

Modeling mass transfer during osmotic dehydration of different vegetable structures under vacuum conditions

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Abstract

Osmotic dehydration (OD) is a method to partially reduce moisture of food, aiming to improve the shelf life and the stability of the products. The mass transfer that occurs during this process can be enhanced with the application of reduced pressure in the first minutes of the osmotic dehydration (pulsed vacuum osmotic dehydration - PVOD). The present work aimed to study the vacuum effect on the kinetics of osmotic dehydration of vegetables (carrot, eggplant and beetroot) in terms of water loss, solids gain and water activity, in ternary solution. Moreover, mathematical modeling of experimental data obtained from the osmotic processes, was employed for correlating the mass transfer of the food product under the different conditions. An intensification of mass transfer was observed for carrot and eggplant in the vacuum treatments. This was related to their porous structure. The present work demonstrated a lack of fit of Fick's second law for describing the dehydration kinetics of the eggplant.

Keywords: vacuum application; carrot; beetroot; eggplant; impregnation.

Practical Application: Preservation processes for different vegetable structures.

1 Introduction

The dehydration of food is an usual technique to extend the shelf life and the stability of perishable products. The osmotic dehydration (OD) is a simple process, in which the material is immersed in a hypertonic liquid medium. Due to the osmotic pressure gradient, two mass transfer fluxes are generated, the water present in the tissues migrates to the osmotic solution (dehydration), and the solutes of the osmotic solution are incorporated to the food matrix (impregnation) (Luchese et al., 2015; Ramya & Jain, 2017).

The mass transfer rates can be intensified if in the beginning of the process a reduced pressure is applied, in the operation known as pulsed vacuum osmotic dehydration (PVOD). The partial vacuum promotes internal gas expansion, and with the atmospheric pressure restoration, these gases are removed through the hydrodynamics mechanism (HDM), accelerating the process (Fito, 1994; Junqueira et al., 2017a; Moreno et al., 2016).

The internal structure of the food material influences the dehydration behavior. Mass transfer through porous structure is related to the higher fluxes of water removal and solids incorporation during the osmotic dehydration. This is observed due to the larger internal surface area. During the PVOD, this effective surface for mass transfer is extended due to the expulsion of the occluded fluids on account of the pressure gradient created (Fito, 1994; Junqueira et al., 2018; Şahin & Öztürk, 2016).

Numerous studies have been conducted to improve knowledge regarding the mass transfer phenomena during the OD for modeling the relevant mechanisms of the process. Furthermore, the mass transport mechanisms during the OD are complex and not completely understood. Theoretical, semi-theoretical and semi-empirical models are mathematical models employed to define the dehydration behavior of agricultural products (Horuz et al., 2017; Silva et al., 2012).

Dehydration models have been lead by considering the diffusion as the majority mechanism for describing the whole mass transfer through the food and the osmotic solution. These semi-theoretical models are mainly derived from the direct solution of Fick's second law (Crank, 1975). This model allows the determination of the diffusivity coefficient, and it was satisfactorily used during the OD of pineapple (Silva et al., 2014), tomatoes (Abbasi Souraki et al., 2013) and jackfruit (Kaushal & Sharma, 2016).

Because the phenomenological models must portray the physical mechanism of the processes, Fito & Chiralt's model has been widely used for coupling the diffusional and hydrodynamics effects during the OD (Corrêa et al., 2016; Fito & Chiralt, 1997). However, because of the complex plant tissue and the structural changes after the OD, some models may present a lack of fit, due to the development of the analytical solution (Simpson et al., 2015).

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In this sense, the aim of this study was to evaluate the effects of vacuum application in different material structures (carrot, eggplant and beetroot) on the dehydration kinetics of water loss, solid gain and water activity and to fit the experimental data to semi-theoretical models.

2 Materials and Methods

2.1 Material

The carrot (*Daucus carota* L.), eggplant (*Solanum melongena* L.) and beetroot (*Beta vulgaris* L.) samples were characterized with respect to the initial moisture content (Association of Official Analytical Chemists, 2010), water activity (a_w) (Aqualab, 3-TE model, Decagon Devices Inc., Pullman, WA, USA), total soluble solids (Tecnal, AR-200 model, São Paulo, Brazil) and pH (Digimed, DMpH-2 model, São Paulo, Brazil). All the analyses were performed in triplicate, and the results are presented in Table 1.

