JOÃO RENATO DE JESUS JUNQUEIRA

THE INFLUENCE OF VACUUM APPLICATION AND VEGETABLE STRUCTURE ON THE OSMOTIC DEHYDRATION

LAVRAS – MG

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Tese apresentada à Universidade Federal de Lavras, como parte das exigências do Programa de Pós-Graduação em Ciência dos Alimentos, área de concentração em Ciência dos Alimentos, para a obtenção do título de Doutor.

Orientador

Dr. Jefferson Luiz Gomes Corrêa

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GENERAL ABSTRACT

Many studies have been conducted to better understand the mechanisms of mass transfer during the osmotic dehydration of food, and to evaluate the quality parameters of the osmodehydrated products and the mathematical modeling of the phenomena involved in this process. Osmotic dehydration is a pre-treatment that extends the shelf life of foods and aids in the development of products with aggregated value and differentiated characteristics. The use of vacuum during the first minutes of the osmotic dehydration (pulsed vacuum osmotic dehydration) enhances the transfer of moisture from product to the osmotic solution, and the transfer of solute to the food matrix. The material structure exercises great influence over the effect of osmodehydration on the characteristics of the final product and on the mass transfer kinetics. The present work was elaborated with the objective of studying the behavior of different vegetable slices (eggplant, carrot and beetroot) during osmotic processes, using different vacuum pressures and osmotic solutions. For all products, quality characteristics and dehydration kinetics, were evaluated, and mathematical model adjustments were conducted. The effect of the application of vacuum over optical, chemical, nutritional and structural parameters of foods was confirmed. The vegetable with greater porosity (eggplant and carrot) were more sensitive to vacuum. The mass transfer parameters (water loss, solid gain and consequent water activity reduction of the final product) were influenced by the type of agent employed as osmotic substance, as was the application of reduced pressure at the beginning of the process. The partial replacement of sodium chloride by other salts as osmotic agents was promising, with no significant changes in the characteristics of the osmodehydrated product, coupled with a decrease in the concentration of sodium chloride incorporated. According to the dehydration kinetics curves, the effective diffusivities of water and solutes were calculated by employing phenomenological mathematical models. In some conditions, a lack of adjustment of the above-mentioned models was observed. In conclusion, vacuum pulse osmotic dehydration affects the final characteristics of the product, and behavior during dehydration.

Keywords: Pulsed vacuum osmotic dehydration, Alternative osmotic agents, Mathematical models, Drying.

RESUMO GERAL

Muitos estudos têm sido realizados visando o melhor entendimento dos mecanismos de transferência de massa durante a desidratação osmótica de alimentos, bem como avaliação dos parâmetros de qualidade dos produtos desidratados osmoticamente e a modelagem matemática dos fenômenos envolvidos durante o processo. Consistindo de um pré-tratamento, a desidratação osmótica auxilia na extensão da vida útil de alimentos, além de proporcionar o desenvolvimentos de produtos com valor agregado e características diferenciadas. A utilização do vácuo durante os primeiros minutos de desidratação osmótica (desidratação osmótica com pulso de vácuo) intensifica a transferência de umidade do produto para a solução osmótica, bem como a transferência de soluto da solução para a matriz do alimento. A estrutura do material exerce grande influência sobre o efeito da desidratação osmótica nas características do produto final e na cinética de transferência de massa. O presente trabalho foi elaborado com o objetivo de estudar o comportamento de fatias de diferentes vegetais (berinjela, cenoura e beterraba) em processos osmóticos utilizando diferentes pressões de vácuo e soluções osmóticas. Para todos os produtos, foram avaliadas características de qualidade, cinética de desidratação e ajustes por modelos matemáticos. Foi confirmado o efeito da aplicação de vácuo nos parâmetros óticos, químicos, nutricionais e estruturais dos alimentos. Vegetais com maior porosidade (berinjela e cenoura) foram mais sensíveis à aplicação do vácuo. Os parâmetros de transferência de massa (perda de água, ganho de sólidos e consequente redução na atividade de água do produto final) foram influenciados pelo tipo de agente empregado como substância osmótica, bem como a aplicação de pressão reduzida no início do processo. A substituição parcial do cloreto de sódio por outros sais, como agentes osmóticos, mostrou-se promissora, sem alterações significativas nas características do produto osmodesidratado, acompanhado de uma redução na concentração de cloreto de sódio incorporado. Através das curvas de cinética de desidratação osmótica, as difusividades efetivas de água e solutos foram calculados de acordo com modelos matemáticos fenomenológicos. Em algumas condições, uma falta de ajuste de tais modelos foi observada. Concluiu-se que a desidratação osmótica com pulso de vácuo interfere nas características finais do produto, bem como na avaliação do comportamento durante a desidratação.

Palavras-chave: Desidratação osmótica com pulso de vácuo, agentes osmóticos alternativos, modelagem matemática, secagem.

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LIST OF ACRONYMS

Abs	Absorbance
a_w	Water activity
BC	Betalain content
BCC	Betacyanin content
BI	Browning index
BXC	Betaxanthins content
CAPES	Coordination for the Improvement of Higher Education Personnel
CC	Carotenoid content
CNPq	National Council for Scientific and Technological Development
d.b	Dry basis
DF	Dilution factor
FAPEMIG	State of Minas Gerais Research Foundation
FLP	Food liquid phase
IMF	Intermediate-moisture food
HDM	Hydrodynamic mechanism
HPLC	High performance liquid efficiency chromatography
MC	Moisture content
MW	Molecular weight
OD	Osmotic dehydration
PVOD	Pulsed vacuum osmotic dehydration
R^2	Correlation coefficient
RMSE	Root mean square error
SE	Estimative standard error
SEM	Scanning electron microscopy
SG	Solid gain
SD	Standard deviation

VA	Vacuum application
VP	Vacuum pressure
WL	Water loss
w	weight

LIST OF SYMBOLS

a^*	Redness
A_0	Initial contact area [m^2]
A_t	Contact area [m^2]
b^*	Yellowness
C_{ab}	Chroma
$CaCl_2$	Calcium chloride
D_{eff}	Effective diffusivity [m^2/s]
D_{effs}	Effective diffusivity of solid [m^2/s]
D_{effw}	Effective diffusivity of water [m^2/s]
ΔE	Total color difference
E	Elasticity modulus [kPa]
$E^{1\%}$	Molar extinction coefficient
F_t	Compression force [N]
H_0	Initial height [m]
H_t	Height at time t [m]
k	Adjustment coefficient
k_1	Peleg's parameter
K_1	Modified Peleg's parameter
KCl	Potassium chloride
L	Characteristic length [m]
L^*	Lightness
mbar	milibar
M_0	Initial sample weight [kg]
M_t	Sample weight at time t [kg]
NaCl	Sodium chloride

P	Sample weight [kg]
t	Time [s]
V	Total extract volume
V_0	Initial sample volume [m^3]
V_f	Final sample volume [m^3]
X_0	Initial water or solid content [kg water or solid/ 100 kg sample]
X_{eq}	Water or solid content at equilibrium [kg water or solid/ 100 kg sample]
X_{w0}	Initial water content [kg water/ 100 kg sample]
X_{st}	Soluble solid content at time t [kg solid/ 100 kg sample]
X_t	Water or solid content at time t [kg water or solid/ 100 kg sample]
X_{wt}	Water content at time t [kg water/ 100 kg sample]
W	Dimensionless water or solid content
W_s	Dimensionless solid content
W_w	Dimensionless water content
Y	Reduced drive force
y_{eq}^{ss}	Mass fraction of soluble solids in the osmotic solution
z	General coordinate [m]
z_{eq}^{ss}	Mass fraction of soluble solids in the food
β	Dimensionless volume
σ	Maximum stress [kPa]

SUMMARY

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THIRD SECTION

FIRST SECTION

1 GENERAL INTRODUCTION

Osmotic dehydration (OD) is a technique that provides partial water removal from food products, with low energy consumption, and conducted at room or moderate temperatures. It is based on the immersion of pieces of fresh fruits or vegetables in a hypertonic solution. The process involves simultaneous counter-current water diffusion from the food to the solution and solute diffusion into the food, under the influence of an osmotic pressure gradient. It is considered a pretreatment to many processes and preserves physical, chemical and sensorial characteristics of food with few changes to its integrity (AHMED; QAZI; JAMAL, 2016; CORRÊA; ERNESTO; MENDONÇA, 2016).

A mass transfer rate improvement can be achieved with the vacuum application in the beginning of the OD, in a process denominated pulsed vacuum osmotic dehydration (PVOD). The reduction in pressure releases the occluded gases in the pores of the fruit and vegetables, due to the action of a hydrodynamic mechanism (HDM) enhanced by pressure difference, increasing the surface area for mass transfer. Different material structures present different responses to vacuum application (FITO, 1994; OLIVEIRA et al., 2016).

This work aimed to evaluate the relation between the vacuum application during the osmotic dehydration of three different food structures (eggplant, carrot and beetroot), regarding the quality parameters of the osmodehydrated vegetables and the mathematical modeling of the processes. The effects of some alternative salts as osmotic agents were also studied as replacement to sodium chloride.

2 THEORETICAL REFERENCE

2.1 Osmotic dehydration

Osmotic dehydration (OD) is a food preservation process used for obtaining final products with intermediate moisture (in vegetables and fruits up to 50 % of the initial content) (ASSIS; MORAIS; MORAIS, 2016). It consists of a treatment done prior other preservation techniques (such as drying), conducted at mild temperatures, improving the overall quality of food characteristics by retaining the vitamins, minerals, color, flavor and taste of the raw material (NIETO et al., 2013).

During OD, the food is immersed in a hypertonic aqueous solution. The difference in the osmotic pressure between the food and the surrounded solution results in a simultaneous countercurrent flow through the cell membranes: the diffusion of water from the food to the osmotic solution (dehydration) and the incorporation of solutes from the solution into the food (impregnation) (RAMYA; JAIN, 2017). Qualitatively, the leaching of water soluble constituents from the food to the osmotic media (carbohydrates, minerals, organic acids and vitamins) may affect the nutritional and sensory quality of the osmodehydrated product (UTKUCAN; KEMAL, 2016).

The vegetable tissues are composed by cells that act as a water source, delimited by the cellular membrane. The water presents a relevant role in structure preservations and in the functional integrity of the tissues. When the cellular tissue is exposed to concentrated solution, the solids migrate to the extracellular volume and the chemical potential gradient induces the impregnation of solutes and the removal of the water through the cellular membranes. The water leaves the intracellular volume, reaching the osmotic solution before transferring this extracellular volume. During OD, the volume of water flowing out of the system is higher than the solid uptake volume (NIETO et al., 2013; PORCIUNCULA et al., 2013).

This process assists in maintaining the physical, chemical and nutritional properties of the raw material, and is an energy efficient method, as water does not change phases (KAUSHAL; SHARMA, 2016).

This technique has been employed as a pretreatment for perishable food, such as bananas (PORCIUNCULA et al., 2013), mangoes (LIN; LUO; CHEN, 2016), yacon (MENDONÇA et al., 2016), tomatoes (DEROSSI et al., 2015), carrots (SINGH et al., 2010), apples (WIKTOR et al., 2014), eggplants (BAHMANI et al., 2016), melons (BARBOSA JÚNIOR; CORDEIRO; HUBINGER, 2013) and beef cuts (BAMPI et al., 2016). In all the presented studies, the preservation of the structure material and the characteristics were related to the reduction in the period of the drying processes and to economy saving, as well as the development of products with aggregated value.

The effects of OD over different material structures and compositions depend on the process variables and characteristics of the product. Some aspects of osmotically dehydrated fruits have been reviewed by various studies concerning osmotic substance and their concentration (HERMAN-LARA et al., 2013), temperature (RUIZ-LÓPEZ et al., 2011), osmotic agents (MENDONÇA et al., 2017), immersion period (GANJLOO; BIMAKR, 2015) and agitation (AMAMI et al., 2014). The improvement of mass transfer rates and of the final osmodehydrated properties can be reached using ultrasound (AMAMI et al., 2017), ohmic heating (MORENO et al., 2016), pulsed electric fields (WIKTOR et al., 2014) and vacuum (OLIVEIRA et al., 2016).

The type of osmotic agent also affects the quality of the final product. Substances used for osmotic dehydration are solutions of sugars (sucrose, fructose, glucose), salts (sodium chloride, calcium chloride), polyols (sorbitol, glycerol, xylitol), or other substances acceptable for consumption, which can produce high osmotic pressure, allowing the reduction of water activity in the dehydrated material (CIURZYŃSKA et al., 2016).

The most employed osmotic substances are sucrose (mainly for fruits) and sodium chloride (mainly for vegetables, meats and fishery products), since they are cheap, present considerable solubility and produce moisture and water activity reduction in the products (MERCALI et al., 2011).

The osmotic agents can be used with water (binary solutions) or in combination with others (ternary or quaternary solutions). When the osmotic substances are blended, a synergic effect can be verified between them. This allows the use of high solute concentration without reaching the saturation limit of the solutions and increasing the water loss (dehydration) when compared to binary solutions (HEREDIA et al., 2012).

2.2 Pulsed vacuum osmotic dehydration

Vacuum application for a short period, at the beginning of the osmotic process, is called pulsed vacuum osmotic dehydration (PVOD). This reduced pressure induces the removal of native gases and liquids from capillary pores and leads to gas expansion. As the atmospheric pressure is restored, voids are instated in the pores. This suctions the osmotic solution inside the pores, with deformation and relaxation phenomenon taking place, which enables the modification of pore dimension, facilitating water release and solute diffusion (AHMED; QAZI; JAMAL, 2016; FITO, 1994).

This process enhances mass transfer rates by osmotic solution penetration into the tissue pores through the hydrodynamic mechanism (HDM). The presented mechanism is controlled by the presence of internal fluids in the open pores (in a porous structure product), which is expanded and compressed with pressure changes. This encourages the exchange between pore liquid/gas and the outside liquid (OLIVEIRA et al., 2016; VIANA; CORRÊA; JUSTUS, 2014). In Figure 1, it is possible to verify a scheme of mass transfer during the PVOD process.

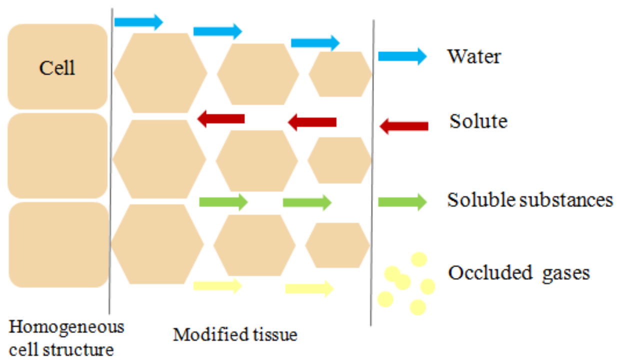

Figure 1 - Mass transfer during PVOD process
Source: Author (2017).

The PVOD has been employed for guavas (CORRÊA et al., 2010), figs (UTKUCAN; KEMAL, 2016), grapefruit (MORAGA et al., 2009), plums (FANTE et al., 2011), tomatoes (CORRÊA; ERNESTO; MENDONÇA, 2016) and yacon (OLIVEIRA et al., 2016). All these authors reported an increase in water loss and solid uptake rates, as well as maintenance of the product structure. Also, using vacuum pressure in OD, accelerates processing times and allows a reduction in energy during process (UTKUCAN; KEMAL, 2016).

2.3 Material structure

The plant tissue is a complex and heterogeneous system considered a three-dimensional solid matrix with liquid phase (generally water). The particle structure of the material and mechanical characteristics of the elements define its volume and shape. When water is removed from the food, a pressure imbalance occurs between the inner and external pressure of the product, creating a stress that leads to a material collapse and size alterations (NAHIMANA et al., 2011).

Each material presents a singular composition and structure, and the effect of the water removal during the OD is particular for each product. Some properties, such as porosity, pore size, bulk density and pore distribution, influence the final characteristics of the osmodehydrated products. Porosity is especially important in the reconstitution of the dehydrated products, effectively controlling the rewetting speed, as well as taste and appearance. Information on pore formation and their characteristics in foods during processing is also essential in estimating transport properties (KOÇ; EREN; KAYMAK ERTEKIN, 2008; NAHIMANA et al., 2011).

2.4 Mathematical modeling

Mathematical modeling is an important tool for evaluating dehydration behavior. Mathematical models can be divided into two main groups: empirical and phenomenological. The empirical models are obtained from mathematical correlations and adjustments of the experimental data, with no physical meaning of the parameters. The phenomenological models must portray the physical mechanism of the processes, and consider the elementary steps of mass transfer (diffusion and convection), with parameters often presenting physical meaning. Generally, the mathematical modeling represents the process trend, even under different experimental conditions, allowing the prediction of food product temperature, changes in moisture content and structural changes (COUTINHO et al., 2010; JUNQUEIRA; CORRÊA; MENDONÇA, 2017; PARTHASARATHI; ANANDHARAMAKRISHNAN, 2014).

The usual mathematical treatment for describing the mass transport, which presents the molecular diffusion as the main step, is related with the second law of Fick (CRANK, 1975). This application and the development of this law is restricted to a few initial and boundary conditions and to the supposition that the dehydration is controlled by the internal diffusion in a homogeneous and isotropic solid. However, due to the complexity of the plant tissue and the structural changes after OD, some models may not fit (PORCIUNCULA et al., 2013; SIMPSON et al., 2015).

Many studies have been conducted to improve the knowledge regarding the mass transfer phenomena during OD for modeling the relevant mechanisms of the process. Furthermore, the mechanisms of mass transport are complex and not completely understood (PARTHASARATHI; ANANDHARAMAKRISHNAN, 2014).

Dehydration models have been lead by considering diffusion as the majority mechanism for describing the whole mass transfer through food and osmotic media. Moreover, some realistic models have been proposed for osmotic processes, as presented by Fito & Chiralt's model (FITO; CHIRALT, 1997), which considers a coupling of diffusional and hydrodynamics effects during OD, and Barbosa Júnior' s model (BARBOSA JÚNIOR; CORDEIRO; HUBINGER, 2013), which considers the effective diffusivity variation during the osmotic process and calculates this parameter for each period, which improves the overall estimates of the response and adjustment of the model.

Some empirical models obtained from mathematical correlations of the experimental data have showed good fits, and are indicated as a simple tool for describing the osmotic processes. Between these models, Page's equation, Midilli and Kuçuk's equation and Weibull' model have been employed for dynamics processes, with suitable agreements (KUÇUK et al., 2014).

