Influence of the process parameters on osmotic dehydration of mapara (*Hypophthalmus edentatus*) fillet

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Abstract The effects of temperature, time, pressure and brine concentration were assessed during osmotic dehydration (OD) of *H. edentatus* fillet. The response surface methodology (RSM) showed that using vacuum during the OD process did not cause a statistically significant change ($p > 0.05$) on the water loss (WL), solid gain (SG) and water activity (a_w). The other factors showed a statistically significant effect ($p \leq 0.05$) on at least one of these responses. Through RSM, the following condition could be defined for the OD process: brine concentration at 25 % NaCl, 25 °C, 120 min and atmospheric pressure. The Azuara model was shown to be effective in predicting the WL and SG kinetics during the OD process. The simplified solution of Fick's second law of diffusion for a slab geometry was used to calculate the average values of apparent diffusion coefficient (D_{eff}), namely $4.73 \times 10^{-10} \text{ m}^2/\text{s}$ for WL and $1.33 \times 10^{-9} \text{ m}^2/\text{s}$ for SG.

Keywords Vacuum · Kinetic · Modelling · Diffusivity · Preservation

Introduction

Mapara (*H. edentatus*) is widely distributed in South America. It figures among the ten highest productive species in the Brazilian continental extractive fishing (MPA 2011). This

species has a tasty musculature and can reach 3 kg with fillet yield above 60 % (Ribeiro et al. 2008).

Fish are considered an important source of nutrients since their tissues have proteins with high biological value, vitamin A, vitamin D and low cholesterol content (Luzia et al. 2003; Guinazi et al. 2006; Simões et al. 2007). On the other hand, fish muscle has high water activity (a_w) and pH close to neutral, besides being rich in unsaturated fatty acids that favour lipid oxidation and, consequently, product rancidity. Thus, fish require that appropriate handling and preservation processes be applied, in order to maintain the quality of the product until it reaches the consumer (Oliveira et al. 2008).

Osmotic dehydration is a unit operation that has been used as a treatment prior to food drying (Raoult-Wack 1994; Fernandes et al. 2006; Souza et al. 2007). Different conditions of this process have been applied to a large number of products, mainly those of vegetal origin (Serenio et al. 2001; Alves et al. 2005; Monnerat et al. 2010). Nevertheless, data on osmotic dehydration of fish are still scarce in the international scientific literature.

In general, osmotic dehydration is carried out at atmospheric pressure, however, several authors have assessed the effect of sub-atmospheric pressures (vacuum) as an alternative to increase the mass transfer rates and decrease the process temperature, which may guarantee better product quality (Barat et al. 2001; Mújica-Paz et al. 2002; Panadés et al. 2006; Ito et al. 2007; Occhino et al. 2011).

The importance of *H. edentatus* to the Amazon region and the lack of data in the scientific literature on the effect of reduced pressures (vacuum) on osmotic dehydration of fish motivated the current research, which studied the osmotic dehydration of *H. edentatus* fillet under different conditions of temperature, process time, NaCl concentration in the osmotic solution and work pressure. This research aimed to assess the influence of these process parameters on osmotic dehydration

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of *H. edentatus* fillet, establish process conditions able to provide the highest water loss and the lowest gain of solids and water activity to the product, as well as the osmotic dehydration kinetics model.

Material and methods

Sample

Thirty *H. edentatus* units were used (35.0 ± 1.7 cm long and 213.0 ± 29.7 g) obtained in the city of Cametá (Pará, Brazil) (latitude $02^{\circ}14'40''\text{S}$ and longitude $49^{\circ}29'45''\text{W}$) in July 2013. The fish were transported in cool boxes with ice to the Fish Products Laboratory of the Federal University of Pará (Belém, Pará, Brazil) (latitude $01^{\circ}27'21''\text{S}$ and longitude $48^{\circ}30'16''\text{W}$), where they were cleaned and manually filleted with stainless steel blades. Slab-shaped samples (50 mm length, 20 mm width and 5 mm thickness) were obtained from the fillet and a representative sample (1 kg of fillet) was reserved for the product's characterization. All samples were packaged under vacuum in nylon poly vacuum bags and stored at -18°C until use.

