

## Rate of Water Loss and Sugar Uptake During the Osmotic Dehydration of Pineapple

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### ABSTRACT

*Water loss, sucrose gain and the variation in concentration of other natural fruit sugars (glucose and fructose) were studied during osmotic dehydration of pineapple slices (0.6 mm thick) in sucrose solution (60 % w/w) at three temperatures (30, 40 and 50°C). As temperature increased from 30 to 50°C, the apparent moisture and sucrose diffusivities ( $D_w$  and  $D_s$ ) increased 3.8 and 2.8 times, respectively; therefore, the dehydration efficiency index ( $D_w/D_s$ ) increased with temperature. The loss of glucose and fructose increased with temperature too. It was found that the solute content was a linear function of the moisture content and this relation was independent of the temperature during the first 600 minutes of dehydration.*

**Key words:** Osmotic Dehydration, Pineapple, Sugars Content, Concentration Profiles

### INTRODUCTION

Osmotic dehydration is a method for the partial dehydration of water-rich foods, such as fruits and vegetables, by immersing them in a concentrate solution of sugar or salt. It results in two simultaneous crossed flows: a water outflow, from the food to the solution and a solute inflow from the solution into the food (Hough et al., 1993; Raoult-Wack et al., 1994; Spiazzi and Mascheroni, 1997). The water flow may carry away other solutes present in the fruit (vitamins, minerals, sugars). Pineapple contains sucrose, fructose and glucose in concentrations that, in combination with acids and other compounds, determine the typical flavor of this fruit (Gherardi et al., 1994).

Osmotic dehydration processes are normally designed with the aim of maximising water removal meanwhile restraining solid uptake, to obtain a product whose taste and flavor have little

changes in respect to that of the fresh food. The ratio of water loss to solid uptake is a good index of the efficiency of the process. When analysing operating conditions, the ratio between the diffusion coefficients of water and sugar may be used as a parameter for the efficiency index of the osmotic dehydration process (Lazarides et al., 1997).

Water loss and solute gain are usually measured as average values in the piece of food. There are a few papers published about the concentration profiles developed in structured foods during osmotic dehydration. There is information about the concentration profiles developed during osmotic dehydration of apple tissue (Salvatori et al., 1998), potato tissue (Lenart and Flink, 1984; Genina-Soto et al., 2001; Mauro and Menegalli, 2003), in gels (Raoult-Wack, 1991) but no published studies were found about the concentration profiles developed throughout the

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thickness of pineapple slices during osmotic dehydration.

The objectives of the present work were to analyse the effect of temperature on water loss, solute gain and glucose and fructose leakage during the osmotic dehydration of pineapple and to study the concentration profiles developed during this process.

## MATERIALS AND METHODS

### Sample preparation

*Ananas Comosus* variety *Cayena Lisa* of commercial ripeness degree, procured from Colonia Aurora, Misiones, Argentina, was used in this work. The fruits were manually peeled, cored, cut into half rings of 0.6 cm thickness and gently blotted with tissue paper to remove surface water. Because the sugar content could be different from one extreme to another of the fruit, only the central zone of each pineapple was used in the experiments.

### Osmotic Dehydration

An aqueous sucrose solution (60 % w/w) was used as dehydrating agent. The weight ratio of solution to fruit was at least 20:1, to avoid significant dilution of the medium during the experiments. Dehydration was run immersing an open mesh plastic basket with the fruit into the stirred osmotic solution at the preselected temperature; the vessel was covered with a sheet of aluminium film to prevent evaporation of syrup. The system was maintained at the selected temperature by immersion in a constant-temperature water bath. At pre-established times, a sample (a half-slice) was removed from the osmotic medium, rinsed with deionised water and blotted with tissue paper to remove excess surface solution. It was divided into two fractions; one was used to determine water content and the other for sugars contents. The process was run at three temperature levels, by triplicate at 30 and 50 °C, and by sextuplicate at 40 °C.

