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Osmotic dehydration of tilapia fillets in limited volume of ternary solutions

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Abstract

In this study, water activity, moisture content reduction and solute uptake kinetics as well as mass transfer in osmotic dehydration of tilapia *(Oreochromis niloticus*) fillets were studied, using limited volumes (solution/fillet ratio: 4/1) of ternary solutions (salt–sugar–water) at different initial sucrose (0–100 g in 100 g of water) and salt (0–35.14 g in 100 g of water) concentrations at 20 °C. The multicomponent effective diffusion coefficients were calculated by fitting the experimental values to analytical solution of mass transfer equations with a limited solution/fillet ratio. Previously, the effective diffusion coefficients of salt and sugar in tilapia muscle were calculated using a binary system. A very good fit between the mathematical model and experimental values was observed. The binary coefficients obtained were: for sodium chloride, D_{11}, $0.91 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$ and sucrose, D_{22}, $0.22 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$, and the calculated cross-term coefficients, D_{12} and D_{21}, were -0.12×10^{-9} and $0.06 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$, respectively. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Sucrose; Salt; Multicomponent diffusion; Effective diffusivity

1. Introduction

Osmotic dehydration, a water removal process used for the concentration of water-rich food by immersion in concentrated solutions of salt, sugar, acids, etc. has been widely studied as a convenient method to improve the economics of dehydration processes [1–3] as well as the nutritional and organoleptic characteristics of the products which usually undergo further drying by conventional means [4–6].

Concentration results primarily from an osmotic water flow, caused by the water and solute activity gradients across the semi-permeable cellular membrane. Since the membrane is partially selective, there is always some solute diffusion into the food [7].

Aqueous binary solutions of NaCl are commonly used in osmotic dehydration or fish salting [8–10]. The NaCl has higher water activity reduction capacity and causes a considerable impregnation in animal tissues, but has limited capacity to improve weight loss and moisture content reduction. Sucrose solution, contrary to the sodium chloride solution, has higher moisture content reduction capacity. In general if the molecular weight of the solutes is slightly increased, it is possible to decrease solute gain for an equivalent water loss, which results in a greater weight reduction [11]. The combination of both solutes may provide the advantages of each one [11,12].

In osmotic dehydration and wet salting, it is common to use the assumption of constant solution concentration, satisfied by employment of high solution to food ratio, which makes the interpretation and modeling easier [13]. This assumption can be good for laboratory scale works. However, the cost of great volumes of solution and the great amount of solution to discard or recycle are factors to be considered in an industrial application. Use of small volumes implies significant changes in the solution composition during the process with the advantage of improvement of the process control, since the rate uptake of solutes by the food can be deduced from observations of the concentration in the solution [14–17].

Until about 30 years ago, little attention was given to the understanding of the basic mechanisms involved in salting fish, but it is now generally accepted that salt migration by diffusion plays an important role in salting [18,19].

The Nile tilapia (*Oreochromis niloticus*), an important farmed fish produced in various parts of the world, very much sought by its low fat meat, shows a growing consumption in Brazil. In this study, the kinetics and mass transfer in tilapia fillets osmotically dehydrated with ternary solutions is presented, as its behavior while passing through some sort of salting processes is not well explored until now. Also

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Nomenclature

a_{w}	water activity
c_i	concentration of species i (kg/m^3)
C_i	concentration of species i in solution (kg/m^3)
D_{ij}	multicomponent diffusion coefficient (m/s^2)
F	fillet volume (m^3)
J	flux (g/m^2 s)
L	solution volume (m^3)
m_i	partition coefficient
N	number of experimental data
q_n	nonzero roots of $\tan q_n = -\alpha q_n$
t	time (h)
x	half-thickness (m)
V_{c}	calculated value
V_{e}	experimental value

Greek symbols

α	volumes ratio
β	parameters of Eq. (13)
σ_i	eigen values of modal matrix
ψ	transformed variable

Sub indices

i, j	solutes
n	series term
∞	at infinite time

tilapia represents a good model system for studying mass transfer in fishes.

