S_1 were estimated from the slope and intercept of the plot (t/WL_t) versus t using the Eq. (8).

Eq. (5) was solved with the same procedure applied to Eq. (4), where the subscript w is replaced by s in Eq. (6). In the same way, the solid gain value at equilibrium can be obtained from Azuara's empirical model (Eq. 8).

Knowing the experimental average values of moisture and solids content in the product at different process times, the diffusion coefficients of water and solids in the product may be calculated, using the six first terms of the Fourier series (Eq. 6) (Singh et al., 2007). The values of the coefficients are calculated as roots of polynomial equation by numerical method of Quasi-Newton method (Telis et al., 2004).

The average relative error (*ARE*) (Eq. 9) was the statistical parameter used to estimate the quality of model adjustment.

$$ARE_j = \sum_i \left| \frac{C_j^{\exp} - C_j^{cal}}{C_j^{\exp}} \right| \tag{9}$$

where the subscript *j* indicates water or solids, the superscript *exp* refers to experimental, while *cal* to calculated and the counter *i* indicates that the sum is made for discrete time steps in which experimental data are available.

b) Assumption of real geometry:

Eqs. (4) and (5) were solved numerically with the finite elements method (FEM) using a commercial software (COMSOL Multiphysics), assuming as valid the following assumptions:

- solution concentration is constant in time;
- diffusive mechanism of water removal is considered as valid;
- fluxes interaction is not considered;
- shrinkage and external resistance to mass transfer are dismissed;
- real geometry of the product is considered (Fig. 1b).

This last assumption is considered valid due to volume variation is low at short process times (the sample loses water but gains soluble solids) and the ratio solution volumen to sample weight is high enough as to secure almost constant solution concentration (Pani et al., 2008; Riva et al., 2008).

The software Matlab 7.10.0 was used for the determination of the effective diffusion coefficients of water and solids, which algorithm considers different combinations of D_w and D_s in a known range. These interval values were selected according to previous data presented in the literature (Panagiotou et al., 2004, Arballo et al., 2012).

Then, the numerical solutions for these combinations could be obtained with the assistance of COMSOL software; this solution was compared with experimental data (C_{w} and C_s as a function of process time) through the average relative error (Eq. 10) for each pair D_w-D_s tested:

$$ARE = ARE_w + ARE_s \tag{10}$$

The pair which minimized the error function (Eq.10) was considered valid for the selected operating conditions. A detailed explanation of the calculation methodology is presented in a previous work (Rodriguez et al., 2013).

Statistical analysis

The influence of the process variables on quality parameters was evaluated through analysis of variance at a significance level of 5%. The analysis was carried out using the software InfoStat (Universidad Nacional de Córdoba, 2004).

RESULTS AND DISCUSSION

Water loss kinetics during osmotic dehydration

Water loss (WL) of samples dehydrated during 120 minutes in glucose and sorbitol solutions are shown in Figure 2a and 2b, respectively. The figures show the kinetics of *WL* for the sixteen different treatments, varying the operating conditions: concentration of glucose (g-40% and g-60%) and sorbitol (s-40% and s-60%), fruit/solution ratio (r1/4 and r1/10) and process temperature (25°C and 40°C). The values of standard deviation between the duplicates are included as vertical bars in the same figures.

The statistical results of the analysis of variance performed to evaluate the effect of the treatments on water loss are shown in Table 1. The independent variables, the degrees of freedom (*df*), the critical values of Fisher (*F*) and the *p*-values are displayed in the same table.

Related the rate of WL during OD of plums, process time, type of solute, its concentration and fruit: solution ratio influenced it significantly, but temperature had statistically no effect.

According the statistical analysis there is significant interaction between process time and type of osmotic agent (p=0.0067). The same is valid for process time and solute concentration (p<0.0001). An increase in WL values along process time is determined both for glucose and sorbitol solutions, which is enhanced at the higher solute concentration of 60% w/w (Fig. 2 a and b).

Interaction between variables type of osmotic agent and concentration influenced WL of plums (*p*=0.0001), having a higher degree of dehydration those treated in sorbitol solution at 60% w/w. These results are equivalent to those obtained by Araujo et al. (2004) and Ispir & Togrul (2009) in OD of apricots in solutions of glucose, sorbitol, fructose, sucrose and maltodextrin and by Ferrari et al. (2009) in OD of pears in sucrose and sorbitol solutions.

There were also significant effects between the ratio fruit:osmotic solution with concentration and process temperature (*p*=0.0020 and *p*<0.0363, respectively). The highest fruit: osmotic solution ratio allowed obtaining – in most tests – plums with lower water content. Khoyi and Hesari (2007) – during their study of OD of apricots – found equivalent results, but also determined that fruit / solution ratios higher than 1:10 increase process costs with low additional increase in WL. Same type of results were reported by Ispir & Togrul (2009) for OD of apricots.

