Combined osmotic dehydration and drying process of pirarucu (*Arapaima gigas*) fillets

Mayara Galvão Martins¹ · Rosinelson da Silva Pena^{1,2}

Revised: 13 June 2017 / Accepted: 7 July 2017 / Published online: 8 August 2017
© Association of Food Scientists & Technologists (India) 2017

Abstract The osmotic dehydration (OD) and complementary drying of pirarucu (*Arapaima gigas*) fillets were studied. Pieces of the dorsal portion of pirarucu (60 mm × 20 mm × 10 mm) underwent OD in a binary solution (NaCl–water) with the application of vacuum pulse following a central rotatable composite design. The effect of the following process variables was assessed: temperature (20–40 °C), osmotic solution concentration (15–25% NaCl), and vacuum pulse pressure (7–101 kPa) on water loss (WL), solid gain (SG), and water activity (a_w). OD kinetics was obtained and the Peleg model was fitted to WL and SG data. The osmotically dehydrated pirarucu was dried (40–70 °C) in a fixed-bed dryer and mathematical models were fitted to the drying data. The optimal operational condition for the OD process was 35 °C, solution with 25% NaCl, and atmospheric pressure, which yielded WL of $14.87 \pm 1.46\%$, SG of $8.56 \pm 0.45\%$, and a_w of 0.87 ± 0.02 . The Peleg model efficiently predicted the WL and SG kinetics. The increase in the water loss in drying was more evident at low temperatures (40–50 °C) with effective diffusivity ranging from 10.85×10^{-9} to 12.30×10^{-9} m²/s. The Midilli and Page models efficiently predicted the drying kinetics.

Keywords *Arapaima gigas* · Vacuum · Desirability · Convective drying · Modeling

Introduction

Pirarucu (*Arapaima gigas*) is one of the five most widely farmed and marketed species in the Amazon and represents a very economically and ecologically important resource for being one of the largest species of the fresh-water ichthyofauna on the planet, reaching up to 3 m in length and weighing up to 200 kg (Castello 2008; Torres et al. 2008; Watson et al. 2013).

The increase in demand for pirarucu farming in aquaculture production systems has highlighted the economic importance of this species in recent years mainly because of its nutritional quality. However, consumption of this beneficial product throughout the year is difficult because of the many changes that occur in the fish's enzymatic and microbial activity, which result in fish spoilage (Baslar et al. 2015).

In order to preserve easily spoiled products, such as fish, different methods have been employed and/or enhanced. The osmotic dehydration (OD) together with hot air convective drying present a promising combination of hurdle technologies leading to more stable fish and fish products (Corzo et al. 2006a, b; 2007).

Convective air drying is the most common method of food preservation and consists in removing part of the moisture contained inside the product through evaporation, usually caused by forced convection of heated air, which simultaneously promotes heat and mass transfer (Rastogi et al. 2002; Chen et al. 2016).

OD is the most often reported technique used in combination with convective air drying. The OD process is

✉ Rosinelson da Silva Pena
rspena@ufpa.br

¹ Graduate Program in Food Science and Technology, Technology Institute, Federal University of Pará (UFPA), Belém, PA 66075-110, Brazil

² Faculty of Food Engineering, Institute of Technology, Federal University of Pará (UFPA), Rua Augusto Corrêa 01, Belém, PA 66075-110, Brazil

employed to lower the product's initial moisture and may also change the product's tissue structure in a way that speeds up air drying (Fernandes et al. 2008). This process consists in partially removing the water of a food product based on its immersion in hypertonic solutions of one or more solutes, which basically initiates two simultaneous and opposed flows: Water leaving the product to the solution and migration of solutes from the solution to the product. The driving force for water removal is the difference in osmotic pressure between the food and the solution, while the food's complex cellular structure acts as a semi-permeable membrane (Fito 1994; Medina-Vivanco et al. 2002; Corzo and Gómez 2004; Corzo and Bracho 2007b).

The use of vacuum in the OD process is still little explored for fish, unlike what occurs for fruits and vegetables. Osmotic dehydration with vacuum pulse (PVOD) occurs under sub-atmospheric pressure conditions for a given period of followed by another period under atmospheric pressure. Overall, this process aims to maximize water loss and, at the same time, minimize the gain of solids in the product (Corzo et al. 2007; Corzo and Bracho 2007a).

The combination of OD with drying has been indicated as an economic and safe alternative to preserve food products. This combination allows obtaining dehydrated products with better physical, chemical, nutritional, and sensory characteristics compared with products subjected to direct drying (Corrêa et al. 2011; Fante et al. 2011).

Despite the high nutritional and commercial potential, pirarucu muscle is considered a highly perishable resource, which demands the use of techniques for increasing its preservation. In this context, this research presents the first report, in which process conditions for the pirarucu muscle were determined, as a contribution for preservation of the fish muscle. Thus, the main objective of this research was to assess the influence of the osmotic dehydration process parameters of the pirarucu fillets and the effect of the temperature on the complementary drying process of the dehydrated product.

Materials and methods

Raw material

A pirarucu specimen was purchased in an aquaculture farm located in São João de Pirabas (Pará, Brazil) (00°46'29"S and 47°10'38"W). The fish was transported in an isothermic container with ice to the Federal University of Pará (Belém, Pará, Brazil) (01°27'21"S and 48°30'16"W), where it was cleaned and manually filleted with stainless steel blades. The dorsal portion was used in the study for being the most representative fraction of pirarucu muscle.

It was used to obtain the slab-shaped samples (60 mm length, 20 mm width, and 10 mm thickness).

Osmotic dehydration experiments

A central composite design (CCD) was applied to study the effects of the independent variables temperature (T), NaCl concentration in the osmotic solution (C), and vacuum pulse pressure (P) on the responses of water loss (WL), solid gain (SG), and water activity (a_w) (dependent variables). Osmotic dehydration (OD) process employed a 2^3 CCD with 17 assays, being eight linear assays at the levels -1 and $+1$, six axial tests (one variable at levels $\pm\alpha$ and two at 0), and three assays in the central point (the three independent variables at level 0) (first four columns of Table 1).