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SECOND SECTION

ARTICLE 1

INFLUENCE OF SODIUM REPLACEMENT AND VACUUM PULSE ON THE OSMOTIC DEHYDRATION OF EGGPLANT SLICES

Running title: Pulsed vacuum osmotic dehydration of eggplant.

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Abstract - Partial replacement of sodium chloride by potassium and calcium chlorides has been proposed as a strategy for reducing the sodium content of osmodehydrated eggplant. The influence of sodium substitutes and vacuum application (VA) on mass transfer parameters and chemical, mechanical and optical properties were investigated. Kinetics of water loss, solid gain and water activity were performed and fitted by the model of Barbosa Junior et al. This model was satisfactorily adjusted, mainly for the osmotic dehydration at atmosphere pressure. VA and the calcium increased the ascorbic acid retention in 29.33 and 85.06%, respectively. VA increased water loss up to 53% and ions incorporation, especially that of potassium (648%). The VA caused a higher total color difference, maximum stress and elasticity as compared to the osmotic dehydration at atmospheric pressure.

Keywords - Pulsed vacuum osmotic dehydration, Alternative osmotic solutes, Mineral content, *Solanum melongena* L.

1. INTRODUCTION

Eggplant (*Solanum melongena* L.) is an important crop of subtropical and tropical regions. This fruit presents low caloric value and is a good source of minerals, vitamins and anthocyanins. Eggplant has also received great attention due to its high antioxidant activity and medicinal properties, and its consumption is recommended for diabetic patients (Hussain, Omeera, Suradkar, & Dar, 2014; Zaro, Ortiz, Keunchkarian, & Chaves, 2015). The eggplant consists mostly of water, which limits its shelf life. Therefore, techniques that enhance its stability are required (Bahmani, Jafari, Shahidi, & Dehnad, 2015).

Osmotic dehydration (OD) is a water removal treatment that preserves physical, chemical, nutritional, sensorial and functional properties of food with limited changes to the food's integrity. This technique involves the immersion of the product in a hypertonic aqueous solution leading to a loss of water through the cell membranes of the product and subsequent flow along the inter-cellular space before diffusing into the solution. It also allows the diffusion of solutes from the osmotic solution into the food tissue (Junqueira, Corrêa, & Mendonça, 2016; Kaushal & Sharma, 2016; Mendonça, Corrêa, Junqueira, Pereira, & Vilela, 2016; Torreggiani, 1993).

OD is usually employed as a pretreatment before drying. A reduction of the total drying time was observed by Aydogdu, Sumnu, & Sahin (2015) and Osidacz & Ambrosio-Ugri (2013) when using sodium chloride as an osmotic agent during the pretreatment prior to further drying processes. Ganjloo & Bimakr (2015) evaluated the OD of eggplant using sucrose at different concentrations as an osmotic agent and found that the best condition for mass transfer was 30% (w/w) sucrose.

An increase in mass transfer rates can be achieved by the application of vacuum in the first minutes of the osmotic dehydration in a process called pulsed vacuum osmotic dehydration (PVOD). The reduction in pressure causes an expansion of the internal gases in the pores of the fruit and vegetables, expelling them via hydrodynamic mechanisms (HDM) enhanced by the pressure difference, increasing the surface area available for mass transfer (Fito, 1994; Junqueira, Corrêa, & Ernesto, 2016; Oliveira, Corrêa, de Angelis Pereira, Ramos, & Vilela, 2016; Viana, Corrêa, & Justus, 2014).

The osmotic agents employed during OD are responsible for the sensory properties and nutritional value of osmodehydrated vegetables. Sodium chloride (NaCl) is an osmotic agent that presents higher osmotic pressure than sugars because of its ionic characteristics. Consequently, transfers between the solution and the food are favored, and the water activity of the product is significantly reduced (Hamdan, Sharif, Derwish, Al-Aibi, & Altaee, 2015; Terefe, Janakievski, Glagovskaia, Silva, Horne, & Stockmann, 2016).

Since OD is a counter-current diffusional process, the incorporation of sodium can be considered a disadvantage, due to its effects on sensory quality and undesirable health impacts. Excessive sodium intake is linked to hypertension and cardiovascular diseases (Rodrigues, Souza, Mendes, Nunes, & Pinheiro, 2016; Tamm, Bolumar, Bajovic, & Toepfl, 2016).

Therefore, the substitution of NaCl by other chloride salts in processed foods could offer diverse and healthier products and improve consumer acceptance. The most common sodium chloride substitutes in processed food are potassium chloride (KCl), calcium chloride ($CaCl_2$) and magnesium chloride ($MgCl_2$). These salts present promising perspectives since their addition did not significantly affect the physicochemical and sensorial characteristics of the final products compared to NaCl (Bautista-Gallego, Rantsiou, Garrido-Fernández, Cocolin, & Arroyo-López, 2013).

A diet based in low sodium content and high potassium and calcium uptake is suggested to reduce high blood pressure problems and prevent against osteoporosis (Bautista-Gallego, Rantsiou, Garrido-Fernández, Cocolin, & Arroyo-López, 2013).

In processed food, the partial substitution of NaCl by other cationic ions, is usually related for fishery (Faralizadeh, Zakipour, & Khanipour, 2016), meat (Fellendorf, O'Sullivan, & Kerry, 2016), dairy (Khetra, Kanawjia, & Puri, 2016) and bakery (Sayar, Erdoğdu, Eydemir, & Nayman, 2016) products. However, no studies regarding the combination of different salts as sodium substitutes during the osmotic dehydration of fruits and vegetables were published. Moreover, studies about the effects of the combination of different salts as osmotic agents coupled with vacuum application were not found.

The aim of this work was to evaluate the influence of sodium chloride replacers as osmotic solutes (potassium and calcium chlorides) and the vacuum application on mass transfer parameters (water loss, solid gain and water activity reduction), chemical (minerals), mechanical (maximum stress and elasticity), optical (color changes and browning index) and functional (ascorbic acid) properties of osmodehydrated eggplant slices.

2. MATERIAL AND METHODS

2.1. Materials

The eggplant fruits (*Solanum melongena* L.) used in the osmotic processes were purchased in the local market (Lavras, MG, Brazil) and stored in a refrigerator at 8 ± 1 °C for 48 hours prior to the experiments. The fruits were selected based on their size, peel color, appearance and firmness. Fresh eggplant was characterized with respect to the chemical composition (moisture content, crude fat, protein, ash, crude fiber and carbohydrate) according to the methodology proposed by the AOAC (2007) and is presented (on wet basis) as follows:

moisture content 92.24 ± 0.08 kg 100 kg^{-1}; crude fat 0.13 ± 0.01 kg 100 kg^{-1}; protein 1.19 ± 0.02 kg 100 kg^{-1}; ash 0.56 ± 0.01 kg 100 kg^{-1}; crude fiber 1.00 ± 0.01 kg 100 kg^{-1} and carbohydrate 4.86 ± 0.04 kg 100 kg^{-1}. All analyses were performed in triplicate.

Analyses of water activity (Aqualab, 3-TE model, Decagon Devices Inc., Pullman, WA, USA), total soluble solids (Tecnal, AR-200 model, São Paulo, Brazil), pH (Digimed, DMpH-2 model, São Paulo, Brazil) and color parameters of the peel and internal parenchyma (Minolta colorimeter, CR 400 model, Japan) were also performed to characterize the fresh material. The fresh eggplant presented an a_w of 0.992 ± 0.001. The total soluble solids were 3.14 ± 0.46 kg solute 100 kg^{-1} fruit, and the pH was 5.09 ± 0.02. The color parameters observed for peel were L^*= 28.495 ± 1.643; a^*= 4.290 ± 0.564 and b^*= -2.090 ± 0.336, and those for internal parenchyma were L^*= 86.674 ± 0.914; a^*= -5.821 ± 0.677 and b^*= 21.340 ± 2.409.

2.2. Sample preparation and treatments

The fruits were washed in tap water and cut into disk-shaped samples (0.40 ± 0.03 cm thickness and 5.00 ± 0.50 cm diameter). The peel was maintained during the sample preparation in order to avoid the radial diffusion of water and solutes during the osmotic process. The slices were immersed for 3 min in a solution of 1% (w/w) ascorbic acid and 2% (w/w) citric acid to reduce enzymatic browning (Moreno, Simpson, Pizarro, Pavez, Dorvil, Petzold, & Bugueño, 2013).

The osmotic solutions were prepared with distilled water and the osmotic agents. The concentration of all osmotic solutions was 0.1 kg of solid kg^{-1} solution. The formulation and the a_w of each solution are presented in Table 1.

Table 1 - Composition and water activity of the osmotic solutions in osmotic dehydration (OD) and pulsed vacuum osmotic dehydration (PVOD)

	Treatments	NaCl (%)	KCl (%)	$CaCl_2$ (%)	$a_w \pm SD$
OD	1	10.0	-	-	0.945 ± 0.001
OD	2	7.5	2.5	-	0.949 ± 0.001
OD	3	7.0	2.5	0.5	0.949 ± 0.002
OD	4	5.0	4.0	1.0	0.953 ± 0.002
PVOD	5	10.0	-	-	0.945 ± 0.001
PVOD	6	7.5	2.5	-	0.949 ± 0.001
PVOD	7	7.0	2.5	0.5	0.949 ± 0.002
PVOD	8	5.0	4.0	1.0	0.953 ± 0.002

2.3. Osmotic dehydration processes

Osmotic experiments were performed at atmospheric pressure (OD) and under vacuum (PVOD). The experiments were conducted in a temperature-controlled oven (Solab SL104/40, Piracicaba, Brazil) coupled with a vacuum pump. For the PVOD treatments, a vacuum pressure of 145 mbar was applied to the system during the first 10 minutes of process, after which the local atmospheric pressure of 755 mbar (Lavras, MG, Brazil) was restored.

The temperature was set at 30.0 ± 1 °C, and the ratio of solution to fruit was 1:10 (w/w) to prevent dilution of the osmotic solution during the experiments (Corrêa, Dev, Gariepy, & Raghavan, 2011). At preset times (10; 20; 30; 40; 60; 90; 120; 180; 240; 300 and 360 min), the samples were removed from solution. Each removed sample was then immersed in a bath of cold distilled water for 10 seconds to stop the osmotic process, and the surface of the sample was gently wiped with absorbent paper to remove excess solution. The sample was weighed and submitted to moisture content determination according to AOAC method 934.06 (AOAC, 2007) (Junqueira, Corrêa, & Mendonça, 2016). All the experiments were performed in four replicates.

The Equations 1 and 2 were used for obtaining the water loss (WL) and the solid gain (SG), respectively.

$$WL\ (\%) = \frac{(M_0 X_{w0}) - (M_t X_{wt})}{M_0} x100 \tag{1}$$

$$SG\ (\%) = \frac{(M_t X_{st} - M_0 (1 - X_{w0}))}{M_0} x100 \tag{2}$$

where M_0 is the weight of the sample at time t= 0 s [kg], X_{w0} is the initial water content [kg water 100 /kg sample], M_t is the weight of the sample at time t [kg], X_{wt} is the water content [kg water /100 kg sample] at time t and X_{st} is the soluble solid content [kg solid /100 kg sample] at time t.

2.4. Mathematical model

Mass transfer during osmotic treatments has been modeled using Fick's second law of diffusion. The experimental data were fit to the unidirectional diffusion model (Crank, 1975) to estimate the effective diffusivity of the water and solutes transferred during the osmotic dehydration. Fick's second law for unidirectional unsteady state diffusion is given by Equation 3:

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

where X_t is water or solid content [kg water or solid /100 kg sample] at time t, D_{eff} is the effective diffusivity [m^2 /s], z is a general coordinate [m] and t denotes the time [s].

Due to the shape and dimensions of the slices, it is possible to assume that there was not diffusion in the radial or angular directions (the peel promoted a high resistance to transport phenomena), and the sample may be considered as an infinite slab. For this geometry, and considering the experiment as a brief process and the internal transfer of moisture unidirectional, Equation 4 was used for calculating the effective diffusivity:

$$W_{wor\,s} = \left(\frac{8}{\pi^2} \sum_{i=0}^{\infty} \frac{1}{(2i+1)^2} \exp\left(-(2i+1)^2 \pi^2 D_{eff} \frac{t}{4L^2} \right) \right) \tag{4}$$

where $W_{w\ or\ s}$ is the dimensionless water or solid content and L is the characteristic length (half of 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, $\left.\frac{\partial X_t}{\partial t}\right|_{z=0} = 0$, and the equilibrium content at the surface, $X_{(L,t)} = X_{eq}$ (Corrêa, Rasia, Garcia-Perez, Mulet, Junqueira, & Cárcel, 2016).

The dimensionless water or solid content is given by Equation 5:

$$W_{wor\,s} = \frac{X_t - X_{eq}}{X_0 - X_{eq}} \tag{5}$$

where X_{eq} is the water or solid content at equilibrium [kg water or solid /100 kg sample] and X_0 is the initial water or solid content [kg water or solid /100 kg sample].

The data related to the mass transfer parameters could be fitted using the equation presented by Peleg (Palou, Lopez-Malo, Argaiz, & Welti, 1994) and modified by Barbosa Junior, Mancini, & Hubinger (2013) (Equation 6):

$$X_t = \frac{t}{K_1 + K_2 t} \tag{6}$$

where K_1 and K_2 are the modified Peleg's equation parameters (Barbosa Junior, Mancini, & Hubinger, 2013).

Barbosa Junior, Mancini, & Hubinger (2013) developed a simple mathematical model which considers the effective diffusivity variation during the osmotic process and calculates an effective diffusion value for each time point, which improves the overall estimates of the response and the adjustment of the model, as demonstrated in Equation 7 (Corrêa, Viana, Mendonça, & Justus, 2016; Junqueira, Corrêa, &Mendonça, 2016).

$$\overline{D_{eff}} = \frac{\int_0^t D_{eff}(t)dt}{\int_0^t dt} \qquad (7)$$

where $\overline{D_{eff}}$ is the average effective diffusivity [m^2 /s] supposed to represent the D_{eff} at each pair of time-water loss or time-solid gain.

Peleg's equation parameters and the effective diffusivities were obtained using the nonlinear estimation (Quasi-Newton method) in the software Statistica 8.0 (StatSoftInc., Tulsa, USA). The fitting of the model was performed by root mean square error (RMSE) estimation and calculation of the correlation coefficient (R^2).

2.5. Quality analyses

The time of the experimental osmotic dehydration was chosen as 120 minutes, because, according to the classic literature (Torreggiani, 1993), after this time, the osmotic process tends to equilibrium. During the first two hours, the mass transfer is intensified, as demonstrated by Porciuncula, Zotarelli, Carciofi, & Laurindo (2013) and by Bahmani, Jafari, Shahidi, & Dehnad (2015) during the OD of bananas and eggplants, respectively. Therefore, the following analyses were performed after this period.

2.5.1. Mechanical properties

The rheological parameters of fresh and osmodehydrated eggplant were measured by uniaxial compression tests using a texturometer (Stable Micro

Systems, TA-X2T, Surrey, England). The sample dimensions were standardized with the aid of a cylindrical mold. The diameter and the thickness of the disk samples were 13.80 ± 0.01 and 4.00 ± 0.03 mm, respectively. The texture of eggplant slices was obtained from load and strain curves recorded during the compression of samples to 70% of their initial height using two horizontal parallel plates (70 mm length) with the sample placed on the center of the lower plate. The crosshead speed was 60 mm /min. The maximum stress (σ) was calculated according to Equation 8 (Oliveira, Mendonça, Corrêa, Junqueira, & Justus, 2016).

$$\sigma = \frac{F_t}{A_t} \qquad (8)$$

where F_t and A_t represent the compression force [N] and contact area of the sample with the probe [m^2] at each time t, respectively.

The contact area at each time t during the compression test was obtained from the area of the eggplant sample measured before compression (A_0), the initial height (H_0) and the height at each time t (H_t), assuming constancy of sample volume during compression, according to Equation 9 (Ferrari, Arballo, Mascheroni, & Hubinger, 2011; Mayor, Moreira, Chenlo, & Sereno, 2007)

$$A_t = \frac{A_0 H_0}{H_t} \qquad (9)$$

The elasticity modulus was calculated as the initial slope of the stress/strain curve as described by Thybo & Martens (1999).

2.5.2. Color parameters

The color parameters of fresh and osmodehydrated eggplant slices were measured using a colorimeter (Minolta, Model CR-400, Osaka, Japan). The CIELAB coordinate system (defined by CIE-Commission Internationale d'Eclairage) L^*, a^* and b^* with D65 as illuminant was used. The total color difference (ΔE) was calculated according to Equation 10 based on the color of the osmodehydrated samples and fresh eggplant parenchyma as a reference. The color parameters were used to calculate the chroma (C_{ab}) and the browning index (BI) according to Equations 11 and 12 (Araújo, Oliveira, Ramos, Brandão, & Silva, 2016; Corrêa, Braga, Hochheim, & Silva, 2012). Seven samples were evaluated for each treatment.

$$\Delta E = \sqrt{(L_t - L_0)^2 + (a_t - a_0)^2 + (b_t - b_0)^2} \tag{10}$$

$$C_{ab} = \sqrt{(a_t)^2 + (b_t)^2} \tag{11}$$

$$BI = \frac{100\,(x - 0.31)}{0.17} \tag{12}$$

where

$$x = \frac{a_t + 1.75 L_t}{5.645 L_t + a + 3.012 b_t} \tag{13}$$

where L^* indicates the lightness (100 for white to 0 for black), a^*= red when positive and green when negative, and b^*= yellow when positive and blue when negative; the subscripts 0 and t indicate the values for the samples after two hours of treatment and the values for fresh samples, respectively.

2.5.3. Determination of mineral content

The determination of sodium, potassium and calcium contents was performed according to Malavolta (1997). The osmodehydrated eggplant slices were dried at 70 °C for 24 hours. The samples were mashed, and the powder material was subjected to nitric-perchloric digestion. The sodium and potassium contents were analyzed by atomic emission spectrometry, and the calcium content was determined by atomic absorption spectrometry (Micronal, B-262 model, São

Paulo, Brazil). The fresh samples presented the following average values: Na^{+}=1.485 ± 0.075 mg g^{-1}, K^{+}= 16.833 ± 0.757 mg g^{-1} and Ca^{2+}= 3.500 ± 0.529 mg g^{-1}(dry basis).

2.5.4. Ascorbic acid content

Quantification of the ascorbic acid (vitamin C) concentration was performed by the colorimetric method according to Strohecker & Henning (1967). The ascorbic acid content was expressed as mg per 100 g (dry basis). The ascorbic content of the fresh eggplant was 23.913 mg /100 g (dry basis). The retention of ascorbic acid was calculated as the ratio of the percentage of ascorbic acid in the treated sample to that in the fresh sample.

