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POPULATION MODEL OF BACTERIAL SPORES FOR VALIDATION OF DYNAMIC THERMAL PROCESSES¹

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ABSTRACT

Data are presented supporting a new model of spore populations during isothermal and dynamic lethal heat treatments. The model incorporates activation, injury, and preliminary inactivation of less-resistant fractions as well as the usual predominant inactivation. Rate constants of those transformations were determined experimentally for Bacillus subtilis strain A and were found to vary with temperature according to Arrhenius equations. Model generated and experimental isothermal survivor curves compared well. Comparison of model and experimental survivor curves for this species in a time-varying temperature regime showed the model to be a potentially good predictor of survivors during dynamic lethal heat treatment. The new model could be particularly important in simulating sterilization and pasteurization processes, especially short duration UHT treatments, and microbiological validation of arbitrary, dynamic thermal processes.

INTRODUCTION

Traditional design of thermal sterilization processes has been based on the assumption that thermal inactivation of bacterial spores predominates and can be modeled as a single, first order reaction (Bigelow 1921; Ball and Olson 1957). As such, the semilogarithmic survivor curve (logarithm of the number of surviving spores plotted against time of exposure) for constant lethal temperature

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should be described by a straight line. However, survivor curves for laboratory data frequently deviate considerably from a straight line early in an exposure interval (Stumbo 1965). That is a consequence of additional, concomitant processes such as heat activation and preliminary inactivation (early inactivation of less heat-resistant fractions). Deviations from a straight line response often make it difficult to test or validate thermal processes microbiologically.

The kinetics of thermal inactivation of bacterial spores were investigated by Wang *et al.* (1964) for high temperature, short time (HTST) treatments. The HTST treatments were applied to suspensions of *B. stearothermophilus* spores injected into a tubular flow reactor. The authors tested the Arrhenius equation, absolute rate theory, and thermal death time (TDT) curves and found the first two to be significantly better predictors of the temperature dependence of rate constants than the third.

Observed departures of survivor curves from logarithmic linearity have motivated a series of studies. Cerf (1977), in a review on deviations from the logarithmic order of death, mentioned several often-found deviations: shoulder, sigmoid, upward concavity, and a biphasic curve with a tail. The shoulder appears due to combined activation and inactivation. Vitalistic (individuals not identical, with a distribution of resistance attributes) and mechanistic (first order reaction) approaches to explaining the tail were discussed with guidelines and procedures for obtaining unquestionable survival curves.

The modeling of complex systems of irreversible first-order chemical reactions was investigated by Lee (1978); a lumped structure allowed synthesis of kinetically consistent systems of organic chemical reactions. Prokop and Humphrey (1970) presented a review paper on models of disinfection kinetics; the proposed models shared a consecutive-reaction mechanism. Shull *et al.* (1963) proposed consecutive reaction mechanisms for spore inactivation, postulating the existence of activated, intermediate and inactivated states. As a consequence, activation and inactivation were dependent, i.e., the spores had to be activated first and then inactivated. No biochemical basis for this assumption was presented. Hayakawa *et al.* (1981) and Hayakawa (1982) developed empirical equations for estimating nonlinear survivor curves for thermally vulnerable factors.

More recently, Kalinina and Motina (1984) developed a kinetic model for *B. stearothermophilus* that also assumed sequential reactions. Arabshahi and Lund (1985) published procedures for calculating kinetic parameters from experimental data. Toda and Aiba (1966) investigated the thermal characteristics of spore clumps and found that the increase in life span of a clump was proportional to the logarithm of the number of single spores within the clump. Aiba and Toda (1965) analyzed thermal death rates of bacterial spores and found that a normally distributed individual life span produced thermal death in a population of spores consistent with a first order reaction. The life span of a spore was defined as the

time a spore remained viable at a given temperature. The authors recommended supplementing the first-order reaction rate model with probability theory. Berry and Bradshaw (1982) compared lethalties predicted by Bigelow's model with microbiological enumerations of survivors for thermal sterilization processes with specified thermal histories. They found calculated lethalties to be as much as 7.2 min larger than experimental values and concluded the two sterilization values were not directly comparable.

From increasing knowledge about microbiological changes (transformations) during exposure of spores to high temperature, various transformations have been identified that may occur concurrently with thermal inactivation. Of these, the most significant are thermal activation (Keynan *et al.* 1965) and injury (Edwards *et al.* 1965a, b; Adams 1978; Hurst 1984). Those transformations significantly influence the number of survivors and they are not taken into consideration in the traditional mathematical model used in practice.

Based on the above work, Rodriguez *et al.* (1988) developed a new kinetic mode that better accounted for the situation extant in bacterial spore populations treated at lethal temperature. They hypothesized that a model consisting of activation and preliminary inactivation of dormant spores, as well as inactivation of activated spores (the traditional model), would significantly improve predicted survivor curves for commercial sterilization of food and pharmaceutical products. With experimentally determined estimates of the rate constants for all transformations in the model, predicted and experimental survivor curves compared well for suspensions of *C. botulinum* 62A heated in olive oil at 120 °C and *B. subtilis* heated in water at 93 °C.

The objectives of this research were (1) to establish preliminary relations for the temperature dependence of reaction rate constants of *B. subtilis* and (2) to examine further the potential of the new model with constant and dynamic (time-varying) thermal regimes.

MATERIALS AND METHODS

Description of Model

The model of bacterial spore populations proposed by Rodriguez *et al.* (1988) was developed using techniques of systems analysis and population dynamics described by Smerage (1979). The conceptual model is depicted in Fig. 1 and discussed only briefly here, since details are given in their paper. Circular symbols labeled N_1 and N_2 are stores representing, respectively, populations of mature, dormant spores and mature, activated spores. The former are potentially capable of producing colonies after activation; the latter are immediately capable of producing bacterial colonies in an enumeration medium. The store labeled N_3

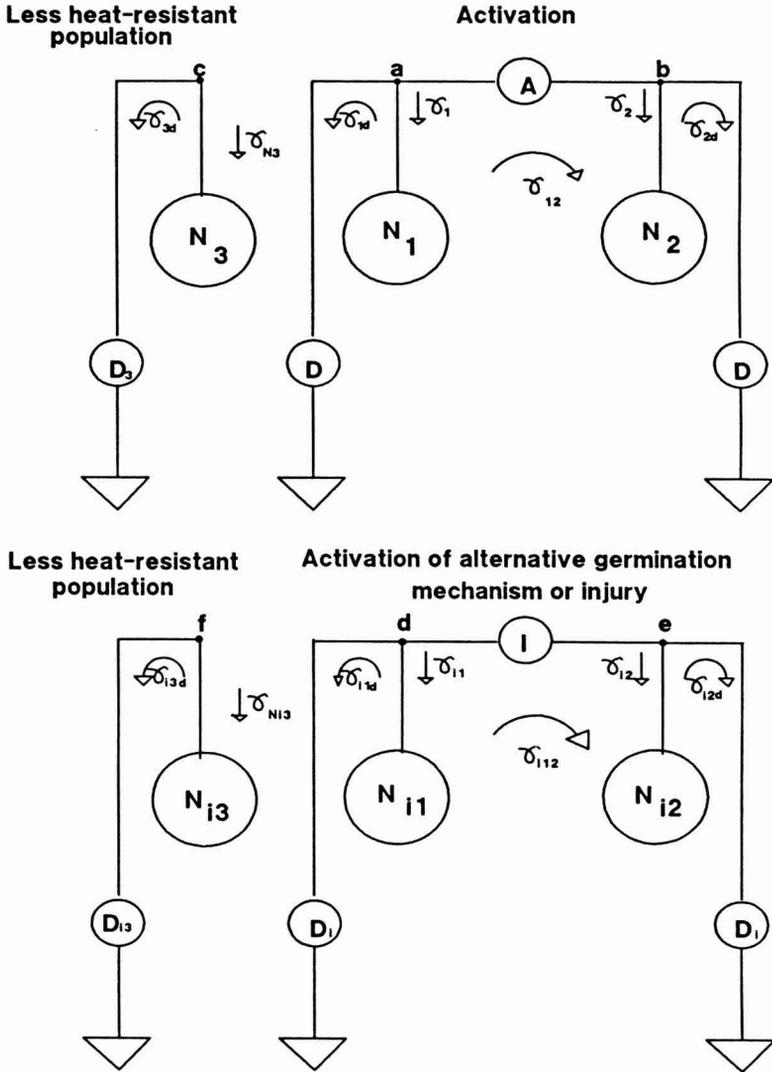


FIG. 1. SYSTEM DIAGRAM OF A BACTERIAL SPORE POPULATION EXPOSED TO LETHAL TEMPERATURE SHOWING THE NUMBER OF INDIVIDUALS (N) IN VARIOUS STAGES (CIRCLES) AND REACTION PATHWAYS (LINES) BETWEEN STAGES THAT CAN OCCUR SIMULTANEOUSLY AND INDEPENDENTLY. Gamma represents reaction rate (dN/dt) and triangles represent death sinks.

contains microorganisms present that are less resistant to high temperature and, therefore, inactivate much faster than members of N_1 and N_2 . They may be vegetative cells or, in the case of multiple genome bacteria, unusually heat sensitive spores. Upon exposure to lethal temperature, only one response, rapid inactivation, is available to them. Triangles in Fig. 1 denote sinks for nonviable spores. Maturation of spores during thermal processing would alter the dynamics of spore numbers in both N_1 and N_2 , but it was assumed to be negligible in these experiments.

Populations are linked by transformations representing reaction pathways through which individuals transfer from one stage to another. Two transformations were identified to be major determinants of the dynamics of spore populations, activation and inactivation. Inactivation renders spores incapable or reproducing in an enumeration medium. In contrast, activation enables spores to germinate and produce colonies. Both transformations were assumed to be independent and described by pseudo first-order reaction kinetics. In Fig. 1, transformation A represents activation of mature, dormant spores; D and D_3 represent inactivation of associated populations.

Thermal injury of bacterial spores during heat treatment may modify their requirements for growth and germination. In particular, an alternative germination mechanism may be activated by heat shock that enables *Bacillus subtilis* strain A spores to grow at 32 °C, whereas spores that have not received heat shock (normal spores) incubate best at 45 °C. Edwards *et al.* (1965b) showed that when no heat shock treatment was given, the optimum growth temperature for the strain of *B. subtilis* spores used in their work was 45 °C. Injury may also affect members of a less heat-resistant fraction. Spores affected by injury and relevant transformations are denoted in Fig. 1 by subscript i ; thus, nonactivated, activated, and less resistant fractions are denoted by N_{i1} , N_{i2} , and N_{i3} , respectively.

Only activated spores and the less heat-resistant fraction produce colonies when incubated in an enumeration medium; they are the survivors of heat treatments. Populations measured by microbiological enumeration are $N = N_2 + N_3$ for normal spores and $N_i = N_{i2} + N_{i3}$ for injured spores. Unactivated, viable spores (N_1 and N_{i1}) cannot be measured directly; a procedure to estimate their numbers in untreated populations is described later.

Once this conceptual model was established, the system was analyzed to derive the mathematical model given in Table 1. The solution to those equations for constant temperature is the summations of exponentials given in Table 2. It follows from Table 2 that the number of normal survivors during isothermal processing is given by

$$N(t) = N_{30} \exp(-K_{d3} t) + N_{10} [1 - \exp(-K_a t)] \exp(-K_d t) + N_{20} \exp(-K_d t) \quad (1)$$

where N_{10} , N_{20} , and N_{30} denote initial populations in N_1 , N_2 , and N_3 , respectively. Similarly, the number of survivor spores for which an alternate, injury-related germination mechanism has been activated is

$$N_i(t) = N_{i30} \exp(-K_{d3i} t) + N_{i10} [1 - \exp(-K_{ai} t)] \exp(-K_{di} t) + N_{i20} \exp(-K_{di} t) \quad (2)$$

with N_{i10} , N_{i20} , and N_{i30} denoting initial populations.

TABLE 1.
A NEW MATHEMATICAL MODEL OF NORMAL AND INJURED BACTERIAL
SPORE POPULATIONS EXPOSED TO LETHAL TEMPERATURES

$\frac{dN_1}{dt} = -(K_d + K_a) N_1$	$N_1(0) = N_{10}$
$\frac{dN_2}{dt} = K_a N_1 - K_d N_2$	$N_2(0) = N_{20}$
$\frac{dN_3}{dt} = -K_{d3} N_3$	$N_3(0) = N_{30}$
$\frac{dN_{i1}}{dt} = -(K_{di} + K_{ai}) N_{i1}$	$N_{i1}(0) = N_{i10}$
$\frac{dN_{i2}}{dt} = K_{ai} N_{i1} - K_{di} N_{i2}$	$N_{i2}(0) = N_{i20}$
$\frac{dN_{i3}}{dt} = -K_{d3i} N_{i3}$	$N_{i3}(0) = N_{i30}$

The first term in Eq. (1) describes rapid decay of the initial, less heat-resistant fraction, N_{30} ; it produces any initial, rapid decline in the number of survivors. The second term describes thermal activation and subsequent inactivation of dormant spores initially in N_1 . It is a pulse rising rapidly from an initial value of zero, peaking, and then decaying slowly toward zero. This term generates the "shoulder" of survivor curves. The third term describes inactivation of initially active spores, N_{20} ; it corresponds to the classical, single exponential model of thermal inactivation. Similar interpretation applies to Eq. (2) for injured spores.

Predictive applications of Eq. (1) and Eq. (2) require values of rate constants k_a , K_d , K_{d3} and initial conditions N_{10} , N_{20} , and N_{30} of the normal populations and similar parameters of injured populations prior to heat treatment. They are determined by fitting Eq. (1) and Eq. (2) to corresponding isothermal survivor curves from laboratory data. In this research, the Levenberg-Marquardt method of nonlinear regression (see the Appendix and Press *et al.* (1986)) was employed to fit the curves and estimate the initially unknown parameters. The Levenberg-Marquardt method requires preliminary estimates of all unknown parameters. Those estimates of initial populations and rate constants were obtained in this research by fittings Eq. (1) and Eq. (2) to survivor data using the method of successive residuals (Mohsenin 1978), a method particularly suited to an equation summing exponential with widely separated rate constants.

Application of the method of successive residuals to Eq. (1) and an isothermal survivor curve proceeds as follows under the assumption that $K_{d3} \gg K_a \gg K_d$, as is usually the case. Treatment of Eq. (2) and relevant data is similar. After an initial $5/K_a$ interval of isothermal heating, activation of N_1 and inactivation of the less heat-resistant fraction are completed and

$$N(t) \approx (N_{10} + N_{20}) \exp(-K_d t) \quad t > 5/K_a \quad (3)$$

The intercept and slope of a semi-log plot of this latter portion of the survivor curve provide $(N_{10} + N_{20})$ and K_d , respectively. Subtraction of Eq. (3) from the survivor curve Eq. (1), yields first residual N_{r1} defined by

$$N_{r1}(t) \equiv N(t) - (N_{10} + N_{20}) \exp(-K_d t) = N_{30} \exp(-K_{d3} t) - N_{10} \exp(-(K_a + K_d) t) \quad (4)$$

After the initial $5/K_{d3}$ interval, inactivation of the less heat-resistant fraction to N_{r1} is negligible and

$$N_{r1}(t) \approx -N_{10} \exp(-(K_a + K_d) t) \quad t > 5/K_{d3} \quad (5)$$

TABLE 2.
ISOTHERMAL SOLUTION OF THE MATHEMATICAL MODEL IN TABLE 1

$$N_1(t) = N_{10} \exp[-(K_d + K_a)t]$$

$$N_2(t) = (N_{10} + N_{20}) \exp(-K_d t) - N_{10} \exp[-(K_d + K_a)t]$$

$$N_3(t) = N_{30} \exp(-K_{d3} t)$$

$$N_{i1}(t) = N_{i10} \exp[-(K_{di} + K_{ai})t]$$

$$N_{i2}(t) = (N_{i10} + N_{i20}) \exp(-K_{di} t) - N_{i10} \exp[-(K_{di} + K_{ai})t]$$

$$N_{i3}(t) = N_{i30} \exp(-K_{d3i} t)$$

The intercept and slope of a semi-log plot of this latter portion of N_{r1} provide N_{10} and $(K_a + K_d)$, respectively. Subtraction of Eq. (5) from the first residual curve, Eq. (4), yields second residual N_{r2} defined by

$$N_{r2}(t) \equiv N_{r1}(t) - (N_{10} \exp(-K_d t)) = N_{30} \exp(-K_{d3} t) \quad (6)$$

The intercept and slope of a semi-log plot of N_{r2} provide N_{30} and K_{d3} , respectively.

After completion of the above steps, initial estimates of all six parameters in Eq. (1) are known and may be used in the Levenberg-Marquardt method to obtain final, and usually more accurate, estimates of those parameters. Microbiological enumeration of a sample of untreated suspension provides $N(0) = N_{30} + N_{20}$, which may be used in conjunction with the calculations described above.

Rodriguez estimated rate constants for suspensions of *C. botulinum* 62A heated in olive oil at 120 °C and *B. subtilis* heated in water at 93 °C and reported the methodology and results (Rodriguez *et al.* 1988). Research reported here utilized the same experimental and analytical procedures in two sets of laboratory experiments that determined for *B. subtilis* (1) the temperature dependence of rate constants and (2) how well the model predicted real responses to a dynamic thermal processes.

Experiment Design and Rationale

All experiments consisted of exposing *B. subtilis* spore suspensions to carefully controlled lethal thermal regimes, frequently sampling each suspension over the exposure interval, and incubating the sequences of samples at two different temperatures to produce survivor curves for both normal and injured spores. A key feature of the methodology was laboratory apparatus that allowed accurate control and recording of exposure time to lethal temperature. This apparatus, depicted in Fig. 2, consisted of a three-neck flask equipped with a needle thermocouple in one neck, a reflux condenser in the center neck, and a bundle of nine needles in the third neck. The purpose of the reflux condenser was to maintain constant volume of spore suspension in the flask during heating, while the needles in the third neck provided the means for injecting and withdrawing liquid samples without compromising sterility.

Experimental procedure attempted to minimize contamination of the medium and inaccuracy of measurement. Convective transport of spores in water vapor did not occur because the contents of the flask were not boiled. This was confirmed through visual observations recorded on videotape and temperature measurements above and below the water surface. Also, subsequent enumeration data showed no unusual numbers of survivors. Aluminum foil on the top of the condenser prevented contamination of the air above the flask contents, as was confirmed by the observed morphologies of colonies in the agar plates. A second potential difficulty, contamination of the suspension by the close proximity of inoculating and sampling needles did not occur because the inoculating needle was used only once (at the beginning) in an experiment, and it was withdrawn immediately thereafter. Furthermore, each sampling needle was immersed in the medium until withdrawn with a sample, and it was not reinserted or used again in the experiment.

The entire flask assembly rested on an electric heater controlled by a microcomputer through an analog-to-digital (a/d) amplifier board. The needle thermocouple in the flask was connected to the microcomputer for recording and controlling the temperature of the spore suspension. The suspension was continuously agitated by a magnetic stirrer to assure rapid, uniform mixing and heating of the relatively small volume (< 1%) of spore inoculum. Although mixing time was not measured, previous experience with similar apparatuses provided confidence that the suspension was thoroughly mixed before withdrawal of the first sample, even at high temperature. All timed events and the thermal history of the spore suspension during each experiment were accurately recorded by the computer. Additional details on laboratory procedure, materials, and methods are given in Rodriguez *et al.* (1988).