The OD assays were carried out in an apparatus composed of a jacketed system to guarantee temperature control and properly sealed to guarantee sub-atmospheric pressures (vacuum pulse) were maintained. A gauge-equipped vacuum pump was coupled to the system to ensure pressure control. Process temperature was controlled using an ultra thermostatic bath (Q214M2, Quimis, Brazil) by circulating a thermal fluid in the jacketed system, while the temperature of the osmotic solution was monitored by a thermocouple connected to a digital multimeter (Metex 506, Voltcraft, Germany). The jacketed system was placed on a stirring table (225.21, Quimis, Brazil) to ensure constant stirring at 70 rpm, enough to promote the circulation of the solution and remove the surface layer around the sample. The osmotic solutions were prepared by dissolving commercial sodium chloride in distilled water according to the CCD.

The slab-shaped samples were weighed in an analytical balance (AY220, Shimadzu, Brazil) and immersed in the osmotic solution at working temperature and concentration. The system was immediately closed and subjected to the pressure condition (vacuum pulse) defined by the experimental design. Each treatment was performed using a sample mass:osmotic solution volume ratio of 1:20 (m/v) to guarantee that the osmotic solution's concentration remained constant throughout the process.

After 30 min of contact time, the system's vacuum condition was interrupted and, by the end of the total process time (90 min), the samples were removed from the osmotic solution, superficially rinsed with 20 mL of water and dried in absorbent paper to remove excess water and salt from the surface, and finally weighed in an analytical balance. Sample moisture was determined before and after the OD process (AOAC 1997), when a_w was also determined using a thermohygrometer (Aqualab 3TE, Decagon, USA). The WL and SG responses were determined according to Eq. (1) and (2), respectively.

Table 1 Central composite design in original and coded forms of the independent variables temperature (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)	P (kPa)	C (% NaCl)	WL (%)	SG (%)	a_w
1	24 (−1)	26 (−1)	17 (−1)	1.21	5.05	0.93
2	24 (−1)	26 (−1)	23 (+1)	7.18	8.41	0.91
3	24 (−1)	82 (+1)	17 (−1)	0.97	5.30	0.92
4	24 (−1)	82 (+1)	23 (+1)	8.63	8.58	0.90
5	36 (1)	26 (−1)	17 (−1)	3.76	5.80	0.94
6	36 (1)	26 (−1)	23 (+1)	11.39	9.87	0.90
7	36 (1)	82 (+1)	17 (−1)	2.49	7.41	0.92
8	36 (1)	82 (+1)	23 (+1)	11.38	8.03	0.88
9	20 (−1.68)	54 (0)	20 (0)	3.93	7.48	0.91
10	40 (+1.68)	54 (0)	20 (0)	6.73	7.21	0.92
11	30 (0)	7 (−1.68)	20 (0)	6.78	7.11	0.92
12	30 (0)	101 (+1.68)	20 (0)	3.79	5.50	0.92
13	30 (0)	54 (0)	15 (−1.68)	−0.28	5.27	0.94
14	30 (0)	54 (0)	25 (+1.68)	11.03	7.11	0.88
15	30 (0)	54 (0)	20 (0)	4.75	7.40	0.92
16	30 (0)	54 (0)	20 (0)	4.18	7.54	0.92
17	30 (0)	54 (0)	20 (0)	4.04	7.02	0.93

Values between parentheses are the coded forms of the variables

$$WL(\%) = \frac{WM_o - WM_t}{M_o} \times 100 \quad (1)$$

$$SG(\%) = \frac{SM_t - SM_o}{M_o} \times 100 \quad (2)$$

where WM_o = initial water mass in the product (g), WM_t = water mass in the product at a time (t) of OD (g), M_o = initial mass of fish fillet slab (g), SM_o = initial dry mass of fish fillet slab (g), SM_t = dry mass of fish fillet slab (g) after time (t) of OD (g).

A second-order polynomial model (Eq. 3) was fitted to the responses of the experimental design to define the effects of the independent variables using the software Statistica (version 7.1, StatSoft Inc., USA). The analysis of variance (ANOVA), lack-of-fit test, and determination of the regression coefficients were carried out for the responses of the experimental design.

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_{ij} \quad (3)$$

where Y represents the response variables (WL, SG, and a_w); β_0 (for the intercept), β_i (for the linear terms), β_{ii} (for the quadratic terms), and β_{ij} (for the interaction terms) are the regression coefficients; x_i (for the linear terms), x_i^2 (for the quadratic terms), and x_{ij} (for the interaction terms) indicate the effect of the independent variables (T, C, and P).

Optimization of the osmotic dehydration process

Based on the results obtained by the CCD and using the desirability function proposed by Derringer and Suich (1980), the set of optimized conditions for the OD process

was obtained with the goal was maximizing WL and minimizing SG and a_w . The optimization of multiple responses consists in converting each response into an individual desirability function ($d(x_i)$), in the range of $0 \leq d(x_i) \leq 1$, where the value 0 is attributed to an undesirable response and the value 1, to a desirable response. This point is the optimized condition and is obtained by combining the individual desirabilities.

Osmotic dehydration process kinetics

In order to assess osmotic dehydration process kinetics, slab-shaped samples underwent the dehydration process under the conditions of temperature, pressure, and NaCl concentration in the osmotic solution that led to the highest WL and lowest SG and a_w levels (optimal condition). The effect of the contact time (20, 40, 60, 90, 120, 240, and 360 min) on WL, SG, and a_w were determined to assess OD process kinetics. The assays were carried out according to “Osmotic dehydration experiments” section.

