



Short communication

Predicting heat process efficiency in thermal processes when bacterial inactivation is not log-linear



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ABSTRACT

The food industry widely uses the F -value which considers microbial log-linear inactivation, while microbial heat inactivation may result in a non-log-linear inactivation pattern due to genetic or phenotypical heterogeneity. This may yield discrepancies in predicting microbial heat inactivation under dynamic conditions of heat treatment. In this paper, we suggest the calculation of the equivalent time of heat treatment at a given temperature to overcome these constraints. To validate our proposal, the heat inactivation of *Bacillus pumilus*, showing non-log-linear behavior, was predicted for 4 different heat inactivation profiles and bacterial enumeration was performed to determine whether prediction errors were acceptable. When the proportion of residuals in an acceptable zone from 1 log (fail safe) to 0.5 log (fail dangerous) was greater or equal to 70%, the model was considered as acceptable for predictions of the tested data. The new approach gave four different temperature profiles, with 96, 85, 85 and 100% of the residuals in the acceptable zone, indicating satisfactory prediction. Thus the proposed practical alternative to simulate microbial heat inactivation kinetics is able to extend the F -value to non-log-linear inactivation patterns.

1. Introduction

To respond to safety concerns as well as to consumer concerns, the food industry uses various food preservation methods (Leistner, 2000). Preservation methods ensure either microbial growth inhibition (Negi, 2012) or microbial inactivation (Mañas and Pagán, 2005). Thermal processing is the main preservation method used in the food industry. It aims to inactivate pathogen and spoilage microorganisms and/or enzymes which could alter food or make it unfit for human consumption. The effect of heat treatment on food is related to the time/temperature combination, the targeted microorganism and the food properties such as pH and a_w (Akterian et al., 1999). To assess the efficiency of heat treatments, food industries widely use the term F -value to define the intensity of thermal treatments under practical conditions. The F -value is defined as the number of minutes at a defined lethal reference temperature. To calculate the F -value, food manufacturers consider microbial log-linear inactivation. Nevertheless, the genetic or phenotypical heterogeneity of microbial populations may result in nonlinear inactivation and in this case, calculations of thermal death of microorganisms become more complicated (Smelt and Brul, 2014). This is

why, over the past few years, predictive microbiology has begun to propose more accurate models to describe non-log-linear inactivation kinetics (Corradini et al., 2006; Peleg and Pechina, 2000; Peleg et al., 2001; Valdramidis et al., 2011). In spite of that, the F -value remains the reference criterion in the food industry for its ease of use and the possibility to compare different temperature profiles. In addition most of the available microbial heat resistance data concern heat resistance parameters obtained in buffer under static conditions of heat treatment whereas the heat process step in food industries is non-isothermal. Thus despite heat treatments have been used for years for food safety and quality purposes, investigating thermal models that may accurately predict heat inactivation for non-isothermal heat treatments remains a challenge for the food industry. One way for improving the heat process step is to allow the use of non-log-linear heat resistance parameters acquired in isothermal conditions and the well-known F -value to predict heat inactivation kinetics under non-isothermal heating. Herein, we proposed a practical modelling method for transforming non-isothermal heating into isothermal heating which may be used for both log-linear and non-log-linear inactivation kinetics. To do so, we combined our practical method with the Weibull-type model and overcame

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the temperature variation by calculating the equivalent time of treatment at a reference temperature, using the z -value related to the microorganism. To the best of our knowledge, this is the first time that these models have been combined to simulate microbial heat inactivation under dynamic conditions, especially when heat inactivation is non-log-linear. This results in a practical method for the food industry to evaluate the impact of the heat process step, while continuing to use the well-known F -value parameter.

2. Material & methods

2.1. Microorganism and spore production

Bacillus pumilus E71 was chosen as a bacterial model for its shoulder inactivation behavior. The strain was stored in frozen aliquots stored at $-20\text{ }^{\circ}\text{C}$ consisting of nutrient broth supplemented with 50% (v/v) glycerol. From these aliquots, the strain was grown in Brain Heart Infusion Broth (Biokar Diagnostics, Beauvais, France) and sporulated in Sporulation Minimal Buffer following the protocol proposed by Baril et al., 2012. The Sporulation Minimal Buffer consists of phosphate buffer (pH 7, 30 mM) supplemented with CaCl_2 , 2 H_2O (8.0 mg/L) and MnSO_4 , H_2O (1.5 mg/L).

