OSMOTIC DEHYDRATION OF PUMPKIN (*Cucurbita moschata*) IN SUCROSE AND SUCROSE-SALT SOLUTIONS. EFFECT OF SOLUTION COMPOSITION AND SAMPLE SIZE

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Abstract --- Mass transfer during osmotic dehydration (OD) of Anco pumpkin cubes was studied as a function of sample size and composition and concentration of sucrose dehydrating solution. Models of Azuara and Hawkes & Flink were adjusted to the experimental data and optimal dehydrating conditions could be specified. For equal solutes concentration, higher weight losses were reached for smaller size samples. The addition of 2% NaCl increased Water Loss (WL) and lowered Solids Gain (SG) for the solution of higher sucrose content. Prediction capacity of the simplified models was very good under the tested working conditions. In brief, OD can be optimized using 1 cm side cubes dehydrated in binary or ternary solutions at the higher sucrose content (55°Bx), maximizing WL and minimizing SG.

Keywords— Anco pumpkin; osmotic dehydration; size; sucrose; sodium chloride.

I. INTRODUCTION

During last decades, per capita consumption of fruits and vegetables has noticeably increased; besides, buyers prefer products with high quality, low processing and easy to prepare and consume. That is why, numerous processing methods have been studied that allow obtain new products, nutritious, innocuous and of fresh appearance (Simpson et al., 2007). Osmotic Dehydration (OD) is one of them. It is a Unit Operation used to partially withdraw water from a high-water content material by dipping it in a hypertonic solution. In cellular foods, water withdrawal is based on the phenomenon of osmosis through cell membranes (Rastogi et al., 2005). Water diffusion from the food to the solution is accompanied by a counter-diffusion of solutes from the solution to the interior of the food (Chenlo et al., 2008). Both transfers are due to the gradient in osmotic pressure (chemical potential) between food and solution (Salvatori and Alzamora, 2000; Rastogi et al., 2002). Osmotic dehydration usually precedes other preservation processes like refrigeration, freezing, final dehydration (hot air, vacuum, microwaves), preserves, etc.; it is also used in certain minimally processed foods (Chiralt et al., 2001).

Pumpkin of the variety anco (Cucurbita moschata) is a vegetable broadly produced and consumed in Central and South America. It is one of the vegetables most consumed in the Northwest of Argentina due to its low cost and availability along all the year. It has a high content of carotenoids, constituting an important input of vitamin A (González *et al.*, 2001), potassium, vitamins B₂, C and E and vegetable fiber (de Escalada Plá *et al.*, 2007).

Anco is highly consumed fresh or cooked due to low cost, availability, nutritious quality, good taste and easiness of cleaning, peeling and cooking. Notwithstanding, OD of anco pumpkin –as a possible first stage to further processing - is a little studied subject. There is only about half a dozen of papers related to it. But no paper makes a study intended for the later processing of the OD anco in refrigerated storage (as a ready to eat vegetable, for salads or for pot stews). Osmotic dehydration can be used as a pretreatment (first stage of processing) to later obtain a more stable final product, by refrigeration with or without modified atmosphere or by freezing (dehydrofreezing), constituting an interesting alternative for the development of local industries. As said, literature relative to OD of anco pumpkin is very scarce (Castilho Garcia et al., 2007; Lee and Lim, 2011; Arballo et al., 2012; Bambicha et al., 2012; Bellocq, 2013), compared to that of other products like apples, mango, potatoes, carrots, meats and fishes, etc. That is why it is very important to characterize mass transfer during OD under specified working conditions feasible to be adopted for industrial processing.

Mass transfer rate depends on numerous factors such as composition, concentration, temperature and stirring of osmotic solution; type, composition, structure, shape and size of food material, process time, etc. (Rastogi *et al.*, 2002; Sablani and Rahman, 2003).

Numerous studies have been developed to better understand mass transfer during OD of vegetable tissues and mathematically model the process. In fact, the high structural complexity of cellular foods makes difficult the use of complex mathematical models, due to the need of numerous parameters that are difficult to measure or predict. That is why, phenomenological, empirical or semiempirical methods are applied that relate WL and SG with some of the process variables (time, and mostly solution concentration) through very simple mathematical relations, whose parameters are only valid for the case under study (Ochoa-Martínez and Ayala-Aponte, 2005). An empirical model, widely used, is that of Azuara. It is based in the adjustment of experimental data to simplified equations derived of mass balances for WL and SG, that require adjustable parameters or values for relative rate constants (Azuara *et al.*, 1992). Another model frequently used is that of Hawkes and Flink, based in Fick's diffusion law. It assumes constant solution concentration and negligible external resistance to mass transfer (Hawkes and Flink, 1978). Both models allow the calculation of mass transfer coefficients. No published studies using these methods related to OD of pumpkin were found.