The main objectives were to study the influence of each solute (salt and sucrose) on the equilibrium, kinetics and mass transfer into the tilapia fillet using small volumes of binary and ternary solutions, with the purpose of: (1) analyzing the water activity and moisture content reduction and solutes uptake kinetics of fish fillets during osmotic dehydration with binary (sucrose/water or sodium chloride/water) and ternary (sucrose + sodium chloride/water) solutions; (2) determining the binary and multicomponent diffusion coefficients by fitting the experimental values to the analytical solution of a mass transfer equation with a limited solution/fillet ratio.

2. Theory

Usually, the classical diffusion equation, Eq. (1), is sufficient to model a binary diffusion process or calculate the binary diffusion coefficient in this same process

$$\frac{\partial c_i}{\partial t} = D_{ij}\frac{\partial^2 c_i}{\partial x^2} \tag{1}$$

The use of the above equation is possible when species (i) diffuses into a fixed solid matrix (*j*) in a stagnant liquid without shrinkage and the diffusion coefficient is constant during the process.

However, osmotic dehydration in a ternary solution, e.g. sugar and salt in water, produces diffusion of sucrose (in), sodium chloride (in) and water (out) into product. In this case ternary diffusion equation could describe the system assuming that it consists of two solutes and a matrix (homogeneous) [20]. When simultaneous diffusion of sodium chloride and sucrose into the fish fillet occurs, the diffusion process of each solute is affected by the presence of the other component [11,12]. For each solute, the continuity equation is:

$$\frac{\partial c_i}{\partial t} = -\frac{\partial j_i}{\partial x} + c_i v^0, \quad i = 1, 2 \tag{2}$$

where c_i are the concentrations, v^0 the volume-average velocity.

To the multicomponent diffusion, the constitutive equation is the generalized Fick's law [20]:

$$j_i = \sum_{j=1}^{n-1} D_{ij}\nabla c_j, \quad i, j = 1, \ldots, n-1 \tag{3}$$

where j_i is the flux of species "i" relative to volume-average velocity, D_{ij} the multicomponent coefficient. In general the diffusion coefficient are not symmetrics, i.e. $D_{ij} \neq D_{ji}$. The diagonal terms, D_{ii} are called the main-term coefficients and generally equal to the binary coefficients. The off-diagonal terms, called the cross-term coefficient, are of a lower value than the main-term coefficient.

Substituting Eq. (3) into Eq. (2) and assuming absence of chemical reaction, one-dimensional diffusion, volume-average velocity (v^0) zero, diffusion coefficients independent of solute concentration and neglecting the convective flux, the ternary diffusion equation is obtained:

$$\frac{\partial c_i}{\partial t} = \sum_{j=1}^{2} D_{ij}\frac{\partial^2 c_j}{\partial x^2}, \quad i = 1, 2 \tag{4}$$

The analytical solution of the above equation may be more easily achieved, defining a new concentration Ψ_k, according to the method developed by Toor in 1964 [20] that implies in a modified ternary diffusion equation:

$$\frac{\partial \Psi_i}{\partial t} = \sigma_i\frac{\partial^2 \Psi_i}{\partial x^2}, \quad i = 1, 2 \tag{5}$$

where σ_i are the eigen values of modal matrix.

The initial and boundary conditions of Eq. (5) to solve this uncoupled differential equation are:

$$C_i = C_{i0} \quad \text{and} \quad \Psi_i(x, 0) = 0, \quad t = 0,\ i = 1, 2 \tag{6}$$

$$\frac{\partial \Psi_i}{\partial x} = 0, \quad x = 0,\ t > 0,\ i = 1, 2 \tag{7}$$

$$\alpha_i l\frac{\partial C_i}{\partial t} = \pm\sum_{j=1}^{2}\sigma_i\left(\frac{\partial \Psi_i}{\partial x}\right), \quad x = \pm\, l,\ t > 0,\ i = 1, 2 \tag{8}$$