2.2 Sample and osmotic solution preparation

Fresh vegetables were purchased from a local market (Lavras, MG, Brazil) and stored in a refrigerator at $8^\circ\text{C} \pm 1^\circ\text{C}$ until experimental use. All vegetables were washed with tap water, peeled, and sliced (2.00 cm length \times 2.00 cm width \times 0.40 ± 0.03 cm thickness) by using a stainless steel mold. The osmotic solutions were prepared with distilled water, sucrose [40 kg/100 kg (w/w)], and sodium chloride [10 kg/100 kg (w/w)]. The a_w of the ternary solution was 0.836 ± 0.001 .

2.3 Osmotic processes

The OD experiments were performed in an osmotic dehydrator with temperature and inner pressure control, as presented by Viana et al. (2014). The experiments were conducted at atmospheric pressure (OD) and under vacuum conditions (PVOD). For the PVOD treatments, vacuum pressures (VP) of 40 or 80kPa were applied to the system during the first 10 minutes of process, and then, the local atmospheric pressure (Lavras, MG, Brazil) was restored, 101.35 kPa (VP = 0) (Junqueira et al., 2018).

The temperature was set at $35 \pm 1^\circ\text{C}$ and ratio of solution to fruit was 1:10 (w/w) to prevent the dilution of the osmotic solution during the experiments. At set times (10; 20; 30; 40; 60; 90; 120; 180; 240 and 300 minutes) the samples were removed from the solution. Each removed sample was immersed in a bath of cold distilled water for 10 seconds to stop the osmotic process, and the surface of the samples was gently wiped with absorbent paper to remove excess solution. Finally, the samples were weighed, and the moisture content was calculated according to AOAC (Association of Official Analytical Chemists, 2010). All of the experiments were performed in four replicates.

Table 1. Physical characteristics of fresh vegetables.

	Carrot	Eggplant	Beetroot
Initial moisture content [kg water/kg]	0.870 ± 0.003	0.927 ± 0.004	0.875 ± 0.004
a_w	0.982 ± 0.003	0.989 ± 0.003	0.981 ± 0.003
pH	6.28 ± 0.02	5.84 ± 0.02	6.02 ± 0.01
Total soluble solids [kg solid/ kg]	0.112 ± 0.004	0.028 ± 0.004	0.133 ± 0.005

The water loss (WL) and solids gain (SG) were calculated in accordance with Equations 1 and 2, respectively,

$$WL = \frac{(M_0 \times X_{w0}) - (M_t \times X_{wt})}{M_0} \quad (1)$$

$$SG = \frac{(M_t \times X_{st}) - M_0 \times (I - X_{w0})}{M_0} \quad (2)$$

where X_{w0} is the initial water content (on wet basis), X_{wt} and X_{st} are the water and soluble solids content, respectively, at time t , and M_0 and M_t are the initial and final sample mass, respectively.

2.4 Mathematical models

The experimental kinetic data were fit to semi-theoretical models, with the effective diffusivity estimation.

Fick's second law for unidirectional unsteady state diffusion is given by Equation 3:

$$\frac{\partial X_t}{\partial t} = \frac{\partial}{\partial z} \times \left(D_{eff} \frac{\partial X_t}{\partial z} \right) \quad (3)$$

where X_t is water or solid content (on wet basis) at time t , D_{eff} is the effective diffusivity of water or solid [m^2/s], z is a general coordinate [m] and t denotes the time [s].

For infinite slab geometry, considering the experiment as a brief process and the internal transfer of moisture unidirectional, the D_{eff} is calculated according to Eq. 4

$$W = \left(\frac{8}{\pi^2} \sum_{i=0}^{\infty} \frac{1}{(2i+1)^2} \exp\left(- (2i+1)^2 \pi^2 D_{eff} \frac{t}{4L^2}\right) \right) \quad (4)$$

where W is the dimensionless water or solids content and L is the characteristic length (half the thickness) [m].

The initial condition is a uniform initial amount of water or solid, $X(z, 0) = X_0$. The boundary conditions are the symmetry of concentration, $\left. \frac{\partial X_t}{\partial z} \right|_{z=0} = 0$, and the equilibrium content at the surface, $X(L, t) = X_{eq}$ (Crank, 1975). The dimensionless water or solid content is given by Equation 5:

$$W = \frac{X_t - X_{eq}}{X_0 - X_{eq}} \quad (5)$$

where X_{eq} is the water or solid content at equilibrium (on wet basis) and X_0 is the initial water or solid content (on wet basis).

