Generalized (Semi)-Empirical Formulae for Optimal Sterilization Temperatures of Conduction-Heated Foods with Infinite Surface Heat Transfer Coefficients

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ABSTRACT

A computer program was developed to model heat sterilization of one-dimensional conduction-heated foods with negligible surface resistance to heat transfer.

The optimal sterilization temperature, defined as the processing temperature that results in a food product with minimum surface cook-value after achieving the desired degree of sterility, was calculated as a function of food properties (thermal diffusivity, Z-value of the quality factor), processing conditions (dimensions and geometry of the food or container, initial temperature of the product, heating medium come-up-time) and processing criteria (target F_0 -value).

The initial temperature of the food and the heating medium come-up-time have a minor effect on the optimal temperature. Generalized (semi)-empirical formulae relating optimal sterilization temperatures and all relevant variables were developed.

NOTATION

A	Area (m^2)
A_i	Surface area associated with node i ($i = 1, \dots, N$) (m^2)
c_p	Specific heat (J/kg K)

C	Cook-value (min)
CUT	Time required for the heating medium to reach the sterilization temperature (min)
D	Decimal reduction time (min)
D_{ref}	Decimal reduction time at a reference temperature of 121.15°C (min)
f_h	Slope factor of a heating curve (min)
F_0	Sterilizing value at reference temperature 121.15°C and $Z_m = 10^\circ\text{C}$ (min)
F_t	Target sterilizing value at reference temperature 121.15°C and $Z_m = 10^\circ\text{C}$ (min)
j	Heat penetration parameter, heating lag-factor (dimensionless)
L	Characteristic length (radius of an infinite cylinder or a sphere and half thickness of an infinite slab) (m)
RF	Reduced Fourier number (s^{-1})
t	Time (s)
t_h	Holding time (s)
t_p	Holding time + come-up-time (s)
T	Temperature ($^\circ\text{C}$)
T_c	Temperature in the geometric centre of the product ($^\circ\text{C}$)
T_h	Holding temperature ($^\circ\text{C}$)
T_i^n	Temperature at node i and iteration step n ($^\circ\text{C}$)
T_0	Initial temperature of the product ($^\circ\text{C}$)
T_{op}	Optimal processing temperature ($^\circ\text{C}$)
T_{ref}	Reference temperature ($^\circ\text{C}$)
T_s	Temperature at the surface ($^\circ\text{C}$)
T_∞	Heating medium temperature ($^\circ\text{C}$)
V	Volume (m^3)
V_i	Control volume for node i ($i = 1, \dots, N$) (m^3)
x	Spatial coordinate (m)
X	Level of microorganisms, enzymes or quality index
Z	Z-value ($^\circ\text{C}$)
Z_m	Z-value for the microorganism ($^\circ\text{C}$)
Z_q	Z-value for the quality factor ($^\circ\text{C}$)
α	Thermal diffusivity (m^2/s)
Δt	Time increment (s)
Δx	Space between nodes (m)
ρ	Density (kg/m^3)
λ	Thermal conductivity ($\text{W}/\text{m K}$)
λ_{xx}	Thermal conductivity in the x -direction ($\text{W}/\text{m K}$)

INTRODUCTION

Thermal processing of foods is a preservation technique already known from the beginning of the 19th century. Nevertheless, the idea of modelling and optimizing this process is relatively recent.

Several aspects should be taken into account when considering the optimization of a heat sterilization process. Quality and nutrient retention, economical viability as well as the formation of toxic compounds (Lund, 1982) are most relevant in improving the overall result. Controlling the heating medium time-temperature profile can affect various factors, e.g. energy consumption, process time, retention of nutrients and cost of control (Lund, 1982).

Barreiro *et al.* (1984) developed a procedure to estimate the energy consumption of batch processes. They applied the method to determine the minimum energy consumption for sterilizing canned pea puree, while maintaining the same reduction of microorganisms. However, economical viability is only one step in the optimization procedure. Processing foods under conditions of minimum energy consumption, neglecting its effect on quality, can lead to an unacceptable final product. This is the reason why most of the work done on optimizing thermal processes aims at obtaining a high quality product with maximum nutrient retention.

