MASS TRANSFER MODELING DURING OSMOTIC DEHYDRATION OF CHUB MACKEREL (SCOMBER JAPONICUS) SLICES IN SALT AND GLYCEROL SOLUTION AT DIFFERENT TEMPERATURES

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ABSTRACT

The use of osmotic dehydration to preserve fish products could be an interesting option in order to maintain the characteristics of food. The objective of this research was to study the mathematical modeling of water loss and solute gain during the cooking-infusion of chub mackerel (*Scomber japonicus*) slices at different temperatures. Osmotic dehydration was performed by cooking-infusion in solutions containing 54% (w/w) glycerol and 7% (w/w) salt (solution $a_w = 0.64$) at different temperatures (50, 70 and 90C). Experimental results were adjusted to the Crank, Weibull, and Zugarramurdi & Lupín models. Mass transfer kinetics of water, salt and glycerol during osmotic dehydration of chub mackerel slices were adequately adjusted, with similar accuracy, by the three models. However, the Zugarramurdi & Lupín model was preferred because it also allowed the prediction of water and solids contents at equilibrium, which is relevant for the development of this kind of products.

PRACTICAL APPLICATIONS

This article deals with the mathematical modeling of mass transfer during osmotic dehydration (OD) of chub mackerel (*Scomber japonicus*) slices. Based on the importance of developing new preservation technologies of this species, we consider that OD by immersion in osmotic solutions represents a good alternative for the industrialized production of chub mackerel. The models proposed in this work allowed the accurate prediction of water and soluble solid transfer kinetics during processing at different temperatures. Moreover, Zugarramurdi & Lupín model satisfactorily predicted the equilibrium values. The development of new products and the optimization of OD process depend on the adequate understanding of the moisture and solids contents and on the knowledge of the diffusion coefficients during OD. The results of this work are of sum importance for the fish processing industry to develop intermediate moisture fish products, which, in turn, can be ready-to-eat products, as well as input material of further processes.

INTRODUCTION

Chub mackerel (*Scomber japonicus*) is found in warm and temperate waters of the Atlantic, Indian and Pacific Oceans (Buratti *et al.* 2011). The geographic area of chub mackerel

along the Atlantic coast of South America is bounded on the north by the latitude of 23°S (Rio de Janeiro) and on the south by the latitude of 39°S (Bahía Blanca) (Angelescu 1980). In Argentina, during 2010 and 2011, the fishing fleet reached the highest historical landings with 27.558 and

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28.253 tons, respectively (MAGYP 2011). At present, the usual preservation processing of chub mackerel consists mainly of freezing and canning. However, these processes affect the nutritional properties as well as quality attributes such as texture and flavor. The heat-labile vitamins A, C, biotin, pyridoxine, pantothenic acid, thiamine, riboflavin and niacin are the most damaged nutrients by the canning and sterilizing processes (Pigott and Tucker 1990). Considering the aforementioned, it is important to develop new processing alternatives for this species.

Osmotic dehydration (OD) is used as a pretreatment to improve the sensory, nutritional and functional properties of food without changing its integrity (Lemus-Mondaca et al. 2009b). OD consists of the immersion of solid food, either whole or in parts, in aqueous solutions of high concentration of sugars and/or salts to cause at least two main flows, simultaneous and cross-current: the flow of water from the food to the solution and the simultaneous transfer of solute from the solution into the food (Fante et al. 2011). These flows are due to the concentration gradients of water and solute existing at either side of the membranes that constitute the tissue of the food. During this process, the mass transfer rates are increased or decreased until equilibrium is reached (Tortoe 2010).

Knowledge of the kinetics of water and salt transfer during processing has great technological importance because it allows estimating the immersion time of food in an osmotic solution to obtain products with determined salt and moisture contents and permits an adequate process design (Ochoa-Martínez and Ayala-Aponte 2005; Schmidt et al. 2008). Several solutes, including sugars, salts and humectants, can be used to obtain intermediate moisture foods by reducing the $a_{\rm w}$ in the range of 0.6–0.9. The selection of the proper solutes for the stabilization of the food involves consideration about the aw lowering capacity, flavor impact, texture, cost and safety. Taking into account these characteristics, a combination of sodium chloride and glycerol has been conveniently used to generate intermediate moisture products (Brockman 1970; Favetto et al. 1981; Sánchez Pascua et al. 1994; Moreira et al. 2007).