The Fito and Chiralt hydrodynamics model (Fito & Chiralt, 1997) considers an equilibrium approach (Equation 6):

$$z_{eq}^{ss} = y_{eq}^{ss} \quad (6)$$

where z_{eq}^{ss} is the mass fraction of the soluble solids in the food and y_{eq}^{ss} is the mass fraction of the soluble solids in the osmotic solution, and both are at an equilibrium state. Therefore, the effective diffusivity (D_{eff}) is the same for both the water and solids, and the changes in composition are functions of the reduced drive force, Y , which is given by Equation 7:

$$Y = Y_t^w = Y_t^s = \frac{z_t^w - z_{eq}^w}{z_0^w - z_{eq}^w} \quad (7)$$

The variation in the food liquid phase (FLP) composition related to the HDM occurs at the beginning of the process ($t = 0$ to $t = t_{HDM}$) where this mechanism is predominant and is dependent on the pressure gradients (Equation 8)

$$1 - Y_t^w \Big|_{t=t_0}^{t=t_{HDM}} \approx k \quad (8)$$

After this period, the phenomena are modeled with Fick's equation for a semi-infinite slab and a short time (Crank, 1975) with the approach suggested by Fito & Chiralt (1997) (Equation 9).

$$1 - Y_t^w \Big|_{t=t_{HDM}}^{t=t} = 2 \sqrt{\frac{D_{eff} t}{\pi L^2}} \quad (9)$$

These two effects were coupled to consider the effect of the diffusional and HDM (Equation 10).

$$1 - Y_t^w \Big|_{t=t_0}^{t=t} = k + 2 \sqrt{\frac{D_{eff} t}{\pi L^2}} \quad (10)$$

The D_{eff} and k parameters were obtained for each experiment from a linear fitting of the experimental $1 - Y_t^w$ versus \sqrt{t}

2.5 Statistical evaluation of the models

Data were analyzed using Statistica software (Statistica 8.0, Statsoft Inc., Tulsa, OK). The equation parameters were estimated using a non-linear regression procedure. The terms used to evaluate the goodness of fit were the correlation coefficient (R^2), root mean square error (RMSE) and reduced chi-square (χ^2). Higher R^2 and lower RMSE (Equation 11) and χ^2 (Equation 12) values indicated a better fit of the experimental data to the model (Horuz et al., 2017; Junqueira et al., 2017b).

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (MR_{pre,i} - MR_{exp,i})^2} \quad (11)$$

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{pre,i} - MR_{exp,i})^2}{N - n} \quad (12)$$

where the subscripts exp,i and pre,i denote experimental and predicted dimensionless water or solid content respectively, N is number of observations and n is the number of constants.

3 Results and Discussion

3.1 Water loss kinetics

The kinetics of WL in osmodehydrated vegetables are shown in Figure 1a-c. For all the vegetables, the water removal rate was higher in the first 120 min of the process. Initially, the dehydration

driving force between the product and the hypertonic solution is the strongest, and as the osmotic process extends, an osmotic pressure reduction occurs, due to mass transfer between the phases (Junqueira et al. 2017c; Lombard et al., 2008; Ramya & Jain, 2017).

For the carrot (Figure 1a) and eggplant (Figure 1b), the influence of the vacuum application (VA) during the first minutes was clearly observed. During the PVOD, the presence of the hydrodynamic mechanism (HDM) in the first minutes of process improves the mass transfer due to the expulsion of the internal gases and liquids occluded in the pores of the plant tissue and the replacement of this native material by the external osmotic solution (Ahmed et al., 2016; Fito, 1994; Moreno et al., 2016).

The carrot presents a heterogeneous composition, and even though it is considered a low porosity product, morphologically it is composed of an inner xylem (core) and an outer phloem (cortex) (Nahimana et al., 2011). The central stele presents a fibrous and porous structure, which may be related to the HDM action facilitation. After the first 10 minutes, the OD treatment presented lower WL, and the treatment PVOD 80 kPa showed higher WL (Figure 1b). The VA enhances the mass transfer in the initial period of the OD (Deng & Zhao, 2008), but at the end of the osmotic processes, no remarkable differences in the WL were observed. According to Figure 1a, the WL tended towards the equilibrium condition after 3 h for all the treatments.

