

# Evaluation of mass transfer coefficients and volumetric shrinkage during osmotic dehydration of apple using sucrose solutions in static and non-static conditions

Ramón Moreira <sup>a</sup>, Alberto M. Sereno <sup>b,\*</sup>

<sup>a</sup> Department of Chemical Engineering, Universidade de Santiago de Compostela, Avda. das Ciencias s/n, E-15706 Santiago de Compostela, Spain

<sup>b</sup> CEQUPI/Department of Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias s/n, 4200-465 Porto, Portugal

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## Abstract

Effects of temperature, solution concentration and solution flow rate on osmotic dehydration/impregnation rate observed during immersion of apple cylinders in sugar solutions at low temperature ( $\leq 25$  °C) are presented. Water and sucrose mass transfer coefficients are calculated and correlated using Magee's model; an extension of the model is proposed to account for solution flowrate; in both cases average relative deviations of less than 3% are obtained. Analysis of results obtained suggests that solute gain by the sample be controlled by diffusion inside the material while water loss is governed by mixed internal–external flow.

Additionally, volumetric shrinkage of apple cylinders treated by osmotic solutions is predicted by measuring the change of the moisture content or the net mass loss due to the treatment. Volume changes observed in samples were correlated linearly with moisture content (dry basis) and with the net change in sample weight. These results suggest that shrinkage be essentially due to water removal/solid gain and offer a simple way to predict such changes during industrial processing.

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## 1. Introduction

Osmotic treatment is a common method to improve product quality and stability (Le Maguer, 1988; Ramaswamy & Nsonzi, 1998; Krokida, Karathanos, & Maroulis, 2000a), to modify its functional properties (Shipman, Rahman, Segras, Kaspalis, & West, 1972; Spiess & Behnlian, 1998) and to reduce energy required for dehydration (Raoult-Wack, Guilbert, & Lenart, 1992). Some models have been proposed to predict water and solute transfer during osmotic treatment (Hawkes & Flink, 1978; Magee, Hassaballah, & Murphy, 1983; Toupin & Marcotte, 1989; Saurel, Raoult-Wack, Rios, & Guilbert, 1994; Yao & Le Maguer, 1996), but experimental data are necessary to use them both to improve the understanding of the phenomena and the process design. It is also known that mass transfer rate is affected by shrinkage of the product (Hough, Chirife, & Marini, 1993) and volume changes are dependent of several

factors as geometry (Moreira, Figueiredo, & Sereno, 2000; Mulet, García-Reverter, Bon, & Berna, 2000), drying method (Krokida & Maroulis, 1997) and experimental conditions (McMinn & Magee, 1996). Physical properties as bulk density and porosity change due to the shrinkage (Misra & Young, 1980; Roman, Urbicain, & Rotstein, 1982; Lozano, Rotstein, & Urbicain, 1983; Sjöholm & Gekas, 1995) and transport properties as thermal and mass coefficient of diffusion are related to changes in density and porosity of material during dehydration (Mattea, Urbicain, & Rotstein, 1990; Sereno & Medeiros, 1990; Karathanos, Kanellopoulos, & Bellessiotis, 1996). Textural properties are also influenced by osmotic treatment leading to an increased plasticity of the structure (Bourne, 1986; Krokida, Karathanos, & Maroulis, 2000b).

During the soaking process, two main countercurrent flows take place simultaneously: water flow out of the product into the osmotic medium, while the solute is transferred from the medium into the product. In addition, losses of vitamin, minerals, and other product solutes are normally observed. Final product characteristics and mass transfer kinetics are largely affected by

\* Corresponding author. Tel.: +351-22-5081655; fax: +351-22-5081449.

E-mail address: sereno@fe.up.pt (A.M. Sereno).

