A Rapid Freezing Experiment to Assess the Effect of Temperature/ Position-Variable Thermal Conductivity on Freezing Time Estimation

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Freezing time is an important factor in the design of an industrial freezing process for quality of fish, process productivity and economic reasons. A number of workers have compared freezing times experimentally with those predicted by mathematical models, with varying degrees of success. However, perfect agreement between the measured freezing time and that predicted does not occur. In this paper we propose a procedure for calculating the freezing time with a mathematical model and which takes into account a temperature/position variable feature of effective thermal conductivity. In an attempt to assess the mathematical model proposed, experiments at considerably high freezing rates were performed using sodium chloride aqueous solution as sample material, since precise thermal data are available. The experimental apparatus used was a specially designed plate freezer which enabled us to assure the soundness of the conditions used in the mathematical model. The results of experiment supported the mathematical model proposed.

Freezing is an important storage method which maintains the characteristics of fish. In fish freezing processes, high rate freezing is often recommended because high rate freezing followed by appropriate storage can maintain the characteristics almost unchanged over extended periods.¹⁾ For the quality of fish, process productivity and economic reasons, the freezing time (the time required to lower the product temperature from its initial temperature to a given temperature at its thermal center)²⁾ is one of the most important factors in the design of an industrial freezing process.

The freezing time for a process can be determined experimentally or can be predicted, with varying degrees of success, by mathematical models. A number of workers have compared measured and calculated freezing times but perfect agreement between a measured freezing time and that predicted does not occur.³⁾

The factors responsible to the disagreement between prediction and measured data were summarized by Cleland³⁾ as:

(1) imprecise knowledge about conditions in the freezer or chiller (2) imprecise thermal data for the product (3) use of a prediction method beyond its range of applicability, and (4) shortcomings in the prediction method.

In this paper, we propose a mathematical meth-

od which takes into account the temperature/position variable features of effective thermal conductivity in the freezing time estimation. In an attempt to assess this prediction method, experiments at considerably high freezing rate were performed, eliminating imprecise factors related to equipment conditions as well as the thermal data of the sample material. We used sodium chloride aqueous solution as the sample material because precise thermal data are available.^{4,5)} A specially designed plate freezer was constructed which enabled a sharp step-wise temperature change which was assumed in the prediction. Selected conditions of the freezer was precisely examined using model calculations and experiments.

Theoretical Consideration

In a freezing process of a biological product, a freezing zone is formed which is a mixed layer containing ice and aqueous solution. In the course of freezing, the concentration of dissolved solids in the aqueous phase increases as water is removed as ice, bringing about a change of the effective thermal properties.

Combining a heat balance equation and a Fourier' heat conduction equation, the heat conduction, accompanied with phase transition

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taking place in a freezing zone, may be described using apparent volumetric specific heat capacity, C_{ap} , and effective thermal conductivity, λ_{ef} , as:

$$\frac{\partial}{\partial t}\{C_{ap}(T)T\} = \frac{\partial}{\partial X}\left\{\lambda_{ef}(T)\frac{\partial T}{\partial X}\right\} \quad (1)$$

where

$$C_{ap} = C_{p,ef} + (\rho_{ice}L)(d(1-S)/dT) \quad (2)$$

$C_{p,ef}$ is effective specific heat capacity, ρ_{ice} is density of pure water ice, L is latent heat of fusion and S is volumetric ice fraction.

The initial and boundary conditions are as:

$$T = T_0 \quad \text{at} \quad -D \leq X \leq D, t = 0 \quad (3)$$

$$h(T - T_A) = \mp \lambda_{ef} \frac{\partial T}{\partial X}, \quad \text{at} \quad x = \pm D, t \geq 0 \quad (4)$$

When C_{ap} varies little with t and X , Eq. (1) may be simplified as

$$\frac{\partial T}{\partial t} = \frac{\partial}{\partial X}\left\{\alpha(T)\frac{\partial T}{\partial X}\right\} \quad (5)$$

where $\alpha = \lambda_{ef}/C_{ap}$ (6)