Fish characterization

The recommended methods of the Association of Official Analytical Chemists (AOAC 1997) were adopted in order to determine the moisture, ashes, total lipids, total proteins and salt (NaCl) contents of the fresh *H. edentatus* fillet and dehydrated product. The thiobarbituric acid (TBA) value was determined as a measure of the product's oxidative rancidity (Vyncke 1970; Osawa et al. 2005).

Osmotic dehydration of *H. edentatus* fillet

The study of *H. edentatus* fillet osmotic dehydration employed a 2^4 central composite design with 27 assays, being 16 linear assays at the levels -1 and $+1$, eight axial tests (one variable at levels $\pm\alpha$ and three at 0), and three assays in the central point (the four independent variables at level 0) (first five columns of Table 1). The effects of the independent variables temperature, time, pressure (vacuum) and NaCl concentration in the osmotic solution on the responses of water loss (WL), solid gain (SG) and water activity (a_w) (dependent variables) were assessed.

The osmotic dehydration assays were carried out in an purpose-built apparatus, composed of a jacketed system to guarantee temperature control and properly sealed to guarantee sub-atmospheric pressures (vacuum) were maintained. An ultra thermostatic bath (Q214M2, Quimis, Brazil) was used to cool down/warm up the system using a thermal fluid flowing through the

apparatus's jacket. The osmotic solution temperature was monitored using a thermocouple linked to a digital multimeter (Metex 506, Voltcraft, Germany). The process pressure was controlled using a gauge-equipped vacuum pump (131, Primatec, Brazil) connected to the system.

The osmotic dehydration assays were performed according to the following steps: The slab-shaped *H. edentatus* fillets were weighed in an analytical balance (AY220, Shimadzu, São Paulo, Brazil) and immersed in the osmotic solution prepared with distilled water and analytical-grade sodium chloride (NaCl) at the working concentration (5 to 25 %) and temperature (10 to 30°C). The system was immediately closed and the vacuum pump was turned on to verify the working pressure. A stirring table (225.21, Quimis, Brazil) was used to keep the set at constant 60 rpm agitation to guarantee homogeneous osmotic solution and temperature. The ratio between the sample's mass and the osmotic solution's volume was maintained at 1:80 (m/v) to guarantee that the osmotic solution's concentration remained constant throughout the process. After the contact time (30 to 150 min), defined by the experimental design, the system's vacuum condition was interrupted. The samples were then removed from the solution, washed superficially with 10 mL distilled water to remove the excess salt from the surface, dried in paper towels, and weighed in the analytical balance. The samples' moisture was determined before and after the osmotic dehydration process and a_w was determined after the process using a thermohygrometer (Aqualab 3TE; Decagon, Pullman, WA, USA).

The water loss (WL) and solid gain (SG) response were determined according to Eqs. (1) and (2) (Falade et al. 2007).

$$\text{WL}(\%) = \frac{(M_0 - m_0) - (M - m)}{M_0} \times 100 \quad (1)$$

$$\text{SG}(\%) = \frac{m - m_0}{M_0} \times 100 \quad (2)$$

where M_0 = initial mass of fish fillet slab (g), M = mass of fish fillet slab after time (t) of osmotic dehydration (g), m_0 = initial dry mass of fish fillet slab (g), m = dry mass of fish fillet slab (g) after time (t) of osmotic dehydration.

Osmotic dehydration process kinetics

In order to assess the *H. edentatus* fillet osmotic dehydration process kinetics, fresh fish fillet slab samples underwent the dehydration process under the conditions of temperature, pressure (vacuum) and NaCl concentration in the osmotic solution that led to the highest WL and lowest SG and a_w levels. The effect of the process's contact time on WL, SG and a_w were assessed. The assays were carried out in triplicate.