### Nomenclature

<i>a</i>	parameter of Eq. (8)	<i>w</i>	weight (g)
<i>b</i>	parameter of Eq. (9)	WL	water loss
<i>C</i>	solution concentration (mass fraction)	WR	weight reduction
<i>d</i>	parameter of Eq. (9)	<i>x</i>	parameter of Eqs. (8) and (9)
<i>e</i>	parameter of Eq. (9)	<i>X</i>	moisture content in dry basis
<i>k</i>	mass transfer coefficient (s <sup>-1</sup> )	<i>y</i>	parameter of Eqs. (8) and (9)
NMC	normalised moisture content	<i>z</i>	parameter of Eq. (9)
NSC	normalised solid content	<i>Greek symbols</i>	
<i>Q</i>	volumetric flow rate (m <sup>3</sup> s <sup>-1</sup> )	<i>θ</i>	time of osmotic treatment (s)
SG	solids gain	<i>Subscripts</i>	
<i>S<sub>v</sub></i>	relative volumetric shrinkage	0	initial
<i>T</i>	temperature (°C)	w	water
<i>u</i>	solid content (g)	s	solute
<i>V</i>	volume (m <sup>3</sup> )		

this osmotic solute uptake (Lazarides & Mavroudis, 1996).

Osmotic dehydration is usually conducted with agitation of the liquid solution in order to reduce external resistance and increase overall mass transfer rate. However, under such conditions the dynamic liquid flow pattern around the material is difficult to characterise. In order to achieve a better hydrodynamic control an experimental test installation was built that kept the samples immersed in a continuous flow of solution inside a cylindrical column at a specified flow-rate.

This work presents the results of mass transfer coefficients and volume shrinkage observed during the osmotic treatment of apple samples by sucrose solution, both in a static vessel and in the continuous contact apparatus, as a function of temperature, concentration and flow-rate of the solution. In order to minimise structural change of the material a low temperature range between 5 and 25 °C was maintained.

## 2. Procedure

Experimental solutions were prepared with commercial sucrose and distilled water to concentrations of 40%, 50% and 60% (w/w). Apple samples (*Golden delicious* variety) were purchased in a local market and stored under refrigeration (5 °C) until use. Initial moisture content was between 0.85–0.89 (w/w) wet basis. Apple cylinders 24 mm long by 8 mm diameter ( $L/D = 3$ ), were obtained employing a metallic cork borer and a cutter.

In order to obtain samples of greater possible homogeneity in texture and behaviour, the cylinders were always cut parallel to the apple axis, in the central parenchyma tissue. The samples were analysed for moisture content, total solids and soluble solids. Water loss and net solids gain were determined for each cylinder at

different moisture contents. Both static and non-static dehydration at 5 and 25 °C were studied.

Continuous contact experiments were conducted in an apparatus consisting of an acrylic column (height: 0.80 m, diameter: 0.10 m) where sample cylinders were immersed, hanging from the top. This was achieved by introducing a thin stainless steel wire (0.5 mm diameter) along the axis of three sample cylinders; the number of sets of 3-sample cylinders equalled the number of contact times to be studied; after each time step was reached one of the wires with three cylinders was removed and analysed, without significant disturbance of the others. The column is inserted in a circuit allowing a continuous flow of the solution using a centrifugal pump; a number of K-thermocouples were inserted in some of the samples to follow their temperature. A diagram of this installation is presented in the Fig. 1. Four flow rates were employed ranging from zero (static condition) up to  $6.67 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ , corresponding in all cases to laminar flow conditions ( $Re < 100$ ). It should be noted that temperature has a significant effect on solution viscosity and its effect is included in the Reynolds number. The column and a holding tank for the solution are enclosed in a modified domestic freezing cabinet, where low temperatures can be reached and maintained. With this arrangement very well defined hydrodynamic conditions can be established. The temperature of the solution was measured by K-type thermocouples. All piping is made of plastic material.

Each run for mass transfer studies lasted 4 h and samples were taken out of the osmotic solution at different times (10, 20, 30, 60, 120 and 240 min) enough to reach stable conditions (Lerici, Mastracola, & Nicola, 1988). To evaluate the relative volumetric shrinkage runs up to 8 h were carried out. In both cases, after removal, the samples were blotted with blotting paper to remove superficial osmotic solution. For the range of