2.6. Statistical analysis

The results were evaluated by one-way ANOVA at the 95% probability level. In the case of significant effects ($p \leq 0.05$), the means were compared using Tukey's test. These analyses were performed using Statistica 8.0 (StatSoft Inc., Tulsa, OK, USA). All experiments were carried out in triplicate.

3.RESULTS AND DISCUSSION

3.1 Mass transfer parameters

The kinetics of WL in OD and PVOD of sliced eggplant are shown in Figures1a and 1b, respectively, for both conditions. It can be seen that the rate of water removal was higher in the first 30 minutes of the process, which corresponds to the greater dehydration driving force between the fruit and the hypertonic solution. As the osmotic process continues, the osmotic pressure is diminished because of the transfer of water and solutes between the phases (Ahmed, Qazi, & Jamal, 2016; Junqueira, Corrêa, & Mendonça, 2016; Silva, Fernandes, & Mauro, 2014). According to Figures 1a and 1b, the WL tends to the equilibrium condition after two hours. Similar results were reported by Bahmani, Jafari, & Dehnad (2015) during the OD of eggplants with sodium chloride as osmotic agent.

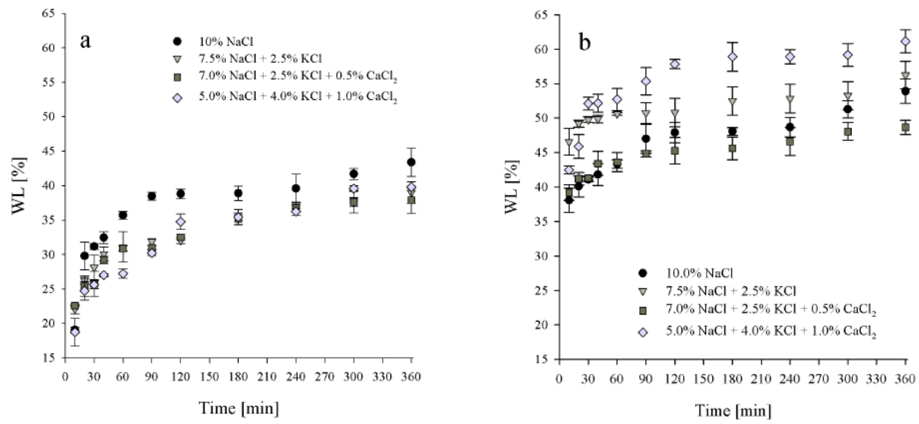

Figure 1 Kinetics of water loss (WL) of eggplant slices during (a) the osmotic dehydration and (b) pulsed vacuum osmotic dehydration, in different osmotic solutions.

Higher WL was achieved in PVOD for all osmotic solutions from the beginning of the process. The vacuum pulse was applied at the first minutes and changed the WL during all the osmotic period. After 6 hours of processing, the samples subjected to vacuum application presented an increase in the WL of 35; 44; 28 and 53%, for treatments 5, 6, 7 and 8, respectively, when compared to OD at atmospheric pressure. The eggplant is a high porous fruit. Its porosity is approximately 46% (Russo, Adiletta, & Di Matteo, 2013). As pointed by Ahmed, Qazi, & Jamal (2016) the porosity favors the effects of the vacuum application. The reduction in pressure causes the expansion and escape of gases in the pores. When the pressure is restored, the pores may be occupied by the osmotic solution, with an increased surface area available for mass transfer (Corrêa, Dev, Gariepy, & Raghavan, 2011; Fito, 1994; Rastogi, Raghavarao, Niranjan, & Knorr, 2002).

For the OD (Figure 1a), the treatment 1 (10% NaCl) presented the highest WL, with respect to the other treatments (2, 3 and 4), that presented similar WL during the osmotic process. In the PVOD process (Figure 1b), the treatment 8 presented the highest WL after six hours of dehydration, and the lowest WL was observed for the treatment 7.

According to Figures 2a and 2b, there were small differences among the SG in all treatments. It was observed that SG increased rapidly during the first 120 minutes. The solid uptake forms resistant layers as the penetration of salt into the tissue continues and leads to structural changes (Bahmani, Jafari, Shahidi, & Dehnad, 2015).

In OD process (Figure 2a), the samples treated in osmotic solutions with calcium (Treatments 3 and 4) presented higher SG during the entire process period. Lower SG values were obtained in samples treated in an osmotic solution without this salt (Treatments 1 and 2). Initially, in PVOD processes (Figure 2b), treatment 7 presented the highest SG rate and treatment 8 the lowest, but during the final processing period, the SG rates of both treatments presented similar values.

In general, the application of vacuum slightly limited the uptake of solids undergoing osmotic dehydration. At the end of the process, treatment with 10% NaCl presented an SG of approximately 4.5% in both OD and in PVOD. It was expected that the vacuum pulse aids the impregnation (Lima, Tribuzi, Souza, Souza, Laurindo, & Carciofi, 2016). Although this was not confirmed in the present study, the SG obtained for both cases were similar. As pointed bellow, vacuum presents different influence on each ion. Corrêa, Ernesto, & Mendonça (2016) also observed small differences in SG during the OD of tomatoes with and without vacuum application in ternary osmotic solutions of NaCl with sucrose and maltodextrin.

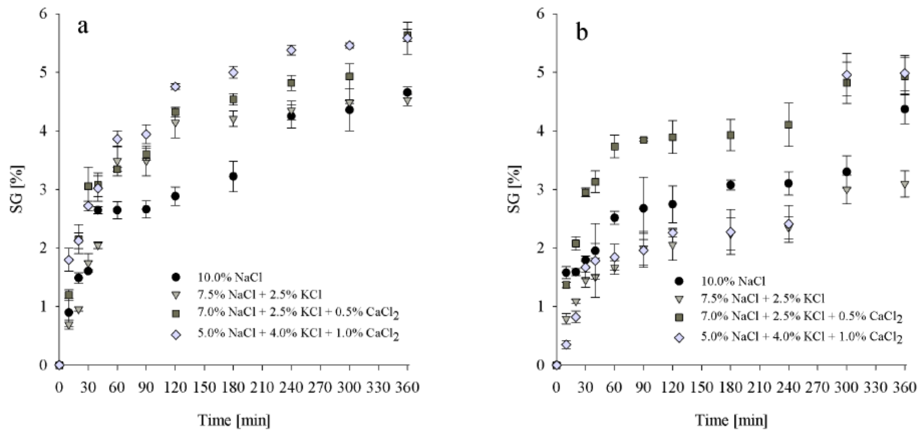

Figure 2 Kinetics of solid gain (SG) of eggplant slices during (a) the osmotic dehydration and (b) pulsed vacuum osmotic dehydration, in different osmotic solutions.

Figures 3a and 3b show the decrease in a_w for samples treated with different solutions as a function of the OD time. When the vacuum pulse was applied at the beginning of the osmotic process, a faster reduction of a_w was observed. This is a consequence of the WL, which increased with the vacuum application. According to Figures 3a and 3b, regardless of the osmotic agent used, the a_w was reduced to less than 0.96 in PVOD in the first hour of processing; the same a_w was obtained after 180 minutes in OD.

In PVOD, the mass transfer improvement achieved by the removal of internal gas and the solution intake into the internal pores reduced the a_w (Corrêa, Pereira, Vieira, & Hubinger, 2010; Corrêa, Ernesto, & Mendonça, 2016). At the end of the process (360 min), PVOD promoted an a_w reduction, related to the increased water removal in this process.

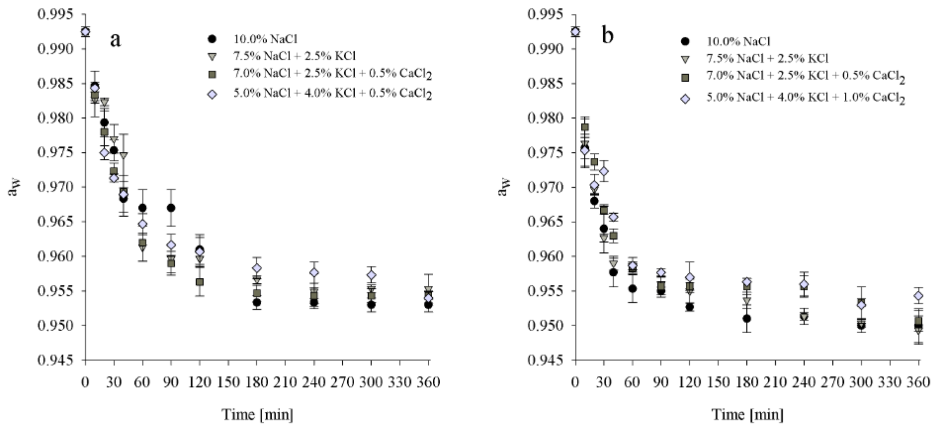

Figure 3 Kinetics of water activity reduction (a_w) of eggplant slices during (a) the osmotic dehydration and (b) pulsed vacuum osmotic dehydration, in different osmotic solutions.

The a_w of all solutions was very similar (Table 1), and no difference in the reduction of a_w at the end of processing was observed between the osmotic solutions employed. The low molecular weight, high dissociation of ions and solubility of the salts were responsible for reduction of the a_w, since they exhibit high permeability in biological materials (Corrêa, Rasia, Garcia-Perez, Mulet, Junqueira, & Cárcel, 2016).

Due to the larger size of the potassium ion compared to the sodium ion, the a_w reduction capacity of KCl was lower than that of NaCl. The higher ion molecular weight, the lower osmolarity, and consequently the lower osmotic pressure difference (Atkins, & Paula, 2012). Bampi, Domschke, Schmidt, & Laurindo (2016) observed during the OD of beef cuts that higher KCl concentration promoted a lower a_w reduction.

3.2 Effective diffusivity

The effective water diffusivities obtained according to the Barbosa Junior model are shown in Table 2. It can be noted that this model represented the water loss behavior with $R^2 > 0.98$ and low SE values. The D_{effw} values ranged from 4.110 x 10^{-10} m^2 /s to 5.464 x 10^{-10} m^2 /s. The values of the effective diffusion coefficients are of the same order of magnitude as those found in the literature (Corrêa, Ernesto, & Mendonça, 2016). Higher D_{effw} values were found in PVOD treatments. Vacuum application promoted higher water loss, as previously demonstrated in Figures 1a and 1b.

Table 2 - Effective diffusion coefficients [m^2 /s] for water and solids in osmotically dehydrated eggplant.

Treatment	D_{effw} x10^{10}	R^2	SE x 10^3	D_{effs} x10^{10}	R^2	SE x 10^3
1	4.345	0.995	1.611	2.545	0.970	5.910
2	4.592	0.988	5.021	2.567	0.983	3.978
3	4.535	0.984	6.260	3.118	0.987	2.665
4	4.110	0.980	8.722	3.049	0.993	1.435
5	5.044	0.985	6.975	3.251	0.945	4.533
6	5.464	0.996	1.869	2.867	0.970	9.617
7	5.329	0.995	5.408	3.547	0.985	15.241
8	4.988	0.995	1.207	1.107	0.899	27.511

The D_{effs} values ranged from 1.107 x 10^{-10} m^2 /s to 3.547 x 10^{-10} m^2 /s, and the D_{effs} values were higher in treatment 3 for OD and 7 for PVOD (Table 2). The calcium may have two opposite effects on plant cells; it may improve the firmness by cross-linking with both cell wall and middle lamella pectin polymers, but it may also cause severe internal disruption, probably because cell membranes are damaged as the process continues (Anino, Salvatori, &

Alzamora, 2006; Silva, Fernandes, & Mauro, 2014). This observation suggests that the increase in calcium concentration promoted damage to the eggplant tissue and diminished the effective solid diffusivities.

The D_{effw} values were higher than the D_{effs} values because the ionic components have a higher resistance to transport than water (Aliño, Grau, Baigts, & Barat, 2009). Bahmani, Jafari, & Dehnad (2015) obtained values of 8.83 x 10^{-10} m^2 /s and 5.81 x 10^{-10} m^2 /s for effective moisture and solid diffusivities, respectively, for the OD of eggplant samples in brine (10% w/w) at 30 °C. Similarly, Abbasi Souraki, Ghavami, & Tondro (2013) found values of 4.73 x 10^{-10} m^2 /s and 3.97 x 10^{-10} m^2 /s for effective moisture and solid diffusivities, respectively, for the OD of cherry tomatoes in brine (10% w/w) at 30 °C.

In regard to Barbosa Junior's model, some considerations were pointed: It is more realistic than the Crank's model (1975) because considers the diffusivity variation during the osmotic process. However, it should be noted that the model is a diffusive one and PVOD presents a hydrodynamics mechanism (Fito, & Chiralt, 1997). This reduces the accurate of the model in the treatments with vacuum application. Besides, the model is not so accurate as the numerical ones that consider the diffusivity as a function of moisture (Porciuncula, Zotarelli, Carciofi, & Laurindo, 2013).

This model presents several advantages as its easy implementation, and despite of lower R^2 values in some treatments, reaching even 0.899, it is a good tool to describe the behavior of osmotic dehydration. The suitability of the model was also confirmed by Junqueira, Corrêa, & Mendonça (2016) during the OD of sweet potato in binary osmotic solutions (sucrose, sorbitol and fructose), and by Corrêa, Viana, Mendonça, & Justus (2016) during the OD of tomatoes in ternary solutions (NaCl and sucrose).

3.3 Mechanical properties

Typical stress/strain curves were extracted from uniaxial compression tests of fresh samples as well as eggplant slices after two hours of osmotic dehydration. The results of maximum stress and elasticity are demonstrated in Figures 4 and 5. The Tukey test results showed a significant difference ($p \leq 0.05$) between the maximum stress of fresh eggplant and samples treated at different pressures (Figure 4). A reduction of the maximum stress was observed in all treated samples. Water loss causes cellular collapse, resulting in cell deformation and shrinkage, affecting the physical properties of the food (Junqueira, Corrêa, & Mendonça, 2016). Cellular disruption leads to a reduction of the turgor pressure and plasmolysis, causing the softening of the tissue and reducing the stress (Castelló, Igual, Fito, & Chiralt, 2009; Mayor, Moreira, Chenlo, & Sereno, 2007; Oliveira, Mendonça, Corrêa, Junqueira, & Justus, 2016).

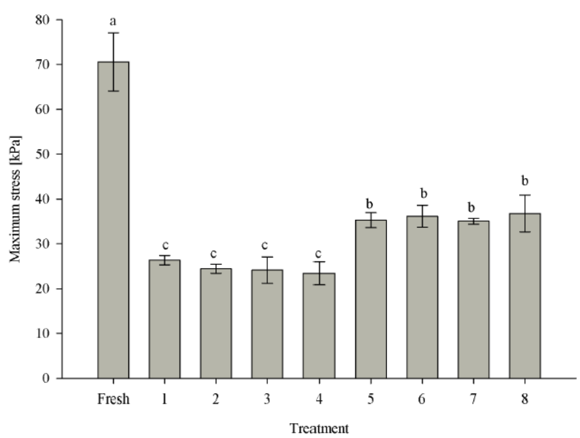

Figure 4 Maximum stress of the fresh and osmodehydrated eggplant slices in different processes. Mean followed by different letters in the row differs significantly ($p \leq 0.05$), according to Tukey's test.

According to Figure 4, an influence of vacuum application on maximum stress values was observed. The PVOD helped maintaining the firmness of the fruit, in comparison with the atmospheric pressure, and a significant difference ($p \leq 0.05$) between these two processes was observed. Vacuum promotes the replacement of the internal gas with the osmotic solution, enhancing the mass transfer, resulting in a stronger and more compact structure (Deng & Zhao, 2008; Ferrari, Arballo, Mascheroni, & Hubinger, 2011).

It can be noted that the type of osmotic solution had no influence on the maximum stress (Figure 4). Significant differences ($p \leq 0.05$) in the maximum stress were not observed between the treatments with and without calcium addition. Similar results were reported by Silva, Fernandes, & Mauro (2014) on the OD of pineapple slices.

A significant difference ($p \leq 0.05$) was observed in the elastic modulus between the treatments (Figure 5).

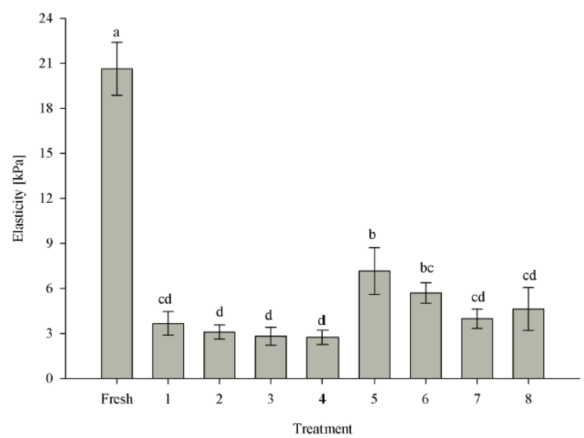

Figure 5 Elasticity modulus of the fresh and osmodehydrated eggplant slices in different processes. Mean followed by different letters in the row differs significantly ($p \leq 0.05$), according to Tukey's test.

As with maximum stress, the elastic modulus is also diminished in osmodehydrated samples. The same observation was recorded by Oliveira, Mendonça, Corrêa, Junqueira, & Justus (2016) during the osmotic dehydration of pear slices. In general, elasticity was higher in PVOD treatments. The high porosity of the eggplant causes the solid matrix to suffer minor deformation and less cell disruption when vacuum is applied, preserving the food structure (Mújica-Paz, Valdez-Fragoso, López-Malo, Palou, & Welti-Chanes, 2003). Such as the maximum stress, the presence of calcium (Treatments C and D) did not improve the mechanical behavior. Similar results were also observed by Castelló, Igual, Fito, & Chiralt (2009) and Moraga, Moraga, Fito, & Martínez-Navarrete (2009) during osmotic processing of apple and grapefruit, respectively.

3.4. Color parameters

Table 3 shows the mean values and standard deviations of the luminosity (L^*), chroma (C_{ab}), total color difference (ΔE) and browning index (BI) obtained for the osmodehydrated eggplant slices. Generally, due to the physicochemical similarities between the osmotic agents, the osmotic solution did not affect the color parameters.

Small differences in L^* values were found between samples treated at atmospheric pressure and the fresh samples (86.674 ± 0.914). However, when the eggplant slices were subjected to vacuum application, a significant decrease ($p \leq 0.05$) in L^* values was found. This effect was caused by the pressure reduction. Similar results were reported by Moreno, Simpson, Pizarro, Parada, Pinilla, Reyes, & Almonacid (2012) and Moreno, Simpson, Pizarro, Pavez, Dorvil, Petzold, & Bugueño (2013) during the OD of strawberry and apple, respectively. Those authors associated the difference in L^* values with the transparency gain of the samples due to the removal of internal gases and the impregnation of the tissues with osmotic solution after the restoration of atmospheric pressure.