Table 1 Central composite design setting in original and coded forms of the independent variables temperature (T), time (t), pressure (P) and NaCl concentration (C), and experimental results for water loss (WL), solid gain (SG) and water activity (a_w)

Run order	T (°C)	t (min)	P (kPa)	C (%)	WL (%)	SG (%)	a_w
1	15(−1)	60(−1)	30(−1)	10(−1)	−5.65	1.88	0.97
2	15(−1)	60(−1)	30(−1)	20(+1)	2.50	2.45	0.94
3	15(−1)	60(−1)	78(+1)	10(−1)	−8.08	0.44	0.96
4	15(−1)	60(−1)	78(+1)	20(+1)	0.64	4.50	0.93
5	15(−1)	120(+1)	30(−1)	10(−1)	−9.36	2.91	0.95
6	15(−1)	120(+1)	30(−1)	20(+1)	1.87	6.03	0.92
7	15(−1)	120(+1)	78(+1)	10(−1)	−10.82	1.79	0.95
8	15(−1)	120(+1)	78(+1)	20(+1)	3.73	7.33	0.91
9	25(+1)	60(−1)	30(−1)	10(−1)	−10.06	0.20	0.97
10	25(+1)	60(−1)	30(−1)	20(+1)	4.98	6.23	0.92
11	25(+1)	60(−1)	78(+1)	10(−1)	−4.28	1.47	0.96
12	25(+1)	60(−1)	78(+1)	20(+1)	2.88	6.74	0.92
13	25(+1)	120(+1)	30(−1)	10(−1)	−9.35	2.51	0.96
14	25(+1)	120(+1)	30(−1)	20(+1)	8.35	8.85	0.89
15	25(+1)	120(+1)	78(+1)	10(−1)	−8.23	3.10	0.95
16	25(+1)	120(+1)	78(+1)	20(+1)	7.17	8.21	0.92
17	10(− α)	90(0)	54(0)	15(0)	−3.94	4.05	0.94
18	30(+ α)	90(0)	54(0)	15(0)	−2.71	5.48	0.93
19	20(0)	30(− α)	54(0)	15(0)	−0.98	2.89	0.96
20	20(0)	150(+ α)	54(0)	15(0)	1.18	7.14	0.93
21	20(0)	90(0)	7(− α)	15(0)	−3.99	6.08	0.93
22	20(0)	90(0)	101(+ α)	15(0)	−2.04	2.71	0.94
23	20(0)	90(0)	54(0)	5(− α)	−10.61	0.42	0.98
24	20(0)	90(0)	54(0)	25(+ α)	10.08	9.07	0.89
25	20(0)	90(0)	54(0)	15(0)	−2.95	4.71	0.94
26	20(0)	90(0)	54(0)	15(0)	−2.41	5.36	0.94
27	20(0)	90(0)	54(0)	15(0)	−1.90	4.25	0.94

Values between parenthesis are the coded forms of the variables

Osmotic dehydration process modelling

The two-parameter model proposed by Azuara et al. (1992) (Eq. (3)) was used in order to predict the osmotic dehydration process kinetics and to determine the final equilibrium point. Mathematical modelling estimated the values of parameters S , WL_∞ and SG_∞ for the process.

$$WS = \frac{S(WS_\infty)t}{1 + St} \quad (3)$$

where WS = fraction of water lost (WL) or solid gained (SG) by the product at time t , S = constant related to the rate of output of water (S_1) or input of solids (S_2) by the product, and WS_∞ = fraction of water lost (WL_∞) or solid gained (SG_∞) by the product at equilibrium.

Based on Fick's second law, Crank (1975) proposed an equation for one-dimensional diffusion in a slab-shaped sample in contact with an infinite amount of

solution. Its simplified form when t is small is presented in Eq. (4). This equation is only applicable in the early stages of the process, when diffusion is assumed to occur in a semi-infinite medium. In this stage, both the amount of water leaving and solids entering the product are directly proportional to the square root of time ($t^{1/2}$). The value of apparent diffusion coefficient (D_{eff}) calculated from Eq. (4) is the average D_{eff} for the range of concentrations during the diffusion process. The values of D_{eff} for WL and SG were calculated from the angular coefficients of the linear regions of the curves WL/WL_∞ and SG/SG_∞ versus $t^{1/2}$, respectively.

$$\frac{WS}{WS_\infty} = 2 \left(\frac{D_{eff}t}{\pi L^2} \right)^{1/2} \quad (4)$$

where WS = fraction of water lost (WL) or solid gained (SG) by the product at time (t), WS_∞ = fraction of water lost (WL_∞) or solid gained (SG_∞) by the product

at equilibrium, D_{eff} = apparent diffusion coefficient, and L = half-thickness of the slab.