2.2. Heat inactivation under static heat treatment

Survival curves were obtained at static temperature in order to estimate heat resistance parameters. 10 μL of spores were suspended in 2 mL of buffered casein-peptone broth (10 g/L casein peptone, 5 g/L NaCl, 4.5 g/L K_2HPO_4 , 1.8 g/L KH_2PO_4 , pH = 7) yielding an initial concentration close to 7 log. Then, 100 μL of the suspension were introduced into capillary tubes, sealed, and immersed in a glycerol-water thermostatically-controlled bath. After given heating times, the tubes were cooled in a water bath and the content was collected to enumerate the surviving spores by plating in Brain Heart Infusion agar. The isothermal heat treatments were conducted at 68 $^{\circ}\text{C}$, 74 $^{\circ}\text{C}$, 77 $^{\circ}\text{C}$, 80 $^{\circ}\text{C}$, 83 $^{\circ}\text{C}$, 86 $^{\circ}\text{C}$, 89 $^{\circ}\text{C}$, 92 $^{\circ}\text{C}$, 95 $^{\circ}\text{C}$, 98 $^{\circ}\text{C}$ and 101 $^{\circ}\text{C}$ and led to at least a 3 log reduction of the bacterial population. These experiments were conducted using high inoculum levels (10^6 CFU per mL) and the limit of quantitation (LoQ) does not allow for a lower inoculum to be used. It should be borne in mind that the contamination levels obtained in real food are substantially different.

2.3. Estimation of spores' heat resistance

2.3.1. Primary model fitting

Two primary models were used to quantify the microbial resistance. These were fitted on each isothermal inactivation kinetic data.

The first-order model was the log-linear model (Bigelow, 1921):

$$\log_{10} N(t) = \log_{10} N_0 - \frac{t}{D} \quad (1)$$

The non-linear inactivation model was the Weibull model (Mafart et al., 2001):

$$\log_{10} N(t) = \log_{10} N_0 - \left(\frac{t}{\delta}\right)^p \quad (2)$$

where D is the classical decimal reduction time while δ is the first-decimal reduction time (min) and p is a shape parameter. A single p -value was estimated for the whole set of data. Indeed, Weibull model parameters are dependent and a single p -value estimated from the whole set of kinetics eliminates the structural correlation (Couvert et al., 2005). N_0 and N are the initial population and the survival population at time t , respectively.

The minimization of the sum of square error was used to fit inactivation kinetics with both models. The minimum values and the 95% confidence interval were computed with non-linear modules

(LSQCURVEFIT and NLPARCI, MATLAB R2016b, Optimization Toolbox, The Mathworks, Massachusetts, USA).

2.3.2. Primary model fitting performances

To evaluate the model-fitting performances the MSE_{model} and the AIC_C were computed. According to the mean square error of the model value (MSE_{model}), the lower the value is, the better the model is able to describe the experimental data (den Besten et al., 2006). The corrected Akaike Information Criterion (AIC_C) (Hurvich and Tsai, 1995) allows comparison of models by taking both the goodness of fit and the parsimony into account. The lower the AIC_C value, the better the model.

2.3.3. Secondary model fitting

The temperature effect is described by Eq. (3) (Hansen and Riemann, 1963) in which δ is the first decimal reduction time at the temperature T , δ_{ref} is the first decimal reduction time at the reference temperature T_{ref} , and z is the temperature increase leading to a ten-fold reduction of δ .

$$\log \delta = \log \delta_{ref} - \frac{T - T_{ref}}{z} \quad (3)$$

The model was fitted to the whole set of $\log \delta$ -values by minimizing the sum of square error using the *nlinfit* function (Optimization Toolbox; MATLAB R2016b; The Math-works). The 95% confidence interval was computed with the *nlparci* function (MATLAB R2016b, Optimization Toolbox, The Mathworks, Massachusetts, USA).