Prediction of apparent diffusivity

Based on Fick’s second law, Crank (1975) proposed an equation for unidirectional diffusion in a sample with flat slab geometry in contact with an infinite amount of solution, used to describe the OD kinetics. The simplified form of this model (Eq. 4) for short contact times (t) is applied to the initial phase of the process, when diffusion is assumed to occur in a semi-infinite medium. The value of the apparent diffusivity coefficient (D), calculated from Eq. (4), is the mean of the values of D for the solution’s

concentration range during the diffusion process. The values of D for WL and SG were calculated from the angular coefficients of the linear regions of the curves WL_t/WL_∞ and SG_t/SG_∞ versus $t^{1/2}$, respectively.

$$\frac{WS_t}{WS_\infty} = 2 \left(\frac{Dt}{\pi L^2} \right)^{1/2} \quad (4)$$

where WS_t = amount of WL or of SG the product at a given time (t), WL_∞ = amount of WL_∞ or of SG_∞ the product after an infinite time (equilibrium), D = apparent diffusivity (m^2/s), L = half-thickness of the sample (m).

Osmotic dehydration process modeling

The two-parameter model proposed by Peleg (1988) (Eq. 5) was used to predict the OD process kinetics. The model's parameters for WL and SG were estimated from the linear coefficient (k_1) and from the slope of the curves t/WL versus t and t/SG versus t (k_2) by linear regression. The model's fit to the experimental data was confirmed by the coefficient of determination (R^2) and by the root mean square error (RMSE) (Eq. 6). For a condition of $t \rightarrow \infty$, the values of water loss (WL^∞) and solid gain (SG^∞) at equilibrium were estimated from the $1/k_2$ ratio for the respective processes.

$$X_w = X_{w0} \pm \frac{t}{k_1 + k_2 t} \quad (5)$$

where X_w = product moisture at a time t (h) of OD (g/g dry base-db), X_{w0} = product's initial moisture (g/g db), k_1 = Peleg rate constant (h/(g/g db)), k_2 = Peleg capacity constant ((g/g db) $^{-1}$). “ $\pm$ ” was assumed “ $+$ ” for SG and “ $-$ ” for WL.

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (Y_{exp,i} - Y_{pre,i})^2 \right]^{1/2} \quad (6)$$

where Y_{pre} = predicted WL or SG value, Y_{exp} = experimental WL or SG value, N = number of experimental measurements.

Drying process kinetics

Pirarucu fillets osmotically dehydrated under the optimized condition were dried in a fixed-bed dryer with airflow velocity of 1.5 m/s at 40, 50, 60, and 70 °C, to assess drying kinetics. Six sample pieces (60 mm $\times$ 20 mm $\times$ 10 mm) were placed side by side on a wire tray to allow the perpendicular airflow (contact area = 1200 mm 2) to simulate the thin-layer drying process. For monitoring purposes, the samples were weighed every 5 min during the first 30 min of drying, every 10 min in the following 60 min, and every 20 min from that point onwards until the sample's mass

variation was below 1% (equilibrium condition). The products were ground in a knife mill (A11 basic, IKA, Germany), stored in vacuum-sealed packaging (5 °C). Sample moisture was determined before and after drying. The drying curves were built from the correlation between the moisture ratio (MR) (given by the quotient of the difference between moisture at a time t and moisture at equilibrium and the difference between initial moisture and moisture at equilibrium) and the process time.

Prediction of effective diffusivity

In order to model the drying curves, it is assumed that the main mechanism during the downward rate period is moisture diffusion. When moisture diffusion controls the drying rate during a downward rate period, the diffusion equation, represented by Fick's second law of diffusion at non-steady state, can be used with Cartesian coordinates and in the non-dimensional form (Eq. 7).

$$\frac{\partial MR}{\partial t} = \frac{\partial}{\partial y} \left(D_{eff} \frac{\partial MR}{\partial y} \right) \quad (7)$$

where D_{eff} = effective diffusivity coefficient, t = time, y = rectangular coordinate, MR = moisture ratio (non-dimensional).

The analytical solution of Eq. (7) is given by Eq. (8), assuming uniform initial moisture, constant effective diffusivity, and sample with flat slab geometry (Crank 1975). For a sufficiently long drying time, Fick's second law of diffusion truncated in the first term (Eq. 9) provides a good estimate of the solution. This way, the value of D_{eff} was calculated from the slope of the curve $\ln(MR)$ versus t by linear regression.

$$MR = \frac{8}{\pi^2} \sum_{i=0}^{\infty} \frac{1}{(2i+1)^2} \exp \left(\frac{-(2i+1)^2 \pi^2 D_{eff} t}{4L^2} \right) \quad (8)$$

$$MR = \frac{8}{\pi^2} \exp \left(\frac{-\pi^2 D_{eff} t}{4L^2} \right) \quad (9)$$

where MR = moisture ratio (non-dimensional), D_{eff} = effective diffusivity coefficient (m^2/s), L = half-thickness of the slab (m), t = process time (s).