Water loss kinetics during OD has been modeled with different equations such as the Fick diffusion law (Gerla and Rubiolo 2003; Gou et al. 2003; Telis et al. 2003; Graiver et al. 2006; Corzo and Bracho 2007; Casales et al. 2009, Corrêa et al. 2010), empiric relations like Peleg and Zugarramurdi & Lupín models (Zugarramurdi and Lupín 1980; Turhan et al. 2002; Corzo and Bracho 2005, 2006; Schmidt et al. 2009; Czerner and Yeannes 2010) and probabilistic models like Weibull (Corzo and Bracho 2006). Empiric and probabilistic models are based on the mathematical representation of natural processes; they have simple application and solve the lack of precision of the Crank model (Ochoa-Martínez and Ayala-Aponte 2005; Corzo and Bracho 2006).

The information in the literature about the application of such empirical and probabilistic models for describing OD of fish products in glycerol–salt solutions is very scarce. The objective of this work was to study and model the mass transfer kinetics during OD of chub mackerel slices in glycerol–salt solutions at different temperatures, using the Crank, Weibull and Zugarramurdi & Lupín models.

MATERIALS AND METHODS

Raw Materials

Chub mackerel (*S. japonicus*) caught in Mar del Plata, Argentina, in October 2011 and stored at -18C was used in this study. The skin, head, tail and viscera were withdrawn from the frozen samples. The frozen trunk was cut into slices with a stainless steel knife. Slices had an average thickness of 0.72 ± 0.05 cm and an average diameter of 5.15 ± 0.15 cm.

Infusion Solution

Infusion solution composition (w/w) was as follows: 54% glycerol (Biopack, Zárate, Buenos Aires, Argentina, 99.5 g/ 100 g of purity), 38.3% water, 7% sodium chloride (Biopack, 99 g/100 g of purity) and 0.7% potassium sorbate (DQI, Medellín, Colombia, 99 g/100 g of purity). The $a_{\rm w}$ of cooking-infusion solution was 0.64. The fish: solution was 1:10 (w/w) to avoid significant dilution of the osmotic solution as a result of water loss and solute uptake.

Cooking-Infusion

The slices of chub mackerel were thawed under refrigeration until a temperature of 8C and placed in the infusion solution at temperatures of 50 ± 1 , 70 ± 1 and 90 ± 1 C until the equilibrium was reached (a maximum immersion time of 3 h). At specified time intervals (5, 10, 15, 20, 25, 40, 60, 80, 120, 150 and 180 min), samples were removed from the solution for physicochemical analysis. The slices were drained, superficially rinsed with distilled water, dried with absorbent paper and weighed. Three slices were extracted for each sampling time at each of the temperatures tested. Two experiences were carried out for each chosen temperature.

Physicochemical Analyses

The water content was determined at 105C until constant weight (AOAC 1990; Sec. 984.25) using a drying oven (Marne, 644, Córdoba, Argentina). The ashes were determined by calcinations at 550C as described by AOAC (1990), Sec. 945.46, using an electric oven (Indef, 332,

Córdoba, Argentina). The lipid extraction was according to AOAC (1990), Sec 922.06. The total proteins were determined as described by AOAC (1990), Sec. 920.152. The sodium chloride content was determined using the Mohr method adapted for food (Kirk *et al.* 1996). The glycerol content was determined using an enzymatic UV method (BoehringerMannheim/R-Biopharm, Darmstadt, Germany) with a spectrophotometer (Shimadzu UV-1601 PC, Kyoto, Japan), and $a_{\rm w}$ was determined as described by AOAC (1990), Sec. 978.18, using an Aqualab hygrometer (CX-2T, Decagon, Pullman, WA).

All analyses were carried out in triplicate.