When λ_{ef} varies little with X , Eq. (1) may be simplified as

$$\frac{\partial T}{\partial t} = \alpha(T) \frac{\partial^2 T}{\partial X^2} \quad (7)$$

Since the thermal conductivity of ice is about four times larger than that of the aqueous phase, the effective thermal conductivity, λ_{ef} , of freezing zone varies with the extent of freezing. The extent of freezing varies with temperature, and temperature varies with position, thus implying that the effective thermal conductivity varies with position. However, most mathematical models for freezing biological products neglect the temperature/position-variable feature of effective thermal conductivity, even though they take into account temperature-variable effective thermal conductivity as is shown by Eq. (7).

The right-hand side of Eq. (1) may be expanded as:

$$\frac{\partial}{\partial X}\left(\lambda_{ef}\frac{\partial T}{\partial X}\right) = \frac{d\lambda_{ef}}{dT}\left(\frac{\partial T}{\partial X}\right)^2 + \lambda_{ef}\frac{\partial^2 T}{\partial X^2} \quad (8)$$

Hence we have:

$$\frac{\partial}{\partial t}\{C_{ap}(T)T\} = \frac{d\lambda_{ef}}{dT}\left(\frac{\partial T}{\partial X}\right)^2 + \lambda_{ef}\frac{\partial^2 T}{\partial X^2} \quad (9)$$

where, an equation

$$\frac{\partial \lambda_{ef}}{\partial X} = \frac{d\lambda_{ef}}{dT} \frac{\partial T}{\partial X} \quad (10)$$

is used. This equation clearly shows that the effect of position-variable thermal conductivity caused by temperature-variable thermal conductivity may be concealed when the temperature gradient inside the food body is very small. This implies that the reason why most mathematical models for freezing biological products have neglected the position-variable feature of effective thermal conductivity is that most of experimental results have been obtained at low and moderate freezing rates.⁶⁾

In the present study, time/temperature curves in a sample material were measured at a considerably high freezing rate, which might generate a noticeable temperature gradient in the sample, to assess the temperature/position-variable feature of effective thermal conductivity in the freezing time estimation.

Materials and Methods

Materials

We used 5% sodium chloride aqueous solution as the sample material because precise thermal data, including temperature coefficients and concentration coefficients which were necessary for our assessing the temperature/position-variable thermal conductivity model, are available only for sodium chloride aqueous solution.^{4,5)}

The physical property data, used in this work to predict time/temperature curves, are as follows: Volumetric ice fraction, S , in the freezing zone at an arbitrary temperature was calculated using the phase diagram⁷⁾ for sodium chloride aqueous solution. Effective specific heat capacity, $C_{p,ef}$, effective thermal conductivity, λ_{ef} , and effective density, ρ_{ef} , which are the average values of solid and liquid phases in a freezing zone, were estimated using additive prediction equations⁸⁾

$$C_{p,ef} = SC_{p,ice} + (1-S)C_{p,uf} \quad (11)$$

$$\lambda_{ef} = S\lambda_{ice} + (1-S)\lambda_{uf} \quad (12)$$

$$\rho_{ef} = S\rho_{ice} + (1-S)\rho_{uf} \quad (13)$$

where the subscripts ice and uf refer to pure water ice and sodium chloride solution, respectively.

Cooling System

In order to perform a sharp and intense step-wise change of temperature, a specially designed

Fig. 1. A schematic diagram of experimental apparatus. A: cooling plate, D: end plate, I: stop valve, J: stop valve, j: surge control valve, K: dump tanks, L: suction line, M: compressor, N: discharge, P: air cooled condenser, Q: liquid line, R: flow meter, S: stop valve, T: constant pressure expansion valve, Y: evaporator, Z: food sample.

plate freezer was constructed (Fig. 1). A sufficient amount of liquid refrigerant was stored in the evaporator, Y, before freezing. At the start of freezing, we abruptly reduced the pressure in the evaporator which commenced a vigorous boiling which rapidly removed a great amount of heat; hence rapid cooling of cooling plate, A, took place. The large amount of cooling gas generated was absorbed into dump tanks, K, in a short period of time.