Table 3 - Values of color parameters obtained for eggplant slices (fresh and osmodehydrated in different osmotic solutions; processing time = 2 h).

Treatments	L^*	C_{ab}	ΔE	BI
1	84.37±1.60 a	20.43±2.36 b	4.37±1.29 c	23.21±3.04 b
2	85.68±0.96 a	19.42±1.04 b	3.35±0.85 c	20.75±1.49 b
3	85.85±1.11 a	21.51±1.49 ab	2.61±0.88 c	23.79±2.71 b
4	85.08±1.59 a	20.52±1.17 b	2.96±1.17 c	21.74±1.45 b
5	62.93±2.58 bc	22.28±2.81 ab	24.39±2.33ab	39.32±3.13 a
6	65.11±2.61 bc	21.82±1.31 ab	22.04±2.70ab	37.49±3.79 a
7	61.67±2.16 c	22.59±1.01 ab	25.12±2.09a	36.68±3.45 a
8	66.15±1.59 b	24.12±1.47 a	20.77±1.45 b	35.29±3.07 a

Mean followed by different letters in the column differs significantly (p=0.05), according to Tukey's test.

Significant differences ($p \leq 0.05$) in C_{ab} values (Table 3) were observed. A slight increase in the yellow color of samples treated with vacuum was observed, and this promoted the increase in color saturation.

According to Table 3, the vacuum pulse promoted higher ΔE. The results demonstrated that the OD at atmospheric pressure preserved better the color of the samples. As previously noted, a reduction in L^*, due to the loss of clarity, was reflected as a more pronounced deterioration of the color. It is interesting to note that the larger ΔE was observed when the vacuum pulse was used. It can be associated to the greater variation in the L^* values in PVOD, indicating the browning of the samples (Oliveira, Corrêa, Pereira, Ramos, & Vilela, 2016). Corrêa, Ernesto, Alves, & Andrade (2014) reported that the color variation during the OD is related to water loss, solids uptake and pigment concentration.

The application of vacuum also influenced the browning index, as demonstrated in Table 3. Samples treated without vacuum presented lower BI than those treated with vacuum, and the difference was significant ($p \leq 0.05$). The high values of luminosity in this treatment was related to this fact, as noted previously.

These results indicated that the pressure of the process directly influenced color properties. Two main mechanisms are responsible for this color difference: enzymatic and non-enzymatic reactions, which are responsible for the degradation of components during processing (Schulze, Hubbermann, & Schwarz, 2014). The use of chemical treatment before the osmotic process (ascorbic acid and citric acid) prevents enzymatic browning (Moreno, Simpson, Pizarro, Pavez, Dorvil, Petzold, & Bugueño, 2013), which suggests that non-enzymatic reactions was predominant in PVOD processes.

3.5 Mineral content

Osmodehydrated samples presented higher sodium content than fresh eggplant (1.485 ± 0.075 mg /g), resulting from solid uptake during the immersion into the osmotic solutions (Table 4). The incorporation of solids during the osmotic process has been extensively discussed (Brochier, Marczak, & Noreña, 2015; Nahimana, Zhang, Mujumdar, & Ding, 2011; Vieira, Pereira, & Hubinger, 2012) and is attributable to the counter-current mass diffusion that occurs on the osmotic processes.

Table 4 shows a significant difference ($p \leq 0.05$) in sodium content between the treatments. Vacuum did not significantly affect total sodium uptake. This corroborated the slight difference in SG (Figures 2a and 2b) between atmospheric and reduced-pressure osmotic processes. In general, as the concentration of sodium decreased, a proportional reduction in its content was observed. Comparing the treatments using 10% NaCl (1 and 5) to treatments using 5% NaCl + 4% KCl + 1% $CaCl_2$ (4 and 8), sodium content was reduced by approximately 50%, regardless of the vacuum application.

Table 4 - Mineral contents (dry basis) obtained for eggplant slices osmodehydrated in different osmotic solutions (processing time = 2 h).

Treatment	Na [mg /g]	K [mg /g]	Ca [mg /g]
1	299.648± 6.543 b	14.466± 0.577 e	2.067± 0.251 b
2	224.273± 9.968 d	16.766± 0.513 d	2.167± 0.231 b
3	196.863± 4.374 e	17.167± 0.924 d	9.800± 0.519 ab
4	142.652± 1.448 f	17.366± 0.351 d	13.466± 1.738 a
5	325.681± 6.904 a	10.266± 1.193 f	2.433± 0.153 b
6	265.288± 9.972 c	65.350± 1.250 c	2.233± 0.115 b
7	227.553± 9.026 d	92.800± 0.400 b	11.400± 0.608 ab
8	160.630± 4.289 f	136.300± 0.600 a	17.633± 1.986 a

Mean followed by different letters in the column differs significantly (p=0.05), according to Tukey's test.

The potassium content of the fresh samples (16.833 ± 0.757 mg /g) was higher than the values observed in the treated samples in solutions without potassium (Table 4). Osmotic dehydration presents a leaching flow of natural minerals from the fruit to the osmotic solution (Ahmed, Qazi, & Jamal, 2016; Guiamba, Ahrné, Khan, & Svanberg, 2016; Mendonça, Corrêa, Junqueira, Pereira, & Vilela, 2016) and this leaching was higher in the PVOD (treatment 5). The potassium content was statistically different ($p \leq 0.05$) between the treatments, but with small differences in the incorporation in OD at atmospheric pressure.

As observed in Table 4, higher potassium content was observed in vacuum treatments. Comparing samples dehydrated in osmotic solution with higher potassium content (Treatments 4 and 8), in the OD process, the potassium ion content increased by 20 %, and in the PVOD process, it increased by 1227 %. This result suggests that exchanging gas in the pores for the osmotic solution promotes higher levels of potassium in cell tissue.

The calcium content of the fresh samples was 3.500 ± 0.529 mg /g. As pointed for potassium, the osmotic treatment also lead to the leaching of this mineral. This could be observed in the treatments 1, 2, 5 and 6, that did not present calcium on the osmotic composition (Table 4). A significant difference ($p \leq 0.05$) in calcium content was observed among the treatments. As expected, higher calcium incorporation were obtained for samples treated with osmotic solution which were composed with calcium in the formulation.

The vacuum pulse did not significantly influence the calcium concentration after two hours of processing when compared to OD samples, showing that the vacuum pulse removes moisture without changing the calcium concentration (Lima, Tribuzi, Souza, Souza, Laurindo, & Carciofi, 2016). Those authors reported a higher concentration of calcium in vacuum impregnation of pineapple slices. They attributed this to a higher mass transfer into the pineapple pores via the hydrodynamic mechanism, which could indicate a very quick interaction between the calcium and the fruit tissue.

As a general remark, it can be noted that the replacement of NaCl by KCl and $CaCl_2$ led to a reduction in sodium content and an increase in the calcium content, showing that this treatment is a potential alternative for improving the quality of osmodehydrated fruit, increasing health benefits without significant changes in global mass transfer. The potential of OD and PVOD as sustainable processes for enrichment and production of healthy food is highlighted by Ciurzyńska, Kowalska, Czajkowska, & Lenart (2016).

3.6 Ascorbic acid retention

The retention of ascorbic acid after the osmotic treatments is shown in Table 5. A higher retention of vitamin C ($p < 0.05$) was observed in the treatments with added calcium (3, 4, 7 and 8) compared to treatments without this ion. In PVOD, higher calcium uptake was achieved (Table 5) and retentions of 69.68 % (Treatment 7) and 76.97 % (Treatment 8) were observed. The calcium interacts with the plant cellular matrix, improving cell wall integrity by forming bonds between wall components, reducing tissue permeability (Guiamba, Ahrné, Khan, & Svanberg, 2016; Gras, Vidal, Betoret, Chiralt, &Fito, 2003) and consequently reducing the leaching of ascorbic acid.

Table 5 – Ascorbic acid retention of eggplant slices osmodehydrated in different osmotic solutions (processing time = 2 h).

Treatment	Ascorbic acid retention [%]	Treatment	Ascorbic acid retention [%]
1	39.66 ± 1.59 e	5	49.04 ± 0.92 d
2	33.04 ± 1.63 f	6	41.59 ± 0.53 e
3	57.26 ± 1.09 c	7	69.68 ± 0.93 b
4	59.51 ± 0.30 c	8	76.97 ± 0.88 a

Mean followed by different letters in the column differs significantly (p=0.05), according to Tukey's test.

Reductions in ascorbic acid content were observed in all osmodehydrated samples (Table 5). The loss of ascorbic acid during osmotic processes occurs through oxidation and leaching from the fruit to the aqueous solution, since it is a water soluble bioactive compound found in the cell sap (Germer, Morgano, Silva, Silveira, & Souza, 2015; Nambi, Gupta, Kumar, & Sharma, 2016). Araya-Farias, Macaigne, &Ratti (2014) reported losses in the ascorbic acid content of osmotically dehydrated sea buckthorn fruits.

According to Table 5, it was observed that fruits treated under vacuum presented a higher retention of ascorbic acid. This is probably due to the removal of occluded gases and the substitution by osmotic solutions, which reduced the aerobic degradation of the vitamin C.

4. CONCLUSION

The application of vacuum in the OD of eggplant slices increased the WL and reduced the a_w. The model of Barbosa Junior et al. model was satisfactorily adjusted for the osmotic treatment of eggplant, mainly for the OD at atmosphere pressure. Consequently, effective diffusivities were lower for vacuum-treated samples. Higher values of maximum stress and elasticity were achieved when the reduced pressure vacuum was applied. The color parameters were better preserved in OD processes (without vacuum). The vacuum application increased the ion incorporation, especially for the potassium content. The addition of calcium increased the ascorbic acid retention. The substitution of NaCl by KCl and $CaCl_2$ was shown to be feasible, potentially producing a healthy product without negatively affecting the nutritional, physical or mass transfer parameters.

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ARTICLE 2

PULSED VACUUM OSMOTIC DEHYDRATION OF CARROTS, EGGPLANT AND BEETROOT SLICES: EFFECT OF VACUUM PRESSURE ON THE KINETICS AND MATHEMATICAL MODELING

Running title: Pulsed vacuum osmotic dehydration of vegetables.

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(Elaborated in accordance to the journal Food and Bioproducts Processing)

Abstract - The 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 OD, in a process named 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, solid gain and water activity reduction, in ternary solution (distilled water, sucrose and sodium chloride) for 300 minutes. Moreover, mathematical modeling of experimental data obtained from the OD processes, was employed for correlating the water loss and solid gain of the food product in the different conditions. An intensification of water loss and solid gain were observed for carrot and eggplant in PVOD processes. This was related to their porous structure. The present work demonstrated a lack of fit from the Fick's second law. The Midilli's semi-empirical equation satisfactorily described the dehydration kinetics with the highest R^2.

Keywords Pulsed vacuum osmotic dehydration; carrot; beetroot; eggplant.

1. INTRODUCTION

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

The rates of mass transfer 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 the internal gases expansion, and with the atmospheric pressure restoration, this gases are removed trough the hydrodynamics mechanism (HDM), accelerating the process (Fito, 1994; Junqueira et al., 2017; 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 solid incorporation during the osmotic dehydration. It is observed due to the higher of the 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; Utkucan & Kemal, 2016; Viana, Corrêa, & Justus, 2014).

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 mechanisms of mass transport during the OD are complex and not completely understood (Silva et al., 2012).

Dehydration models have been lead by considering the diffusion as the majority mechanism for describing the whole mass transfer trough the food and the osmotic solution, in unsteady state by the use of the Fick's law (Crank, 1975). This model allows the determination of the diffusivity coefficient, and it was satisfactorily used during the OD of pineapple (Silva, Fernandes, & Mauro, 2014), tomatoes (Abbasi Souraki, Ghavami, & Tondro, 2013) and jackfruit (Kaushal & Sharma, 2016). Moreover, different approaches have been employed to describe the osmotic process, as the semi-empirical models (obtained from mathematical correlations of the experimental data) and other phenomenological models (Kucuk et al., 2014).

Between the semi-empirical equations the Page's equation (Page, 1949) with two constants and the Midilli's (or Midilli & Kuçuk's) with four constants have been determined to the most suitable models employed for various products in dynamic process (including OD) with good agreements (Kucuk et al., 2014).

As the phenomenological models must portray the physical mechanism of the processes, the Fito & Chiralt's model have been widely used for coupling the diffusional and hydrodynamics effects during the OD (Corrêa, Ernesto, & Mendonça, 2016; Fito & Chiralt, 1997). However, 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).

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 reduction and to fit the experimental data to phenomenological (Fick's law and Fito & Chiralt' model) and semi-empirical equations (Page and Midilli).

2. MATERIAL 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 (AOAC, 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.

Table 1 - Physical characteristics of fresh vegetables.

	Carrot	Eggplant	Beetroot
Initial moisture content [kg /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

2.2 Sample and osmotic solution preparation

The vegetables were washed in tap water, peeled and cut into slices of 2.00 cm length x 2.00 cm width x 0.50 cm thickness 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 300 or 600 mmHg were applied to the system during the first 10 minutes of process, and then, the local atmospheric pressure (Lavras, MG, Brazil) was restored, 755 mmHg (VP = 0).

The temperature was set at 35 ± 1 ºC 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 during 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 (2010). All of the experiments were performed in four replicates.

The water loss (WL) and solid gain (SG) were calculated in accordance with Eqs. 1 and 2, respectively,

$$WL\,[\%] = \frac{\left(M_0\,X_{w0}\right) - \left(M_t\,X_{wt}\right)}{M_0}100 \qquad (1)$$

$$SG\,[\%] = \frac{\left(M_t\,X_{st}\right) - M_0\left(1 - X_{w0}\right)}{M_0}100 \qquad (2)$$

where M_0 is the weight of the sample at time t= 0 s [kg], X_{w0} is the initial water content [kg water /100 kg sample], M_t is the weight of the sample at time t [kg], X_{wt} is the water content [kg water /100 kg sample] at time t and X_{st} is the soluble solid content [kg solid /100 kg sample] at time t.

2.4 Mathematical models

The experimental kinetic data were fit to phenomenological models, with the estimation of the effective diffusivity and semi-empirical models, with the estimation of the empirical coefficients.

2.4.1 Fick's second law

Fick’s second law for unidirectional unsteady state diffusion is given by Eq. 3:

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

where X_t is water or solid content [kg water or solid /100 kg sample] 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 the Eq. 4

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

where W is the dimensionless water or solid content and L is the characteristic length (half of 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, $\left. \partial X_t \middle/ \partial t \right|_{z=0} = 0$, and the equilibrium content at the surface, $X_{(L,t)} = X_{eq}$ (Crank, 1975). The dimensionless water or solid content is given by Eq. 5:

$$W = \frac{X_t - X_{eq}}{X_0 - X_{eq}} \tag{5}$$

where X_{eq} is the water or solid content at equilibrium [kg water or solid /100 kg sample] and X_0 is the initial water or solid content [kg water or solid /100 kg sample].

The equilibrium values, were estimated according to Peleg's equation (Peleg, 1988), Eq. 6:

$$X_t = X_0 + \frac{t}{k_1 + k_2\, t} \tag{6}$$

where k_1 is the Peleg rate constant, related to dehydration rate at the initial time ($t=t_0$) and the constant k_2 is related to WL and SG at equilibrium conditions ($t=\infty$), according to Eq. 7

$$X_{eq} = X_0 \pm \frac{1}{k_2} \tag{7}$$

In the above equations, "±" becomes "–" for WL and "+" for SG.

2.4.2 Fito and Chiralt's model

The Fito and Chiralt hydrodynamics model (Fito & Chiralt, 1997) considers an equilibrium approach (Eq. 8):

$$z_{eq}^{ss} = y_{eq}^{ss} \tag{8}$$

where z^{ss}_{eq} is the mass fraction of the soluble solids in the food and y^{ss}_{eq} 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 the Eq. 9:

$$Y = Y_t^w = Y_t^s = \frac{z_t^w - z_{eq}^w}{z_0^w - z_{eq}^w} \tag{9}$$

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 (Eq. 10)

$$1 - Y_t^w \Big|_{t=t_0}^{t=t_{HDM}} = k \tag{10}$$

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 and Chiralt (1997) (Eq. 11)

$$1 - Y_t^w \Big|_{t=t_{HDM}}^{t=t} = 2\sqrt{\frac{D_{eff}\, t}{\pi\, L^2}} \tag{11}$$

These two effects were coupled to consider the effect of the diffusional and hydrodynamics mechanisms (Eq. 12)

$$1 - Y_t^w \Big|_{t=t_0}^{t=t} = k + 2\sqrt{\frac{D_{eff}\, t}{\pi\, L^2}} \tag{12}$$

The D_{eff} and k parameters were obtained for each experiment from a linear fitting of the experimental $1\text{-}Y^w_t$ versus $t^{0.5}$.

2.4.3 - Semi-empirical equations

Selection of semi-empirical equations was based on those previously proposed to describe the characteristics of agricultural products during osmotic dehydration (Kucuk et al., 2014; Nuñez-Mancilla et al., 2011). In this case, the selected mathematical models are: Page (Eq. 13) and Midilli (Eq. 14).

$$W = \exp\left(-k\, t^n\right) \tag{13}$$

$$W = a \exp\left(-k\, t^n\right) + bt \tag{14}$$

where k, n, a and b are adjustment coefficients and t denotes the time [s].