Mathematical modeling of the drying curves

The Newton (Eq. 10), Page (Eq. 11), Modified Page (Eq. 12), Henderson and Pabis (Eq. 13), Two-term exponential (Eq. 14), Logarithmic (Eq. 15), Diffusion approximation (Eq. 16), Verma (Eq. 17), Midilli (Eq. 18), and Two-term (Eq. 19) models, classically used to describe the kinetics of thin-layer drying processes (Akpınar et al. 2003), were fitted by nonlinear regression to the experimental drying data using the software Statistica (version

7.1, StatSoft Inc., USA). The Levenberg–Marquardt algorithm was used with a convergence criterion of 10^{-6} .

$$MR = e^{-k \cdot t} \quad (10)$$

$$MR = e^{-k \cdot t^n} \quad (11)$$

$$MR = e^{-(k \cdot t)^n} \quad (12)$$

$$MR = a \cdot e^{-k \cdot t} \quad (13)$$

$$MR = a \cdot e^{-k \cdot t} + (1 - a) \cdot e^{-k \cdot a \cdot t} \quad (14)$$

$$MR = a \cdot e^{-k \cdot t} + c \quad (15)$$

$$MR = a \cdot e^{-k \cdot t} + (1 - a) \cdot e^{-k \cdot b \cdot t} \quad (16)$$

$$MR = a \cdot e^{-k \cdot t} + (1 - a) \cdot e^{-g \cdot t} \quad (17)$$

$$MR = b \cdot t + a \cdot e^{-k \cdot t^n} \quad (18)$$

$$MR = a \cdot e^{-k_0 \cdot t} + b \cdot e^{-k_1 \cdot t} \quad (19)$$

where MR = moisture ratio (non-dimensional), t = process time (s), and a , b , c , g , k , n , k_0 and k_1 are constant of the models.

The values of R^2 , RMSE (Eq. 6), and reduced Chi squared (χ^2) (Eq. 20) were used to determine the model that best described the drying curves.

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{\text{exp},i} - MR_{\text{pre},i})^2}{N - n} \quad (20)$$

where $MR_{\text{exp},i}$ and $MR_{\text{pre},i}$ are the MR determined from the experimental data and predicted by the fitted models, respectively, N = number of experimental measurements, n = number of parameters in the model.

Results and discussion

Osmotic dehydration process

Pirarucu fillets ($77.24 \pm 0.51\%$ moisture content and $0.996 \pm 0.01 a_w$) underwent the OD process and the values of the responses WL (-0.28 to 11.39%), SG (5.05 – 9.87%), and a_w (0.88 – 0.94) for the experimental domain employed are presented in the final three columns of Table 1. The maximum WL and minimum a_w were observed for the experiments carried out at higher temperatures and more concentrated osmotic solutions, which may be attributed to the increase in osmotic gradient between the product and the solution and to the increase in cell membrane permeability, as well as to the reduction in viscosity of the osmotic solution, which leads to lower external resistance against mass transfer (Uddin et al. 2004).

In the experiment carried out with the lowest osmotic concentration (15% NaCl), a swelling effect of the sample was observed (increase in volume due to water gain,

WL < 0), which is undesirable in OD processes. According to Oozumi et al. (2003), the swelling effect observed is attributed to the solubilization of myofibrillar proteins, which is favored when osmotic solution with low NaCl concentration are employed in the OD process.

The results of the analysis of variance (Table 2) were used to assess the linear (L), quadratic (Q), and interaction effects of the factors temperature (T), NaCl concentration in the osmotic solution (C), and vacuum pulse pressure (P) on the WL, SG, and a_w responses, as well as the significance ($p \leq 0.05$) of each effect. The coefficient of determination values ($R^2 = 0.98$ for WL and $R^2 = 0.90$ for a_w) show good fits of the second-order polynomial model to WL (Eq. 21) and a_w (Eq. 22). This way, besides being significant, the models may be used for predictive purposes in the experimental domain studied. The fact that the statistical lack-of-fit test was non-significant ($p > 0.05$) for these responses (Table 2) confirms the applicability of the statistical methodology used. On the other hand, the R^2 value (73%) and the statistical lack-of-fit test significant ($p \leq 0.05$) to the SG response indicates that the polynomial model (Eq. 23) cannot be used with 95% confidence for predictive purposes, however, it can be used with good significance to estimate the SG trend (Khuri and Cornell 1996).

$$\begin{aligned} \text{WL} = & 24.14 - 0.899T + 0.013T^2 - 1.81C \\ & + 0.054C^2 - 0.107P \\ & + 5.6 \times 10^{-4}P^2 + 0.020TC - 1.9 \times 10^{-3}TP \\ & + 4.4 \times 10^{-3}CP \end{aligned} \quad (21)$$

$$\begin{aligned} a_w = & 0.496 + 0.012T - 8.6 \times 10^{-5}T^2 + 0.029C \\ & - 6.4 \times 10^{-4}C^2 - 6.1 \times 10^{-4}P \\ & - 2.0 \times 10^{-6}P^2 - 3.0 \times 10^{-4}TC - 1.3 \times 10^{-5}TP \\ & - 4.0 \times 10^{-6}CP \end{aligned} \quad (22)$$

$$\begin{aligned} \text{SG} = & -22.39 + 0.049T + 4.9 \times 10^{-3}T^2 + 2.12C \\ & - 0.027C^2 + 0.140P \\ & + 2.0 \times 10^{-4}P^2 - 0.014TC - 5.0 \times 10^{-4}TP \\ & - 5.3 \times 10^{-3}CP \end{aligned} \quad (23)$$

According to the statistical analysis (Table 2), the linear effects of T and C, as well as the quadratic effect of C, were statistically significant for the WL response ($p \leq 0.05$). In turn, the linear effect of C and the combined effect of C and P were significant for SG ($p \leq 0.05$). For the a_w response, the linear effect of C and P, the quadratic effect of C, and the combined effect of T and C were statistically significant ($p \leq 0.05$). The NaCl concentration in the osmotic solution was the parameter that had the greatest effect on all responses and, consequently, on the OD process (Table 2), being positive for WL and SG and negative

