

The effects of Sucrose/NaCl/Time interactions on the osmotic dehydration of banana slices

Efeitos de interações de Sacarose/NaCl/Tempo na desidratação osmótica de fatias de banana

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Abstract

The objective of this work was to study the mass transfer kinetics during the osmotic dehydration of banana slices in an osmotic solution prepared by combining sugar with salt. Two levels of sucrose concentration (50 and 60 °Bx), three levels of NaCl content (0, 5% and 10% w/v) and four time levels (90, 180, 270 and 360 min) were applied according to the full factorial technique. The experiments were carried out with three replications and the means analyzed using response surface methodology (RSM). The experimental data revealed that the water loss increased with increase in time, sucrose and salt contents. According to the data obtained the minimum and maximum water losses observed were 9.0% (at 50 °Bx, 0% salt and 90 min) and 46.5% (at 60 °Bx, 10% salt and 360 min), respectively. Furthermore, a small portion of salt was found to reduce the solids gain while the sugar content and time increased it. The effects of all the parameters were significant for water loss, while only those of sucrose content, time and the interaction of salt with sucrose were significant for solids gain. Based on Fick's second law, the effective diffusivity of water in banana slices was evaluated in the range from 5.67×10^{-9} to 9.11×10^{-9} m²/s for the solutions studied.

Keywords: *Banana; Effective diffusivity; Osmotic dehydration; Response surface methodology; Statistical analysis; Mass transfer.*

Resumo

O objetivo deste trabalho foi estudar a cinética de transferência de massa durante a desidratação osmótica de fatias de banana numa solução osmótica preparada pela combinação de açúcar e sal. Dois níveis de concentração de açúcar (50 e 60 °Brix), três níveis de conteúdo de NaCl (0, 5% e 10% p/v) e quatro níveis de tempo (90, 180, 270 e 360 min) foram aplicados de acordo com a técnica fatorial completa. Os experimentos foram conduzidos com três réplicas e as médias foram analisadas usando a metodologia de superfície de resposta (MSR). Os dados experimentais revelaram que a perda de água aumentou com aumento em tempo e conteúdos de sacarose e sal. Conforme os dados obtidos, as perdas mínimas e máximas de água observadas foram de 9,0% (com 50 °Brix, 0% sal e 90 min) e 46,5% (com 60 °Brix, 10% sal e 360 min), respectivamente. Ademais, uma porção pequena de sal reduziu o ganho de sólidos enquanto o conteúdo de açúcar e o tempo aumentaram o mesmo. Os efeitos de todos os parâmetros foram significantes para perda de água, enquanto apenas os efeitos de conteúdo de sacarose, tempo e a interação entre o sal e a sacarose foram significativos para o ganho de sólidos. Baseado na Segunda Lei de Fick, a difusividade efetiva de água nas fatias de banana foi avaliada na faixa de $5,67 \times 10^{-9}$ a $9,11 \times 10^{-9}$ m²/s para as soluções estudadas.

Palavras-chave: *Banana; Difusividade efetiva; Desidratação osmótica; Metodologia de superfície de resposta; Análise estatística; Transferência de massa.*



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Nomenclature

C	molar concentration (mol/m ³)
D	effective diffusivity (m ² /s)
M_0	initial mass (g)
S_0	initial dry mass (g)
S_1	constant related to the rate of water diffusion out (h ⁻¹)
S_2	constant related to the rate of solid diffusion in (h ⁻¹)
SG	solids gain
SG_{∞}	solids gain at equilibrium (%)
SG_t	solids gain at any time (%)
S_t	dry mass at time t (g)
t	time (h)
W_0	initial water content (g)
WL	water loss
WL_{∞}	water loss at equilibrium (%)
WL_t	water loss at time t (%)
W_t	water content at time t (g)
X_0	initial moisture (g _{water} /g)
X_{eq}	moisture at equilibrium (g _{water} /g)
X_t	moisture at time t (g _{water} /g)

1 Introduction

The aim of the osmotic dehydration process is to partially remove the water content of fruits or vegetables immersed in a hypertonic solution. Osmotic dehydration is gaining attention due to its important role in food industries since an osmotic pre-treatment prior to other drying methods improves the colour, flavour and texture of the final product; and is also not an energy intensive process (RASTOGI; RAGHAVARAO, 1997; FERNANDES et al., 2006; SUTAR; GUPTA, 2007).