The analytical solution is similar to that obtained by Crank [14]:

$$f(\sigma_i) = \frac{\Psi_{it} - \Psi_{i\infty}}{\Psi_{i0} - \Psi_{i0}} = \sum_{n=1}^{\infty} \frac{2\alpha_i(1+\alpha_i)}{1+\alpha_i+\alpha_i^2 q_{ni}} \exp\left(\frac{-\sigma_i q_{ni}^2 t}{l^2}\right),$$
$$i = 1, 2;\ n = 1, \dots, \infty \tag{9}$$

where α_i is the solution and fillet volumes ratio ($\alpha_i = m_i L/F$), m_i the partition coefficient, l the half-thickness and q_n the nonzero roots of $\tan q_n = -\alpha q_n$. The concentrations in Eq. (9) are not those actually measured; so converting this one into the actual concentrations observed results [21]:

$$\frac{c_{it}}{c_{i\infty}} = 1 - \{A_i f(\sigma_i) + (1 - A_i)[f(\sigma_j)]\} \tag{10}$$

$$A_i = \frac{D_{ii} - \sigma_j + \kappa_{ij} D_{ij}}{\sigma_i - \sigma_j}, \quad i, j = 1, 2;\ i \neq j \quad \text{and}$$
$$\kappa_{ij} = \frac{c_{j0} - c_{j\infty}}{c_{i0} - c_{i\infty}}, \quad i, j = 1, 2;\ i \neq j \tag{11}$$

where c_i are solutes concentration in the fillet.

3. Material and methods

The fillets of Nile tilapia (*O. niloticus*), acquired at a local market, were obtained and processed up to 24 h after capture. Their physical characteristics were: fish weight 300–350 g, length 23–25 cm, average thickness and specific mass of the fillets 1.33–1.55 cm and 1.065–1.072 g/cm^3, respectively.

The binary osmotic solutions were composed by 0, 25 and 35.14 g of sodium chloride in 100 g of water or 0, 66.67 and 100 g of sucrose in 100 g of water, and ternary solutions were composed by 25 + 66.67, 25 + 100, 35.14 + 66.67 and 35.14 + 100 (g of NaCl + g of sucrose)/100 g of water. The solutions were prepared by dissolution of required amounts of these solutes, alone or combined, in distilled water.

The fillets and solutions were previously conditioned at 20 °C. Afterwards, one fillet properly dried on absorbent paper and weighted was placed in 500 ml flasks with the solution at the required concentration with a solution/fillet ratio, 4/1 (v/v). Finally, the flasks were placed in a thermostatic shaker, assuring high Biot number, at different times up to 48 h to determine equilibrium conditions: concentration and partition coefficient calculations, which are both necessary to calculate the diffusion coefficients (Eq. (10)). However, the recommended process time can be determined after analysis of variables rates (a_w, moisture content or solute uptake), establishing as final time of osmotic dehydration process that one where the rate of reduction (of water activity or moisture content) or solutes uptake is not changing anymore.

Twelve flasks were used for each trial to assure that the established ratio volumes between the solution and the fillet were constant in each point (time) of the sampling. At different processing times, the flasks were removed from the shaker and the solution was drained and weighted. The fillets were gently blotted to remove surface moisture, weighted and finally ground and homogenized.

The water activity, for fillet and solution, was measured in duplicate with three readings per sample with an Aqualab CX-2T (Decagon, USA) device linked to a 25 °C water bath. Fillet water activity at equilibrium was obtained by comparison to the solution water activity. Equilibrium was considered when their values were equal [17]. The moisture, sodium chloride and the sucrose contents were determined in duplicate by using the AOAC Official Method [22], Na$^+$ quantification, using flame photometer analyzer 910 M and by the Lane and Eynon method, described by Banganna [23], respectively. Equilibrium values (moisture content, sodium chloride and sucrose concentrations) were calculated by extrapolating plotted experimental points as a function of time with a two-exponential decay equation.

In order to obtain the binary diffusion coefficients of both solutes (D_{11} and D_{22}) the procedure described by Medina-Vivanco et al. [17] was used. For the ternary diffusion coefficients calculations, D_{11} and D_{22} values were employed and different D_{12} and D_{21} were selected, to get the least root mean square (RMS) percentage deviation between theoretical and experimental solute contents.

$$\text{RMS}\,(\%) = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\frac{V_{ei} - V_{ci}}{V_{ei}}} \tag{12}$$

where V_{ei} is the experimental value, V_{ci} the calculated value and N the number of experimental data points.

