Determination of apparent diffusion coefficient in balls made from haddock mince during brining

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Key words

Salting, fish mince, diffusion coefficient, NaCl

1 Introduction

Salting is one of the oldest methods of food preservation. Sodium chloride concentration influences the properties of food systems, including water holding properties, viscosity, texture, emulsification etc., by influencing the properties of the proteins. In addition, salt is important for taste. The daily intake of salt is around 8 – 11 g/day, which is more than twice the amount estimated as necessary. Since excess of NaCl intake has negative health effects, dietary advice is to reduce the amount of consumed salt.

Industrially prepared foods contribute with 70 – 80% of the daily intake of salt (FSA, 2003; Wheelock and Hobbiss, 1999; WHO, 2006). There is therefore an interest in developing products with reduced salt content , while retaining high quality and safety in the end products. Stricter regulations and more focus on quality and traceability, increase the demand for scientific verification of product claims. Applying scientific methods to document and model food processing operations can give competitive advantages for the manufacturers. Modifications of raw materials and process parameters are used to create new products and optimise existing processes. Studying and modelling technological processes in laboratory conditions (for instance simulation of salt diffusion) may allow controlling food processing, increasing productivity and improving the quality of the products. In salting it is important to understand the distribution and mobility of salt in the product, as these parameters influence the release of ions from the matrix and thereby the salt perception.

Many studies have focused on the diffusion of salt in muscle foods. The effective diffusion is an important mass transfer mechanism, responsible for sodium and chloride transport. Mass transport between solid food and brine is generally controlled by the diffusion rate of the solutes. Diffusion rates are calculated using effective diffusion coefficients of solutes into the solid. Food materials are often irregular in shape and can also present various inner regions of different composition. These aspects give some extra challenges to the mathematical modelling, and various approaches are applied to deal with heterogeneous materials. In particular, description of the geometry is of great importance when the aim of modelling is to obtain internal profiles of temperature, concentration and/or pressure. The geometry, including surface area and volume, as well as (apparent) density influences gradients of physical quantities, especially when irregularly shaped objects are involved. In foods with heterogeneous structure, it is often difficult to estimate the volume into which the solute can penetrate accurately. Tissue composition affects effective diffusion coefficients (D_e) by making the pathway of salt ions longer. A tight network of fibres and proteins, high content of lipids and low water content are also factors reducing diffusivity rate. The physical state of the material affect the conditions of the diffusion process (Cierach and Modzelewska-KapituŁA, 2011; Floury et al., 2009; Puolanne et al., 2001).

Hashiba and co-workers modeled the dual mode of diffusion and sorption of NaCl through the tissue as diffusion through a heterogeneous medium. The muscle is modelled as consisting of two types of bulk water, with different diffusion rate in the two phases. The main part of water in tissue is present in small droplets (1-2 µm), where diffusivity of the ions is approximately the same as in pure water. In the so-called water swollen protein region, where water is bound to protein chains, the diffusivity is considerably reduced, affecting the rate of the diffusion process. However, in their model of salting of different fish, Zugarramurdi and Lupin assumed that the flesh in cut fish only acts as an inert support, and that the influence of the inner membranes is negligible.

Earlier studies have shown that salt diffusivity is affected by the NaCl concentration because the NaCl concentration affects the microstructure of muscle. Knowledge on how salt affects the muscle microstructure is needed both to model diffusion and to obtain the desired salt concentration in the product. At low salt concentrations the tissue gains water (salting in), maximum water uptake in muscle is observed at NaCl concentration of 5-6%, while higher salt concentrations (above 9-10%) gradually lead to dehydration. The increase in water holding capacity is related to an expansion of the myofibrillar network and this is coupled to protein solubility. Increase in water binding in salted muscle foods is attributed to enhanced electrostatic repulsion, leading to swelling of the proteins, and a more open network capable of retaining more water. A tight network of fibres and proteins, high content of lipids and low water content are also factors reducing diffusivity rate.