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), 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)	18.18	1	130.66	0.008	0.81	1	10.93	0.081	1.1×10^{-7}	1	0.01	0.914
T (Q)	2.43	1	17.43	0.053	0.34	1	4.53	0.167	1.0×10^{-4}	1	14.45	0.063
2 C (% NaCl) (L)	177.16	1	1273.54	0.001	15.26	1	205.68	0.005	3.9×10^{-3}	1	534.34	0.002
C (Q)	2.60	1	18.71	0.049	0.63	1	8.44	0.101	3.6×10^{-4}	1	49.58	0.020
3 P (kPa) (L)	1.90	1	13.67	0.066	0.46	1	6.18	0.131	1.5×10^{-4}	1	20.72	0.045
P (Q)	2.26	1	16.22	0.056	0.43	1	5.81	0.137	3.9×10^{-5}	1	5.30	0.148
1L by 2L	1.04	1	7.50	0.111	0.47	1	6.37	0.128	2.3×10^{-4}	1	31.67	0.030
1L by 3L	0.78	1	5.61	0.141	0.05	1	0.70	0.491	3.7×10^{-5}	1	5.15	0.151
2L by 3L	1.10	1	7.91	0.107	1.56	1	21.00	0.044	1.1×10^{-6}	1	0.14	0.740
Lack-of-fit	4.84	5	6.96	0.130	7.48	5	20.17	0.048	5.3×10^{-4}	5	14.70	0.065
Pure error	0.28	2			0.15	2			1.5×10^{-5}	2		
Total SS	209.86	16			28.01	16			5.3×10^{-3}	16		

Bold values indicate statistical significance *p* value ($p \leq 0.05$)

SS Sum of squares; DF Degree of freedom; R_{WL}^2 0.98; R_{SG}^2 0.73; R_{aw}^2 0.90; L Linear; Q Quadratic

for a_w . This behavior was observed by Maciel et al. (2015) for OD of highwaterman catfish (*Hypophthalmus edentatus*) and by Medina-Vivanco et al. (2002) for OD of tilapia (*Oreochromis niloticus*).

Optimization of the osmotic dehydration process

The desirability function was employed to define the best operational condition for the OD process of pirarucu fillets in the experimental domain studied. Figure 1 shows the desirability values as a function of each independent variable (WL, SG, and a_w) when the other two were fixed at the central point of the CCD. The profiles presented in the column desirability show the acceptable range of the desirability response ($0 \leq d(x_i) \leq 1$). In turn, the profiles presented in the last row show the individual desirability for each factor, from which the global desirability is obtained. The results showed a maximum desirability of 0.999 obtained using the following experimental conditions: 35 °C, 25% NaCl, and 101 kPa. This desirability value corresponded to WL = 14.82%, SG = 6.15%, and $a_w = 0.85$.

In order to validate the models, experimental assays were carried out under the conditions indicated as optimal for the OD process. Under those conditions, the product has 66.53% (± 1.86) moisture content and the experimental values of WL ($14.87 \pm 1.46\%$) and a_w ($0.87 \pm 0.02\%$) converged well to the values estimated by the desirability function and by Eq. (21) and (22), respectively. On the other hand, the experimental value of SG ($8.56 \pm 0.45\%$) had lower convergence to the estimated value, which is attributed to the lower correlation of the polynomial model

(Eq. 23) to the experimental data of SG in the CCD. Maciel et al. (2012, 2015) established as optimal conditions 49 °C and 28% NaCl for the OD of pirarucu at atmospheric pressure and 25 °C, 25% NaCl for the OD of highwaterman catfish, respectively.

Osmotic dehydration process kinetics

The evolution of WL, SG, and a_w over time during OD of pirarucu fillets under the optimized condition is presented in Fig. 2. During the OD process, WL and SG increased, which was more marked in the first 2 h. This behavior is attributed to the driving force responsible for the diffusion of water and solids between the hypertonic solution and the product in the beginning of the process. The osmotic pressure and concentration gradients between the osmotic solution and the product are higher in the beginning of the process, which favors the WL and SG rates. Over time, those gradients gradually decrease until the equilibrium condition is reached (Rastogi et al. 2002; Medina-Vivanco et al. 2002). In turn, the increase in WL and SG in the first 2 h of the process caused a marked reduction in the product's a_w , which remained virtually unchanged from that point onwards and reached the value of 0.84.

The apparent diffusivity (*D*) value for the OD of pirarucu fillets was $3.59 \times 10^{-9} \text{ m}^2/\text{s}$ for WL and $3.49 \times 10^{-9} \text{ m}^2/\text{s}$ for SG. Those values are of the same order of magnitude ($10^{-9} \text{ m}^2/\text{s}$) as the one found by Medina-Vivanco et al. (2002) for the OD of tilapia fillets and above those observed by Corzo and Bracho (2007a, b) for the OD of sardine fillets and by Gallart-Jornet et al. (2007) for the OD of cod and salmon (10^{-10} – $10^{-12} \text{ m}^2/\text{s}$).

Fig. 1 Profile of desirability of osmotically dehydrated pirarucu fillets. Data obtained by changing each independent variable while the other two were fixed at the central point of the CCD

Fig. 2 Evolution of water loss (Circle, WL), solid gain (Square, SG), and water activity (Diamond, a_w) over time during the osmotic dehydration process under the optimized condition (35 °C, atmospheric pressure, and 25% NaCl) and the Peleg model fitted (Solid line) to the WL and SG data

The values of R^2 (>0.96) and RMSE (<2.34) indicate the model proposed by Peleg (1988) fitted the experimental data of OD kinetics for pirarucu fillets and can be used with good precision to predict the kinetics of WL and SG in the experimental domain studied (Fig. 2). The constant k_1 for WL ($0.59 \text{ h}/(\text{g/g db})$) and for SG ($1.11 \text{ h}/(\text{g/g db})$) is related to the initial transfer rate of water and solids, respectively, and the lowest its value, the higher those rates. The value of k_2 ($1.34 (\text{g/g db})^{-1}$ for WL and $1.99 (\text{g/g db})^{-1}$ for SG), in turn, is inversely related to the water content and solute concentration at equilibrium (Corzo and Bracho 2006). The values of WL^∞ and SG^∞ estimated for the OD process of pirarucu fillets were 21.32 and 11.83%. Corzo and Bracho (2006) reported approximately the same values of Peleg constants for WL [$k_1 = 0.39 \text{ h}/(\text{g/g db})$ and $k_2 = 1.03 (\text{g/g db})^{-1}$] and slightly higher for SG [$k_1 = 1.63 \text{ h}/(\text{g/g db})$ and $k_2 = 3.56 (\text{g/g db})^{-1}$] for the OD process of sardine fillets at 34 °C in an osmotic solution with 24% NaCl.