A factorial experimental design of two factors and three levels (nine experimental runs, performed in random order) was used aiming the evaluation of initial solution concentration effects of salt (0, 25 and 35.14 g in 100 g of water) or/and sucrose (25, 66.67 and 10 g in 100 g of water) on the equilibrium values. The dependent variables were moisture content (MC), water activity (a_w), sodium chloride and sucrose contents in the fillet at equilibrium. A second order polynomial model was used (Eq. (13)) to describe the response variables Y_i (a_w, MC, NaCl and sucrose) as a function of the variable X:

$$Y_i = \hat{\alpha}_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 \tag{13}$$

where X_1 and X_2 are NaCl and sucrose concentrations in osmotic solution, respectively.

The program Statistica 5.0 Copyright© StatSoft (1995) was used for the experimental design statistic analysis and establish the response surfaces with a significance level of 95%.

4. Results and discussion

The kinetics of water activity reduction for the different concentrations of binary and ternary solutions are shown in

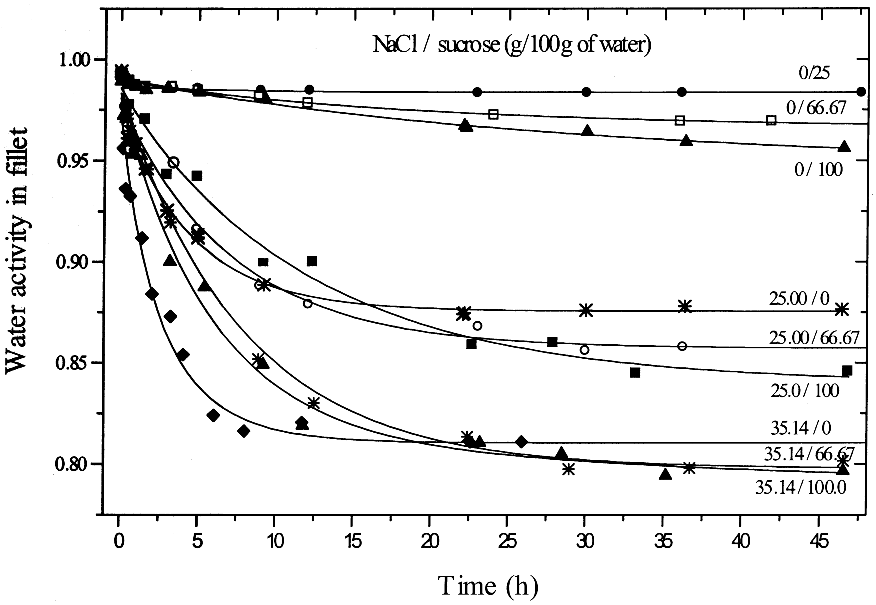

Fig. 1. Kinetics of water activity in fillet using binary and ternary solutions.

Fig. 1. In all cases, water activity (a_w) values decreased with time and for a long time decreased with the increase of initial concentration. Equilibrium water activity values, when sucrose solutions were used, are very high compared to those obtained when employing sodium chloride solutions. For ternary solutions, the influence of sucrose is minimal. This may be due to a greater potential to reduce water activity shown by brine, compared to sucrose solutions, as evidenced by the initial water activity values of binary solutions: 0.981, 0.962 and 0.934 for the aqueous sucrose solution concentrations of 25, 66.67 and 100 g in 100 g of water, and 0.843 and 0.762 for aqueous sodium chloride solution concentrations of 25 and 35.14 g in 100 g of water, respectively.

The initial water activity values for ternary solutions were: 0.831, 0.808, 0.790, 0.753 and 0.728 for NaCl/sucrose, 25/25, 25/66.67, 25/100, 35.14/25 and 35.14 g/100 g in 100 g of water, respectively. These results seem to demonstrate that the salt has a greater influence on these values due to its molecular weight and ionization capacity. Other important behavior is referred to the time necessary to reach the equilibrium state. When sucrose concentration is increased in the binary or ternary solutions, higher times are necessary to reach equilibrium. This difference in water activity reduction behavior presumably arises from differences in molecular size of ionized salt versus the larger and unionized sugar [7] that causes a low diffusion of this solute and, probably, also affecting and decreasing sodium chloride diffusion [12]. Statistic analysis indicated that the effects of initial sodium chloride and sucrose solution concentrations (alone or mixed) on equilibrium a_w in tilapia fillet were negative and significant (95%), although the effect of initial sodium chloride solution concentration was 16 times greater than sucrose solution concentration (Table 1).