From a physical point of view, curing of meat depends on mass exchange by diffusion and osmosis, where the driving force is the chemical potential of the substances involved in the process, for instance, study of diffusion in gels helps to extract information on polymer network structures, to predict transport by diffusion. Absorption and migration rates of salt ions depend not only on external factors including curing method, time, temperature, brine concentration, and pH, but also as explained above on internal factors such as chemical composition and the biochemical state of the muscle, the microstructure, direction of muscle fibers, viscoelastic properties, and how the water is distributed in the muscle.

The size and shape of the penetrating ions and the diameter of their hydration coats also affect the process of diffusion. According to various studies, D_e for Cl^- in water is higher than for Na^+, therefore chloride ions in the NaCl solutions should diffuse faster than the sodium ions. This would lead to a charge gradient in the solution, due to the slower movement of the chloride ions while the sodium ions move more rapidly in the medium. Effective diffusion coefficient for NaCl in infinite dilution is close to the average of D_e for Cl^- and Na^+.

The large number of factors that influence the diffusion rate in foods have led to a wide range of different measured values for diffusion coefficients. The choice of the system used to determine diffusion is crucial, since there is no method suitable for all purposes. Data on salt diffusivity in different food matrices are available in literature.

A key prerequisite for modelling mass transfer is the knowledge of the boundary conditions. To estimate salting time, it is necessary to take into account the nature of the ions used in the process, their diffusivity, temperature and concentration.

The aim of this study was to develop a simple model system to determine diffusion coefficients during controlled brining of minced foods, and to use this model to study the effect of freezing on fresh and chilled raw materials, salt concentration and stirring on the diffusion rate. Fish balls are traditional products in Norway, therefore they were chosen for the research. We chose gelled fish balls to have a model system where mathematical equations existed, and also we wanted to avoid the problem of fiber directions.

2 Material and methods

2.1 Modeling diffusion of salt in the fish ball model

Two different models of meat or fish tissue structure are usually employed when considering transport mechanisms:

1. Muscle is a network of permeable membranes composed of large organic molecules, thus the transport mechanism may be described by Fick's laws of diffusion;

2. Muscle consists of impermeable barriers forming a complex capillary system, thus salt can be transported by diffusion through the water inside the capillaries.

Minced meat and fish has a complex structure. However the whole process is usually considered as molecular diffusion and therefore concentration profile method has often been used for determination of diffusivity. The method based on the exact solution of the Fick equation for a semi-infinite medium has been used by Doulia et al. (1993).

The effective diffusion properties is an important mass transfer mechanism responsible for sodium and chloride transport. In steady state conditions the flux per unit of time and area is proportional to the concentration gradient. In non-stationary conditions, the mass balance can be modeled by an extension of Fick's second law:

$$\frac{\partial c}{\partial \tau} = D \frac{\partial^2 c}{\partial x^2} , \qquad \text{(Eq.1)}$$

where the input must be equal to the output plus accumulation in the system. D is the salt diffusivity (m^2/s), $\frac{\partial c}{\partial \tau}$ is the concentration change, depending on time of treatment (kg/m^3 / h), x is the space coordinate.

At constant temperature conditions, salt diffusion depends on brining time. At the end of the process the system is approaching equilibrium between the salt content in the ball and in the brine. However, heterogeneities in the ball prevent a full equilibrium. The equilibrium for NaCl in the system will be:

Concentration of salt in the brine = Concentration of salt in water phase in the meat ball

In an inhomogeneous system with pores, fibers and layers, molecular diffusion will be different from the one described by the Fick's law, influencing the effective diffusivity coefficient D_e (m^2/s).

Brine and solid food are normally modeled as a pseudo-binary system (solute – tissue). However, due to the complex nature of the food matrix, and its strongly heterogeneous structure, precise analytical solutions for binary systems cannot always be applied.

The ball is an environment with characteristics different from the brine: in general, they have different density, viscosity, and structure. The activation energy of diffusion varies considerably and is closely correlated with water content. The temperature was kept constant in this experiment. The process can be described as a non-steady-state diffusion from a well-stirred solution of limited volume. The fish balls have a structure formed by fish muscle filaments and network of alginate and calcium. The ball was placed into the brine with a desired constant concentration of salt, continuously in contact with the surface of the ball and slowly diffusing into the ball.