### Concentration Profiles

Two experiments were carried out at 40 °C with slices 10 mm thick. At different osmosis periods (30, 60, 75, 90 and 150 minutes), one sample was removed from the solution, rinsed and blotted. Then, it was cut with scalpel into four slices of

approximately 1 mm thickness, starting from the osmosed surface. Slices were identified by their order of slicing. Moisture and sugars content were determined in each slice.

### Measurement of Moisture Content

Moisture content was determined gravimetrically by vacuum oven drying at 70 °C until constant weight (AOAC).

### Sugars Content Determination (glucose, fructose and sucrose)

**Extraction Process:** Each sample was weighed and crushed in mortar for 10 minutes; 80 mL of ethanol-water solution (80:20 % v/v) for each 2.5 g of fruit was added. The mixture was let to settle in a water bath at 75 °C for one hour with sporadic stirring, and then filtered through filter paper (the solid residue was washed with 20 mL of ethanol-water solution, and added to the main solution). The final volume of filtered liquid was registered and this solution was kept under refrigeration until its chromatographic analysis.

**Quantitative Determination:** Sugars content was evaluated by liquid chromatography (HPLC). The filtered solution was injected into the chromatograph (Shimadzu LC6A) with an Amino NH<sub>2</sub> column (LiChrospher® 100 NH<sub>2</sub> 5 µm, 250 mm x 4.6 mm), and a Refraction Index detector. Assay conditions were: mobile phase of acetonitrile:water (75:25 % v/v) and flow rate: 1.5 mL/min. The system was thermostated at 30°C. The quantification was made using the technique of external standard.

### Water and Sucrose Diffusion Coefficients

One-dimensional diffusion was assumed to estimate the diffusion coefficients (Beristain et al., 1990; Hough et al., 1993; Waliszewski et al., 1997; Kosegarten-Conde et al., 1999; Rastogi et al., 2000). The first term of the analytical solution (Crank, 1975) for solute diffusion in a plane plate with constant surface concentration, size and diffusion coefficient was used. A detailed description of the procedure and considerations is given in Ramallo et al. (2004). The simplified form of analytical solution is:

$$\frac{\bar{X} - X_e}{X_0 - X_e} = \frac{8}{\pi^2} \exp\left\{-D \frac{\pi^2}{l^2} t\right\} \quad (1)$$

where  $\bar{X}$  is the actual (at time  $t$ ) average water (or solute) content in the fruit,  $X_0$  is the initial water (or solute) content,  $X_e$  is the equilibrium water (or solute) content,  $D$  is the effective (apparent) diffusion coefficient for water or sucrose and  $l$  is the half thickness of the pineapple slices.

### Sucrose hydrolysis within the fruit

To verify that the measured glucose and fructose contents were not the result of the hydrolysis of the sucrose that migrated towards the interior of the fruit during the dehydration process, tests for sucrose hydrolysis in pineapple juice were done. The experiences tried to resemble the true conditions inside the fruit. Two types of sample and two extraction conditions were used.

**Extract 1:** Juice was left to drift from the fruit, simulating to work with the liquids contained in the extracellular space of the fruit tissue.

**Extract 2:** Fruit was manually crushed in mortar during 15 minutes, so as to also extract compounds from the interior of cells.

Juices were filtered through Whatman N° 3 paper. An aliquot (natural juice) was extracted for analysis by HPLC, and 2 % w/w of sucrose was added to the remaining solution. Another aliquot (time zero for hydrolysis) was taken for analysis. The flasks containing the sugary juices were kept covered in a water bath at  $45 \pm 1$  °C during 20 h. One mL aliquots were withdrawn for immediate analysis at predetermined time intervals during that period.

## RESULTS

### Water and sucrose content

Mass transfer in osmotic dehydration is a combination of simultaneous water loss and solute uptake (Hough et al., 1993; Rahman and Perera, 1996; Spiazzi and Mascheroni, 1997). For that reason, the experimental data of water loss and sucrose gain were analysed and displayed in joint way in the present study.