Banana is a good source of energy with a high nutritional value which makes it one of the most consumed fruits in the world (WALL, 2006; CASTELO-BRANCO et al., 2017). Due to its high respiration rate, bananas are extremely perishable; therefore osmotic dehydration followed by drying is a good technique to prevent the decay of this valuable product (FERNANDES et al., 2006; CABRERA-PADILHA et al., 2014). During the last decade, researchers carried out experiments and developed models to test novel aspects of the osmotic dehydration of the banana (OLIVEIRA et al., 2006; CHAVAN et al., 2010; KADAM; DHINGRA, 2011; MD SALIM et al., 2016).

The rate of osmotic dehydration depends on factors such as the concentration and composition of the osmotic solution, temperature, immersion time, pre-treatments, agitation, nature of the food and the ratio of solution to sample (CORZO; GOMEZ, 2004; CHAUHAN et al., 2010; DA SILVA et al., 2014; LIU; PENG, 2017). Amongst others,

ultrasonic pre-treatment has been reported to improve osmotic dehydration (XIN et al., 2013). Different shapes and sizes of fruits and vegetables have also been studied in the literature (PORCIUNCULA et al., 2013; DA SILVA et al., 2014). The mass diffusion of banana in the transient form was studied with a cylindrical shape. The one-dimensional diffusion equation was obtained to simulate diffusive processes and to determine the thermo-physical parameters (DA SILVA et al., 2011). The water diffusivity of bananas in cylindrical coordinates was estimated using different approaches (PORCIUNCULA et al., 2013).

Numerous studies have been carried out to better understand internal mass transfer and to model the mechanism of the process. In one of these studies, the process of the osmotic dehydration of bananas followed by air-drying was used (OLIVEIRA et al., 2006). It was concluded that a moderate to high sucrose concentration could be used for the osmotic treatment of bananas to reduce the processing time. Banana dehydration according to the thickness of the banana slices, sugar syrup concentrations and sample-to-sugar ratios in the syrup was studied in another work (KADAM; DHINGRA, 2011). They found that the most significant factors were sucrose concentration and the thickness of the slices. In other research project, a process to prepare ripe banana slices using osmotic dehydration was developed and standardized (CHAVAN et al., 2010). The banana slices were successfully osmo-dried, obtaining improves colour, appearance, flavour, texture, taste and overall acceptability, and could be stored for 6 months at ambient conditions without any adverse effect on quality.

Experimental designs have been used by some researchers to study the parameters affecting osmotic dehydration. For example, optimizing the osmotic dehydration of banana with respect to temperature, salt and sucrose concentrations has been investigated (MERCALI et al., 2011). Of the parameters studied, the temperature and osmotic solution composition showed significant effects on the dehydration process. Increasing the temperature and sucrose concentration led to an increase in the effective diffusivities of sucrose, while increasing the NaCl concentration had adverse effects.

The objective of this work is to analyze the osmotic dehydration of banana slices. The effects of time and the osmotic solution composition (sucrose and NaCl concentrations) were investigated with respect to the effective diffusivity, water loss and solids gain. The experiments were carried out based on the full factorial method, and response surface methodology (RSM) was applied to analyze the results obtained. Pareto charts, the interactions and surface plots were used to discuss the effects of the parameters.

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2 Materials and methods

2.1 Samples and pre-treatment

Fresh and ripe bananas (*Musa* spp.) were obtained from the local market daily, prior to each set of experiments. The bananas were peeled and cut into 1 mm thick slices and immediately immersed in a 20 g/L solution of ascorbic acid (Merck) for 5 min to control browning. The thickness to diameter ratio was about 1:20; therefore, radial mass transfer can be assumed to be negligible.

2.2 Osmotic Dehydration

The initial moisture content of the samples was determined by direct heating in a drying oven at 95 °C to 100 °C for 24 h, according to the AOAC method (HELRICK, 1990). To obtain the osmotic solutions, the specified amounts of both sucrose and NaCl (food grade from Merck) were dissolved in distilled water. To study the effects of time and the sucrose and salt concentrations on water loss, solids gain and the diffusion coefficient of water, two levels of sucrose (50 °Bx and 60 °Bx), three levels of salt concentration (0, 5% and 10% w/v) and four time levels (90, 180, 270 and 360 min) were selected, based on the literature review and preliminary experiments. Thus the desired osmotic solutions were prepared as a result of the combination of sugar and salt.