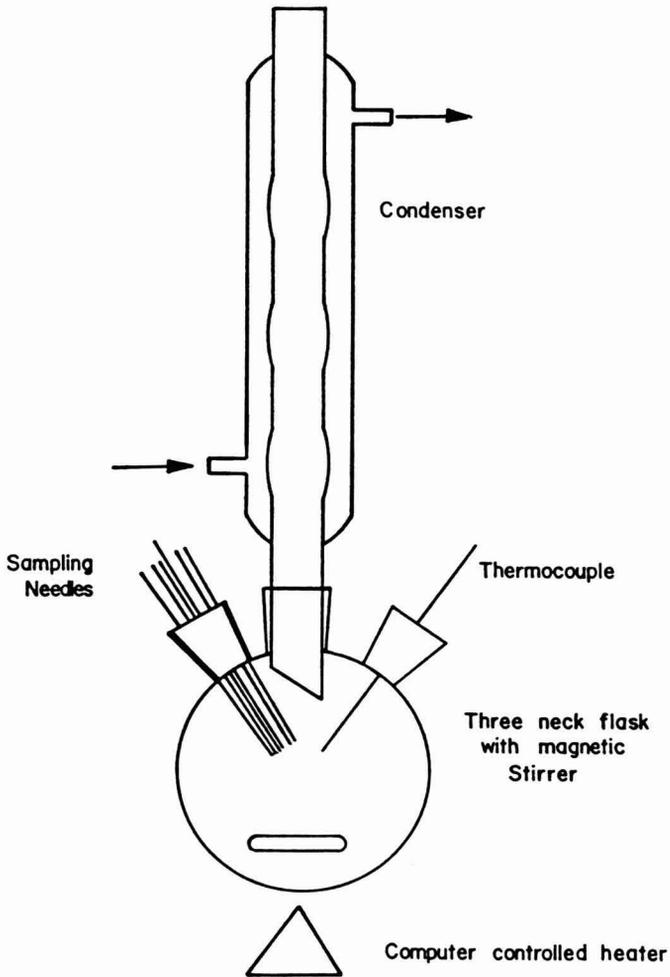


FIG. 2. SCHEMATIC OF LABORATORY APPARATUS SHOWING THREE-NECK FLASK USED AS REACTOR VESSEL FOR SUBJECTING SPORE SUSPENSIONS TO COMPUTER-CONTROLLED TIME-TEMPERATURE EXPOSURES

Temperature-Dependence of Rate Constants

Reaction rate constants of the model and their temperature dependencies were determined from isothermal survivor curves obtained at 87, 93, 95, and 99 °C. Identically prepared spore suspensions were exposed to each temperature in the three-neck flask reactor. Exposure time was selected to encompass the major

dynamics and to reduce the number of survivors in each suspension by approximately two orders of magnitude from the peak value. Eight samples were withdrawn from each suspension at intervals determined by preliminary experiments to be well spaced for defining the survivor curve over the exposure. Each of the four isothermal treatments was done once. However, an additional, independent series of four experiments was conducted at 95 °C to establish measurement accuracy; in each experiment, a sample was withdrawn from the suspension at the same time about midway through the exposure, incubated, and enumerated. Means and variances of normal and injured survivor counts in the four samples were calculated, and their standard errors of 20% and 30%, respectively, were considered estimates of the accuracy of the measurement procedure in all experiments.

From decimal dilution of each sample, aliquots were prepared for incubation at 45 °C, to enumerate survivor spores whose normal germination mechanism had been activated, and at 32 °C, for survivor spores whose injury-related mechanism had been activated (Rodriguez *et al.* 1988). Data for each constant temperature were plotted as experimental survivor curves of mean normal and injured *B. subtilis* spores over the exposure interval.

Reaction rate constants for normal and injured spores at each of the four constant temperatures were determined by fitting Eq. (1) and Eq. (2) to relevant experimental survivor curves as described in the subsection above. Graphs of each rate constant versus temperature were analyzed by regression to determine the relationship between the two variables. Survivor curves plotted from Eq. (1) and Eq. (2) for each temperature, using experimentally determined rate constants and initial populations, were compared with corresponding experimental curves as a test of the model.

All computations in this research were performed on personal computers using programs written by the authors in Turbo Pascal (Anon, 1983). One program implemented the procedures discussed above for estimating rate constants and initial conditions from isothermal experimental survivor curves; another determined parameters for Arrhenius descriptions of variations of rate constants with temperature. The criterion used for convergence of the Levenberg-Marquardt method was $< 10^{-14}$ change in the absolute value of Chi-squared as described in Press *et al.* (1986); the number of iterations required varied with temperature. The third program simulated by standard methods the model in Table 1 for specified rate constants, initial conditions, and experimental temperature regimes for both isothermal and dynamic (see below) processes. The time step of integration was variable, ranging from 13.5 to 18 s.

Response to a Dynamic Thermal Process

The apparatus previously described was used to subject a suspension of *B. subtilis* spores (from the same stock used throughout all previous tests) to a

dynamic thermal process. The three-neck flask, containing sterile distilled water, was heated to 99 °C and injected with *B. subtilis* spore suspension at time zero. Then, under computer control, the heater was cycled on and off to achieve a dynamic temperature history between 85 and 99 °C for 2.5 h. That exposure permitted a full cycle of population dynamics, with the final number of survivors approximately two orders of magnitude below the peak value. The actual temperature of the suspension was recorded by the computer from the thermocouple in the flask. Samples withdrawn from the flask at intervals determined by preliminary experiments to be well spaced during the exposure were prepared for enumeration, as described above, to determine the number of survivors in the suspension at each sample time.

With the recorded experimental time-temperature history as input, population response curves of surviving normal and injured spores were predicted by computer simulation of the mathematical model. During simulations, the rate constants of activation and inactivation were varied with the recorded temperature regime according to the relationships established in the isothermal experiments. Initial populations used were averages of initial populations calculated during the four isothermal experiments for rate constant determination. Model predicted and experimental survivor curves were compared to establish the model's accuracy and potential for use in microbiological validation of commercial thermal processes.

RESULTS AND DISCUSSION

The *B. subtilis* spore suspension used throughout these experiments did not exhibit a fraction (N_3) less resistant to high temperature. That was confirmed by the absences of preliminary inactivation in all isothermal survivor curves and vegetative cells and phase dark spores in untreated samples inspected with a phase contrast microscope. Accordingly, initial population $N_{30} = 0$ and $N_{i30} = 0$ were assumed throughout this work, and Eq. (1) and Eq. (2) reduced to

$$N(t) = (N_{10} + N_{20}) \exp(-K_d t) - N_{10} \exp(-(K_a + K_d)t) \quad (7)$$

and

$$N_i(t) = (N_{i10} + N_{i20}) \exp(-K_{di} t) - N_{i10} \exp(-K_{ai} + K_{di})t \quad (8)$$

Only K_a , K_d , N_{10} , and N_{20} for normal spores and their counterparts for injured spores could be determined from isothermal survivor curves; K_{d3} and K_{d3i} did not apply. Direct microscopic counts of untreated suspension equaled N_{20} and N_{i20} , so N_{10} and N_{i10} were easily calculated, as discussed above for Eq. (3) from $(N_{10} + N_{20}) - N_{20}$ and $(N_{i10} + N_{i20}) - N_{i20}$. Samples were incubated at 45 °C to enumerate normal survivor spores or at 32 °C to enumerate injured survivors.

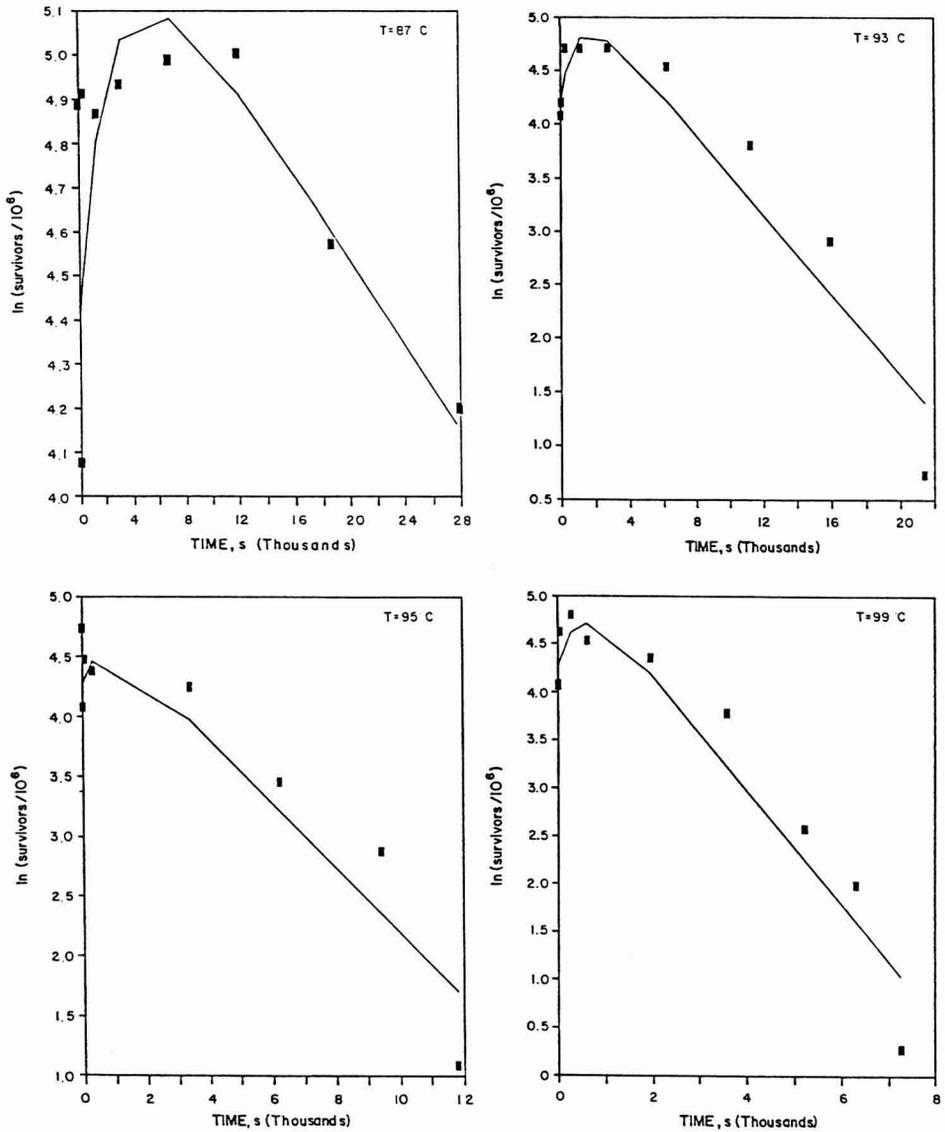


FIG. 3. SURVIVOR CURVES SHOWING $\ln(\text{SURVIVORS}/10^6)$ VS TIME (s/ 10^3) AT FOUR LETHAL TEMPERATURES (T) PREDICTED BY NEW KINETIC MODEL (____) SUPERIMPOSED ON EXPERIMENTAL DATA (■) FOR NORMAL SPORES OF *BACILLUS SUBTILIS* -STRAIN A INCUBATED AT 45°C PRIOR TO ENUMERATION

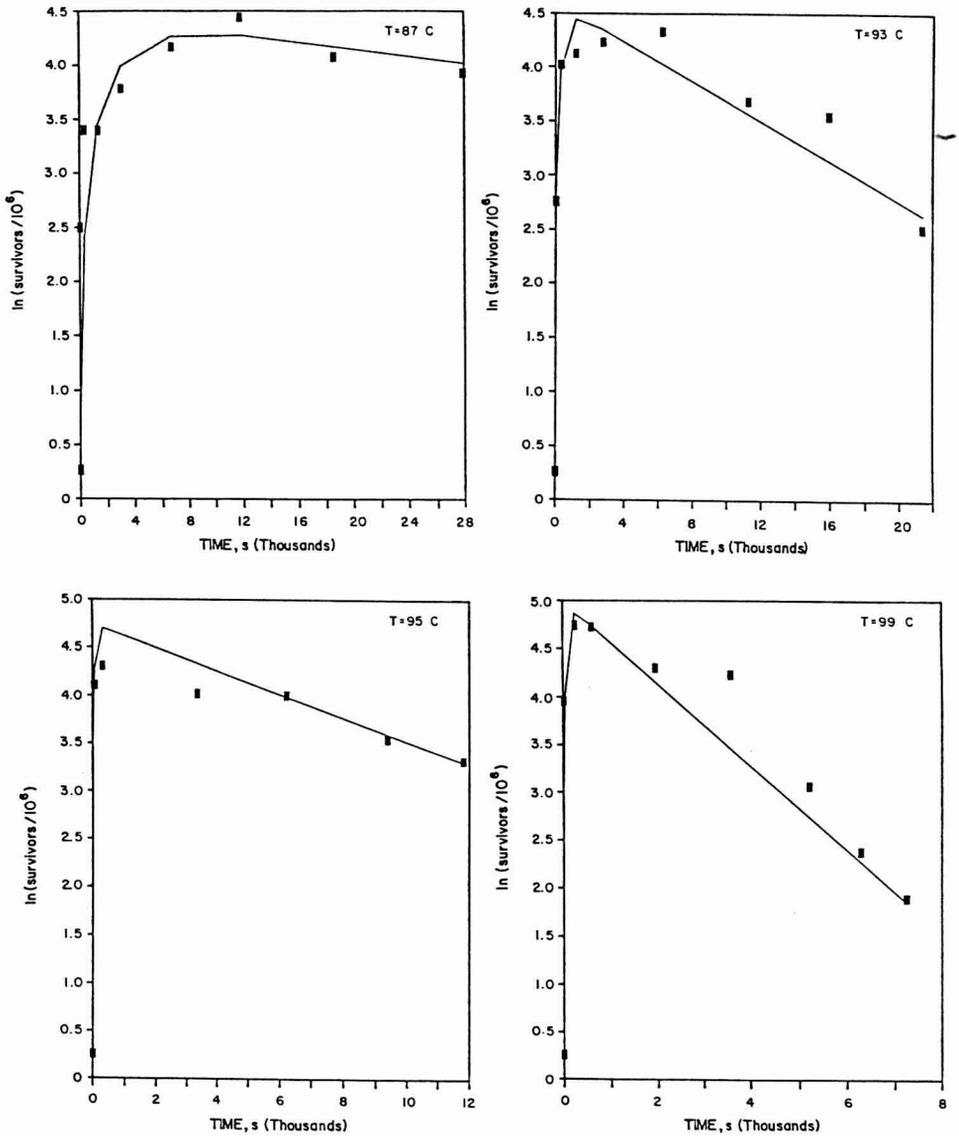


FIG. 4. SURVIVOR CURVES SHOWING $\ln(\text{SURVIVORS}/10^6)$ VS TIME ($s/10^3$) AT FOUR LETHAL TEMPERATURES (T) PREDICTED BY NEW KINETIC MODEL (____) SUPERIMPOSED ON EXPERIMENTAL DATA (■) FOR INJURED SPORES OF *BACILLUS SUBTILIS* - STRAIN A INCUBATED AT 32 °C PRIOR TO ENUMERATION

Isothermal Survivor Curves

Since microbiological enumeration data were obtained at two incubation temperatures (to reveal both normal and injured spores) for each of four constant treatment temperatures, a total of eight survivor curves were generated from the laboratory experiments. Experimental curves for normal spores at the four temperatures are shown in Fig. 3; those for injured spores are shown in Fig. 4. Each curve is defined by eight data points marked by dark rectangles corresponding to the eight samples taken during an exposure.

In all isothermal experiments, activation dominated early in the treatments, as evidenced by large initial increments in the numbers of survivors. However, at higher temperatures, samples could not be taken sufficiently frequently to demonstrate well how the rapid dynamics of activation affected initial increments in survivors. It followed that estimated parameters and measured and simulated populations were not as accurate as they might otherwise have been.

Parameters K_a , K_d , N_{10} , and 87, 93, 95, and 99 °C and counterparts for injured spores were estimated by fitting Eq. (7) and Eq. (8) to the experimental survivor curves with the successive residual and Levenberg-Marquardt procedures discussed earlier and illustrated in Table 3 for normal spores at 93 °C. Stability of Levenberg-Marquardt nonlinear regression required scaling of time and population variables to $X = t/10$ and $Y = N/10^6$. With those changes Eq. (7) became

$$Y(X) = (Y_{10} + Y_{20}) \exp(-K'_d X) - Y_{10} \exp(-(K'_a + K'_d) X) \quad (9)$$

where $Y_{10} = 10^{-6}N_{10}$, $Y_{20} = 10^{-6}N_{20}$, $K_a = 10 K'_a$, and $K_d = 10 K'_d$. Similar scaling was applied to Eq. (8). This scaling also reduced computer round-off error.

Survivor curves calculated from Eq. (7) and Eq. (8) with estimated values of the rate constants and initial populations are plotted as solid curves in Fig. 3 and 4 for comparison with the experimental data. They compared well except early in exposures due to inadequate sampling frequency then. A scatter diagram of all corresponding calculated and experimental population numbers had a linear regression equation with 0.9634 correlation coefficient.

Temperature Dependence of Rate Constants

Graphs of natural logarithms of experimental K_a , K_d , K_{ai} , and K_{di} for normal and injured *B. subtilis* spores versus inverse absolute temperature ($1/T$) are shown in Fig. 5. They were observed to follow the Arrhenius relationship

$$\ln K = A + m(1/T) = A + (-E_a/R) (1/T) \quad (10)$$

TABLE 3.
ESTIMATION OF PARAMETERS FOR NORMAL SPORES (45 °C INCUBATION)
OF *B. SUBTILIS* -STRAIN A WITH THE EXPERIMENTAL SURVIVOR DATA
FOR 93 °C LETHAL TEMPERATURE

Time (s)	Survivors (million)	X=time/10	Y=survivors/10 ⁶	ln Y
0	59	0	59	4.08
60	67	6	67	4.205
300	111	30	111	4.71
1140	111	114	111	4.71
2760	112	276	112	4.72
6240	94	624	94	4.54
11280	45	1128	45	3.81
15960	18.4	1596	18.4	2.91
21420	2.08	2142	2.08	0.732

$$Y(X) = Y_{10} \exp(-(K'_a + K'_d)X) + (Y_{10} + Y_{20}) \exp(-K'_d X)$$

$$\text{where } Y_{10} = 10^{-6} N_{10}, Y_{20} = 10^{-6} N_{20}, K'_a = 10 K_a, \text{ and } k'_d = 10 K_d.$$

Linear regression of last six data points with $\ln Y = \ln(Y_{10} + Y_{20}) - K'_d X$ yields

Intercept $\ln(Y_{10} + Y_{20})$	5.393	$Y_{10} + Y_{20} = 219.8$
Std Err of Y estimate	0.571	
R ²	0.892	
slope $-K'_d$	-0.00186	
Std Err of slope	0.000323	

Levenberg-Marquardt nonlinear regression using the initial estimates above of $Y_{10} + Y_{20}$ and $-K'_d$ yields the estimate

$$Y(X) = 156.6 \exp(-0.00915 X) + 219.0 \exp(-0.00186 X)$$

from which, with reversal of the scaling, the desired parameters are estimated to be

$$K_a = (0.00915 - 0.00186)/10 = 0.000728$$

$$K_d = 0.00186/10 = 0.000186$$

$$N_{10} = 156.6 \times 10^6$$

$$N_{20} = (219.0 - 156.6) \times 10^6 = 62.4 \times 10^6$$

a straight line with slope $m = -E_a/R$ and intercept A , where E_a = activation energy and $R = 8.314 \text{ J/g-mol K}$ is the universal gas constant. The logarithmic form was chosen for ease of use in simulations. Slopes, intercepts, and activation energies for Arrhenius descriptions of the four rate constants of *B. subtilis* were determined by regressing Eq. (10) to each of the four experimental graphs; they are listed in Table 4.