According to Fig. 1, the adequate process time, where no more water activity reduction is observed, could be fixed among 5–10 h, depending on solution concentration used.

The kinetics of moisture content reduction for binary and ternary solutions can be seen in Fig. 2. The moisture content, as the water activity values, decreased with time and for higher initial solution concentration, but contrary to the observed for water activity, the moisture content values achieved were lower when sucrose was added. These results are in agreement with those related by literature [8,9,24] and confirm that sucrose is a good dehydrating agent, due to its high molecular weight, that makes possible an enhanced water output. As this solute diffuses slowly, remaining for a longer time at the fillet surface, it probably causes a high concentration gradient between the product and the solution allowing greater water loss [11]. Statistic analysis of equilibrium values showed negative and significant effects for both solutes (95%), being the sucrose effect twice as greater as sodium chloride effect (Table 1).

Table 1
Regression coefficients and analysis of variance of the second-order polynomial equation

Coefficient	a_w	Moisture content	NaCl content	Sucrose content
β_0	0.99158	0.835959	0.31822	0.356074
β_1	−0.00448*	−0.003869*	0.00924*	−0.007814*
β_2	−0.00028*	−0.006797*	−0.01226	0.011722
β_{11}	−0.00002*	−0.000037	0.00009	−0.000029
β_{22}	−0.000001*	0.000028	0.000093	−0.000084
β_{12}	−0.000007*	0.000035	0.000024	0.000026
R^2	0.999	0.987	0.856	0.932

* Significant effects ($P \leq 0.05$) of different variables.

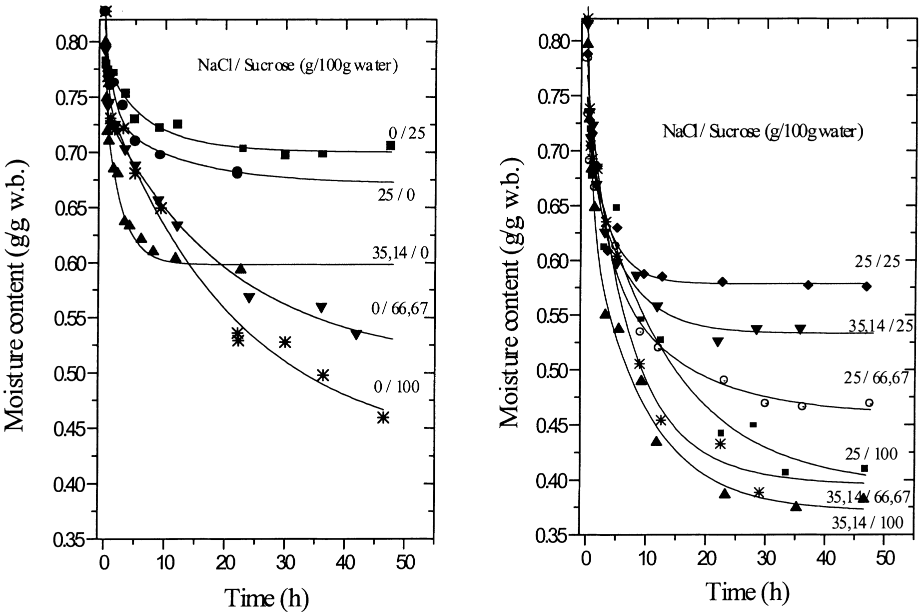

Fig. 2. Kinetics of moisture content reduction for binary and ternary solutions.

Considering the moisture content reduction as the most important variable, the adequate process time was between 10 and 15 h, depending on solution concentration used.

The NaCl uptake kinetics is presented in Fig. 3. There is a fast increase of salt level during the first few hours, characterizing a first period of high transfer rates. Probably this is a consequence of a larger initial driving force, i.e. a larger difference between the solution osmotic pressure and the fillet, caused by a strong difference between the concentrations of salt in the brine and fillet. The driving force decreases while the process goes on, not only due to an increment of NaCl concentration in the fillet, but also as a consequence of the brine dilution by water migration.