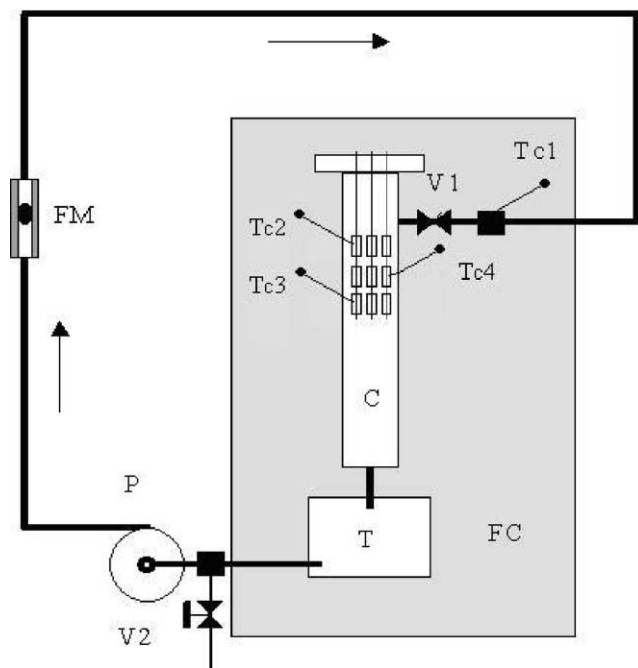


Fig. 1. Diagram of continuous flow unit for osmotic dehydration. (C: column; FC: freeze chamber; FM: flowmeter; P: pump; T: tank; Tc: thermocouple; V: valve).

flowrate and contact times used, no significant change in sample texture was observed.

Volume change (shrinkage) of the cylinders during osmotic dehydration was determined by immersing samples of different moisture content, in *n*-heptane. Triplicate results for each moisture content were obtained. Moisture contents of the samples were determined in an oven at 70 °C at less than 0.1 atm, during 24 h.

### 3. Theory

#### 3.1. Mass transfer model

Osmotic dehydration rate depends on several variables such as type of material, maturity state, shape, size and pre-treatments employed, as well as on process variables (solution composition and concentration, solution/product ratio, temperature, contact method and immersion time) (Saurel et al., 1994; Lazarides, Gekas, & Mavroudis, 1997). To describe mass transfer, Fick's approximation is traditionally used, employing a concentration gradient as driving force. Some simplifications are normally assumed namely the use of effective diffusion coefficient which takes into account all transport mechanisms contributing to diffusion.

Three main process variables are usually measured: moisture content, change in weight and change in soluble solids. From these, water loss (WL), weight reduction (WR), solids gain (SG), normalised moisture

content (NMC) and normalised solid content (NSC) were calculated as follows:

$$WR = (w_0 - w)/w_0 \quad (1)$$

$$SG = (u - u_0)/w_0 \quad (2)$$

$$WL = WR + SG \quad (3)$$

$$NMC = X/X_0 \quad (4)$$

$$NSC = u/u_0 \quad (5)$$

where  $w$  is sample weight;  $w_0$  the initial sample weight;  $u$  the solid weight;  $u_0$  the initial solid weight;  $X$  the moisture content and  $X_0$  the initial moisture content (dry basis). A model was proposed by Hawkes and Flink (1978) to describe the kinetics of moisture loss and solid gain:

$$NMC = 1 - k_w \theta^{0.5} \quad (6)$$

$$NSC = 1 + k_s \theta^{0.5} \quad (7)$$

where  $k_w (s^{-0.5})$  and  $k_s (s^{-0.5})$  represent overall mass transfer coefficients for water and solute respectively, and  $\theta$  (s) is the dehydration time.

Magee et al. (1983) suggested that, under static conditions, mass transfer coefficients,  $k_i$  ( $k_w$ , water;  $k_s$ , solute), depend on solution concentration,  $C$  (% w/w) and the contact temperature,  $T$  (°C):

$$k_i = aC^x T^y \quad (8)$$

where  $a$ ,  $x$  and  $y$  are parameters of the model in static conditions.

To account for non-static contact, an extension of Magee's model is proposed here by including solution flow rate  $Q$  ( $m^3 s^{-1}$ ):

$$k_i = bC^x T^y (d + eQ^z) \quad (9)$$

where  $b$ ,  $d$  and  $z$  are the new parameters of the model in non-static conditions.

Evaluation of the effect of agitation rate during osmotic dehydration of apple (and other fruits) was analysed by (Panagiotou, Karathanos, & Maroulis, 1999) presenting a model where  $k_s$  is dependent on this variable, but  $k_w$  is independent of the level of agitation.

#### 3.2. Volumetric shrinkage

A linear relationship between  $S_v = V/V_0$  and NMC during apple drying, during the whole process or at least part of it, is reported in several works using different drying procedures (Lozano et al., 1983; Ratti, 1994; Sjöholm & Gekas, 1995). Such linear relationship was also observed during osmotic dehydration of apple, banana, potato, carrot or blueberries (Krokida & Maroulis, 1997; Nsonzi & Ramaswamy, 1998), but an analysis of the effect of non-static osmotic conditions on shrinkage should be made as happened with the study of the effect of air rate in convective drying (McMinn & Magee, 1996; Khraisheh, Cooper, & Magee, 1997).