Thermal inactivation of microorganisms is much more temperature dependent than quality and nutrient degradation (Lund, 1977). Higher-temperature/shorter-time (HTST) processes, for low viscous fluid foods, use this knowledge to maximize quality and nutrient retention. However, the applicability of this principle to conduction heated foods is limited (Lund, 1982). Furthermore, the high thermal resistance to inactivation of enzymes (e.g. peroxidase), can limit the applicability of the HTST-principle (Saguy, 1988).

The first computer optimization of sterilization processes for conduction-type heated foods, using a finite difference numerical method, was done by Teixeira *et al.* (1969). They proved that it is possible, when using the required lethality as a state variable, to find a process with constant temperature which is optimal from the point of view of minimizing overall thiamine degradation. In their case study, this optimum was in the range of conventional processes.

Several authors determined optimal constant processing temperatures (Teixeira *et al.*, 1975; Thijssen *et al.*, 1978; Ohlsson, 1980a, b; Tucker & Holdsworth, 1990, 1991) in order to minimize average or surface quality and nutrient degradation. Variable sterilization temperature was studied as a possible means of improving the process (Teixeira *et al.*, 1975; Saguy & Karel, 1979; Martens, 1980; Nadkarni & Hatton, 1985). The

general conclusion is that variable sterilization temperature is nearly as good as constant sterilization temperature in terms of the final level of nutrients and quality factors.

Among calculation procedures, a general, easy and quick method of determining optimal sterilization temperatures in terms of known experimental parameters and processing criteria is lacking. Furthermore, there is a need for generalized equations relating optimal sterilization temperatures to the relevant variables. These types of equations would both have a practical use and improve our scientific knowledge on this topic.

Hendrickx *et al.* (1989) developed a (semi)-empirical formula to calculate the optimal temperature to minimize surface quality and nutrient losses. Surface quality is of interest when appearance and odour are considered, while volume average quality is of interest when taste, consistency or nutrient retention are considered (Ohlsson, 1980a). Their model did not take into account the lethality gained during the cooling period and the existence of external resistance to heat transfer (finite Biot number). Nevertheless, the approach of an infinite heat transfer coefficient is valid for a wide range of practical thermal processes, e.g. condensing steam sterilization and processing of metal containers (Tucker & Holdsworth, 1991).

Therefore, the purpose of the research presented here was to develop generalized equations relating optimal temperatures and all relevant variables. The (semi)-empirical formula of Hendrickx *et al.* (1989) was extended to calculate optimal sterilization temperatures for processes with infinite Biot number, including: (i) the cooling period in the lethality calculation and (ii) the effect of a come-up-time. One-dimensional heat transfer models covering extreme shapes — infinite slab, infinite cylinder and sphere — were used.

METHODOLOGY

To obtain optimal sterilization temperatures, the following procedure was set up. First, a mathematical model for the sterilization process was developed. Secondly, the relevant variables affecting the optimal temperature were studied. Finally, simulations were executed and a regression analysis was performed on the data obtained.

Development of the model

Modelling sterilization processes mainly involves the mathematical description of two important phenomena: (i) heat transfer into the food

and (ii) kinetics of thermal inactivation of microorganisms, enzymes and quality changes. These phenomena are typically described by differential equations expressing the rate of change for a property (e.g. temperature, viable cell number) with respect to time and distance. The energy consumed by the thermal destruction of cells and food quality is extremely small. Therefore, the heat transfer equation can be solved separately from the kinetic equations (Clark, 1978).

Assumptions of the model

In the development of the model some simplifying assumptions were made:

1. Heat transfer into the food is by pure conduction.
2. Only one-dimensional shapes, such as infinite slab, infinite cylinder and sphere, were considered.
3. The food product is homogeneous and isotropic, i.e. its properties are constant with location and temperature.
4. The initial temperature of the food (T_0) is uniform.
5. The heating medium time-temperature profile is as shown in Fig. 1, consisting of a come-up-time with a linear increase of temperature from the initial temperature (equal to the initial temperature of the food) to the constant holding temperature, from which it is

Fig. 1. Heating medium time-temperature profile assumed in the development of the model: T_∞ , heating medium temperature; T_h , holding temperature; T_0 , initial temperature of the product.

cooled to 20°C. This type of time-temperature profile approximates the characteristic temperature profile for static vertical and horizontal retorts, agitating retorts and fluids with solid particles in aseptic processing, while the same profile with zero come-up-time is typical for continuous retorts.