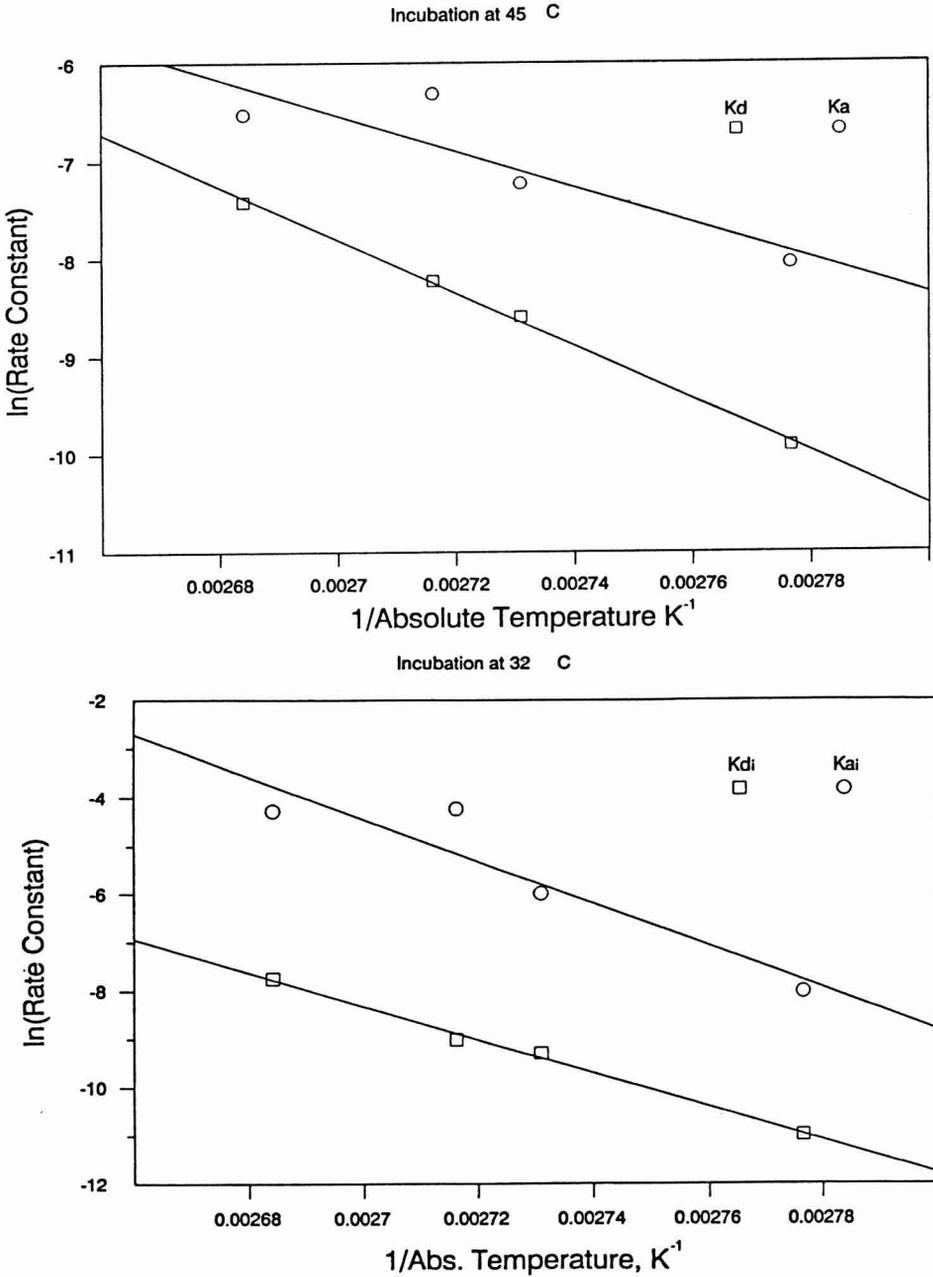


FIG. 5. ARRHENIUS PLOTS SHOWING TEMPERATURE DEPENDENCE OF PREDOMINANT INACTIVATION RATE CONSTANTS K_d and K_{di} AND ACTIVATION RATE CONSTANTS K_a and K_{ai} FOR NORMAL (UPPER) AND INJURED (LOWER) SPORES OF *BACILLUS SUBTILIS* - STRAIN A ENUMERATED AFTER INCUBATION AT 45 and 32 °C, RESPECTIVELY. Data (■) and regression lines (—) are shown.

TABLE 4.
SLOPE (m), INTERCEPT (A), ACTIVATION ENERGY (E_a), AND CORRELATION
COEFFICIENT (R) FOR ARRHENIUS TEMPERATURE DEPENDENCE OF
REACTION RATE CONSTANTS FOR *B. SUBTILIS* EXPOSED TO LETHAL TEMPERATURE

Reaction	m (°K)	A	E_a (J/g-mol)	R
activation*	-17809	41.50	1.48×10^8	-0.928
Inactivation*	-27201	65.64	2.26×10^8	-0.999
Activation**	-43568	113.20	3.62×10^8	-0.935
Inactivation**	-34529	84.90	2.87×10^8	-0.997

* normal spore; incubation at 45 C

** injured spore; incubation at 32 C

Dependence of inactivation rate constants on temperature was described well by Arrhenius equations for both normal and injured *B. subtilis* spores; dispersion of the data was very low. However, the previously mentioned inability to sample sufficiently often early in high temperature treatments caused data and equations to differ significantly at 115 and 120 °C and emphasized the importance of rapid sampling early in high temperature treatments to accurate survivor curves and activation rate constants. Although estimates of activation rate constants were not as accurate as for inactivation, the magnitudes of correlation coefficients were higher than 0.9 and all estimates should be good enough for most practical purposes.

For the range of temperature used in this study, the Bigelow method for expressing temperature dependency of thermal death rates could have been used instead of the Arrhenius description with similar results. In that method, each rate constant, K , would have been expressed in terms of decimal reduction time $D = 2.303/K$, and its temperature dependence would have been expressed by a Z -value instead of activation energy. However, for integrity of the theories of chemical and biological system dynamics underlying this research and for wider and higher ranges of temperature, use of rate constants and Arrhenius descriptions was preferred.

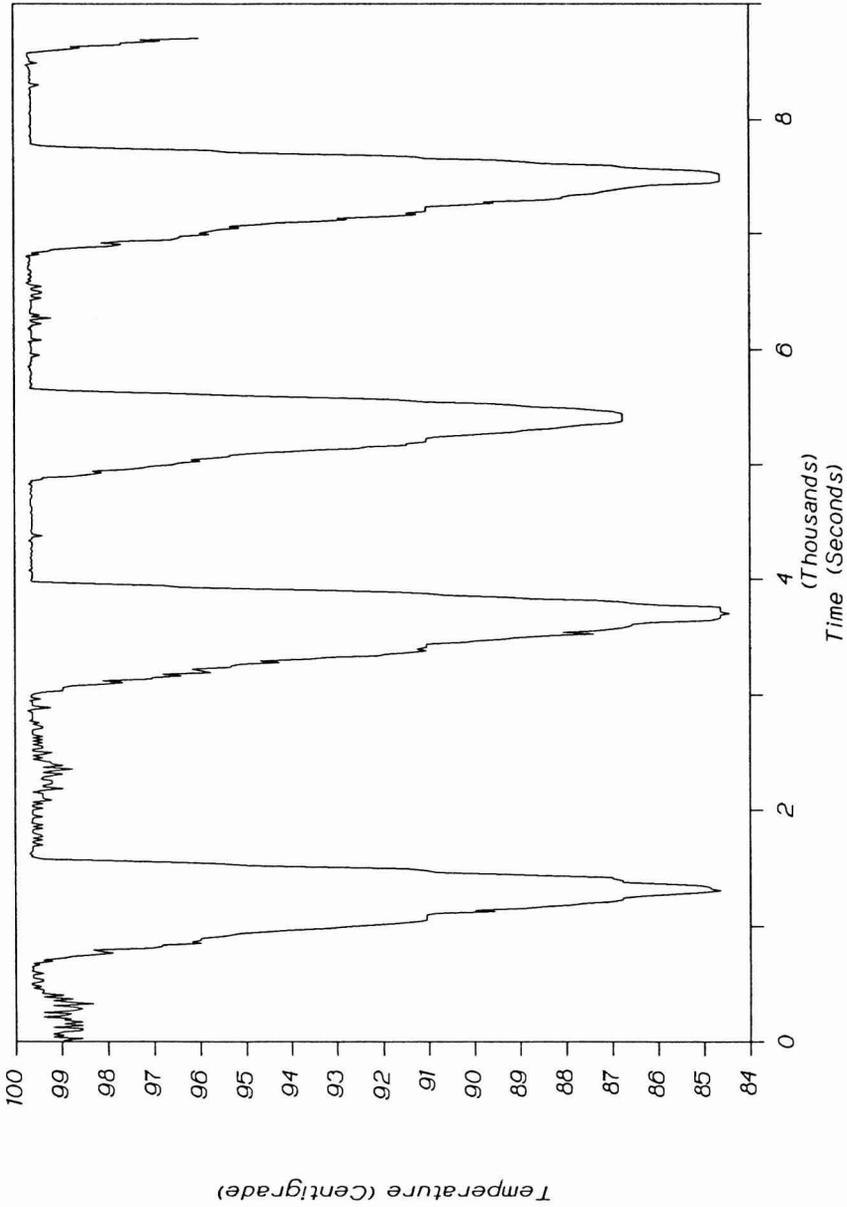


FIG. 6. TEMPERATURE HISTORY EXPERIENCED BY *B. SUBTILIS* SPORES DURING DYNAMIC ON-OFF HEATING PROCESS

Response to a Dynamic Thermal Process

A suspension of *B. subtilis* spores was exposed for 2.5 h to the dynamic thermal process shown in Fig. 6, and the responses of normal and injured spore populations were measured by incubation and microbiological enumeration of samples withdrawn from the suspension at eight instants well spaced over the exposure. Corresponding model responses to the temperature history in Fig. 6 were obtained by simulating the mathematical model with the four initial populations listed in Table 5; they are shown in Fig. 7. Note the significant initial increases in numbers of survivors due to activation, the variations with temperature of the rates of subsequent decreases, and the plateaus when the temperature was below 87°C. The quality of model predictions was demonstrated by the scatter diagram in Fig. 8 of pairs of model predicted and experimental population numbers at corresponding instants. The numbers were always the same order of magnitude with both positive and negative deviations from the ideal.

TABLE 5.
INITIAL POPULATIONS OF VIABLE *B. SUBTILIS* - STRAIN A SPORES, BY STAGE,
FOR SIMULATION ANALYSES, CALCULATED AS MEANS OF VALUES
DETERMINED FOR THE FOUR ISOTHERMAL EXPERIMENTS

<u>Spore Stage</u>	<u>Mean</u> (million)	<u>Std. dev.</u> (million)
Normal Activated, N ₂	69.75	5.16
Normal Nonactivated, N ₁	136.75	44.32
Injured & Activated, N _{i2}	1.75	0.43
Injury-susceptible, N _{i1}	112.0	22.76

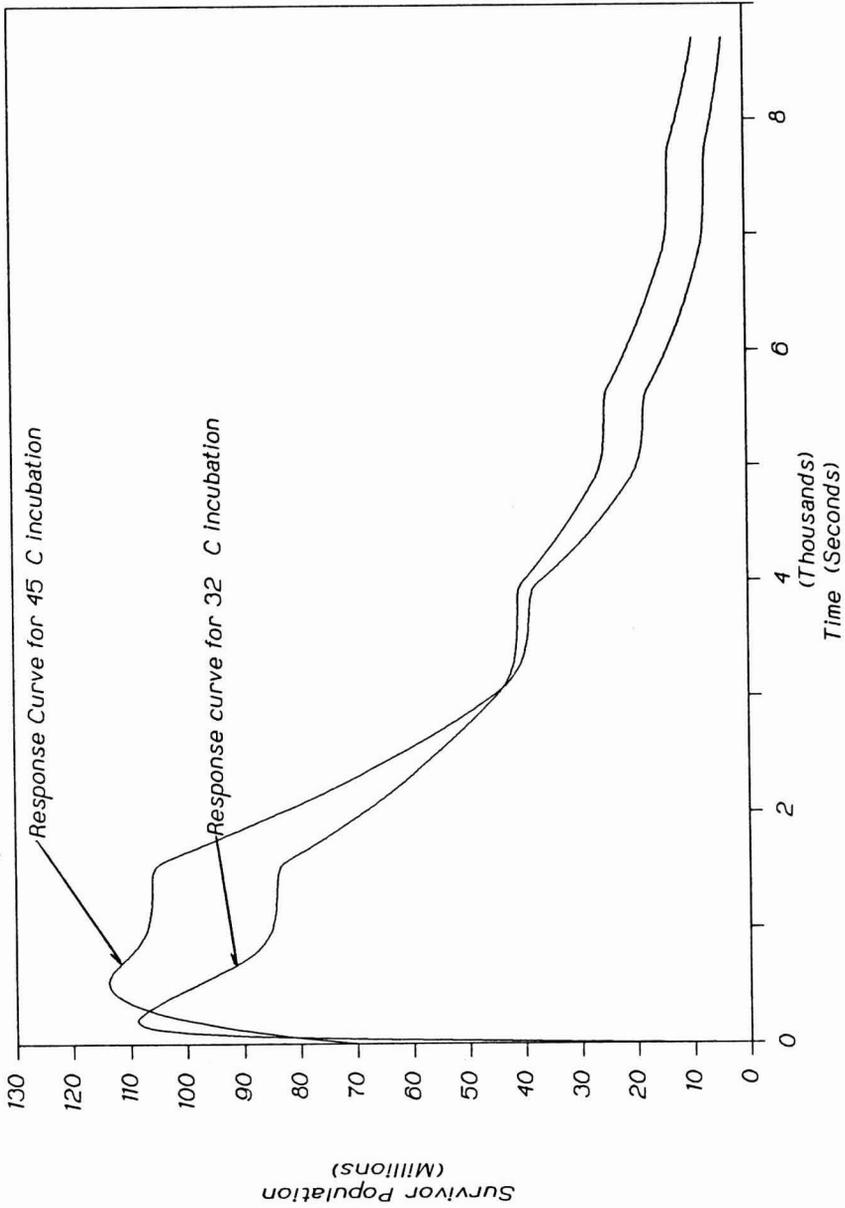


FIG. 7. SURVIVOR RESPONSE OF NORMAL AND INJURED *B. SUBTILIS* SPORES TO TEMPERATURE HISTORY OF PROCESS IN FIG. 6.

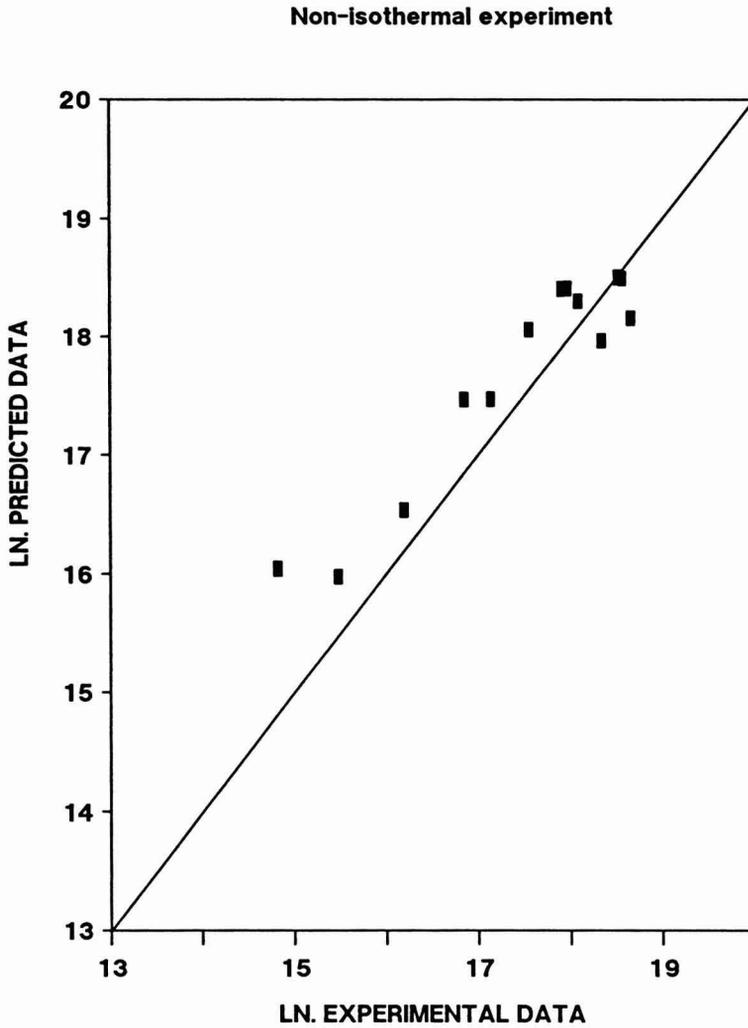


FIG. 8. SCATTER DIAGRAM COMPARING MODEL PREDICTED AND EXPERIMENTALLY MEASURED NUMBERS OF SURVIVOR SPORES AT VARIOUS INSTANTS DURING THE DYNAMIC TEMPERATURE PROCESS IN FIG. 6.

Comparison with Traditional Model

The traditional model of a population of normal spores (free of less heat resistant fraction N_3) at lethal temperature pertains only to size N of the subpopulation of surviving, activated spores affected by a single process, inactivation. It applies, therefore, only to the declining phase of the subpopulation and is given by

$$\frac{dN}{dt} = -K_{dt} N \quad (11)$$

with rate constants K_{dt} and initial population $N(0) = N_0$.

The two-process model for the same population pertains to subpopulations N_1 and N_2 of mature, dormant spores and mature, activated spores, respectively; it involves activation in addition to inactivation. From Table 1, it is given by

$$\frac{dN_1}{dt} = -(K_d + K_a) N_1 \quad (12)$$

$$\frac{dN_2}{dt} = K_a N_1 - K_d N_2 \quad (13)$$

with rate constants K_a and K_d and initial populations N_{10} and N_{20} . Survivor population $N = N_2$ has, contrary to the traditional model, the potential to both increase and decrease and applies throughout the exposure, not just the declining phase. In practice, K_{dt} and K_d may not equal due to the influences discussed below of activation on survivor response and different regression analyses for the two models.

Comparison of survivor population responses of the two models is insightful and done here in the absence of less heat resistant fraction N_3 for the dynamic temperature regime in Fig. 6. Response curves of the two models are given in Fig. 9 and 10 for normal and injured spore populations, respectively. Two curves in each figure pertain to the traditional model for different initial conditions, one for $N_0 = N_{20}$ and another for $N_0 = N_{10} + N_{20}$. Specification of N_0 for the traditional model often has plagued food microbiologists. Since N pertains to activated spores only, $N_0 = N_{20}$ should be chosen. However, Fig. 9 and 10 indicate that choice may cause the traditional model to predict a significantly low population over time because initially inactive spores, N_{10} , present at the beginning of heat treatment quickly activate and add to the population inactivated during the subsequent slow decline. This initial increment in the activated spore population may be large and cause the shoulder often observed in data, with the size of the shoulder reflecting the relative sizes of N_{10} and N_{20} .

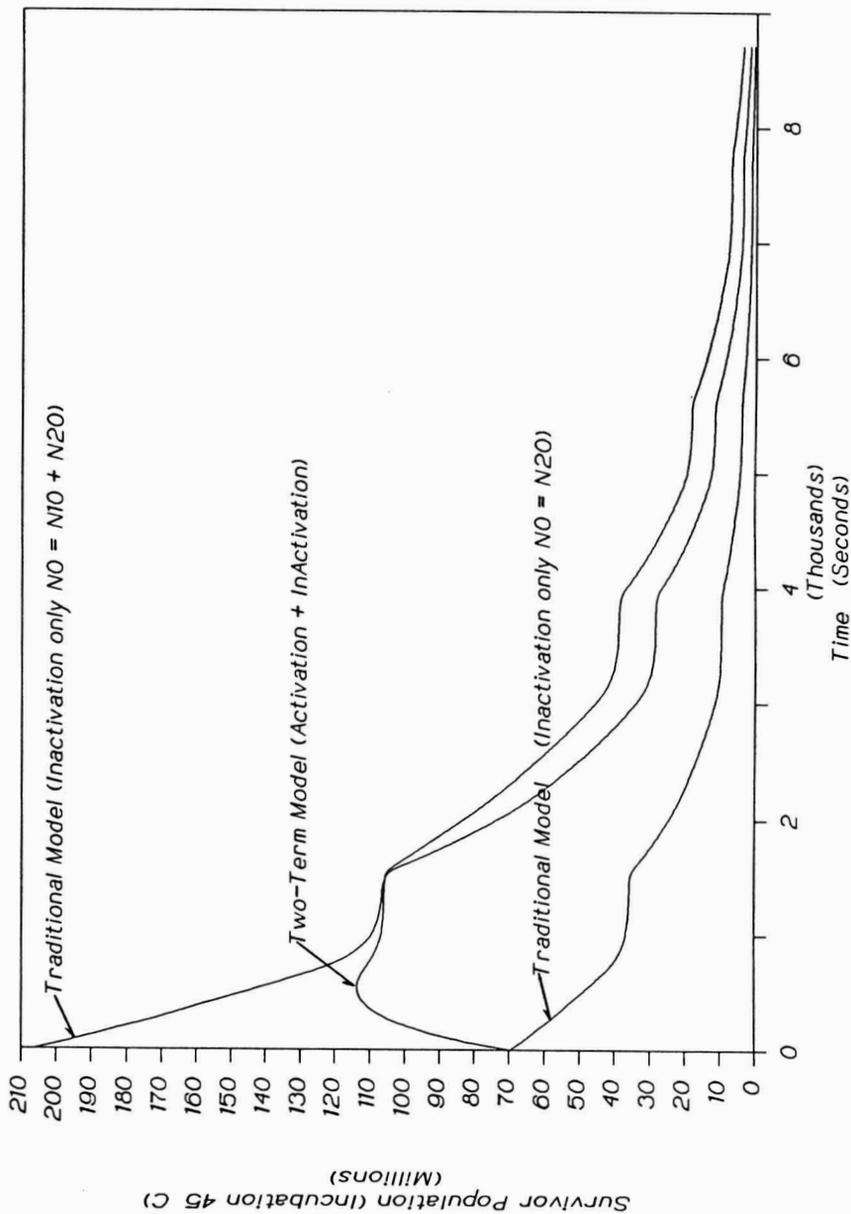


FIG. 9. POPULATION RESPONSES OF TRADITIONAL AND TWO-PROCESS MODELS FOR NORMAL *B. SUBTILIS* SPORES EXPOSED TO THE TEMPERATURE HISTORY IN FIG. 6.

