Original

Saline distribution during multicomponent salting in pre-cooked quail eggs

Distribuição salina durante a salga multicomponente de ovos de codorna pré-cozidos

Dionísio BORSATO[1]*, Mariete Barbosa MOREIRA[1], Ivanira MOREIRA[1], Marcos Vinicios Roberto PINA[1], Rui Sergio dos Santos Ferreira da SILVA[2], Evandro BONA[3]

Abstract

The relationship of NaCl with problems of arterial hypertension has led to a reduction in the levels of this salt in food production. KCl has been used as a partial substitute for NaCl since it cannot be completely substituted without affecting the acceptability of the end product. In this study, the diffusion that occurs during quail egg salting in static and stirred brine was simulated. The mathematical model used was based on a generalization of the Fick's 2nd law, and the COMSOL Multiphysics software was used to simulate the diffusion in the NaCl-KCl-water system. The deviations in the simulated data and experimental data were 2.50% for NaCl and 6.98% for KCl in static brine, while in the stirred brine they were 3.48% for NaCl and 4.72% for KCl. The simulation results presented good agreement with the experimental values and validated the predictive capacity of the model.
Keywords: *three-dimensional modeling; finite element method; mass transfer Biot number; effective diffusion coefficients; quail egg.*

Resumo

A relação de NaCl com problemas de hipertensão arterial tem levado a uma redução dos níveis de sal presente na produção de alimentos. O KCl tem sido usado como substituto parcial do NaCl, porque este não pode ser totalmente substituído sem afetar a aceitabilidade do produto final. Neste estudo, foi simulada a difusão que ocorre durante o processo de salga do ovo de codorna em salmoura estática e agitada. O modelo matemático utilizado teve como base uma generalização da segunda lei de Fick. O programa COMSOL Multiphysics foi usado para simular a difusão do sistema água-KCl-NaCl. Os desvios entre os dados simulados e experimentais foram de 2,50% para o NaCl e 6,98% para o KCl em salmoura estática, enquanto na salmoura agitada foram de 3,48% para o NaCl e 4,72% para o KCl. Os resultados da simulação apresentaram boa concordância com os valores experimentais e validando a capacidade preditiva do modelo proposto.
Palavras-chave: *modelagem tridimensional; método de elementos finitos; número de Biot de massa; coeficiente de difusão efetivo; ovo de codorna.*

1 Introduction

Diffusion is the physical process through which mass is transferred under the influence of concentration gradients (VARZAKAS et al., 2005). The need for producing and collecting data in the field of physical properties of food industries, has led food scientists to study the mechanisms of heat and mass transfer. Material diffusion is important in at least three fields of food conversion: introduction or removal of solutes, drying, and aroma retention. Food scientists are also interested in diffusivity in order to understand the influence and preservation on food quality (ROQUES, 1987). Therefore, estimating diffusion coefficients or diffusivity is important for the determination of mass transfer rate (VARZAKAS et al., 2005).

When several solutes diffuse simultaneously a generalized Fick's law can be assumed (CUSSLER, 1976), and in this case, in addition to the main diffusion coefficient of each solute, the cross-diffusion coefficient is included to relate the influence of one solute on the flow of another solute (ONSAGER, 1945).

Fick's equation establishes a relationship between the component flow and the concentration gradients (GHEZ, 1988; RAOULT-WACK, 1994). The ideas and theories on diffusion are well-established (CRANK, 1975; GEANKOPLIS, 1972). Varzakas et al. (2005) reviewed comprehensively the determination of solute effective diffusivities in foods.

New computers and softwares have enabled more realistic modeling of mass transfer in food processes (WELTI-CHANES; VERGARA-BALDERAS; BERMÚDEZ-AGUIRRE, 2005; BONA et al., 2007). These advances also enable the application of numerical techniques such as the Finite Element Method (FEM). The FEM is a set of efficient techniques to obtain numerical solutions of differential equations originating in the most varied fields of science and especially in problems of engineering, physics and chemistry (CHUNG, 1978; ZIENKIEWICZ; MORGAN, 1983). According to Puri and Anantheswaran (1993), the main advantages of FEM are as follow: the spatial variation in material properties can be easily manipulated, irregular regions can be modeled with great precision, it is the best for nonlinear problems, the dimensions of the elements can be easily altered, the spatial interpretation is very realistic,

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[1] Chemistry Department, State University of Londrina – UEL, PO Box 6001, CEP 86051-990, Londrina, PR, Brazil, e-mail: dborsato@uel.br
[2] Food Science Department, State University of Londrina – UEL, PO Box 6001,CEP 86051-990, Londrina, PR, Brazil
[3] Food Engineering Department, Federal University of Technology – UTFPR, PO Box 271, CEP 87301-006, Campo Mourão, PR, Brazil
**Corresponding author*

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and problems with the most diverse boundary conditions can be easily studied.