The banana slices were immersed individually in plastic containers, maintaining the 1:5 banana/osmotic solution ratio in order to minimize changes in the solution concentration, which could lead to a local reduction in the osmotic driving force during the process (POKHARKAR; PRASAD, 2002; AZOUBEL; ELIZABETH, 2004). The experiments were carried out at room temperature (26±2 °C) in a shaker, with 100 shakes per minute for all containers. At intervals of 90, 180, 270 and 360 min, three samples were removed from the osmotic solution, rinsed, and the free water of the surfaces carefully collected using filter paper. The samples were weighed using a 4-Digit Laboratory Weighing Balance (AND HR-200, Japan) to determine the moisture loss by comparing the weights obtained at any time with the initial mass of the banana slices. The results reported are the average values of three replications in all cases.

The photos of the banana slices taken during osmotic dehydration under the different conditions can be seen in Figure S1 of the **Supplementary Materials**.

2.3 Water loss and solids gain calculations

The water loss at any time (WL) was calculated using the following equation (Equation 1):

$$WL_t (\%) = \frac{W_0 - W_t}{M_0} \times 100 \quad (1)$$

Where W_0 and W_t are the water contents at zero time and time t , respectively, and M_0 is the initial mass of the banana slices.

Typical osmotic dehydration tests require several hours to reach equilibrium. In some cases, it is not possible to attain equilibrium due to biological, chemical and/or physical instability. Therefore, a two-parameter model for moisture loss (Equation 2), which describes the mass transfer patterns based on a short duration osmosis treatment, was developed (AZUARA et al., 1992). This model also predicts the equilibrium moisture loss. Since the banana structures are biologically and physically sensitive, this model should be used to calculate the equilibrium water loss values.

$$WL_t = \frac{S_1 t (WL_\infty)}{1 + S_1 t} \quad (2)$$

where WL_t is the water loss at time t , WL_∞ is the corresponding quantity at equilibrium, S_1 is the constant related to the rate of water diffusing out from the product and t is time. During the osmotic dehydration of banana slices, the constant S_1 and moisture loss at equilibrium can be determined by linear regression, using the experimental data obtained. The linear form is presented in Equation 3:

$$\frac{t}{WL_t} = \frac{1}{S_1 (WL_\infty)} + \frac{t}{WL_\infty} \quad (3)$$

The solids gain SG_t of the fruit after time t of osmotic treatment is defined as Equation 4 (SINGH et al., 2007):

$$SG_t (\%) = \frac{S_t - S_0}{M_0} \times 100 \quad (4)$$

where S_t is the dry mass of banana after time t of osmotic dehydration and S_0 is the dry mass of the fresh banana. Similar to the water loss calculation, the following equation (in the linear form) for solids gain during the osmotic dehydration of fruits was developed by Azuara et al. (1992) as Equation 5:

$$\frac{t}{SG_t} = \frac{1}{S_2 (SG_\infty)} + \frac{t}{SG_\infty} \quad (5)$$

where SG_t is the solids gain at any time, S_2 is the constant related to the rate of solids diffusion in the banana and SG_∞ the solids gain at equilibrium.

2.4 Mathematical procedure

There are several methods reported in the literature for the estimation of diffusivity for water transport during osmotic dehydration, based on the solution of Fick's second law. Fick's second law for unidirectional unsteady state diffusion is given by Equation 6.

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial y^2} \quad (6)$$

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This equation can be applied for infinite slabs of fruit dehydrated from both sides. By applying the following boundary conditions (Equation 7), the analytical solution of Equation 6 can be obtained for diffused water during osmotic treatment (Equation 8):

$$\begin{aligned} X &= X_0 \quad @ t = 0 \quad \& -L < y < L \\ X &= X_t \quad @ t > 0 \quad \& y = \pm L \end{aligned} \quad (7)$$

$$\frac{X_t - X_{eq}}{X_0 - X_{eq}} = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-D(2n+1)^2 \pi^2 t}{L^2}\right) \quad (8)$$

Where X_t is the moisture at time t , X_0 the initial moisture and X_{eq} the moisture at equilibrium. D is the effective diffusivity of water and L half the thickness of the slices. The least square error method was used to find the best D for this process. Equation 8 is a complex equation and the proper D cannot be obtained using regular methods such as linearization. Therefore, the parameter Z was defined from Equation 9 and the plot of Z versus D was used to find the minimum quantity of Z . A Visual Basic for Application (VBA) source code was written to carry out these calculations.

$$Z = \sum \left(\frac{X_{cal} - X_{eq}}{X_0 - X_{eq}} - \frac{X_{exp} - X_{eq}}{X_0 - X_{eq}} \right)^2 \quad (9)$$

2.5 Experimental design

In this study, the Minitab software (version 14.12.0) was applied for a full factorial experimental design and the data analyzed using Response Surface Methodology (RSM). The analysis of variance (ANOVA) was also used to analyze the data to investigate the interaction between the process variables at a 95% confidence level.