The objectives of this research work were:

- To study the effect of product size and of nature and concentration of the osmotic solution over mass transfer kinetics during OD of anco pumpkin cubes; adjust experimental data to empirical models to determine mass transfer rates, calculate effective diffusion coefficients and evaluate the adequacy of these methods for simulating experimental data.
- To select the most appropriate conditions for the OD of anco cubes - intended for further processing -, based on the previous data.

II. MATERIALS AND METHODS

A. Materials

Anco pumpkins, acquired in a local market, were selected based in their soluble solids content, measured with a hand refractometer and expressed in °Bx, having between 8 and 14 °Bx. Products were washed, peeled and cut into cubes of 1.0 or 1.5 cm side.

Six osmodehydrating solutions were tested: binary solutions of sucrose of 45 and 55 °Bx, and ternary solutions of sucrose-sodium chloride obtained adding 1 or 2 % NaCl to the binary solutions.

B. Osmotic Dehydration

Anco samples, previously weighed, were placed in metallic grid baskets that were dived into the osmotic solution, contained in a thermostatized bath. Constant temperature (30 °C) and stirring (480 rpm) were used, maintaining a ratio solution: product higher than 30:1 to avoid the effect of dilution.

At different immersion times (0.5, 1, 2, 3, 5 and 8 h), samples of at least 5 cubes were randomly withdrawn, washed in distilled water to eliminate traces of solution and dried with tissue paper. Samples were weighed in analytical balance and their content of sol-ids was determined drying to constant weight in a vacuum oven at 60°C (AOAC, 2006).

C. Mass transfer parameters

W

Weight Reduction (*WR*), Water Loss (*WL*) and Solids Gain (*SG*) were calculated according to Agnelli *et al.* (2005):

$$R_t(\%) = \left(\frac{m_0 - m_t}{m_0}\right) \times 100 \tag{1}$$

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

$$SG_t(\%) = \left[\left(1 - \frac{WR_t}{100} \right) \frac{TS_t}{100} - \frac{TS_0}{100} \right] \times 100$$
(3)

where m_0 and m_t are, respectively, the mass of sample, initial and dehydrated during a period t; TS_0 and TS_t represent, respectively, the total solids content, initial and after a dehydration period t.

Process efficiency was evaluated using the index WL/SG (Mayor *et al.*, 2006), as high values of this ratio mean that water loss was favored respect to solids gain.

D. Mathematical modeling

Models of Azuara and Hawkes and Flink were used to describe mass transfer from and to the food during OD. The mathematical model established in Azuara *et al.* (1992) for WL and SG is given in Eq. 4 and 5:

$$WL_t = \frac{s_1 t WL_\infty}{1+s_1 t} \tag{4}$$

$$SG_t = \frac{s_2 t \, S\bar{G}_{\infty}}{1 + s_2 t} \tag{5}$$

where WL_t , WL_{∞} and SG_t , SG_{∞} are water loss and solids gain after a dehydration time t and at equilibrium, respectively; s_1 and s_2 are rate constants related to water loss and solids gain, respectively.

Linearizing Eq. 4 and 5 as function of time, parameters s_1 , s_2 , WL_{∞} and SG_{∞} re obtained from their graphical plot. Besides, relating the model to the first root solution of Fick's law, the effective diffusion coefficient (D_e) can be calculated as given in Eq. 6 (Azuara *et al.*, 1992).

$$D_e = \frac{\pi t}{4} \left[\left(\frac{s_1 l}{1 + s_1 t} \right) \left(\frac{W L_{\infty}^{mod}}{W L_{\infty}^{exp}} \right) \right]^2 \tag{6}$$

In Eq. 6 WL_{∞}^{mod} and WL_{∞}^{exp} correspond to experimental and modeled water loss at equilibrium. Due to the fact that food shape is cubic, *l* in Eq. 6 must be one third of cube side (Rastogi and Niranjan, 1998).

Hawkes and Flink model is given in Eq. 7:

$$\frac{WL}{WL_{\infty}} = 2 \left(\frac{D_e t}{\pi l^2}\right)^{1/2} \tag{7}$$

Plotting WL vs. $t^{1/2}$, D_e is obtained from the slope of the straight line (Hawkes and Flink, 1978).