The most significant changes in the sucrose and water content took place in the first 600 minutes of osmotic dehydration process (Ramallo et al., 2004). Consequently, the relation between water

and sucrose content during osmotic dehydration of pineapple was studied during the first 600 minutes of process. However, the treatment was prolonged until the system reached the equilibrium. Equilibrium water and sucrose contents were experimentally determined, as the values reached when their rate of change dropped to zero – which occurred after about 25 h of immersion. The average values of equilibrium contents for the three temperatures are given in Table 1, with their standard deviations (SD). The statistical study of these values showed that the equilibrium water content was independent of temperature, meanwhile the degree of sugar impregnation increased with temperature (from 45.12 to 54.29 %). These results fully agreed with those reported by Panagiotou et al. (1998) for osmotic dehydration of banana, apple and kiwi pieces in sucrose solution.

Fig. 1 shows the behaviour of the sucrose concentration vs. moisture content ratio during osmotic dehydration of pineapple slices in sucrose solution at three temperatures. In all cases, the results fit to a linear function in the generalized form of Equation (2), displaying a high adjustment ( $p \leq 0,001$ ).

$$s = a + b w \quad (2)$$

Where  $s$  is the sucrose content, g/100 g of fruit and  $w$  is the water content, g/100 g of fruit.

In table 2 are the average values of the parameters  $a$  and  $b$  of Equation (2) obtained by fit of experimental data –sucrose and water content- to a linear function, for each independent experience. Statistical analysis (ANOVA) showed that these parameters are temperature independent. Then, by adjustment of all the experimental data to the linear model Equation (3) was obtained:

$$s = 79.051 - 0.8365 w, \quad r^2 = 0.985 \quad (3)$$

These results indicated that the solute uptake was function of the water content in the fruit, being this ratio independent of temperature during the first 600 minutes of dehydration, within the range of tested temperatures. Nevertheless, the temperature influences on the rate at which the ratio  $s/w$  was reached, as shown by the efficiency index (Table 3).

**Table 1** - Equilibrium water and sucrose contents (g/100 g of material) in pineapple slabs, after 25 h dehydration in a stirred sucrose solution (60 % w/w).

Temperature (°C)	Equilibrium sucrose content ± SD	Equilibrium water content ± SD
30	45.12 ± 3.14	35.95 ± 0.07
40	53.00 ± 0.41	34.32 ± 2.52
50	54.29 ± 2.39	35.68 ± 2.28

**Table 2** - Results from adjustment of experimental data to a linear function, during osmotic dehydration of pineapple slice of 0.6 cm thickness.

Temperature, °C	Intercept ± DS (a)	Slope ± DS (b)	Average Correlation Coefficient
30	87.040 ± 3.479	-0.948 ± 0.0523	0.942
40	76.047 ± 8.231	-0.794 ± 0.1072	0.984
50	83.197 ± 9.820	-0.896 ± 0.1244	0.986

The plot of experimental data of s/w versus time showed that the ratio changes with the temperature, with similar behaviour at 40 and 50 °C with respect to that at 30 °C, as could be observed in Figure 2. These results agreed with the observed ones through the efficiency index of process (Table 3). The slope obtained by regression analysis could be considered as a measure of the rate of change in sucrose-water content ratio; average values of slope were:  $0.000700 \pm 0.000078 \text{ min}^{-1}$ ;  $0.00185 \pm 0.00025 \text{ min}^{-1}$  y  $0.002007 \pm 0.00040 \text{ min}^{-1}$  at 30, 40 and 50 °C, respectively.

The values calculated for the Apparent Moisture Diffusivity ( $D_w$ ) and the Apparent Sucrose Diffusivity ( $D_s$ ) are given in Table 3. As temperature increased from 30 to 50 °C,  $D_w$  increased 3.8 times and  $D_s$ , 2.8 times. The ratio  $D_w/D_s$  could be used as a measure of dehydration efficiency (Lazarides et al., 1997). As shown in Table 3, when temperature was increased from 30 to 40 °C, this parameter also increased, but remained constant when temperature passed from 40 to 50 °C.