RSM is a technique used to evaluate the effects of the operational conditions and to achieve the best

conditions for desirable responses with a limited number of planned experiments (HEYDARI et al., 2013). A regression design was used to model a response as a mathematical function of factors. Each response can be expressed as a second-order polynomial, according to Equation 10.

$$R = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \sum_{j=i+1}^k \beta_{ij} x_i x_j + \sum_{i=1}^k \beta_{ii} x_i^2 \quad (10)$$

where R is the predicted response used as a dependent variable (R_1 = water loss in g/100 g of fresh fruit, R_2 = solids gain in g/100 g of fresh fruit and R_3 = the effective diffusivity); k is the number of independent variables (factors), which is 3 in this case; x represents the independent factors (x_1 = sucrose concentration, x_2 = salt concentration and x_3 = process time); β_0 is the constant coefficient, and β_i , β_{ij} and β_{ii} are the coefficients of linear, interaction and quadratic terms, respectively.

According to Equation 10 the quadratic equations for effective diffusivity (D), solids gain (SG) and water loss (WL) were obtained using Equations 11, 12 and 13 respectively:

$$D = 10^{-9} (-9.02 + 0.58x_1 - 5.69 \times 10^{-3} x_1^2 + 0.72x_2 - 6.65 \times 10^{-3} x_2^2 - 5.79 \times 10^{-3} x_1 x_2) \quad (11)$$

$$SG = 1.33 - 0.23x_1 + 4.86 \times 10^{-3} x_1^2 + 1.61x_2 + 2.11 \times 10^{-3} x_2^2 + 2.71 \times 10^{-3} x_3 - 5.81 \times 10^{-6} x_3^2 - 3.16 \times 10^{-2} x_1 x_2 + 1.33 \times 10^{-4} x_1 x_3 + 3.08 \times 10^{-4} x_2 x_3 \quad (12)$$

$$WL = -8.13 - 0.25x_1 + 3.69 \times 10^{-5} x_1^2 + 5.48x_2 - 0.21x_2^2 + 1.45 \times 10^{-2} x_3 - 5.59 \times 10^{-5} x_3^2 - 3.81 \times 10^{-2} x_1 x_2 + 1.08 \times 10^{-3} x_1 x_3 + 1.76 \times 10^{-4} x_2 x_3 \quad (13)$$

3 Results and discussion

3.1 Effects of time, sucrose and salt on water loss

To find the effect of time on water loss, dehydrated samples were weighed after different periods. All the reported data are averages of the results obtained. Figure 1 shows that the water loss increased non-linearly with time for all

