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Analysis of operating conditions on osmotic dehydration of plums (Prunus domestica L.) and numerical real-shape determination of effective diffusion coefficients

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Complete List of Authors:	Campañone, Laura; Consejo Nacional de Investigaciones Cientificas y Tecnicas, CIDCA (CONICET UNLP) Rodríguez, M.; Universidad Nacional del Centro de la Provincia de Buenos Aires, Departamento de Ingeniería Química, Facultad de Ingeniería Arballo, Javier; Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), Mascheroni, Rodolfo
Classifications:	
Keywords:	Plums, osmotic dehydration, diffusion coefficients
Abstract:	The objective of this work was to analyze the relevant process conditions on osmotic dehydration of plums and to determine the diffusion coefficients related to this process. The influence of solution (type and concentration of solute, temperature, fruit/solution ratio) and process time on water loss, water content and solutes gain were studied. 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 phenomena. Experiments were carried out with glucose and sorbitol solutions (40-60% w/w), dehydrating plum pieces during 2h at temperatures of 25 and 40ºC, with fruit/solution ratios of 1/4 and 1/10. For calculating effective diffusion coefficients, a novelty inverse-method was applied, the real shape of food-pieces was considered using Finite Elements Method. Calculated diffusion coefficients ranged from 1.13x10-09 to 4.71x10-09 m2 s-1 and 0.44x10-09 to 3.46x10-09 m2 s-1, for water and solutes, respectively.

Reviewer(s)' Comments to Author:

Reviewer: 1

Comments to the Author

The purpose of this paper was to analysis of operating conditions on osmotic dehydration of plums (Prunus domestica L.) and numerical real-shape determination of effective diffusion coefficients. A number of corrections/additions are suggested to improve the quality of this manuscript:

1-The current research has no novelty. The novelty of this study should be add clearly.

Answer: This manuscript is performed in plums, these fruits are interesting for our study due to their nutrient content: numerous phenolic phytochemicals, such as flavonoids and phenolic acids, which may function as effective natural antioxidants (Page 3, lines 15-21).

Besides, the numerical method for calculating diffusivity parameters is a new procedure; it considers the real geometry of the treated samples. It is included in the new version of the manuscript (red color).

"In the present work, this novelty technique was adapted to plums pieces considering the new product geometry and different operating conditions".

2- This manuscript has critical problem in writing. I advise the authors that ask native editor for proofreading.

Answer: English revision was done in the whole manuscript.

3-Introduction: This section is too long.

Answer: The following parts of the introduction section were removed from the previous version of the manuscript (page 4 lines 23-30 "However, preservation is necessary…" and lines 43-49 "This process implies the water…")

4-Introduction: It is better to compare antioxidant activity of plum with synthetic antioxidant such as BHA or BHT.

Answer: the objective of the present work does not include the antioxidant capacity determination. In this section, we only wanted to highlight the nutritional properties of plums.

5-Result and discussion: In this manuscript, the results just reported and the there was no justification. I advise the authors compare their result with the other work.

Answer: in Sections 3.1 and 3.2, the results of the osmotic dehydration process were shown and in Section 3.4 we discuss the values of diffusion coefficients according to several operating conditions (Page 11, previous version of the manuscript).

The authors thank Reviewer #1 for the comments and suggestions that help us to improve the manuscript.

Reviewer: 2

Comments to the Author

1.Figures 2 and 3. WL and SG data should be presented as regular kinetic plots, with data separation in time axis proportional to values differences (in current plots the same spacing is used for a time interval of 15 and 30 min).

Answer: These Figures were modified and they are included in the new version of the manuscript.

2. Figure 4. Text in figure is too small and cannot be easily read. Time units? It seems that time is in seconds, so plots are for 7200 s or 120 min.

Answer: The Font size was increased. In this new version of the Figure, it is easy to read 7200 seconds of time. Besides, this information was included in the list of Figures legends.

3. Figure 4. According to this figure water concentration at its highest value is about 650 mol/m3, that is, (650 mol/m3)(18 g/mol)(1 kg/1000 g) = 11.7 kg/m3. This value is too small!!! On the other hand, glucose concentration at its highest value is about 220 mol/m3, that is, (220 mol/m3)(180 g/mol)(1 kg/1000 g) = 39.6 kg/m3. This value is higher than water concentration!!!

Answer: We thank to the reviewer, the units of the water and solid concentration were corrected, and correspond to 650 kg/m^3 and 220 kg/m^3 for water and solid, respectively. Then, Figure 4 were modified.

4. Azuara's plot to evaluate final WL and SG must be given to allow the reader check the adequacy of this model to evaluate WL and SG at equilibrium for these particular data. Moreover, values for WL and SG at equilibrium must be included.

Answer: In the present version of the manuscript, the final value of water loss and sugar content were used as equilibrium values. This procedure was better than the Azuara´s technique, because the final values are close to the asymptotic ones working on 2 hours of process. Azuara´s equations were removed in the new version of the manuscript and from the mathematical calculation. Besides, this new version presents the predicted and experimental values and the goodness of fit through R2 factor (see Table 5).

The paragraph was modified, and the following text was included:

"The microscopic mass balances (Eqs. (4) and (5)) were solved taking into account initial uniform concentration in all domain and Dirichlet boundary condition at the surface (equilibrium concentration). These equilibrium values (water and sugar) were obtained from the experimental kinetic curves, considering the asymptote at 2 hours of process."

5. TS is referred as total solids of sample, but it seems it is a mass fraction. The units of all variables should be included.

Answer: The units of the variables were included in the new version of the manuscript (red color).

6. Initial conditions for model solution (Cw0 and Cs0) should be given.

Answer: Specific initial conditions are different for each experiment and they are considered in the simulations. The initial conditions of Figure 4 were Cw0 = 799.4 kg/m^3 and Cs0= 187.5 kg/m^3. The following paragraph was modified according to the consideration made by the reviewer:

"Typical water and soluble solids predicted concentration profiles using the real geometry considering specific initial condition - Glucose concentration 40%, fruit/solution ratio 1/4 and medium temperature of 25ºC - (Cw0= 799.4 kg/m3 and Cs0= 187.5 kg/m3) are presented in Figure 4."

7. Is there a particular reason to not use the sum of squares for error as fitness criterion?

Answer: There is not a particular reason, but it is important to note that the sum includes the absolute value of the differences between calculated and experimental values. This procedure is similar to take the sum of squares of the differences, therefore, the errors will not cancel among them. This fitness criterion is published by the authors in a previous work:

-Rodríguez, M. M., Arballo, J. R., Campañone, L. A., Cocconi, M. B., Pagano, A. M.,Mascheroni, R.H. (2013). Osmotic dehydration of nectarines: Influence of the operating conditions and determination of the effective diffusion coefficients. Food Bioprocess Technol., 6:2708–2720.