2.5. Statistical evaluation of the models

Data were analyzed using Statistica software (Statistica 8.0, Statsoft Inc., Tulsa, OK). The parameters of equations were estimated using a non-linear regression procedure. The fit quality of the experimental data to proposed models was evaluated using the regression coefficient (R^2) and estimative standard error (SE, Eq. (15)).

$$SE = \frac{1}{N}\sum_{i=1}^{N}\left(W_{pre,\mathrm{i}} - \mathrm{W}_{\mathrm{exp,i}}\right)^2 \quad (15)$$

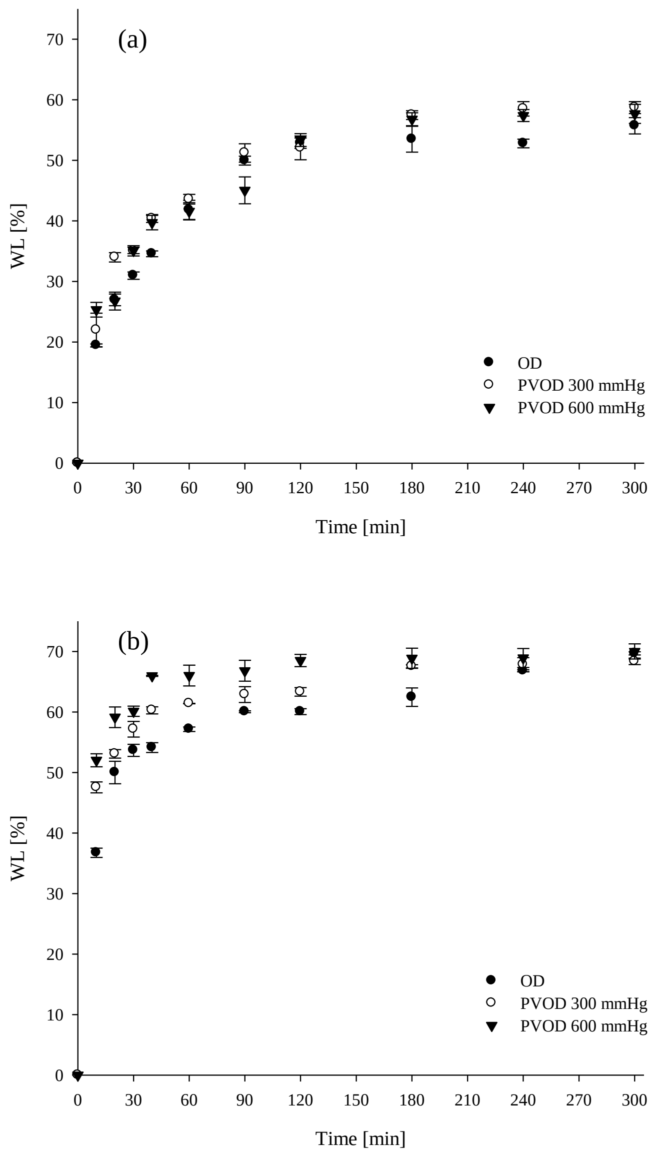
where the subscripts exp,i and pre,i denotes experimental and predicted dimensionless water or solid content respectively and N is the number of observations. Higher R^2 and lower SE values indicated a better fit of the experimental data to the model (Kaushal & Sharma, 2016).

3. RESULTS AND DISCUSSION

3.1 Water loss kinetics

The kinetics of WL in osmodehydrated vegetables are shown in Fig. 1 a-c. For all the vegetables, the rate of water removal was higher in the first 120 min of the process. Initially, the dehydration driving force between the product and the hypertonic solution is the greatest one, and as the osmotic process extends, a reduction on the osmotic pressure occurs, due to mass transfer between the phases (Junqueira, Corrêa, & Mendonça, 2017; Lombard, Oliveira, Fito, & Andrés, 2008; Ramya & Jain, 2017).

For the carrot (Fig. 1a) and eggplant (Fig. 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 porous of the plant tissue and the replacement of these native material by the external osmotic solution (Ahmed, Qazi, & Jamal, 2016; Fito, 1994; Moreno et al., 2016).



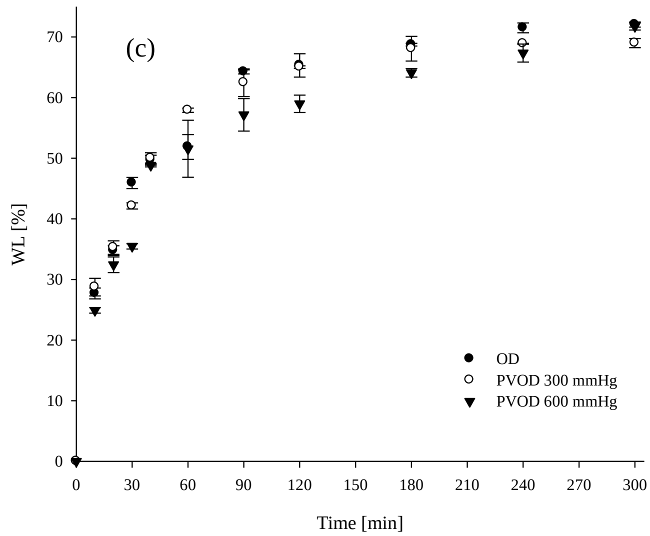


Figure 1 Kinetics of water loss (WL) of carrot (a), eggplant (b) and beetroot (c) slices during the osmotic dehydration.

The carrot, present a heterogeneous composition, and even though it is considered a low porosity product, morphologically it is composed by an inner xylem (core) and a outer phloem (cortex) (Nahimana, Munjumdar, & Zhang, 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 600 mmHg showed higher WL (Fig. 1b). The VA enhances the mass transfer in the initial period of the OD (Deng & Zhao, 2008), but the final of the osmotic processes, no remarkable differences in the WL were observed. According to the Fig. 1a, the WL tended to the equilibrium condition after 3 h for all the treatments.

According to the Fig. 1b, the eggplant WL was strongly influenced by the VA, and higher WL was achieved in PVOD processes. After 10 min of process, the samples subjected to VA presented an increase in the WL of approximately 30 % (PVOD 300 mmHg) and 40% (PVOD 600 mmHg), respectively, when compared to OD at atmospheric pressure. Similar results were reported by Junqueira et al. (2017) during the OD and PVOD of eggplant slices in salt solutions.

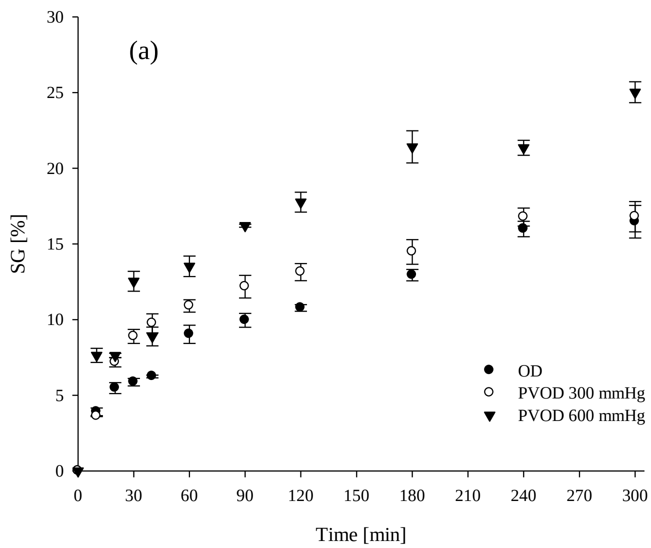
The higher water removal response of the eggplant when the vacuum was applied, is related to its very porous structure (Russo, Adiletta, & Di Matteo, 2013). The pressure gradients during the PVOD, promote the outflow mainly of the occluded gases. When the atmospheric pressure is restored, the compression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

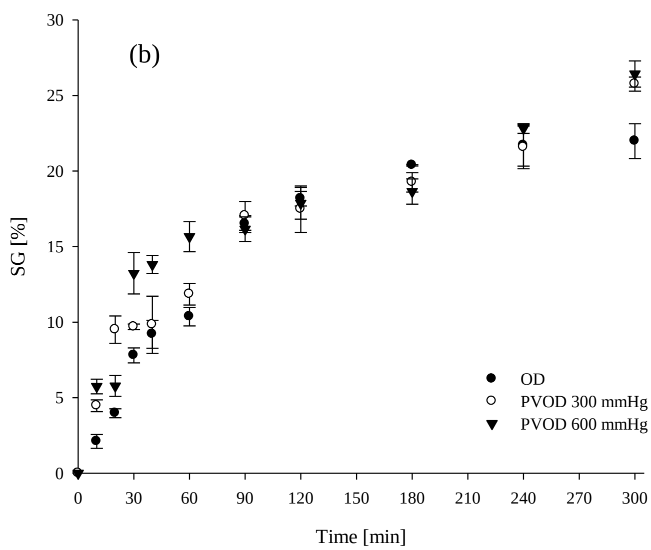
The effects of VA during the OD of beetroot (Fig. 1c) was not evidenced after the 10 initial minutes, in which the PVOD was conducted. During all the osmotic process, the treatments presented similar behavior, and the influence of the VA was not noted. At the final time (300 min), no remarkable differences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

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 with the quite hard texture could hinder the exchanges established by the VA, with consequent insufficiency for reducing the mass transfer parameters. (Corrêa, Ernesto, & Mendonça, 2016) reported that regardless of the VA, little differences in the WL during the OD and PVOD of tomatoes slices were observed.

3.2 Solid gain kinetics

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





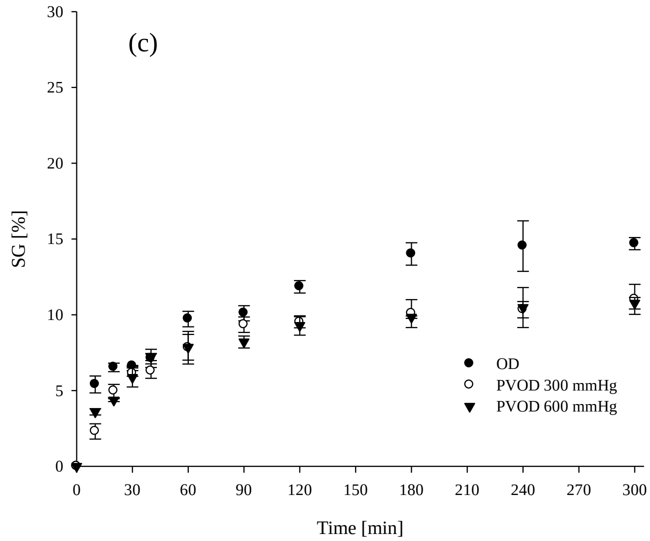


Figure 2 Kinetics of solid gain (SG) of carrot (a), eggplant (b) and beetroot (c) slices during the osmotic dehydration.

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 600 mmHg promoted higher solid uptake rates. At the end of the process (300 min) no differences were reported to the treatments OD and PVOD 300 mmHg, 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 (Fig. 2b). After the VA, in the first 10 min, the SG was doubled in PVOD treatments, compared with those obtained in the OD. At the end of the process (300 min), it was noted that both vacuum treatments presented similar values, also higher than the OD observed 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).

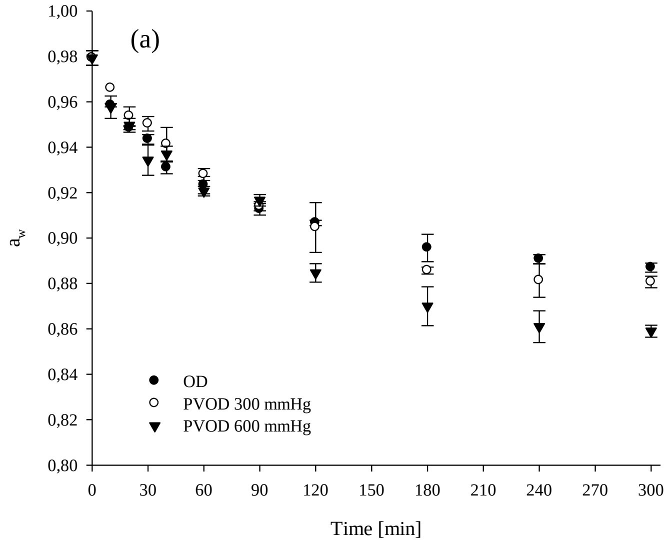
For the beetroot (Fig. 2c), the VA reduced the SG, contrary to the literature reports. Corrêa, Ernesto, & Mendonça (2016) related that this phenomena was possible during the OD and PVOD of tomatoes in ternary osmotic solutions. The treatment conducted at atmospheric pressure presented higher SG during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

3.3 Water activity reduction kinetics

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

In osmotic processes, the a_w decreased due to the moisture reduction (related to the WL) and the SG (Corrêa, Ernesto, & Mendonça, 2016; Lech et al., Michalska et al., 2018). For the carrot (Figs. 3a) and eggplant (Fig. 3b), higher a_w reduction was obtained in the vacuum treatments, with emphasis for the PVOD 600 mmHg. It was also related with the higher SG observed in this treatment (Figs. 2a and 2b). For the beetroot, the OD promoted SG (Fig. 2c), which reflected in a further reduction in the a_w for this treatment (Fig. 3c).

It should be noted that the reduction in the a_w of the products do not provide them microbiologically stable, and as a pretreatment, the osmotic process requires a further preservation technique as the drying (Kowalski & Łechtańska, 2015; Sette, Salvatori, & Schebor, 2016; Utkucan & Kemal, 2016).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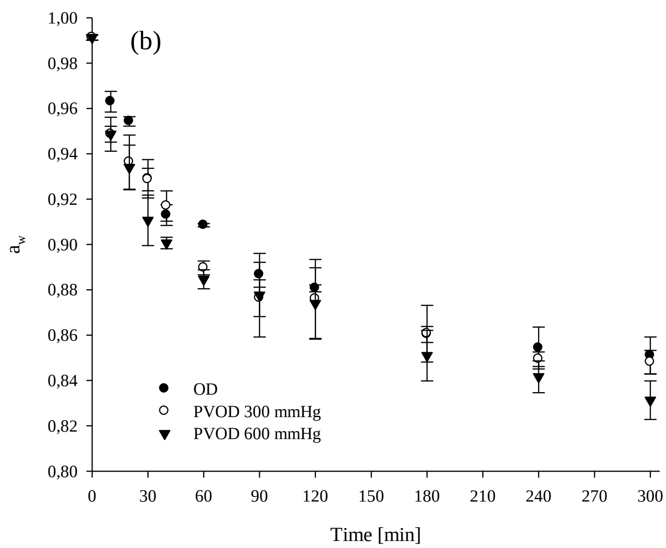
OD
PVOD 300 mmHg
PVOD 600 mmHg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PVOD 300 mmHg
PVOD 600 mmHg

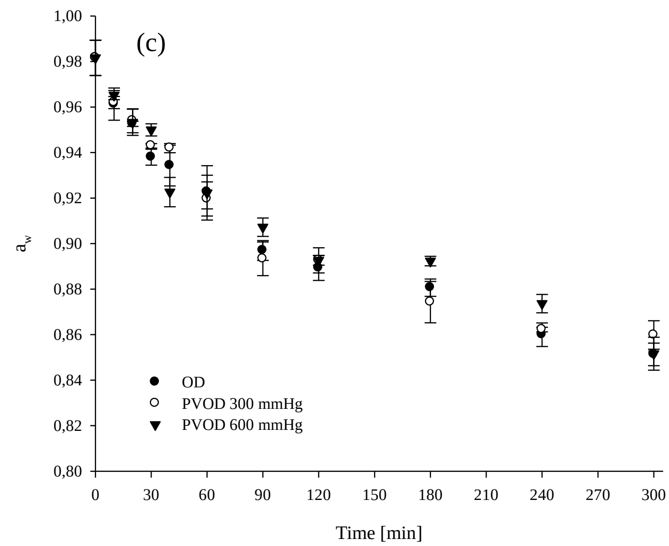

Figure 3 Kinetics of water activity reduction (a_w) of carrot (a), eggplant (b) and beetroot (c) slices during the osmotic dehydration.

3.4 Mathematical modelling

The OD kinetics were adjusted by four models in the literature, and the effective diffusivities, adjustment constants and statistical parameters are provided in Tables 2–5.

The effective diffusivities obtained according to the Fick's model was shown on Table 2. For the carrot, the water effective diffusivity (D_{effw}) values ranged from 6.497 x 10^{-10} to 7.769 x 10^{-10} m^2 /s, and the solid effective diffusivity (D_{effs}) ranged from 1.634 x 10^{-10} to 4.389 x 10^{-10} m^2 /s. For this vegetable, it was obtained $R^2 > 0.95$. Lower effective diffusivities were observed for the samples treated with OD at atmospheric pressures (except for the beetroot), as previously presented by the Figures 1a and 2a.

Table 2 - Effective diffusivities of water (D_{effw}) [m^2 /s] and solids (D_{effs}) [m^2 /s] of osmodehydrated vegetables, according to Fick's model.

Treatment	D_{effw} x10^{10}	R^2	SE x 10^3	D_{effs} x10^{10}	R^2	SE x 10^3
Carrot						
OD	6.497	0.964	2.758	1.634	0.972	1.616
PVOD300	7.769	0.961	3.040	4.389	0.962	2.863
PVOD600	6.721	0.954	3.660	2.348	0.951	3.436
Eggplant						
OD	12.15	0.872	9.684	2.177	0.961	2.565
PVOD300	12.20	0.738	19.76	2.150	0.965	2.324
PVOD600	12.22	0.640	32.21	3.340	0.926	5.729
Beetroot						
OD	7.385	0.962	2.907	3.246	0.963	2.751
PVOD300	8.362	0.966	2.595	5.494	0.958	3.240
PVOD600	6.145	0.956	3.399	5.729	0.961	3.017

For the eggplant, slight differences were observed for the D_{effw} values, which ranged from 12.14 x 10^{-10} to 12.22 x 10^{-10} m^2 /s, showing similar behavior to those presented by the Fig. 1b, although it was observed lower R^2 values (< 0.87). The D_{effs} ranged from 2.150 x 10^{-10} to 3.340 x 10^{-10} m^2 /s and R^2 > 0.92. For the beetroot, the D_{effw} ranged from 6.145 x 10^{-10} to 8.362 x 10^{-9} m^2 /s, and the D_{effs} ranged from 3.246 x 10^{-10} to 5.729 x 10^{-10} m^2 /s with R^2 > 0.95 (Table 2).

Although the effective diffusivities varies with the food material and the experimental conditions, which makes it difficult its comparison in terms of exact values, the D_{eff} obtained according to the Fick' model presented the analogous order of magnitude of food materials subjected to the OD (Bahmani et al., 2015; Mendonça et al., 2017; Souraki, Ghavami, & Tondro, 2014).

According to the Table 2, Fick's model showed a 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 the Fick's diffusive model for the osmotic processes (Barbosa Júnior, Cordeiro Mancini, & Hubinger, 2013; Corrêa et al., 2010; Corrêa, Ernesto, & Mendonça, 2016; Zielinska, Zielinska, & Markowski, 2018). Simpson et al. (2015) pointed that the complexity of the mass transfer process due to the heterogeneous nature of plant tissues can reflect to a lack of fit of the 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, Marczak, & Noreña, 2015).

Moreover, the osmotic dehydration is not a simple diffusional process, which means that the mass transport of water and solids is not homogeneous. There are other mechanisms than diffusion involved in this process, as the capillary flow, volumetric shrinkage, the removal of trapped gases and the hydrodynamic process during the vacuum application. The dehydration is a complex process, and it can be observed that all the physical phenomena affects the development and the suitability of the models for representing the process (Azarpazhooh & Ramaswamy, 2012; Deng & Zhao, 2008; Miano & Augusto, 2018).