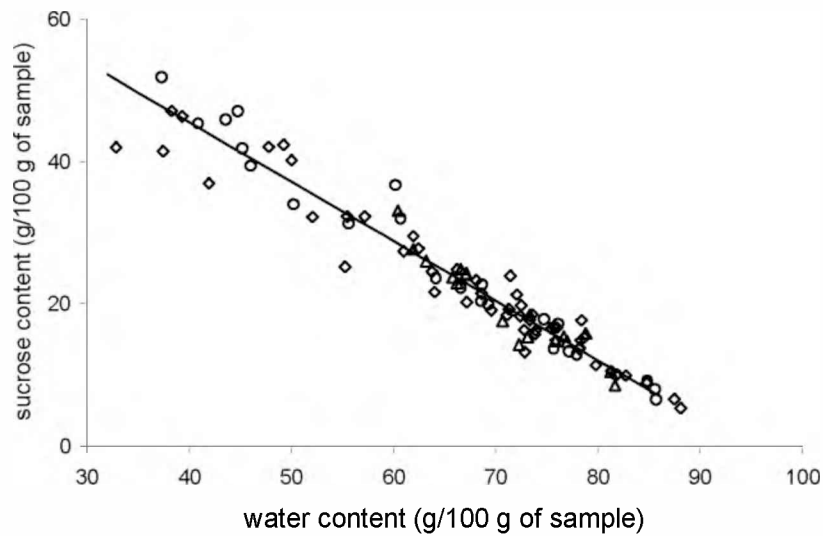
While dehydrating apple slices, Lazarides et al. (1997) found that this index was constant with temperature. The same was reported by Rastogi and Raghavarao (1998) for carrot dehydration. Castro et al. (1998) and Saputra (2001), for

pineapple in sucrose solution, found that higher temperatures favoured water loss meanwhile solids uptake remains practically constant, in partial coincidence with results. Differences could arise from pineapple varieties, temperature ranges and from differences in structure respect to other fruit.

#### Concentrations of glucose and fructose

Experimental data of glucose and fructose content, expressed in g per g of the initial wet matter of the sample (g/g i.w.m.), during osmotic dehydration of pineapple slabs in sucrose syrup are shown in Fig. 3. A similar behaviour was observed at 40 and 50 °C: there was no meaningful variation in sugar contents during the first 600 minutes of process. However, results determined from longer time of process were different: glucose and fructose contents remained almost constant at 40 °C and decreased at 50 °C.

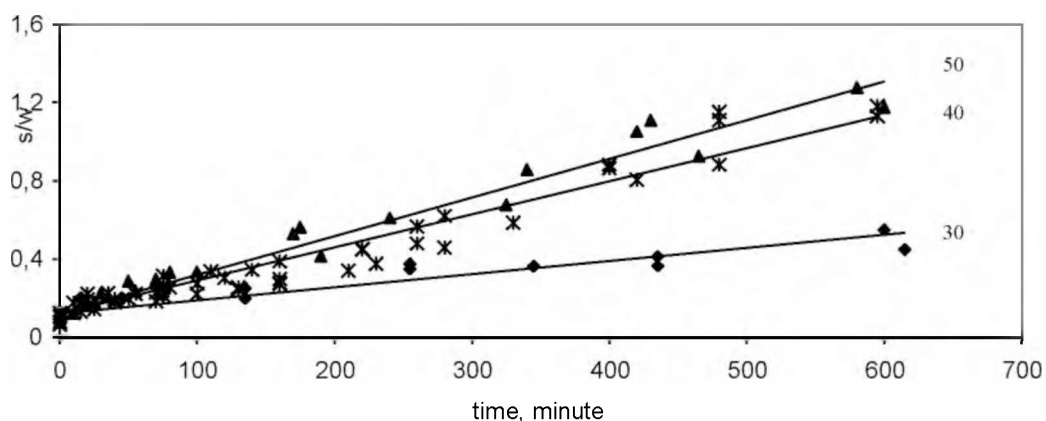
This net loss – higher for fructose – could be due to solute sweeping away by the higher volumetric water flow during dehydration at higher temperatures (leaching). These results coincided with those of Bolin and Huxsoll (1993) in osmotic dehydration of pears in sucrose solution and with those of Saurel et al. (1994) in osmotic dehydration of apples.