Mathematical Models

Crank Model. Crank model is based on a group of solutions of Fick's diffusion law for different geometries (Crank 1975). The diffusion of sodium chloride and glycerol through the mackerel slices can be considered as diffusion through a plane sheet of thickness 2l. At time t = 0, the region -1 < x < 1 is at uniform concentration (C_0) and the surfaces are kept at a constant concentration (C_1). The diffusion equation for one-dimensional motion can be expressed as follows (Crank 1975) (Eq. 1):

$$\frac{\partial C_t}{\partial t} = \frac{\partial \left(D\frac{\partial C_t}{\partial x}\right)}{\partial x} \tag{1}$$

where C_t is the concentration at time t and coordinate x, and D is the diffusion coefficient. If D is constant, Eq. (1) can be expressed as

$$\frac{\partial C_t}{\partial t} = D \frac{\partial^2 C_t}{\partial x} \tag{2}$$

Equation (2) solution for semi-infinite plane sheet for short times is given by

$$(C_t - C_0/C_{\infty} - C_0) = \frac{M_t}{M} = 2\left(\frac{Dt}{\pi l^2}\right)^{\frac{1}{2}}$$
 (3)

where M_t and M_{∞} are the water loss or solutes content (expressed as g on a non-salt and non-glycerol dry matter basis, g/g dm) at time t and at infinite time, respectively, and l is half of the thickness of the slice.

The period of time that was analyzed to adjust the experimental values to Crank model was up to 80 min.

This model assumes that concentration at the surface remains constant, and external resistance is worthless against internal resistance. Furthermore, sample geometry is a semi-infinite sheet.

Zugarramurdi & Lupín (Z & L) Model. Zugarramurdi and Lupín (1980) proposed a mathematical model, with an

exponential approach to the salt and water equilibrium values:

$$\frac{\partial x_t}{\partial t} = K(X^{\text{eq}} - X_t) \tag{4}$$

where X_t and X^{eq} are the water, salt or glycerol contents (expressed as g on a non-salt and non-glycerol dry matter basis, g/g dm) at a given time t (h) and at equilibrium, respectively, and K is the specific rate constant (h⁻¹).

Integrating Eq. (4) with the initial condition $X_{t=0} = X_0$, the following is obtained:

$$X_{t} = X_{0}e^{-Kt} + X^{eq}(1 - e^{-Kt})$$
(5)

Weibull Model. The Weibull model was used to describe the behavior of systems or events that have some degree of variability such as the OD kinetics. The probability density function of the Weibull distribution may be described as follows (Cunha *et al.* 1998; Corzo and Bracho 2008):

$$f(t) = \begin{cases} \frac{\beta}{\alpha} \left(\frac{t}{\alpha}\right)^{\beta - 1} \exp\left(-\left(\frac{t}{\alpha}\right)^{\beta}\right), t > 0 \\ 0, elsewhere \end{cases}$$
 (6)

where α is the scale parameter of the Weibull model, β is the shape parameter and t is the sampling time. If one considers that n_t corresponds to the fractional amount of a given component X, changing from an initial value (X_0) to a final equilibrium value (X^{eq}) , and that the time required to reach a certain value of n_t is represented by the continuous random variable G, with probability density function F(t), where F(t) is the Weibull distribution function, then n_t can be defined as the probability of having a certain fractional amount of X for, at least, a specified time t, under specified experimental conditions (Cunha et al. 1998). Therefore,

$$n_t = P(G > t) = \int_{t}^{\infty} f(u) du = 1 - F(t) = \exp\left(-\left(\frac{t}{\alpha}\right)^{\beta}\right)$$
 (7)

where F(t) is the corresponding cumulative distribution. The fractional amount of moisture or solute content during OD can be expressed as (Cunha *et al.* 1998)

$$n_t = \frac{X_t - X^{\text{eq}}}{X_0 - X^{\text{eq}}} = \exp\left(-\left(\frac{t}{\alpha}\right)^{\beta}\right)$$
 (8)

where X_0 , X_t and X^{eq} are the water or solute contents (expressed as g on a non-salt and non-glycerol dry matter basis, g/g dm) at t = 0, at a time t and at equilibrium, respectively, α is the scale parameter of the Weibull model, β is the shape parameter (dimensionless) and t is the sampling time.

Arrhenius Equation

Dependence of water, salt and glycerol diffusion coefficients as well as of the kinetic parameters of models on temperature is represented by the linearized Arrhenius equation (Eq. 9):

$$\ln(A_n) = \ln(A_0) - \left(\frac{E_a}{RT}\right) \tag{9}$$

where A_n is the parameter to be studied, A_0 is the Arrhenius factor (cm²/s), E_a is the activation energy (kJ/mol), R is the universal gas constant (8.314 J/[mol K]) and T is the absolute temperature (K). From this equation, the activation energy, E_a (kJ/mol), can be estimated by plotting $\ln(A_n)$ against T^{-1} .