Changing in Eq. 6 and 7 WL for SG and s_1 by s_2 , the diffusivity of solids can be obtained.

E. Statistical Analysis

Average value and standard deviation of data were calculated as well as the fitting of models to experimental data by the correlation coefficient (R^2) and the Root Mean Square (RMS). The statistical significance of effects was analyzed through their variances (ANOVA) and, as a comparison method, the test of Least Significant Differences (LSD) or Fisher test with a confidence level of 95% was used.

III. RESULTS AND DISCUSSION

Experimental values for WR, WL and SG are plotted in Fig. 1 that shows the prevalence of WL respect to SG. In all tests main variations occur during the first three hours of dehydration, with a later lower rate tending to equilibrium.

WR was higher for the smaller cubes for equal concentration of osmotic solution; e.g., after 3 h dehydration in 45°Bx-2% solution, 1 cm side cubes lost 48.03% of weight meanwhile those of 1.5 cm decreased

their weight by only 29.04% (Fig. 1.a). In most cases higher NaCl content in the osmodehydrating solutions meant higher WR and WL values, independently of sucrose concentration, with the same trends as those determined by other authors (Sacchetti *et al.*, 2001; Telis *et al.*, 2004; Ozdemir *et al.*, 2008; Monnerat *et al.*, 2010; Mercali *et al.*, 2011). Similar values of WL were obtained by Kowalska and Lenart (2001) for 1.0cm side cubes of pumpkin cv Melonowa OD in sucrose solution at 61.5%.

The effect of sample size on dehydration rate was analyzed by Van Nieuwenhuijzen *et al.* (2001) in apple rings. They attributed the increase of WL for samples with lower characteristic dimension to the higher specific area (surface/volume ratio). This behavior was verified for other products (Chavarro-Castrillon *et al.*, 2006; Salvatori and Alzamora, 2000).

Adding of salt at the lower concentration (1%) did not produce noticeable increase in SG. When NaCl concentration was 2%, in most cases, an increase in SG

was determined. Same results were found by other authors working with ternary solutions with low salt content (Lerici et al., 1984; Sacchetti et al., 2001; Jokié et al., 2007). For higher amounts of salt addition (5% or higher) an increase in SG with salt concentration is evident (Azoubel and Xidieh Murr, 2004, Telis et al., 2004). The effect of salt addition on SG seems to depend of salt concentration via an increase in the osmotic pressure gradient and the consequent loss of functionality of cell membrane, what allows the input of solids (Jokié et al., 2007, Herman-Lara et al., 2013). On the other hand, significant differences were found for SG under diverse sucrose concentrations (Fig. 1c and c'), in accordance with results obtained by Singh et al. (2008) with carrot cubes 1.0 cm side, Mújica-Paz et al.(2003) for apple slices OD under vacuum and Pereira et al. (2006) for red guavas. Lower values of SG were determined when using binary and ternary solutions of sucrose at



Figure 1: Evolution of characteristic variables during the osmotic dehydration of anco cubes of (---) 1.0 cm and (-.-) 1.5 cm with sucrose solutions of 45°Bx (a, b and c) and 55°Bx (a', b' and c') without and with added Sodium Chloride.



Figure 2: WL/GS index in cubes of (a) 1.0 and (b) 1.5 cm side de-hydrated in binary and ternary solutions.

55°Bx. Ferrari and Hubinger (2008) attributed this behavior to the increase of resistance to soluble solids penetration owed to higher solution viscosity, which determines the formation of a surface layer of sugar (candying effect).

WL/SG index is given in Fig. 2 a and b for cubes of 1.0 and 1.5 cm, respectively. The highest values of the index were obtained for binary and ternary solutions with 55°Bx of sucrose. Furthermore, the index remains almost constant after 3 h of dehydration. According to the intended later use of the product this is an important characteristic. Mayor et al. (2006) observed a similar trend during OD of 1.5 cm diameter cylinders of pumpkin of other variety (Cucurbita Pepo L.) in solutions of NaCl at 10%. The highest values were for binary solutions of 55°Bx, being 11.22 and 10.72 for cubes of 1 and 1.5 cm, respectively. Kowalska and Lenart (2001) obtained lower values (8.09) after 3 h of OD of pumpkin cubes of a different cv (Melonowa), and with a lower ratio solution: product which means lower dehydrating power due to solution dilution during processing.