Statistical treatment

Equation (4) was fitted to the osmotic dehydration data using linear regression in the software Microsoft Excel 2013 to estimate the apparent diffusion coefficient (D_{eff}) for WL and SG. Meanwhile, the parameters of Eq. (3) were estimated by nonlinear regression using the

Levenberg-Marquardt algorithm and a convergence criterion of 10^{-6} . The analysis of variance (ANOVA), lack-of-fit test, determination of the regression coefficients, and the generation of surface graphs were carried out for the responses of the experimental design. The software Statistica Kernel Release 7.1 was used for these procedures and for the nonlinear regression. A second-order polynomial model (Eq. (5)) was fitted to the responses of the experimental design (Khuri and Cornell 1996).

$$\text{WL, SG or } a_w = \beta_0 + \beta_1 T + \beta_{11} T^2 + \beta_2 t + \beta_{22} t^2 + \beta_3 P + \beta_{33} P^2 + \beta_4 C + \beta_{44} C^2 + \beta_{12} Tt + \beta_{13} T P + \beta_{14} T C + \beta_{23} t P + \beta_{24} t C + \beta_{34} P C \quad (5)$$

where WL, SG or a_w are the respective response variables, namely, water loss, solid gain and water activity; T , t , P and C represent the independent variables, namely, temperature, time, pressure and NaCl concentration, respectively; and the regression coefficients are β_0 (for the intercept); β_1 , β_2 , β_3 and β_4 (for the linear terms); β_{11} , β_{22} , β_{33} and β_{44} (for the quadratic terms); and β_{12} , β_{13} , β_{14} , β_{23} , β_{24} and β_{34} (for the interaction terms).

Results and discussion

Characterization of *H. edentatus* fillet

The *H. edentatus* fillet studied had the following proximate composition: 76.90 % (± 0.27) moisture, 15.57 % (± 0.64) protein, 5.38 % (± 0.09) lipids, 0.93 % (± 0.02) ash and 0.10 % (± 0.01) salt contents. Based on this composition, the *H. edentatus* used can be classified as a fatty fish (>5 % lipids) with a high protein content (>15 % protein) (Pigott and Tucker 1990). Ribeiro et al. (2008) and Costa et al. (2010) observed the following contents for this same fish species: 64.01–65.18 % moisture, 11.72–17.85 % protein, 17.85–21.21 % lipids and 0.75–0.91 % ash. The protein and ash contents of the fish studied match the values from those authors, however, the moisture content was higher due to the lower lipid content, which may be related to the fish's age and place and time of capture, which occurred in the peak Amazon summer. Luzia et al. (2003) reported 62 % lower lipid content in sardine (*Sardinella* spp.) muscle in fish captured in the summer when compared to those captured in the winter. The low TBA value (0.18 ± 0.01 mg malondialdehyde/kg fresh fish) observed in the fresh fish muscle showed that the product had excellent preservation regarding lipid oxidation (Al-Kahtani et al. 1996).

Osmotic dehydration process

The values of responses WL (−10.82–10.08 %), SG (0.20–9.07 %) and a_w (0.89–0.98) for the experimental domain of *H. edentatus* fillet osmotic dehydration (OD) process (columns two to five of Table 1) are presented in the last three columns of this same table. In the assays performed with osmotic solution concentration up to 15 % NaCl, the sample gained between 0.98 % and 10.82 % water, which is undesirable in the OD process.