Statistical Analysis

The fitting of the models to the experimental data was performed using OriginPro 8 (OriginLab, Northampton, MA). The goodness of fit was determined using the determination coefficient (R^2) and the root mean square error (RMSE, Eq. 10):

$$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (X_{ip} - X_i)^2}$$
 (10)

where X_i is the experimental value, X_{ip} is the predicted value and n is the number of data pairs.

RESULTS AND DISCUSSION

Proximate Analysis of Raw Chub Mackerel Slices

The main constituents of raw chub mackerel slices were water content (73.85%), crude protein (21.13%), lipid content (3.69%) and ashes (1.33%).

OD Kinetics

The effect of the temperature on the water loss and solute gain was analyzed. The water loss (expressed as initial water content – water content at time $t [X_0 - X_1]$) and salt and glycerol content during the OD at different temperatures are shown in Fig. 1. It was observed that an increase in the process temperature improved the water loss and the salt and glycerol gain. Higher temperatures seem to promote faster water loss through swelling and plasticizing of cell membranes as well as better water transfer characteristics on the product surface due to lower viscosity of the osmotic medium (Burhan Uddin et al. 2004; Tortoe 2010). A similar behavior was also reported by Corzo and Bracho (2006) during the OD of sardine sheets. The decrease in the water content and the increase in the salt and glycerol contents had a maximum rate at the early stages of the process due to higher concentration gradients of water and solute existing at either side of the membranes that constitute the food. Then, a decrease in the rates of water and solute diffusion was observed until the equilibrium was reached (equilibrium values not shown in Fig. 1).

As shown in Fig. 2, the $a_{\rm w}$ of the slices decreased with process time due to the transfer of solutes from the solution into the food and to the flow of water from the food to the solution. Higher temperatures generated a faster decrease of $a_{\rm w}$ due to the improvement of mass transfer kinetics. It can be seen that the time required to reach a determined $a_{\rm w}$ decreased as the process temperature increased.

Estimation of Diffusion Coefficients

The salt diffusivity values of fish depend on species, temperature, muscle orientation, fat content, the presence or absence of skin and other factors (Zugarramurdi and Lupín 1980; Collignan *et al.* 2001).

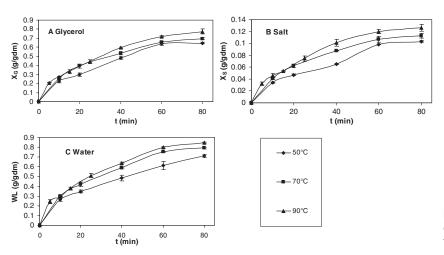


FIG. 1. OSMOTIC DEHYDRATION KINETICS OF CHUB MACKEREL SLICES AT DIFFERENT TEMPERATURES

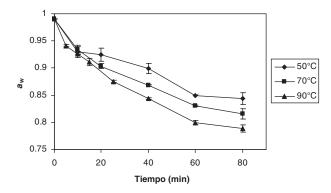


FIG. 2. DECREASE OF A_W AT DIFFERENT TEMPERATURES

The diffusion coefficient (D) values of water, salt and glycerol obtained for chub mackerel slices are shown in Table 1. Results found in this work indicated that the experimental data were adequately fitted to the Crank model, as indicated by the high determination coefficients $(0.942 < R^2 < 0.995)$ and the low RMSE (0.0159 < RMSE < 0.0757) observed (Table 1). The values of $D_{\rm salt}$ and $D_{\rm water}$ (Table 1) for chub mackerel slices are in agreement with the results reported by Uribe *et al.* (2011) regarding the OD of jumbo squid at temperatures between 75 and 95C. Nevertheless, lower $D_{\rm salt}$ values were reported during the OD of Atlantic salmon (Wang *et al.* 2000).

It is important to notice that information about glycerol diffusivity in fish could not be found in the literature. However, a diffusion coefficient ($D_{\rm gly}$) of 0.47 × 10⁻⁹ m²/s was found in osmotically dehydrated beef at 30C (Favetto *et al.* 1981).