According to the Figure 1b, the eggplant WL was strongly influenced by the VA, and higher WL was reached in PVOD processes. After 10 min of process, the samples subjected to VA presented a WL increase of approximately 30% (PVOD 40 kPa) and 40% (PVOD 80 kPa), respectively, when compared to OD at atmospheric pressure. Similar results were reported by Junqueira et al. (2017a) during the OD and PVOD of eggplant slices in salt solutions.

The higher water removal response of the eggplant, when the vacuum was applied, is related to its very porous structure (Russo et al., 2013). The pressure gradients during the PVOD, promote the outflow mainly of the occluded gases. When the atmospheric pressure is restored, the compression of the residual gas leads to an uptake of the external osmotic solution, increasing the WL (Atarés et al., 2008; Gras et al., 2002; Oliveira et al., 2016). It was also observed that even the VA increased the WL at the beginning of the processes, after 300 min, all the different treatments present similar values.

The effects of VA during the OD of beetroot (Figure 1c) were not evidenced after the 10 initial minutes, in which the PVOD was conducted. During all the osmotic process, the treatments presented similar behavior, and the influence of the VA was not noted. At the final time (300 min), no remarkable differences were found for all the treatments. This occurred probably due to the compact structure of this tissue, which is considered a low porous vegetable (Boukouvalas et al., 2006; Junqueira et al., 2018).

Viana et al. (2014) observed that the VA did not present a significant influence on the WL during OD and PVOD of fodder palm. Those authors reported that the absence of soft and porous structure, in addition to the quite hard texture, could hinder the

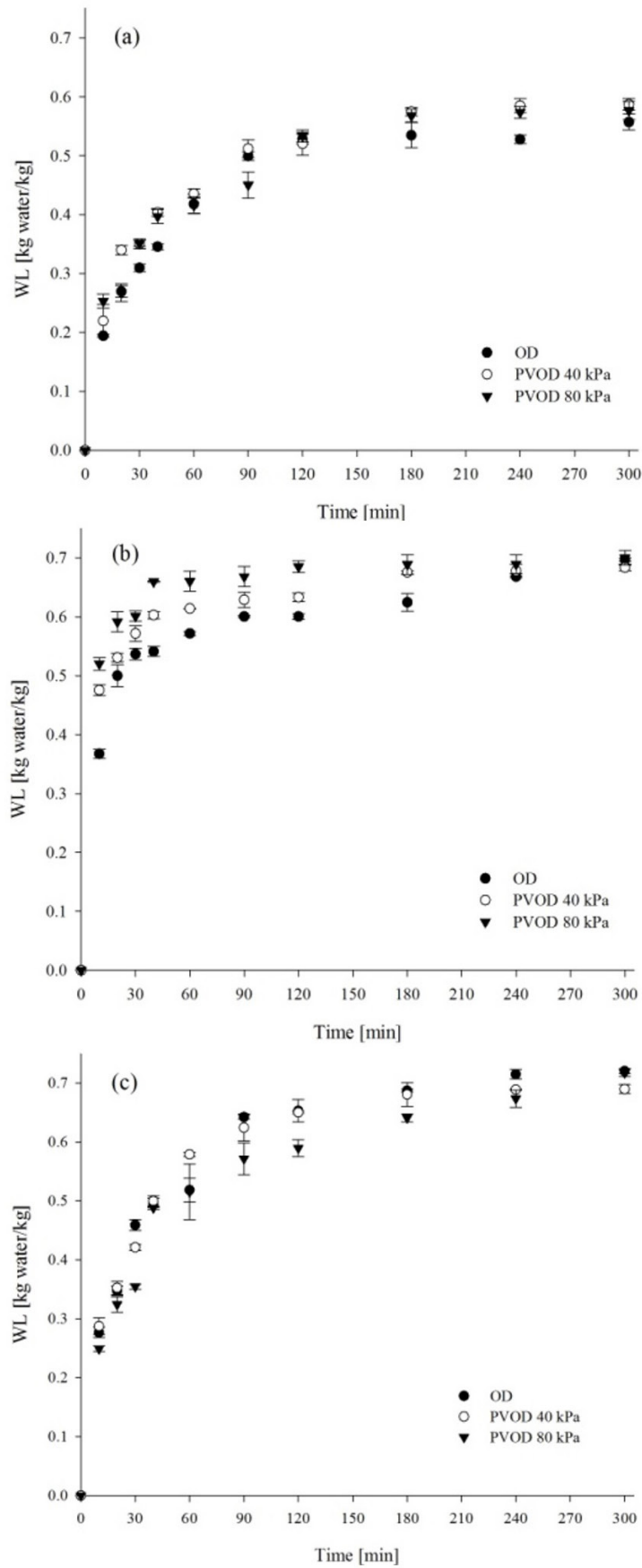


Figure 1. Kinetics of water loss (WL) of carrot (a), eggplant (b) and beetroot (c) during the OD.

exchanges established by the VA, with consequent insufficiency for reducing the mass transfer parameters. Corrêa et al. (2016) reported that regardless of the VA, little differences in the WL during the OD and PVOD of tomato slices were observed.