6. There is no heat transfer resistance at the product surface (infinite Biot number).
7. First-order kinetics apply for the thermal inactivation of spores of microorganisms and enzymes and for the thermal degradation of quality and nutrients.
8. Kinetic parameters are described by the empirical *D-Z* model.

Modelling heat transfer

The governing differential equation for transient heat transfer by conduction in one direction, the 2nd law of Fourier, can be written as

$$\rho c_p \frac{\delta T}{\delta t} = \frac{\delta}{\delta x} \left(\lambda_{xx} \frac{\delta T}{\delta x} \right) \quad (1)$$

using cartesian coordinates (Carslaw & Jaeger, 1959). This equation describes the heat flow into one-dimensional shapes: (i) sphere; (ii) infinite cylinder (height to radius ratio greater than five); and (iii) infinite slab (length to width ratio greater than five).

Applying the following initial and boundary conditions:

$$\begin{array}{llll} \text{(i)} & T = T_0 & \text{at } t = 0 & \\ \text{(ii)} & T = T_\infty & \text{at } t > 0 & \text{and } x \in S \\ \text{(iii)} & \delta T / \delta x = 0 & \text{at } t > 0 & \text{and } x \in P \end{array} \quad (2)$$

where *S* denotes surface and *P* denotes planes of symmetry, an explicit finite difference solution, for eqn (1), with a non-capacitance node at the surface can be derived (Clausing, 1989). The equations to calculate the temperature distribution as a function of space (Fig. 2) and time are presented in Table 1.

Modelling kinetics of thermal inactivation

Thermal process design is traditionally based on the hypothesis that thermal inactivation of microorganisms, of the same strain and the same history and age, and degradation of quality can be modelled by a single first-order reaction. Even though only 40% of the thermal resistance studies on microorganisms can be correctly described with this simple approach (Maesmans *et al.*, 1990), in most of the cases it leads to acceptable results (Hayakawa, 1978).

Fig. 2. Discretization of space for one-dimensional heat transfer shape using a non-capacitance surface node.

First-order inactivation kinetics can be written mathematically as

$$\frac{dX}{dt} = -\frac{2.303X}{D} \quad (3)$$

Within a given temperature range, the temperature dependence of D can be expressed according to the empirical D - Z model:

$$D = D_{\text{ref}} \times 10^{(T_{\text{ref}} - T)/Z} \quad (4)$$

where the Z -value is the temperature rise needed to reduce the decimal reduction time by one log-unit.

Optimizing the processing temperature

A Pascal program was developed in order to implement the proposed model. A target sterilization value, F_t -value, was specified at the coldest point of the product (geometric centre) and calculated by the general method (Ball & Olson, 1957)

$$F_t = \int_0^t 10^{(T_c - T_{\text{ref}})/Z_m} dt \quad (5)$$

where $Z_m = 10^\circ\text{C}$, $T_{\text{ref}} = 121.15^\circ\text{C}$ and t is total processing time, using the centre time-temperature profile (T_c) including the cooling part. Cooling was allowed to continue until there was no further significant contribution to the lethality. For a given sterilization temperature (T_h) the F_t -value was obtained iteratively as is schematically shown in the flow diagram of Fig. 3.

TABLE 1

Finite-Difference Numerical Solution for One-Dimensional Heat Transfer Problems with Infinite Surface Heat Transfer Coefficient

$$T_N^{n+1} = \frac{\Delta t \times \alpha \times A(N)}{\Delta x \times V(N)} T_{N-1}^n + \left(1 - \frac{\Delta t \times \alpha \times A(N)}{\Delta x \times V(N)} \right) T_N^n$$

$$T_i^{n+1} = \frac{\Delta t \times \alpha \times A_i}{\Delta x \times V_i} T_{i-1}^n + \frac{\Delta t \times \alpha \times A_{i+1}}{\Delta x \times V_i} T_{i+1}^n + \left(1 - \frac{\Delta t \times \alpha \times A_i}{\Delta x \times V_i} - \frac{\Delta t \times \alpha \times A_{i+1}}{\Delta x \times V_i} \right) T_i^n$$