8. What is the R2 (generalized determination coefficient) for each regression procedure? ARE has not an upper limit and its value depends on the magnitude order of fitted variable so their values do not allow to verify the fitness quality in comparison to other studies. Fitted plots should be presented jointly with experimental data.

Answer: Minimize ARE is used as criterion to obtain the diffusion coefficients, a value lower than 15% is considered as upper value for good prediction criterion:

-Rodríguez, M. M., Arballo, J. R., Campañone, L. A., Cocconi, M. B., Pagano, A. M.,Mascheroni, R.H. (2013). Osmotic dehydration of nectarines: Influence of the operating conditions and determination of the effective diffusion coefficients. Food Bioprocess Technol., 6:2708–2720.

Besides, R2 is included in the new version of the manuscript (calculated from MS Excel software), showing a good concordance between predicted and experimental data. Table 5 includes the predicted and experimental curves and presents a summary of this information.

This paragraph was included in the new version of the manuscript:

"Table 5 shows the experimental and predicted values using the water and sugar diffusion coefficients. The R2 coefficients for the water loss were greater than 0.92 implies a high degree of adjustment. In the case of soluble solids, a good fit can be observed, however, the range was broad (0.72-0.99), this behavior can be attributed to the experimental data, which are more dispersed."

9. In my experience diffusivity values presented by the authors are on the high side for OD products.

<u>Answer</u>: There are several manuscripts in literature, those articles support our results. A summary of some data are summary bellow:

- Derossi et al., 2008: Dw: $0.2\text{x}10^{-09}$ – $0.5\text{x}10^{-09}$ m^2/s OD of apples.
- Rodríguez et al., 2013: Dw: $0.70\text{x}10^{-09}$ – $4.8\text{x}10^{-09}$ m^2/s and Ds: $0.26\text{x}10^{-09}$ – $1.70\text{x}10^{-09}$ m^2/s OD of nectarines.
- Khoyi and Hesari, 2007: Dw: $1.07\text{x}10^{-09}$ – $4.06\text{x}10^{-09}$ m^2/s and Ds: $0.77\text{x}10^{-09}$ – $3.13\text{x}10^{-09}$ m^2/s OD of apricots.
- Park et al., 2002: Dw: $0.35\text{x}10^{-09}$ – $1.92\text{x}10^{-09}$ m^2/s and Ds: $0.20\text{x}10^{-09}$ – OD of pears.
- El-Aouar et al., 2003: they informed diffusion coefficients of 10^{-09} m^2/s during OD of papaya.
- Barrera et al., 2004: Dw: $0.12\text{x}10^{-09}$ – $0.23\text{x}10^{-09}$ m^2/s, OD of apples.
- Rodrigues et al., 2003: Dw: $0.31\text{x}10^{-09}$ – $0.65\text{x}10^{-09}$ m^2/s and Ds: $0.11\text{x}10^{-09}$ – $0.93\text{x}10^{-09}$ m^2/s OD of papaya.
- Manafi et al., 2011: Dw: $1.37\text{x}10^{-09}$ m^2/s and Ds entre $1.15\text{x}10^{-09}$ m^2/s OD of apricots.
- Porciuncula et al., 2013: Dw: $3\text{x}10^{-09}$ -$7\text{x}10^{-09}$ m^2/s OD of banana.
- Rastogi et al., 2004: Dw: $0.66\text{x}10^{-09}$ m^2/s and Ds: $0.41\text{x}10^{-09}$ m^2/s OD of potato.
- Simpson et al., 2015: Dw: $0.15\text{x}10^{-09}$ -$0.51\text{x}10^{-09}$ m^2/s OD of apples.
- Togrul and Ispir, 2007: Dw: $5.139\text{x}10^{-09}$ - $10.342\text{x}10^{-09}$ m^2/s and Ds: $0.767\text{x}10^{-10}$ - $1.755\text{x}10^{-10}$ m^2/s OD of apricots.

Barrera, C., Betoret, N., & Fito, P. (2004). Ca2+ and Fe2+ influence on the osmotic dehydration kinetics of apple slices (var. Granny Smith). Journal of Food Engineering, 65: 9–14.

Derossi, A., De Pilli, T., Severini, C- (2008). Mass transfer during osmotic dehydration of apples. Journal of Food Engineering, 86:519-528.

El-Aouar, A. A., Azoubel, P. M., & Murr, F. E. X. (2003). Drying kinetics of fresh and smotically pretreated papaya (Carica papaya L.). Journal of Food Engineering, 59: 85–91.

Khoyi, M. R., Hesari, J. (2007). Osmotic dehydration kinetics of apricot using sucrose solution. Journal of Food Engineering, 78: 1355-1360.

Manafi, M., Hesari, J., Peighambardoust, S. H., Damirchi, S. A., Khoyi, M. R. (2011). kinetic study of osmotic dehydration of apricot using salt solutions. CyTA – Journal of Food, 9(3): 167–170.

Park, K. J., Bin, A., Brod, F. P. R., & Park, T. H. K. B. (2002). Osmotic dehydration kinetics of pear D'anjou (Pyrus communis L.). Journal of Food Engineering, 52: 293–298.

Porciuncula, B. D. A., Zotarelli, M. F., Carciofi, B. A. M., Laurindo, J. B. (2013). Determining the effective diffusion coefficient of water in banana (Prata variety) during osmotic dehydration and its use in predictive models. Journal of Food Engineering, 119: 490–496.

Rastogi, N. KRaghavarao, K. S. M. S. (2004). Mass Transfer During Osmotic Dehydration. Determination of Moisture and Solute Diffusion. Coefficients from Concentration Profiles. Food and Bioproducts Processing, 82(C1): 44–48.

Rodrigues, A. C. C., Cunha, R. L., & Hubinger, M. D. (2003). Rheological properties and color evaluation of papaya during osmotic dehydration processing. Journal of Food Engineering, 59: 129–135.

Rodríguez, M. M., Arballo, J. R., Campañone, L. A., Cocconi, M. B., Pagano, A. M., Mascheroni, R.H. (2013). Osmotic dehydration of nectarines: Influence of the operating conditions and determination of the effective diffusion coefficients. Food Bioprocess Technol., 6: 2708–2720.