Sodium chloride plays a fundamental role in many foods currently consumed and, because of this, its complete substitution is not advisable (LYNCH, 1987). Examples of its importance in the food industry are conserved products, in which sodium chloride is responsible for flavour enhancement in addition to promoting chemical and nutritional changes; it has an antimicrobial function (BIDLAS; LAMBERT, 2008). However, NaCl -hypertension relationship has led to a reduction in the levels of this salt in food production (JAMESON, 1986).

Potassium chloride (KCl) has been used together with NaCl during salting since sodium chloride cannot be eliminated or substituted in great quantity without affecting the acceptability of the end product. A diet rich in potassium can be considered as a preventive action against hypertension or can even be effective in the treatment of hypertension. A high ingestion of potassium increases sodium excretion by the kidneys resulting in an anti-hypertensive effect (HE; MCGREGOR, 2001).

Furthermore, Bidlas and Lambert (2008) reported that potassium chloride presents a similar antimicrobial action and can partially substitute sodium chloride with this purpose.

Potassium is found in almost all foods of plant and animal origin (banana, orange, grape, chocolate, milk and meat). Potassium deficiency due to inadequate intake is unlikely in healthy individuals. The minimum daily requirement for potassium intake is 1.6 to 2.0 g/day for adults, but higher levels are recommended because of the possible protective effect of potassium against hypertension, and thus more than 4.7 g/day is recommended (AUGUSTO; ALVES; MANNARINO, 1995).

Among the different types of foods essential for a healthy diet, eggs are sources of high quality protein, phosphate, riboflavin, selenium, and vitamins A, B12, D and K (HERRON; FERNANDEZ, 2004).

Recent findings have destroyed an old myth that eggs cause cardiovascular diseases mainly because they contain high amounts of cholesterol (QURESHI et al., 2007). In the human body, cholesterol can be obtained by endogenous production or from foods. In addition to being a structural component of membranes, it is the pre-cursor of the bile acids, steroid hormones and vitamin D. Although it has vital functions, cholesterol has been considered a health concern because of the correlation between increased plasmatic cholesterol levels and the occurrence of arteriosclerosis (STRYER, 1988).

According to Qureshi et al. (2007), eating up to six hen eggs a week does not present this risk. Jiang, Noh and Koo (2001) suggested that the phosphatidylcholine present in eggs sharply reduces cholesterol absorption by the intestine. Even with the ingestion of high amounts of cholesterol, through the ingestion of eggs for example, much of this sterol becomes unavailable for absorption due the presence of phospholipid.

The cholesterol content of quail egg is around 12.0 $mg.g^{-1}$, which is very similar to that found in hen eggs (BRAGAGNOLO; RODRIGUEZ-AMAYA, 2003).

Panda and Singh (1990) demonstrated that brining of cooked and peeled eggs offers an opportunity to improve quail egg marketing. These salted eggs can be consumed without any further processing or for the preparation of food specialties. Salting of quail eggs, for their conservation, is usually done by immersion in saline sodium chloride solution (MURAKAMI; ARIKI, 1998).

Recently, Hashiba, Gocho and Komiyama (2008) presented NaCl concentration profiles for one-dimensional diffusion in precooked egg white, at various temperatures, considering the phenomenon of the Languimir-type sorption. As far as we know, there are no reports of FEM use to simulate multicomponent diffusion in whole and pre-cooked eggs using the Fick generalized equation.

The aim of the present study was to investigate the multicomponent diffusion of the NaCl-KCl-water system in the salting process of quail eggs using FEM and analyze the diffusion mechanisms of the different solutes present. The present study also aimed at determining the sensory difference between conserved quail eggs salted with sodium chloride and eggs prepared with sodium chloride partially substituted for potassium chloride.

2 Materials and methods

2.1 Egg preparation

The quail eggs (*Coturnix coturnix japonica)* used were purchased from Comércio de Produtos Hortigranjeiros Yamada Ltda, Londrina-PR. They were cooked in boiling water for 15 minutes and, after cooling in an icebox, the shells and membranes were removed (MURAKAMI; ARIKI, 1998).

2.2 Brine

The brines were prepared with a saline concentration of approximately 3% ($m.V^{-1}$), in which the quantity of salt was divided into portions of 30% (0.90 $g.100\ mL^{-1}$) of potassium chloride and 70% (2.1 $g.100\ mL^{-1}$) of sodium chloride. In order to guarantee a constant brine concentration throughout the salting process, the volume used was approximately 10 times greater than the volume occupied by the eggs (GERLA; RUBIOLO, 2003). Before beginning each brining, a sample of the initial brine was removed to quantify the salts. Static and stirred brines were used. A pump with a circulation flow of 500 L/hour was used for agitation. For the sensory evaluation, a brine solution containing sodium chloride only, at concentration of 3% ($m.V^{-1}$), was used as a control sample. The eggs were placed in 500 mL recipients containing the brine at pH = 4.3 adjusted with 0.5% citric acid and then further heated for 30 minutes in a boiling water bath. They were hermetically sealed for 15 days until sensory analysis (MURAKAMI; ARIKI, 1998).