2.4. Prediction of spore heat inactivation kinetics under non-isothermal heating

2.4.1. Prediction

The heat treatment efficiency given by the decimal reduction number (n) depends on heating time (Eq. (4)). The non-linear inactivation curve supposes that n depends on time and δ -value:

$$n = \log\left(\frac{N_0}{N}\right) = \left(\frac{t}{\delta}\right)^p \quad (4)$$

However, depending on the shape parameter p , the *instant* changes with heat treatment time:

$$dn = p \cdot \delta^{-p} \cdot t^{p-1} \cdot dt \quad (5)$$

Eqs. (3) and (5) could be associated to predict the non-isothermal destruction. This has previously been carried out (Mafart et al., 2010; Peleg et al., 2001) but it led to a complex equation (Chen et al., 2007). We suggested here converting the non-isothermal treatment to an equivalent treatment in isothermal conditions knowing the impact on bacterial inactivation. Indeed, in view of the relationship between the time and the temperature (Eq. (3)), the dynamic temperature profile changes in an equivalent time at a reference temperature using the following integration:

$$t_{T_{ref}} = \int_0^t 10^{\frac{T-T_{ref}}{z}} \cdot dt \quad (6)$$

where $t_{T_{ref}}$ is the equivalent time at the temperature T_{ref} leading to the same thermal log reduction that is obtained by exposure to the non-isothermal treatment (T, t). z is the estimated z -value of the bacterial strain.

And according to a numerical integration:

$$t_{T_{ref}} = \int_{i=1}^m 10^{\frac{T_i - T_{ref}}{z}} \cdot \Delta t_i \quad (7)$$

T_i is the temperature of treatment at each time variation Δt_i .

Thus the evolution of the bacteria could be achieved combining Eqs. (4) and (6) as follows:

$$\log N(t_{Tref}) = \log N_0 - \left(\frac{t_{Tref}}{\delta} \right)^p \quad (8)$$

where N is the bacterial population at time t_{Tref} ; t_{Tref} is the equivalent time at the temperature T_{ref} ; δ is the first decimal reduction time and p is a shape parameter.

2.4.2. Performance evaluation of heat inactivation prediction

To validate the performance of the model (Eq. (8)), prediction errors (PE ; in log) or residuals for individual prediction cases were calculated as described by Oscar, 2009. Briefly, a PE value > 0 means that the model is fail-dangerous whereas a PE value lower than 0 means that the model is fail-safe.

$$PE = O - P \quad (9)$$

where O is the observed value (in log), P is the predicted value (in log). To determine whether individual prediction errors were acceptable, an acceptable prediction zone (APZ) was used (Oscar, 2005). The percentage of PE in the APZ was calculated and used as an overall measure of model performance.

$$\%PE = \left(\frac{PE_{in}}{PE_{total}} \right) \times 100 \quad (10)$$

where PE_{in} is the number of PE in the APZ and PE_{total} is the total number of PE in the evaluation. A $\%PE$ of $\geq 70\%$ indicated a tertiary model that provided acceptable predictions for a given data set (Oscar, 2005).

2.4.3. Experimental data obtained under non isothermal heating

Four non-isothermal heat treatments were tested (Fig. 1). The first profile was inspired by an industrial temperature profile used for the preservation of refrigerated processed foods of extended durability (Profile 1). This temperature profile was modified to simulate an “incident” during the process (Profile 2) in which the heating is interrupted for a few minutes before resuming the normal profile. The third temperature profile corresponds to a low increase in temperature encountered in the delicatessen baking process. The last profile (Profile 4) corresponds to a sigmoidal heat treatment. To perform each heat treatment, a thermostatically-controlled water bath was used (FD40-HE, Julabo, Seelbach, Germany). The temperature in the water bath was controlled by a Pt100 sensor. Capillary tubes with *Bacillus pumilus*