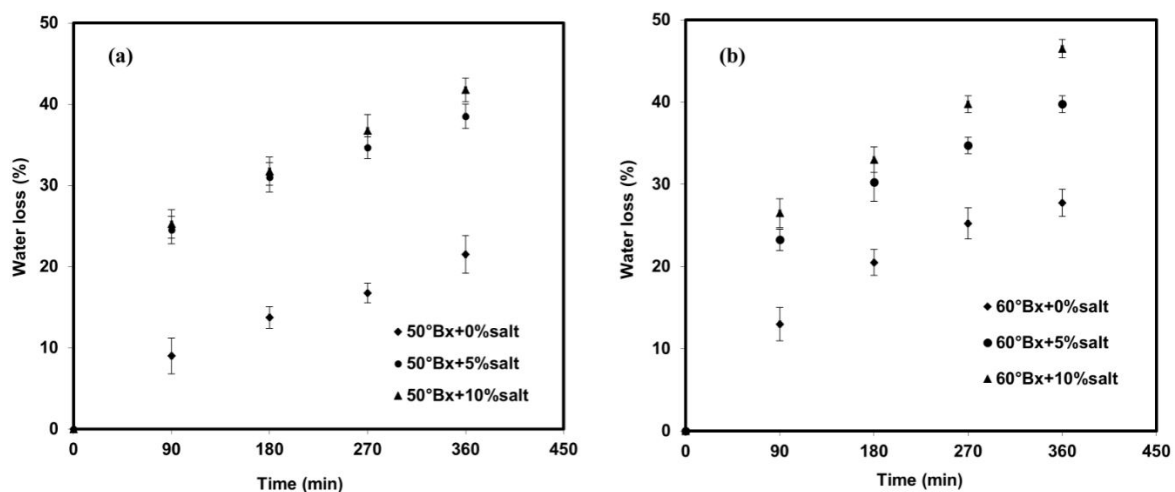


Figure 1. Variation of water loss with percent salt in (a) 50 °Brix sucrose and (b) 60 °Brix sucrose.

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osmotic solution concentrations, being faster at the start of osmosis and decreasing thereafter. This implies that the driving force of water diffusion decreases with time, since during the osmotic process the water moves gradually from the sample towards the bulk solution. In addition, the rapid mass transfers near the surface at the beginning of the process caused some structural changes, leading to compaction of these surface layers followed by increased mass transfer resistance as the process continued (LENART; FLINK, 1984). Similar results were also reported for the osmotic dehydration of potatoes (LENART; FLINK, 1984).

According to Figure 1, the water loss increased with increasing sucrose concentration (from 50 °Bx to 60 °Bx) and salt percentage (from 0 to 10%). This increase was attributed to the mass transfer intensifying in high sucrose and salt concentrations. Similar results were reported for the osmotic dehydration of carrots (SINGH et al., 2007) and of onion slices (SUTAR; GUPTA, 2007), and another work with the osmotic dehydration of banana slices reported that the weight loss increased with increasing time, sugar content and the solution concentration to sample ratio for different solutions (PREETHI, 2014).

The response surfaces of the water loss presented in Figure S2 of the **Supplementary Materials** also confirmed

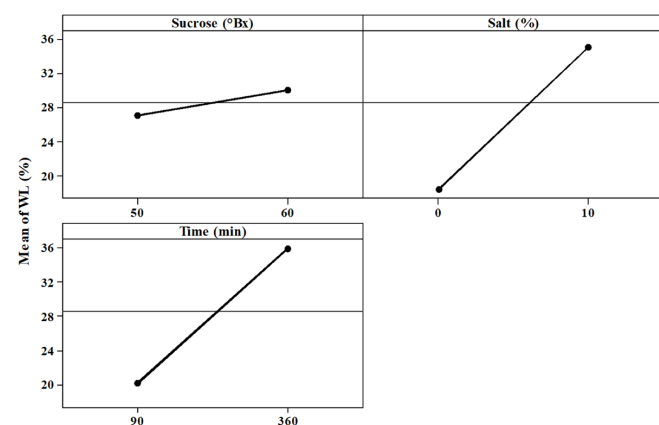


Figure 2. Main effects plot for water loss (%).

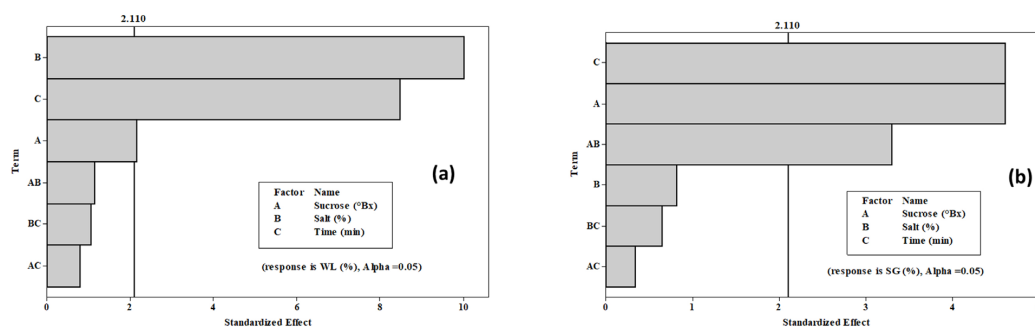


Figure 3. Pareto chart of the standardized effects of NaCl and sucrose on (a) water loss and (b) solids gain.

that the water loss increasing with increase in sucrose concentration, salt percentage and time.