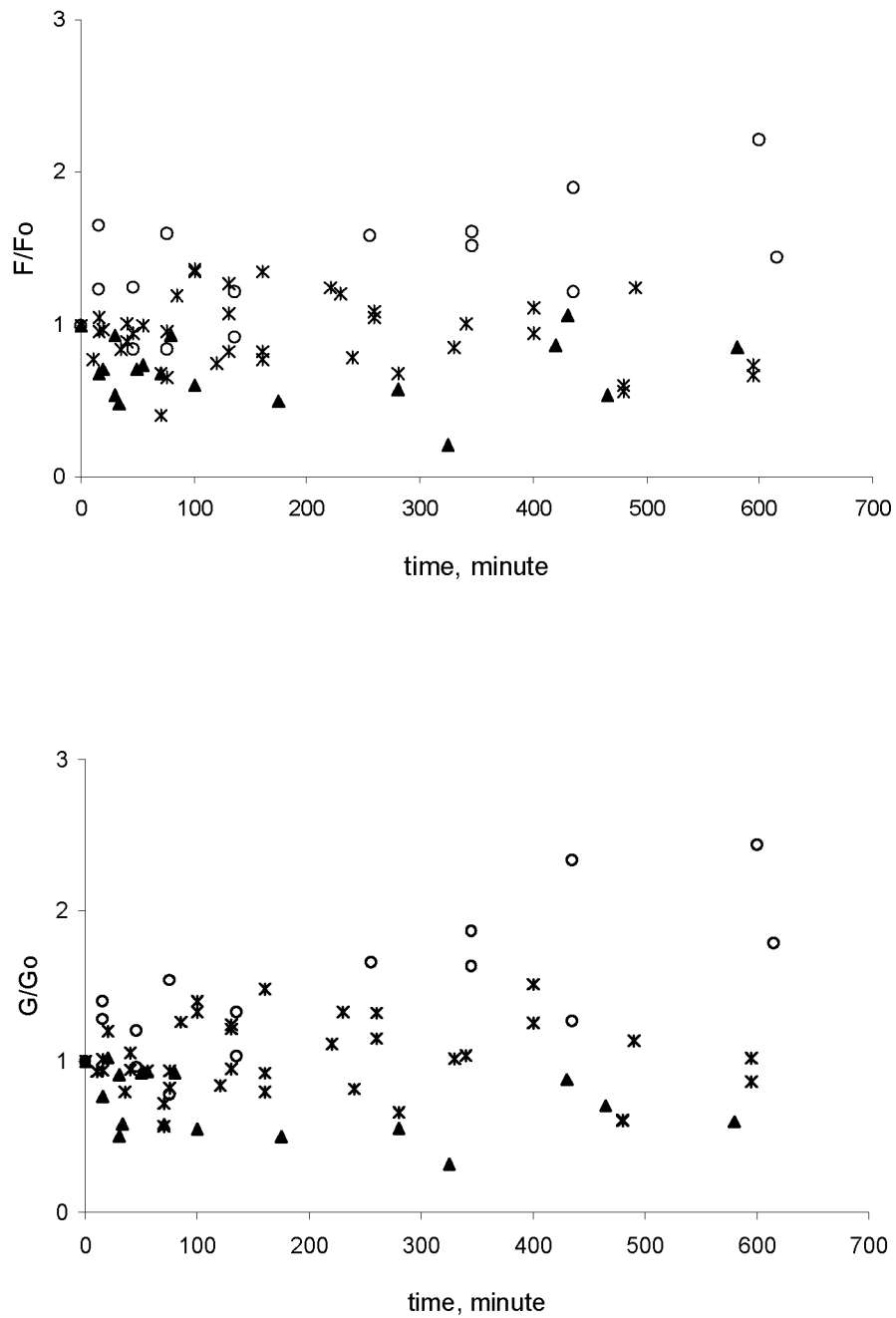
**Figure 1** - Experimental data of sucrose concentration versus moisture content and the representation of the linear model (full line). (o) 30°C; (◊) 40°C; (Δ) 50°C.