#### 4. Results and discussion

Typical results of the change of the dehydration parameters (NMC and NSC) for apple cylinders immersed in aqueous sucrose solutions (40%, 50% and 60%) at 5 °C are shown in Fig. 2 under static conditions. Due to the increase of osmotic pressure difference with increase of the solution concentration, a corresponding increase in dehydration rate was observed. Water mass transfer was important during all dehydration period (2 h), but solids gain rate was greater at the beginning (during the first hour) and then stopped. Similar behaviour is reported by other authors employing a glucose solution as osmotic agent (Salvatori & Alzamora, 2000).

Eqs. (6) and (7) were fitted to experimental data up to 2 h. It is observed that in the case of solid gain, the kinetics is acceptably represented during the first hour only. Temperature has an important effect on apple dehydration rate, which was found to increase with processing temperatures. Mass transfer coefficients in static conditions, with sucrose solution as osmotic agent, were reported by Sereno, Moreira, and Martinez, 2001; and were correlated with temperature and concentration of the sucrose solution (Eq. (8)) obtaining the following expressions (with average relative deviations of less than 3%):

$$k_w s^{-0.5} = 0.00172 C^{1.36} T^{0.371} \quad (10)$$

$$k_s s^{-0.5} = 0.00101 C^{0.621} T^{0.137} \quad (11)$$

It was observed that mass transfer coefficient for solute,  $k_s$ , was less dependent on the temperature and concentration in the range of experimental conditions used than  $k_w$ . A dashed extension to the line representing Eq. (11) is shown in Fig. 2 to highlight the lack of fit of the model beyond 1 h of contact.

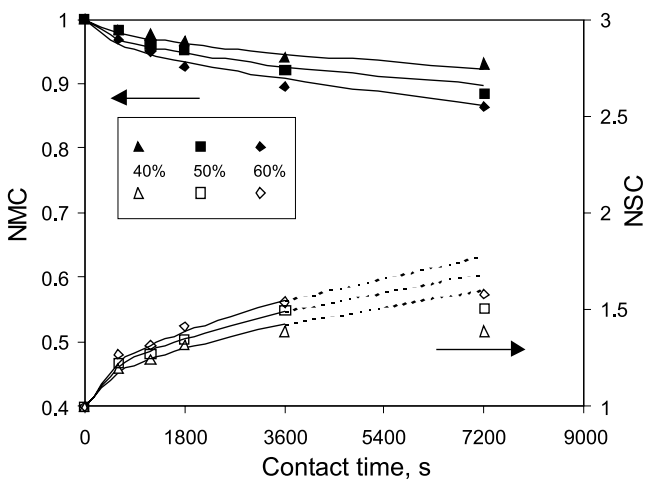


Fig. 2. Kinetics of simultaneous water removal/sucrose impregnation at 5 °C: effect of solution concentration (dashed lines represent model predictions).

With respect to experiments in non-static conditions, as expected, dehydration rates increase with solution flow rate and tend to a lower equilibrium moisture content than the one observed in static conditions, due to the reduction of the external resistance for removal water. The following equation describing water mass transfer coefficient was obtained:

$$k_w = 4.25 \times 10^{-5} (40.5 + 24.7 \times 10^3 Q^{0.67}) C^x T^y \quad (12)$$

The parameters  $x$  and  $y$  have the same values as for static conditions (Eq. (11)). The good agreement between predicted and experimental values of the overall mass transfer coefficients at different temperatures when a 60% (w/w) sucrose solution is employed is shown in Fig. 3.

With respect to solid gain, results were similar to the ones obtained in static conditions (Fig. 4). This shows that while the rate of water removal is improved by the use of flow conditions, sucrose gain is not affected, suggesting that in this case the internal rate of mass transfer control the process. These results differ from Panagiotou et al. (1999) where the experiments conducted at a higher temperature and in turbulent regime resulted in a solid acquisition that was a function of speed of agitation. In the present case sucrose impregnation of the sample surface lasted for about 1 h at the same rate as previously observed under static experiments until a pseudo equilibrium was reached, after which solute content remained essentially constant.