Finally – as mentioned - the increase in solution temperature from 25 to 40ºC had no significant effect (*p*=0.5937) over *WL* (Table 1). These results are in accordance with those obtained by Fernandes et al. (2006) for OD of bananas.

Solids gain kinetics during osmotic dehydration

Solid gain (SG) of samples dehydrated during 120 minutes in glucose and sorbitol solutions are shown in Figure 3a and 3b, respectively. The figures include the kinetics of *SG* for all treatments, varying the operating conditions.

Related the rate of SG during OD of plums, process time, type of solute, its concentration and fruit: solution ratio influenced it significantly, but temperature had statistically no effect (Table 2). Besides, time interacted significantly with the type of osmotic agent (p=0.0106) and with its concentration (p<0.0001). An interaction between these last two variables can also be detected (p=0.0001). For all experimental conditions a continuous increase in SG with time was determined, reaching higher values when sorbitol was the osmotic agent and its concentration was the highest (60% w/w) (Figure 3b).

SG was also affected by the interactions between temperature and concentration (p=0.0024) and temperature with the ratio fruit/osmotic solution (p=0.0133) (Table 2). The increase in the ratio fruit: solution clearly favored SG, being this effect more noticeable when using sorbitol as dehydrating agent (Fig. 3b). Finally, *SG* was independent of temperature (p=0.8181). These results are in agreement with those obtained by Ponting et al. (1966); Hawkes and Flink (1978); Islam and Flink (1982); Fito et al. (1992) and Ozen et al. (2002), where these authors remark that a mild increase in process temperature has no effect on SG.

Determination of Process Efficiency

To discriminate between the results of different operating conditions of the OD process, the index of efficiency as defined by Lazarides (2001) (ratio between WL and SG) was calculated. Table 3 presents the results obtained for all the experimental conditions tested.

For all the conditions tested, water transfer was higher than solutes transfer, giving efficiency indexes much higher than 1. This means that the low solutes income to the food should have little influence on taste and flavor, producing partially dehydrated plums with sensory properties similar to fresh ones.

During OD the highest efficiency (6.57) was obtained in assay 2 using glucose solution at 40% w/w, with a ratio fruit:solution of 1/4 at 40 ºC, meanwhile the lowest value (2.55) was for assay 15 when using sorbitol solution at 60% w/w, ratio 1/10 and 25 ºC.

The combined negative effect of high concentration and low temperature over efficiency index may be attributed to the collapse of cell structure when working under such conditions, producing pore contraction and – consequently – the reduction of free volume for soluble solids impregnation (Barat et al., 2001). Similar behavior was observed working on nectarines in the same operating conditions (Rodriguez et al., 2013).

When comparing efficiency indexes in function of type of solute, in general, dehydration process was more efficient when using glucose. Sorbitol induced higher WL and SG, from whose ratio efficiency indexes were lower. These results are in accordance with those of Ferrari et al. (2009) during OD of pears in sucrose and sorbitol solutions and Rodriguez et al. (2013) working on nectarines.

Diffusion coefficients of water and solids

The effective diffusion coefficients were calculated using the numerical solution as applied to the real sample geometry as described in materials and methods section as well as using the analytical solution applied to an equivalent fruit slab shape.

Typical water and soluble solids predicted concentration profiles using the real geometry are presented in Figure 4. To obtain diffusion coefficients using the numerical method, water and solids concentration profiles within the samples were calculated using COMSOL Multiphysics software (version 3.5a). By volumetrically integration of these profiles, time variation of average water and solute concentrations can be obtained.

Table 4 presents the effective diffusion coefficients for water (D_w) and solids (D_s), calculated using the numerical method, as well as their relative errors (ARE).

The effective diffusion coefficients for water varied between 1.38x10^{-09} and 4.19x10^{-09} m^2 s^{-1}; the effective diffusion coefficients for solids ranged between 0.58x10^{-09} and 3.55x10^{-09} m^2 s^{-1}. The values of ARE were lower than 0.06 for both components, showing the high quality of the numerical fitting between experimental and predicted ones. It can be seen in Table 4 that predicted diffusion coefficients for water were higher than for solids, which implied higher WL than SG, as effectively it can be seen in all the experiences.

In Table 5 are presented the effective diffusion coefficients for water (*D_w)* and solids (D_s), calculated using the analytical solution together with their average relative errors (AR*E*).

From the reported results, the effective diffusion coefficients for water ranged between 3.98x10^{-09} and 1.1x10^{-08} m^2 s^{-1}. In the same sense, the effective diffusion coefficients for solids varied between 1.21x10^{-09} and 9.89x10^{-09} m^2 s^{-1}. ARE values were lower than 0.05.