3.2 Solid gain kinetics

Figure 2 a-c present the SG kinetics of the osmodehydrated vegetables. The VA induced the SG for carrot and eggplant (Figure 2a and 2b), but reduced the SG for beetroot (Figure 2c). This behavior is explained in terms of different structural effects induced by the VA (Lin et al., 2016).

As presented for the WL, carrot and eggplant were directly influenced by the VA. For the carrot, the higher SG was observed for PVOD treatments during all the experimental period. The treatment PVOD 80 kPa promoted higher solid uptake rates. At the end of the process (300 min) no differences were reported for the treatments OD and PVOD 40 kPa, even though the VA application at this pressure increased the SG during the osmotic process.

For the eggplants, the VA also enhanced the SG rates (Figure 2b). After the VA, in the first 10 min, the SG doubled in PVOD treatments, compared with that obtained in the OD. At the end of the process (300 min), both vacuum treatments presented similar values, and also higher than the observed OD values. This result is consistent with the HDM action, coupled with diffusional osmotic phenomena, which accelerate mass transfer, due to the exchange of native internal gases and liquids with external solution solids (Betoret et al., 2015; Lin et al., 2016; Moreno et al., 2011).

For the beetroot (Figure 2c), the VA reduced the SG, contrary to the literature reports. Corrêa et al. (2016) related that this phenomenon was possible during the OD and PVOD of tomatoes in ternary osmotic solutions. The treatment conducted at atmospheric pressure presented higher SG during all the osmotic process and regarding the PVOD treatments, no significant differences were recorded (Figure 2c).

3.3 Water activity kinetics

A reduction in the a_w parameter occurred in all treatments for all vegetables (Figure 3a-c). Such a reduction was also observed for kiwifruit (Nowacka et al., 2017), yacon (Mendonça et al., 2017) and sweet potato (Junqueira et al., 2017c), during osmotic processes.

In osmotic processes, the a_w decreased due to the moisture reduction (related to the WL) and the SG (Corrêa et al., 2016; Lech et al., 2018). For the carrot (Figure 3a) and eggplant (Figure 3b), higher a_w reduction was obtained in the vacuum treatments, with emphasis for the PVOD 80 kPa. It was also related to the higher SG observed in this treatment (Figure 2a and 2b). For the beetroot, the OD promoted SG (Figure 2c), which reflected in a further reduction in the a_w for this treatment (Figure 3c).

It should be noted that the reduction in the a_w of the products does not make them microbiologically stable, and as a pretreatment, the osmotic process requires a further preservation technique

such as drying (Kowalski & Łechtańska, 2015; Sette et al., 2016; Şahin & Öztürk, 2016).

3.4 Mathematical modeling

The effective diffusivities were calculated according to Fick's model and Fito & Chiralt's model and their results are presented in Tables 2 and 3.

The effective diffusivities obtained according to Fick's model are shown in Table 2. For the carrot, the water effective diffusivity (D_{effw}) values ranged from 6.497×10^{-10} to 7.769×10^{-10} m²/s, and the solid effective diffusivity (D_{effs}) ranged from 1.634×10^{-10} to 4.389×10^{-10} m²/s. For this vegetable, the results showed that R^2 values were mainly above 0.95, RMSE and χ^2 values were under 6.050×10^{-2} and 4.026×10^{-3} respectively. Lower effective diffusivities were observed for the samples treated with OD at atmospheric pressures, as previously presented by Figures 1a and 2a.

For the eggplant, slight differences were observed for the D_{effw} values, which ranged from 1.214×10^{-9} to 1.222×10^{-9} m²/s, showing behavior similar that presented in Figure 1b, although lower R^2 values (< 0.87) and higher RMSE and χ^2 values were observed. The D_{effs} ranged from 2.150×10^{-10} to 3.340×10^{-10} m²/s and $R^2 > 0.92$. For the beetroot, the D_{effw} ranged from 6.145×10^{-10} to 8.362×10^{-9} m²/s, and the D_{effs} ranged from 3.246×10^{-10} to 5.729×10^{-10} m²/s with $R^2 > 0.95$, with lower RMSE and χ^2 values for the PVOD 80 treatment (5.829×10^{-2} and 3.738×10^{-3} , respectively). (Table 2).