In colloid chemistry a brine is defined as a solution of an electrolyte, that is dissociated ions of Na^+ and Cl^-, dispersed in the diluter, H_2O, which is not dissociated. Intact muscle food such as meat or fish without grinding pretreatment is a capillary, porous and colloidal solid. However, the transport of salt ions by convection (mixing process) is faster than transport by diffusion. In the salting process balls are typically surrounded by a thin layer of stagnant liquid. The thickness of this layer depends on the movement of the solution (brine) and the properties of the surface of the balls. To study how the stagnant layer affected diffusion, experiments were carried out both in dynamic and static conditions. Salt concentration near the surface was assumed to be constant. The sphere was initially free from solute. Transport of salt and water is governed by various mechanisms difficult to study separately: the whole process was therefore modeled as molecular diffusion, and can be described by Fick's law.

Mathematical models can give a better understanding of transport phenomena, and a better control of process variables such as immersion times and salt concentrations. Modeling diffusion as a simple Fick's model is usually not possible because the diffusion coefficient is affected by salt concentration, swelling and heterogeneous movement of water in the system. Graiver therefore suggested a model where Fick's law is used for modeling the diffusion and "convective" terms have been included to model the global flux of brine due to electrostatic forces.

Mass transfer of salt into a meat ball in brine is described by Graiver et al. (2006). Provided that D_e is constant, the concentration in the brine does not change significantly in the salting process, the volume of brine is large compared to the volume of the ball, the brine is well mixed with no concentration gradients, and the surface concentration increases instantaneously to the concentration in the brine (C_0), the amount of diffusing substance entering or leaving the sphere is given by:

$$\frac{M(t)}{M_\infty} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp(-D_e n^2 \frac{\pi^2 t}{a^2}) , \quad \text{(Eq. 2)}$$

where **M(t)** (kg) is the mass of diffusive substance (salt) that enters the ball during the time **t** (s), M_∞ is the maximum amount of solute that could enter at infinite time and corresponds to the mass in equilibrium, **a** (m) is the radius of the sphere, $\mathbf{D_e}$ (m^2/s) is the effective diffusivity and **t** is the salting time.

Coefficients of diffusion could be different for various kinds of materials and treatment methods, and it is therefore important to increase the knowledge on the relationship between the properties of the raw material and the diffusivity.

2.2 Materials

Haddock (*Melanogrammus aeglefinus*) fillets were bought in a local fish store in February 2013. The fish was one day old when bought and had been stored at 0-2°C on ice. One half of the fillets was frozen and stored at -20°C for 8 days; the other half were used to prepare mince as described below. Before preparing mince from frozen fillets, the fillets were thawed in a cold room (+ 4°C) over night.

Before preparation of mince, skin and bones were removed from the fillets. The fillets were then minced in a food processor at low speed regime. During the mincing, sodium alginate, Protanal LF 10/60S12727 G 0.65 (1% to raw material) supplied by FMC Biopolymer, calcium carbonate ($CaCO_3$ precipitate, Merck) (0.25% to raw material), Glucono -δ- Lactone (0.89% to raw material) and distilled water (20 g) were added as shown in Figure 1.

Figure 1. In here

Mince and balls were prepared in a cold room (+4ºC) and kept at this temperature over night for gel stabilization.

Design of the experiment

Conditions of the experiment were chosen based on results of preliminary experiments, (results not published).

A special system was designed for the experiment (Figure 2). Three centrifuge tubes (volume 50 ml) were connected parallel to two plastic bottles (volume 0.5 l) from both sides. The balls had a diameter of: 1.5 cm and mass – 5.5 ± 0.6 g., corresponding to the size of the tubes. The brine circulation was driven by peristaltic pump Masterflex, easy-load, model 7518-00; processing speed was 550 rpm.

Figure 2. in here

The whole system was closed, with a constant brine volume of 1 litre, without air, to minimize the factors affecting the diffusion. The temperature was kept constant (T = 26ºC) and three different salt concentrations (3, 5, 10%) were used. The brine was made by dissolving sodium chloride (NaCl pro analysis, GMB Germany) in distilled water.

The system was stirred to get a constant solute concentration by circulation pumping, or kept at static conditions in the laboratory glass. At different time steps (2.5, 5.0, 7.5 hours), three balls were removed from the brine and the salt concentration was determined.