**Table 3** - Average values of  $D_w$  and  $D_s$  with their corresponding standard deviation SD and Efficiency Index during the osmotic treatment of pineapple half slices 0.6 cm-thick at different temperatures.

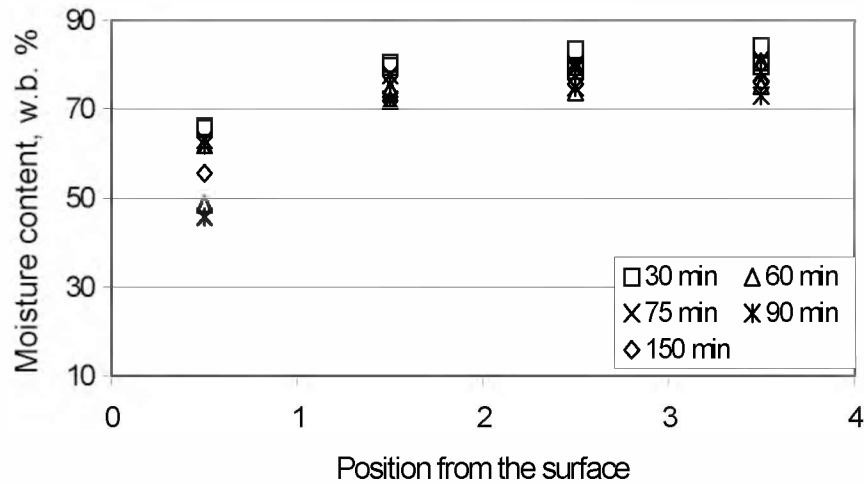
Temperature (°C)	$(D_w \pm SD) \times 10^{11} \text{ (m}^2/\text{s)}$	$(D_s \pm SD) \times 10^{11} \text{ (m}^2/\text{s)}$	Efficiency	Index
30	$5.80 \pm 1.60$	$7.68 \pm 3.68$	0.754	
40	$17.17 \pm 2.83$	$16.10 \pm 4.18$	1.066	
50	$22.22 \pm 2.55$	$21.83 \pm 5.67$	1.017	



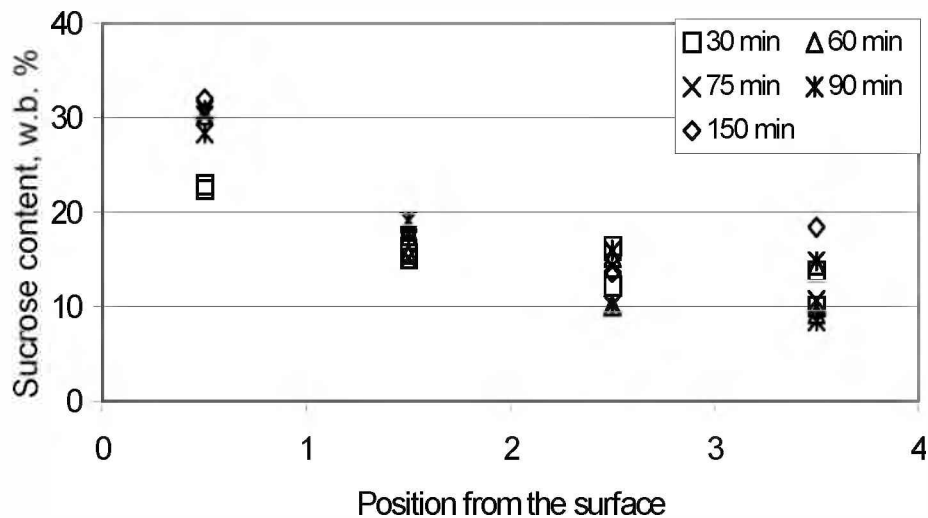
**Figure 2** - Ratio of sucrose content to water content,  $s/w$ , during osmotic dehydration of pineapple slabs in sucrose syrup (60% w/w) at 30°C (◆), 40°C (\*) and 50°C (▲).