Simpson, R., Ramírez, C., Birchmeier, V., Almonacid, A., Moreno, J., Nuñez, H., Jaques, A. (2015). Diffusion mechanisms during the osmotic dehydration of Granny Smith apples subjected to a moderate electric field. Journal of Food Engineering, 166: 204–211.

Togrul, I, T., Ispir, A., I. (2007). Effect on effective diffusion coefficients and investigation of shrinkage during osmotic dehydration of apricot. Energy Conversion and Management 48: 2611–2621.

The authors thank Reviewer #2 for the comments and suggestions that help us to improve the manuscript.

Analysis of operating conditions on osmotic dehydration of plums (*Prunus domestica* L.) and numerical real-shape determination of effective diffusion coefficients

María M. Rodríguez[1]; Javier R. Arballo[2,3] ; Laura A. Campañone[2,3] and Rodolfo H. Mascheroni[2,3]

[1] TECSE, Facultad de Ingeniería, Universidad Nacional del Centro de la Provincia de Buenos Aires – (7400) Olavarría (Argentina)

[2] CIDCA (Centro de Investigación y Desarrollo en Criotecnología de Alimentos) (CONICET LA PLATA-CIC-U.N.L.P.). 47 y 116 - (1900) La Plata (Argentina)

[3] Facultad de Ingeniería – U.N.L.P. 116 y 47 - (1900) La Plata (Argentina)

Corresponding author: Javier Ramiro Arballo, jrarballo@conicet.gov.ar; Tel +54 221 4249287, Fax +54 221 4890741

Abstract. The objective of this work was to analyze the relevant process conditions on osmotic dehydration of plums and to determine the diffusion coefficients related to this process. The influence of solution (type and concentration of solute, temperature, fruit/solution ratio) and process time on water loss, water content and solutes gain were studied. 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 phenomena. Experiments were carried out with glucose and sorbitol solutions (40-60% w/w), dehydrating plum pieces during 2h at temperatures of 25 and 40ºC, with fruit/solution ratios of 1/4 and 1/10. For calculating effective diffusion coefficients, a novelty inverse-method was applied, the real shape of food-pieces was considered using Finite Elements Method. Calculated diffusion coefficients ranged from 1.13×10^{-09} to 4.71×10^{-09} m^2 s^{-1} and 0.44×10^{-09} to 3.46×10^{-09} m^2 s^{-1}, for water and solutes, respectively.

Keywords: Plums, osmotic dehydration, diffusion coefficients.

1. Introduction

Nowadays, the consumers demand nutritious and natural foods such as fruits and vegetables, in belief that foods contribute directly to their health [1]. The incorporation of whole fruits in the diet is a topic of interest due to scientific agreement that they may help to lower the incidence of certain types of cancer, cardiovascular and neurodegenerative diseases, and DNA damage and even may have anti-aging properties [1, 2].

In particular plum (*Prunus domestica* L.) is the most numerous and diverse group of fruit tree species. Plums contain numerous phenolic phytochemicals, such as flavonoids and phenolic acids, which may function as effective natural antioxidants in our daily diet [3]; Wang et al. [4] demonstrated that plums had 4.4 times higher total antioxidant capacities than apples.

During the last years numerous studies have been developed to osmotic dehydration of stone fruits, like plums [6, 7], cherries [8], peaches [9], apricots [10] and nectarines [11] 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 – [12].

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 [13]. 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/solution ratio, solution temperature and agitation and process time [14].

Mass transfer parameters, such as diffusivity and transfer coefficient, must be obtained for an efficient analysis of dehydration process [11]. 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 common means to describe dehydration processes and as is known as "diffusive mechanism" [15]. 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. Sareban and Abbasi Souraki [16] investigated osmotic dehydration 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 strictly valid for irregular shaped 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 [11].

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 [11]. The study revealed that diffusional coefficients calculated by the analytical method were higher than those calculated considering the real geometry, overestimating the rate of diffusion for the same values of water loss and solid gain.

In the present work, this novelty technique was adapted to plums pieces considering the new product geometry and different operating conditions.

In agreement to the expressed so far, to our knowledge, a deep study of this particular process in plums has not been done. Therefore, the present work deals with the study of osmotic dehydration of plums as a function of process conditions; besides the effective diffusion coefficients for water and solutes transfer were predicted through the use of computational tools that allow consider the real shape of food pieces.

2. Materials and Methods

2.1 Preparation and characterization of samples

Experiments were carried out with Plums of the variety D'ente (*Prunus doméstica* L.). The fruits were harvested from the Chacra Experimental at the Facultad de Agronomía of UNCPBA located in the city of Azul, Buenos Aires (Argentina). Initial moisture of the fresh fruit was 4.205±1.218 g water g/dry solid (84.43% to 74.92%, w.b.) [17], and the initial content of soluble solids was 18.75±1.48% (w.b.), determined by Abbe refractometer (accuracy±0.01). 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 1).

2.2 Osmotic Dehydration

The dehydration process was done for 2 h– initial period of high water removal [18] – 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. To prevent flotation, samples were kept immersed in the solutions using a stainless steel mesh; two fruit/solution ratios: 1/4 or 1/10 were employed. The experiments were carried out at two temperatures: 25 and 40ºC. 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 [11]:

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

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

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

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 (kg); W_t is the mass of sample (kg) at time t.

2.3 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 [19]:

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

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

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.

Eqs. (4) and (5) were solved using the following assumptions:

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

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 volume to sample weight is high enough as to secure almost constant solution concentration [20].

The microscopic mass balances (Eqs. (4) and (5)) were solved taking into account initial uniform concentration in all domain and Dirichlet boundary condition at the surface (equilibrium concentration). These equilibrium values (water and sugar) were obtained from the experimental kinetic curves, considering the asymptote at 2 hours of process.

For the determination of the effective diffusion coefficients of water and solids, the software Matlab 7.10.0 was used developing an algorithm that 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 [21]. Then, the numerical solutions for these combinations could be obtained with the assistance of COMSOL software (COMSOL Multiphysics); this solution was compared with experimental data (C_w and C_s as a function of process time) through the average relative error (Eq. 6) for each pair D_w-D_s tested:

$$ARE = ARE_w + ARE_s \tag{6}$$

The average relative error (ARE) (Eq. 7) was the statistical parameter used to estimate the quality of model adjustment.

$$ARE_j = \sum_i \left| \frac{C_j^{exp} - C_j^{cal}}{C_j^{exp}} \right| \tag{7}$$

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.