Concerning the analysis of volumetric shrinkage Fig. 5 presents the results obtained for relative volumetric shrinkage ( $S_v = V/V_0$ ) of the samples vs. moisture content ( $X$ , dry basis) in static conditions at 5 and 20 °C, using 50% and 60% (w/w) sucrose solution (to extend the range of moisture content, contact times up to 8 h were reached in this study). Some dispersion of the results

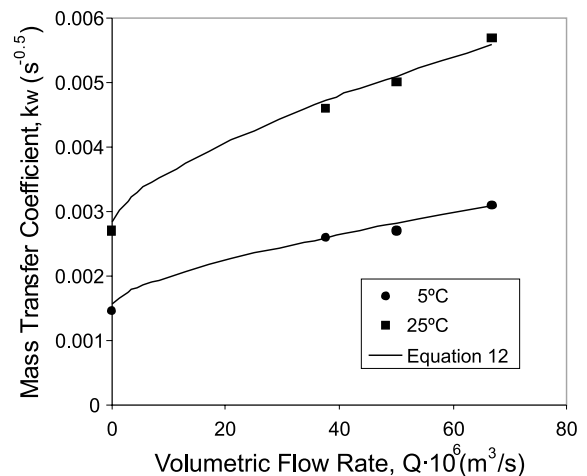


Fig. 3. Mass transfer coefficients for water during osmotic dehydration of apple cylinders immersed in a circulating 60% sucrose solution.

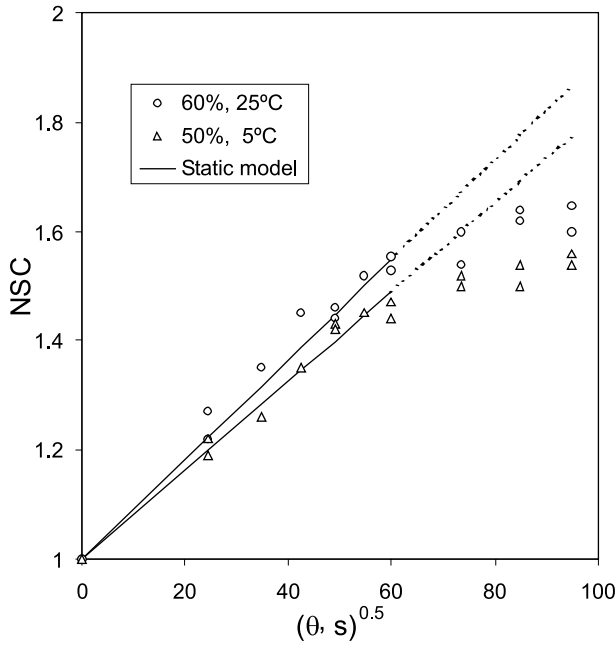


Fig. 4. Comparison of kinetic data of sucrose uptake in flow conditions ( $Q = 0.12 \text{ m}^3 \text{ h}^{-1}$ ) at two solute concentration and temperature (symbols) with static conditions (line); (dashed lines represent model predictions).

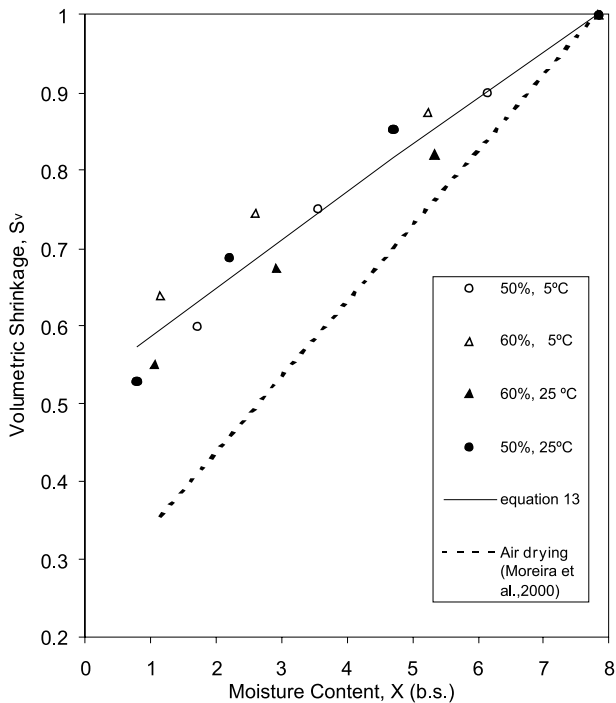


Fig. 5. Relative shrinkage vs. moisture content during osmotic and convective drying.