2.3 Sampling and moisture determination

After removing the shells and membranes, the whole pre-cooked eggs were completely immersed in the brine. The samples were collected at intervals of 120 hours. In each sampling, two eggs were randomly removed and left on a plastic plate at 20 ± 1 °C and dried on filter paper to remove adhering brine. Each sample was analyzed separately. Measurements were taken using a pachimeter and an analytical balance. The eggs were then cut in half and taken to dry in a stove, at 105 °C, to determine the moisture (RICHARDSON, 1985; ASSOCIATION…, 1984).

2.4 Sodium and potassium

Sodium and potassium content was determined by atomic emission (flame photometry) using a Celm FC-280 flame photometer after incinerating the samples and extracting the salts with HCl 0.5 mol.L^{-1} (ASSOCIATION…,1984).

2.5 Sensory analysis

A microbiological analysis using eggs and the brine mixed together in a blender was conducted before the sensory analysis. The count of yeasts and molds was performed in a potato dextrose agar medium (PDA, Quimiolab). The total mesophiles content was determined using the standard plate count agar method (PCA, Laborclin).

The sensory analysis was performed using the triangle difference test to measure the specific effects by simple discrimination indicating whether the samples were equal (DUTCOSKY, 2007). The test was carried out by a team of 51 tasters, consisting of undergraduate students, graduate students, and staff of the Chemistry Department at the State University of Londrina, in an illuminated environment.

After a short orientation session, each judge was presented with a series of three codified samples, and they informed that two samples were the same and one was different. Each judge was asked to identify the different sample in each group. For this, always beginning from the left to the right and washing their mouth between each tasting with room temperature mineral water, with a pen, the judges circled on the answer sheet the respective number of the egg sample they found to be different. The number of correct responses necessary to establish significant differences was based on the chi-square test (DUTCOSKY, 2007).

The research protocol CONEP 268 nº 041/08 CAAE 0012.0.268.000.08 was approved by the ethics committee on research involving humans.

2.6 Multicomponent diffusion model

This study considered the simultaneous three-dimensional mass transfer of two solutes in an egg that occupied a volume of $\Omega \subset R^3$ associated with a system of Cartesian coordinates *x*, *y*, *z* with the origin located at the geometric center of the egg. The process occurs under approximately isothermal conditions, and the sample contraction was negligible. Under these conditions, multicomponent diffusion during salting at a constant temperature of 20 ± 1 °C was modeled.

The $C_1(x, y, z, t)$ and $C_2(x, y, z, t)$ are the concentrations of the solutes NaCl and KCl, respectively, defined at point $P \equiv (x, y, z) \in \Omega$, at instant t, and they can be described by the Onsager equations (1945) for the solute concentrations:

$$\begin{aligned}\frac{\partial C_1}{\partial t} &= D_{11}\nabla^2 C_1 + D_{12}\nabla^2 C_2 \\ \frac{\partial C_2}{\partial t} &= D_{21}\nabla^2 C_1 + D_{22}\nabla^2 C_2\end{aligned} \quad (1)$$

where $\nabla^2(.) = \nabla \times \nabla(.)$ is the Laplacian operator and D_{ii} are the main diffusion coefficients and D_{ii} are the crossed diffusion coefficients that combine the flows.

The initial condition is represented by Equation 2, where $C_{1,0}$ e $C_{2,0}$ are the initial solute concentrations of NaCl and KCl in eggs.

$$\begin{aligned}C_1(x,y,z,0) &= C_{1,0} \\ C_2(x,y,z,0) &= C_{2,0}\end{aligned} \qquad x,y,z \in \Omega \quad (2)$$

The boundary conditions used for the salting process in well-stirred brine are, in mathematical terms called Dirichlet boundary conditions and are given by Equation 3, where $\partial\Omega$ is the set of surface points of the quail egg and $C_{1,s}$ and $C_{2,s}$ are the solute concentrations in the brine.

$$\begin{aligned}C_1(x,y,z,t) &= C_{1,S} \\ C_2(x,y,z,t) &= C_{2,S}\end{aligned} \qquad x,y,z \in \partial\Omega, \ \text{e} \ t \geq 0 \quad (3)$$

The Cauchy boundary condition for static brine was (LUNA; BRESSAN, 1986):

$$\begin{aligned}\frac{\partial C_1(\pm R,t)}{\partial \eta} &= \frac{h_m}{\lambda_m}\left[C_1 - C_{1,s}\right] \\ \frac{\partial C_2(\pm R,t)}{\partial \eta} &= \frac{h_m}{\lambda_m}\left[C_2 - C_{2,s}\right]\end{aligned} \qquad \text{with } x,y,z \in \partial\Omega, \ t > 0 \quad (4)$$

where h_m (kg.m^{-2}/seconds) is the mass transfer coefficient; λ_m (kg.m^{-1}/seconds) is the mass conductivity; and $\partial/\partial\eta$ is the normal derivative operator.