The values given in Table 4 and 5 are in accordance with those published by different researchers. According to Ispir and Togrul (2009), D_w varied between 0.77x10^{-10} and 1.75x10^{-10} m^2 s^{-1} in OD of apricots, meanwhile Sabarez and Price (1999) obtained values in the range between 4.30x10^{-10} and 7.60x10^{-10} m^2 s^{-1} in OD of plums. On the other side, Khoyi and Hesari (2007) reported data ranged between 1.07x10^{-09} and 4.06x10^{-09} m^2 s^{-1} for D_w and between 7.69x10^{-10} and 3.13x10^{-09} m^2 s^{-1} for D_s in apricots, calculated using the analytical solution for plane plate. Besides, Azuara et al. (2009) obtained diffusion coefficients in apples after 1 h of OD, of the order of 1.53x10^{-10} and 1.05x10^{-10} m^2 s^{-1} for water and solids, respectively.

Finally, Rodríguez et al. (2013) during OD of nectarines reported values of D_w between 1.27x10^{-10} and 1.37x10^{-08} m^2 s^{-1} considering the fruit piece as a plane plate and between 0.70x10^{-09} and 4.80x10^{-09} m^2 s^{-1} when the true shape was considered. These authors reported values of D_s calculated using the analytical solution of between 1.14×10^{-10} and 1.08×10^{-08} m^2 s^{-1}, while those calculated using the true sample shape ranged between 0.26×10^{-09} and 1.70×10^{-09} m^2 s^{-1}.

The analysis of paired means through *T* test was employed to compare the diffusion coefficients of water and soluble solids obtained using analytical solution and numerical calculation. The values of experimental *T* obtained by comparing in pairs the 16 diffusion coefficients of water and solids calculated for slab and real geometry were 11.52 (p=7.48×10^{-9}) and 8.79 (p=2.61×10^{-7}), respectively. Therefore, there are significant differences ($p<0.05$) between values determined by analytical and numerical calculation.

From the obtained results it can be observed the coefficients calculated by the analytical method are higher relative to those obtained by the numerical method. It can be explained considering that the fluxes assigned to a single direction overestimate the rate of diffusion for *WL* and *SG* values*;* to consider the real and irregular geometry involves a different spatial distribution and a lower rate of diffusion (Rodriguez et al., 2013).

Besides, an analysis of variance was carried out to evaluate the influence of system variables on the diffusion coefficients of mass, by which it was determined that the operating variables (type of osmotic agent, concentration, fruit to syrup ratio and temperature) did not exert a significant influence ($p<0.05$) on D_w and D_s and values obtained from the two calculation techniques.

CONCLUSIONS

Osmotic dehydration (OD) pre-treatment of plums was studied with under different operating conditions through water loss, solid gain and efficiency parameters. Analysis of the experimental data revealed that water loss was significantly dependent on process time, type and concentration of solution and fruit: solution ratio, but temperature had statistically no effect. According the statistical analysis, there were also significant effects between the ratio fruit:osmotic solution with concentration and process temperature, process time with type of osmotic agent and process time with solute concentration. Besides, the interaction between the kind of osmotic agent and concentration influenced WL of plums, having a higher degree of dehydration those treated in sorbitol solution at 60% w/w.

Related the rate of SG during OD of plums, the results showed a significantly influence of process time, type of solute, its concentration and fruit: solution ratio, but temperature had statistically no effect, similarly to water loss parameter. Besides, time interacted significantly with the type of osmotic agent and its concentration. An interaction between these last two variables can also be detected. SG was also affected by the interactions between temperature, concentration and temperature with the ratio fruit/osmotic solution. For all experimental conditions a continuous increase in SG with time was determined, reaching higher values when sorbitol was the osmotic agent and its concentration was the highest (60% w/w).

During OD the highest efficiency (6.57) was obtained using glucose solution at 40% w/w, with a ratio fruit:solution of 1/4 at 40 ºC, meanwhile the lowest value (2.55) was for the process using sorbitol solution at 60% w/w, ratio 1/10 and 25 ºC. The numerical effective diffusion coefficients obtained through the numerical technique for water varied between 1.38×10^{-09} and 4.19×10^{-09} $m^2\ s^{-1}$; the effective diffusion coefficients for solids ranged between 0.58×10^{-09} and 3.55×10^{-09} $m^2\ s^{-1}$. The values of ARE were lower than 0.06 for both components, showing the high quality of the numerical fitting between experimental and predicted ones. A statically analysis show there are significant differences between values determined by analytical and numerical calculation.

Finally, OD allowed the efficient partial withdrawal of water maintaining sensory properties under mild dehydrating conditions. This methodology must be complemented by another preservation technique to reach true stability.

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