was observed, probably due to differences during sample blotting with absorbing paper. Nevertheless, a uniform behaviour is observed, which is essentially independent of each set of experimental conditions. This behaviour

suggests a simple linear relation between net mass loss by the samples and volumetric shrinkage. Each data point in the plot represents the average of three experiments. Similar results were obtained using a non-static method (up to  $6.67 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$  solution flow rate). Eq. (13) represents the linear function observed between relative volumetric shrinkage and moisture content in dry basis:

$$S_v = 0.524 + 0.062X(\text{db}) \quad (13)$$

Earlier results obtained by drying similar apple samples in warm air are presented in the same plot (Moreira et al., 2000). In this case a higher value for the slope (0.096) was obtained. The lower value of the slope found for osmotic treated samples may be due to the already mentioned solute uptake which fills part of the internal volume left by the water lost. It should be noted that after 2 h of osmotic treatment, during which the proposed model is valid and thought to exceed the large majority of actual industrial application of this process, the moisture content is about 6 kg of water/kg db corresponding to only 10% of volume shrinkage. This allows the use of a simplified mass transfer model that does not include the effect of volumetric shrinkage, as presented. In the case of longer osmotic dehydration time, the volumetric shrinkage should be taken into account.

Donsi, Ferrari, and Nigro (1998) studied (without modelling) the shrinkage of apple during osmotic treatments with agitation (100 rpm) at low temperature during a long dehydration period (up to 90 h) and observed a lower deformation at 4 °C than at 25 °C (with

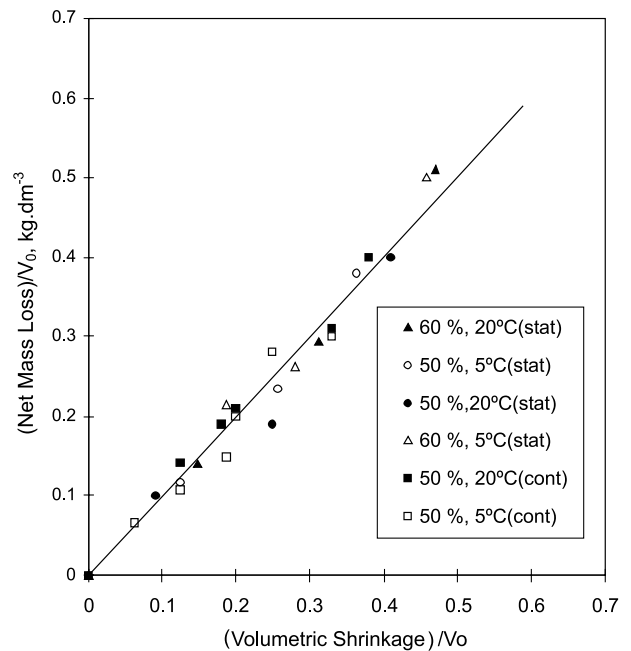


Fig. 6. Relationship between shrinkage and net mass loss (solid line represents the diagonal, not a data fitting).

the same total solid gain and total water loss); however, during the early steps of the study, no difference was recorded.

Fig. 6 shows that in spite of different experimental conditions (static/non-static; temperature and solution concentration) a good linear correlation between the net mass flow (difference between water coming out and solute going into the material) expressed in terms of net mass loss and volume shrinkage was found.

## 5. Conclusions

Osmotic treatment of apple in sucrose solution was studied in the present work in static and non-static conditions and a model for mass transfer (water removal and solid acquisition) is presented with satisfactory agreement up to 2 h of osmotic treatment. In all cases, a higher solution concentration gives higher water loss and higher solid acquisition.

The use of a laminar flow of the osmotic treating solution leads to an increase of the rate of water loss, but no change in solid acquisition with respect to the use of static conditions. This is an interesting result for the industrial processing of these materials, as it represents a way of accelerating water removal without increasing sucrose impregnation.

The results about relative volumetric shrinkage indicate that it may be easily estimated from changes in moisture content of the sample, independently of drying rate. Inversely, determination of volumetric shrinkage would give an indirect indication of moisture content of the product.

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