Although the effective diffusivity varies with the food material and the experimental conditions, which makes comparison difficult in terms of exact values, the D_{eff} obtained according to Fick's model presented the analogous order of magnitude of food materials subjected to the OD (Bahmani et al., 2016; Junqueira et al., 2017c; Mendonça et al., 2017; Souraki et al., 2014).

According to Table 2, Fick's model showed lower R^2 values (even 0.640) which indicates its low acuity in portraying the experimental data. Other works have reported a lower fitting capacity for Fick's diffusive model for the osmotic processes (Barbosa et al., 2013; Corrêa et al., 2010, 2016; Zielinska et al., 2018). Simpson et al. (2015) pointed out that the complexity of the mass transfer process due to the heterogeneous nature of plant tissues can reflect a lack of fit of Fick's second law. This can be because to the initial and boundary assumptions employed for the analytical development of this diffusive model, as initial moisture content is distributed uniformly in the product, negligible shrinkage and the consideration of D_{eff} constant and homogeneous during process (Brochier et al., 2015).

Moreover, the OD is not a simple diffusional process, which means that the mass transport of water and solids is not homogeneous. There are mechanisms other than diffusion involved in this process, such as capillary flow, volumetric shrinkage, removal of trapped gases and hydrodynamic process during the VA. The dehydration is a complex process, and it can be observed that all the physical phenomena affect the development and the suitability of the models for representing