The h_m and λ_m coefficients are related to Biot mass-exchange number by

$$Bi = \frac{h_m \times R_i}{\lambda_m} \qquad for\ i = 1,2,3 \quad (5)$$

where R_i is the characteristic length (m).

2.7 Diffusion simulation

The COMSOL Multiphysics software installed in a Core 2 Duo computer with 2 Ghz memory was used to simulate the multicomponent saline diffusion process. The solution of the equation system formed by Equations 1 and 2 and the selected boundary condition was obtained by the conjugate gradient method. The starting point for the Finite Element Method is a mesh, a partition of the domain into small elements of a simple shape. The COMSOL Multiphysics automatically generated an unstructured mesh containing tetrahedral elements (COMSOL MULTIPHYSICS, 2005).

2.8 Statistical test

The estimated and experimental data of salting were compared by the percentage of deviation (BONA et al., 2007).

$$\%deviation = 100\sqrt{\sum_{k=1}^{N}\left[\left(\frac{\bar{C}_{est} - \bar{C}_{\exp}}{\bar{C}_{\exp}}\right)_k\right]^2 \frac{1}{N}} \quad (6)$$

where $\bar{C}_{est}$ = mean concentration estimated by the numerical solution; $\bar{C}_{\exp}$ = experimental mean; and N = number of observations considered.

2.9 Fit of the diffusion coefficients and Biot number

The effective diffusion coefficients and Biot number were fitted by the Simplex optimization method (WALTERS et al., 1999) associated with the desirability functions (DERRINGER; SUICH, 1980). The upper and lower limits of the parameters used are shown in Table 1. The lower limit value was used to start the simplex optimization.

3 Results and discussion

Diffusion during the salting process in static and well-stirred brine was simulated using the Finite Element Method. A tetrahedron standard mesh consisting of 20179 elements and 17138 degrees of freedom was used. Figure 1 shows the mesh, the conventions adopted for the imaginary axis, and the mean dimensions with their standard deviation.

Using the values of the concentrations obtained experimentally, the principal diffusion coefficients (effective), crossed coefficients, and the Biot number were fitted by the simplex optimization method (WALTERS et al., 1999) associated with the Derringer and Suich (1980) desirability functions.

An optimization algorithm (BONA et al., 2000) proposed combinations for the diffusion coefficients, which were assessed by the Finite Element Method. The diffusion coefficients and the Biot number were repeatedly reassessed by the optimization method that gave new value combinations to minimize the deviations among the estimated and experimental values for NaCl and KCl concentrations.

The procedure was repeated until the difference between three consecutive deviations was less than or equal to 10^{-2}. Figure 2 shows this stabilization by applying the simplex method when well-stirred brine was used.

Table 2 shows the main coefficients, crossed coefficients, and the Biot number obtained for the static and stirred brine using the simplex method after 40 iterations.

Similar results for diffusion coefficients were reported by Hashiba, Gocho and Komiyama (2008), when they studied sodium chloride diffusion in pre-cooked egg white. The

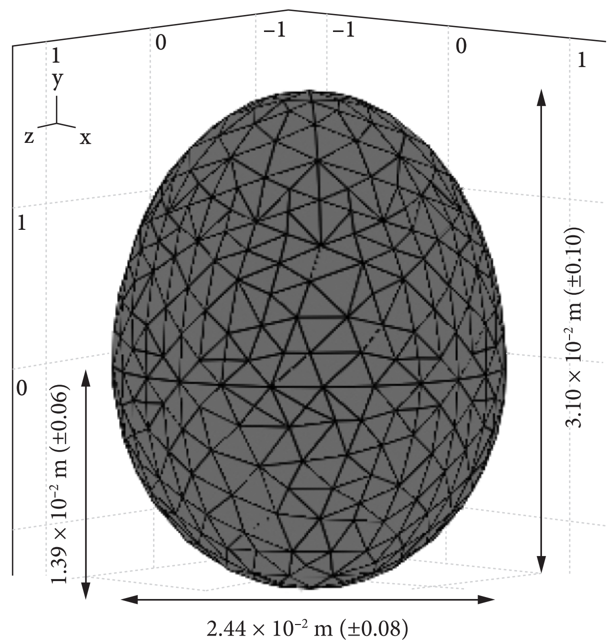

Figure 1. Mean dimensions of the quail eggs used in the simulation; convention adopted for the imaginary axis and the tetrahedron mesh used.