Analytical methods

The sodium chloride content was determined with the Volhard method.

The numerical solution of the coefficient of diffusivity was developed using Scilab software based on the Equation 2. Coefficients of determination (R^2) for models were in range 0.867 – 0.999, except the one ("frozen static 5%".) with R^2 = 0.787. Curve fitting was done using Scilab *datafit* function utilizing non-linear optimization routine based on quasi-Newton BFGS algorithm. This model is useful for calculating the concentration distribution in the early stages of diffusion, since the series (n) converges rapidly and two or three terms give sufficient accuracy for most practical purposes.

3 Results and discussion

3.1 Effective diffusion coefficient of NaCl in the ball, model and numerical solution

The solution of Equation 2 was calculated by Scilab – 5.4.1., with graphic results shown in Figure 3a, b, c; the diffusion coefficients are presented in Tables 1 and 2.

Figure 3. in here

Numerical simulations of the diffusion rate, parametrized on our theoretical model, show good agreement with experimental results (Figure 3), especially with higher solute concentration and/or active circulation. For the brine concentration of 10%, the deviation of the experimental result from the theoretical curve was negligible. The sampling time was determined by previous trial experiment (results not published) and by the results of , who found that salt concentration increased in the first 2 hours of brining.

Nguyen and co-workers found that, with increasing salt concentration in the brine, water exudation and salt diffusion increased and weight gain decreased. The highest process yield was found in 6% brine. The highest effective diffusion value was found with 15% brine, while brines at 18% and 24% had identical effective diffusion coefficients.

This is in agreement with (Gallart-Jornet et al., 2007), where it was found that pretreatment (chilling, freezing or superchilling) of the raw material influence the salting time needed to reach a certain concentration of salt.

Table 1. In here

Diffusion coefficient of the balls made from frozen material was nearly twice higher during dynamic treatment than during static treatment. For the balls made from fresh raw material, with the exception of brining at 5% with stirring, the diffusion coefficients were found to be higher when brining conditions were more turbulent compared to for static conditions.

Generally results show correlation between diffusion coefficient and the material of the balls and salting time, which is in agreement with the results of Costa-Corredor et al. (2010), Gallart-Jornet et al. (2007b); Hansen et al. (2008). Diffusion coefficient of the balls made from frozen material was lower than the ones for balls made from fresh material, with the exception of brining at 3% and 5% during dynamic treatment. This may be due to a lower water holding capacity caused by protein denaturation during freezing. It is difficult to compare the results of this experiment with results from the studies reported in the literature because different salting methods have been used for determination of the diffusion coefficients. Different authors have determined effective diffusion coefficients in different food systems, such as fish and pork tissue and variable results were reported (Table 2).

Table 2. In here

4 Conclusion

Distribution and mobility of salt in the food product is an important mass transfer mechanism, which can be studied by determination of apparent diffusion. The diffusion coefficients (D_e) for the fish balls brined at different stirring conditions and made from both fresh and frozen raw material were determined. A numerical solution for the coefficients of Fick's second law was proposed. Critical experimental parameters for the estimation of diffusion coefficients were found to be the size of balls, the initial solute concentration in the bulk of brine and the state of raw material. The stirring process increased the transport of salt into balls as it was expected. The results of experiments may be used in further studies of modelling of salt diffusion.

Acknowledgements

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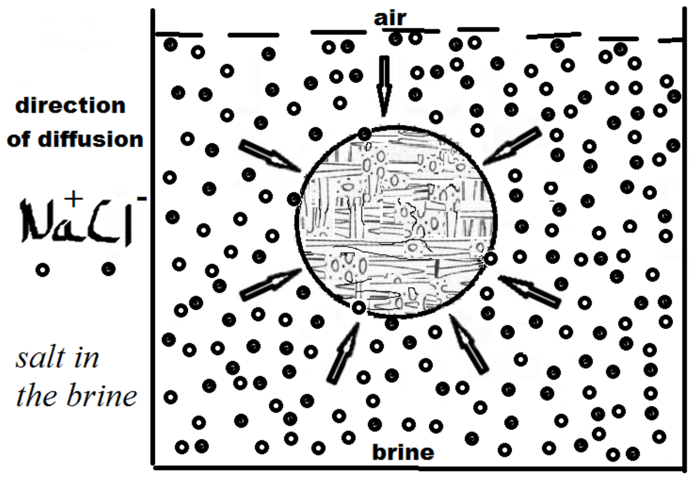


Figure 1.