for $2 < i < N$

$$T_2^{n+1} = \frac{\Delta t \times \alpha \times A_3}{\Delta x \times V_2} T_3^n + \frac{\Delta t \times \alpha \times A_2}{\Delta x/2 \times V_2} T_1^n + \left(1 - \frac{\Delta t \times \alpha \times A_3}{\Delta x \times V_2} - \frac{\Delta t \times \alpha \times A_2}{\Delta x/2 \times V_2} \right) T_2^n$$

$$T_1^{n+1} = T_\infty$$

Fig. 3. Schematic flowsheet of the computer program to calculate the F_t -value and corresponding surface cook-value for a given holding temperature (T_h).

$$^{(1)}\Delta t_h = [(F_t - F_o) / 10^{(T_h - 121.15^\circ\text{C})/Z_q}] \times 60 \text{ (s)}$$

The optimal temperature was defined as the sterilizing (holding) temperature which results in a product with a minimum surface cook-value. The objective function then becomes:

$$C = \int_0^t 10^{(T_s - T_{\text{ref}})/Z_q} dt \quad (6)$$

where $T_{\text{ref}} = 100^\circ\text{C}$. T_s is the temperature at the surface as a function of time and Z_q is the z -value for the quality index. To estimate the optimal temperature, the computer program was integrated in an optimization routine, using the Davis, Swann and Campey method (Saguy, 1983).

Development of (semi)-empirical formulae

The optimal temperature can be correlated to the heat penetration parameter f_h (Ohlsson, 1980a, b; Hendrickx *et al.*, 1989). For the three different geometries studied, f_h is given by (Ball & Olson, 1957):

$$f_h = u \times L^2 / (\alpha \times 60) \quad (7)$$

where u is a constant; 0.933, 0.398 and 0.233 respectively for an infinite slab, an infinite cylinder and a sphere. L is the characteristic dimension (half-thickness or radius).

In this work, a new parameter, defined as the ratio of thermal diffusivity/square of the characteristic dimension, called the Reduced Fourier number, RF , will be used:

$$RF = \alpha / L^2 = u / (f_h \times 60) \quad (8)$$

Several optimization problems were executed in order to study the influence of the various parameters on the optimal temperature. Parameter values were chosen in such a way to simulate a wide range of practical sterilization processes.

The heat penetration factor f_h was allowed to vary from 0.5 to 600 min, so as to consider products with a thermal diffusivity around $1.6 \times 10^{-7} \text{ m}^2/\text{s}$ (George, 1990) and a characteristic dimension between 0.5 and 7.5 cm.

Z -values for quality and nutrient degradation between 15 and 45°C cover most kinetic data available (Lund, 1977; Ohlsson, 1988).

Simulation cases, considering a warehouse and distribution temperature below 30°C , had F_t -values within 3 to 15 min. This range was calculated from the minimum and maximum recommended F_0 -values and safety-factors (Pflug, 1987). Although, there is some variability associated with the Z -value for the thermal inactivation of *Clostridium botulinum* spores, a value of 10°C is still the best recommendation for calculating low-acid food sterilization process from the safety point of view (Pflug & Odlaug, 1978).

Initial product temperatures from 10 to 70°C and heating medium come-up-time from 0 to 15 min are values which include almost all the practical situations.

RESULTS AND DISCUSSION

(Semi)-empirical formulae

To study the influence of the four variables: rate of heat penetration (f_h or RF number), F_t -value, Z -value for the quality factor (Z_q) and initial temperature of the product (T_0); a total of 3360 optimization problems were solved, 1120 per geometry (infinite slab, infinite cylinder and sphere). Simulations of all combinations of the values presented in Table 2 were performed. Because of log-linear relation between optimal processing temperatures and the parameter f_h is expected, this variable was varied logarithmically. Some typical calculation results are shown in Figs 4 and 5.