Table 1. Lower limit (L) and upper limit (U) of the diffusion coefficients and Biot number used in the simplex optimization.

	Static brine		Well-stirred brine	
	L	U	L	U
NaCl diffusion coefficient (m^2/s)	6.0×10^{-10}	1.0×10^{-9}	6.0×10^{-10}	1.0×10^{-9}
KCl diffusion coefficient (m^2/s)	1.0×10^{-10}	2.5×10^{-10}	1.0×10^{-10}	2.5×10^{-10}
NaCl crossed coefficient (m^2/s)	1.0×10^{-12}	1.0×10^{-10}	1.0×10^{-12}	1.0×10^{-10}
KCl crossed coefficient (m^2/s)	1.0×10^{-12}	1.0×10^{-10}	1.0×10^{-12}	1.0×10^{-10}
Mass Biot number	1	100	1	200

Table 2. Fitted values for diffusion coefficients, mass Biot number, and percentage of deviation.

	Static brine		Well-stirred brine	
	NaCl	KCl	NaCl	KCl
Main coefficients (m^2/s)	8.047×10^{-10}	1.185×10^{-10}	8.047×10^{-10}	1.185×10^{-10}
Crossed coefficients (m^2/s)	5.787×10^{-11}	5.752×10^{-11}	5.787×10^{-11}	5.752×10^{-11}
h_m/λ_m (m^{-1})	3821		13770	
Mass Biot number	46.61*		167.99*	
% deviation	2.50	6.98	3.48	4.72

*Related to X axis.

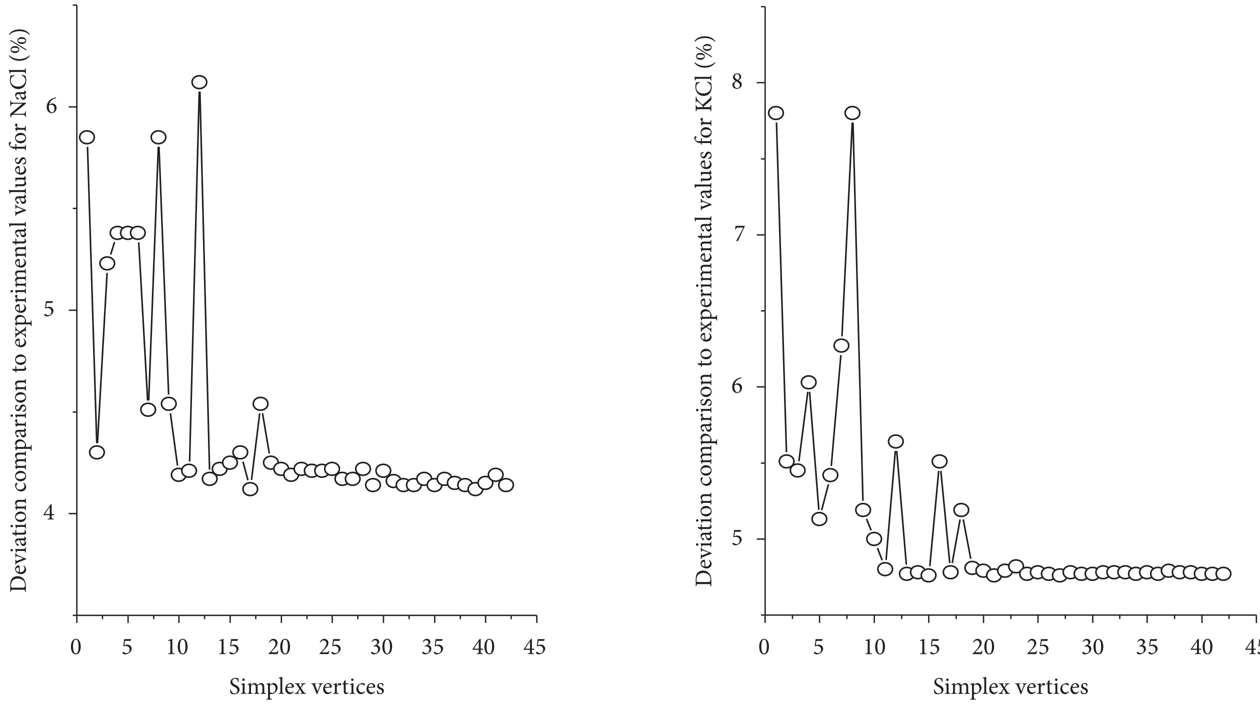

Figure 2. Deviation stability during simplex method application for well-stirred brine.

pre-cooked quail egg presented a large quantity of water (74 g. 100 g^{-1}), distributed in two different heterogeneous phases; one in which the water was bonded to the proteins, and the other phase was dispersed in the solid matrix. The fact that the diffusion coefficient of sodium, observed by Hashiba, Gocho and Komiyama (2008) in pre-cooked egg white is similar to that found in pre-cooked quail egg suggests a similar diffusive behavior to the whole quail egg studied in this work.