The sample material to be frozen, Z, was placed between the two parallel cooling plates in the evaporator, Y.

For safety reasons, the freezer should be operated at atmospheric pressure during the period of freezing. We used CFC-22 (chloro-difluoromethane) as the refrigerant, because it has a reasonably low boiling temperature (-40.8°C) at atmospheric pressure, large in latent heat ($233 \text{ J}\cdot\text{kg}^{-1}$ at 1 atm.) and is not particularly corrosive to metal.

Checking the Dimension of the Test Section

The width of the sample plate was determined as follows so that the assumption of one-dimensional heat-transfer might be acceptable in the

freezing of the sample material. A preliminary calculation on heat conduction without phase change (see Appendix I) suggests that the aspect ratio, which is the thickness of the object being frozen divided by its width, should not be larger than 0.40 in order to restrict the edge effect within 1%. The sample thickness, on the other hand, was set to 60 mm in this experiment, because the thickness of fishery products often applied to plate freezers was reported to be in the range of 25 mm to 60 mm.⁹⁾ Using the aspect ratio of 0.40 and the sample thickness of 60 mm, we decided the sample width as 150 mm.

We made an evaporator as depicted in Fig. 2. A couple of 8 mm thick copper plate were used as the cooling plates. In order to make sure the potential error⁹⁾ caused by the deformation of the food body being frozen, we estimated the deflection and the stress of the cooling plate, the deflection and stress generated by the load of significant pressure at the start of cooling (see Appendix II). The result implied that you should operate the evaporator with a pressure load within 1 MPa so that you can avoid a permanent deflection on the cooling plate.

Fig. 2. A plan and elevation view of evaporator. A: cooling plate (copper), B: rubber packing, C: vessel frame (steel), D: vessel plate (steel), E: vessel assembling bolt (M8), F: rubber packing, G: sample holding frame, H: stay bolt (M8), Y: evaporator, Z: food sample.

Capacity of the Dump Tanks and the Compressor

The cooling heat load, to cool down the cooling plate stepwise at the start of cooling, may be shared by the dump tanks and the compressor. We decided the capacity of the dump tanks so that they covered the heat to be removed from the evaporator in order to lower its temperature from 20°C to -20°C. The amount of this heat is 497 J, and hence the volume of refrigerant gas to be stored into the dump tanks is 0.211 m³. We connected six dump tanks (36 liters for each) in parallel.

The compressor was assigned to cool down the evaporator from -20°C to -40°C. The heat to be removed is 249 J and hence the volume of refrigerant gas to be removed is 0.227 m³.

We used a compressor which displaced the gas at the rate of 0.0028 m³·s⁻¹. It takes the compressor 80 seconds to displace the volume of gas calculated above. When the total freezing time was about 4000 seconds, we could operate the compressor with a doubled frequency using a frequency converter and completed the stepwise temperature change within 1% of the total freezing time.

Experimental Procedure

We added 3% weight agar to the sodium chloride solution in order to immobilize the solution. The solution was cast into the test section, *i. e.* between the cooling plates in the evaporator. Casting sample solution enhanced the reproducibility of the experiment, diminishing the unstable contact resistance between the sample and the cooling plates. We closed the valve, I, and started the compressor. When the pressure came up to a preset pressure (*e. g.* 500 kPa) in the evaporator and down to a preset pressure (*e. g.* 0.5 kPa) in the dump tanks, we opened the valve I, to start freezing. We operated the freezer in order to keep the evaporator temperature constant, manipulating the expansion valve, T, and the rotating frequency of the compressor. The temperature at the center of the sample solution was recorded with a copper/constantan thermocouple (1 mm diameter).

Results and Discussion

The time/temperature curves of the refrigerant in the evaporator measured in the time course of freezing (Fig. 3), showed sharp step-wise tem-

Fig. 3. Selected time/temperature curves of the flashing refrigerant in the evaporator.