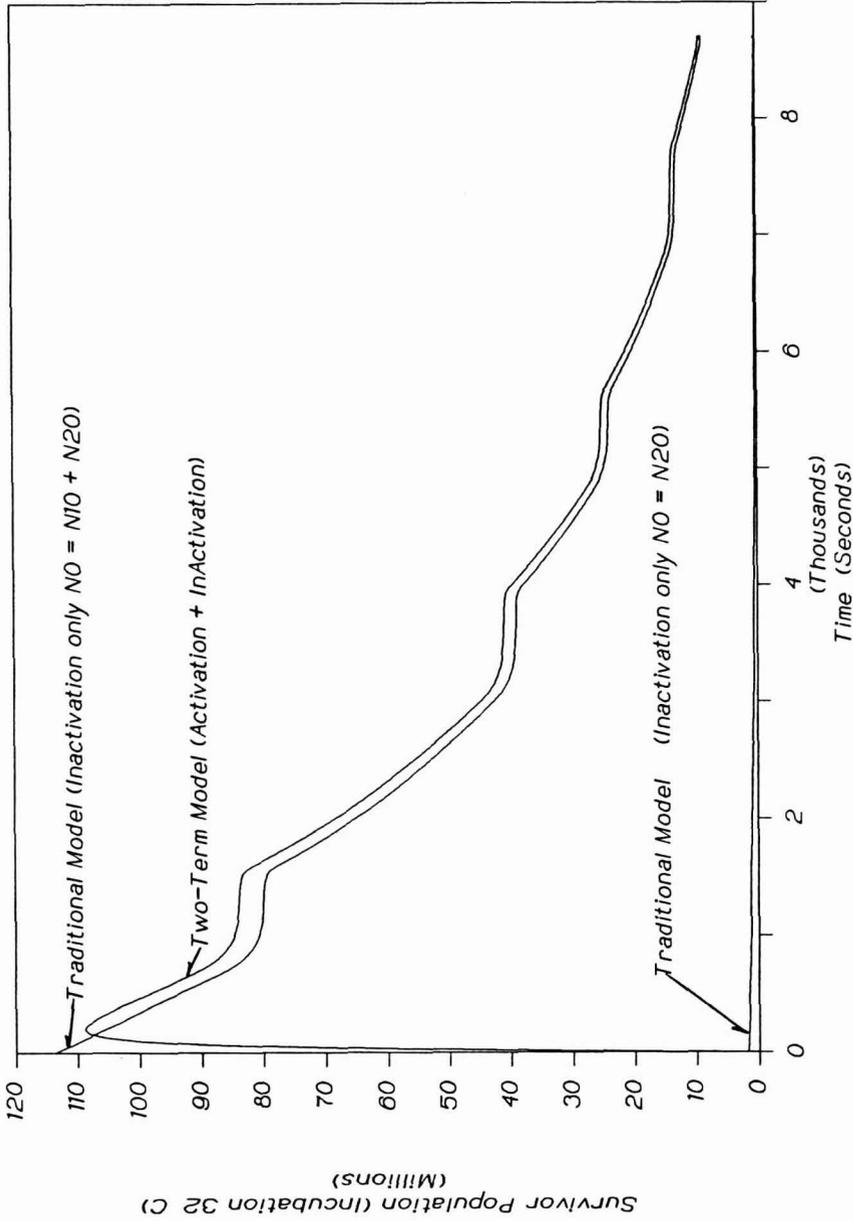


FIG. 10. POPULATION RESPONSES OF TRADITIONAL AND TWO-PROCESS MODELS FOR INJURED *B. SUBTILIS* SPORES EXPOSED TO THE TEMPERATURE HISTORY IN FIG. 6.

A more appropriate initial population for the traditional model would be total spores, $N_0 = N_{10} + N_{20}$, as indicated by responses of the traditional model in Fig. 9 and 10 that compare better with those of the two-process model with the same values of N_{10} and N_{20} . However, the response of the traditional model still remains below that of the two-process model because it fails to account for the delayed appearance in N_2 of activated members of N_{10} , and it inactivates them prematurely.

Isothermal responses of populations free of less heat resistant fraction N_3 provide further proof that $N_0 = N_{10} + N_{20}$ is more appropriate for the traditional model. The long decline in a semilog plot of isothermal survivor population is due to predominant inactivation. Conventional linear regression of the decline fits the solution of Eq. (11).

$$N(t) = N_0 \exp(-K_d t) \quad (14)$$

to that portion of the survivor data. The response of mature, activated population N in the two process-model is given by Eq. (7); its first term

$$(N_{10}+N_{20}) \exp(-K_d t) \quad (15)$$

pertains to the long decline in activated spore population due to predominant inactivation and may be regressed to it. Clearly Eq. (14) and Eq. (15) describe the same survivor data, and pseudo initial spore population $N_0 = N_{10} + N_{20}$. Initial activated spore population N_{20} is obtained experimentally by laboratory enumeration of an untreated sample; pseudo initial population N_0 is found from the intercept of the linear regression line fitting the declining population. It follows that the initial, inactivated population is found from $N_{10} = N_0 - N_{20}$.

Conventional linear regression of a semilog plot of population data for isothermal treatment ignores any shoulder occurring early in the treatment and only fits Eq. (14) to the subsequent, declining portion of the response. From the two-process model and the discussion above, it is clear that the amplitude of the declining phase and, therefore, N_0 for the traditional model depend directly on formation of that shoulder, i.e., on initial population N_{10} and the speed and degree of its activation. The latter depend on relative sizes of K_a and K_d . Common heat shock of a spore suspension attempts to minimize the shoulder and reduce uncertainty in N_0 by inducing a highly activated population prior to testing a lethal treatment, but the result is $N_{20} < N_0 < N_{10} + N_{20}$, and uncertainty remains about the degree to which heat shock has activated N_{10} . Predictions by both models should agree closely after the shoulder and well into the decline with $N_0 = N_{10} + N_{20}$. This was the case in Fig. 9 and 10. The new, two-process model precludes need for heat shock; indeed, it applies to any distribution of N_{10} and N_{20} and may even be used to predict results of sequential heat shock and lethal heat treatment.

In summary, the traditional model is a dependable predictor of the dynamics of a population of activated spores only during its decline late in a thermal sterilization process. A more complex model, such as the one presented here, is needed for accurate prediction early in a process when premature inactivation and activation can cause real population dynamics to deviate significantly from those produced by the conventional model. The two-process model applies to both shoulder formation and the decline. The choice of model must take into consideration the duration of a process, relatively long in retort sterilization of most canned foods or short for ultra high temperature (UHT) sterilization of liquid food, initial conditions N_{10} and N_{20} , and the effect of the temperature regime on relative magnitudes and dynamics of K_a and K_d .

CONCLUSION

This paper elaborates upon a new, two-process kinetic model for predicting the dynamics of survivors in a spore suspension exposed to lethal heat treatment. It differs from the traditional, single first-order reaction model by having up to three simultaneous, independent first-order reactions and, consequently, up to three exponential terms, instead of one, in its isothermal survivor curve. The difference arises from taking into account preliminary inactivation and activation reactions that may occur concurrently with predominant inactivation early in an exposure. The new model, therefore, is more accurate than the traditional model in its representation of the thermal mechanisms and initial conditions affecting dynamics of spore populations in lethal, sublethal, and composite thermal regimes. It precludes need for heat shock of an indicator population prior to testing a lethal heat treatment. Furthermore, it applies to injured as well as normal spores.

As with the traditional model, parameters of the new model must be determined from experimental survivor curves for several constant, lethal temperatures. However, instead of describing a semilog survivor curve by a single straight line with one reaction rate constant, it is described more elaborately with up to three exponentials. Experiments reported here provided preliminary values of rate constants of activation and predominant inactivation for normal and injured *B. subtilis* spores in the lethal range of 87–99 °C and indicated that temperature dependencies of those rate constants follow Arrhenius relationships. Slopes, intercepts, and activation energies of those Arrhenius relationships were determined.

Model generated isothermal survivor curves compared well with corresponding experimental data. Small discrepancies early in exposure intervals, when the rapid dynamics of activation and preliminary inactivation dominate, pointed to the importance of frequent, accurate sampling then. Less frequent sampling is appropriate to the slower, longer lasting, predominant inactivation.

Tests with a dynamic process showed the capability of the new model, using experimentally determined dependencies of reaction rate constants on temperature, to produce survivor curves comparing well with experimental curves and to improve understanding and microbiological validation of arbitrary, dynamic thermal sterilization processes.

Overall, the results of this study indicated high potential for the new model to describe well the dynamics of bacterial spore populations exposed to constant and dynamic lethal temperatures, to enhance understanding of those dynamics, and to become an important tool of basic research and applied practice. They also indicated need for more extensive, replicated experiments for full validation of the model.

APPENDIX

The Levenberg-Marquardt method has become a standard for nonlinear, least-square parameter evaluation. The model fitted to data is

$$y = y(x; \mathbf{a})$$

where \mathbf{a} is a parameter set. The X^2 (chi squared) merit function is used in the determination of parameters \mathbf{a} .

$$\chi^2 = \sum_i \left(\frac{y_i - y(x_i; \mathbf{a})}{\sigma_i} \right)^2$$

where

x_i	is i th sample of x
y_i	is corresponding experimental value of y
$y(x_i; \mathbf{a})$	is corresponding calculated value of y
σ_i	is corresponding standard deviation of y_i
\mathbf{a}	is the parameter set to be determined

Values of parameters \mathbf{a} for which the gradient of X^2 with respect to \mathbf{a} equals zero determine the minimum of X^2 .

For the data reported here, the parameters were the rate constants, and standard deviations of the y_i were determined experimentally to be 30% of corresponding mean values. However, using the data with values of the σ_i , between 20 to 100% of corresponding means led to the same rate constants.

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DISTRIBUTION OF HEAT TRANSFER RATE AND LETHALITY IN A SINGLE BASKET WATER CASCADE RETORT

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ABSTRACT

Temperature and heat distribution studies were carried out in a single basket horizontal water cascading retort at two temperatures and two air over-pressure levels under fully loaded operating conditions. Various heat transfer parameters (heating and cooling rate indices, f_h and f_c), lag factors (j_{ch} and j_{cc}) and process lethality (F_0) were used as indicators of the retort performance. Time-temperature data gathered from 24 LexanTM transducers were used for calculation of the heating and cooling rate indices as well as lethality. There were significant variations in the temperature distribution and heat transfer parameters at the different tray levels; however, these variations did not contribute to large variations in the accumulated overall process lethality.

INTRODUCTION

New retorting systems and processing media for food sterilization need to provide adequate temperature and heat transfer distribution in order to promote safety of the processed food as well as the control of its quality. Several studies have been carried out on the evaluation of retort systems, and guidelines have been published on the efficacy of a retort based on temperature distribution (NFPA 1985; Lopez 1987). When heating in pure steam, the surface resistance to heat transfer is generally neglected and the process is primarily assumed to be dependent on retort temperature performance. However, with several over-pressure retort systems, the effective heat transfer rates could be dependent on several additional factors such as medium flow rate, flow direction, loading pattern, etc. Steam heated water with air over-pressure and steam/air are two

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heating media commonly used to sterilize foods packaged in glass containers, flexible and semi-rigid packages such as retort pouches and thermostable plastic trays. Both of these media have been studied in several over-pressure retort systems with reference to temperature and heat transfer distribution (Adams and Hardt-English 1990; Berry 1979; McGinnis 1986a,b; Ramaswamy 1983; Ramaswamy *et al* 1983; Tung and Ramaswamy 1986; Tung *et al.* 1984, 1990; Yamano 1976).

Guidelines have been established by the NFPA (National Food Processor's Association, Washington, DC) and recently by the IFTPS (Institute for Thermal Processing Specialists, Washington, DC) for temperature distribution verification of all retorts for assurance of adequate performance. Typical recommendations are:

- (1) The heating media circulation pattern should be compatible with the racking system design. "Ideally, the circulation of the heating medium should be parallel to the container length or width" (NFPA 1985).
- (2) The temperature within the retort after one minute following come-up should have a maximum range of 3F (1.7C) and should be within 1.5F (0.8C) of the reference temperature device (NFPA 1985).
- (3) Heating rate (f_h) distribution throughout the loading area should be verified.

Among the several new retorts introduced to the market is the Steriflow™ (Barriquand, Paris, France), based on a unique cascading water flow principle. In their study of 4- to 6-basket models of the Steriflow retort, Adams and Hardt-English (1990) reported temperature ranges (maximum *minus* minimum temperature) of 2.2C during the first minute after come-up, and a reduction of the temperature range to 1.0C by the third minute following the come-up period. In previous studies with a 1-Basket Standard Model of Steriflow retort (Ramaswamy *et al.* 1991), much larger temperature ranges (3.5C) were observed even several minutes after come-up. In the same study however, the maximum difference between mean temperatures at various locations within the retort during the cook period were small ($\sim 0.6C$), indicating good temperature distribution. The seemingly different observations were attributed to out of phase cyclic temperature oscillations, resulting in relatively large temperature deviations centered about similar means.

The temperature distribution data described above raises a concern as to the performance of the Steriflow retort, especially with respect to the NFPA (1985) recommendations. The objective of this study was to further examine the Steriflow retort with reference to the distribution of various effective heat transfer parameters such as heating and cooling rate indices (f_h and f_c), lag factors (j_{ch} and j_{cc}) and, more importantly, the resulting process lethality (F_0).

MATERIALS AND METHODS

Description of Retort System

Unlike typical over-pressure steam-air processes, in which homogeneity of the processing environment is ensured by means of powerful fans or constant venting, water cascading retorts utilize a spray of superheated water as the heat transfer medium. The water, showered on top trays, trickles down to each consecutive level through numerous perforations in the trays, releasing heat as it travels. Once at the bottom of the retort, the water is recycled, passing through a steam-supplied heat exchanger. Due to the high heat transfer coefficient associated with condensing steam, the process water temperature is raised to the operating condition in about 6–8 min (come-up time) which could possibly be reduced further by injecting steam in to the retort during the come-up period. In the Steriflow™ retort (manufactured by Barriquand, Paris, France), a pump recycles the relatively low volume of heating water (about 100 L/loading car) at about once every 9 s (Manufacturer's Data, Steriflow, Barriquand, Paris, France). Cooling is achieved by providing the heat exchanger with cold water, thus using the same water to cool the product as was used for heating. The cooling water temperature, however, falls at a rate much lower than heating (come-down periods required more than 20 min), thereby causing the product cooling rates to be much lower than the heating rates in the same run.

As the water flows from top to bottom of the loading car of a water-cascading retort, heat is transferred to the contents of the retort, resulting in a temperature gradient from top to bottom of the retort. The occurrence of such a temperature gradient was confirmed by Adams and Hardt-English (1990), who reported that the slowest heating area of the retort basket was on the bottom tray.

The system used for this study was a 1-door, 1-basket horizontal Steriflow retort located at the Agriculture Canada Food Research and Development Center at St. Hyacinthe, PQ. The loading car of the unit contains 8 trays of dimensions 0.81×0.81 m, providing a load area of 0.65 m^2 . Each tray was 2 cm thick, with a 10.2 cm of clearance between trays.

Design of Food-Simulating Units

Lexan™ bricks (Canadian General Electric, Montreal, Canada) were used for gathering heat penetration data. Food-simulating test units were fabricated by tightly sandwiching a thermocouple (Type T) between two half-thickness Lexan slabs ($10 \times 15 \times 1.0$ cm) with the thermocouple tip located at the geometric center of the brick as detailed in Ramaswamy *et al.* (1983). NFPA (1985) suggested that Lexan polycarbonate could be used for fabricating test bricks for heat distribution studies and this was chosen as a substitute for the relatively expensive Teflon™. Further, the transparent Lexan slab permitted visual observation of the thermocouple wire for failure without actually dismantling the brick.

Experimental Layout

To simulate the fully loaded retort operation, each of the eight trays were loaded with 24 bricks of Lexan polycarbonate, with dimensions of about 10 cm \times 15 cm \times 2 cm. The eight trays were numbered 1 to 8 from top to bottom. The medium temperature was measured using 24 type-T thermocouples (six each on trays numbered 1, 3, 6, and 8) by carefully tying the thermocouples to the perforated trays so that their tips did not touch any brick or tray material.

With 24 Lexan test transducers placed on the top, middle or bottom tray (tray #1, 5, or 8; all transducers loaded onto one tray), experiments were carried out in duplicates at two temperatures, 115.6 and 121.1C, and two pressure levels, corresponding to steam/air fractions of 65/35, and 75/25 (air over-pressure 170 and 130 kPa at 115.6C, 220 and 180 kPa at 121.1C, respectively). During retort operation, both medium temperature from the 24 bare thermocouples and test brick temperature from the thermocouples in the 24 Lexan bricks were obtained at 15 s intervals throughout the entire process. Each process run was continued for a minimum of 30 min (cook period) following retort come-up time. All thermocouples were calibrated against an ASTM mercury-in-glass thermometer. A Metrabyte Dash-8 with 3 EXP-16 expansion boards (Metrabyte Corp., Taunton, MA) interfaced to a personal computer was used to acquire temperature data from the 48 thermocouples.

Heating Rate and Lethality Calculations

Slight variations in retort temperature and in the initial product load temperature from one run to the next were unavoidable. In commercial retorting operations, such variations are usually compensated by adjusting the processing times to ensure adequate lethality. In heat distribution evaluation, even these small variations in the retort and initial product load temperatures may cause considerable nonuniformity with reference to the delivered lethality. For this reason, the equations of Schultz and Olson (1940) were employed to adjust the initial product load temperature and processing temperature to their respective setpoint temperatures.

The adjusted time-temperature data from Lexan bricks were employed to evaluate the heating and cooling rate indices (f_h and f_c), obtained as negative reciprocal slopes of the straight line portion of their heat penetration curves (Ball 1923). Similarly, the heating and cooling lag factors (j_{ch} and j_{cc}) were calculated from the initial portions of the heating and cooling curves (Ball 1923).

The accumulated lethality of the process (F_O) was obtained from center temperatures of test bricks by numerical integration of the lethal rate equation (Ball 1923) as performed by Patashnik (1953). The process lethality was divided into heating and cooling lethality (F_{Oh} and F_{Oc}) with the heating lethality calculated from steam on to Ball's process time of 30 min (based on a come-up

period efficiency of 42%), at which time all Lexan transducers had reached process temperature. Cooling lethality was calculated from the initiation of the cooling cycle for the two processing temperatures (115.6C and 121.1C), and two steam contents (65% and 75%).

RESULTS AND DISCUSSION

Temperature Distribution in the Retort

The temperature distribution in terms of mean retort temperature, the overall standard deviation and maximum deviation between two locations in the retort during the cook period (excluding the come-up) are summarized in Table 1. The retort mean cook-temperatures were close to the setpoint temperatures and the standard deviations in the retort temperature at various locations were small (0.58–0.71C). As detailed in Ramaswamy *et al.* (1991), the instantaneous temperature differences between locations were large and their maximum during the cook-period varied from 2.6 to 3.9C. However, the mean cook temperatures on the top, middle and bottom trays of the retort were very close, with a maximum tray to tray mean temperature deviation of 0.3C.

TABLE 1.
SUMMARY OF OVERALL TEMPERATURE DISTRIBUTION IN STERIFLOW RETORT

Setpoint Temperature (C)	Air over- pressure (kPa)	No. of Replicates	Retort Temperature Distribution (C)		
			Mean	Std. Dev.	Max. Dev.
115.6	130	6	115.5-116.0	0.59-0.63	2.9-3.7
115.6	170	6	115.5-116.0	0.59-0.67	2.6-3.6
121.1	180	6	120.8-121.0	0.56-0.71	2.6-3.9
121.1	220	6	120.8-121.1	0.58-0.69	2.7-3.3

Heating and Cooling Rates

The distribution of heating and cooling rate indices (f_h and f_c) at the three tray levels and at two temperatures are shown in Fig. 1 and 2. The horizontal lines on these plots represent the mean values of the parameter at each tray level. These figures demonstrate a considerable spread in the f_h and f_c values at the three tray levels with a somewhat similar distribution pattern at the two temperatures.