The diffusion coefficients of the sodium and potassium ion, in very diluted aqueous solutions, are greater than those verified in food solids (GUINEE, 2004). The egg is characterized by presenting a certain porosity and tortuosity, and it contains a liquid immobilized in a solid protein matrix and, since the diffusion was verified in the water, these factors interfere in salt diffusion resulting in much smaller effective coefficients (GUINEE, 2004; HASHIBA; GOCHO; KOMIYAMA, 2008).

The main sodium diffusion coefficient was greater than that of potassium during multicomponent salting of quail eggs (Table 2). The greater mobility of the sodium and smaller mobility of potassium may be related to the ionic ray because the sodium ionic ray is smaller than that of potassium. However, it is known that the hydrated sodium ray is greater than the hydrated potassium ray (LEE, 1991) suggesting that a smaller interaction of sodium ion, with pre-cooked quail egg components, would be responsible for increased mobility.

The values of the crossed coefficients of sodium were similar to those detected for potassium and were much smaller than the principal coefficients indicating that the diffusion, in relation to the gradient itself, was more important than the interference of a solute in the flow of the other solute.

The value of the Biot number adjusted for the process using well-stirred brine was high (167.99). This indicated that external resistance may be rejected and since the diffusion coefficients do not depend on whether the brine was agitated or not, the values adjusted for well-stirred brine were the same considered in the salt diffusion simulation during salting in static brine (Table 2). In the optimization using the simplex method, the value found for the Biot number in the static brine was 46.61, which indicated that the resistance should be taken into consideration because of the film formed on the egg surface (SCHWARTZBERG; CHAO, 1982). Furthermore, according to Bona et al. (2007), the external resistance may influence mass transference, and therefore the Biot number should be considered.

Figure 3 shows the experimental and simulated average saline concentrations for salting with and without agitation. It can be observed that in the first 15 hours, the salt diffusion in the agitated brine was faster than in the static brine, confirming that the influence of the film formed on the quail egg surface should be considered in static brine. The same figure also shows that the sodium chloride equalization was verified after 25 hours after salting in both brines and that, in the agitated brine, the potassium chloride equalization was verified after 60 hours of salting and after approximately 110 hours in the static brine.

Murakami and Ariki (1998) recommended a period of 15 days of salting (3% solution) before marketing. The simulation during salting showed that the equalization of KCl in quail egg occurs in 5 days.

After simulating 25 hours of salting in a well-stirred brine, using COMSOL Multiphysics, a slice plot showing the distribution profile of sodium chloride and potassium chloride can be seen in Figure 4.

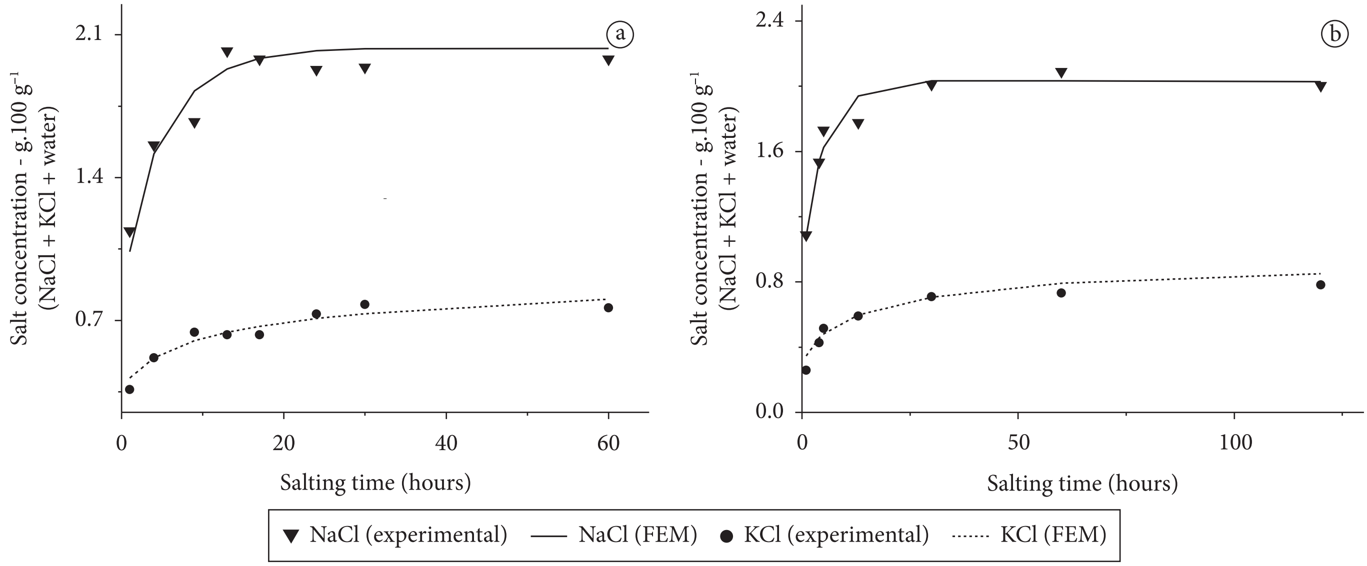

Figure 3. Experimental and FEM estimated saline profiles for NaCl and KCl during salting of quail eggs in well-stirred (a) and at static (b) brine.