ANOVA was used to assess the linear (L), quadratic (Q) and interaction effects of the factors temperature (T), time (t), pressure (P) and NaCl concentration (C) on the responses of WL, SG and a_w , as well as the significance of each effect (Table 2). The values of the coefficients of determination ($R^2=0.96$ for WL, $R^2=0.92$ for SG and $R^2=0.95$ for a_w) show good fits of the second-order polynomial model (Eq. (5)) and that most of the variabilities in responses were explained by the model. The fact that the statistical lack-of-fit test was non-significant ($p>0.05$) for all responses confirms the applicability of the statistical methodology used (Khuri and Cornell 1996).

According to the statistical analysis (Table 2 and Fig. 1), the linear effects of temperature (T) and NaCl concentration (C) were statistically significant for all responses (WL, SG and a_w). The linear effect of time (t) was significant for the responses of SG and a_w and the quadratic effects of t and of the interactions $C \times T$ and $C \times t$ were significant only for the response WL ($p \leq 0.05$). The positive sign of the significant effects on the responses WL and SG and the negative sign of the significant effects on the response a_w (Fig. 1) show that the increase in the respective factors led to higher WL and SG and lower a_w . The increase in WL and decrease in a_w are highly desirable in OD processes, while the increase in SG must be controlled. According to Fig. 1, NaCl concentration was the most important factor influencing the OD process in the experimental domain. Similar behaviours were observed by

Table 2 Analysis of variance of the parameter estimates obtained from the multiple linear regression and for the lack-of-fit test for temperature (T), time (t), pressure (P) and NaCl concentration (C)

Factor	Water loss (WL)				Solid gain (SG)				Water activity (a_w)			
	SS	DF	F	<i>p</i>	SS	DF	F	<i>p</i>	SS	DF	F	<i>p</i>
1 T (°C) (L)	15.18	1	54.63	0.018	6.89	1	22.26	0.042	1.5×10^{-4}	1	20.40	0.046
T (Q)	2.01	1	7.23	0.115	0.29	1	0.93	0.436	1.1×10^{-5}	1	1.52	0.342
2 t (min) (L)	0.93	1	3.35	0.208	26.67	1	86.16	0.011	1.3×10^{-3}	1	168.84	0.006
t (Q)	6.44	1	23.16	0.041	0.06	1	0.20	0.701	8.0×10^{-6}	1	1.11	0.402
3 P (kPa) (L)	0.55	1	1.97	0.296	0.74	1	2.38	0.263	1.0×10^{-6}	1	0.20	0.698
P (Q)	1.13	1	4.05	0.182	0.93	1	3.01	0.225	2.3×10^{-5}	1	3.12	0.219
4 C (%) (L)	808.81	1	2,910.01	<0.001	118.57	1	383.07	0.003	1.0×10^{-2}	1	1,388.55	0.001
C (Q)	4.49	1	16.14	0.057	0.31	1	1.02	0.420	3.0×10^{-6}	1	0.39	0.596
1L by 2L	4.44	1	15.99	0.057	0.04	1	0.12	0.767	4.2×10^{-5}	1	5.71	0.139
1L by 3L	3.54	1	12.74	0.070	0.06	1	0.18	0.712	1.0×10^{-4}	1	13.99	0.065
1L by 4L	10.02	1	36.04	0.027	5.57	1	18.01	0.051	1.2×10^{-4}	1	16.78	0.055
2L by 3L	0.06	1	0.21	0.692	0.32	1	1.04	0.416	1.6×10^{-5}	1	2.21	0.275
2L by 4L	24.53	1	88.24	0.011	1.09	1	3.51	0.202	6.2×10^{-5}	1	8.41	0.101
3L by 4L	2.44	1	8.77	0.098	0.96	1	3.09	0.221	3.9×10^{-5}	1	5.25	0.149
Lack-of-fit	33.52	10	12.06	0.079	13.42	10	4.33	0.202	6.0×10^{-4}	10	8.08	0.115
Pure error	0.56	2			0.62	2			1.5×10^{-5}	2		
Total SS	924.48	26			176.00	26			1.3×10^{-2}	26		

SS Sum of squares, DF Degree of freedom, R^2_{WL} 0.96, R^2_{SG} 0.92, R^2_{aw} 0.95, L Linear, Q Quadratic statistical significance ($p \leq 0.05$)

Medina-Vivanco et al. (2002) and Ribeiro et al. (2008) during OD of tilapia (*Oreochromis niloticus*) and mapara (*H. edentatus*) fillets.