It was observed in this work that the temperature influenced the values of the diffusion coefficient of water, salt and glycerol. When the temperature was increased, higher

TABLE 1. DIFFUSION COEFFICIENTS (*D*) AND STATISTICAL PARAMETERS

Temperature (C)	Solute	D (m²/s)	R^2	RMSE	
50	Salt	9.81×10^{-10}	0.979	0.0457	
70		1.30×10^{-09}	0.985	0.0321	
90		1.50×10^{-09}	0.990	0.0256	
50	Glycerol	8.46×10^{-10}	0.972	0.0414	
70		1.05×10^{-09}	0.971	0.0518	
90		1.15×10^{-09}	0.995	0.0159	
50	Water	9.60×10^{-10}	0.994	0.0204	
70		1.16×10^{-09}	0.942	0.0757	
90		1.34×10^{-09}	0.991	0.0228	

RMSE, root mean square error.

values of D were obtained. These results are in agreement with the values obtained by Uribe *et al.* (2011) in jumbo squid, Hashiba *et al.* (2009) in pork meat, and Sarang and Sastry (2007) in vegetable tissue for $D_{\rm salt}$ at different temperatures.

The comparison of experimental and estimated contents of water, glycerol and salt during OD of chub mackerel slices is shown in Fig. 3. It can be seen that all points have a good proximity to the bisector line, indicating the goodness of the predictions. So, the Crank model could be used to explain the mass transfer kinetic during OD, which was also verified by the statistical parameters R^2 and RMSE (Table 1).

The Arrhenius equation (Eq. 9) was applied to study the influence of temperature on the diffusion coefficients. It was observed that D values increased with process temperature following the Arrhenius equation for water loss and solute gain. The R^2 values were 0.977, 0.962 and 0.998 for $D_{\rm salt}$, $D_{\rm glycerol}$ and $D_{\rm water}$, respectively. The $E_{\rm a}$ values were 10.401, 7.532 and 8.139 kJ/mol for salt, glycerol and water, respectively. Favetto *et al.* (1981) reported a similar $E_{\rm a}$ value for

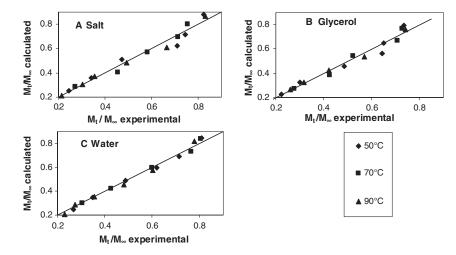


FIG. 3. COMPARISON BETWEEN EXPERIMENTAL AND CRANK ESTIMATED DATA AT DIFFERENT TEMPERATURES

Water Salt Glycerol Temperature (C) Model Parameter 50 70 50 70 90 50 70 90 Z & L Κ 1.230 2 080 2.520 1 140 1.530 1.820 1.010 1.320 1 400 Xec 1.270 1.230 1.180 0.140 0.140 0.150 0.950 0.900 1.010 R^2 0.860 0.980 0.980 0.960 0.970 0.950 0.970 0.980 0.960 **RMSE** 0.054 0.088 0.038 0.055 0.006 0.008 0.006 0.059 0.055 В 0.710 0.710 Weibull 0.680 1 050 0.660 0.810 0.690 0.770 0.680 α 0.730 0.450 0.520 0.940 0.770 0.640 1 190 0.860 0.890 R^2 0.710 0.950 0.940 0.990 0.990 0.970 0.970 0.990 0.970 RMSF 0.058 0.049 0.020 0.022 0.031 0.030 0.019 0 125 0.036

TABLE 2. PARAMETERS OF ZUGARRAMURDI & LUPÍN (Z & L) AND WEIBULL MODELS

RMSE, root mean square error.

glycerol diffusion in beef. E_a value for salt diffusion is in agreement with that reported by Favetto *et al.* (1981) in beef and Del Valle and Nickerson (1967) in sward fish. Nevertheless, this value is lower than that reported by Uribe *et al.* (2011) in jumbo squid.

Z & L and Weibull Models

The parameters of the Z & L and Weibull models are presented in Table 2.

It was observed that the specific rate constant (*K*) of the Z & L model increased with higher temperature in all cases. This could be due to the increase of the driving force for both water and solutes and also to the decrease of brine viscosity at higher temperatures, which affects the external mass transport rate. Similar results were reported by Corzo and Bracho (2005) in sardine sheets.

The shape parameter (β) of Weibull model did not show a clear pattern with respect to process temperature neither for salt and glycerol nor for water. The scale parameter (α)

for salt, glycerol and water showed a decrease with increasing temperatures. Similar results were obtained by Uribe *et al.* (2011) during the OD of jumbo squid and by Schmidt *et al.* (2009) during OD of chicken breast with 5% NaCl.