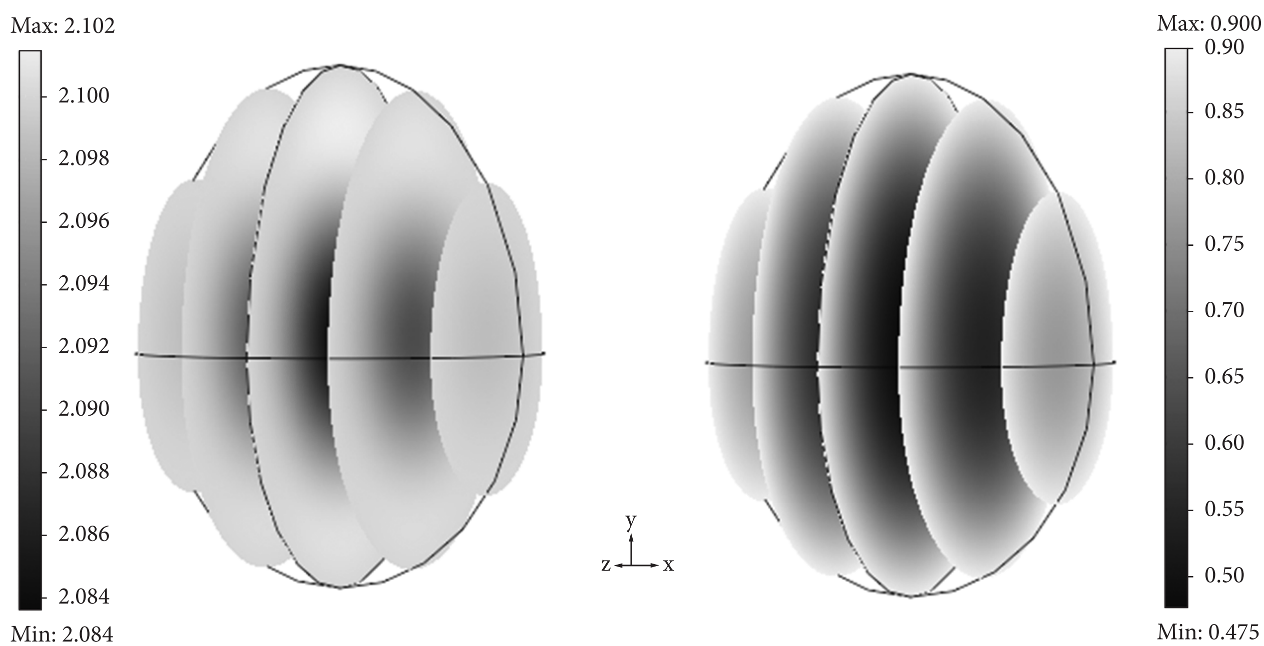

Figure 4. Saline profile of NaCl and KCl after 25 hours in stirred brine.

The centre of the egg (darker color) should represent a NaCl concentration equal to 2.08 g.100 g$^{-1}_{(water+NaCl+KCl)}$, corresponding to 99% of the content of this salt in the brine. However, for KCl, at the centre of the sample (darker color), after the same salting time, its concentration should be 0.50 g.100 g$^{-1}_{(water+NaCl+KCl)}$, value corresponding to 55.5% of the content of this salt in the brine indicating that KCl diffusion was slower, as shown by its principal diffusion coefficient presented in Table 2.

The distribution profile at the end of 25 hours of salting in static brine, simulated by COMSOL, presented similar results to those observed in the diffusion simulation during salting with agitation. However, the KCl value detected at the centre of the sample corresponded only to 44.0% of the value of its concentration in the brine.

Figure 5 shows the KCl saline distribution profile after 100 hours in agitated brine and that its concentration should be equal to 0.82 g.100 g$^{-1}_{(water+NaCl+KCl)}$, which corresponds to 91% of this salt in brine. A similar concentration value was detected in static brine after 110 hours salting. The longer time observed when using static brine was due to the resistance of the superficial film that seems to influence the entry of the potassium ion and delay its equalization.