The statistical analysis showed that the linear and quadratic effects and the interaction effect for the pressure factor were statistically non-significant ($p > 0.05$) (Table 2 and Fig. 1) for all responses assessed (WL, SG and a_w) in the range between very low pressure (7 kPa) and the local atmospheric pressure (101 kPa). Schmidt et al. (2008) observed an increase in SG in chicken breast fillet (animal tissue) subjected to OD at vacuum; however, the difference was only significant after 4 h of contact with the osmotic solution (15–20 % NaCl).

Equations (6), (7) and (8), obtained after multiple linear regressions for the response variables WL, SG and a_w , respectively, were carried out to generate the overlapping of

response surface and contour plots presented in Fig. 2. According to this figure, the highest values of WL (10–15 %) and SG (8–10 %) (Fig. 2a and c) and the lowest values of a_w (0.88–0.90) (Fig. 2b and d) were observed in the lighter area, from which the following process conditions were defined for *H. edentatus* osmotically dehydrated fillet, within the experimental domain: osmotic solution concentration of 25 % NaCl, temperature of 25 °C, time of 120 min and atmospheric pressure. According to Fig. 2, using an osmotic solution with 25 % salt content enables using lower temperature and shorter OD times, which is advantageous to control the fish's degradative conditions, such as lipid oxidation, as well as lower process costs. Hence, the temperature of 25 °C was established since it also restricts the need for using a heating system, besides a time of 120 min.

$$WL = 8.01 - 0.356T - 0.012T^2 - 0.302t + 6 \times 10^{-4}t^2 + 0.013P - 4 \times 10^{-4}P^2 - 0.587C + 0.018C^2 + 0.003Tt + 0.004TP + 0.032TC + 1 \times 10^{-4}tP + 0.008tC - 0.003PC \quad (6)$$

$$SG = -1.24 - 0.059T - 0.005T^2 + 0.037t - 1 \times 10^{-4}t^2 + 0.010P - 4 \times 10^{-4}P^2 - 0.149C - 0.005C^2 - 3 \times 10^{-4}Tt + 5 \times 10^{-4}TP + 0.024TC - 2 \times 10^{-4}tP + 0.002tC + 0.002PC \quad (7)$$

$$a_w = 1.03 + 2 \times 10^{-4}T - 3 \times 10^{-5}T^2 - 5 \times 10^{-4}t + 7 \times 10^{-7}t^2 - 6 \times 10^{-4}P - 2 \times 10^{-6}P^2 - 0.001C - 1 \times 10^{-5}C^2 + 1 \times 10^{-5}Tt + 2 \times 10^{-5}TP - 1 \times 10^{-4}TC + 1 \times 10^{-6}tP - 1 \times 10^{-5}tC + 1 \times 10^{-5}PC \quad (8)$$

Fig. 1 Pareto charts of the standardised effects for **a** water loss (WL), **b** solid gain (SG) and **c** water activity (a_w). T: temperature; t: time; P: pressure; C: NaCl concentration; L: linear effect; Q: quadratic effect

In order to validate the models, experimental assays were carried out under the conditions indicated as optimal for the OD process (25 % salt content, 25 °C, 120 min, and atmospheric pressure). The results presented in Table 3 show that the values predicted for WL (Eq. (6)), SG (Eq. (7)) and a_w (Eq. (8)) were approximately the same as the experimental values, which confirms the validity and adequacy of the predictive models, with a 95 % confidence interval.

The dehydrated product obtained under the optimized condition had the following proximate composition: 62.06 % (± 0.68) moisture, 17.0 % (± 0.24) protein, 6.37 % (± 0.38) lipids, 12.80 % (± 0.44) ash and 12.21 % (± 0.05) salt contents. The product had a_w of 0.87 (± 0.01) and TBA value of 0.66 mg malondialdehyde/kg (± 0.02). Therefore, the dehydrated product did not reach a_w that could guarantee microbiological stability ($a_w < 0.6$) (Salwin 1963; Rockland and Nishi 1980). Thus, to ensure its preservation, the product must be stored under refrigeration or undergo another processing step such as smoking or drying.