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

In 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). The Table 3 provides de D_{eff} obtained for all the vegetables employing this phenomenological model.

Lower diffusivities were obtained by applying this model (Table 3). It ranged from 1.904 x 10^{-10} m^2 /s to 2.057 x 10^{-10} m^2 /s for carrot, from 2.109 x 10^{-10} m^2 /s to 2.131 x 10^{-10} m^2 /s for eggplant and from 1.824 x 10^{-10} m^2 /s to 2.076 x 10^{-10} m^2 /s for beetroot. According to the Table 3, in a general way, it was observed higher R^2 and lower SE, when compared with those obtained by the Fick's model (Table 2).

During the period of the vacuum application, the gases and free internal liquid flow out and the molecular diffusion can be enhanced. After the atmospheric pressure restoration, the matrix pore volume reduces and occurs the expulsion of occluded fluids. Fito & Chiralt's model considers the diffusion and hydrodynamic mechanisms during PVOD, achieving a better fit to the experimental data (Ahmed et al., 2016; Fito et al., 2001).

Semi-empirical equations has been successfully used for describing the dehydration behavior with high good agreement to the experimental data. Simple equations without a reasonable theoretical basis can adjust the results more efficiently than complex models, based on physical mass transfer phenomena (Kemp, 2011). Tables 4 and 5 show the mean values of the statistical tests for WL and SG to the selected equations.

Table 3 - Effective diffusivities (D_{eff}) [m^2 /s] of osmodehydrated vegetables, according to Fito & Chiralt's model.

Treatment	D_{eff} x10^{10}	R^2	SE x 10^3
	Carrot		
OD	1.904	0.974	1.682
PVOD 300	2.057	0.966	1.715
PVOD 600	1.942	0.960	1.691
	Eggplant		
OD	2.131	0.928	1.730
PVOD 300	2.109	0.878	1.726
PVOD 600	2.110	0.890	1.762
	Beetroot		
OD	1.824	0.973	1.665
PVOD 300	2.076	0.951	1.719
PVOD 600	1.879	0.981	1.678

According to Tables 4 and 5, the high R^2 (even reaching 0.99) and low SE values for the Page and Midilli's equation were acceptable to characterize the behavior of the osmodehydrated vegetables in all treatments. It was concluded that both equations were suitable for predicting the dehydration kinetics probable due to the limited theoretical basis and the absence of physical meaning of the adjustment parameters (Assis, Morais, & Morais, 2016; Nuñez-Mancilla et al., 2011).

Table 4 - Adjustment parameters of osmodehydrated vegetables, according to Page's equation.

Treatment		Parameters	R^2	SE x 10^3
		Carrot		
OD	WL	k= 0.0078; n= 0.6057	0.987	1.036
	SG	k= 0.0079; n=0.5963	0.990	0.737
PVOD 300	WL	k= 0.0125; n= 0.5632	0.994	0.456
	SG	k= 0.0056; n= 0.6193	0.986	1.062
PVOD 600	WL	k= 0.0117; n= 0.5612	0.986	1.105
	SG	k= 0.0024; n= 0.6671	0.959	2.847
		Eggplant		
OD	WL	k= 0.0489; n= 0.4526	0.987	0.990
	SG	k= 0.0013; n= 0.7324	0.976	1.589
PVOD 300	WL	k= 0.0939; n= 0.3974	0.997	0.206
	SG	k= 0.0019; n= 0.6869	0.972	1.845
PVOD 600	WL	k= 0.1043; n= 0.4019	0.998	0.176
	SG	k= 0.0040; n= 0.6389	0.943	4.479
		Beetroot		
OD	WL	k= 0.0109; n= 0.5761	0.992	0.605
	SG	k= 0.0043; n= 0.6272	0.977	1.731
PVOD 300	WL	k= 0.0113; n= 0.5799	0.991	0.672
	SG	k= 0.0059; n= 0.6291	0.983	1.337
PVOD 600	WL	k= 0.0092; n= 0.5835	0.988	0.908

	SG	k= 0.0079; n= 0.5963	0.990	0.738

Table 5 - Adjustment parameters of osmodehydrated vegetables, according to Midilli's equation.

Treatment		Parameters	R^2	SE x 10^3
		Carrot		
OD	WL	a= 1.0041; k= 0.0036;	0.994	0.506
		n= 0.7138; b= 4.673x10^{-6}		
	SG	a= 1.0165; k= 0.0075;	0.991	0.684
		n= 0.6091; b= 1.171x10^{-6}		
PVOD 300	WL	a= 1.0139; k= 0.0121;	0.994	0.424
		n= 0.5707; b= 7.232 x10^{-7}		
	SG	a= 1.0194; k= 0.0077;	0.987	1.008
		n= 0.5809; b= -1.426x10^{-6}		
PVOD 600	WL	a= 1.0289; k= 0.0160;	0.987	1.018
		n= 0.5240; b= -1.097x10^{-6}		
	SG	a= 1.0709; k= 0.0146;	0.968	2.215
		n= 0.4467; b=-9.985x10^{-6}		
		Eggplant		
OD	WL	a= 1.0037; k= 0.0696;	0.988	0.874
		n= 0.4024; b= -2.081x10^{-6}		
	SG	a= 0.9832; k= 0.0002;	0.992	0.551
		n= 0.9888; b=1.111x10^{-5}		
PVOD 300	WL	a= 1.0023; k= 0.1238;	0.998	0.134

		n= 0.3575; b= - 1.475x10^{-6}		
	SG	a= 1.0511; k= 0.0083;	0.978	1.424
		n= 0.5050; b= 8.482x10^{-6}		
PVOD 600	WL	a= 1.0005; k= 0.0958;	0.998	0.169
		n= 0.4145; b= 3.920x10^{-7}		
	SG	a= 1.0300; k= 0.0116;	0.948	4.033
		n= 0.5008; b= -7.543x10^{-6}		
		Beetroot		
OD	WL	a= 1.0120; k= 0.0085;	0.994	0.495
		n= 0.6125; b= 2.013x10^{-6}		
	SG	a= 1.0676; k= 0.0151;	0.984	1.232
		n= 0.4744; b= -6.153x10^{-6}		
PVOD 300	WL	a= 1.0053; k= 0.0062;	0.996	0.280
		n= 0.6652; b= 3.692x10^{-6}		
	SG	a= 0.9984; k= 0.0026;	0.989	0.847
		n= 0.7404; b= 4.876x10^{-6}		
PVOD 600	WL	a= 1.0199; k= 0.0102;	0.989	0.870
		n= 0.5741; b= 1.255x10^{-7}		
	SG	a= 1.0165; k= 0.0075;	0.991	0.684
		n= 0.6091; b= 1.171x10^{-6}		

In a general way, the Midilli's equation presented higher R^2 values (Table 5), than those observed for the Page's equation (Table 4). This model has been chosen as most suitable for describing different drying processes and material structures as yacon (Shi, Zheng, & Zhao, 2013), figs (Filho et al., 2015), sweet potato (Junqueira, Mendonça, & Corrêa, 2016) and lemon peel (Tasirin et al., 2014). And even this semi-empirical equation has been widely employed for portraying the moisture reduction during the drying, according to the Table 5, this equation can also be used for describing the water loss and the solid gain during the OD of vegetables.

4. CONCLUSION

The osmotic dehydration of different material structure (carrot, eggplant and beetroot) was studied, and the effect of VA was evaluated. In general way, vegetables with a porous structure (carrot and eggplant) were more sensitive to the pressure reduction, with notable intensification of mass transfer. Four mathematical models were employed for correlate the experimental WL and SG. The effective diffusivities were obtained according to the Fick's model and Fito & Chiralt's model. It was observed the same magnitude order of this parameter for all the vegetables and treatments. The 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. The Midilli semi-empirical equation presented higher R^2 (> 0.95) and it was related for describing the osmotic dehydration behavior of all the vegetables in all conditions.

5. ACKNOWLEDGMENTS

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ARTICLE 3

EFFECTS OF VACUUM PRESSURE AND MATERIAL STRUCTURE ON THE OSMOTIC DEHYDRATION VEGETABLES

Running title: Pulsed vacuum osmotic dehydration of vegetables

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Abstract - Pulsed vacuum osmotic dehydration (PVOD) is a widely used technique for reducing moisture content and water activity in biological products. This study aimed to analyze the effect of vacuum application (VA) on PVOD of beetroot, carrot, and eggplant slices, with respect to chemical (moisture, water activity, specific pigments, polyphenols, and sodium content), optical (color), mechanical (shrinkage, maximum stress, and elasticity), and structural (microstructure) properties. PVOD was conducted at three different vacuum pressures (0, 300 and 600 mmHg, for 10 min), during a total process time of 300 min. Osmotic processing was performed at 35°C by using a ternary osmotic solution [40% sucrose + 10% sodium chloride (w/w)]. Eggplant and carrot samples were more sensitive to VA, compared to beetroot. This was related to their porous and less compact structure. In general, VA reduced the moisture content and water activity, and preserved the carotenoid content. VA caused loss of betalain and phenolic acid, favored sodium uptake, and induced significant changes in the optical, mechanical, and structural properties, compared to the osmotic processing conducted at atmospheric pressure.

Keywords - Beetroot; carrot; eggplant; pulsed vacuum osmotic dehydration.

1. INTRODUCTION

Osmotic dehydration (OD) is often applied as a pretreatment, which induces the exchange of water and solutes, mainly in fruits and vegetables, allowing partial removal of water, solute uptake, and decrease in water activity (a_w). It offers some benefits such as reducing heat-induced damage to the nutritional and sensorial characteristics (preserving flavor, color, and inhibiting enzymatic browning) and energy saving (Herman-Lara et al. 2013; Ramya and Jain 2017).

This process is influenced by associated process variables and the characteristics of the material (moisture content, structure, porosity, and the geometric shape of the samples) (Dimakopoulou-Papazoglou and Katsanidis 2016; Lech et al. 2018; Sette et al. 2016). However, the influence of vacuum application (VA) on the structure of the material is related to the effects on mass transfer parameters. Few studies have focused on the changes to the chemical, optical, mechanical, and structural properties (Corrêa et al. 2016; Junqueira et al. 2017a). A reduction in the OD pressure during the initial period promotes an increase in the rate of mass transfer, by a process called pulsed vacuum osmotic dehydration (PVOD). VA causes the internal gases in the pores of the material to expand, expelling them by hydrodynamic mechanism (HDM) and increasing the surface area available for mass transfer because of the pressure difference (Fito 1994; Viana et al. 2014).

Vegetables show relevant differences in the composition and physicochemical properties. Beetroots (*Beta vulgaris* L.) are roots rich in polyphenols and a water-soluble nitrogenous pigment group, the betalains. This vegetable presents a compact structure, associated with lower porosity (Boukouvalas et al. 2006; Ravichandran et al. 2013). Carrot (*Daucus carota* L.) is an important vegetable rich in phytonutrients, in particular, carotenoids, minerals, and vitamins. Morphologically, this root consists of two fractions, namely, an inner xylem surrounded by an outer phloem (Kaur and Sogi 2016; Nahimana et al. 2011a). Eggplants (*Solanum melongena* L.) are fruits and a source of minerals, vitamins, and anthocyanins. Approximately 50% of its volume comprises voids, and therefore, it has been classified as a highly porous fruit (Russo et al. 2013).

These vegetables are basically composed of water, which limits their shelf life. Therefore, it is necessary to employ techniques that enhance their stability, such as dehydration. The aim of this study was to evaluate the influence of different vacuum pressure (VP; 0, 300, and 600 mmHg) on the chemical (moisture, a_w, specific pigments, and polyphenol and sodium content), physical (color), mechanical (shrinkage, maximum stress, and elasticity), and structural (microstructure) properties of osmodehydrated beetroot, carrot, and eggplant slices.

2. MATERIALS AND METHODS

2.1 Sample preparation

Fresh beetroots, carrots, and eggplants were purchased from a local market (Lavras, MG, Brazil) and stored in a refrigerator at 8°C ± 1°C until experimental use. All vegetables were washed with tap water, peeled, and sliced (2.00-cm length × 2.00-cm width × 0.40 ± 0.03-cm thickness) by using a stainless steel mold.

2.2 Osmotic processes

The osmotic solution was 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.Osmotic processing was performed in an osmotic dehydrator with temperature and inner pressure controls. The dehydrator was composed of a stainless steel chamber covered by a jacket, with water acting as the thermal fluid (Viana et al. 2014).

The total process time was 300 min. This period was chosen based on the results obtained in previous tests, considering the higher water loss and lower solid uptake (data not shown) during the dehydration of these vegetables. No pre-osmosis blanching was performed because blanching was reported to be detrimental to OD because of the loss of the semi-permeability of the cell membrane and reduction of nutrients and pigments (Kargozari et al. 2010). For all vegetables, the experiments were conducted at atmospheric pressure (OD) and under vacuum conditions (PVOD). For PVOD, a VP of 300 or 600 mmHg was applied to the system during the first 10 min of processing, and then, the local atmospheric pressure (101,32 kPa) was restored, and OD was conducted for the remaining process period (290 min).

The temperature was set at 35°C ± 1°C, and the solution-to-vegetable ratio was maintained at 1:10 (w/w) to prevent the dilution of the osmotic solution during experiments (Junqueira et al. 2017a). After osmotic processing, the samples were removed from the solution and immersed in a cold distilled water bath for 10 s to halt osmosis and to remove the excess solution. The surface of the samples was gently wiped with absorbent paper, and they were weighed and submitted for moisture content determination (AOAC 2010). All experiments were performed in four replicates.

2.3 Quality analyses

Quality analyses were performed using fresh and osmodehydrated vegetables.

2.3.1 – Moisture content and a_w

The moisture content of the vegetables was determined using a vacuum drying oven (SL 104/40; Solab, Piracicaba, Brazil) at 70°C until constant weight was achieved (AOAC 2010). a_w determination was performed using a hygrometer (Aqualab, 3-TE model; Decagon Devices, Inc., Pullman, WA, USA). The analyses were performed in triplicate.

2.3.2 – Betalain content

Betalain content (BC) was separately determined as betacyanin content (BCC) and betaxanthin content (BXC). This analysis was performed for beetroot samples. Approximately 1.0 g of the sample was dissolved in 10.0 mL of 50% ethanol. The mixture was stirred in a shaker at 800 rpm for 10 min, and the homogenate was centrifuged at 6000 rpm for 10 min. The supernatant was collected at two repeat stages of centrifugation to ensure maximum extraction, which was further used for the determination of BC (Nistor et al. 2017; Ravichandran et al. 2013). The BCC and BXC in the extracts were determined spectrophotometrically (Cary 50;Varian, Australia) at 538 and 480 nm, respectively. The absorbance value was used to calculate the concentration of betalain in each sample (Eq. 1), by using the following formula:

$$BC = \frac{Abs\ DF\ MW}{\mathrm{E}^{1\%}_{1\ \mathrm{cm}}} 1000 \tag{1}$$

where Abs is the absorbance, DF is the dilution factor, MW is the molecular weight [g /mol], and $E^{1\%}$ is the molar extinction coefficient in water. The MW of BCCs (red-violet color) was 550 g /mol, while that of BXCs (yellow-orange color) was 308 g /mol. The $E^{1\%}$ of each betalain in water ($E^{1\%}{}_{1cm}$) was 60000 (BCCs) and 48000 (BXCs). The results of four replicates were expressed as mg /100 g of betalains (d. b.).

2.3.3 – Carotenoid content

Carotenoid content (CC) was determined for carrot according to the method described by Rodriguez-Amaya (2001), with modifications wherein 1.0 g of carrot was used. The carotenoids were exhaustively extracted with cold acetone, partitioned into petroleum ether, and washed with distilled water. Absorbance was measured by UV/Vis spectrophotometry (Cary 50, Varian, Australia) at 444, 450, and 470 nm, which correspond to α-carotene, β-carotene, and lycopene determination, respectively. These are the most abundant carotenoids found in carrots (Nahimana et al. 2011a). Carotenoid content was determined according to Eq.2:

$$CC = \frac{Abs\ V}{E^{1\%}_{1\ cm}\ P} 1000 \qquad (2)$$

where Abs is the absorbance, V is the total extract volume [mL], and P is the sample weight [g]. The $E^{1\%}$ of each carotenoid in petroleum ether ($E^{1\%}_{1\ cm}$) was 2800 (α-carotene), 2592 (β-carotene), and 3450 (lycopene). The results of four replicates were expressed as mg /100 g of carotenoids (d. b.).

2.3.4 - Polyphenol content

The polyphenols for high-performance liquid chromatography (HPLC) were extracted from 2.5 g of pulp and a 20 mL solution containing 70 % methanol in water (v/v), according to Ramaiya et al. (2013) with modifications. Briefly, the samples were homogenized and placed in an ultrasonic bath at 20 °C for 60 min. The extracts were centrifuged at 1400 × g for 15 min at 4 °C and filtered through Whatman no. 2 filter paper. The extracts were refiltered through regenerated cellulose filters (0.45-μm-thick; Millipore, Bedford, MA, USA) and stored at -18°C until analyses.

Chromatographic analyses were performed using an Ascentis C18 5-lm (250 mm × 4 mm) column. The mobile phase consisted of 2% (v/v) acetic acid in water (mobile phase A) and 70:28:2 methanol/water/acetic acid (mobile phase B), set to a flow rate of 1.0 mL /min and conducted using a gradient elution program and a 65-min-long run time. The injection volume was 20 μL.

Analyses were performed at 15 °C. The phenolic compounds generated a UV–Vis spectrum in the HPLC chromatogram at 280 nm. Quantitative determination of compounds was performed by comparison of the dose–response curves based on the *m/z* data by using authentic polyphenol standards. The results of three replicates were expressed as mg /100 g (d. b.).

2.3.5 – Sodium content

Sodium content was determined according to Malavolta (1997). The dried samples were mashed, and the powdered material was subjected to nitric-perchloric acid digestion, the material being analyzed by flame photometer (Micronal, B-262 model; São Paulo, Brazil). The results of three replicates were expressed as mg /100 g of sodium (d. b.).

2.3.6 – Color parameters

Color parameters were measured using a colorimeter (Minolta, Model CR-400; Osaka, Japan). The parameters were recorded using CIE $L^*a^*b^*$ uniform color space (CIE-Lab) with D65 as the illuminant. L^*, a^*, and b^* were quantified for each sample. These color parameters were used to calculate the total color difference (ΔE) (Eq. 3) (Junqueira et al. 2017b). Seven samples were evaluated for each treatment.

$$\Delta E = \sqrt{(L^*_t - L^*_0)^2 + (a^*_t - a^*_0)^2 + (b^*_t - b^*_0)^2} \qquad (3)$$

where the subscripts 0 and t indicate the color parameters of the samples after 300 min of treatment and fresh samples, respectively.