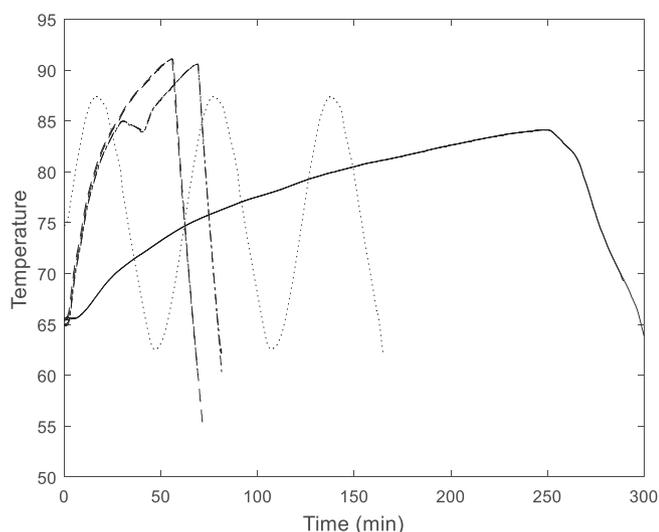


Fig. 1. Temperature profiles of mild heat processes. Profile 1 (–) inspired by an industrial process used for the preservation of Refrigerated Processed Food of Extended Durability. Profile 2 (– ·) follows the same outline as profile 1 with an interruption during heating. Profile 3 (continuous line) corresponds to a delicatessen baking process, and profile 4 (· ·) represents a sinusoidal heat treatment to try out the proposed approach.

E71 spores were prepared as explained above for the isothermal treatment, and immersed in the water bath. The heat treatment was stopped at different heating times by removing a capillary and cooling it in a cold water bath and enumerating spores on a BHI agar plate. The microbial enumeration was performed in two replicates for profile 1, profile 2 and profile 3 and once for profile 4.

3. Results

In this study, the heat resistance of *Bacillus pumilus* E71 spores was investigated under 11 static heating temperatures from 68 °C to 101 °C. The inactivation kinetics were fitted using a linear and a Weibull model. The fitting performances of these models, MSE_{model} and AIC_C , were calculated for each kinetic profile (Table 1). Comparison of the performance indices obtained for all temperatures shows that the inactivation data were best fitted using the Weibull model (Fig. 2A) as shown by the lowest MSE_{model} and AIC_C obtained with the Weibull model. As recommended by Couvert et al., 2005, the improvement of the fitting quality for the Weibull model was performed using a global p -value that was estimated with the whole set of data at 1.41 ± 0.09 . Thus a shoulder in heat inactivation kinetics was observed meaning that the bacterial resistance decreased gradually once the heat treatment had begun. The first decimal reduction time was higher than the second, the second higher than the third, and so on. That is why the linear model will overestimate the bacterial decrease at the beginning of the heat treatment, and underestimate it at a later stage.

To quantify the influence of temperature on bacterial resistance, the secondary Bigelow-like model was used. The sensitivity of spores to temperature variation was estimated by the z value, which represents the temperature increase leading to a 10-fold reduction of the D or δ values (Fig. 2B). The reference heat resistance at 100 °C was estimated at 0.52 min ($\log D = -0.28 \pm 0.05$) for the linear model and at 0.86 min ($\log \delta = -0.06 \pm 0.05$) for the Weibull model. The z value was estimated at $9.30 \text{ °C} \pm 0.26 \text{ °C}$ and at $9.35 \text{ °C} \pm 0.22 \text{ °C}$ for the linear and the Weibull model, respectively, with correlation coefficients equal to 0.999. The heat sensitivity z was not significantly affected by the primary model used ($p = 0.88$).

These heat resistance parameters were further used to predict the heat inactivation kinetics (Fig. 2) of the *Bacillus pumilus* spores submitted to the four studied temperature profiles. The concept of the proposed model is to overcome the temperature variation by calculating the equivalent time of treatment at a reference temperature, using the z -value related to the microorganism (Eq. (6)). The heat inactivation resulting from the in vitro simulation is shown in Fig. 3. The simulation was performed using Eq. (8) combined with a Weibull model and using heat resistance parameters previously determined in isothermal conditions, meaning that p was equal to 1.41, $\log \delta_{ref}$ to -0.06 and z to 9.30 °C . Similarly, Eq. (8) was combined with the linear model (p equal to 1) whereby $\log D_{ref}$ was equal to -0.28 .