NaCl diffusion from brine to a fish/meat ball. The direction of diffusion into the sphere is perpendicular to the surface.

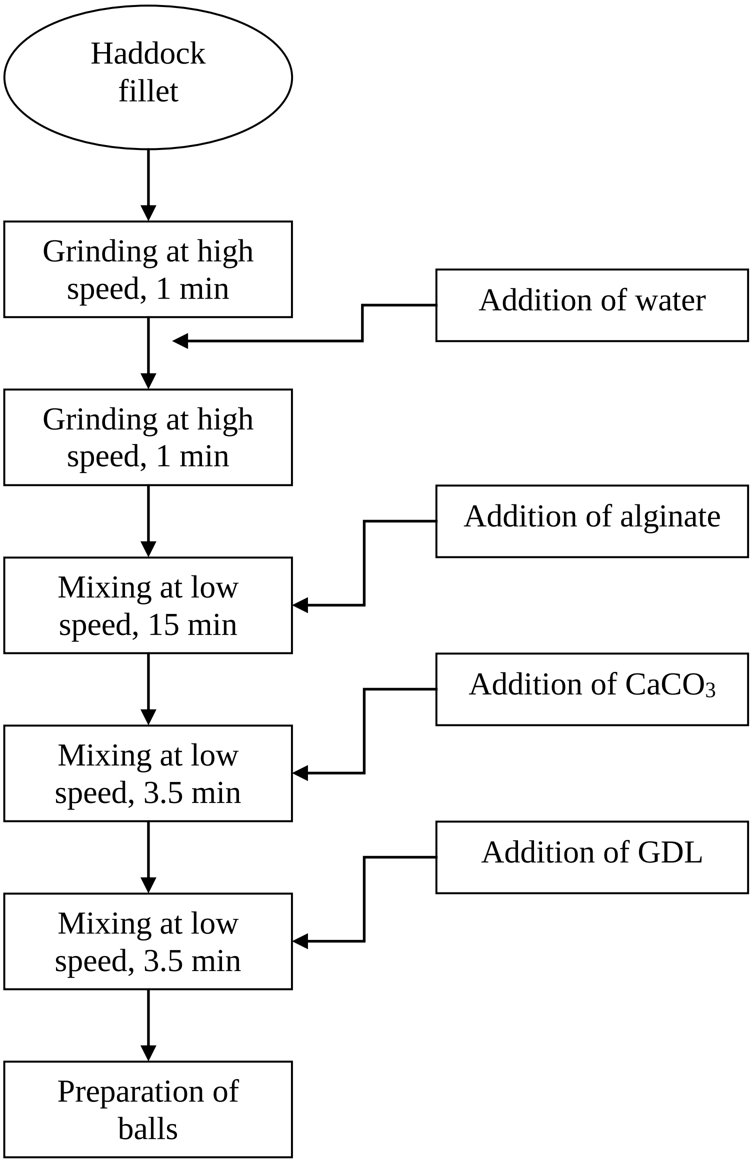


Figure 2. Preparation of mince for fish balls.