The mean values and standard deviations of heating and cooling rate indices at the two temperatures and two over-pressure levels are summarized in Table 2. The two levels of air over-pressure (corresponding to 65% and 75% steam) had no significant effect on the heating or cooling rates of test bricks. For this reason, the lethality analyses were performed only with respect to retort temperature (115.6C and 121.1C) and tray level (top, middle, bottom) within the retort.

The effect of temperature (115.6 and 121.1C) on the heating or cooling rate indices in the Steriflow retort (Table 2) was not significant ($p > 0.01$) on the top tray of the retort. However, the temperature effect was significant ($p < 0.01$) on the cooling rate index of bricks on the middle tray, and on both heating and cooling rate indices on the bottom tray. These statistical differences were considered to be possible artifacts in the sensitivity analyses due to the inclusion of large number of data pairs (96). The maximum mean difference on a given tray between the two temperatures was 2.5% with the heating rate index and 10% with the cooling rate index, which were within the tray to tray variations observed with the retort.

Although the Steriflow retort utilizes the same medium for both heating and cooling of the retort load, the relative heating rates of bricks on the three trays did not follow the same trend as the cooling rates. This is illustrated in Fig. 3 in which the average heating and cooling rate indices (and their overall standard deviations as vertical bars) are plotted as a function of tray level. During heating, the test bricks on the top tray heated most rapidly (lowest f_h value), while the most rapid response to cooling (lowest f_c value) occurred on the bottom tray. For both heating and cooling the middle tray provided the slowest rate. If the locational trends in heating and cooling rates during processing were similar, the location receiving the lower lethality during heating (higher f_h) would acquire the higher lethality during cooling (higher f_c). Conversely, the location of higher heating lethality (lower f_h) would receive the lower cooling lethality (lower f_c). However, locational differences existed in the retort between heating and cooling rates. The maximum difference between the mean f_h values with reference to the tray levels was $\sim 10\%$ while the variation within a tray was 3–4%. With reference to the cooling rates, the maximum tray level difference was $\sim 15\%$ with 3–7% variation within a tray. These results generally indicated relatively less uniformity of cooling rates compared to heating rates.

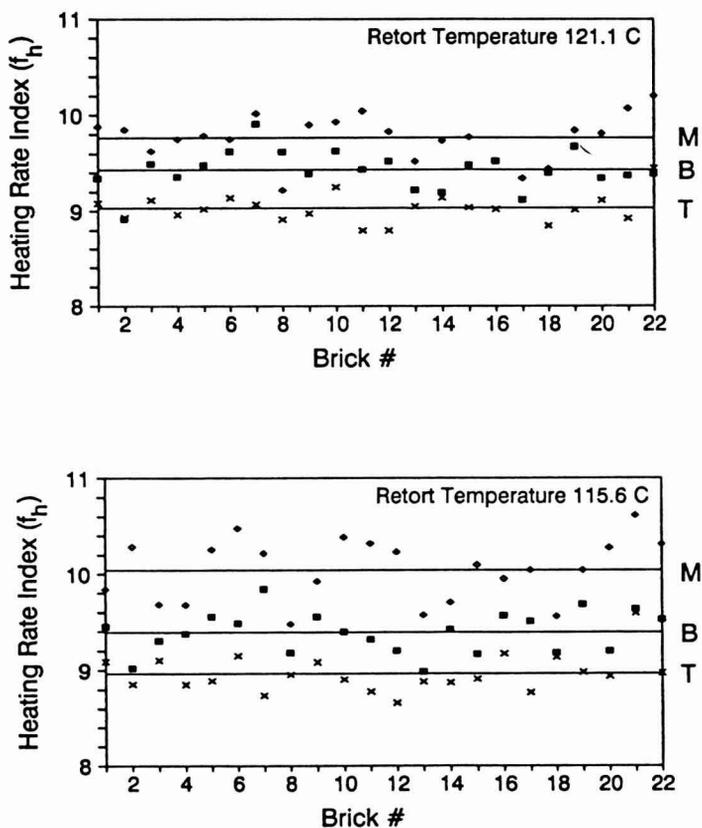


FIG. 1. DISTRIBUTION OF HEATING RATE INDEX (f_h) AT TOP (T), MIDDLE (M) AND BOTTOM (B) TRAYS AT TWO TEMPERATURES

In all cases, rates of cooling were much slower than heating, indicated by the cooling rate indices, which were over 50% higher than the heating rate indices. This was a result of the relative come-up and come-down profiles of the retort. While the retort come-up period was characteristically 6–8 min, water temperature in the retort during the cooling changed relatively slowly as shown in Fig. 4 for a typical run. The result was a significantly larger cooling lag as well as lowered temperature difference potential for the test bricks during cooling which was the main cause for the higher f_c value.

Heating and Cooling Lag Factors (j_{ch} and j_{cc})

The distribution of heating and cooling lag factors in typical runs at the two temperatures are shown in Fig. 5, and the mean values with standard deviations of the lag factors are summarized in Table 2. As with the heating and cooling

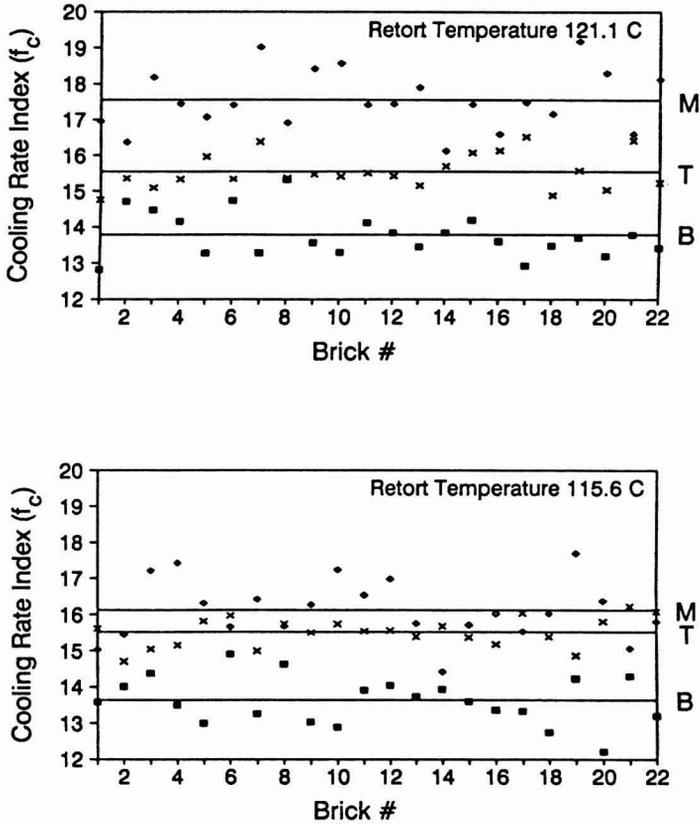


FIG. 2. DISTRIBUTION OF COOLING RATE INDEX (f_c) AT TOP (T), MIDDLE (M) AND BOTTOM (B) TRAYS AT TWO TEMPERATURES

rate indices, the influence of air over-pressure and retort temperature were non-significant with reference to the lag factors (Table 2). The tray level, however, significantly influenced the lag factors with the bottom tray of the retort showing the largest scatter as well as the highest lag value for both heating and cooling (Fig. 5). The lag was lowest on the top tray during heating and the middle tray during cooling. Unlike most retorts, which use fresh cold water for cooling, the Steriflow retort cools the product by recycling the same water used for heating through a heat exchanger supplied with cold water. This slower cooling of process water through the indirect heat exchanger (Fig. 4) resulted in apparent j_{cc} values almost four times as large as j_{ch} .

TABLE 2.
MEAN VALUES (AND STANDARD DEVIATIONS) OF HEAT PENETRATION
PARAMETERS (f_h , f_c , j_{ch} , j_{cc}) ON DIFFERENT TRAYS IN THE STERIFLOW RETORT
AT TWO TEMPERATURES AND TWO AIR OVER-PRESSURES

Temperature (C)	Overpressure (kPa)		f_h (min)	f_c (min)	j_{ch}	j_{cc}
121.1	220	Top	9.0(0.27)	15.3(0.61)	1.1(0.09)	4.2(0.21)
		Middle	10.1(0.45)	16.7(0.85)	1.1(0.08)	3.6(0.32)
		Bottom	9.6(0.34)	15.1(0.50)	1.2(0.05)	3.7(0.27)
121.1	180	Top	8.8(0.24)	14.4(1.20)	1.0(0.05)	4.0(0.20)
		Middle	9.8(0.45)	17.2(0.84)	1.0(0.16)	3.4(0.26)
		Bottom	9.6(0.37)	15.3(0.67)	1.2(0.08)	3.9(0.27)
115.6	170	Top	9.1(0.27)	15.4(1.69)	1.1(0.12)	3.9(0.15)
		Middle	10.1(0.47)	16.0(0.79)	1.2(0.07)	3.6(0.49)
		Bottom	9.5(0.39)	13.4(0.77)	1.3(0.08)	4.1(0.43)
115.6	130	Top	8.9(0.33)	15.2(0.34)	1.1(0.12)	4.1(0.18)
		Middle	10.0(0.31)	16.3(0.87)	1.2(0.05)	3.8(0.34)
		Bottom	9.3(0.48)	14.4(1.13)	1.1(0.15)	4.3(0.43)

Process Lethality

The lethality distribution of top, middle and bottom trays during typical retort processes at 121.1C (Fig. 6) demonstrated the same characteristic scatter previously observed with the heat penetration parameters. The mean and standard deviations in lethality contributed during heating, cooling and combined heating and cooling are summarized in Table 3. In order to keep the table simple, the results at the two over-pressure levels have been pooled, since their influence on process lethality was not significant. The lethality achieved during the heating period of a 30 min process was highest for the bricks on the top tray and lowest on the middle tray (Fig. 6). This result is consistent with the distribution of heating rate indices which were lowest on the top tray and highest on the middle tray. The lethality contributed during cooling (Fig. 6) was consistent

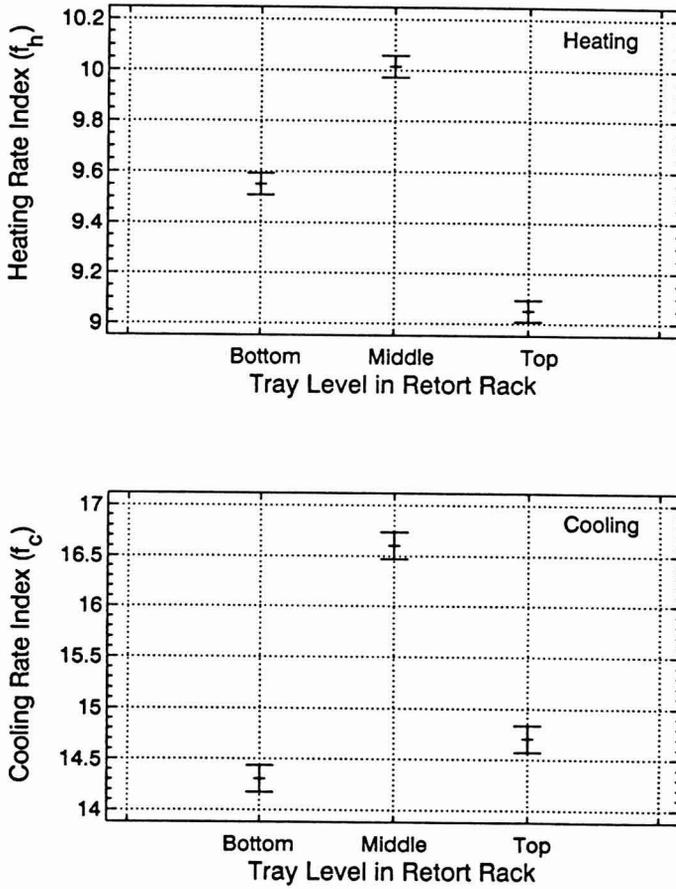


FIG. 3. AVERAGE AND STANDARD DEVIATIONS (VERTICAL BARS) IN HEATING AND COOLING RATE INDICES (f_h AND f_c) AS A FUNCTION OF TRAY LEVEL

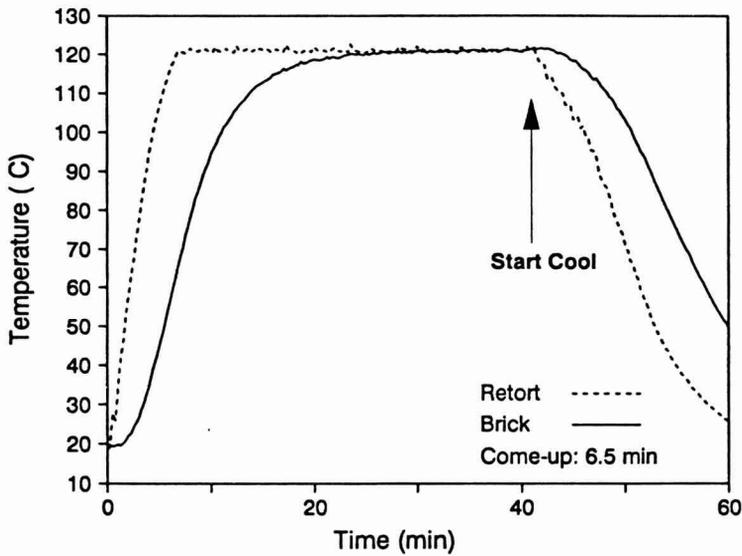


FIG. 4. RETORT AND PRODUCT TEMPERATURE HISTORY DURING A TYPICAL PROCESS AT 121°C

TABLE 3.
MEAN VALUES (AND STANDARD DEVIATIONS) OF LETHALITY DURING HEATING,
COOLING AND COMBINED (F_{oh} , F_{oc} , F_{ot}) ON DIFFERENT TRAYS
IN THE STERIFLOW RETORT AT TWO TEMPERATURES

Temperature (C)		F_{oh} (min)	F_{oc} (min)	F_{ot} (min)
121.1	Top	15.62 (0.49)	4.10 (0.27)	19.72 (0.50)
	Middle	14.29 (0.73)	4.16 (0.22)	18.45 (0.63)
	Bottom	14.67 (0.67)	3.95 (0.16)	18.62 (0.49)
115.6	Top	4.45 (0.14)	1.16 (0.04)	5.61 (0.18)
	Middle	3.75 (0.27)	1.27 (0.07)	5.02 (0.29)
	Bottom	4.20 (0.11)	1.23 (0.06)	5.43 (0.14)

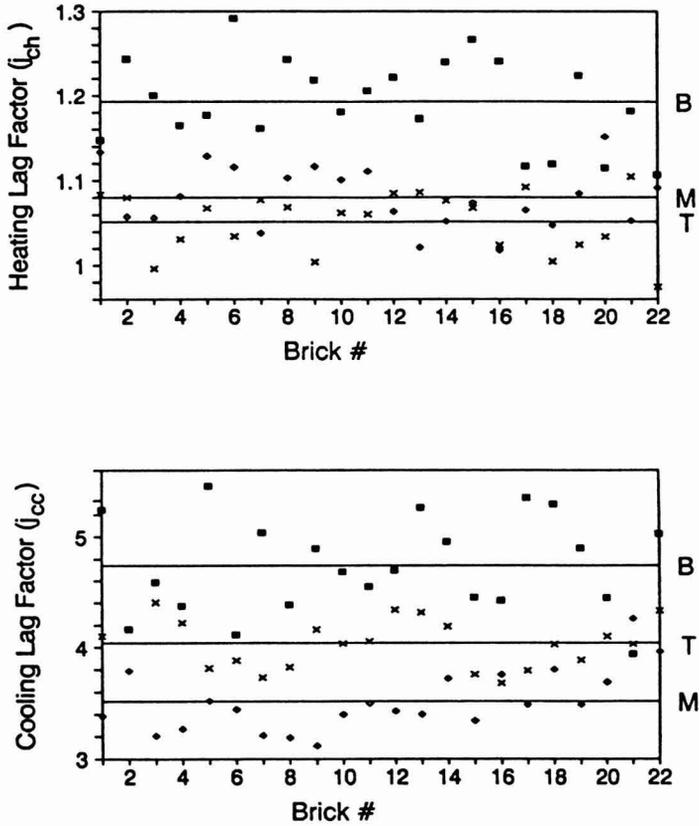


FIG. 5. DISTRIBUTION OF LAG FACTORS (j_{ch} AND j_{cc}) AT TOP (T), MIDDLE (M) AND BOTTOM (B) TRAYS IN A TYPICAL RUN AT 121C

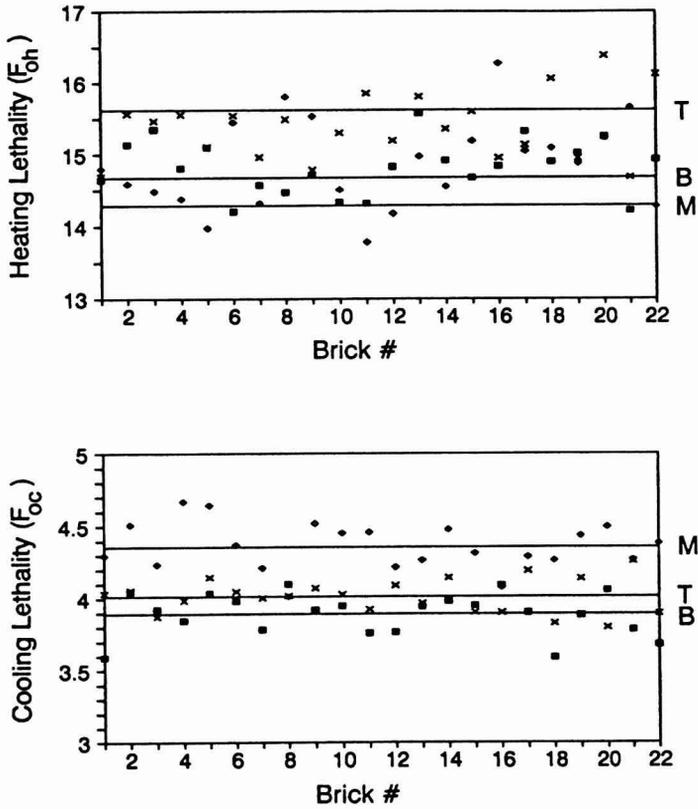


FIG. 6. DISTRIBUTION OF HEATING LETHALITY (F_{oh}) AND COOLING LETHALITY (F_{oc}) AT TOP (T), MIDDLE (M) AND BOTTOM (B) TRAYS DURING A TYPICAL RUN AT 121.1°C

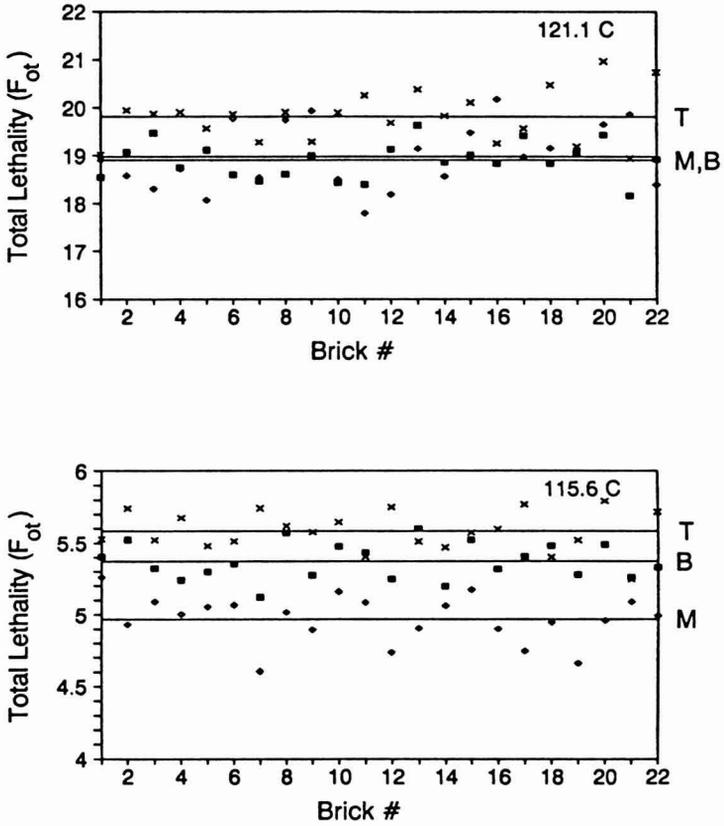


FIG. 7. DISTRIBUTION OF TOTAL LETHALITY (F_{ot}) AT TOP (T), MIDDLE (M) AND BOTTOM (B) TRAYS DURING A TYPICAL RUN AT 121.1C

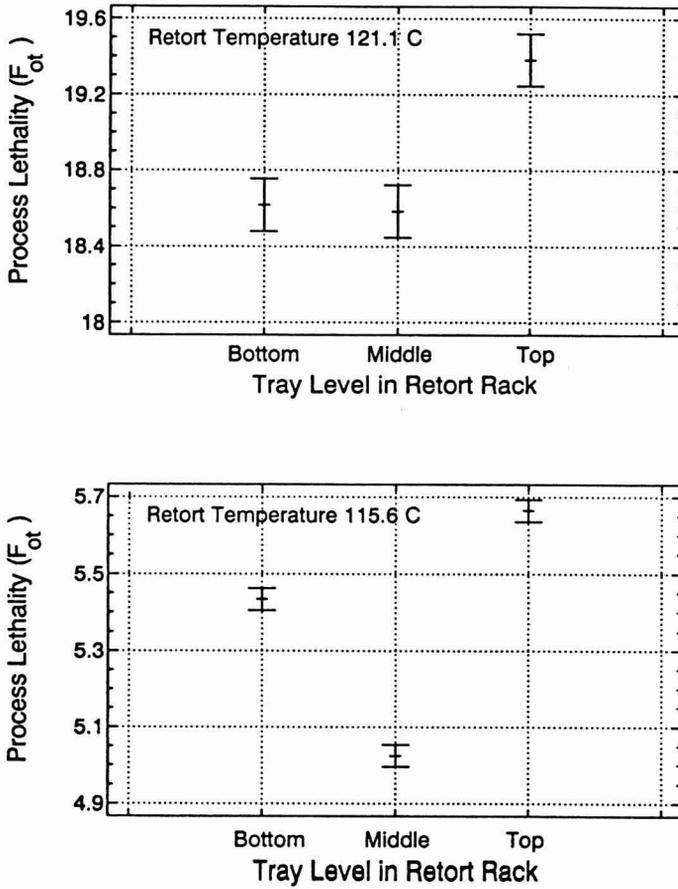


FIG. 8. AVERAGE AND STANDARD DEVIATIONS (VERTICAL BARS) TOTAL PROCESS LETHALITY (F_{ot}) AS A FUNCTION OF TRAY LEVEL

with the trend exhibited by the cooling rate: the highest on the middle tray of the rack corresponding to the slowest cooling rate. These observations were similar at the other retort temperature, 115.6C (Table 3). The cooling lethalties were similar for all runs at a temperature, mostly because the thin profile test bricks used in these studies generally reached the retort temperature during the 30 min process given.