In parallel to these simulations, spore enumerations were performed under the 4 heat temperature profiles (Fig. 3). According to Fig. 3A, bacterial enumeration was spread out around the prediction made using Eq. (8) combined with a Weibull model, whereas above they were predicted using the linear model. To further evaluate model prediction, an acceptable prediction zone (APZ) was defined (Oscar, 2005). The APZ was comprised between -1 and $+0.5 \log \text{ CFU} \cdot \text{mL}^{-1}$. The APZ was twice as wide in the fail-safe direction because greater error can be accepted in the fail-safe direction when the model is used to fulfill food safety criteria (Ross et al., 2000). Indeed if the observed value minus the predicted value is > 0 this means that microbial population prediction is underestimated or, in other words, that the impact of the heat treatment is overestimated. Fig. 3B represents the PE in the APZ as an overall measure of model performance. A percentage of $PE > 70\%$ indicated that the model provided acceptable predictions for the tested data set (Oscar, 2005). The acceptable value was based on the gold standard in the U.S. education system meaning $-1.0 \log < \text{acceptable}$

Table 1

Estimated primary inactivation models and performance criteria of studied models (MSE_{model} and $AICc$). The shape parameter of the Weibull model (p) was estimated for the whole set of kinetics to the unique value of 1.41 ± 0.09 .

T (°C)	Models	Estimated parameters \pm IC 95%			MSE	AICc
		N_0 (log CFU·mL ⁻¹)	D (min)	δ (min)		
68	Linear	7.4 ± 0.3	1421 ± 162		0.006	-93
	Weibull	7.0 ± 0.2		2254 ± 204	0.004	-115
74	Linear	7.9 ± 0.4	286 ± 34		0.007	-84
	Weibull	7.2 ± 0.2		485 ± 47	0.004	-137
77	Linear	7.6 ± 0.3	164 ± 28		0.002	-156
	Weibull	7.3 ± 0.2		236 ± 23	0.001	-219
80	Linear	7.5 ± 0.3	70.5 ± 8.0		0.007	-39
	Weibull	7.1 ± 0.2		114 ± 11	0.004	-96
83	Linear	7.4 ± 0.3	37.4 ± 4.9		0.008	-34
	Weibull	7.1 ± 0.2		60.6 ± 5.7	0.002	-196
86	Linear	7.7 ± 0.4	18.9 ± 2.8		0.007	-14
	Weibull	7.3 ± 0.2		30.0 ± 3.0	0.003	-102
89	Linear	7.4 ± 0.4	8.73 ± 1.38		0.004	-43
	Weibull	7.0 ± 0.3		13.8 ± 1.4	0.001	-217
92	Linear	7.3 ± 0.3	4.33 ± 0.55		0.005	-92
	Weibull	6.9 ± 0.2		6.87 ± 0.64	0.002	-180
95	Linear	7.9 ± 0.4	1.84 ± 0.24		0.006	-27
	Weibull	7.4 ± 0.3		3.10 ± 0.32	0.001	-160
98	Linear	7.7 ± 0.4	0.79 ± 0.11		0.006	-57
	Weibull	7.1 ± 0.3		1.33 ± 0.14	0.002	-172
101	Linear	7.6 ± 0.5	0.36 ± 0.07		0.003	-91
	Weibull	7.1 ± 0.3		0.58 ± 0.07	0.001	-177