The increase in TBA value from 0.18 mg malondialdehyde/kg in fresh fillet to 0.66 mg malondialdehyde/kg in the dehydrated product suggests a slight increase in lipid oxidation reactions, which can be attributed to the salt incorporated

into the product (Vyncke 1970; Osawa et al. 2005). However, according to Connell (1995), only TBA values between 1 and 2 mg malondialdehyde/kg fresh fish are usually regarded as the limit of acceptability due to the formation of unacceptable odour and flavour. Additionally, Tsironi and Taoukis (2010) observed sensory rejection of gilthead seabream (*Sparus aurata*) fillets at an average TBA value of 0.9 mg malondialdehyde/kg fresh fish. Chaijan (2011) observed TBA values of 3 mg malondialdehyde/kg fresh tilapia (*Oreochromis niloticus*) muscle, which was about twice as much in the product subjected to wet salting in brine with 25 % NaCl at 25 °C for 180 min. Similar behaviour was observed by Espe et al. (2001) for salmon fillets subjected to wet salting followed by cold-smoking.

Osmotic dehydration process kinetics

The evolution of WL, SG and a_w over time during osmotic dehydration of *H. edentatus* fillet under the optimized condition is presented in Fig. 3. In the first 120 min of the process, WL markedly increased until it reached 15.8 %, while in the last 120 min of the process, this increase was by only 1.90 %. The sharp osmotic pressure gradient between the product and

Fig. 2 Overlapping of response surface and contour plots at 90 min and 52.7 kPa for **a** PA (areas) and SG (lines) and **b** PA (areas) and a_w (lines), and at 20 °C and 52.7 kPa for **c** PA (areas) and SG (lines) and **d** PA (areas) and a_w (lines)

the osmotic solution in the beginning of OD leads to high WL levels, but, as the process advances, this gradient gradually levels off, limiting WL until equilibrium is reached (Rastogi and Raghavarao 1997). For SG, the most marked increase was seen in the first 60 min of the process, when it reached 8.0 %, and the maximum value of 11.7 % was observed at 180 min of contact. In this case, the behaviour observed is attributed to the solute's concentration gradient between the osmotic solution and the product's aqueous phase,

Table 3 Comparison of predicted and experimental values for the responses water loss, solid gain and water activity at the optimized condition

Response	Observed value ^a	Predicted value
Water loss (WL, %)	15.84±0.56	16.09
Solid gain (SG, %)	10.83±0.28	10.65
Water activity	0.87±0.01	0.89

^a Mean±SD of triplicate determinations of experimental values for osmotic dehydration carried out at 25 % salt content, 25 °C, 120 min and atmospheric pressure

which is high at the beginning of the process and gradually decreases until equilibrium is reached. The product's a_w significantly decreased until 180 min of the process (from 0.99 to 0.85), when it stabilized at 0.85.

Fig. 3 Evolution of water loss ($\circ$, WL), solid gain ($\square$, SG) and water activity ($\diamond$, a_w) over time during the osmotic dehydration process under the optimized condition (25 % salt content, 25 °C and atmospheric pressure), and the Azuara model (—) fitted to the WL and SG data

A similar behaviour was observed by Medina-Vivanco et al. (2002) during OD of tilapia fillets.

Osmotic dehydration modelling

The values of the parameters of the model proposed by Azuara et al. (1992), and of the statistical parameters correlation coefficient (R^2) and root-mean-square error (RMSE), used to assess the fit of Eq. (3) to the experimental data of the WL and SG kinetics under the optimized condition are presented in Table 4. The values of $R^2 > 0.96$ and $RMSE < 1.90$ show that the Azuara model was able to predict with good precision the WL and SG kinetics in OD of *H. edentatus* fillet under the conditions assessed, as seen in Fig. 3.