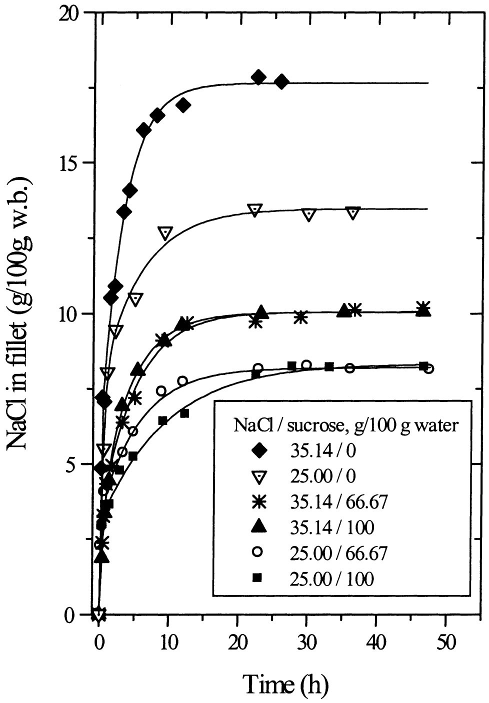

Fig. 3. Kinetics of NaCl content in fillet for initial solution concentration.

Statistic analysis pointed positive and significant effect of initial sodium chloride solution concentration. When ternary solution began to be used initial sucrose solution concentration influenced negatively on the NaCl content in fillet, with a decrease of fillet NaCl contents, if compared to binary solutions. Sucrose addition cause “dilution” of solution, as a consequence of sugar addition and of the resulting competition between the two solutes. Sucrose effect on NaCl content in fillet among concentration range 66.67–100 g in 100 g water is not obvious, and the different moisture content (Fig. 2, right) may be affecting this behavior.

Sucrose uptake kinetic presents similar behavior of salt uptake (Fig. 4) however with lower penetration rates. Similarly to the previous situation, sucrose content in fillet was negatively influenced by increase of NaCl solution concentration in the range 0–25 g in 100 g water. For higher concentrations, this effect disappeared.

Experimental and predicted values for sucrose and sodium chloride contents in tilapia fillet osmotically treated by ternary solutions (NaCl/sucrose, 35.14 g/100 g in 100 g of water) are showed in Fig. 5. The model used, Eqs. (9) and (10), described very well the behavior of sucrose and sodium chloride diffusion into the fillet. Lombardi and Zaritzki [25] and Gerla and Rubiolo [21] used similar models to determine cross-diffusion coefficients for citric and

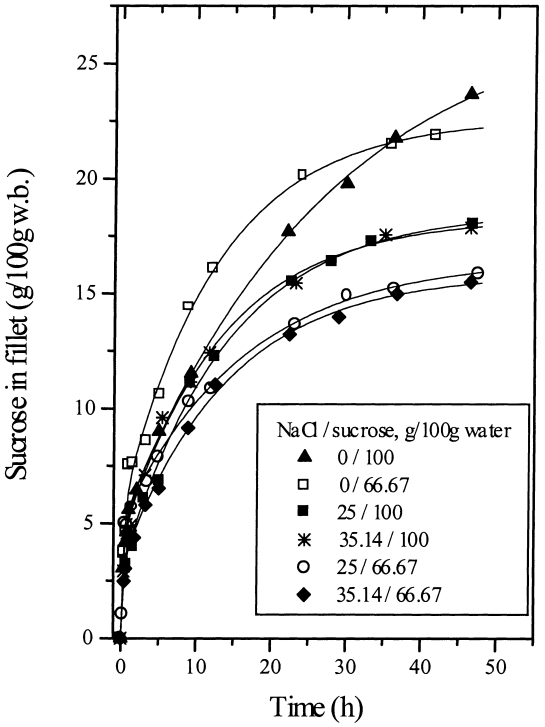

Fig. 4. Kinetics of sucrose content in fillet for initial solution concentration.