The pair which minimized the error function (Eq. 6) was considered valid for the selected operating conditions. A more detailed explanation of the calculation methodology was developed by the authors in a previous work [11].

2.4 Statistical analysis

The statistical study of the results was performed using the analysis of variance (ANOVA) with a significance level (SL) of 5% or p-value<0.05. This analysis was performed using the InfoStat software (Universidad Nacional de Córdoba, 2004).

3. Results and Discussion

3.1 Water loss kinetics during osmotic dehydration

Water loss (WL) of samples dehydrated during 120 minutes in glucose and sorbitol solutions are shown in Figure 2. 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), are drawn. 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, osmotic agent, its concentration and fruit/solution ratio influenced it significantly considering their main effect.

Also, combined effects were analyzed. 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 the 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. [22] and Ispir and Togrul [23] in OD of apricots in solutions of glucose, sorbitol, fructose, sucrose and maltodextrin and by Ferrari et al. [24] in OD of pears in sucrose and sorbitol solutions.

Significant influence was found between process temperature and concentration (p=0.0020) and temperature with fruit/solution ratio (p=0.0363). Individual ANOVA tests for each osmotic agent showed that the interaction between temperature and concentration affects significantly the WL in the process when using glucose solutions (p=0.0002); on the other hand, using sorbitol solutions WL was influenced by the interaction between temperature and fruit/solution ratio (p=0.0266). The highest fruit/solution ratio allowed obtaining – in most tests – plums with lower water content. Khoyi and Hesari [10] – 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. Similar results were reported by Ispir and Togrul [23] for OD of apricots.

3.2 Solids gain kinetics during osmotic dehydration

Solid gains (SG) of samples dehydrated during 120 minutes in glucose and sorbitol solutions are shown in Figure 3.

Related the rate of SG during OD of plums, process time, type of solute, its concentration and fruit/solution ratio influenced it significantly (Table 2). Main effect of temperature is not significant on SG and WL. This result is in agreement with those obtained by Islam and Flink [25] and Ozen et al. [26], where these authors remark that a mild increase in process temperature has no effect on SG. 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). In the same way as WL, individual analysis of variance for each solute showed that the interaction between temperature and concentration is significant when glucose is used (p=0.0002) and the combined effect of the fruit/solution ratio and temperature affects significantly SG (p=0.0266). The increase in the ratio fruit/solution clearly favored SG, being this effect more noticeable when using sorbitol as dehydrating agent (Figure 3b).

3.3 Determination of Process Efficiency

To analyze the obtained results of different operating conditions of the OD process, the index of efficiency as defined by Lazarides [27] (ratio between WL and SG) was calculated. Table 3 presents the results obtained for all the experimental conditions tested.

For all the analyzed conditions, water loss was higher than solutes gain, 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.

The efficiency of osmotic treatment varied according operating conditions. Solutions with the lower concentration (40%) gave higher efficiency index than those with 60%, for both solutes. In the same way highest efficiencies were obtained with a fruit/solution ratio of 1/4.

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, but ratio efficiency indexes were lower. These results are in accordance with those of Ferrari et al. [24] during OD of pears in sucrose and sorbitol solutions and Rodriguez et al. [11] working on nectarines.

3.4 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.

Typical water and soluble solids predicted concentration profiles using the real geometry considering specific initial condition - Glucose concentration 40%, fruit/solution ratio 1/4 and medium temperature of 25ºC - (Cw_0= 799.4 kg/m^3 and Cs_0= 187.5 kg/m^3) are presented in Figure 4. In same Figure, color bars are included, they indicate the concentration level, red and blue are associated to high and low concentration of this species. 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 volumetric integration of these profiles, time variation of average water and solute concentrations can be obtained.

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

The effective diffusion coefficients for water varied between 1.13x10^{-09} and 4.71x10^{-09} m^2 s^{-1}; the effective diffusion coefficients for solids ranged between 0.44x10^{-09} and 3.46x10^{-09} m^2 s^{-1}. The values of ARE were lower than 5 10^{-2} for both components, showing the high quality of the numerical fitting between experimental and predicted values. 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. Table 5 shows the experimental and predicted values using the water and sugar diffusion coefficients. The R^2 coefficients for the water loss were greater than 0.92 implies a high degree of adjustment. In the case of soluble solids, a good fit can be observed, however, the range was broad (0.72-0.99), this behavior can be attributed to the experimental data, which are more dispersed.

From the obtained results at 40% (w/w), it can be observed that a combined decrease of fruit/solution ratio and temperature provokes an increase of both diffusion coefficients. Besides, at 60% (w/w), the same behavior was observed but associates to an inverse interaction between ratio and temperature. These different scenes can be explained taking into account that at high concentration, the viscosity of the solutions is affected by temperature and promotes a high mass transfer rate; at lower concentration the effect of the temperature is not relevant, demonstrated by the statically analysis performed in the previous section.

The values given in Table 4 are in accordance with those published by different researchers. According to Ispir and Togrul [23], D_w varied between 0.77x10^{-10} and 1.75x10^{-10} m^2 s^{-1} in OD of apricots, meanwhile Sabarez and Price [28] 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 [8] 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. [29] 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. [11] 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 flat 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 1.14x10^{-10} and 1.08x10^{-08} m^2 s^{-1}, while those calculated using the true sample shape ranged between 0.26x10^{-09} and 1.70x10^{-09} m^2 s^{-1}.

4. Conclusions

Analysis of the experimental data revealed the influence of main process parameters on osmotic dehydration of plums. Water loss was significantly dependent on process time, type and concentration of solution and fruit/solution ratio, but temperature had statistically no effect as individual effects. According the statistical analysis, there were also significant effects between process temperature and concentration and temperature with fruit/solution ratio, process time with type of osmotic agent and process time with solute concentration. Besides, the interaction between the type 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 significant 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 high efficiency values were obtained using solutions at 40% w/w and fruit/solution ratio of 1/4.

The calculated effective diffusion coefficients using the numerical technique for water varied between $1.13\text{x}10^{-09}$ to $4.71\text{x}10^{-09}$ m^2 s^{-1}; the effective diffusion coefficients for solids ranged between $0.44\text{x}10^{-09}$ to $3.46\text{x}10^{-09}$ m^2 s^{-1}. The values of ARE were lower than 5 10^{-2} for both components, showing the high quality of the numerical fitting between experimental and predicted ones.

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

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Legends of Figures

Figure 1: Piece of fruit (1/8), 3D model.

Figure 2: Water Loss of plums OD in glucose (a) or sorbitol (b) solutions.

Figure 3: Soluble solids gain of plums OD in glucose (a) or sorbitol (b) solutions.