Fig. 3 Evolution of the moisture ratio of osmotically dehydrated pirarucu fillets over time at 40 °C (Circle), 50 °C (Square), 60 °C (Triangle), and 70 °C (Diamond)

Drying process

The drying curves of the pirarucu fillets dehydrated under the optimal OD condition for the different drying temperatures (40–70 °C) are presented in Fig. 3. According to the drying kinetics, increasing the temperature from 40 to 50 °C led to an increase in drying rates, favoring the elimination of water from the product, which is confirmed by the effective diffusivity value ($D_{\text{eff}} = 10.85 \times 10^{-9} \text{ m}^2/\text{s}$ for 40 °C and $D_{\text{eff}} = 12.30 \times 10^{-9} \text{ m}^2/\text{s}$ for 50 °C). In turn, for the drying performed at 60 °C, the value of D_{eff} remained at virtually the same level ($11.89 \times 10^{-9} \text{ m}^2/\text{s}$), while for the drying at 70 °C, the value of D_{eff} dropped ($10.98 \times 10^{-9} \text{ m}^2/\text{s}$), which indicates a reduction in drying rates at this temperature level. The drying time and

corresponding product moisture for the different drying conditions were: 40 °C (9.8 h and 42.4%), 50 °C (9.2 h and 40.3%), 60 °C (8.8 h and 37.1%), and 70 °C (9.5 h and 30.5%).

The influence of NaCl in reducing the water content in the muscle of salted fish can be attributed to the effects of protein denaturation (Shenouda 1980). According to Sanchez and Lam (1965), and Jonsson et al. (2001), the denaturation of fish proteins, favored by the action of salt, lowers the solubility and water molecule retention capacity of the proteins due to the weakening of the protein-water binding forces. This effect favors the removal of water from the product and was observed in the temperature range of 40–50 °C.

Although protein denaturation favors the elimination of water from fish muscle, at high NaCl concentrations and when higher temperatures are employed in the drying process, the increase in solubility of salt and other compounds in the muscle may be favored, thus increasing the interaction between water molecules and those solutes (Nketsia-Tabiri and Sefa-Dedeh 1995). In this context, the hydrophilic nature of NaCl and the possible structural and biochemical changes may have contributed to the behavior observed for the drying at 60 and 70 °C. The values of D_{eff} observed for the drying process of pirarucu muscle in the experimental conditions studied varied within the range found by Zogzas et al. (1996) for food products (10^{-12} – $10^{-8} \text{ m}^2/\text{s}$) and by Panagiotou et al. (2004) for the drying of fish (10^{-11} – $10^{-9} \text{ m}^2/\text{s}$).

The values of R^2 (>0.950), of χ^2 (<0.0007), and of RMSE (<0.063) presented in Table 3 indicate that, overall, all models tested are able to predict, with good precision, the drying kinetics of osmotically dehydrated pirarucu fillets in the different conditions studied. However, the Midilli model had the best fit to all temperature conditions

Table 3 Coefficient of determination (R^2), reduced Chi square (χ^2), and root mean square error (RMSE) values for the non-linear regressions of the models fitted to the drying data at different temperatures of osmotically dehydrated pirarucu fillets

Model number	Statistical parameters											
	R^2 40 °C	$\chi^2 \times 10^4$	RMSE	R^2 50 °C	$\chi^2 \times 10^4$	RMSE	R^2 60 °C	$\chi^2 \times 10^4$	RMSE	R^2 70 °C	$\chi^2 \times 10^4$	RMSE
1	0.993	7.0	0.026	0.973	24.0	0.049	0.997	4.0	0.019	0.951	41.0	0.063
2	0.998	2.0	0.015	0.997	2.0	0.015	0.997	4.0	0.019	0.996	4.0	0.019
3	0.998	2.0	0.015	0.997	2.0	0.015	0.997	4.0	0.019	0.996	4.0	0.019
4	0.995	5.0	0.022	0.989	1.0	0.032	0.997	3.0	0.017	0.980	17.0	0.040
5	0.998	2.0	0.014	0.994	5.0	0.022	0.997	4.0	0.019	0.984	14.0	0.037
6	0.996	2.0	0.012	0.990	10.0	0.030	0.998	3.0	0.017	0.984	15.0	0.037
7	0.999	2.0	0.013	0.999	1.0	0.010	0.999	3.0	0.017	0.998	2.0	0.013
8	0.999	2.0	0.013	0.999	1.0	0.010	0.997	4.0	0.019	0.998	2.0	0.013
9	0.999	1.0	0.010	0.999	1.0	0.007	0.999	3.0	0.016	0.999	1.0	0.010
10	0.999	2.0	0.012	0.999	1.0	0.010	0.998	3.0	0.016	0.998	2.0	0.013

($R^2 > 0.999$; $\chi^2 < 0.0003$; RMSE < 0.016), followed by the Two-term model ($R^2 > 0.998$; $\chi^2 < 0.0003$; RMSE < 0.016), both with four parameters. In turn, the Page model can be used, for practical effect, in the prediction of drying kinetics of the product as it had very good fits to the experimental data ($R^2 > 0.996$; $\chi^2 < 0.0004$; RMSE < 0.019), besides having only two parameters, which facilitates its mathematical solution. Martins et al. (2015) observed that the Midilli model best predicted the drying kinetics of pirarucu with different salt concentrations, followed by the diffusion approximation and Page models.