2.3.7–Volumetric shrinkage

This parameter was determined by measuring the area and thickness of the samples. The area was measured by image analysis using the free software Image J® 1.45s, which provides the sample area by converting the pixels in the image into real dimensions, from a known scale (Nahimana et al. 2011a). For each sample, the thickness at five different points was determined using a digital caliper (DC-6 model; Western, China). The dimensionless volume (β) was determined according to the Eq. 4. A higher β value indicates lesser shrinkage (Junqueira et al. 2017c).

$$\beta = \frac{V_f}{V_0} \tag{4}$$

where V_f indicates the apparent volume after 300 min of process [m^3] and the V_0 indicates the initial volume [m^3].

2.3.8 – Texture

Textural characteristics were measured by uniaxial compression tests by using a texturometer (TA-X2T; Stable Micro Systems, Surrey, England). The diameter and height of the cylindrical samples were 13.80 ± 0.03 and 3.00 ± 0.50 mm, respectively. Vegetable texture was studied based on the load and strain curves recorded during the compression of samples by using two horizontal parallel plates (70-mm length), with the sample being placed at the center of the lower plate. The crosshead speed was 60 mm /min. For beetroot and carrot slices, the compression was about 40% of the initial height, and for the eggplant samples, it was about 70% of the initial height. Eight replicates were performed for each treatment. Maximum stress (σ) [kPa] and elasticity modulus (E) [kPa] were determined according to the methods described by Ferrari et al. (2011) and Moreira et al. (2008).

2.3.9 - Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to analyze the internal structure of the vegetables, before and after osmotic processing. Initially, all samples were fixed with modified Karnovsky solution for 24 h. These samples were immersed in a cryoprotectant solution (30% glycerol in water) for 30 min and transversely sectioned with a scalpel after immersion in liquid nitrogen. These sections were rinsed in distilled water and post-fixed in 1% osmium tetroxide aqueous solution for 1 h, and then dehydrated in an increasing series of acetone solution (25%, 50%, 70%, and 90% for 10 min, and thrice for 10 min at 100%). After dehydration, the samples were subjected to drying on a Critical Point Drier (CPD 030®; Balzers, Germany). Then, the samples were placed on aluminum supports and covered with a film of aluminum foil by using double-sided carbon tape. Finally, all samples were covered with a layer of gold using a Sputter coater (SCD 050®; Balzers, Germany) and observed using a microscope (LEO Evo 40; Zeiss, Cambridge, UK) operating at 20 kV, with a working distance of 8.5 mm (Freitas et al. 2017).

2.4 – Statistical analyses

The results were evaluated by one-way ANOVA at a probability level of 0.95. In case of significant effects ($p \leq 0.05$), the means were compared using the Tukey's test. These analyses were performed using the software Statistica 8.0 (Statsoft Inc., Tulsa, USA). All experiments were carried out in triplicate.

3 . RESULTS AND DISCUSSION

3.1 Moisture content and a_w

The moisture content (MC) and a_w of the vegetables are presented in Table 1. A reduction in the moisture content and a_w ($p \leq 0.05$) was observed for all osmodehydrated products. The products subjected to OD presented a reduction because of dewatering and solute impregnation (sugar and sodium chloride, in this study), as extensively discussed in available literature (Ketata et al. 2013; Mendonça et al. 2016; Porciuncula et al. 2013). According to Fellows (2006), the moisture content and a_w of intermediate-moisture foods (IMF) ranges from 15 kg /100 kg to 50 kg /100 kg and from 0.65 to 0.90, respectively. Thus, all osmotically treated vegetables can be classified as IMF, showing that osmotic processing is an efficient method for pretreatment of the evaluated vegetables (Table 1).

Table 1 –Moisture content (MC) [kg /kg] and water activities (a_w) of fresh and osmodehydrated vegetables

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
		Beetroot		
MC	0.892±0.008[a]	0.422±0.003[bc]	0.442±0.004[b]	0.408±0.013[c]
a_w	0.981±0.002[a]	0.851±0.005[b]	0.854±0.003[b]	0.852±0.007[b]
		Carrot		
MC	0.872±0.009[a]	0.496±0.006[b]	0.493±0.007[b]	0.460±0.004[c]
a_w	0.980±0.002[a]	0.887±0.002[b]	0.881±0.002[c]	0.859±0.002[d]
		Eggplant		
MC	0.931±0.003[a]	0.506±0.007[b]	0.472±0.007[c]	0.442±0.009[d]
a_w	0.991±0.004[a]	0.860±0.003[b]	0.843±0.002[c]	0.826±0.003[d]

Beetroot showed slight differences after OD. The MC of osmodehydrated samples was similar to that of samples subjected to PVOD; no difference was observed in the final a_w (Table 1). Carrot presented a significant difference in the MC ($p \leq 0.05$) after OD. A lower MC was observed in case of samples subjected to PVOD at 600 mmHg. The a_w also reduced, as the VP increased. In case of eggplant, VP directly influenced the reduction in moisture and a_w ($p \leq 0.05$). During PVOD, the capillary pores of the vegetables are filled, resulting in an increase in the solid–liquid surface contact area, enhancing the mass transfer rate (Ahmed et al. 2016; Fito 1994). Carrot and eggplant present a less compact structure than beetroot, and they are more sensitive to VA. Viana et al. (2014) observed little effect of VA during the OD of fodder palm, which has a sturdy structure. On the other hand, porous tissues such as those in eggplants (Junqueira et al. 2017a), pears (Moreno et al. 2011), and mangoes (Lin et al. 2016), are related to significant reduction in moisture and a_w after vacuum osmotic processing.

3.2 Betalain content

The BCC and BXC in fresh and osmodehydrated beetroots are presented in Table 2.

Table 2 – Betalain content (BC) and carotenoid content (CC) of fresh and osmodehydrated beetroot and carrot [mg /100 g] (d.b).

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
		Beetroot		
BCC	1.36±0.01[a]	0.82±0.01[b] (60.29 %)	0.76±0.02[c] (55.88 %)	0.73±0.03[c] (53.67 %)
BXC	0.96±0.01[a]	0.53±0.01[b] (55.20 %)	0.53±0.01[b] (55.20 %)	0.51±0.02[b] (53.12 %)
		Carrot		
α-carotene	58.40±5.67[a]	20.79±3.38[c] (35.59 %)	41.80±8.61[b] (71.57 %)	36.15±6.84[b] (61.90 %)
β-carotene	64.85±6.17[a]	22.99±3.87[c] (35.45 %)	46.09±9.69[b] (71.07 %)	39.68±7.57[b] (61.18 %)
lycopene	41.68±3.92[a]	14.88±2.42[c] (35.70 %)	24.04±[b] (57.67 %)	25.87±4.96[b] (62.08 %)

Osmotic treatment significantly affected the BCC and BXC in beetroot samples ($p \leq 0.05$). The high BCC and BXC in the fresh product were reduced after osmotic processing. There was a reduction in the BC, with the retention ranging from 53.67% to 60.29% and 53.12% to 55.20% for BCC and BXC, respectively. In general, VA reduced the BCC ($p \leq 0.05$), and VP had no effect on the BXC, as observed in osmodehydrated beetroot samples (Table 2). The loss of these pigments is related to leaching, because both compounds are water-soluble. Moreover, the long processing time and exposure to light, oxygen, and temperature promotes the isomerization, deglycosylation, dehydrogenation, decarboxylation, and hydrolysis of the betalains, resulting in a gradual reduction of the red color and appearance of a brown shade (Celli and Brooks 2016; Nistor et al. 2017; Paciulli et al. 2016).

3.3 Carotenoid content

The CC in fresh and osmodehydrated carrots are presented in Table 2. A significant difference ($p \leq 0.05$) was noted for all known carotenoids (α-carotene, β-carotene, and lycopene). The predominant carotenoid found in the carrot was β-carotene, in accordance with the findings of Kaur and Sogi (2016) and Sulaeman (2001). α-Carotene, β-carotene, and lycopene contents reduced after OD (Table 2). Dehydration causes gradual breakdown in the cellular structure, with subsequent loss of turgor pressure and consequent disruption of carrot cells (Nieto et al. 2013). Mendonça et al. 2017 also reported CC reduction in pequi slices after osmotic processing. They suggested that this reduction could not be attributed to the leaching flux, as these molecules are hydrophobic. Carotenoids are highly unsaturated molecules susceptible to degradation or isomerization followed by cleavage, in particular under the influence of heat and light during processing (Saini et al. 2015).

Lower CC losses were observed after treatments conducted with VA (PVOD, 300 mmHg, and PVOD, 600 mmHg), according to Table 2, and a significant difference was noted ($p \leq 0.05$) for all the analyzed carotenoids (α-carotene, β-carotene, and lycopene). High carotenoid retention obtained in

PVOD could be attributed to the fact that the creation of vacuum enhances cell breakdown, by internal gas removal, releasing the carotenoids attached to the more available cells, compared to OD, facilitating their extraction. Furthermore, carotenoid degradation depends on the presence of oxidative enzymes, which can be diminished by the lack of oxygen (Arkoub-Djermoune et al. 2016; Carvalho et al. 2014; Corrêa et al. 2016; Guiamba et al. 2016). At mild temperatures, lycopene may isomerize to the cis-isoform during dehydration. At 35°C, lycopene loss primarily occurs through oxidation, which proceeds faster than isomerization and degradation (Xianquan et al. 2005). Lycopene isomerization improves its bioavailability from food matrices (Arkoub-Djermoune et al. 2016).

3.4 Polyphenol content

With respect to the polyphenol profile, significant differences have been noted in the post-processing levels of analyzed phenolic acids ($p \leq 0.05$; Table 3).

Table 3 – Polyphenol content of fresh and osmodehydrated vegetables [mg 100 g^{-1}] (d.b.)

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
		Catechin		
Beetroot	215.48±4.31^{a}	90.17±3.93^{c}	116.67±4.20^{b}	75.17±2.88^{d}
Carrot	36.89±2.89^{a}	28.72±1.44^{b}	31.42±2.06^{b}	21.09±0.10^{c}
Eggplant	21.26±1.66^{a}	12.22±1.24^{b}	13.42±0.61^{b}	2.40±0.15^{c}
		Chlorogenic acid		
Eggplant	367.71±11.43^{a}	219.07±11.42^{b}	183.17±8.29^{c}	82.92±3.62^{d}

Average value ± standard deviation. Mean followed by different letters in the row differs significantly ($p < 0.05$), according to Tukey's test.

Catechin was identified in all vegetables, with a higher concentration in beetroot samples, but chlorogenic acid was identified only in eggplant samples. In this study, other analyzed acids (trans-cinnamic, caffeic, vanillic, p-coumaric, ferulic, and o-coumaric) were not identified, neither in fresh nor osmodehydrated samples. A reduction in the catechin content was observed in all osmodehydrated samples (Table 3). This could be attributed to the lixiviation. During osmotic processing, as the water is removed, some hydrophilic bioactive compounds migrate from the plant cells to the osmotic solution (Luchese et al. 2015).

Moreover, some loss can be attributed to degradation, because the treatment was conducted for longer time at a temperature close to the optimum temperature for enzymatic action, favoring oxidation (Gonçalves et al. 2017). Moreno et al. (2016) observed significant differences in catechin content in fresh and osmodehydrated blueberries. They concluded that VA reduced catechin content, compared to OD. Chlorogenic acid is the main antioxidant phenol in eggplants (Lo Scalzo et al. 2016), whose content is reduced by PVOD, by almost 50~75% (Table 3). This result can be attributed to mass transfer intensification by VA. Nevertheless, the loss of this polyphenol acid was also observed during OD, and was associated with oxidative and hydrolytic modifications. In a general way, vacuum creation reduced the polyphenol content, compared to OD.

As a general remark, the retention of betalains, carotenoids, and polyphenols is desirable, because they present a wide range of biological activity, including antioxidant, anti-inflammatory, and anti-cancer properties (Celli and Brooks 2016; Del Rio et al. 2013).

3.5 Sodium content

The sodium content of beetroot, carrot, and eggplant (fresh and osmodehydrated) is shown in Table 4. There was a significant increase ($p \leq 0.05$) in the sodium content of all vegetables after osmotic processing. During osmosis, solute flux occurs from the osmotic solution to the material, increasing its concentration in the food material (Brochier et al. 2015; Corrêa et al. 2010; Ramya and Jain 2017).

Table 4 – Sodium content of fresh and osmodehydrated vegetables [mg /100 g] (d.b.)

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
Beetroot	6.94± 0.64[c]	4169.64± 17.07[a]	4016.93± 55.14[b]	4232.06± 80.67[a]
Carrot	69.03± 5.92[d]	4789.66± 18.52[c]	5030.26± 30.09[b]	5217.84± 51.75[a]
Eggplant	7.52± 0.61[c]	6024.55± 154.63[b]	6242.29± 31.43[ab]	6547.07± 333.88[a]

Average value ± standard deviation. Mean followed by different letters in the row differs significantly ($p < 0.05$), according to Tukey's test.

In general, VP increased sodium incorporation. Eggplant is a highly porous entity and seemingly more sensitive to the HDM occurring during VA, which improves the surface area for mass transfer, by replacing the internal gases by an osmotic solution (Betoret et al. 2015; Boukouvalas et al. 2006; Vallespir et al. 2018). Junqueira et al. (2017a) reported significant differences in the sodium content of osmodehydrated eggplant slices with and without VA.

Beetroot and carrot are considered low porosity products (Boukouvalas et al. 2006), and might be less sensitive to VA, presenting lower sodium incorporation (compared to eggplant) (Table 4). Although carrot is a low porosity product, it is composed of a peripheral cortex layer and a central stele (vascular tissue) that presents a fibrous and porous structure to facilitate HDM action (Aguiló-Aguayo et al. 2017; Nahimana et al. 2011a; Parthasarathi and Anandharamakrishnan, 2014). On the other hand, Corrêa et al.(2016) observed a reduction in sodium content during the osmotic dehydration of tomatoes in ternary solutions in the presence of vacuum, which suggests that the structure and composition of the product significantly affect the solute gain response.

3.6 Color

The color of the osmodehydrated vegetables can be indicative of the retention of pigment-based nutrients as betalains, carotenoids, and chlorophyll (Aral and Beşe 2016). Table 5 presents the mean values and standard deviations of the luminosity (L^*), redness (a^*), yellowness (b^*), and total color difference (ΔE) observed for beetroot, carrot, and eggplant (fresh and osmodehydrated) slices.

The beetroot slices subjected to OD and PVOD presented a significant decrease ($p \leq 0.05$) in the L^* and a^* values (Table 5).When BC decreases (Table 2), the decrease in lightness and redness are recorded. Fresh beetroot contains very high amounts of red-violet pigments, and after osmotic processing, the sample surface became darker; hence, the redness reduces (Gokhale and Lele 2014).Statistical difference was observed for the b* parameter ($p \leq 0.05$); VP treatment with 0 and 300 mmHg presented higher yellowness values, while the treatment conducted at 600 mmHg tended to move toward blueness, compared to fresh beetroot subjected to other treatments. According to Table 5, VP promoted higher ΔE ($p \leq 0.05$) in beetroot samples. In OD, color variation is a very complex aspect related to the moisture content, solute incorporation, and pigment content (loss or concentration).

Moreover, in PVOD, the presence (or absence) of occluded gas affects this quality parameter (Corrêa et al. 2014). Since significant differences were observed in the L^*, a^*, and b^* color parameters, ΔE differences were expected.

Higher ΔE values, for the beetroot samples, were probably caused by the intensification of mass transfer occurring because of application of reduced pressure during the initial period of the process, which lead to leaching of pigments (Table 2).

Table 5 - Values of color parameters (L^*, a^*, b^* and ΔE) obtained for the vegetables (fresh and osmodehydrated).

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
		Beetroot		
L^*	32.23±0.56^a	28.69±0.64^b	26.13±1.45^c	27.22±1.51^{bc}
a^*	21.85±1.71^a	17.09±0.89^b	17.55±1.87^b	10.29±0.91^c
b^*	0.28±0.12^b	1.94±0.47^a	2.25±0.50^a	-0.56±0.09^c
ΔE	-	5.46±1.37^b	7.42±0.77^a	7.87±0.42^a
		Carrot		
L^*	56.45±0.81^a	51.65±1.87^c	53.36±0.63^b	53.86±0.65^b
a^*	16.83±0.52^d	21.65±1.23^c	23.59±0.36^b	28.17±0.82^a
b^*	28.66±1.19^d	39.12±1.20^c	42.55±1.10^b	46.46±0.86^a
ΔE	-	3.60±0.62^c	15.75±1.91^b	20.29±0.87^a
		Eggplant		
L^*	84.75±0.94^a	73.13±1.47^b	66.24±1.57^c	63.60±1.84^d
a^*	-4.24±0.26^c	2.07±0.54^a	1.32±0.84^{ab}	0.92±0.61^b
b^*	14.93±1.07^d	16.93±1.02^c	18.92±1.28^b	20.98±1.07^a
ΔE	-	14.25±1.19^c	20.97±0.79^b	23.26±1.86^a

Average value ± standard deviation. Mean followed by different letters in the row differs significantly ($p < 0.05$), according to Tukey's test.

For carrot samples, the L^* value decreased significantly ($p \leq 0.05$) in all osmodehydrated samples. An increase was observed in the a^* and b^* values (Table 5), compared to the fresh samples. An increase in redness and yellowness was also reported by Amami et al. (2014) during the OD of carrots, which was related to the intensification of orange coloration. However, the reason underlying these changes in the color parameters (Table 5) and CC (Table 2) is not clear. However, the presence of heat and light causes cis/trans isomerization of the carotenoids, altering their biological activities and discoloring the food (Saini et al. 2015).

For carrot, the higher ΔE was due to the intensification of the a^* and b^* values (Table 5). Higher the pigment retention, higher was the ΔE (VP = 600 mmHg). As observed for beetroot samples, the pigments are unstable during osmotic processing, contributing to the browning. Moreover, oxidative enzymatic activity makes the sample darker (Betoret et al. 2015; Paciulli et al. 2016). Statistical analysis showed a significant difference ($p \leq 0.05$) in the color parameters of the osmodehydrated eggplant samples (Table 5). When the eggplant slices were subjected to OD and PVOD, a significant decrease ($p \leq 0.05$) was noted in the L^* values. Darker samples were obtained after PVOD. Similar results were reported by Junqueira et al.(2017a) after the OD of eggplant slices.