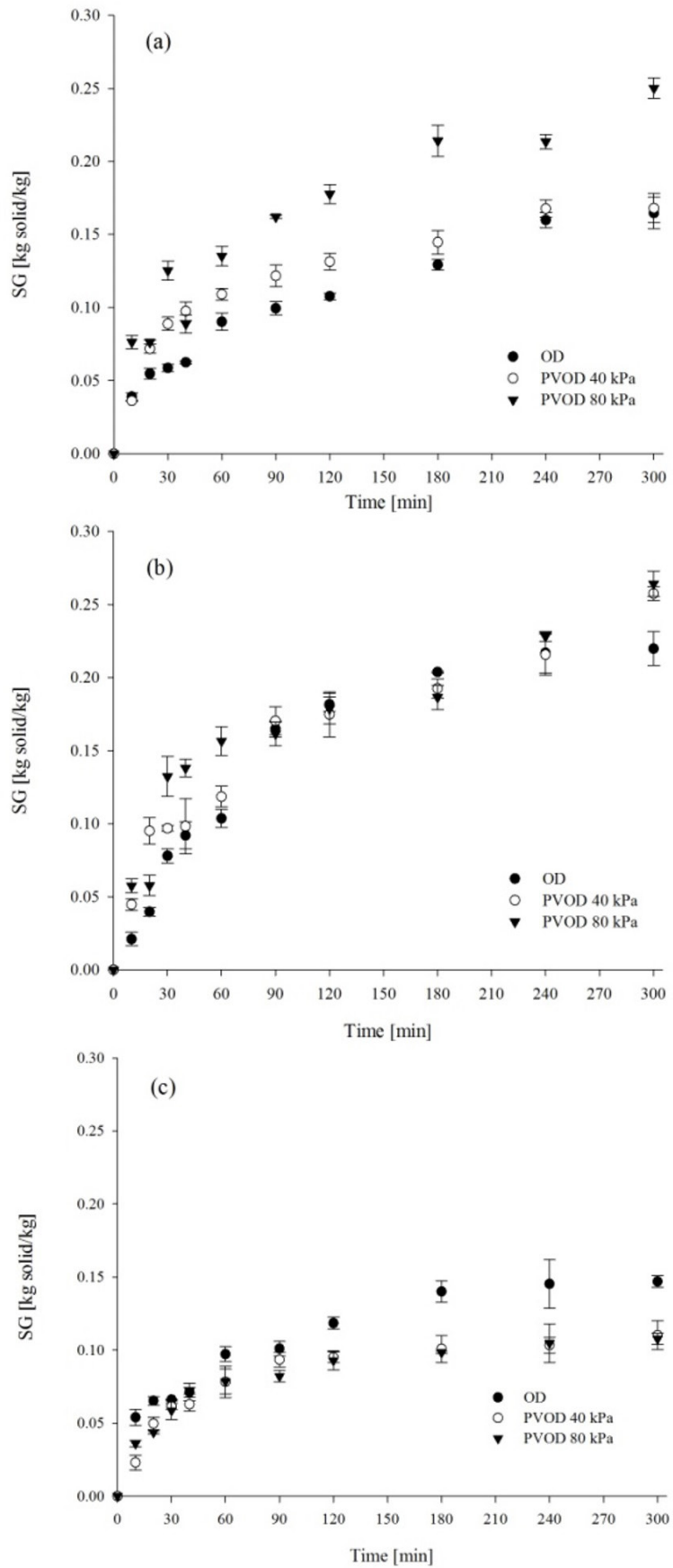


Figure 2. Solid gain (SG) kinetics of carrot (a), eggplant (b) and beetroot (c) during the OD.

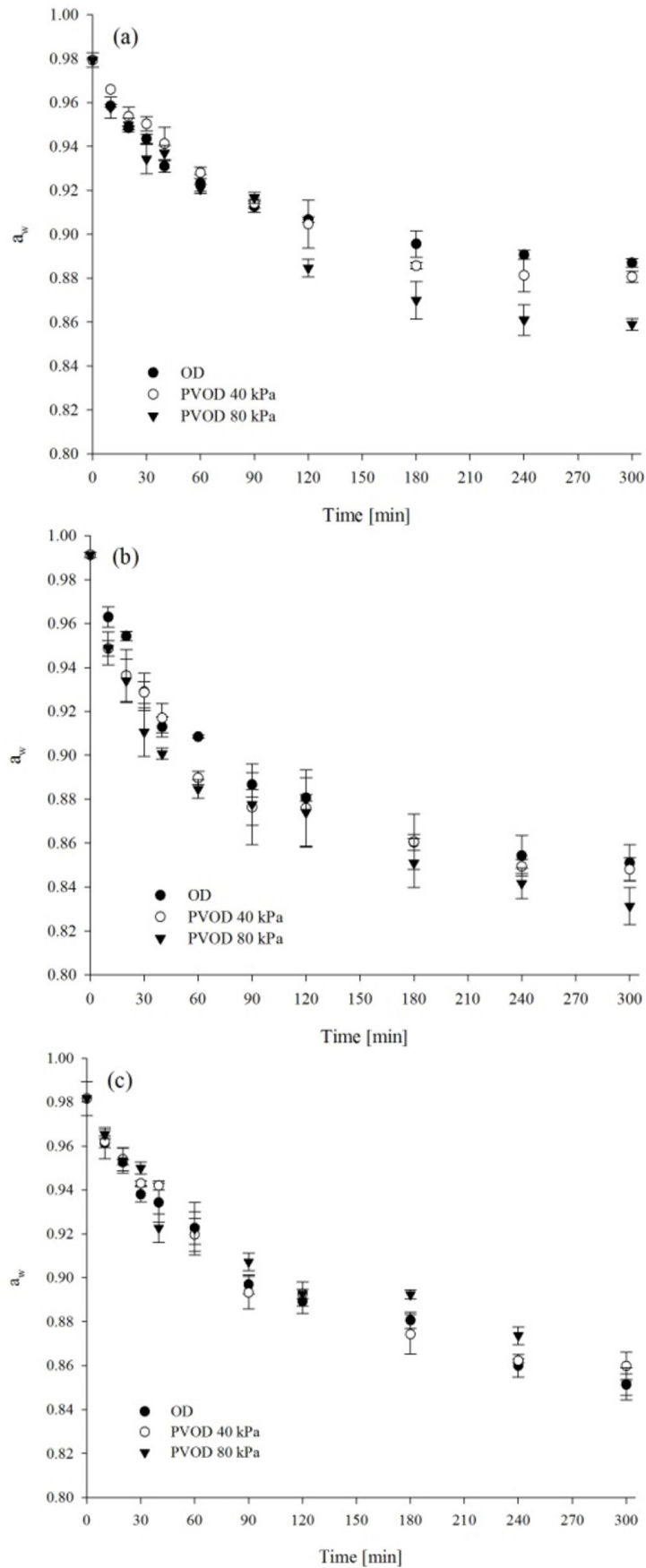


Figure 3. Water activity kinetics (a_w) of carrot (a), eggplant (b) and beetroot (c) during the OD.