Conclusions

The osmotic dehydration (OD) and complementary drying of pirarucu fillets were studied. The concentration of the osmotic solution was the factor with the greatest impact on the increase of water loss (WL) and solid gain (SG) and on the decrease in water activity (a_w) during the OD process. The optimal operational condition defined for the OD process was 35 °C at atmospheric pressure in an osmotic solution with 25% NaCl. The diffusivity for the OD process was $3.59 \times 10^{-9} \text{ m}^2/\text{s}$ for WL and $3.49 \times 10^{-9} \text{ m}^2/\text{s}$ for SG and the Peleg model proved effective in predicting the WL and SG kinetics of the OD process. The study of the drying of osmotically dehydrated pirarucu fillets showed the process is not favorable when temperatures above 60 °C are employed. For the temperature range studied (40–70 °C), the drying process had diffusivity values between $10.85 \times 10^{-9} \text{ m}^2/\text{s}$ and $12.30 \times 10^{-9} \text{ m}^2/\text{s}$ and the Page model proved able to predict, with very good precision, the product's drying kinetics.

Acknowledgements The authors would like to thank FINEP (Financiadora de Estudos e Projetos) and SEBRAE (Serviço de Apoio às Micro e Pequenas Empresas) for the financial support (028/2010).

References

- Akpinar EK, Bicer Y, Yildiz C (2003) Thin layer drying of red pepper. *J Food Eng* 59:99–104. doi:[10.1016/S0260-8774\(02\)00425-9](https://doi.org/10.1016/S0260-8774(02)00425-9)
- AOAC (1997) Official methods of analysis, 16th edn. Association of Official Analytical Chemists, Gaithersburg
- Baslar M, Kılıçlı M, Yalınkılıç B (2015) Dehydration kinetics of salmon and trout fillets using ultrasonic vacuum drying as a novel technique. *Ultrason Sonochem* 27:495–502. doi:[10.1016/j.ultsonch.2015.06.018](https://doi.org/10.1016/j.ultsonch.2015.06.018)
- Castello L (2008) Nesting habitat of pirarucu *Arapaima gigas* (Schinz) in floodplains of the Amazon. *J Fish Biol* 72:1520–1528. doi:[10.1111/j.1095-8649.2007.01778.x](https://doi.org/10.1111/j.1095-8649.2007.01778.x)
- Chen Z-G, Guo X-Y, Wu T (2016) A novel dehydration technique for carrot slices implementing ultrasound and vacuum drying methods. *Ultrason Sonochem* 30:28–34. doi:[10.1016/j.ultsonch.2015.11.026](https://doi.org/10.1016/j.ultsonch.2015.11.026)
- Corrêa JLG, Dev SRS, Garipey Y, Raghavan GSV (2011) Drying pineapple by microwave-vacuum with osmotic pretreatment. *Dry Technol* 29:1556–1561. doi:[10.1080/07373937.2011.582558](https://doi.org/10.1080/07373937.2011.582558)
- Corzo O, Bracho N (2006) Application of Peleg model to study mass transfer during osmotic dehydration of sardine sheets. *J Food Eng* 75:535–541. doi:[10.1016/j.jfoodeng.2005.05.001](https://doi.org/10.1016/j.jfoodeng.2005.05.001)
- Corzo O, Bracho N (2007a) Determination of water effective diffusion coefficient of sardine sheets during vacuum pulse osmotic dehydration. *Food Sci Technol* 40:1452–1458. doi:[10.1016/j.lwt.2006.04.008](https://doi.org/10.1016/j.lwt.2006.04.008)
- Corzo O, Bracho N (2007b) Water effective diffusion coefficient of sardine sheets during osmotic dehydration at different brine concentrations and temperatures. *J Food Eng* 80:497–502. doi:[10.1016/j.jfoodeng.2006.06.008](https://doi.org/10.1016/j.jfoodeng.2006.06.008)
- Corzo O, Gómez ER (2004) Optimization of osmotic dehydration of cantaloupe using desired function methodology. *J Food Eng* 64:213–219. doi:[10.1016/j.jfoodeng.2003.09.035](https://doi.org/10.1016/j.jfoodeng.2003.09.035)
- Corzo O, Bracho N, Marjal J (2006a) Color change kinetics of sardine sheets during vacuum pulse osmotic dehydration. *J Food Eng* 75:21–26. doi:[10.1016/j.jfoodeng.2005.03.046](https://doi.org/10.1016/j.jfoodeng.2005.03.046)
- Corzo O, Bracho N, Marjal J (2006b) The use of fractional conversion technique on firmness change kinetics of vacuum pulse osmotic dehydration sardine sheets. *J Food Eng* 73:358–363. doi:[10.1016/j.jfoodeng.2005.01.038](https://doi.org/10.1016/j.jfoodeng.2005.01.038)
- Corzo O, Bracho N, Rodríguez J, González M (2007) Predicting the moisture and salt contents the sardine sheets during vacuum pulse osmotic dehydration. *J Food Eng* 80:781–790. doi:[10.1016/j.jfoodeng.2006.07.007](https://doi.org/10.1016/j.jfoodeng.2006.07.007)
- Crank J (1975) The mathematics of diffusion. Clarendon Press, Oxford
- Derringer G, Suich R (1980) Simultaneous optimization of several response variables. *J Qual Technol* 12:214–219
- Fante C, Corrêa J, Natividade M, Lima J, Lima L (2011) Drying of plums (*Prunus* sp, c.v Gulfbreeze) treated with KCl in field and subjected to pulsed vacuum osmotic dehydration. *Int J Food Sci Technol* 46:1080–1085. doi:[10.1111/j.1365-2621.2011.02619.x](https://doi.org/10.1111/j.1365-2621.2011.02619.x)
- Fernandes FAN, Gallão MI, Rodrigues S (2008) Effect of osmotic dehydration and ultrasound pre-treatment on cell structure: melon dehydration. *Food Sci Technol* 41:604–610. doi:[10.1016/j.lwt.2007.05.007](https://doi.org/10.1016/j.lwt.2007.05.007)
- Fito P (1994) Modeling of vacuum osmotic dehydration of food. *J Food Eng* 22:313–328. doi:[10.1016/0260-8774\(94\)90037-X](https://doi.org/10.1016/0260-8774(94)90037-X)
- Gallart-Jornet L, Barat JM, Rustad T, Erikson U, Escribano I, Fito PA (2007) Comparative study of brine salting of Atlantic cod (*Gadus morhua*) and Atlantic salmon (*Salmo salar*). *J Food Eng* 79:261–270. doi:[10.1016/j.jfoodeng.2006.01.053](https://doi.org/10.1016/j.jfoodeng.2006.01.053)
- Jonsson A, Sigurgisladottir S, Hafsteinsson H, Kristbergsson K (2001) Textural properties of raw Atlantic salmon (*Salmo salar*) fillets measured by different methods in comparison to expressible moisture. *Aquac Nutr* 7:81–89. doi:[10.1046/j.1365-2095.2001.00152.x](https://doi.org/10.1046/j.1365-2095.2001.00152.x)
- Khuri AI, Cornell JA (1996) Response surfaces: designs and analyses. Marcel Dekker, New York
- Maciel RA, Viana CMC, Simões MG, Fontelles MJP (2012) Osmotic dehydration of fillets pirarucu (*Arapaima gigas*). *Rev Bras Tecnol Agroind* 6:618–629. doi:[10.3895/S1981-36862012000100001](https://doi.org/10.3895/S1981-36862012000100001)
- Maciel RA, Rodrigues AMC, Pena RS (2015) Influence of the process parameters on osmotic dehydration of mapara (*Hypophthalmus edentatus*) fillet. *J Food Sci Technol* 53:676–684. doi:[10.1007/s13197-015-1999-5](https://doi.org/10.1007/s13197-015-1999-5)
- Martins MG, Martins DEG, Pena RS (2015) Drying kinetics and hygroscopic behavior of pirarucu (*Arapaima gigas*) fillet with