Figure 4: Final profiles of water (a) y soluble solids concentration (b) simulated during OD of plum portions using COMSOL-Multiphysics (7200 s, glucose at 40% w/w, fruit /solution ratio 1/4 and temperature of 25°C).

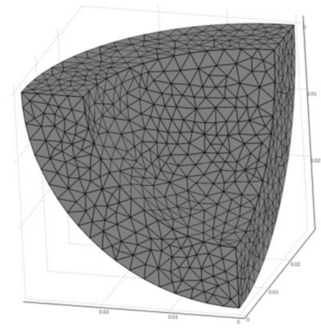


Figure 1

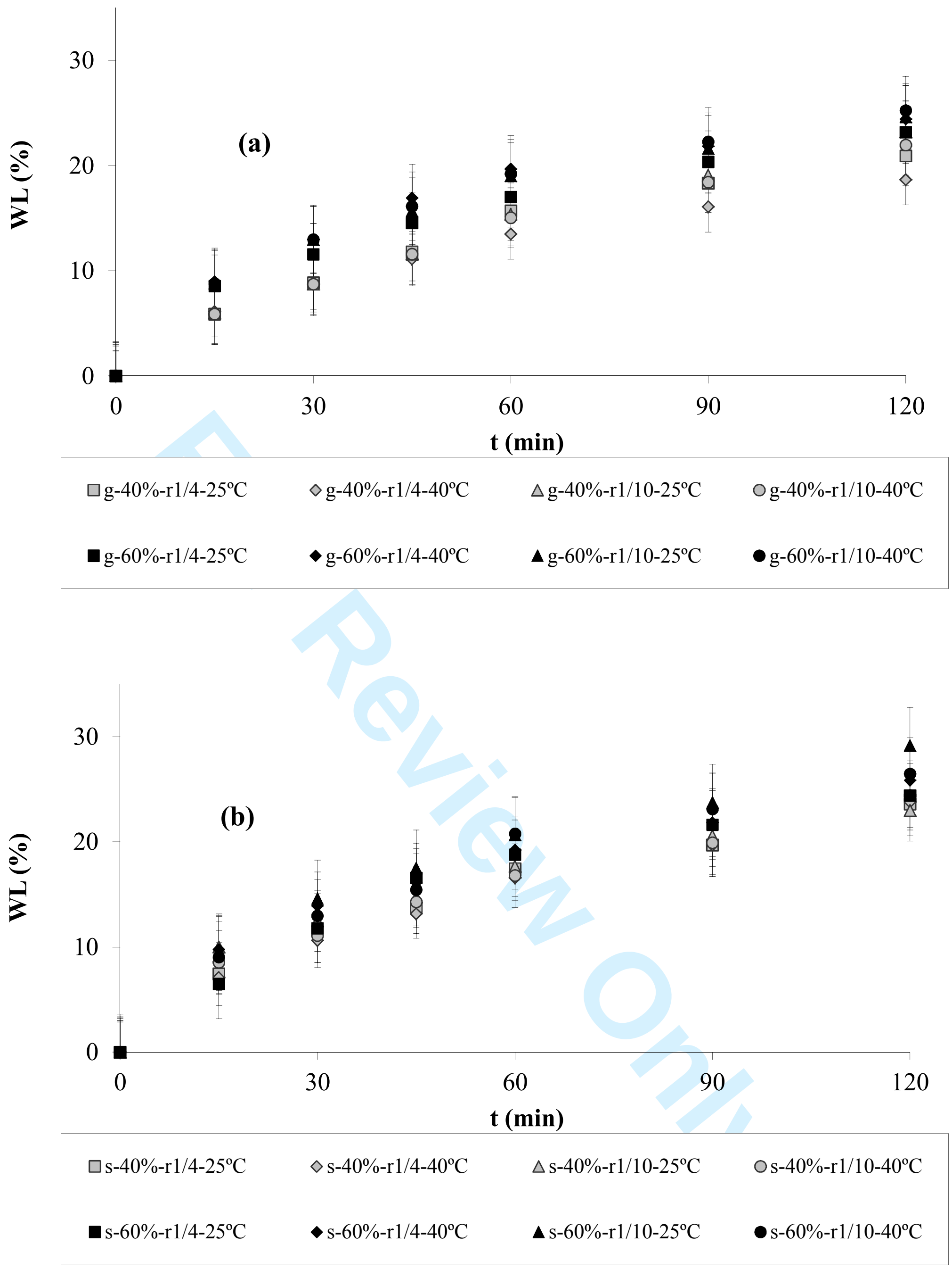
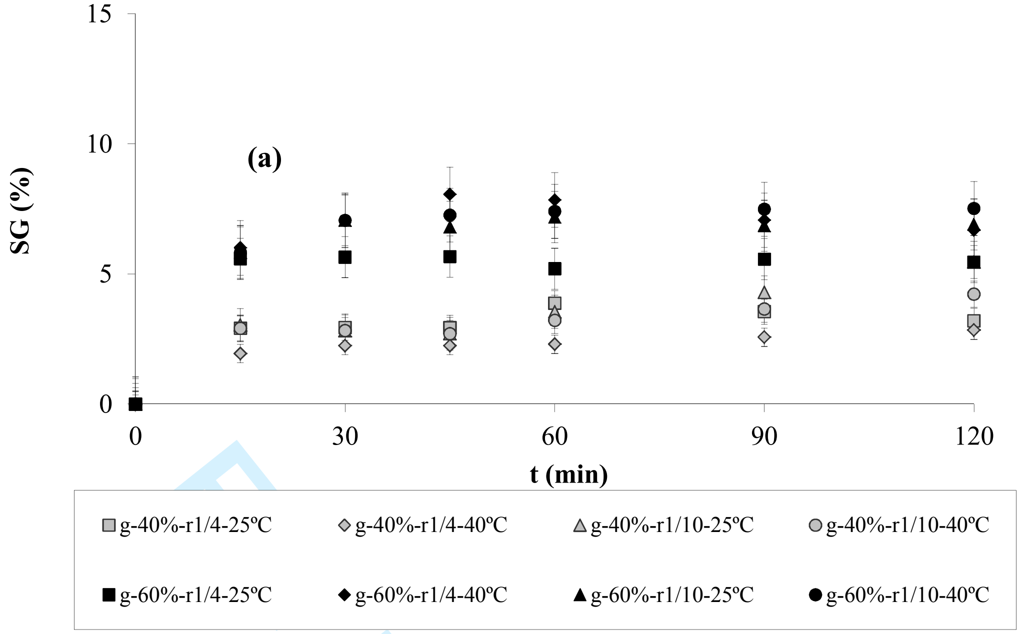

(a)
WL (%)
t (min)
0
10
20
30
0
30
60
90
120
g-40%-r1/4-25ºC
g-40%-r1/4-40ºC
g-40%-r1/10-25ºC
g-40%-r1/10-40ºC
g-60%-r1/4-25ºC
g-60%-r1/4-40ºC
g-60%-r1/10-25ºC
g-60%-r1/10-40ºC
(b)
WL (%)
t (min)
0
10
20
30
0
30
60
90
120
s-40%-r1/4-25ºC
s-40%-r1/4-40ºC
s-40%-r1/10-25ºC
s-40%-r1/10-40ºC
s-60%-r1/4-25ºC
s-60%-r1/4-40ºC
s-60%-r1/10-25ºC
s-60%-r1/10-40ºC

Figure 2.