Since the eggplant is a highly porous fruit (Russo et al. 2013), the reduction in L^* value could be related to the removal of internal gases and the impregnation of the tissues with the osmotic solution after restoration of atmospheric pressure (Moreno et al. 2013). A significant increase in the a^* and b^* values ($p \leq 0.05$) was observed in osmodehydrated eggplant samples. Contrary to that observed for beetroot and carrot, the eggplant presents lower pigment concentration, and the intense browning is mainly related to the action of oxidative enzymes, since the eggplant is rich in polyphenol oxidase, peroxidase and its substrate (phenolic compounds) (Arkoub-Djermoune et al. 2016; Mishra et al. 2013).

During eggplant processing, enzymatic browning starts immediately after cutting and rapidly increases as the fruits are exposed to air. This color change is limited by tissue integrity, rather than by substrate or enzyme availability, which could explain the lower L^* values, with a consequent increase in ΔE ($p \leq 0.05$) after vacuum treatment (Table 5), because VP promotes intense cellular decompartmentalization (Moreno et al. 2012; Zaro et al. 2015). Moreno et al. (2011) noted a higher ΔE in the VA-treated apple samples, when compared to the ΔE of samples subjected to osmotic processing at atmospheric pressure.

3.7 Volumetric shrinkage

The dimensionless volume (β) of the samples after osmotic processing is presented in Fig. 1. All treatments exhibited volume reduction. OD promotes a reduction in the moisture content, decreasing the tension exerted by the liquid against the cell wall, which can cause pressure imbalance between the interior and exterior. This can generate contracting stress, resulting in material shrinkage or collapse (Junqueira et al. 2017c; Nahimana et al. 2011b; Toğrul and İspir 2007).

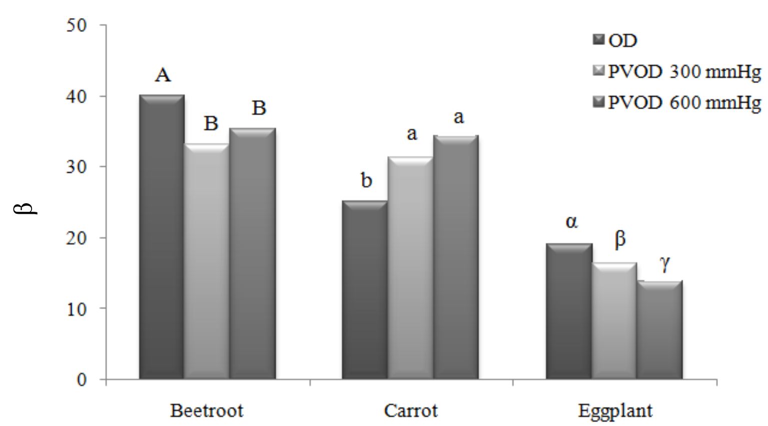

Fig. 1 - Dimensionless volume (β) of osmodehydrated vegetables. Mean followed by different uppercase (beetroot), lowercase (carrot) and greek (eggplant) letter differs significantly ($p < 0.05$), according to Tukey's test.

Regarding volumetric shrinkage, different vegetables showed different patterns. Eggplant presented a lower β (as low as 13.81%), indicating higher shrinkage, whereas beetroot exhibited a higher β (lower shrinkage). This differential behavior could be associated to the different physicochemical characteristics of the vegetables, such as chemical composition, mechanical properties, initial porosity, and apparent volume, which can generate different types of stress during the process (Mayor et al. 2011; Parthasarathi and Anandharamakrishnan 2014). Mújica-Paz et al.(2003) reported that high porosity products present greater susceptibility to mechanical deformation. Higher volume reduction in eggplant can be explained by its high porosity, while the lower reduction in beetroot can be related to its low porosity (Boukouvalas et al. 2006).

VP influenced the extent of shrinkage ($p \leq 0.05$) observed in all vegetables. According to Fig. 1, VA caused a reduction in the β of beetroot and eggplant samples, which suggests that on adopting reduced pressure-based treatment, the expansion and escape of gases that occurs after the restoration of atmospheric pressure also contributes to volumetric shrinkage, although these vegetables exhibit different properties. The opposite behavior was shown by carrot samples (Fig. 1), with an increase in β values and reduced shrinkage being observed when vacuum was employed. This behavior is probably related to the heterogeneous tissue composition of carrot, composed of a peripheral cortex (parenchymatous cells) and a core (xylem tracheary elements),which may cause differential response to OD (Nahimana et al. 2011a). Lin et al. (2016) also concluded that PVOD can reduce the shrinkage observed in mango cubes.

3.8 Texture

The maximum stress (σ) and elasticity modulus (E) of the fresh and osmodehydrated vegetables are shown in Table 6. There was a significant reduction in these parameters after OD ($p \leq 0.05$).

Table 6 – Stress at rupture (σ) and elasticity modulus (E) of fresh and osmodehydrated vegetables [kPa].

	Fresh	OD	PVOD 300 mmHg	PVOD 600 mmHg
		σ		
Beetroot	108.484± 6.007[a]	2.580± 0.359[b]	1.966± 0.283[b]	1.507± 0.043[b]
Carrot	184.920± 3.557[a]	10.128± 1.293[b]	7.333± 0.819[c]	7.432± 1.257[c]
Eggplant	71.424± 2.336[a]	53.051± 2.768[b]	22.218± 2.369[c]	27.083± 6.515[c]
		E		
Beetroot	10.059± 0.782[a]	2.121± 0.202[b]	1.648± 0.203[b]	1.554± 0.016[b]
Carrot	11.798± 1.183[a]	5.070± 0.322[b]	4.066± 0.255[c]	5.040± 0.376[b]
Eggplant	21.457± 0.885[a]	2.142± 0.241[c]	2.576± 0.244[c]	4.043± 0.551[b]

Average value ± standard deviation. Mean followed by different letters in the row differs significantly ($p < 0.05$), according to Tukey's test.

According to Moreno et al. (2013) and (2011), when the vegetable tissue is subjected to osmotic processing, in addition to moisture removal, textural changes are dependent on the physical and chemical changes due to the degradation of the middle lamella and solute incorporation, which causes loss of turgor and movement of ions from the cell wall to the media. These changes create internal stress, leading to cellular disruption and plasmolysis (such as volumetric shrinkage presented in Table 5), causing tissue softening, which might be the reason for the reduction in stress at rupture after osmotic processing (Castelló et al. 2009).

Such a reduction in σ after osmotic treatment, with consequent loss of firmness, was also observed in eggplant (Junqueira et al. 2017a), pear (Oliveira et al. 2016), mango (Lin et al. 2016), pumpkin (Silva et al. 2011), and melon (Ferrari et al. 2011). When fresh and osmodehydrated vegetables are compared, higher firmness maintenance was observed for the eggplant (Table 6). This occurred because of the high porosity of this fruit, which causes the solid matrix to undergo minor deformation, thus preserving the food structure (Gras et al. 2002). For beetroot samples, VP did not have a significant effect on the σ values (Table 6). Comparing OD and PVOD for pear slices, Moreno et al. (2011) concluded that no differences were reported in the firmness values.

However, vacuum creation significantly reduced the σ values for carrot and eggplant slices ($p \leq 0.05$;Table 6). During PVOD, the exchange of internal gases and liquids occurred in the open pores, with the help of the osmotic solution, because of the pressure change-induced action of HDM. More intense deformation and rupture were noted, with a decreasing effect on σ. For these vegetables, VA susceptibility is related to the porous structure of eggplant and the lack of internal structural uniformity and heterogeneity of the carrot, which, in general, presents low porosity. Even the core tissue (mainly represented by xylem-type cells) show some voids (Nahimana et al. 2011a).

Gras et al. (2002) pointed that the carrot tissue presented almost no intercellular space, and that no significant HDM was expected. On the other hand, those authors observed that the vascular system of this vegetable (xylem and phloem elements) might be related to HDM, under conditions of reduced pressure. The system is composed of tracheary elements in the central zone of fresh carrots, wherein empty or full spaces can be found. They also observed that the vessels were separated by water-permeable membranes, and when water pressure in the system decreased, the vessel elements closed to avoid progressive plant dehydration.

With respect to the elastic modulus (E) (Table 6), the osmodehydrated eggplant presented a remarkable decrease (compared to the fresh sample), reaching up to 90% elasticity loss, because of gas replacement during osmotic processing.

3. 9 Microstructure

To evaluate the impact of osmotic processing on vegetable structure, the samples were analyzed using a scanning electron microscope (SEM). The differences between fresh and osmodehydrated tissue are shown in Figs. 2-4.

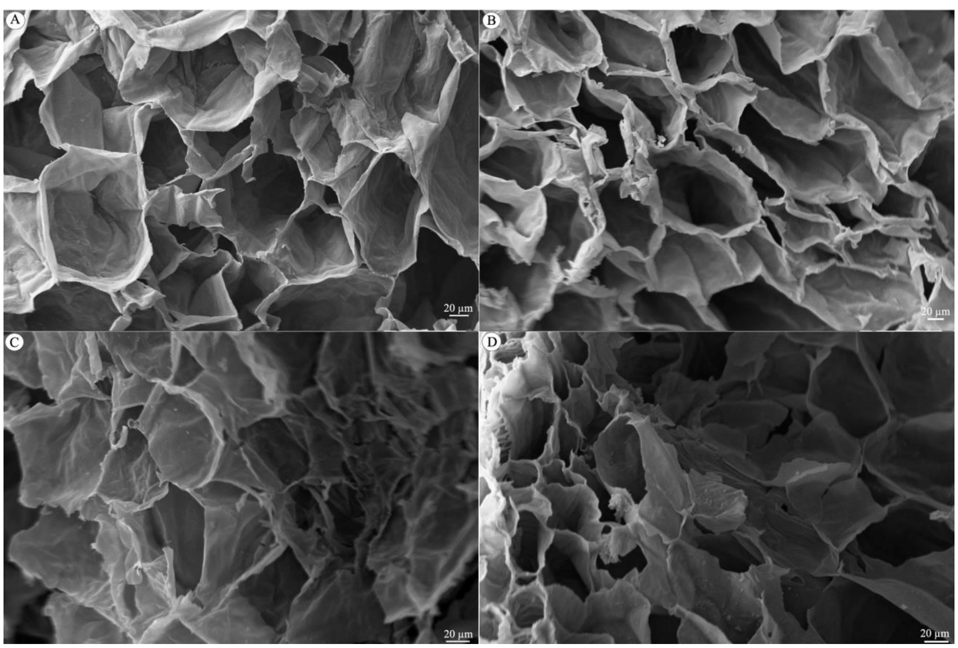

Fig. 2 - Microstructure of fresh (a) and osmodehydrated OD (b), PVOD 300 mmHg (c) and PVOD 600 mmHg (d) beetroot slices.

For fresh beetroot (Fig. 2a), carrot (Fig. 3a), and eggplant (Fig. 4a) tissues, the cells were round and long in shape, and tightly connected to each other. Similar findings were pointed out by Oliveira et al. (2016), Salazar-López et al. (2015), and Corrêa et al. (2015) for fresh tissues of pear, pineapple, and tomato, respectively. For samples subjected to OD (Figs. 2b, 3b, and 4b), the cells were irregularly shaped and moderately collapsed with tissue disruption. During osmotic treatment, the cell walls break down, intercellular contact is decreased, plasmolysis occurs, and the cell structure of the samples collapses (Deng and Zhao 2008; Mayor et al. 2008; Vallespir et al. 2018). Brochier et al. (2015) studied yacon slices treated by OD and observed plasmolysis caused by the loss of water from the cytoplasm, in addition to deformed, collapsed, and nonturgid cells.

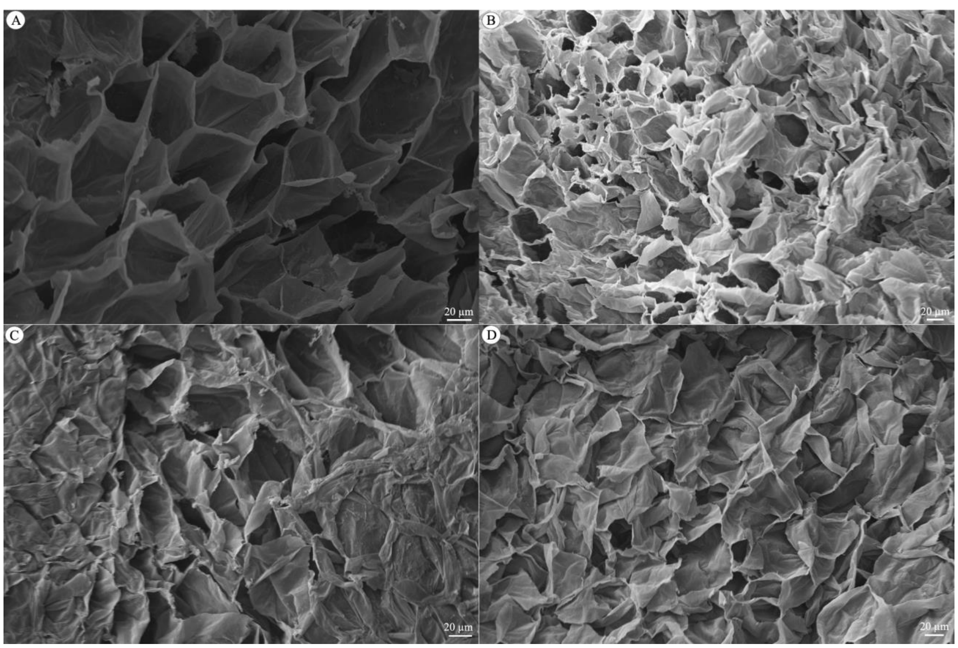

Fig. 3 - Microstructure of fresh (a) and osmodehydrated OD (b), PVOD 300 mmHg (c) and PVOD 600 mmHg (d) carrot slices.

Osmotic dehydration is known to cause changes in the structure of plant tissue. During OD treatment, the mass transfer of water from the tissue into the osmotic solution and the solute from the osmotic solution into the intercellular and extracellular spaces of the tissue leads to structural damage to the cells at the microscopic level (Nowacka et al. 2014; Nowacka and Wedzik 2016).

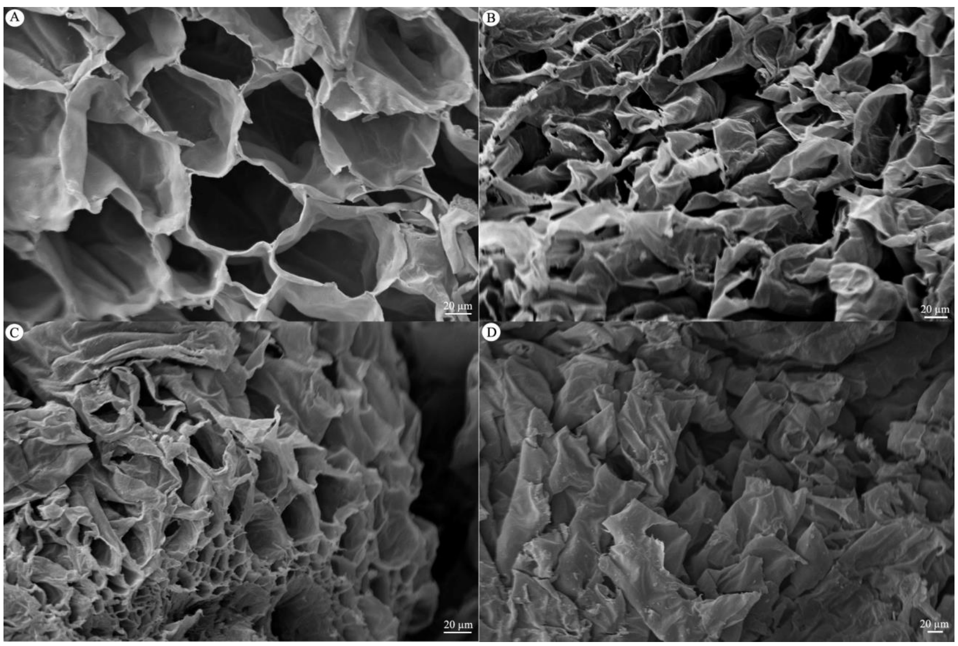

Fig. 4 - Microstructure of fresh (a) and osmodehydrated OD (b), PVOD 300 mmHg (c) and PVOD 600 mmHg (d) eggplant slices.

For PVOD treatment (Figs. 2c, 2d, 3c, 3d, 4c, and 4d), changes to the cellular structure were observed for all vegetables. Such changes are related to the different mass transfer parameters and textural changes (Table 6). In general, higher the water loss and solid gain, higher is the cellular tissue damage during osmotic processing. The removal of occluded gases also influences the cellular structure, and unlike OD, the cells shrank, acquiring an irregular shape (Deng and Zhao 2008; Nieto et al. 2013). Lin et al. (2016) observed changes in the mango tissues subjected to PVOD, who attributed the microstructural changes, occurring after restoration of atmospheric pressure, to cell porosity. They also concluded that the expansion of gases leads to an increase in the intercellular space, in addition to alteration of cell membrane permeability.

4. CONCLUSIONS

The present work showed that the VA promotes distinct responses to different matrix food. The PVOD process induced great changes in the vegetables, but it was more pronounced in eggplant samples, followed by carrot samples, which present a more porous structure, as a result of the expansion of gas in the cell pores of the tissues. The VA reduced the moisture content and the water activity, and preserved the carotenoid content in carrot samples. Compared to OD, the PVOD lead to losses in betalain and polyphenol contents, higher sodium uptake and it influenced the color parameters. The noted variations of vegetables tissues well supported the changes in the mechanical and structural characteristics. It was concluded that retention of food components, shrinkage, texture and microstructure of the osmotically dehydrated products were associated of both VA and biological material structure.

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THIRD SECTION

GENERAL CONCLUSION

The osmotic dehydration of vegetables (eggplant, carrot and beetroot) was achieved in different conditions. In all treatments, changes in optical, chemical and mechanical properties were observed after the osmotic processes.

Evaluating the effects of vacuum application and the use of alternative solutes as osmotic agents during the OD of eggplant slices, it was observed an increase in the water loss and water activity reduction when the reduced pressure was applied in the first minutes of process. The substitution of NaCl by KCl and $CaCl_2$ was shown to be feasible, potentially producing a healthy product without negatively affecting the nutritional, physical or mass transfer parameters.

In general way, it was observed that vegetables with a porous structure (carrot and eggplant) were more sensitive to the pressure reduction, with notable intensification of mass transfer in PVOD processes. According to the mathematical modeling, some established phenomenological presented a lack of fit, in relation to the experimental data obtained in this study.