As was suggested in the formula of Hendrickx *et al.* (1989), a log-linear relation is observed between the optimal processing temperature, to minimize the surface cook-value, and the ratio F_t/f_h (Fig. 4). For products with a high heating rate and large F_t -values, the HTST principle is more applicable. Minimizing the cook-value for a quality index with a high Z -value (higher resistance to thermal degradation) results in a higher optimal processing temperature (Fig. 5). A linear relation between the natural logarithm of the Z_q -value and the optimal temperature is observed. The initial temperature of the food product (T_0) has a small influence on the optimal processing temperature. Furthermore, a linear relation between the optimal temperature and T_0 is observed. Lower optimal processing temperatures correspond to higher optimal surface cook-values. A linear relation is observed between the logarithm of optimal surface cook-value and the logarithm of F_t or the logarithm of f_h .

For a given food product (characterized by an f_h and a Z_q -value) at a given initial temperature, the optimal processing time is insensitive to the F_t -value. This means that, although the optimal temperature changes with the F_t -value, the processing time is constant. This may result in an

TABLE 2
Input Data for the Simulations

Variable	Values							
f_h (min)	0.5	1.4	3.8	10.5	30	79	222	600
F_t (min)	3	6	9	12	15			
Z_q (°C)	15	20	25	30	35	40	45	
T_0 (°C)	10	30	50	70				

important rule of thumb: if you have an optimized process and you want to increase F_t , you just have to use the same processing time and adjust the processing temperature to reach the desired F_t -value.

For each geometry a multiple linear regression analysis, using the software package, SAS (SAS, 1982), was performed. The regression equa-

Fig. 4. Optimal processing temperature (T_{op}) as a function of F_t/f_h . The initial temperature (T_0) of the food is 10°C. Z_0 : $\circ$, 15°C; +, 25°C; $\times$, 35°C; Δ , 45°C.

Fig. 5. Optimal processing temperature (T_{op}) as a function of the Z -value for the quality factor (Z_0). The initial temperature of the food is 30°C. $\text{Log } F_t/f_h$: $\circ$, -2.31; +, -1.27; $\times$, -0.39; Δ , 0.5; $\diamond$, 1.48.

tions are listed in Table 3. An analysis of the equation coefficients listed in Table 3 leads to the conclusion that the optimal processing temperature is independent of geometry when the values of F_t , f_h , Z_q and T_0 are fixed. A general formula resulted from a regression analysis on all the data:

$$T_{op} = 86.68 + 9.73 \times \log (F_t/f_h) + 10.46 \times \ln (Z_q) + 0.025 \times T_0$$

correlation coefficient = 0.999 (9)

This result is explained by the fact that the surface time-temperature profile, when there is no external resistance to heat transfer, is independent of the shape and equal to the heating medium temperature. Furthermore, the heat penetration parameter j (j is the ratio $(T_\infty - T_A)/(T_\infty - T_0)$, where T_A is the ordinate of the origin of the heat penetration curve (Ball & Olson, 1957)), which differs from shape to shape, has a minor impact on the optimal processing temperature.

Equation (9) requires a knowledge of the parameter f_h which can be determined experimentally from a heat penetration study. When the objective is just a theoretical study, and it is not possible to perform a heat penetration study, the equations in Table 3 can be expressed in terms of the variable RF (eqn (8)):

$$T_{op} = a' + b \times \log (F_t \times RF) + c \times \ln (Z_q) + d \times T_0$$

$T_{op}(^{\circ}\text{C}); F_t(\text{min}); RF(\text{s}^{-1}); Z_q(^{\circ}\text{C}); T_0(^{\circ}\text{C})$ (10)

where the coefficients remain the same except for the intercept, which is now a function of geometry: $a' = 103.9, 107.9$ and 110.6 respectively for an infinite slab, an infinite cylinder and a sphere.

TABLE 3

Regression Formulae to Calculate Optimal Processing Temperatures as a Function of Relevant Variables

$$T_{op} = a + b \times \log (F_t/f_h) + c \times \ln (Z_q) + d \times T_0$$

$$T_{op}(^{\circ}\text{C}); F_t(\text{min}); f_h(\text{min}); Z_q(^{\circ}\text{C}); T_0(^{\circ}\text{C})$$

Geometry	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	Correlation coefficient
Infinite slab	86.68	9.74	10.66	0.026	0.999
Infinite cylinder	86.71	9.73	10.44	0.025	0.999
Sphere	86.14	9.72	10.26	0.024	0.999

To obtain a general equation, in terms of Reduced Fourier number (RF), which is independent of geometry, the difference between the intercepts (eqn (10)) suggested the introduction of the empirical term: $2^{(V/AL)}$.

$$T_{op} = 118.1 + 9.71 \times \log(F_t \times RF) + 10.46 \times \ln(Z_q) + 0.025 \times T_0 - 6.87 \times 2^{(V/AL)} \quad \text{correlation coefficient} = 0.998 \quad (11)$$

Equations (9) and (11) allow the calculation of optimal processing temperatures in terms of variables which can be easily determined. Any formula method (Hayakawa, 1978) can be used to evaluate the corresponding processing time.