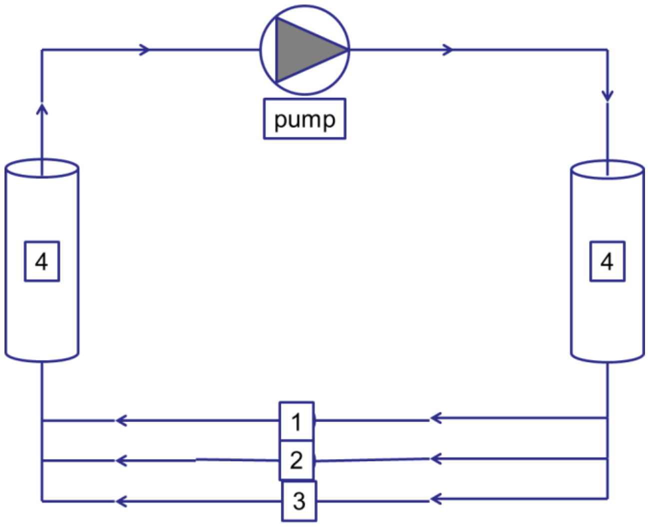
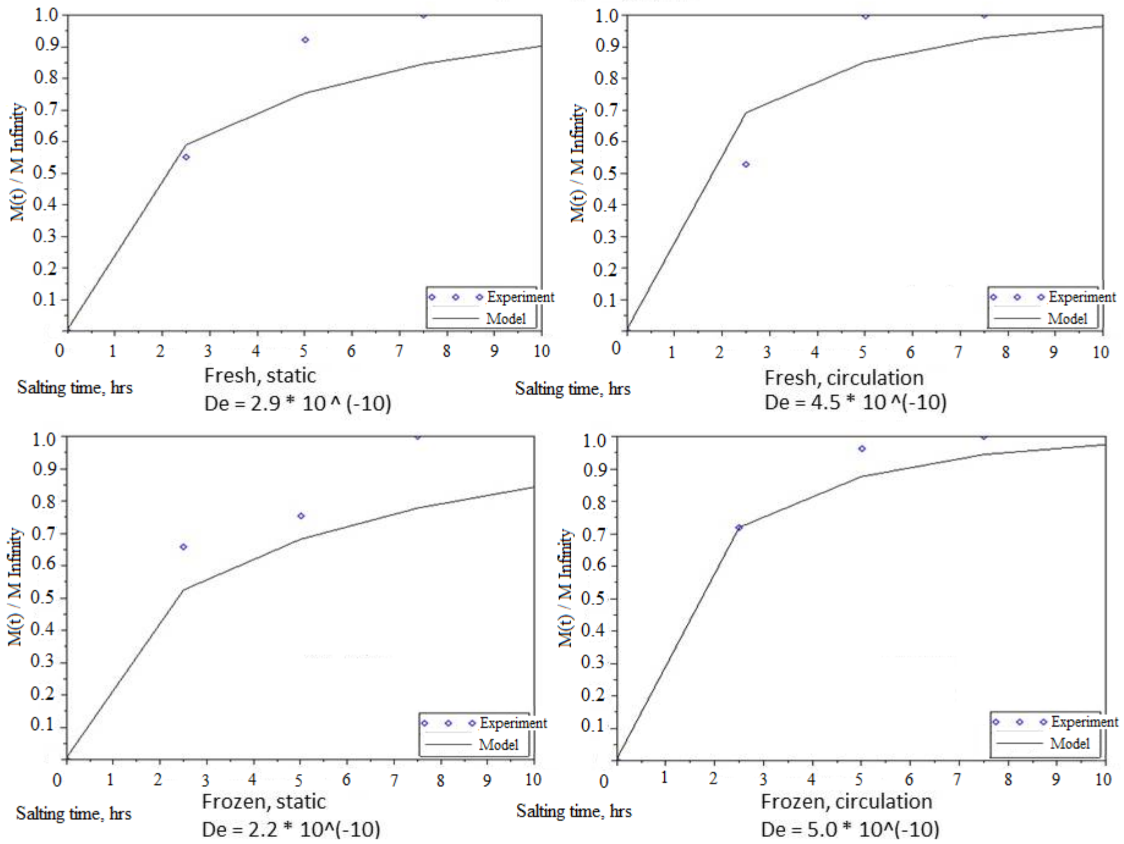
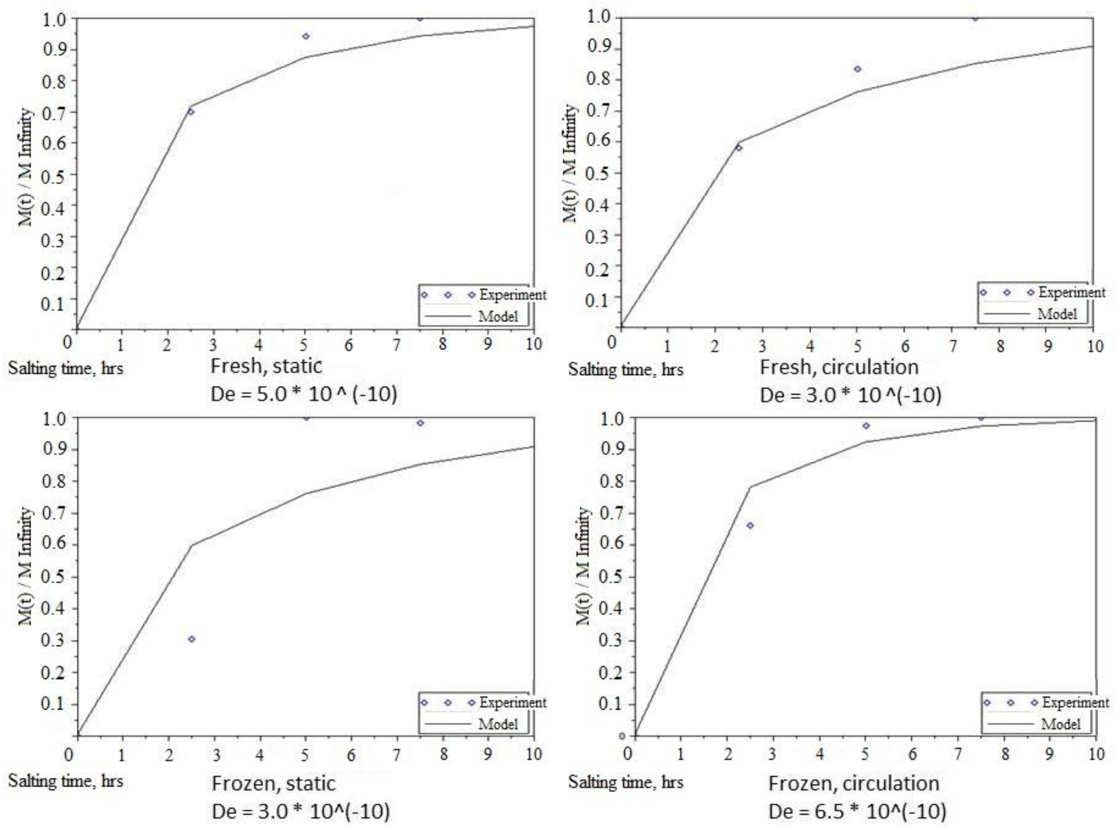