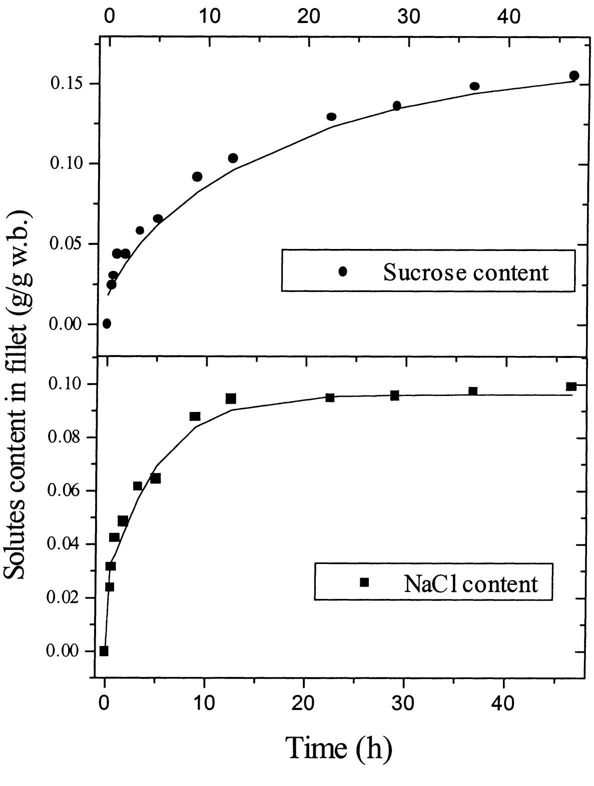

Fig. 5. Experimental and predicted (continuous lines) sucrose and NaCl contents in tilapia fillet.

ascorbic acids in potato spheres and lactic acid and sodium chloride during cheese brining, respectively. The calculated cross-diffusion coefficients revealed that diffusion rate of sodium chloride is independent of the presence of lactic acid in the system; however, the rate of diffusion of lactic acid is accelerated by the presence of sodium chloride. Lombardi and Zaritzki [25] concluded that the diffusion models described efficiently the ascorbic acid and/or citric acid uptake in pre-peeled potatoes dipped in acidified solutions.

The binary effective calculated were: for brine concentration of 35.14 g of NaCl/100 g of water, D_{11}, $0.91 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$ and for solution of 100 g of sucrose/100 g of water, D_{22}, $0.22 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$. The calculated effective diffusion coefficient for NaCl is analogous to those reported in literature by Del Valle and Nickerson [8], for wordfish, and Zugarramurdi and Lupin [9], for "anchovy", but higher than that reported by Wang et al. [19] for Atlantic salmon. The calculated one for sucrose was also higher than that obtained by Bohuon [26]. These differences could be explained by fish species diversity, temperature, muscle orientation, fat content and presence or absence of skin. Storey [27] reported that salt uptake was dependent on fat content, as the presence of fat structures forces salt to take a tortuous path which significantly reduces salt diffusivity [19].

The values of cross-term coefficients calculated by fitting Eq. (10) to the experimental data (ternary solutions, NaCl/sucrose, 35.14 g/100 g in 100 g of water) were: -0.12×10^{-9} and $0.06 \times 10^{-9}\,\mathrm{m^2\,s^{-1}}$ for D_{12} and D_{21}, respectively. The RMS were 0.51 and 0.88% for the first and second values, respectively. This behavior agreed with that observed by Gerla and Rubiolo [21], which studied the salt (1) and lactic acid (2) diffusion in cheese, finding that D_{12} was almost equal to zero and that D_{21} was of the same order but negative. These multicomponent coefficients testify that, for this trial, sucrose gradient affected salt diffusion (negative D_{12}), but salt total flux is mainly the consequence of its own gradient ($D_{11} \gg |D_{12}|$). This can also be observed in Fig. 3. Nevertheless, the effect of the salt gradient on sucrose diffusion is negligible ($D_{21} \sim 0$). So it can be assured that sucrose gradient is the only important driving force in this diffusion component (Fig. 4).

5. Conclusions

The fish fillet processing with ternary solutions showed that the influence of sucrose on fillet water activity is minimal, contrary to sodium chloride that presented great influence. The presence of sucrose in solution favored the moisture content reduction and influenced negatively in sodium chloride uptake in tilapia fillet. The mathematical model described very well the experimental values and the calculated cross-term diffusion coefficients described adequately the behavior of solutes when ternary solutions were used.

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