Fig. 4. Selected time/temperature curves at the center of the sample. $\circ$ & $\times$: 5% sodium chloride aqueous solution, $\blacksquare$ & $\square$: 15% sodium chloride aqueous solution.

perature changes at the early stages of freezing, followed by a stable constant boiling temperature during the rest of freezing period.

At the end of each run of experiments, we examined the cooling plate and found no permanent deflection on it.

Time/temperature curve data collected through a thermocouple inserted into the center of the sample solution showed a remarkable reproduc-

ibility as illustrated in Fig. 4.

We predicted a time/temperature curve, using each mathematical model described by Eqs. (5), (7), or (9), via an explicit finite difference method. The finite difference scheme we used for Eq. (8) is:

$$\frac{\lambda'_f(T_{j+1} - T_{j-1})^2}{2\Delta X^2} + \lambda_j \frac{T_{j+1} + T_{j-1} - 2T_j}{2\Delta X^2} \quad (14)$$

Fig. 5. The effective thermal conductivity of 5% sodium chloride aqueous solution.

Fig. 6. The apparent volumetric specific heat capacity, C_{ap} , of 5% sodium chloride aqueous solution.

where ΔX is an incremental length of finite difference element, T_j is the temperature at the j -th element, λ_j is effective thermal conductivity at the j -th element. Effective thermal conductivity calculated through Eq. (12) is shown in Fig. 5. λ'_j is $d\lambda/dT$ at the j -th element, which was obtained by differentiating λ_j in Fig. 5.

There is some literature¹⁰⁾ in which the right-hand side of Eq. (1) is calculated through a scheme as follows:

$$\frac{\lambda_{j+1/2}(T_{j+1} - T_j) - \lambda_{j-1/2}(T_j - T_{j-1})}{2\Delta X^2} \quad (15)$$

where $\lambda_{j+1/2}$ and $\lambda_{j-1/2}$ are effective thermal conductivity at temperature $(T_{j+1} + T_j)/2$ and $(T_j + T_{j-1})/2$, respectively. The use of Eq. (15) may cause an error when λ varies nonlinearly with temperature as is the case shown in Fig. 5.

Numerical calculation of Eq. (5) was performed in a similar manner as Eq. (7) substituting α for λ in the right-hand side. The apparent specific heat capacity, C_{ap} , calculated by Eq. (2) (shown in Fig. 6) was used in the prediction.

In calculating a time/temperature curve, we need a value for surface heat-transfer coefficient, h , which appears in Eq. (4). The surface heat-transfer coefficient in freezing process operated with commercial type contact freezer is well known to be affected with the surface heat resistance between the cooling plate and the product. In this experiment, however, the surface heat-resistance between the cooling plate and the sample is negligibly small because the sample was cast in between the plates.

The surface heat-transfer coefficient in this experiment may be obtained as:

$$\frac{1}{h} = \frac{t_c}{\lambda_c} + \frac{1}{h_B} = \frac{0.008}{386} + \frac{1}{h_B} = \frac{1}{48000} + \frac{1}{h_B} \quad (16)$$

where t_c and λ_c are the thickness and thermal conductivity of cooling plate, respectively. h_B is a boiling heat-transfer coefficient of the refrigerant in the evaporator. h_B may be estimated as follows: In this experiment, the sample solution was cooled down from room temperature (*e.g.* 15°C) to eutectic temperature (−25°C) in about one hour. The amount of heat, Q , to be removed from the sample may be as follows using specific heat capacity ($4200 \text{ J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$ at room temperature, $2100 \text{ J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$ at subfreezing temperature), latent heat of fusion ($333000 \text{ J} \cdot \text{kg}^{-1}$), density ($1000 \text{ kg} \cdot \text{m}^{-3}$) and cross section area ($S_a \text{ m}^2$):

$$Q = (4200 \times 15 + 2100 \times 25 + 333000) \times 0.06 \times 1000 \times S_a$$

