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# Freezing rate simulation as an aid to reducing crystallization damage in foods

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### Abstract

In food freezing processes the presence of large ice crystals is a serious drawback when a good final quality of the product is desired. To study the size and distribution of those crystals, a large piece of pork muscle has been frozen by liquid nitrogen evaporation. A mathematical model to simulate different cooling rates at the surface of the product was solved using a finite element method; this model satisfactorily fitted experimental data and predicted local freezing rates at different locations in the meat tissue. The model was applied to find the freezing rates that led to a good quality product, related to an optimum distribution of small ice crystals located inside and outside the tissue fibres. © 1999 Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

The main drawback in the use of freezing as a way to preserve food is the risk of damage caused by the formation of ice crystals, not only affecting mechanically the cell membranes and distorting the tissue structure but also producing protein denaturation. Protein denaturation is favored by the increase in ionic strength due to fibre dehydration. Furthermore, the amount of exudate produced by the thawed product shows the influence that ice crystal sizes and distribution have upon the final quality (Gómez & Calvelo, 1982; Martino & Zaritzky, 1988).

The thermal gradient existing between an interior point and the surface determines to a great extent the local cooling/freezing rate of the sample; this value decreases towards the centre of the product and is particularly important in large volume products. Air blast, plate contact, circulating brine and liquid nitrogen (ordered in increasing values of the heat transfer coefficient) are the most common methods used for food freezing (Heldman & Lund, 1992). New methods are being analyzed, e.g. the high-pressure-assisted freezing (Otero, Martino, Sanz, & Zaritzky, 1997, Sanz, Otero, Elvira, & Carrasco, 1997) which, based on the lowering of water melting point with pressure, allows a nucleation of ice throughout the whole volume of the product.

Several studies simulating heat transfer in industrial plate freezing of large pieces have been carried out (Mascheroni & Calvelo, 1986) obtaining thermal recordings at different depths. Remarkable differences in ice crystal sizes, location (intra or extracellular) and morphology were observed these being related to freezing rate values through a characteristic time  $t_c$ , a parameter defined as the necessary time to locally lower the temperature from the initial freezing point -1.1 to  $-7^{\circ}$ C where approximately 80% of the water in the product is frozen (Bevilacqua, Zaritzky, & Calvelo, 1979, Bevilacqua & Zaritzky, 1980).

The objectives of the present work were: (1) to analyze size and location of ice crystals in large pieces of pork frozen by liquid nitrogen evaporation, (2) to develop a numerical model of the freezing process to simulate freezing times distribution along the piece of meat and (3) to apply the model to predict the optimum operating conditions (freezing rate and sample size) that cause the least damage related to ice crystal formation.

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## 2. Materials and methods

Pork loin samples of average dimensions of 16 cm length, 11.5 cm wide and 5 cm. height have been used so, in comparison to usual frozen products, they may be considered as large ones.

The freezing device used is shown in Fig. 1. Liquid nitrogen is uninterruptedly evaporated in a closed chamber, the sample lying on a metal grid of  $39 \times 25$  cm (opening  $6 \times 6$  cm) located 5.5 cm above the floor. The process tracking has been carried out by thermocouples: two on the surface of the product, two more in the centre and another one in the surrounding environment.

Ice crystal sizes and their intra and extracellular location have been determined at different depths from microscopial observations. To know the size and position of the ice crystals produced during the freezing process, histological samples have been taken out at three different depths of the product: at the surface (S), in the centre (C) and in an intermediate area (I) equidistant between (S) and (C).

The sample has been fixed at the final freezing temperature by a freeze-substitution method using Carnoy solution as fixative (Martino & Zaritzky, 1986). Images have been obtained by optical microscopy.

As thermal parameters strongly depend on temperature, the process is highly non-linear so analytical type solutions of the heat transfer with phase change problems are excluded and numerical solutions must be used. On the other hand, the geometry of a 2D crosssection of the product can be approximated by an ellipse (semiaxes 5.74 and 2.36 cm) thus, a finite element technique has been selected as the most suitable one to solve numerically the partial differential equation that rules the process. The domain, a quarter of an ellipse for symmetry reasons (Fig. 2), has been discretised in 624 8noded isoparametric elements giving 1977 nodes. Gauss-Legendre numerical integration rules have been

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Fig. 1. Schematic diagram of the experimental freezing device.



Fig. 2. Finite element discretisation of the two dimensional domain.

adopted, the sampling points being the zeroes of Legendre polynomials.