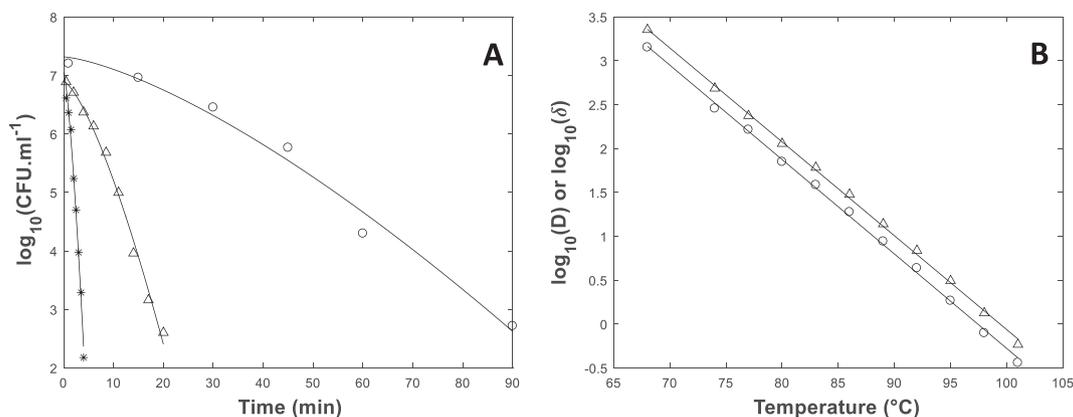


Fig. 2. A - Heat inactivation kinetics of *Bacillus pumilus* E71 at 86 °C (o), 92 °C (Δ) and 98 °C (*). Continuous curves correspond to the fitting of experimental data using a Weibull model. B - Decimal reduction time (○) estimated using a linear model and first decimal decrease (Δ) estimated using a Weibull model as a function of the temperature. Continuous lines correspond to estimated values of the secondary model.

$PE < 0.5$ log. For predictions made using the linear model, the percentage of PE in the APZ was lower than 70% in each studied case with 62%, 48%, 37% and 47% for heat profiles 1, 2, 3 and 4, respectively. Furthermore, looking at PE points (Fig. 3B), it was observed that the PE was only due to fail-dangerous prediction. Thus the linear concept appeared as not acceptable to predict bacterial inactivation with a shoulder as it overestimated the impact of the heat treatment. In contrast for the Weibull model, the percentage of PE in the APZ was equal to 96% for heat profile 1, 96% for profile 2 and 85% and 100% for profiles 3 and 4, respectively. Thus the proposed practical way combined with a Weibull model provided acceptable prediction whatever the dynamic heat treatment used.

4. Discussion

The term widely used to define the intensity of thermal treatment under practical conditions is the F -value. The F -value can be defined as the time or equivalent time taken to reduce initial microbial numbers, at a specified temperature, by a particular value, normally a multiple of the D -value for the target organism. This strategy to calculate the

sterility is based on the assumption that microbial heat inactivation follows a first-order kinetics and that the heat resistance D is log linear dependent on the temperature (z -value). Today, it is assumed that the success of the canning industry in using the F -value as a measure of heat treatment efficiency is mostly due to overprocessing rather than to the accuracy of the model. Various studies (Peleg et al., 2001; Peleg and Penchina, 2000; Periago et al., 2004; Valdramidis et al., 2011; Valdramidis et al., 2006) have pointed out the limits of this approach by underscoring the importance of non-log-linear microbial inactivation behavior and the use of methodologies for determining the model parameters which mainly use isothermal conditions (Geeraerd et al., 2005; Mafart, 2000; Periago et al., 2004; Baril et al., 2011; van Boekel, 2008). Indeed, most survival curves have non-log-linear behaviors and the main available heat resistance parameters were acquired under isothermal conditions. Furthermore, it has been shown that the Weibullian model can describe both isothermal and non-isothermal survival rates (Chen et al., 2007; Mafart, 2000; Mafart et al., 2001, 2010; Peleg et al., 2001; Peleg and Penchina, 2000; Valdramidis et al., 2011). The proposed practical way to extend the F -value to non-log-linear kinetics was shown to overcome these constraints by calculating the equivalent