- different salt contents. *Food Sci Technol* 62:144–151. doi:[10.1016/j.lwt.2015.01.010](https://doi.org/10.1016/j.lwt.2015.01.010)
- Medina-Vivanco M, Sobral PJA, Hubinger MD (2002) Osmotic dehydration of tilapia fillets in limited volume of ternary solutions. *Chem Eng J* 86:199–205. doi:[10.1016/S1385-8947\(01\)00290-X](https://doi.org/10.1016/S1385-8947(01)00290-X)
- Nketsia-Tabiri J, Sefa-Dedeh S (1995) Optimization of process conditions and quality of salted dried tilapia (*Oreochromis niloticus*) using response surface methodology. *J Sci Food Agric* 69:117–127. doi:[10.1002/jsfa.2740690118](https://doi.org/10.1002/jsfa.2740690118)
- Ooizumi T, Kawase M, Kahane Y (2003) Permeation of sodium chloride into fish meat and its effect on moisture content as a function of the osmotic pressure of the soaking solution. *Fish Sci* 69:830–835. doi:[10.1046/j.1444-2906.2003.00694.x](https://doi.org/10.1046/j.1444-2906.2003.00694.x)
- Panagiotou NM, Krokida MK, Maroulis ZB, Saravacos GD (2004) Moisture diffusivity: literature data compilation for foodstuffs. *Int J Food Prop* 7:273–299. doi:[10.1081/JFP-120030038](https://doi.org/10.1081/JFP-120030038)
- Peleg M (1988) An empirical model for the description of moisture sorption curves. *J Food Sci* 53:1216–1217. doi:[10.1111/j.1365-2621.1988.tb13565.x](https://doi.org/10.1111/j.1365-2621.1988.tb13565.x)
- Rastogi NK, Raghavarao KSMS, Niranan K, Knorr D (2002) Recent developments in osmotic dehydration: methods to enhance mass transfer. *Trends Food Sci Tech* 13:48–59. doi:[10.1016/S0924-2244\(02\)00032-8](https://doi.org/10.1016/S0924-2244(02)00032-8)
- Sanchez JT, Lam RC (1965) Principios técnicos de salado y secado del pescado: estudio químico de la sal en el litoral. Institui del Mar del Peru, La Punta
- Shenouda SYK (1980) Theories of protein denaturation during frozen storage of fish flesh. *Adv Food Res* 26:275–311. doi:[10.1016/S0065-2628\(08\)60320-1](https://doi.org/10.1016/S0065-2628(08)60320-1)
- Torres FF, Troncoso OP, Nakamatsu J, Grande CJ, Gomez CM (2008) Characterization of the nanocomposite laminate structure occurring in fish scales from *Arapaima gigas*. *Mater Sci Eng* 28:1276–1283. doi:[10.1016/j.msec.2007.12.001](https://doi.org/10.1016/j.msec.2007.12.001)
- Uddin MB, Ainsworth P, Ibanoglu S (2004) Evaluation of mass exchange during osmotic dehydration of carrots using response surface methodology. *J Food Eng* 65:473–477. doi:[10.1016/j.jfoodeng.2004.02.007](https://doi.org/10.1016/j.jfoodeng.2004.02.007)
- Watson LC, Stewart DJ, Teece MA (2013) Trophic ecology of *Arapaima* in Guyana: giant omnivores in Neotropical floodplains. *Neotrop Ichthyol* 11:341–349. doi:[10.1590/S1679-62252013000200012](https://doi.org/10.1590/S1679-62252013000200012)
- Zogzas NP, Maroulis ZB, Marinos-Kouris D (1996) Moisture diffusivity data compilation in foodstuffs. *Dry Technol* 14:2225–2253. doi:[10.1080/07373939608917205](https://doi.org/10.1080/07373939608917205)