Table 2. Effective diffusivity of water (D_{effw}) and solids (D_{effs}) [m^2/s] and statistical parameters obtained according to Fick's model.

Treatment	$D_{\text{effw}} \times 10^{10}$	R^2	$\text{RMSE} \times 10^2$	$\chi^2 \times 10^3$	$D_{\text{effs}} \times 10^{10}$	R^2	$\text{RMSE} \times 10^2$	$\chi^2 \times 10^3$
Carrot								
OD	6.497	0.964	5.251	3.033	1.634	0.972	4.020	1.778
PVOD 40	7.769	0.961	5.513	3.343	4.389	0.962	5.351	3.149
PVOD 80	6.721	0.954	6.050	4.026	2.348	0.951	5.861	3.779
Eggplant								
OD	12.149	0.872	9.841	10.653	2.177	0.961	5.064	2.821
PVOD 40	12.200	0.738	14.056	21.736	2.150	0.965	4.821	2.557
PVOD 80	12.223	0.640	17.942	35.410	3.340	0.926	7.569	6.303
Beetroot								
OD	7.385	0.962	5.392	3.197	3.246	0.963	5.244	3.026
PVOD 40	8.362	0.966	5.094	2.855	5.494	0.958	5.693	3.565
PVOD 80	6.145	0.956	5.829	3.738	5.729	0.961	5.493	3.319

Table 3. Effective diffusivities (D_{eff}) [m^2/s] and statistical parameters obtained according to Fito & Chiralt's model.

Treatment	$D_{\text{eff}} \times 10^{10}$	R^2	$\text{RMSE} \times 10^2$	$\chi^2 \times 10^3$
Carrot				
OD	1.904	0.974	1.682	1.850
PVOD 40	2.057	0.966	1.715	1.886
PVOD 80	1.942	0.960	1.690	1.859
Eggplant				
OD	2.131	0.928	1.730	1.903
PVOD 40	2.109	0.878	1.725	1.898
PVOD 80	2.110	0.890	1.726	1.899
Beetroot				
OD	1.824	0.973	1.664	1.831
PVOD 40	2.076	0.951	1.719	1.891
PVOD 80	1.879	0.981	1.677	1.844

the process (Azarpazhooh & Ramaswamy, 2012; Deng & Zhao, 2008; Miano & Augusto, 2018).

The differences observed in D_{eff} for all the vegetables is also related to their structure and composition. The eggplant, that presents voids within its structure, showed higher D_{effw} , probably due to the transport of water by capillarity, besides the diffusion (Miano & Augusto, 2018). This behavior was the main reason for the lack of fit of Fick's model for describing the mass transfer in this vegetable (Table 2).

In an attempt to portray the HDM that occurs during the PVOD, the Fito & Chiralt model coupled the effects of the diffusional and the hydrodynamic transport, due to the vacuum application for increasing the quality of the model to fit this process (Fito & Chiralt, 1997). Table 3 provides the D_{eff} obtained for all the vegetables subjected to this phenomenological model.

Lower diffusivities were obtained by applying this model (Table 3) when compared with those obtained by Fick's model (Table 2). They ranged from 1.904×10^{-10} to 2.057×10^{-10} m^2/s for carrot, 2.109×10^{-10} to 2.131×10^{-10} m^2/s for eggplant and 1.824×10^{-10} to 2.076×10^{-10} m^2/s for beetroot. According to Table 3, in general, higher R^2 and lower RMSE and χ^2 values were observed.

As afore mentioned, during the VA period, the gases and free internal liquid flow out and the molecular diffusion can

be enhanced. With the atmospheric pressure restoration, the matrix pore volume reduces and expulsion of occluded fluids occurs. Fito & Chiralt's model considers the diffusion and HDM during PVOD, achieving a better fit to the experimental data (Ahmed et al., 2016; Fito et al., 2001).

4 Conclusion

The osmotic dehydration of different material structures (carrot, eggplant and beetroot) was studied, and the effect of VA was evaluated. In general, vegetables with a porous structure (carrot and eggplant) were more sensitive to the pressure reduction, with notable intensification of mass transfer. The effective diffusivities were obtained according to Fick's model and Fito & Chiralt's model. The same magnitude order of this parameter was observed for all the vegetables and treatments. Fick's model presented a lack of fit to the experimental data, mainly for WL results with R^2 ranging from 0.64 to 0.96.

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