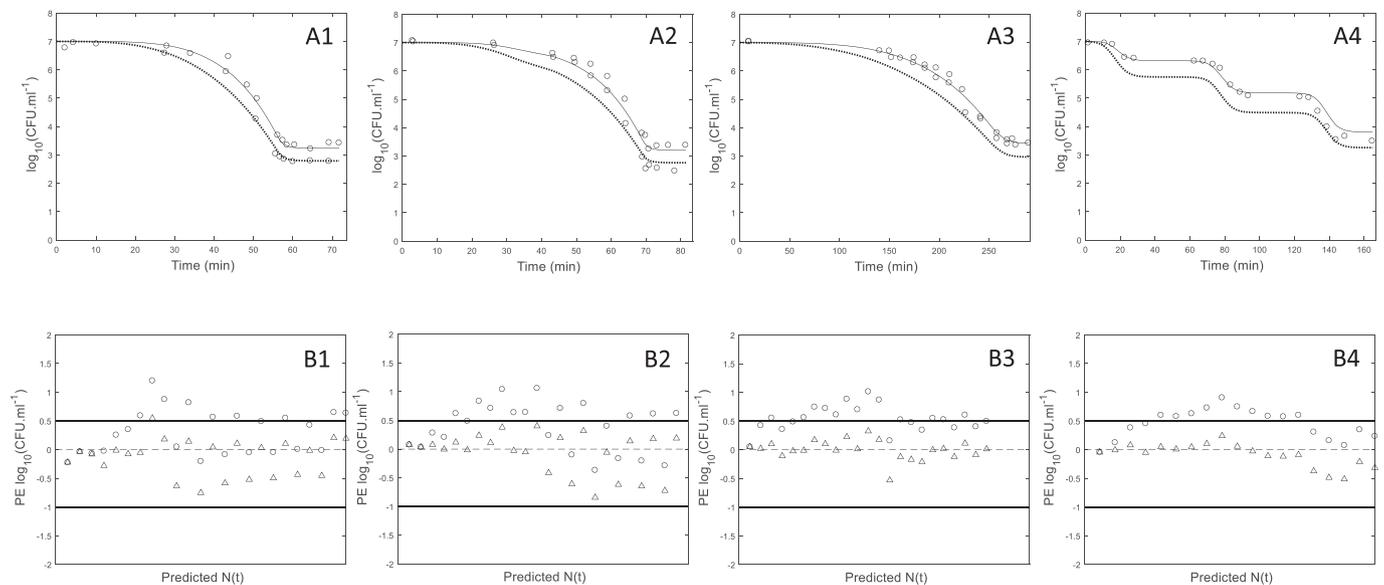


Fig. 3. A - Prediction of heat inactivation kinetics of *Bacillus pumilus* E71 for four dynamic temperatures: profile 1 (A1), profile 2 (A2), profile 3 (A3), and profile 4 (A4) using Eq. (8) combined with a linear model (dotted line) or Weibull model (solid line). B - Prediction Error (PE) plots for Eq. (8) combined with the Weibull (Δ) or the linear (\circ) model for the four dynamic temperatures. Solid lines are the upper and lower boundaries of the acceptable prediction zone.

time of treatment at a reference temperature, using the z-value related to the microorganism. One interesting point is that this approach could be further combined with a linear or a Weibull model and nothing prevents its expanded application to other nonlinear primary models. Nevertheless, one important point to bear in mind, before using heat resistance parameters for further simulation, is the quality of these parameters. For example, *Bacillus pumilus* E71 showed heat inactivation kinetics with a slight shoulder and the performances of both the linear and the Weibull model were sufficient to describe the inactivation (Table 1) and thus characterized heat resistance parameters. However, by using heat resistance parameters obtained with a linear model, further prediction leads to a “fail dangerous” situation for each tested condition, underscoring the necessity of accurately characterizing the bacterial inactivation. Of course the linear model appeared as a simpler concept to easily compare strain resistances since here the resistance is only dependent on temperature. Yet in real life, as shown by the different patterns of inactivation kinetics, heat resistance also depends on heating time. The impact of the time could be modeled by using a Weibull model which adds a shape parameter (p). This parameter is known to be independent of sporulation conditions, microbial physiological state or heating conditions (Baril et al., 2012; Desriac et al., 2013; Mafart et al., 2001). In the example of *Bacillus pumilus* E71 spores, the affined characterization of spore heat resistance allowed further satisfying prediction of inactivation under dynamic heat conditions.

As shown in this article, processing from a non-isothermal to an isothermal temperature profile simplifies the prediction of microbial kinetics, especially when inactivation is non-log-linear. Finally, this article offers a method to make the delta value independent of time, by modifying the time scale. Consequently, based on the modified time scale, the delta value remains constant, and the inactivation kinetics decrease linearly allowing the use of historical linear concepts. Moreover, no additional parameters are required and this method could be combined with different primary models to take different inactivation patterns into account. Furthermore this practical way to extend the F -value to non-log-linear kinetic could be easily integrated in a more general concept for heat prediction which takes other main environmental factors such as pH or aw into account (Mafart et al., 2010). The development of such a model would allow risk managers to estimate microbial contamination during and after heat processing steps and to

compare the efficiency of several processes or food formulations on the inactivation of a targeted microorganism. In terms of model choice, even if a linear model is acceptable, it is important to bear in mind that it may lead to an overestimation of the impact of the heat treatment.

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