The distribution of overall process lethality, shown in Fig. 7 and Table 3, was somewhat similar to the heating and cooling lethality distributions (Fig. 6) with highest values on the top tray of the retort. The overall mean process lethalties and their standard deviations are shown in Fig. 8. Test bricks on the top tray received the highest lethality at both process temperatures. Bricks on the middle tray exhibited the lowest overall lethality, although at 121C, there were no significant differences between the middle and bottom trays (Fig. 8). Based on process lethality, therefore, the middle tray appears to be the most conservative choice for gathering heat penetration data for process calculations. The maximum difference between the mean overall lethalties with reference to the tray levels was ~7% at 121.1 and ~12% at 115.6C while the variation within a tray was 3-6%.

CONCLUSIONS

Locational trends in heat distribution within a retort can be a serious problem in determining accurate process times. The Steriflow retort exhibited definite locational variation of process parameters and resulted in up to 12% overall tray variations in process lethality. The locational differences in process parameters observed in this retort illustrate the difficulty in choosing an appropriate location for gathering time-temperature data for process calculations. One location to avoid for such purposes is the top tray, where the heating rate is rapid and lethality achieved is always highest.

The large temperature deviations (3-4C) observed within the retort (Table 1) did not result in equivalent variations in process lethality. The observed lethality variations of about 12% correspond to a mean temperature deviation of $\pm 0.6\text{C}$ (Ramaswamy *et al.* 1991), which is the average standard deviation in temperature for the retort runs (Table 1). These results suggest that the standard deviation in locational cook-period temperatures may be a better indicator of temperature distribution than temperature deviation approach.

ACKNOWLEDGMENT

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EFFECT OF STEAM/AIR MIXTURES ON THERMAL PROCESSING OF AN INDUCED CONVECTION-HEATING PRODUCT (TOMATO CONCENTRATE) IN A STERITORT

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ABSTRACT

The effect of reel speed, can size, and percent air during steam and steam/air processing in a Steritort on the heating rate of tomato concentrate (an induced convection-heating product) was investigated. A Taylor expansion equation, relating Ball process times with the above three variables, considering first order, second order, and interaction effects, was presented. A correlation equation for the Nusselt number as a function of the Reynolds and Prandtl numbers, the can length over the diameter ratio, and the steam content was also presented. Percent reduction in process lethality increased with increasing can size and air content, and decreasing reel speed and target lethality. Increase in percent air in the steam/air mixture could be compensated by an increase of the rotational speed.

INTRODUCTION

The need for processing under air overpressure, in order to preserve container integrity (plastic, glass, and thin-walled metal containers), to eliminate the insulating effects due to expansion of the entrapped gases in flexible containers (Weintraub *et al.* 1989; Tung *et al.* 1990), to potentially save energy by using steam/air as the heating medium (Milleville, 1981) or to allow for alternative venting and bleeding practices (Kimball and Heyliger 1990), necessitate studies on thermal processing of foods in steam/air environments. Several investigators

have studied the effects of steam/air mixtures on heat transfer to model systems or food products (Pflug *et al.* 1963; Pflug 1964; Pflug and Borrero 1967; Yamano *et al.* 1979; Ramaswamy *et al.* 1983; Tung *et al.* 1984; Kisaalita *et al.* 1985; Ramaswamy and Tung 1986; Deniston *et al.* 1991). With the exception of Deniston *et al.* (1991), who conducted their study in a Steritort (a continuous rotary sterilizer simulator), the above mentioned investigators used various types of stationary batch retorts.

Pflug *et al.* (1963) reported lower f_h values (faster heat transfer rates) for convection or conduction-heating products in rectangular pouches processed in commercial still retorts with 100% steam compared to processing with 90 or 75% steam in steam/air mixtures. Pflug (1964) and Pflug and Borrero (1967) also found decreasing and more uniform f_h values with increasing steam content (75, 90 and 100%) for steam/air processing of water in 303×406 cans or small pouches. However, for the same conditions, for 5% bentonite suspension in water, Pflug (1964) found that heating rates were not affected. For 10 and 30% bentonite suspensions in rectangular pouches processed in steam/air mixtures, Yamano *et al.* (1979) also reported practically constant f_h values for the range of 70 - 100% steam. Below 70% steam they found increasing f_h values with decreasing steam content. Ramaswamy *et al.* (1983) and Tung *et al.* (1984) calculated heat transfer coefficients at the surface of high thermal diffusivity thin profile test bricks (aluminum or stainless steel) processed in several still retorts using steam/air mixtures. They found that heat transfer coefficients were exponential functions of the steam content. For a vertical, positive flow retort, they also reported that the surface heat transfer coefficient was an increasing linear function of the flow rate. Finally, they also found that flow direction had a significant impact on heat transfer rates. Kisaalita *et al.* (1985), using a stainless steel condensing block, found an exponential relationship between surface heat transfer coefficients and the block surface temperature or the temperature difference between surface and heating medium temperatures. Furthermore, for each block surface temperature, they approximated the heat transfer coefficients by a linear function of the air content. Using low thermal diffusivity materials (thin profile silicone rubber and nylon bricks), Ramaswamy and Tung (1986) reported center point f_h values practically unaffected by the steam content of the steam/air heating medium. This indicated that for these materials (with properties similar to food products) the internal resistance is the control resistance to heat transfer. For a convection-heating product, Deniston *et al.* (1991) reported slower heating rates and therefore higher percent lethality reduction for increasing air overpressure, the effects being a function of the can size and the reel rotational speed and being more noticeable for high overpressure values. Finally, Kusak (1958) reported that the surface heat transfer coefficient for a medium consisting of a condensing vapor and a noncondensing gas was a function of the Reynolds number of the medium (raised to the 1/3 power) and an exponential

function of the mole fraction of the noncondensing gas in the gas-vapor system.

Clearly, heat transfer rates are a function of processing system, operational conditions, and product characteristics. The objective of this work was to quantify the effects of steam/air mixtures, as the heating medium, on thermal processing of an induced convection-heating product (tomato concentrate) processed in a Steritort.

MATERIALS AND METHODS

Retort System

Heat penetration tests were conducted in a Steritort with a reel diameter of 1.3 m (FMC Corporation, Chicago, IL). Overpressure was controlled by a combination of an air-to-open control valve on the air line and a mechanical pressure relief valve. For steam/air processing, air was introduced into the steam line after the steam line control valve, to partially mix the steam and air by passage through two elbows and two spreader pipes before introduction into the pressure chamber.

Percent air (by volume) in the steam/air mixtures was determined with a Westinghouse mini probe oxygen analyzer (Model 132, Rosemount Analytical, Inc. Orrville, OH) plumbed through a 3/4-inch pipe to the center of the cylindrical side of the Steritort and connected to a Miniservo Recorder (Esterline Angus Instrument Corp., Indianapolis, IN). The analyzer was calibrated using three concentrations of reference oxygen gas. Percent oxygen was measured during selected heat penetration experiments, and converted to percent air. Percent air (by volume) was also calculated based on total system pressure. Pressure was measured with a high accuracy pressure gauge (± 0.1 psig) from Weksler Instruments, Inc. (Freeport, NY). Air overpressures of 6.9, 20.7, 34.5, and 69.0 kPa which corresponded to 3.2, 9.0, 14.2, and 24.8% air, respectively, in the steam/air mixtures, (0.67, 1.9, 3.0 and 5.2% oxygen, respectively) were used. The variation of the total (steam and air) pressure during the experiments was between 1.4 and 2.8 kPa on either side of the target value. Calculated and measured oxygen concentrations were in good agreement; for the above overpressures, the oxygen concentrations determined experimentally, using the oxygen analyzer, were $0.88 \pm 0.02\%$ (4 trials), $2.19 \pm 0.14\%$ (7 trials), $3.20 \pm 0.08\%$ (4 trials), and $5.88 \pm 0.14\%$ (4 trials).

Heat Penetration Equipment

Heat penetration data were collected (during both the heating and cooling cycles of the process) with equipment from Ecklund-Harrison Technologies, Inc. (Cape Coral, FL) consisting of CNS copper-constantan thermocouples, C-9

locking receptacles, S-28NR rotary contractors, and a 16 circuit slip ring assembly. The thermocouples were calibrated with an ice point cell (Omega Engineering, Inc., Stamford, CT), and against the Steritort mercury-in-glass thermometer which in turn had been calibrated against a NIST thermometer at 121.1C. Retort temperature was measured at the Steritort hub via the thermocouple circuitry. CNS thermocouples measured temperature at the geometric center of the test cans. The signals from the thermocouples were converted from mV to temperature readings and registered at 20 s intervals using a Kaye Digistrip III process monitor (Kaye Instruments, Inc., Bedford, MA) and a Toshiba T 1100 Plus laptop computer (Toshiba America, Inc., Irvine, CA). The time-temperature data were subsequently transferred to a VAX minicomputer (Digital Equipment Corporation, Maynard, MA) for plotting purposes and further analysis. Experimental data were discarded if the rotary contractors or the slip ring assembly caused excessive electrical noise resulting in erroneous temperature readings.

Heat Penetration Tests

The test product used in this study was tomato concentrate, a product which above certain solids concentration heats by conduction in the still mode and by convection in the agitating mode (above a critical rotational speed). Preliminary tests were conducted in order to determine the appropriate solids level of the tomato concentrate and the rotational speed to be used in the principal experiments. Cans (211×300) filled with tomato concentrate adjusted to 5, 7.5, 10 and 12.5 °Brix were processed at 3, 5, 7 and 9 rpm and heating factors were calculated from the time-temperature data. The 7.5 °Brix tomato concentrate showed similar heat transfer rates, as determined from the heating factors as several induced convection-heating products, such as cream style corn or puddings, currently processed in continuous rotary sterilizers, and was selected as the solids level to be used in all subsequent experiments.

Response Surface Methodology (RSM) computer program (Henika 1972) on an IBM personal computer (International Business Machines, Armonk, NY) was used for design of the study. Two RSM studies of 15 experiments each (for 3 independent variables and allowing for second order effects) and an additional confirmatory study of 4 experiments were conducted. Reel speed, air overpressure, and can surface area to volume ratio were chosen as the independent variables. The variables were input into the RSM program and the program sequenced the experiments for the two RSM studies. For both RSM studies, 211×300 , 300×407 , and 307×503 can sizes, were used, corresponding to can surface area to volume ratios of 84.9, 70.2 and 60.9 m^{-1} , respectively. These can sizes cover the range of the majority of can sizes processed commercially in continuous rotary sterilizers for induced convection-heating products. Furthermore,

these surface area to volume ratio values were reasonably equidistant apart and fit the RSM requirements. Air overpressures of 0 kPa (0 psig), 34.5 kPa (5 psig) and 69.0 kPa (10 psig), and reel rotational speeds of 4, 7 and 10 rpm were used for the first RSM study. Air overpressures of 6.9 kPa (1 psig), 20.7 kPa (3 psig) and 34.5 kPa (5 psig) and reel speeds of 6, 8 and 10 rpm were used for the second RSM study. Replicate experiments were included in the RSM design. Five replicate cans were used for each condition. The confirmatory experiments were conducted using the 211 × 300 or the 307 × 503 can sizes processed at 6 rpm with air overpressures of 0 or 69.0 kPa in order to obtain experimental data at conditions that were not tested in the two RSM studies.

The tomato concentrate for each of the three studies was prepared from the same product lot of 603 × 700 cans of tomato paste (from local supplier) adjusted to a target of 7.25 °Brix. For each study, the sauce was heated to 87.8C (190F) and covered to minimize evaporative losses. Tomato sauce was filled hot into cans allowing for a gross headspace of 6.4 mm (8/32 in.) for the 211 × 300 cans, 7.9 mm (10/32 in.) for the 300 × 407 cans, and 9.5 mm (12/32 in.) for 307 × 503 cans, seamed with a Rooney Seamer (Rooney Machine Company, Bellingham, WA), and held overnight to equilibrate the can contents to room temperature. Brookfield consistency and °Brix measurements were conducted on selected cans before and after processing. Degrees Brix, measured with a Bausch and Lomb refractometer (Rochester, NY) held at 20C by a Lo-temptrol water bath (Precision Scientific Company, Chicago, IL), ranged from 7.1 to 7.2, corresponding to an average density of 1014 kg/m³ (Lamb 1977). Product consistency was measured at 20C with a Brookfield viscometer (Brookfield Engineering Laboratories, Stoughton, MA), model RVF, set at 10 rpm using the "Helipath" procedure and the T-A spindle. For samples before processing, Brookfield consistency averaged 5.28 Pa s ± 6.4% for the first RSM study, 3.57 Pa s ± 5.8% for the second RSM study, and 3.85 Pa s ± 4.3% for the confirmatory study.

All heat penetration tests were conducted at 121.1C (250.0 F), and the Steritort vented with the pneumatic steam control valve and the steam by-pass valve open. The drain and vent valves were closed after 45 and 105 s, respectively, from the time steam was admitted to the Steritort. Air overpressure was introduced after the mercury-in-glass thermometer reached 121.1C. Heating was terminated after the slowest heating container reached 120.0C (248.0F), and subsequently the containers were cooled for 30 min with water introduced through the top and the bottom of the Steritort. Cooling was initiated under air overpressure (about 100 kPa) which was gradually released as the temperature of the cans decreased. After the cans were removed, the Steritort door was left wide open for at least 30 min to equilibrate the Steritort shell to room temperature for the subsequent runs (in order to obtain reproducible come-up times).

Data Analysis

Heat penetration factors (j and f_h) were calculated from regression analyses of the temperature-time plots. All heat penetration data were treated as simple heating curves. Process times were calculated by Ball's formula method (Ball 1928), using the VAX minicomputer, for a retort temperature of 121.1C (250.0F), an initial temperature of 21.1C (70C), a target lethality (F_0) value of 10.0 min, and assuming equal cooling and heating rates. A comparison of the results obtained using Ball's formula method with General Method calculations (using actual heating and cooling product temperature data) revealed that Ball's method gave conservative estimates of process times. This was mainly attributed to the delay period between steam-off and beginning of cooling; this delay period is not accounted for by Ball's formula method. Average heating factors and process times and their standard deviations were calculated from the five cans used for each experiment. Overall heat transfer coefficients (U_0) were calculated from the average f_h values (in seconds) of each experiment using the perfect mixing convection model equation (Merson *et al.* 1978).

$$f_h = \frac{2.303mC_p}{U_0A} \quad (1)$$

The experimental responses (f_h value, Ball process time, overall heat transfer coefficient) were input into the computer and, for both studies, the RSM program fit the data (each study separate) using a Taylor expansion equation (Eq. 2) and calculated the coefficient of determination (R^2) and the percent of first order (linear), second order (quadratic) and interaction effects. For example, for Ball process time (t_B),

$$t_B = \alpha_0 + \alpha_1(RS) + \alpha_2(OP) + \alpha_3(AV) + \alpha_4(RS)^2 + \alpha_5(OP)^2 + \alpha_6(AV)^2 + \alpha_7(RS)(OP) + \alpha_8(RS)(AV) + \alpha_9(OP)(AV) \quad (2)$$

The Ball process times from both RSM studies and the additional confirmatory study were also correlated as a single data set using SAS (1990) and the same Taylor expansion equation. Finally, the overall heat transfer coefficients from both RSM and the confirmatory studies were correlated using SAS (1990) for the following dimensionless equation.

$$Nu = \beta_0 + \beta_1 Re^{\beta_2} Pr^{\beta_3} (L_c/D_c)^{\beta_4} (P_s/P_a)^{\beta_5} \quad (3)$$

The above dimensionless correlation equation is similar to those reported by other investigators for axially rotated cans (Lenz and Lund 1978; Rao *et al.* 1985; Soule and Merson 1985) with the addition of the dimensionless term indicating the percent steam (by volume) in the steam/air mixtures. Values for

specific heat and thermal conductivity for the tomato concentrate, needed for the calculation of the various dimensionless numbers in Eq. (3), were obtained from literature correlations (Singh and Heldman 1984) based on the water content. The values of $C_p = 4000 \text{ J/(kgK)}$ and $k = 0.606 \text{ W/(mK)}$ were used.

RESULTS AND DISCUSSION

Heating factors, Ball process times, and overall heat transfer coefficients calculated from experimental time-temperature data for the two RSM studies and the confirmatory study are shown in Tables 1, 2 and 3, respectively. As the percent air increased, the f_h values and Ball process times increased and the overall heat transfer coefficients decreased. However, for the ranges of variables studied, the reel speed had the most pronounced effect. Heating rates increased with increasing reel rpm. In addition, smaller can sizes exhibited consistently lower f_h values. The effect of can size though, on the U_o was not monotonous, indicating a more complex, allowing for interactions between variables, dependency. Unusually large j values (14.81 maximum) can be attributed to a combination of the lower reel speed (4 rpm) used in this study and the assumption of simple, "straight line," heating curve used to calculate the heating factors. Although the preliminary experiments indicated that 4 rpm induced convection-heating, for the tomato concentrate used in the first RSM study (note the considerably higher consistency of 5.28 Pa s, versus 3.57 Pa s for the second RSM study, although concentrate of the same °Brix was used) a "broken-heating curve" was observed, and convection was not induced until viscosity was dropped due to the heating of the product.

Variability between cans within each experiment was measured by comparing the standard deviations of f_h values and Ball process times. Excluding the experiments conducted at 4 rpm, the maximum f_h standard deviation for all three studies was 28.5% (second RSM study, 211 × 300 can, 6 rpm, 20.7 kPa overpressure). Based on our experience, this amount of variability is to be expected for induced convection-heating products. The variability between cans within each experiment for Ball process times was not as large since this involves a calculation procedure and Ball's equations take into account both j and f_h values. The maximum standard deviation for Ball process times was 15.0% (second RSM study, 211 × 300 can, 6 rpm, 20.7 kPa overpressure).