Since the sample was cooled both sides, the heat flux $q \text{ (W} \cdot \text{m}^{-2}\text{)}$ may be:

$$q = Q / (2 \times S_a \times 3600) = 3738 \text{ W} \cdot \text{m}^{-2}$$

Using a nomograph which appears in ASHRAE handbook 1985 (chapter 4, Fig. 4), the boiling heat-transfer coefficient, h_B can be estimated when the heat flux and refrigerant saturation pressure are available. Following this procedure, we obtained $h_B = 511 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$. Substituting this into Eq. (16), we had $h = 506 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$. Hence we used $500 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$ as the surface heat-transfer coefficient in calculating the time/temperature curves.

Fig. 7. Calculated time/temperature curves at the center of a sample, using Eqs. (5), (7), and (9). $\times$ indicates a measured value. h_s^* is assumed to be $500 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$.

Fig. 8. Time/temperature curves calculated using Eq. (9) with varied surface heat-transfer coefficients.

The result of the prediction is shown in Fig. 7. The prediction using Eq. (9) favorably agreed with the measured data, whereas those using Eqs. (5) and (7) failed.

Using Eq. (7), Cleland and Earle¹¹⁾ favorably predicted the time to freeze foods and food substitute in a plate freezer with a varied h (10 – $500 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$). Heldman¹²⁾ found that Eq.

(5) worked when he compared mathematical models for freezing time estimation.

In the freezing experiments used by Cleland and Earle and Heldman to check Eqs. (5) and (7), the freezing rates were low and moderate; hence the temperature gradient induced in the food body might be negligibly small. In the present experiment, on the other hand, a rapid

freezing might induce a considerable temperature gradient in the sample material, which revealed the temperature/position-variable feature of effective thermal conductivity. When a temperature gradient exists in a food body, the temperature-variable thermal conductivity gains the feature of a position-variable as well. This may be the cause of the failure of Eq. (7) in predicting a time/temperature curve measured in the present experiment; in the procedure calculating Eq. (7), effective thermal conductivity varied with time and temperature but not with position.

Equation (5) superficially considers the position-variable feature of effective thermal conductivity. Unfortunately, however, the agreement between that predicted and that measured is poor. This is because the assumption that C_{ap} varies little with t and X , which was used to derive Eq. (5), does not stand as is shown in Fig. 6; C_{ap} varies nearly 40 times in the range of freezing zone temperature.

We examined the effect of surface heat-transfer coefficient, h , on freezing time/temperature curves using Eq. (9). The result, illustrated in Fig. 8, shows that the change of h values significantly influences the time/temperature curves when h is in the range of 200 to 500 $\text{W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$; however, the change of h influences little when h is larger than 500 $\text{W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$.

The conclusion to be drawn from the present results is as follows. In the case of rapid freezing, the use of a mathematical model which takes the temperature/position-variable effective thermal properties into consideration is highly recommended for successful freezing time estimation.

Appendix I

The heat conduction equation for a square plate ($2W$ in width, $2D$ in thickness) may be given by,

$$\rho C_p \frac{\partial T}{\partial t} = \lambda \left(\frac{\partial^2 T}{\partial X^2} + \frac{\partial^2 T}{\partial Y^2} + \frac{\partial^2 T}{\partial Z^2} \right) \quad (1a)$$

where α , C_p , λ are density, specific heat capacity and heat conductivity of the plate, respectively.

When the temperature of the refrigerant changes step-wisely from T_0 to T_A , the boundary conditions are:

$$\left(\frac{\partial T}{\partial X} \right)_{X=D} = \frac{h(T - T_A)}{\lambda} \quad (2a)$$

$$\left(\frac{\partial T}{\partial Y} \right)_{Y=W} = \left(\frac{\partial T}{\partial Z} \right)_{Z=W} = \frac{h_w(T - T_A)}{\lambda} \quad (3a)$$

where h and h_w are the surface heat-transfer coefficients at sample/refrigerant interface and at the heat-sealed edge of the sample, respectively. The initial temperature is

$$T(0, X, Y, Z) = T_0 \quad \text{at } t=0.$$

When we solved Eq. (1a) and calculated the temperature at the center of the square using the values $T_0 = 0^\circ\text{C}$, $T_A = -40^\circ\text{C}$, $h = 500 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$, $h_w = 10 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$, $\lambda = 2.2 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$, we found that the aspect ratio D/W should not be larger than 0.40 in order to restrict the edge effect within 1%.