3.2 Statistical analysis

3.2.1 Water Loss

The first parameter studied was the water loss of the samples as an indicator of the efficiency of the osmotic dehydration. As shown in Figure 2, increases in sucrose content, salt concentration and process time led to increased water loss. This tentatively suggests that these parameters need to be fixed at their maximum levels to achieve maximum water loss. The effect of sucrose was less than that of the other factors. In parallel, the Pareto chart of the standardized effects for water loss in Figure 3(a) revealed that the sucrose content lay on the border of significance while the salt content and time were strongly significant. In addition, the interaction parameters were not significant at the 95% confidence level. These results are in good agreement with another report which showed that the concentrations of NaCl and sucrose significantly affected water loss for the osmotic dehydration of tomato (TELIS et al., 2004).

To provide a better picture, the interaction plot for water loss is presented in Figure 4. According to the interaction plots for water loss, no abnormal effect could be observed for either the maximum or minimum levels of the variables. An increase in level of a variable only resulted in an increase in the intercepts and a gradual change in the slopes of the diagrams can be observed.

3.2.2 Solids gain

Solids gain, considered to be an unfavourable phenomenon in osmotic dehydration, was the second parameter studied. Impregnation of solutes into fruit slices changes the taste of the final product leading to product rejection by consumers (MERCALI et al., 2010). On the other hand, increased weight due to solids gain results in an under-estimation of the amount of dehydration. The main effects plot for solids gain (%) is shown in Figure 5. From Figure 5, it can be concluded that the

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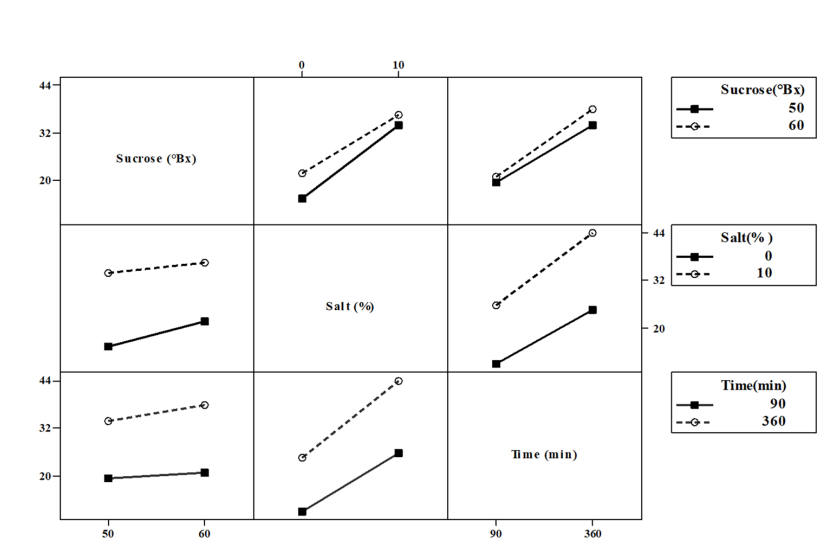


Figure 4. Interaction plot for water loss (%).

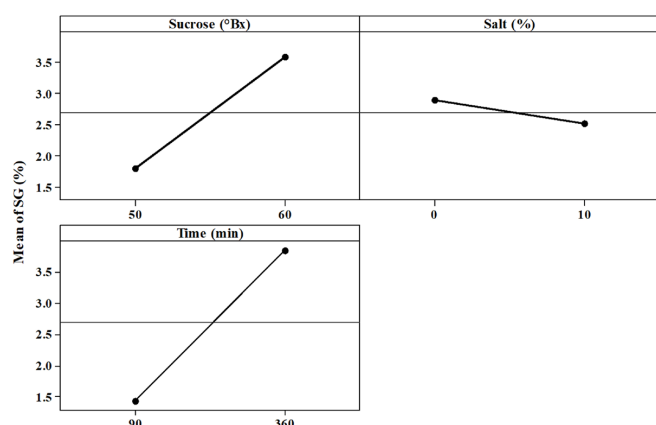


Figure 5. Main effects plot for solids gain (%).

sucrose concentration increases solids gain while NaCl decreases it slightly; and the effect of sucrose was more noticeable than that of NaCl. In comparison to Figure 2, Figure 5 shows that the slope of the sucrose effect for solids gain was greater than that for water loss.