The K and α parameters increased with increasing temperatures following an Arrhenius relationship, according to the high R^2 values obtained (0.89 < R^2 < 0.99).

The E_a values for salt, glycerol and water obtained with the Z & L model were 11.44, 8.05 and 17.62 kJ/mol, respectively. Meanwhile, the E_a for salt, glycerol and water estimated by Weibull model were 9.36, 7.15 and 8.25 kJ/mol, respectively. Based on these results, the K and α parameters could be considered perhaps pseudo-diffusivity coefficients (Lemus-Mondaca *et al.* 2009a).

Water, glycerol and salt values calculated with Z & L and Weibull models were very close to the experimental values (Figs. 4 and 5), both models being able to predict water loss and solute gain during OD of chub mackerel slices. This can also be verified through the high correlation coefficients

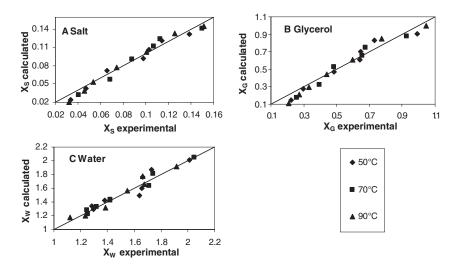


FIG. 4. COMPARISON BETWEEN
EXPERIMENTAL AND ZUGARRAMURDI &
LUPÍN (Z & L) ESTIMATED DATA AT DIFFERENT
TEMPERATURES

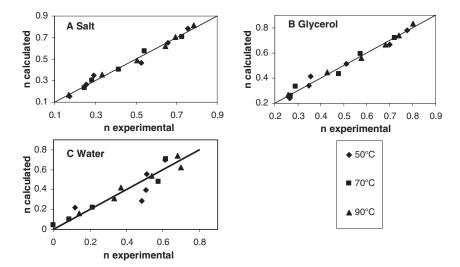


FIG. 5. COMPARISON BETWEEN EXPERIMENTAL AND WEIBULL ESTIMATED DATA AT DIFFERENT TEMPERATURES

and low RMSE (Table 2). The only exception was that of the Weibull model for water at 50C. This model did not predict the experimental values so good ($R^2 = 0.71$ and RMSE = 0.125).

Table 3 shows the equilibrium values obtained experimentally and by the Z & L model. These results were compared with the Peleg equilibrium values obtained by Checmarev et al. (2011) during the OD of chub mackerel slices with the same infusion solutions and temperatures than the ones used in this work. The water loss equilibrium values for longer immersion times estimated from Peleg model were in all cases smaller than the experimental values. The Peleg model tended to underestimate the water loss. Schmidt et al. (2009) reported similar results during the OD of chicken breast. However, salt and glycerol equilibrium values obtained with Peleg model for longer immersion times were higher in comparison with the experimental values. These results indicated that Peleg model also overestimated salt and glycerol gain. The Z & L model gave more accurate estimations than the Peleg model and was able to satisfactorily predict equilibrium values of water loss and solute gain.

CONCLUSIONS

This study demonstrated that higher process temperatures (between 50 and 90C) promoted higher water loss and solute gain in osmotic dehydrated chub mackerel slices. As a result, the time required to reach a determined $a_{\rm w}$ value decreased as the process temperature increased.

The diffusion coefficients (D) and the Z & L and Weibull parameters $(K \text{ and } \alpha)$ all showed a dependence on temperature following an Arrhenius relationship.

The mass transfer kinetics of water, salt and glycerol during the OD of chub mackerel slices were adequately adjusted, with similar accuracy, by the Crank, Z & L, and Weibull models. However, the Zugarramurdi & Lupín model was preferred because it also allowed the prediction of water and solids contents at equilibrium, which is relevant for the development of this kind of products.

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TABLE 3. EQUILIBRIUM VALUES

		Equilibrium values								
		Water		Salt		Glycerol				
	Reference	50C	70C	90C	50C	70C	90C	50C	70C	90C
Experimental	This work	1.295	1.251	1.123	0.138	0.150	0.152	0.993	0.926	1.046
Z & L	This work	1.275	1.226	1.178	0.136	0.143	0.146	0.954	0.898	1.011
Peleg	Checmarev et al. (2011)	1.155	1.072	1.056	0.175	0.173	0.176	1.245	1.100	1.252

Z & L, Zugarramurdi & Lupín.

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