Although, these formulae were developed based on simple geometries (one-dimensional heat transfer shapes) covering extreme values of the parameter j (j is $4/\pi$ and 2 for an infinite slab and a sphere, respectively), it is believed they can be applied to any shape as long as the heat transfer into the product is by pure conduction. For simple geometries the parameter f_h can be calculated as a function of thermal diffusivity and dimensions, and eqn (9) is applicable. As an example, for a finite cylinder (Ball & Olson, 1957):

$$f_h = 0.398 / ((1/a^2 + 0.427/b^2)\alpha) \quad (12)$$

where a and b are the cylinder radius and half-height, respectively. Datta (1990) proved that for any arbitrary shape of a conduction-heated food the heat penetration curve has the classical form, so that it is always possible to determine the parameter f_h experimentally. This means that eqn (9) can be applied to products with any geometry (e.g. cubic particles, retortable pouches) as long as the surface heat transfer coefficient is infinite. However, validation of this statement is needed.

Heating medium come-up-time effect

To study the effect of come-up-time (CUT), 240 optimization problems were executed. Five different values of CUT, six Reduced Fourier numbers and four Z_q -values, in the range of interest, were combined (Table 4). The initial temperature of the product (because it has a minor influence on the optimal processing temperature) was fixed at 10°C and the F_t -value at 3 or 15 min, and a spherical shape was considered, because these were cases where the optimum could be more readily affected. Table 5 shows some results.

For products with a high Reduced Fourier number (i.e. products with a high heat transfer rate) the optimal processing temperature is more

TABLE 4
Input Data for the Simulations

<i>Variable</i>	<i>Values</i>					
RF ($s^{-1} \times 10^6$)	6.5	26.7	110.3	455.6	1881.0	7766.7
Z_0 ($^{\circ}C$)	15	25	35	45		
CUT (min)	0	3.75	7.5	11.25	15	

TABLE 5
Come-Up-Time Effect on the Optimal Processing Temperature (T_{op}) of a Spherical Product: $T_0 = 10^\circ\text{C}$; $F_t = 15$ min

RF ($s^{-1} \times 10^6$)	Z_q ($^\circ\text{C}$)	CUT (min)	C -value (min)	T_{op} ($^\circ\text{C}$)
6.5	15	0	2892.9	99.64
		15	2886.8	99.68
	45	0	1891.5	110.96
		15	1885.2	111.02
455.6	15	0	713.7	117.82
		15	637.6	119.36
	45	0	72.3	129.19
		15	59.1	132.11
7766.7	15	0	280.0	130.03
		15	258.3	129.28
	45	0	8.2	140.98
		15	16.7	137.84

sensitive to the come-up-time. However, the difference in optimal temperatures, for processes with and without come-up-time, is not larger than 2°C . Although differences of 3°C are observed for simulations with large RF numbers and Z_q -values, this is explained by the temperature insensitivity of the C -value which makes exact computation difficult.

Even though the come-up-time has a minor effect on the optimal processing temperature, the surface cook-value can be substantially reduced (especially for products with high RF numbers) with the related disadvantage of an increase in the total processing time and consequently a decrease in productivity.

CONCLUSIONS

Generalized (semi)-empirical formulae were obtained for calculating optimal sterilization temperatures (to minimize surface C -value) of conduction-heated foods, with infinite Biot number, as a function of F_t -value, Z -value of the quality index, initial temperature of the product and the empirical parameter f_h or the more theoretical Reduced Fourier number.

The formula of Hendrickx *et al.* (1989) was extended in order to include the cooling time in the lethality calculation.

The heating medium come-up-time and the initial product temperature have a minor influence on the optimal processing temperature. The come-up-time was not included as a variable in the developed formulae.

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