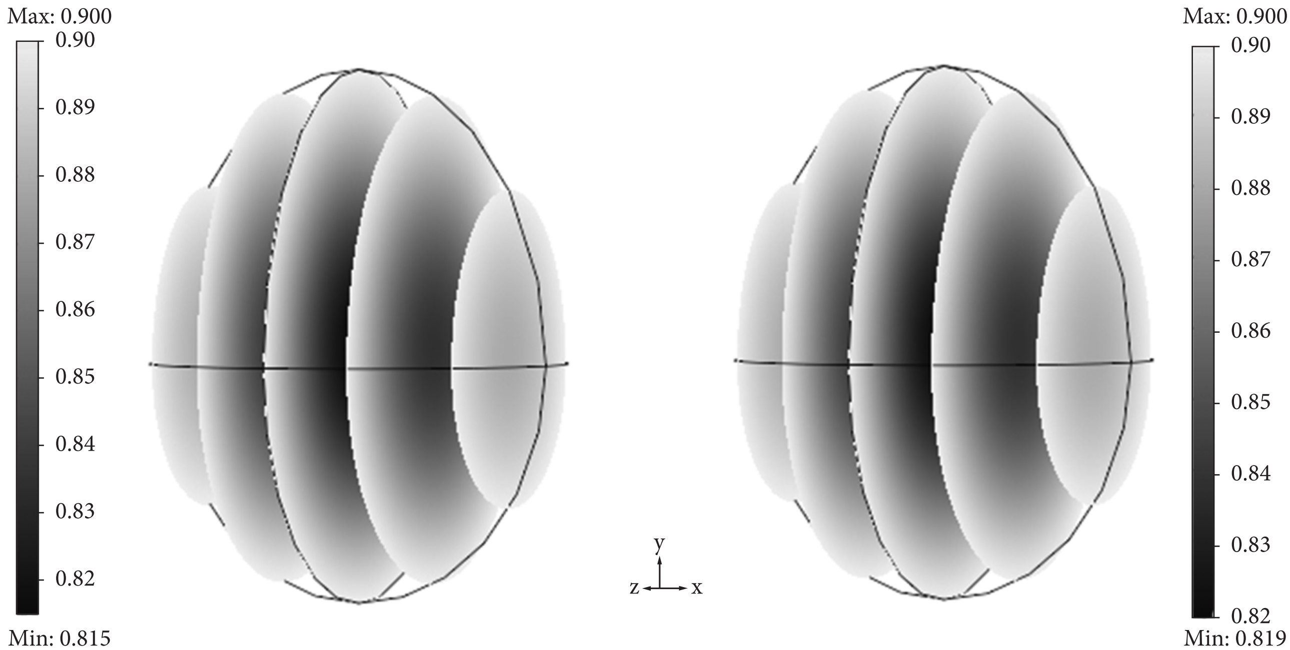

Figure 5. Saline distribution profile of KCl after 100 hours in stirring brine and 110 hours in brine at rest.

Both the experimental data and those obtained by simulation for sodium chloride diffusion showed that the use of agitation did not appreciably reduce the salting time. However, Figure 5 shows that agitation reduced the KCl concentration equalization time in the egg by approximately 10% during salting.

The results of the analysis of yeasts and molds demonstrated that the eggs were in accordance with the value established by legislation, 5.0×10^3 CFU.g^{-1} for this type of foodstuff. The total count of mesophilic bacteria can be used as an indicative of poor product handling, and it depends on the quality of raw material used. The Brazilian food legislation has not established the maximum amount of mesophiles allowed in quail eggs pickles. However, the mesophiles count obtained, 2.5×10^1 CFU.g^{-1} on average, indicate that the eggs could be presented to tasters without risk to health. Since the simulation showed that the equalization of salts occurs in 5 days, microbiological testing started 5 days before the sensory tests.

The triangle test was used to find whether there was significant difference between the eggs salted exclusively with sodium chloride and the eggs salted also with 30% potassium chloride (DUTCOSKY, 2007). Fifty-one judges participated in the analysis, and 22 correct responses were obtained. The table based on the chi-square test (DUTCOSKY, 2007) showed that the minimum number of correct responses to establish a significant difference was 24, and it was concluded that there was no sensory difference among the samples at the level of 5%.

4 Conclusions

The COMSOL allowed simulating the multicomponent diffusion in brines with and without agitation during the salting of quail eggs. The simulated data were reliable and convergent with the experimental results validating the use of the COMSOL for multicomponent diffusion. The deviations calculated were considered acceptable and the diffusion process, under the conditions used, seemed to stabilize in the first 25 hours of salting for NaCl and the first 90 hours for KCl. The system developed to simulate salt diffusion allows the control and modulation of the salt content in quail eggs predicting the final content from the initial conditions.

The sensory test used showed that there was no significant difference, at the level of 5%, among the samples salted with sodium chloride and the quail eggs salted with partial substitution of sodium chloride for potassium chloride.

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