(a)
SG (%)
t (min)
15
10
5
0
0
30
60
90
120
g-40%-r1/4-25ºC
g-40%-r1/4-40ºC
g-40%-r1/10-25ºC
g-40%-r1/10-40ºC
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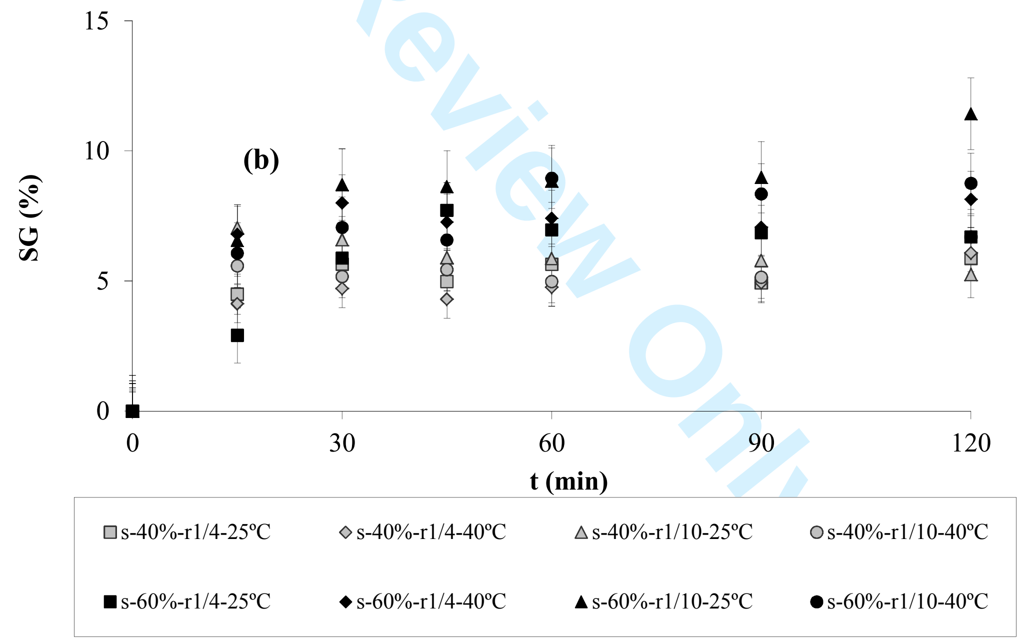
g-60%-r1/10-40ºC


(b)
SG (%)
t (min)
15
10
5
0
0
30
60
90
120
s-40%-r1/4-25ºC
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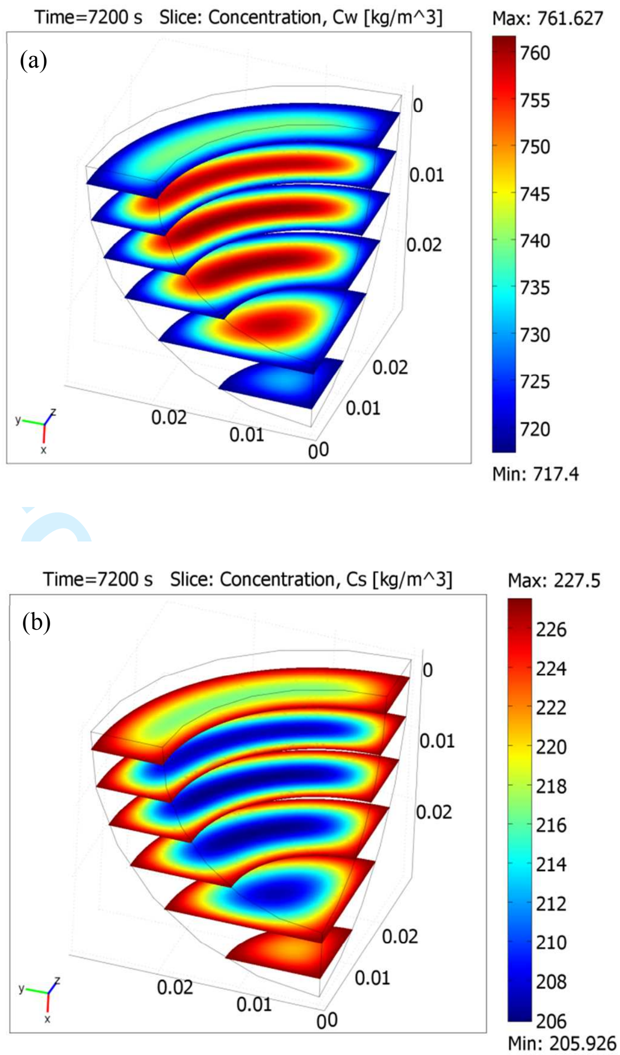
s-40%-r1/10-25ºC
s-40%-r1/10-40ºC
s-60%-r1/4-25ºC
s-60%-r1/4-40ºC
s-60%-r1/10-25ºC
s-60%-r1/10-40ºC

Figure 3.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Max: 761.627
(a)
760
755
750
745
740
735
730
725
720
Min: 717.4
0
0.01
0.02
0.02
0.01
0.02
0.01
00
Time=7200 s Slice: Concentration, Cs [kg/m^3]
Max: 227.5
(b)
226
224
222
220
218
216
214
212
210
208
206
Min: 205.926
0
0.01
0.02
0.02
0.01
0.02
0.01
00

Figure 4.

Table 1. Variance analysis of variables involved in the *WL* of OD plums.