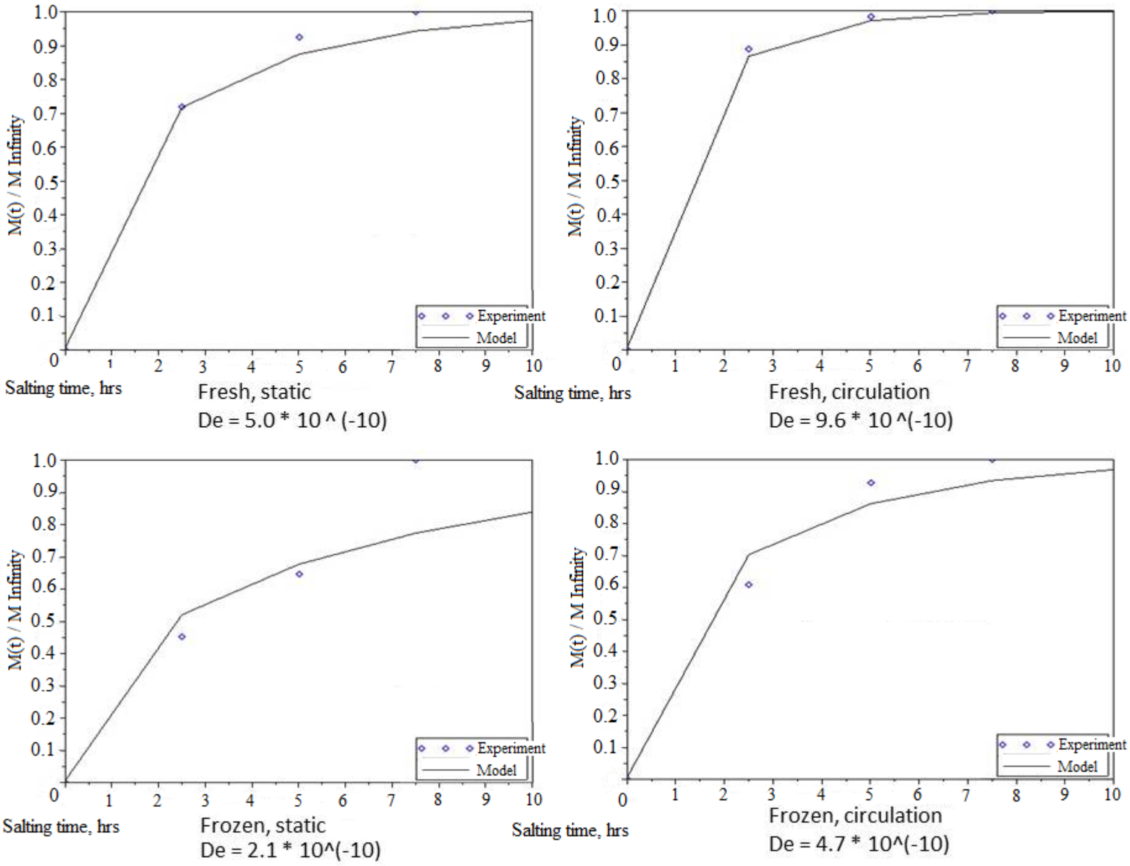
Figure 3. Scheme of experimental setting. 1,2,3: tubes containing a brined ball, 4: bottles containing the brine, pump: imposition of brine circulation.