The thermophysical properties of pork, covering the three well differentiated stages of the whole process i.e. cooling, phase change and tempering, have been taken from literature (Levy, 1977). The convective heat transfer coefficient has been assumed to be constant and equal to  $425 \text{ w/m}^2\text{K}$ . A value of  $-0.6 \,^{\circ}\text{C}$  has been adopted as the initial phase change temperature. Moreover, in applying an enthalpy method to solve numerically the problem, a phase change interval from  $-0.6 \,^{\circ}\text{C}$  has been assumed.

# 3. Results and discussion

Micrographs of pre and postfreezing pork muscle are shown in Fig. 3(a)–(d), the latter showing ice crystals at the surface, intermediate and centre areas. Ice crystal sizes in the surface area [Fig. 3(b)], exposed to a greater freezing rate, are markedly smaller than those observed in the centre [Fig. 3(d)] thus, central zone will show lower final quality. Ice crystals shown in Fig. 3(b) did not alter the shape of tissue fibres as compared to unfrozen tissue [Fig. 3(a)].

With the micrographs corresponding to each zone, a statistical analysis of intra and extracellular ice crystals has been performed and the obtained mean ice crystal diameters are shown in Table 1.

Surface and centre measured temperature along time are shown in Fig. 4, as well as values at the centre predicted by the freezing numerical method with application of the same surface temperature variation. Characteristic freezing times  $t_c$  values in an intermediate and centre

Table 1 Intra and extracellular mean ice crystal diameters ( $\mu$ m) at the surface (S), intermediate location (I) and centre (C) of the muscle

	S	Ι	С
Extracellular Intracellular	$\stackrel{-}{3.03 \pm 1.76}$	$\begin{array}{c} 11.29 \pm 0.48 \\ 5.61 \pm 0.56 \end{array}$	$\begin{array}{c} 18.81 \pm 0.60 \\ 11.37 \pm 1.31 \end{array}$



Fig. 3. Micrographs of raw pork muscle (3a) and of intra and extracellular ice crystals at the surface (3b), intermediate location (3c) and centre (3d) of the frozen muscle.

0.05 mr

points, evaluated from experimental and numerical modelling data are shown in Table 2. A good agreement between experimental data and mathematical model predictions was observed (Fig. 4 and Table 2).

(c)

To get a large number of small intracellular ice crystals that would lead to a good quality product, the value of



Fig. 4. Experimental temperature variation at the surface (EXP S) and in the centre (EXP C) and numerically computed in the centre (NUM C).

 $t_c = 7$  min. corresponding to the sample surface [Fig. 3(b)] has been adopted as a reference value for pork tissue.

(d)

The numerical model was applied to simulate different cooling rates at the surface of the product and to determine the corresponding  $t_c$  values at various locations. The simulated freezing rates have been selected to analyze cases of high refrigerating effects at the surface. Fitting the experimental temperature data at the surface (Fig. 4)

Table 2

Characteristic freezing times  $t_c$  (min) at the surface (S), in an intermediate point (I) and in the centre (C) of the sample

	S	I	С
Experimental	7.0	14.0	21.0
Simulation		14.5	23.9

### Table 3

Characteristic freezing times  $t_c$  (min) computed in an intermediate point (I) and in the centre (C) for different simulated surface temperature variations with time

Surface freezing rates	1.5×m	$2 \times m$	$3 \times m$	4×m
(I) (C)	11.2	8.4	5.6	4.7
$(\mathbf{C})$	12.4	10.5	6.6	5.3



Fig. 5. Simulated freezing rates, their slopes being multiples of the original one (m) obtained after fitting the experimental values (circles) to a straight line.



Fig. 6. Experimental temperature variation in an intermediate point (EXP I) and in the centre (EXP C) and predicted variations in the same points by solving the model (MOD), considering a 3 m slope freeezing rate.

to a straight line (slope m), the four cases that were considered include surface temperature variations of 1.5, 2, 3 and 4 times the former slope m (Fig. 5). The characteristic freezing times obtained in each case are shown in Table 3.

## 4. Conclusion

Considering that liquid nitrogen freezing is, among the conventional freezing methods, the one with the highest heat transfer coefficient, it seems technically difficult to increase the refrigerating power up to levels which would promote in the centre of large sized products, values of  $t_c$  below 7 min. Therefore, it may be concluded that, by using freezing methods based on thermal gradient, the formation of extracellular ice crystals that dehydrate the fibres within large volume products cannot be avoided.

The developed model can also be applied to calculate the maximum size of the meat piece that shows  $t_c = 7$ min. in the centre of the product for different surface freezing rates.

New methods like high-pressure-assisted freezing would help to overcome those difficulties, because the effects of thermal gradients on ice crystal sizes (typical of the conventional freezing methods) are avoided.

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