**Figure 3** - Variation of fructose and glucose content with time (expressed in g per g of the initial wet matter of the sample) during osmotic dehydration of pineapple half slices. (o) 30°C; (\*) 40°C and (▲) 50°C.



**Figure 4** - Moisture content of the slice as a function of the distance from the surface of pineapple in contact with sucrose solution at 40°C.



**Figure 5** - Sucrose content of the sample as a function of the distance from the surface for pineapple in contact with sucrose solution at 40°C.

Results at 30°C showed an increase in glucose and fructose contents during the process (Fig. 3). This increase could be attributed as that these sugars came from sucrose hydrolysis and the rate of hydrolysis was greater than the rate of sugars leakage. Meanwhile, at higher temperatures leakage appeared to be most important than sucrose hydrolysis. Bernardez et al. (2004), using

a similar chromatographic technique, also obtained important dispersion in the glucose and fructose content in varieties of chestnut fruits.

#### Concentration profiles of water and sucrose

The average values and standard deviation of slice thickness used in the study of concentration

profiles were  $0.94 \pm 0.091$  cm. Fig. 4 shows the variation of water content as a function of time and position in the sample, average of two dehydration experiences carried out at  $40^\circ\text{C}$  (the numbers in the spelling axis indicate the 1 mm-slice to which the measured value belongs). Sucrose concentration in a pineapple slice as a function of

distance from surface and time is shown in Fig. 5. Concentration increased in the surface, but its variation decreased to become almost null in the inner part of the sample. These results were in accordance with those of Marcotte and Le Maguer (1992) for potatoes and from Salvatori et al. (1998) for apples.

**Table 4** - Glucose, fructose and sucrose concentrations (g/100 mL) in pineapple juices at  $45^\circ\text{C}$ , as a function of time.

Time (min)	Fruit 1		Fruit 2	
	Extract 1	Extract 2	Extract 1	Extract 2
<b>Fructose</b>				
0	$1.71 \pm 0.12$	$1.47 \pm 0.33$	$1.44 \pm 0.11$	$1.42 \pm 0.32$
75	$1.71 \pm 0.02$	$1.62 \pm 0.05$	$1.55 \pm 0.07$	$1.43 \pm 0.05$
210	$1.69 \pm 0.10$	$1.64 \pm 0.08$	$1.51 \pm 0.09$	$1.34 \pm 0.07$
330	$1.77 \pm 0.14$	$1.63 \pm 0.09$	$1.73 \pm 0.14$	$1.55 \pm 0.09$
1200	$2.04 \pm 0.13$	$1.91 \pm 0.17$	$1.82 \pm 0.11$	$1.96 \pm 0.11$
Natural Juice	$1.49 \pm 0.07$	$1.49 \pm 0.15$	$1.38 \pm 0.06$	$1.25 \pm 0.11$
<b>Glucose</b>				
0	$1.22 \pm 0.09$	$1.02 \pm 0.18$	$1.00 \pm 0.07$	$0.80 \pm 0.13$
75	$1.36 \pm 0.22$	$1.13 \pm 0.19$	$1.16 \pm 0.19$	$0.82 \pm 0.13$
210	$1.40 \pm 0.10$	$1.23 \pm 0.06$	$1.09 \pm 0.06$	$0.85 \pm 0.15$
330	$1.40 \pm 0.28$	$1.27 \pm 0.29$	$1.12 \pm 0.11$	$0.90 \pm 0.05$
1200	$1.71 \pm 0.21$	$1.55 \pm 0.25$	$1.59 \pm 0.15$	$1.52 \pm 0.10$
Natural Juice	$1.09 \pm 0.14$	$1.01 \pm 0.19$	$1.00 \pm 0.12$	$0.79 \pm 0.14$
<b>Sucrose</b>				
0	$9.08 \pm 1.36$	$7.77 \pm 0.64$	$8.81 \pm 1.32$	$9.27 \pm 0.76$
75	$9.08 \pm 0.34$	$8.45 \pm 0.19$	$9.06 \pm 0.34$	$9.35 \pm 0.21$
210	$9.25 \pm 0.37$	$8.58 \pm 0.48$	$8.87 \pm 0.36$	$8.80 \pm 0.49$
330	$8.87 \pm 0.34$	$8.29 \pm 0.99$	$9.23 \pm 0.35$	$9.19 \pm 1.10$
1200	$8.57 \pm 0.49$	$7.91 \pm 0.49$	$8.45 \pm 0.48$	$9.75 \pm 0.60$
Natural Juice	$6.13 \pm 0.12$	$5.93 \pm 0.18$	$6.62 \pm 0.13$	$6.84 \pm 0.20$