The addition of small quantities of sodium chloride (salt) to osmotic solutions increased the driving force of the drying process as well as the synergistic effects between the sucrose and sodium chloride, and the addition of sucrose to the solution resulted in increased water loss, while increases in salt showed the opposite effect (AZOUBEL; ELIZABETH, 2004). In many published papers, sucrose has been the first choice for osmotic dehydration since the change in the taste of the final product from the impregnation of sucrose is negligible. It was concluded that small amounts of salt had a favourable effect on the processes by increasing water loss and water diffusivity and by reducing solids gain.

The Pareto chart of the standardized effects for solids gain in Figure 3(b) revealed that the effects of both time and sucrose content were significant while the effect of salt concentration was not. However, when parameter interactions were considered, only the interaction between NaCl and sucrose was significant.

The interaction plot for solids gain is shown in Figure 6. Figure 6 (a) shows that the presence of a small portion of salt decreases the slope of solids gain against sucrose concentration, suggesting that a concentrated NaCl solution hinders the excessive penetration of sucrose in the banana. Also, it must be noted that solids gain increased with an increase in salt at 50 °Bx sucrose. At the start and end of the process, solids gain increased with increased sucrose levels as shown in Figure 6 (b). Figure 6 (c) shows that an increase in salt content at lower sucrose concentrations increases the solids gain, while at high sucrose levels, the presence of salt decreases the solids gain. This is an important finding, implying that high sucrose levels can be used to accelerate water loss, while the solids gain is controlled by the addition of salt. Figure 6 (d) shows that an increase in salt reduces solids gain in the initial stages of the process, while at equilibrium, changes in the salt concentration have no effect on solids gain. As presented in Figure 6 (e), at both sucrose levels, an increase in dehydration time increases the solids gain at almost the same rate, although solids gain in 60 °Bx is higher than in the 50 °Bx sucrose solution. The effects of higher and lower levels of salt during the process are presented in Figure 6 (f), indicating that solids gain is only slightly affected by the salt concentration.

Figure S3 of the supplementary materials shows the response surfaces for solids gain. This figure confirmed the importance of the sucrose/salt interaction on solids gain and also showed that the sucrose/time and salt/time interactions had no significant effects on solids gain.

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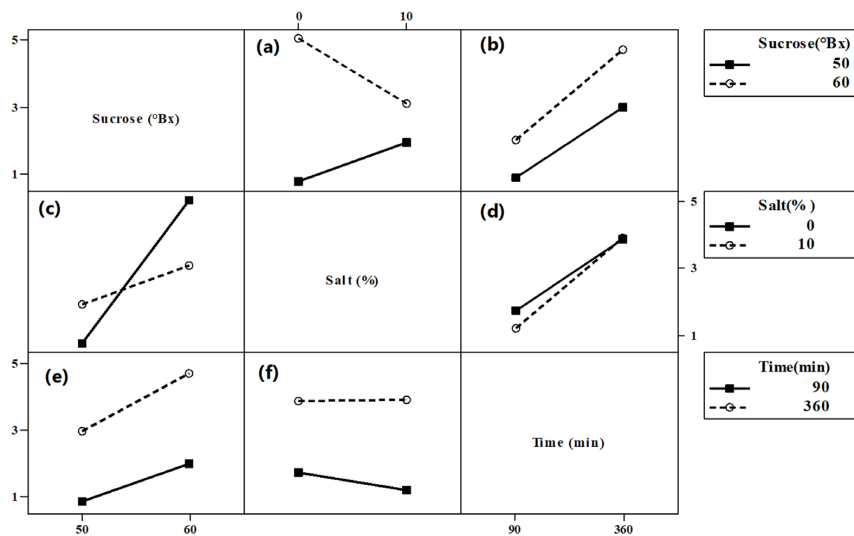


Figure 6. Interaction plot for solids gain (%), (a, b) Sucrose effect, (c, d) Salt effect and (e, f) Time effect.