Variability between replicate experiments was measured by comparing the ranges of average f_h values and overall heat transfer coefficients for the conditions where replicate experiments were performed. The reproducibility of the replicates for the two RSM studies was good. The average f_h values for the first RSM study ranged from 8.11 to 10.31 min. and for the second RSM study ranged from 3.93 to 4.54 min. The overall heat transfer coefficients for the three

TABLE 1.
RSM STUDY 1: EXPERIMENTAL CONDITIONS, HEATING FACTORS, BALL
PROCESS TIMES, AND OVERALL HEAT TRANSFER COEFFICIENTS

Can size	RS rpm	OP kPa	j^1	f_h^1 min	$t_B^{1,2}$ min	U_o W/(m ² K)
211 x 300	4	34.5	8.54 ± 10.6	8.62 ± 2.86	29.4 ± 2.66	155 ± 51*
	7	0.0	1.25 ± 0.40	4.15 ± 0.54	16.6 ± 0.84	317 ± 41
	7	69.0	1.10 ± 0.31	5.86 ± 0.31	19.1 ± 0.65	229 ± 12
	10	34.5	0.66 ± 0.08	3.90 ± 1.04	15.2 ± 1.20	342 ± 91
300 x 407	4	0.0	14.82 ± 7.13	6.51 ± 1.11	27.6 ± 1.74	266 ± 45
	4	69.0	8.87 ± 6.33	9.26 ± 3.53	31.0 ± 4.41	185 ± 71
	7	34.5	1.03 ± 0.11	10.31 ± 1.90	25.9 ± 3.04	170 ± 31
	7	34.5	0.93 ± 0.06	8.11 ± 0.54	22.4 ± 0.65	215 ± 14
	7	34.5	0.92 ± 0.06	8.23 ± 0.59	22.5 ± 0.86	212 ± 15
	10	0.0	0.84 ± 0.04	4.78 ± 0.20	16.9 ± 0.21	367 ± 15
	10	69.0	0.65 ± 0.02	6.21 ± 1.71	18.3 ± 2.35	278 ± 77
307 x 503	4	34.5	14.81 ± 5.84	10.00 ± 2.86	36.0 ± 3.93	206 ± 59
	7	0.0	3.17 ± 0.67	9.38 ± 0.95	29.0 ± 1.55	217 ± 22
	7	69.0	2.53 ± 0.72	10.94 ± 1.00	30.9 ± 2.75	185 ± 17
	10	34.5	0.81 ± 0.03	5.76 ± 0.32	18.3 ± 0.51	353 ± 20

¹ Average values (± one standard deviation) of at least 3 measurements.

² Based on a target $F_o = 10$ min.

* Based on the relative error of one standard deviation of the f_h value.

replicates of the first RSM study ranged from 170 to 215 W/(m²K) and of the second RSM study ranged from 362 to 416 W/(m²K).

Correlation coefficients (R^2) for f_h , Ball process time, and U_o for the Taylor expansion equations, calculated by the RSM program for each RSM study separately, ranged between 0.85 and 0.97. Overall, about 80% of the variation was attributed to first order effects, 10% to second order effects, and less than 1% to interaction effects. Treating Ball process times for both RSM studies and the confirmatory study as a single data set, the following coefficient values of 142.5, -9.075, 0.147, -2.097, 0.284, 0.000808, 0.0111, -0.00171, 0.0388, and -0.00192 for α_0 to α_9 in Eq. (2), respectively, were found with an R^2 of 0.852. It is worthy to note that a four variable model, from Eq. (2), with the

TABLE 2.
RSM STUDY 2: EXPERIMENTAL CONDITIONS, HEATING FACTORS, BALL
PROCESS TIMES, AND OVERALL HEAT TRANSFER COEFFICIENTS

Can size	RS rpm	OP kPa	j^1	f_h^1 min	$t_B^{1,2}$ min	U_o W/(m ² K)
211 x 300	6	20.7	1.30±0.50	5.82±1.66	19.4±2.91	233±66*
	8	6.9	0.74±0.07	3.96±0.27	15.5±0.29	346±24
	8	34.5	0.67±0.04	4.36±0.19	15.9±0.22	319±14
	10	20.7	0.62±0.01	3.40±0.10	14.5±0.10	399±12
300 x 407	6	6.9	1.46±0.25	5.62±0.44	19.5±0.98	295±23
	6	34.5	1.47±0.07	5.87±0.70	20.0±1.40	288±34
	8	20.7	1.00±0.17	3.93±0.22	16.0±0.47	416±23
	8	20.7	0.88±0.14	4.54±0.23	16.6±0.46	362±18
	8	20.7	1.10±0.14	4.19±0.16	16.5±0.16	392±15
	10	6.9	0.68±0.01	3.44±0.12	14.7±0.14	499±17
	10	34.5	0.74±0.15	3.68±0.23	15.1±0.20	455±28
307 x 503	6	20.7	1.88±0.20	8.02±0.65	24.7±1.26	253±21
	8	6.9	0.94±0.20	5.12±0.93	17.6±1.00	391±71
	8	34.5	0.84±0.06	5.32±0.19	17.7±0.19	382±14
	10	20.7	0.70±0.01	4.83±0.25	16.6±0.37	424±22

¹ Average values (\pm one standard deviation) of at least 4 measurements.

² Based on a target $F_o = 10$ min.

* Based on the relative error of one standard deviation of the f_h value.

following coefficients, $\alpha_0 = 67.49$, $\alpha_1 = -6.105$, $\alpha_3 = -0.250$, $\alpha_4 = 0.265$, and $\alpha_5 = 0.000793$ gave an almost equally high R^2 of 0.817. It must be realized that Eq. (2) is a regression equation which provides very limited insight to the physical phenomena under study. Its discussion is limited to the Ball process times as a means of an appropriate, quick way to see the magnitude of process times as they are affected by the changes in the variables within the experimental range studied.

TABLE 3.
CONFIRMATORY EXPERIMENTS: EXPERIMENTAL CONDITIONS, HEATING
FACTORS, BALL PROCESS TIMES, AND OVERALL HEAT TRANSFER
COEFFICIENTS

Can size	RS rpm	OP kPa	j^1	f_h^1 min	$t_B^{1,2}$ min	U_o W/(m ² K)
211 x 300	6	0.0	1.40±0.45	5.54±0.37	19.2±1.14	249±17*
	6	69.0	1.63±0.41	5.88±0.44	20.2±1.28	230±17
307 x 503	6	0.0	2.07±0.23	8.58±1.04	26.0±1.66	230±28
	6	69.0	1.61±0.19	12.12±1.88	30.8±3.22	164±25

¹ Average values (\pm one standard deviation) of at least 4 measurements.

² Based on a target $F_o = 10$ min.

* Based on the relative error of one standard deviation of the f_h value.

The SAS (1990) analysis of the data from all three studies (excluded the results at 4 rpm) yielded the following Nusselt correlation equation,

$$Nu = 0.703 + 0.417 Re^{1.103} Pr^{0.324} (L_c/D_c)^{0.069} (P_s/P_t)^{0.806} \quad (4)$$

with a linear regression (predicted versus experimental Nusselt numbers) R^2 equal to 0.884. A graphical representation of Eq. (4) is shown in Fig. 1.

The experimental ranges of the dimensionless groups appearing in Eq. (4) were: $1.99 \leq Re \leq 5.38$, $23500 \leq Pr \leq 34800$, $1.12 \leq L_c/D_c \leq 1.51$, and $0.75 \leq P_s/P_t \leq 1.00$. In order of increasing importance in the correlation, the dimensionless groups were: constant term (β_0), L_c/D_c , Pr , P_s/P_t , and Re . The surface area to volume ratio gave somewhat lower R^2 (0.837 vs. 0.884) compared to the L_c/D_c term. It is interesting to note that when the experiments at 4 rpm were included for the coefficient determination in Eq. (3), and the constant β_0 term assumed a value of 18.2, a contribution between 30 and 100% of the Nusselt number (recall the different nature of the heating curves obtained at 4 rpm).

As we mentioned earlier, Re (or rpm) had the most pronounced effect on the heat transfer coefficients. Figure 2 illustrates how an increase in the percent air in the steam/air medium can be compensated by increasing the reel rotational speed. Note that increase in percent air at the lower % air values required less rpm adjustment compared to high % air mixtures. The data presented in Fig. 2

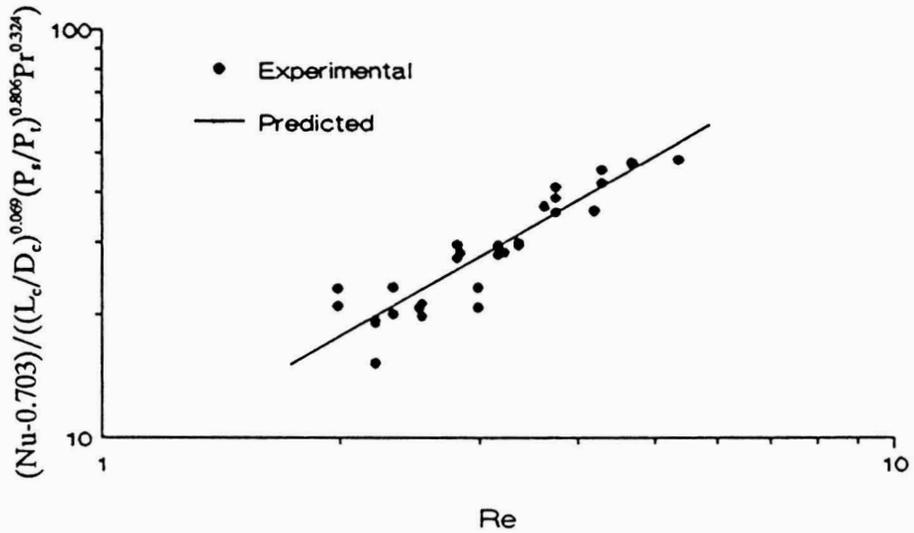


FIG. 1. DIMENSIONLESS CORRELATION FOR THE OVERALL HEAT TRANSFER COEFFICIENT

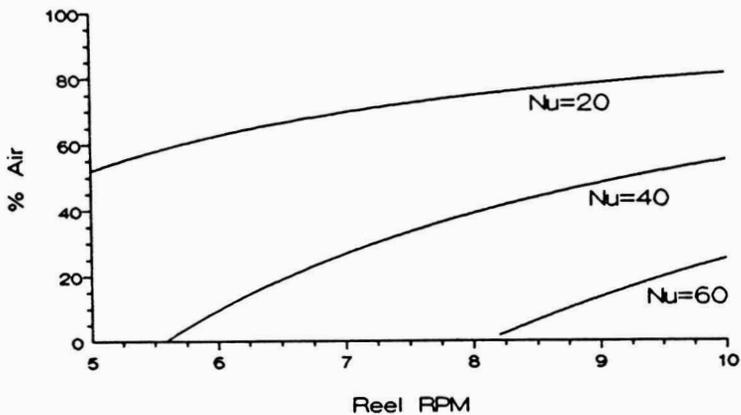


FIG. 2. EFFECTS OF INTERACTIONS BETWEEN REEL ROTATIONAL SPEED AND PERCENT AIR IN THE STEAM/AIR MEDIUM ON TARGET NUSSULT NUMBERS [THROUGH EQ. (4); FOR TOMATO CONCENTRATE WITH 4.5 Pa s CONSISTENCY IN 300 × 407 CANS]

were generated through Eq. (4) for a 300×407 can and a tomato concentrate with consistency of 4.5 Pa s , and these data are only valid for this particular case.

For a chosen can size and reel speed (5 to 10 rpm), an analysis on the percent reduction in process lethality, as a function of the percent air in the heating medium, was made. Once again, Eq. (4) (with $\mu = 4.5 \text{ Pa s}$) was used to generate Nu and hence U_0 values. From these U_0 values, f_h values were calculated through Eq. (1). Next, a Ball process time was calculated for $j = 1.0$, and f_h value corresponding to zero percent air, and a target lethality (F_0) of 10.0 min ($F_0 = 10.0 \text{ min}$ is a value frequently used for induced convection-heating low acid products). This process time was considered the base value for the can size and reel speed under consideration. Using this base process time, lethality values were then calculated for the f_h values corresponding to various % air concentrations (with $j = 1.0$), and percent reduction in lethality was calculated based on the target lethality. The above procedure was repeated for a target lethality of 7.0 min. Example results for two conditions are presented in Fig. 3.

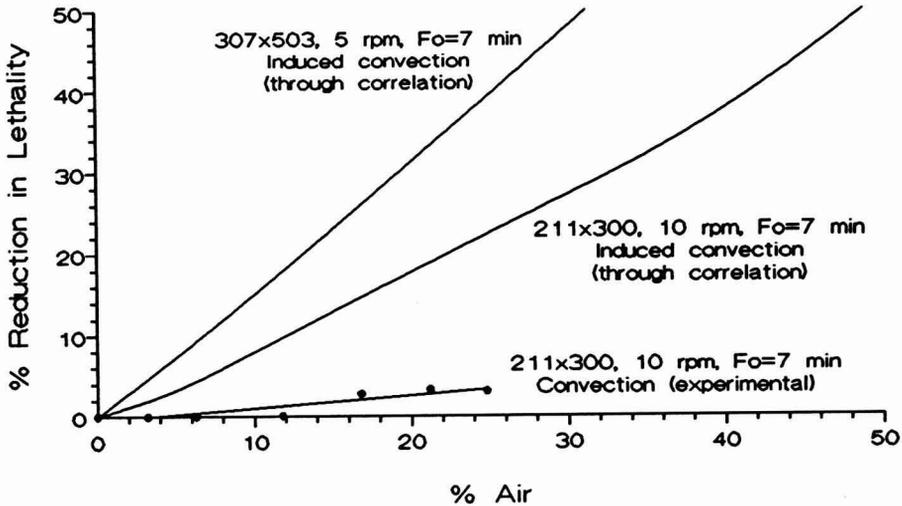


FIG. 3. PERCENT REDUCTION IN PROCESS LETHALITY AS A FUNCTION OF THE PERCENT AIR IN THE STEAM/AIR MEDIUM

For a given % air, higher percent reduction in lethalties were obtained with increasing can size, decreasing reel speed, and decreasing target F_0 value. In Fig. 3, experimental data (Deniston *et al.* 1991) for Washington white beans in brine (convection-heating) processed in 211×300 cans in a Steritort at 10 rpm, are also shown. Percent reductions in calculated lethality were higher for the induced convection-heating product compared to the convection-heating product.

SUMMARY AND CONCLUSION

Tomato concentrate (7.2 °Brix) was processed in a Steritort using steam and steam/air mixtures as the heating medium. The effects of percent air (0 – 25%) in the heating medium, reel rotational speed (4 – 10 rpm), and can size (211×300 , 300×407 , 307×503) on the heat transfer rates were investigated. A correlation equation for the Nusselt number as a function of the Reynolds and Prandtl numbers, the can length over the diameter ratio, and the steam over total processing pressure was developed. Reduced overall heat transfer coefficients were obtained by increasing the air content of the steam/air mixtures. However, this effect could be counterbalanced by an appropriate increase in reel rpm.

Process times were calculated from the heat penetration parameters using Ball's formula method. A Taylor expansion equation, relating Ball process times with percent air, reel rpm, and can surface to volume ratio, considering first order, second order, and interaction effects, was presented. Percent reduction in process lethality (based on a target lethality and the resulting process times for processes using 100% steam) increased with increasing can size and air content, and decreasing reel speed and target lethality. Finally, the percent reductions in process lethality were higher for the induced convection-heating product used in this study compared to literature values for a convection-heating (beans in brine) product.

NOMENCLATURE

Latin Symbols

A	Total external surface area of the can, m^2
AV	Can surface area to volume ratio, m^{-1}
C_p	Product specific heat, $J/(kgK)$
D_c	External can diameter, m
D_r	Internal reel diameter, m

F_0	Time at a constant reference temperature of 121.1C (250.0F) required to destroy a given percentage of microorganisms whose thermal resistance is characterized by $z = 10.0C$ (18.0F), min
f_h	Time required for the difference between heating medium and food temperature to traverse a logarithmic (base 10) cycle, min
j	Lag coefficient defined as $j = (T_{RT} - T_A)/(T_{RT} - T_{IT})$, where T_{RT} is the constant heating medium temperature of the process, T_{IT} the initial can center temperature, and T_A the extrapolated can center temperature, obtained by assuming an exponential function for the entire heating curve, and defined on the 58% of the come-up time axis.
k	Product thermal conductivity, W/(mK)
L_C	External can length, m
m	Product mass, kg
N	Can rotational speed, $N = (RS/60)(D_r/D_c)$, s^{-1}
Nu	Nusselt number, $Nu = U_0 D_c / k$, dimensionless
OP	Air overpressure, kPa
Pr	Prandtl number, $Pr = \mu C_p / k$, dimensionless
P_s	Absolute steam pressure, kPa
P_t	Absolute total pressure, kPa
Re	Reynolds number, $Re = \rho N D_c^2 / \mu$, dimensionless
RS	Reel speed, min^{-1}
t_B	Ball process time, min
U_0	Overall heat transfer coefficient based on the total external can surface, heating medium/can wall/internal fluid, W/(m^2K)

Greek Letters

$\alpha_0 - \alpha_9$	Coefficients in the Taylor expansion equation, Eq. (2), dimensionless
$\beta_0 - \beta_5$	Coefficients in the Nusselt number correlation Eq. (3), dimensionless
μ	Product consistency (as measured with Brookfield viscometer), Pa s
ρ	Product density, kg/m^3

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ROLE OF CULTIVAR AND PRESS AID IN PRESSING CHARACTERISTICS AND JUICE YIELDS OF CRUSHED GRAPES

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ABSTRACT

The stress-volume characteristics of crushed grape cultivars: Baco Noir, Cabernet Sauvignon, Catawba, Cayuga White, Chardonnay, Concord, Delaware, Melody, Niagara, Johannisberg Riesling, and Seyval Blanc, with and without paper and rice hulls press aids were studied in an expression cell and an Instron Universal Testing Machine. The slopes of ln stress vs volume in compression were significantly ($P < 0.001$) dependent on the grape cultivar. Addition of 1–3% press aid by weight decreased the magnitudes of slopes by about 11% with Seyval Blanc to about 51% with Catawba; i.e., the ease with which pressing could be performed increased with the addition of press aid. With Cabernet Sauvignon grapes, addition of press aid 0.5, 1.0, and 2.0% resulted in increase in slopes ranging between 14.7 and 24.9%. The slopes of the ln stress-ln time during the relaxation portion of the experiment were also significantly dependent ($P < 0.001$) on the grape cultivar; the magnitudes of the slopes increased with the addition of 1–3% paper press aid. The juice yields were affected significantly ($P < 0.001$) by the addition of paper press aid, with maximum yields being obtained when 1–2% (w/w) paper press aid was added. As expected, juice yields (w/w%) were significantly dependent ($P < 0.001$) on grape cultivars.

INTRODUCTION

Pressing is widely employed in food processing; in particular, it is an important operation in the production of fruit juices such as grape juice. Typically, in commercial grape juice production, the grapes are crushed prior to pressing and often press aids such as paper or rice hulls may be mixed with the crushed grapes in order to increase the juice yields. Presses used for juice extraction have been described in review papers (e.g., Swindells and Robbins 1966; Peden 1974) and in texts, e.g., Tressler and Joslyn (1961) and Downing (1988); therefore, they

will not be described here. McNulty (1980) discussed plant design for expression of juices that included a discussion of the quality of grape juice as affected by the type of press employed reported earlier by Kemperle and Kerner (1978). Kinzer and Schreier (1980) compared the volatile constituents in grape musts and wines of Morio-Muskat and Mueller-Thurgau grapes harvested in 1978 using a screw press and a bladder press (Willmes press). In the grape musts, the amounts of characteristic aroma compounds and terpene alcohols increased in general with pressure; they were 34–45% less when a cellulose press aid was employed. In the corresponding wines an increase of pressure during pressing resulted in increased formation of ethyl esters and acetates; the concentration of these fermentation by-products were highest in wines made using the cellulose press aid.

Schwartzberg (1983) and Schwartzberg *et al.* (1977, 1985) described in detail the techniques they developed for quantitative studies on the expression or pressing characteristics of many foods. Schwartzberg (1983) also reviewed the relationship between expression and filtration. In this work the two expressions “pressing characteristics” and “expression characteristics” will be used interchangeably. Two important expression characteristics that can provide useful information are the stress vs cake height or volume relationship during the compression stage and pressure vs time relationship during the relaxation stage. In addition, the juice yields and the role of press aids in juice yields are of considerable practical interest.