#### Sucrose hydrolysis during osmotic dehydration of pineapple

The results of the tests done on fruit juices showed that their concentration in natural sugars was constant, at least during the first 5 hours in a water bath at  $45^\circ\text{C}$ , and that there was no sucrose hydrolysis (results not shown). The variations in sugar concentration along the first hours were of the order of the accuracy of the analytical method, so no general trend could be inferred. At the higher period tested (1200 min), the effect of acid hydrolysis on sucrose was leading to an increase in fructose and glucose concentrations and a decrease in sucrose concentration. The extraction method of the juice had no influence on the concentration of sugar in the natural juice neither about the stability of sucrose (similar variation in the concentrations for both extracts).

#### CONCLUSIONS

Results after six hours of osmotic dehydration of pineapple half-slices at 30, 40 and  $50^\circ\text{C}$  in a hypertonic sucrose solution showed that solute content was a linear function of the water content in the pineapple fruit during osmotic dehydration and this ratio was independent of temperature. Nevertheless, the rate to which the final ratio s/w was reached increased with temperature. These results correlated well to the ratio of diffusion rates of water and sucrose ( $D_w/D_s$ ) that increased in a 35 % when augmenting temperature from 30 to  $50^\circ\text{C}$ .

With regard to the variation of sugars content (fructose and glucose) in the fruit during the osmotic process:



- at 30°C, an increase in experimental values until 600 minutes of process was observed;
- at 40°C no appreciable variations in natural sugars content were detected; even for times as long than 3000 minutes;
- at 50°C a slight decrease in natural sugars content was observed, possibly originated in their leaching by the high water flow that was extracted from the fruit at this temperature.

Water and sucrose concentration profiles showed variation only in the first 2 mm from surface, even at the end of the test period at 40°C (150 min). The shape of the concentration profiles is almost constant after the first 60 minutes of dehydration and there was little variation in concentrations even after 150 minutes of processing.

## RESUMO

A perda da água, o ganho do sacarose e a variação na concentração de outros açúcares naturais de fruta (glucose e fructose) foram estudados durante a desidratação osmótica de fatias de abacaxi, com 0.6 milímetro espessura, em solução de sacarose (60 % w/w) em três temperaturas (30, 40 e 50°C). Como a temperatura foi elevada de 30 a 50°C, as difusividades aparentes da água e da sacarose ( $D_w$  e  $D_s$ ) aumentaram 3.8 e 2.8 vezes, respectivamente; conseqüentemente, o índice da eficiência da desidratação ( $D_w/D_s$ ) aumentou com a temperatura.

A perda de glucose e de fructose aumentou com temperatura. O índice do soluto é uma função linear do índice de umidade e esta relação não é função da temperatura durante os primeiros 600 minutos da desidratação.

As experiências da desidratação osmótica foram realizadas em 40°C com fatias de 10 milímetros de espessura para determinar os perfis da concentração da água e da sacarose em função da posição para valores diferentes de tempos da imersão.

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