Variables	***WL***		
	df	**F**	***p***
Time	6	1670.67	<0.0001
Osmotic Agent	1	89.12	<0.0001
Concentration	1	268.79	<0.0001
Fruit/Osmotic Agent ratio	1	26.01	<0.0001
Temperature	1	0.29	0.5937
Time*Osmotic Agent	1	3.28	0.0067
Time*Concentration	1	9.62	<0.0001
Time*Fruit/Osmotic Agent ratio	1	1.83	0.1046
Time*Temperature	1	0.13	0.9929
Osmotic Agent*Concentration	1	17.27	0.0001
Osmotic Agent* Fruit/Osmotic Agent ratio	1	1.00	0.3203
Osmotic Agent*Temperature	1	1.00	0.3208
Concentration*Fruit/Osmotic Agent ratio	1	0.37	0.5455
Concentration*Temperature	1	10.25	0.0020
Fruit/Osmotic Agent ratio*Temperature	1	4.55	0.0363

Table 2. Variance analysis of variables involved in the *SG* of OD plums.

Variables	*SG*		
	df	F	*p*
Time	6	131.79	<0.0001
Osmotic Agent	1	89.76	<0.0001
Concentration	1	278.64	<0.0001
Fruit/Osmotic Agent ratio	1	26.22	<0.0001
Temperature	1	0.05	0.8181
Time*Osmotic Agent	1	3.04	0.0106
Time*Concentration	1	9.72	<0.0001
Time*Fruit/Osmotic Agent ratio	1	1.48	0.1980
Time*Temperature	1	0.08	0.9976
Osmotic Agent*Concentration	1	17.14	0.0001
Osmotic Agent* Fruit/Osmotic Agent ratio	1	1.86	0.1770
Osmotic Agent*Temperature	1	1.47	0.2300
Concentration*Fruit/Osmotic Agent ratio	1	0.91	0.3426
Concentration*Temperature	1	9.93	0.0024
Fruit/Osmotic Agent ratio*Temperature	1	6.45	0.0133

Table 3. Efficiency Index of the OD for plums.

Nº	Osmotic Treatment	Efficiency Index
1	g-40%-r1/4-25 ºC	6.54
2	g-40%-r1/4-40 ºC	6.57
3	g-40%-r1/10-25 ºC	4.25
4	g-40%-r1/10-40 ºC	5.19
5	g-60%-r1/4-25 ºC	4.25
6	g-60%-r1/4-40 ºC	3.65
7	g-60%-r1/10-25 ºC	3.57
8	g-60%-r1/10-40 ºC	3.36
9	s-40%-r1/4-25 ºC	4.02
10	s-40%-r1/4-40 ºC	3.92
11	s-40%-r1/10-25 ºC	4.37
12	s-40%-r1/10-40 ºC	3.65
13	s-60%-r1/4-25 ºC	3.65
14	s-60%-r1/4-40 ºC	3.18
15	s-60%-r1/10-25 ºC	2.55
16	s-60%-r1/10-40 ºC	3.02

Table 4. Effective diffusion coefficients for water and solids during OD of plums, calculated using the numerical method.

		Results of numerical method			
N°	**Osmotic Treatment**	***D_w*** (m^2 s^{-1})	***ARE (x 10^2)***	***D_s*** (m^2 s^{-1})	***ARE (x10^2)***
1	g-40%-r1/4-25°C	$2.06x10^{-09}$	0.57	$1.50x10^{-09}$	3.48
2	g-40%-r1/4-40°C	$4.71x10^{-9}$	0.32	$1.55x10^{-9}$	0.30
3	g-40%-r1/10-25°C	$1.13x10^{-09}$	0.99	$0.44x10^{-9}$	5.00
4	g-40%-r1/10-40°C	$1.33x10^{-09}$	0.66	$0.57x10^{-09}$	3.91
5	g-60%-r1/4-25°C	$2.63x10^{-09}$	0.49	$1.76x10^{-09}$	0.91
6	g-60%-r1/4-40°C	$3.41x10^{-09}$	0.32	$2.93x10^{-09}$	1.54
7	g-60%-r1/10-25°C	$2.79x10^{-09}$	0.34	$2.13x10^{-09}$	1.05
8	g-60%-r1/10-40°C	$2.49x10^{-09}$	0.23	$1.83x10^{-09}$	1.27
9	s-40%-r1/4-25°C	$2.18x10^{-09}$	0.55	$1.24x10^{-09}$	2.03
10	s-40%-r1/4-40°C	$1.45x10^{-09}$	0.57	$0.87x10^{-09}$	2.52
11	s-40%-r1/10-25°C	$4.07x10^{-09}$	0.68	$3.46x10^{-09}$	1.25
12	s-40%-r1/10-40°C	$1.55x10^{-09}$	0.85	$0.87x10^{-09}$	1.79
13	s-60%-r1/4-25°C	$2.70x10^{-09}$	0.91	$2.10x10^{-09}$	4.11
14	s-60%-r1/4-40°C	$2.07x10^{-09}$	0.89	$1.72x10^{-09}$	1.56
15	s-60%-r1/10-25°C	$1.50x10^{-09}$	0.94	$1.13x10^{-09}$	2.11
16	s-60%-r1/10-40°C	$2.18x10^{-09}$	0.83	$1.46x10^{-09}$	3.13

Table 5. Predicted and experimental curves of water loss and soluble solids under different operating conditions.