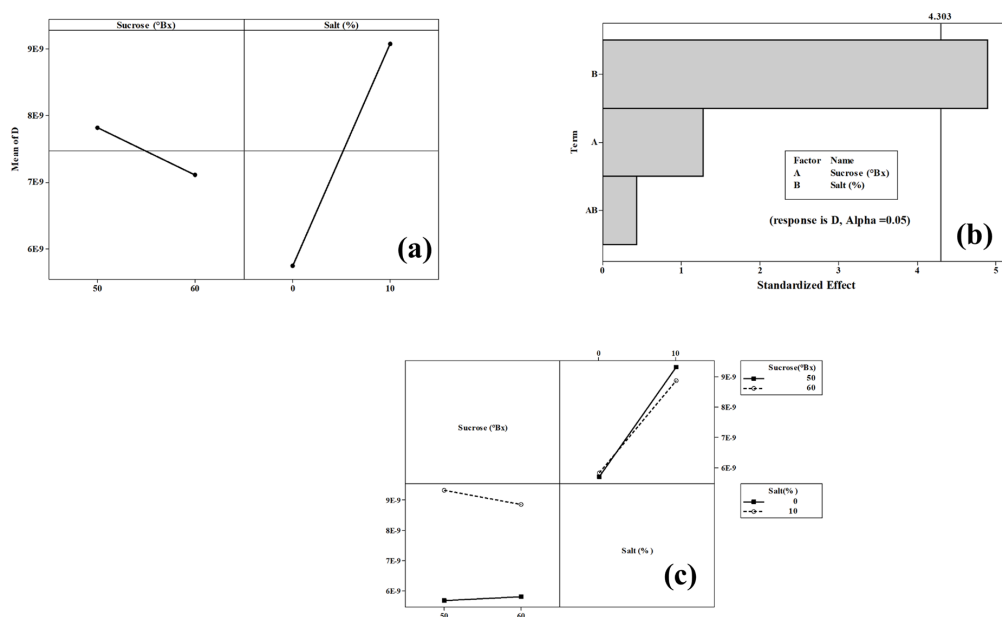


Figure 7. (a) The main effects, (b) Pareto chart and (c) Interaction plots obtained for effective diffusivity (m^2/s).

3.3 Effective diffusivity of water

Figure 7 (a) (as well as Figure S4 in the **Supplementary Materials**) shows the main effects for effective diffusivity. The figures clearly show that salt increases diffusivity while sucrose decreases it and that the effective diffusivity of water varied from 5.67×10^{-9} to $9.11 \times 10^{-9} m^2/s$. The results are in agreement with those of other studies, for example, the results obtained for the osmotic dehydration of tomatoes in different solutions (TELIS et al., 2004) and for the osmotic dehydration of banana slices (SANKAT et al., 1996; WALISZEWSKI et al., 1997; MERCALI et al., 2010). The values calculated for effective diffusivity in the present study are of the same order of magnitude (i.e. $O(-9)$) as

those reported in other studies (SANKAT et al., 1996; WALISZEWSKI et al., 1997; MERCALI et al., 2010), and the slight differences may be attributed to the thicknesses of the slices and to the compositions of the osmotic solutions. The results obtained for the effect of salt are also in agreement with the results obtained for the osmotic dehydration of sardine slices in brine (CORZO; GOMEZ, 2004). The opposite behaviour determined for the increase in sucrose concentration was validated by the osmosis of jenipapo in sucrose syrup (CARDOSO ANDRADE et al., 2007).

Furthermore, according to the Pareto charts shown in Figure 7 (b), the effective diffusivity was related to the percentage of salt, which presented a significant effect.

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Compared to salt, the presence of sucrose did not present a significant influence on the effective diffusivity.

The interaction plot for effective diffusivity is also presented in Figure 7 (c), from which it can be concluded that the presence of salt alters the dependency of effective diffusivity on the sucrose content. According to the data obtained, adding salt to the solution leads to an increase in the effective diffusivity of water of up to 1.5 times (i.e. from $6E-9$ to $9E-9$ m²/s).

4 Conclusion

The rates of water loss and solids gain in the osmotic dehydration of banana slices were related to the processing time and composition of the solution. The system was modelled based on Fick's second law. The resulting equation was solved analytically and the effective diffusivity was found for each of the experiments. From the results it was observed that water loss increased non-linearly with time at all concentrations and was faster in the initial period of osmosis, followed by an almost constant rate, which might be due to a reduction in the osmotic driving force during the process. The findings obtained imply that the effects of sucrose on water loss and solids gain can be controlled by NaCl and vice versa. According to the statistical analyses, all the parameters studied had significant effects on water loss, while only the sucrose content, process time and the interaction of salt and sucrose significantly affected solids gain. For effective diffusivity, only the salt content was found to be a significant parameter, at the 95% confidence level.

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■ **Supplementary material**

The following online material is available for this article:

Supplementary Material containing Figures S1, S2, S3 and S4.

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