The parameter S is a constant related to the rate of output of water or input of solids by the product during OD. Thus, the higher value of S observed for SG suggests the salt diffusion from the solution into the product tends to reach equilibrium in a shorter time than the diffusion of water from the product into the solution (WL) (Azuara et al. 1992), which is shown in Fig. 3. In turn, the parameter WS_{∞} estimates the fraction of water lost (WL_{∞}) or fraction of salt gained (SG_{∞}) by the product at equilibrium. Hence, the highest value predicted for WL_{∞} (31.38 %) suggests that, at the equilibrium condition of *H. edentatus* fillet OD at the optimized condition, the amount of water that leaves the product will be much higher than the amount of salt impregnated into the product ($SG_{\infty} = 14.98$ %).

Although the time of 120 min was defined as the optimized condition for the OD process of *H. edentatus* fillet through the response surface methodology, the OD kinetics shows that using process times above 120 min may favour $WL > 16$ % while maintaining SG at approximately 11 % (Fig. 3). This observation was possible since longer contact times (up to 240 min) than that defined as the upper limit in the experimental design (150 min) were used to obtain the OD kinetics. This behaviour suggests that OD of *H. edentatus* fillet in brine with 25 % NaCl content, at 25 °C and atmospheric pressure, could be carried out with contact times above 120 min; however, lipid oxidation must be observed.

The average values of apparent diffusion coefficient (D_{eff}) estimated by Eq. (4) were $4.73 \times 10^{-10} \text{ m}^2/\text{s}$ ($R^2 = 0.98$) for WL and $1.33 \times 10^{-9} \text{ m}^2/\text{s}$ ($R^2 = 0.99$) for SG during OD of *H. edentatus* fillet. These values confirm that the rate of input of solids into the products was higher than the rate of output of water during the OD process. Corzo and Bracho (2007a, b) reported approximately the same D_{eff} values for WL ($1.82 \times 10^{-10} \text{ m}^2/\text{s}$) when vacuum pulse was used during OD of sardine sheets in brine with 24 % NaCl at 30 °C. Boudhrioua et al. (2009) reported higher D_{eff} values for WL (3.00×10^{-9} to $8.40 \times 10^{-9} \text{ m}^2/\text{s}$), however, approximately the same value for SG ($3.80 \times 10^{-9} \text{ m}^2/\text{s}$) during salting of sardine fillets in brine with 26.5 % NaCl at 20 °C. On the other hand, lower D_{eff} values for SG were observed by Gallart-Jornet et al. (2007) during salting of salmon ($5.19 \times 10^{-10} \text{ m}^2/\text{s}$) and cod ($1.51 \times 10^{-10} \text{ m}^2/\text{s}$) muscle in brine with 25 % NaCl at 4 °C.

Conclusions

In this study, the response surface methodology (RSM) was used to establish an optimized condition for osmotic dehydration (OD) process of *H. edentatus* fillet. In addition, the process kinetics under this condition was assessed and modelled. The methodology showed that the NaCl concentration in the brine was the factor with the greatest impact on the increase of the product's water loss (WL) and solid gain (SG) and on the decrease in water activity (a_w), besides showing that the use of vacuum during the process did not significantly impact these responses. The RSM indicated the following optimized condition for the OD process: brine concentration at 25 % NaCl, temperature at 25 °C, time of 120 min and atmospheric pressure, in the experimental domain. The Azuara model predicted with good precision the WL and SG kinetics during the OD process. The average values of apparent diffusion coefficient (D_{eff}) were $4.73 \times 10^{-10} \text{ m}^2/\text{s}$ for WL and $1.33 \times 10^{-9} \text{ m}^2/\text{s}$ for SG.

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Table 4 Parameters of mathematical modelling of osmotic dehydration of *H. edentatus* fillet

Process	Parameter			
	WS_{∞}	S	R^2	RMSE
Water loss (WL)	31.38	0.0063	0.96	1.90
Solid gain (SG)	14.98	0.0171	0.97	0.43

WS_{∞} and S parameters of the Azuara model (Eq. (3)), R^2 correlation coefficient, RMSE root-mean-square error

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