Table 1 shows the values of diffusion coefficients calculated using the different models. Calculated values had a very good fitting for both models ($R^2 > 0.98$), in no case RMS was higher than 10%. In general, water diffusion coefficients calculated using Hawkes & Flink model resulted significantly higher ($\alpha = 0.05$) than those given by the model of Azuara. On the other hand, no significant differences were determined for solids diffusion coefficients calculated by both methods. Cubes size had a significant effect on diffusion coefficient, being this higher for the bigger sizes. Falade *et al.* (2007) found similar results when working with melon slices of different thicknesses. This behavior could be attributed to the fact that diffu-sion coefficient varies with water content. Bigger samples lose less water and gained less solids, simplified prediction models leading to higher values of the coefficients (Singh et al., 2007).

Table 1: Diffusion coefficients of water and solids obtained with the models of Azuara and Hawkes & Flink for OD anco cubes

		Water				Solids			
		Azua	ara	Hawkess & Flink		Azuara		Hawkess & Flink	
	Size	$D_{e,w}$	RMS	$D_{e,w}$	RMS	$D_{e,w}$	RMS	$D_{e,w}$	RMS
	(cm)	$\times 10^{10}$	(%)	$\times 10^{10}$	(%)	$\times 10^{10}$	(%)	$\times 10^{10}$	(%)
		(m ² /s)		(m ² /s)		(m ² /s)		(m ² /s)	
45°Bx									
0%	1.0	3.55	1.95	4.52	4.28	3.05	5.58	3.96	5.95
	1.5	7.02	3.38	9.06	3.36	6.05	5.13	7.03	4.05
1%	1.0	3.50	1.34	4.46	4.11	1.82	6.10	2.25	4.50
	1.5	9.11	2.13	11.48	3.36	4.98	7.75	6.68	2.55
2%	1.0	3.93	3.29	4.83	3.83	2.06	5.31	2.60	3.19
	1.5	6.98	2.55	9.63	2.56	6.15	9.41	7.70	2.80
55°Bx									
0%	1.0	2.14	7.28	2.40	2.05	2.12	6.81	2.53	5.18
	1.5	5.90	7.06	6.36	1.93	6.02	3.89	6.98	5.47
1%	1.0	3.38	1.94	4.15	3.50	1.61	6.48	2.70	9.59
	1.5	6.29	2.59	8.13	4.71	6.65	5.93	5.08	8.08
2%	1.0	2.48	3.92	4.67	3.64	3.70	6.15	1.72	4.42
	1.5	7.16	3.13	8.73	2.62	6.52	3.25	6.16	5.01
	210		2.10	2.70	0 _		2.20		2.01

Furthermore, lower sucrose concentration lead, in general, to higher water diffusion coefficients. In the same sense, Rodrigues and Mauro (2008) found - when working with apple slices in sucrose solutions - that water diffusion coefficient lowers when sucrose concentration increases meanwhile solids diffusion coefficient remains almost constant. On the other hand, Castilho Garcia et al. (2007) did not find a dependence of $D_{e,w}$ with the type of osmotic solution, attributing this behavior to a conjunction of opposing factors. The addition of NaCl did not modify noticeably the value of $D_{e,w}$. Surely, higher water loss due to NaCl implies higher volume contraction and - at the same time - lower diffusion coefficient (that is proportional to water content). These factors may counterbalance to give similar results with the approximate methods.

IV. CONCLUSIONS

Higher solution concentration and process time and lower sample size increase WR and WL. SG is favored by low solution concentration.

The higher rates of WL and SG during OD of anco cubes were obtained during the first three hours of treatment; at higher process times variation rates lower and these variables tend to constant values.

At equal concentration of the OD solution, smaller samples underwent higher *WL*.

It was observed that the addition of 2% NaCl increases *WL* and that *SG* are lower when sucrose concentration is the highest.

The higher efficiency of OD, calculated with the index WL/SG was for binary and ternary sucrose solutions of 55°Bx (without or with addition of NaCl).

The accuracy of prediction models was very high.

Mass diffusivities calculated with the models by Azuara and Hawkes & Flink, both for water and solids, increase with sample size and lower with sucrose concentration. Water diffusion coefficients calculated with Hawkes & Flink model were significantly higher. In the range of working conditions tested, OD can be optimized using cubes of 1.0 cm side and binary or ternary sucrose solutions at $55^{\circ}Bx$, when the objective is to reach the highest water loss and – simultaneously – the lower solids uptake.

Additional studies are necessary to determine the influence of partial dehydration in later shelf life of the final refrigerated or frozen product and on its quality characteristics.

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