M(t) / M Infinity
Salting time, hrs
Experiment
Model
Fresh, static
De = 2.9 * 10 ^ (-10)
Fresh, circulation
De = 4.5 * 10 ^(-10)
Frozen, static
De = 2.2 * 10^(-10)
Frozen, circulation
De = 5.0 * 10^(-10)

a


M(t) / M Infinity
Salting time, hrs
Experiment
Model
Fresh, static
De = 5.0 * 10 ^ (-10)
Fresh, circulation
De = 3.0 * 10 ^(-10)
Frozen, static
De = 3.0 * 10^(-10)
Frozen, circulation
De = 6.5 * 10^(-10)

b



c

Figure 4. Results of experiments in different conditions: **a**.- balls made from fresh / frozen minces, brined in static / dynamic conditions at room temperature, brine concentration 3% NaCl; **b**.- balls made from fresh / frozen minces, brined in static / dynamic conditions at room temperature, brine concentration 5% NaCl; **c**.- balls made from fresh / frozen minces, brined in static / dynamic conditions at room temperature, brine concentration 10% NaCl. The solid line shows the calculated concentration based on the model of the diffusion, data points show the experimental results. The relative standard deviation for the determination of salt concentration was found to be 12%. The calculated De for each condition is shown below respective figures.

Table 1. Diffusion coefficient (D_e) (m^2/s) in fish balls made from fresh and frozen haddock fillets. Comparison of forced circulation (pumping) and static conditions. Temperature: 26º C. Brine concentration: 3%, 5%, 10%.

NaCl brine concentration	Fresh		Frozen	
	Static	Dynamic	Static	Dynamic
3 %	**2.9*10^(-10)**	**4.5*10^(-10)**	**2.2*10^(-10)**	**5.0*10^(-10)**
5 %	**5.0*10^(-10)**	**3.0*10^(-10)**	**3.0*10^(-10)**	**6.5*10^(-10)**
10 %	**5.0*10^(-10)**	**9.6*10^(-10)**	**2.1*10^(-10)**	**4.7*10^(-10)**

Table 2. Literature values for diffusion coefficient (D_e) for brining of different materials.

Material	Temperature, ºC	$D_e \cdot 10^{-10}$, (m^2/s)	Reference
Pork ham	5 – 26	0.1 – 1.1	
Fresh Atlantic salmon	10	0.3 – 3.4	
Atlantic salmon	5 – 25	1.6 – 3.9	
Sword fish	25	9.5 – 14.5	
Cod	23 – 33	9.3 – 14.1	
Baltic herring	2 - 20	1.1 – 2.4	
Tuna	30	10.4	