Appendix II

We have a significant change of pressure in the evaporator as we start freezing in this experiment. Suppose the refrigerant is 300 K (26.9°C) at the beginning of freezing; its equilibrium pressure is 1.097 MPa (11 atm). Hence, an about 1 MPa (10 atm) pressure change may be loaded on the evaporator at the start of freezing, which might cause the deflection of cooling plates. This deflection may result in some change in the sample dimension. The deflection and the stress to be generated in the cooling plate was estimated using a small deflection model for a rectangular plate.¹³⁾

The evaporator had rubber packing at the connecting part, hence the cooling plate had elastic supporting points. Since it is difficult to calculate the deflection of cooling plate with an elastic boundary condition, we calculated with a free boundary condition as well as a rigid boundary condition, expecting that the result of calculation with the elastic boundary condition might be in between those with a free boundary and a fixed boundary.

Fig. 9 illustrates the predicted deflection. We measured deflection of the cooling plates of our evaporator which was loaded with an oil-pressure loading machine. The measured values of the deflection plotted in Fig. 9 shows that the deflection was successfully estimated.

The stress generated by the pressure load was also estimated using the small deflection model,¹³⁾ as is shown in Fig. 10. A stress of 230 MPa, which was predicted to be induced at the center of the cooling plate by a 1 MPa pressure load, is equal to the yield stress of copper plate.

If the evaporator is operated with a pressure load over 1 MPa, the stress generated at the

Fig. 9. Deflection at the center of a rectangular plate. Solid line: calculated using a small deflection model with a free boundary, chain line: calculated using a small deflection model with a rigid boundary.

Fig. 10. Stress at the center of a rectangular plate. Solid line: calculated using a small deflection model with a free boundary, chain line: calculated using a small deflection model with a rigid boundary.

center of the plate may exceed the yield stress and permanent deflection may take place. Hence, the experimental procedure not to exceed this pressure limit should be carefully managed.

Nomenclature

C_{ap}	$[J \cdot m^{-3} \cdot K^{-1}]$	Apparent volumetric heat capacity
C_p	$[J \cdot kg^{-1} \cdot K^{-1}]$	Heat capacity due to constant pressure
D	[m]	Half-thickness of food body

h	$[W \cdot m^{-2} \cdot K^{-1}]$	slab Surface heat-transfer coefficient
h_B	$[W \cdot m^{-2} \cdot K^{-1}]$	Boiling heat-transfer coefficient
L	$[J \cdot kg^{-1}]$	Latent heat of fusion
Q	[J]	Heat load
S	$[m^3 \cdot m^{-3}]$	Volumetric ice fraction
T	$[^{\circ}C]$	Temperature
t	[s]	Time
t_c	[m]	Thickness of cooling plate
v	$[m^3]$	Volume of refrigerant gas
W	[m]	Half-width of food body slab/cooling plate
x, y, z	[m]	Position coordinate
α	$[m^2 \cdot s^{-1}]$	Thermal diffusivity
Δx	[m]	Incremental length of finite difference element
λ	$[W \cdot m^{-1} \cdot K^{-1}]$	Thermal conductivity
λ_c	$[W \cdot m^{-1} \cdot K^{-1}]$	Thermal conductivity of cooling plate
ρ	$[kg \cdot m^{-3}]$	Density
Subscripts		
A		ambient condition
ef		effective value of freezing zone
		frozen state
ice		pure water ice
j		the j -th finite element
uf		unfrozen state
W		at wall
0		initial state
1		first stage
2		second stage

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