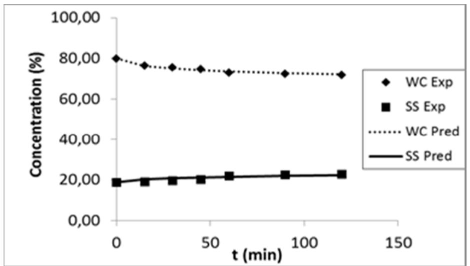
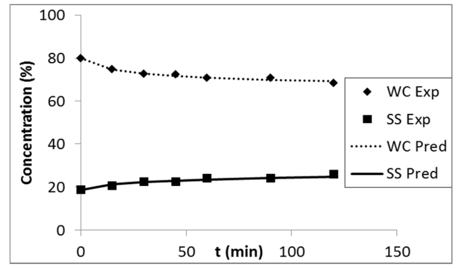
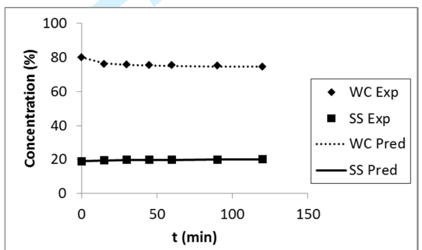
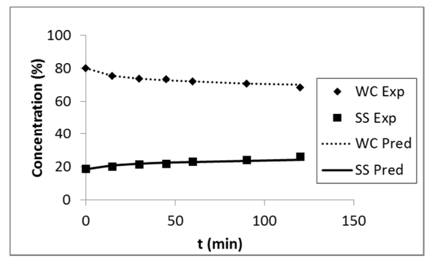
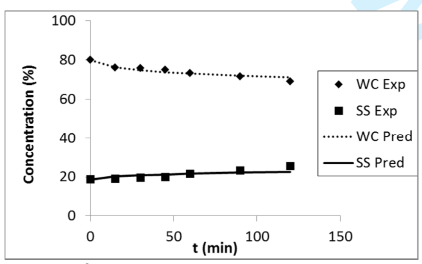
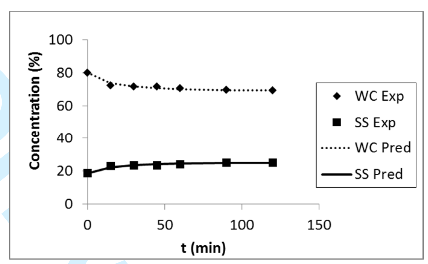
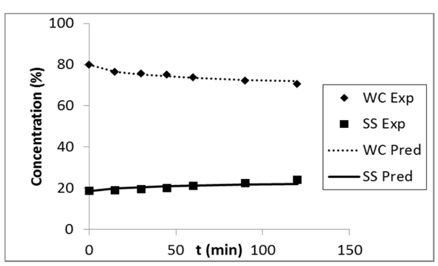
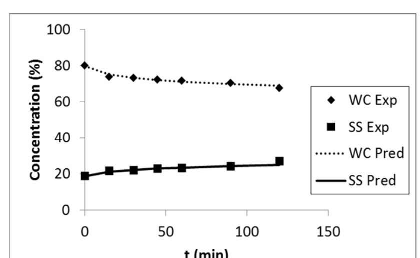
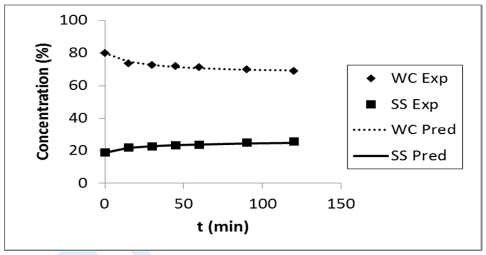
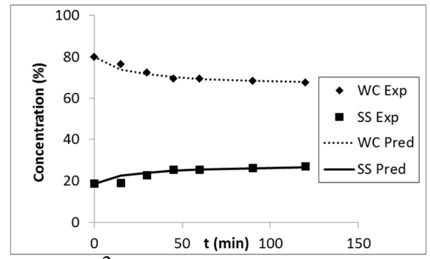
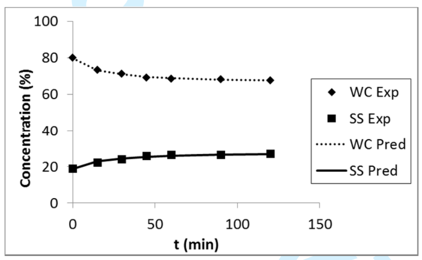
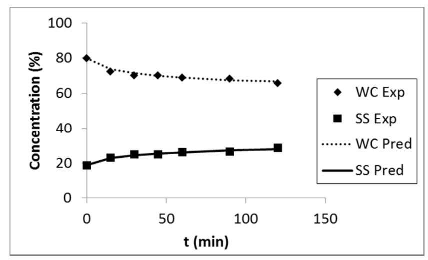
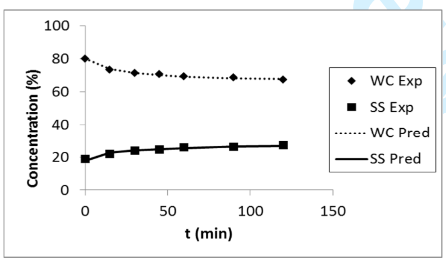
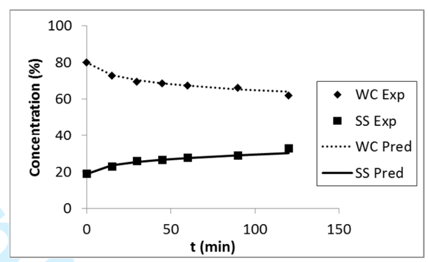
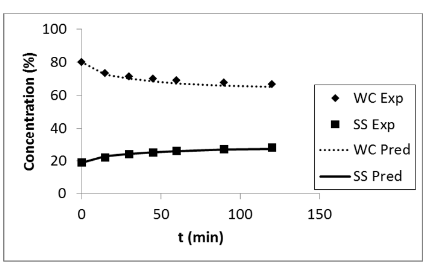
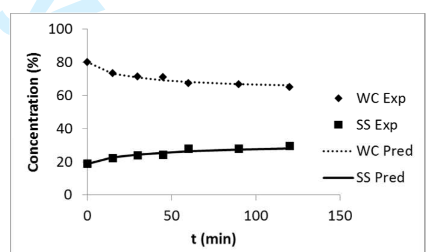
Osmotic Treatment	Glucose Solution	Sorbitol Solution
40%-r1/4-25°C	 R^2 WC/SS=0.97 / 0.78	 R^2 WC/SS=0.98 / 0.94
40%-r1/4-40°C	 R^2 WC/SS=0.97 / 0.95	 R^2 WC/SS=0.97 / 0.89
40%-r1/10-25°C	 R^2 WC/SS=0.92 / 0.74	 R^2 WC/SS=0.96 / 0.95
40%-r1/10-40°C	 R^2 WC/SS=0.95 / 0.77	 R^2 WC/SS=0.95 / 0.91

Table 5. Predicted and experimental curves of water loss and soluble solids under different operating conditions (Cont.).

Osmotic Treatment	Glucose Solution	Sorbitol Solution
60%-r1/4-25°C	 R^2 WC/SS=0.98 / 0.98	 R^2 WC/SS=0.95 / 0.83
60%-r1/4-40°C	 R^2 WC/SS=0.99 / 0.98	 R^2 WC/SS=0.97 / 0.97
60%-r1/10-25°C	 R^2 WC/SS=0.99 / 0.97	 R^2 WC/SS=0.97 / 0.95
60%-r1/10-40°C	 R^2 WC/SS=0.99/0.99	 R^2 WC/SS=0.97/0.94