Several grape cultivars are grown in New York State for the production of either grape juice or wine. Because the New York State Agricultural Experiment Station produces on its vineyards each year the grape cultivars, an unique opportunity exists for examining the role of cultivar in pressing characteristics. The objectives of this study were: (1) to determine the pressing characteristics of several grape cultivars: Baco Noir, Cabernet Sauvignon, Catawba, Cayuga White, Chardonnay, Concord, Delaware, Niagara, Johannisberg Riesling, and Seyval Blanc in order to classify the cultivars with respect to the relative ease of pressing, (2) to ascertain the role of press aids in pressing characteristics and juice yields, and (3) to determine the role of maturity of Concord grapes on ease of pressing and juice yields.

MATERIALS AND METHODS

Grapes and Press Aids

Grapes of each cultivar: Baco Noir, Cabernet Sauvignon, Catawba, Cayuga White, Chardonnay, Concord, Delaware, Melody, Niagara, Johannisberg Riesling, and Seyval Blanc were hand picked from the vineyards of New York

State Agricultural Experiment Station and brought to the laboratory. In order to mix press aid with the grapes, weighed amounts of grapes and press aid were placed in a one liter beaker and mixed (Lightnin mixer, Rochester, NY) at 65 rpm for 3 min; in tests without press aid also the grapes were subjected to the mixing action, so that the mixing operation was a common denominator in all the tests.

During the 1988 growing season, we studied: (1) the role of the amount (0, 0.5, 1.0, 2.0, and 3.0% w/w) of paper press aid on pressing characteristics of all the cultivars, and (2) the role of paper and rice hulls as press aids was studied using Cayuga White grapes. The paper press aid was obtained by grinding a roll of paper (Georgianeer J, ITT Raynier Inc., New York) in a hammer mill (Fitzpatrick Co.) using blunt hammers at 4600 rpm, 0.25 in screen, paper fed at 3 ft/min). During the 1986 growing season, the role of grape maturity was studied using Concord grapes harvested on September 4 and 17, and October 14.

Expression Experiments

Figure 1 is a schematic diagram of the expression cell that was employed for obtaining force-ram movement data with the aid of an Instron Universal Testing Machine (Model TTCM, Canton, MA). The cell was a test unit (Fred S. Carver, Inc., Menomonee Falls, WI) whose base plate was cut to permit the out flow of juice. The procedure employed for recording force-ram distance data was similar to that described in detail by Schwartzberg *et al.* (1977) and Schwartzberg (1983). Briefly, a weighed amount of the grape pulp wrapped in cheese cloth was placed in the barrel and the initial position of the ram was noted; the grape mass was compressed by operating the ram at a fixed speed of 0.5 cm/min. The compression was carried out until a peak force of 2 kN was reached during which period the force-distance data were recorded on a chart paper at 2 cm/min. Following compression, the stress relaxation portion of the cycle was recorded as a function of time for about 5 min. Force-distance and the force-time records were digitized (MacTablet, Summagraphics, Fairfield, CT) and the data analyzed on a Macintosh Plus computer (Apple Computer, Inc., Cupertino, CA). The spent grape pulp samples were weighed at the end of the expression experiment and again after freeze-drying. All experiments were performed in duplicate so that the effects of different variables on observed results could be examined by analysis of variance (ANOVA) using a statistics program (GENSTAT, Numerical Algorithms Ltd., Oxford, UK) on a Prine 9750 computer.

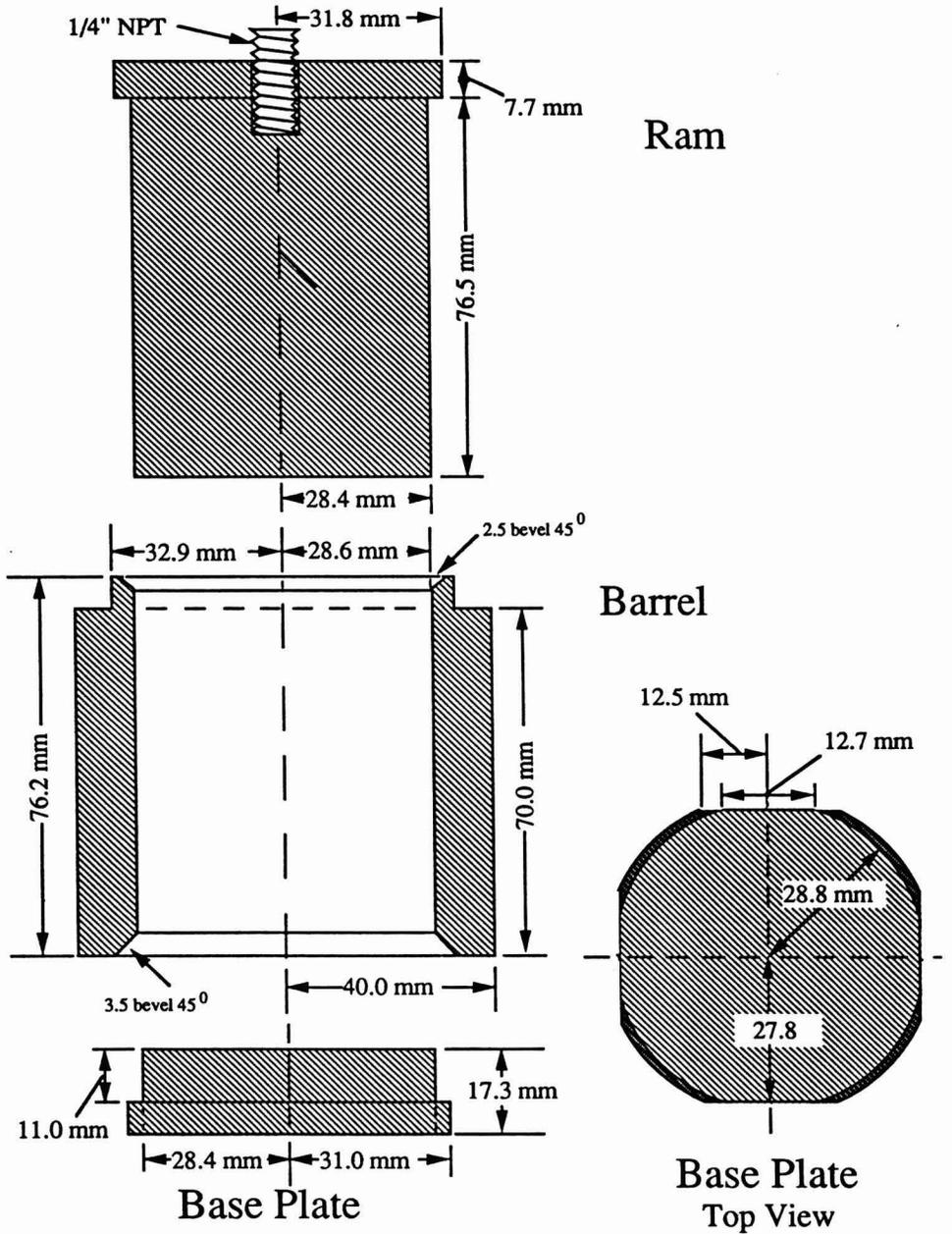


FIG. 1. THE PRINCIPAL PARTS OF THE EXPRESSION CELL EMPLOYED: RAM, BARREL, AND BASE PLATE; ALL DIMENSIONS ARE IN MM.

RESULTS AND DISCUSSION

The magnitudes of replicates of some of the experimental quantities that were recorded for Concord grapes are in Table 1. Similar data were obtained with the other grape cultivars. Typical force-distance curves during the compression and relaxation stages for Baco Noir and Concord grapes are shown in Fig. 2. Because the crushed grape mass was not homogeneous and the compression/relaxation strains were extremely large, data analysis based on application of linear viscoelastic theory would not be appropriate.

TABLE 1.
DATA RECORDED WITH CONCORD GRAPES

Test ^a	Ram Travel (cm)	Wt. Pulp and Cloth ^b (g)	Wt. of All Juice (g)	Dried Pulp and Cloth (g)	Total initial mass (g)
Rep1 control	1.47	67.2	55.1	17.0	125.5
Rep1 0.5%	1.75	44.3	78.9	14.0	125.4
Rep1 1%	2.22	39.9	83.5	13.9	125.2
Rep1 2%	2.56	44.6	79.3	15.7	125.5
Rep1 3%	2.85	40.0	84.3	15.6	125.6
Rep2 control	1.66	66.6	55.1	16.7	125.4
Rep2 0.5%	1.90	48.2	74.1	14.8	125.4
Rep2 1%	2.10	42.3	80.5	14.0	125.2
Rep2 2%	2.75	42.2	81.0	15.5	125.4
Rep2 3%	3.04	37.5	85.9	14.9	125.5

^aRep is replicate; control test is without any press aid; 0.5, 1, 2, and 3% represent wt% paper press aid.

^bWeight of grape pulp and cloth at end of expression test.

Analysis of Compression Portion of Curves

The force-time data during the compression stage were converted to force-distance data using the cross head speed. The ln stress-ram distance data were analyzed in terms of a linear equation and a second order polynomial equation. While the polynomial equation provided better magnitudes of R^2 , the coefficients a, b, and c did not show clear trends with respect to press aid aided. This is seen in Table 2 with data on Concord, Cayuga White, and Johannisberg Riesling grapes. In comparison to the exponential relationship, linear regression of ln stress-ln ram distance data yielded lower magnitudes of R^2 . Analysis of the compression data with other stress-distance equations was not undertaken because, as discussed later, valuable information was obtained with the relaxation curves.

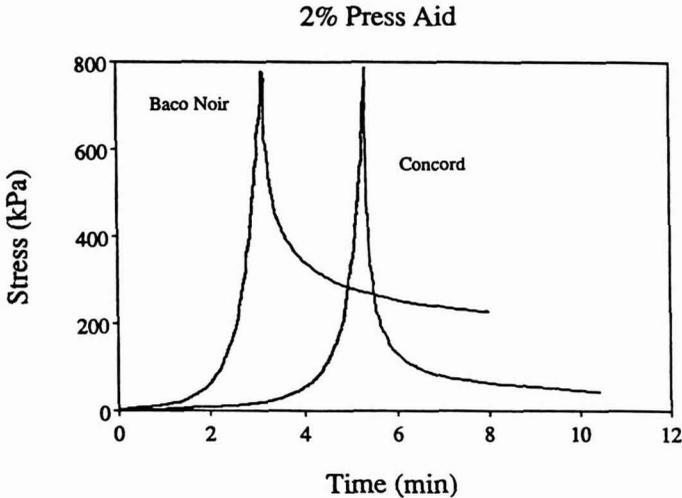


FIG. 2. STRESS (kPa) VS TIME (min) RECORDS OF BACO NOIR AND CONCORD GRAPES CONTAINING 2% PAPER PRESS AID; EACH CURVE HAS FIRST A COMPRESSION STAGE FOLLOWED BY A RELAXATION STAGE

The magnitudes of slopes, intercepts, and of R^2 of the \ln stress-ram distance during compression, and time of compression of all grape cultivars are in Table 3. The magnitudes of R^2 ranged from 0.88 to 0.99. Because the same ram was used in all the tests and it had a constant area of cross-section, the slopes of \ln stress-ram distance during compression are equal to those of \ln stress-volume plots.

The slopes were significantly ($P < 0.001$) dependent on the grape cultivar; in order of decreasing slopes, the cultivars were: Baco Noir, Catawba, Cayuga White, Chardonnay, Concord, Melody, Niagara, Seyval Blanc, Johannisberg Riesling, and Cabernet Sauvignon; there was a more than two-fold difference in the magnitudes of slopes of Baco Noir and Cabernet Sauvignon; in terms of ease of pressing, Baco Noir was the most difficult cultivar, while Cabernet Sauvignon was the least difficult.

They also were affected significantly ($P < 0.001$) by the addition of paper press aid. Addition of press aid in the amount of 3% by weight decreased the magnitudes of slopes by about 11% for Seyval Blanc to about 51% with Catawba, i.e., the ease with which pressing could be performed increased with the addition of press aid. The only exception to this general observation was with Cabernet Sauvignon grapes where addition of press aid 0.5, 1.0, and 2.0% resulted in increase in the \ln stress-volume slopes ranging between 14.7 and 24.9%, and decrease with the addition of 3% press aid. Therefore, with Cabernet Sauvignon grapes addition of paper press aid up to about 2% did not improve the ease of pressing.

TABLE 2.
COMPRESSION OF LINEAR VS POLYNOMIAL EXPRESSIONS
FOR COMPRESSION CURVES^a

Concord					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	4.05 ± 0.07	-3.23 ± 0.25	-	0.96	3.51 ± 0.27
0.5%	4.45 ± 0.79	-2.83 ± 0.12	-	0.98	3.15 ± 0.41
1.0%	2.87 ± 0.12	-2.45 ± 0.49	-	0.95	4.32 ± 0.13
2.0%	2.34 ± 0.11	-2.36 ± 0.07	-	0.96	5.26 ± 0.12
3.0%	2.35 ± 0.20	-2.59 ± 0.52	-	0.97	5.52 ± 0.08
Polynomial					
0.0%	-0.22 ± 0.66	-1.33 ± 0.92	2.29 ± 0.99	1.00	3.51 ± 0.27
0.5%	-3.33 ± 3.93	3.49 ± 6.29	0.88 ± 1.92	1.00	3.15 ± 0.41
1.0%	-0.18 ± 1.44	-1.63 ± 1.65	1.71 ± 0.56	1.00	4.32 ± 0.13
2.0%	-0.35 ± 0.04	-0.91 ± 0.06	1.01 ± 0.04	1.00	5.26 ± 0.12
3.0%	-0.89 ± 0.24	-0.21 ± 0.23	0.75 ± 0.07	1.00	5.52 ± 0.08
Cayuga White					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	4.66 ± 0.64	-1.89 ± 0.69	-	0.98	2.71 ± 0.11
0.5%	3.91 ± 0.79	-3.32 ± 0.88	-	0.96	3.68 ± 0.28
1.0%	4.73 ± 0.91	-3.26 ± 0.63	-	0.98	3.17 ± 0.83
2.0%	2.96 ± 0.38	-3.00 ± 0.32	-	0.96	4.74 ± 0.20
3.0%	2.66 ± 0.03	-2.77 ± 0.62	-	0.96	4.97 ± 0.42
Polynomial					
0.0%	-4.15 ± 0.40	8.30 ± 1.63	-1.43 ± 1.12	0.99	2.71 ± 0.11
0.5%	0.05 ± 0.01	-2.62 ± 0.53	2.72 ± 0.55	1.00	3.68 ± 0.28
1.0%	-0.69 ± 0.35	-0.24 ± 0.15	2.36 ± 1.13	1.00	3.17 ± 0.83
2.0%	-0.95 ± 0.87	-0.78 ± 1.00	1.27 ± 0.18	1.00	4.74 ± 0.20
3.0%	0.00 ± 0.10	-1.23 ± 0.52	1.25 ± 0.28	1.00	4.97 ± 0.42
Johannisberg Riesling					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	3.05 ± 1.09	-2.61 ± 0.38	-	0.92	4.22 ± 0.98
0.5%	3.92 ± 0.93	-2.63 ± 0.01	-	0.96	3.38 ± 0.83
1.0%	3.65 ± 0.48	-2.89 ± 0.70	-	0.97	3.74 ± 0.11
2.0%	2.61 ± 0.12	-2.87 ± 0.17	-	0.95	5.13 ± 0.16
3.0%	2.54 ± 0.37	-2.55 ± 0.04	-	0.96	5.05 ± 0.63
Polynomial					
0.0%	1.15 ± 0.70	-4.08 ± 0.43	2.77 ± 0.85	0.99	4.22 ± 0.98
0.5%	-0.29 ± 0.13	-1.81 ± 0.06	2.86 ± 1.07	1.00	3.38 ± 0.83
1.0%	-0.58 ± 0.49	-1.21 ± 1.39	2.06 ± 0.71	1.00	3.74 ± 0.11
2.0%	-0.55 ± 0.47	-1.26 ± 0.25	1.23 ± 0.03	1.00	5.13 ± 0.16
3.0%	-0.69 ± 0.25	-0.69 ± 0.06	1.07 ± 0.23	1.00	5.05 ± 0.63

^a Linear expression: $\ln(\text{Stress}) = a + b(\text{Distance})$

Polynomial expression: $\ln(\text{Stress}) = a + b(\text{distance}) + c(\text{Distance})^2$

TABLE 3.
CALCULATED VALUES OF SLOPES AND INTERCEPTS OF COMPRESSION DATA^a

Press Aid	Slope	Intercept	R ²	Interval (min)
		Baco Noir		
0.0%	6.15 ± 0.24	-2.11 ± 0.32	0.98	2.02 ± 0.03
0.5%	4.35 ± 0.24	-1.81 ± 0.13	0.99	2.72 ± 0.03
1.0%	4.17 ± 0.37	-2.20 ± 0.27	0.98	2.99 ± 0.30
2.0%	3.62 ± 0.02	-1.58 ± 0.06	0.98	3.15 ± 0.02
3.0%	3.38 ± 0.19	-2.07 ± 0.68	0.98	3.66 ± 0.56
		Seyval Blanc		
0.0%	3.26 ± 0.85	-2.71 ± 0.43	0.88	4.03 ± 1.22
0.5%	4.46 ± 0.69	-2.72 ± 0.37	0.98	3.03 ± 0.17
1.0%	4.13 ± 0.15	-3.10 ± 0.50	0.98	3.43 ± 0.14
2.0%	2.90 ± 0.12	-2.13 ± 0.06	0.96	4.08 ± 0.11
3.0%	2.62 ± 0.40	-2.45 ± 0.07	0.97	4.88 ± 0.68
		Niagara		
0.0%	3.77 ± 0.64	-1.96 ± 0.00	0.96	3.23 ± 0.55
0.5%	3.08 ± 0.02	-2.37 ± 0.18	0.94	3.97 ± 0.11
1.0%	3.78 ± 0.16	-3.75 ± 1.55	0.98	4.07 ± 1.01
2.0%	2.71 ± 0.56	-2.55 ± 0.26	0.97	4.83 ± 1.10
3.0%	2.30 ± 0.35	-2.44 ± 0.00	0.97	5.56 ± 0.82
		Delaware		
0.0%	3.35 ± 0.50	-3.24 ± 1.45	0.94	4.10 ± 0.24
0.5%	2.88 ± 0.51	-2.68 ± 0.44	0.95	4.46 ± 0.47
1.0%	3.40 ± 0.16	-4.22 ± 0.80	0.98	4.77 ± 0.34
2.0%	2.43 ± 0.59	-3.17 ± 0.77	0.96	5.78 ± 0.56
3.0%	2.12 ± 0.15	-2.49 ± 0.10	0.96	5.92 ± 0.25
		Concord		
0.0%	4.05 ± 0.07	-3.23 ± 0.25	0.96	3.51 ± 0.27
0.5%	4.45 ± 0.79	-2.83 ± 0.12	0.98	3.15 ± 0.41
1.0%	2.87 ± 0.12	-2.45 ± 0.49	0.95	4.32 ± 0.13
2.0%	2.34 ± 0.11	-2.36 ± 0.07	0.96	5.26 ± 0.12
3.0%	2.35 ± 0.20	-2.59 ± 0.52	0.97	5.52 ± 0.08
		Catawba		
0.0%	4.90 ± 1.18	-2.39 ± 0.22	0.98	2.70 ± 0.52
0.5%	3.07 ± 0.25	-2.58 ± 0.46	0.95	4.13 ± 0.07
1.0%	2.86 ± 0.67	-2.00 ± 0.01	0.96	4.18 ± 0.81
2.0%	2.37 ± 0.06	-2.14 ± 0.06	0.95	5.03 ± 0.14
3.0%	2.08 ± 0.07	-2.13 ± 0.06	0.95	5.69 ± 0.12
		Cayuga White		
0.0%	4.66 ± 0.64	-1.89 ± 0.69	0.98	2.71 ± 0.11
0.5%	3.91 ± 0.79	-3.32 ± 0.88	0.96	3.68 ± 0.28
1.0%	4.73 ± 0.91	-3.26 ± 0.63	0.98	3.17 ± 0.83
2.0%	2.96 ± 0.38	-3.00 ± 0.32	0.96	4.74 ± 0.20
3.0%	2.66 ± 0.03	-2.77 ± 0.62	0.96	4.97 ± 0.42
		Chardonnay		
0.0%	4.61 ± 1.93	-2.80 ± 0.81	0.95	3.01 ± 0.81
0.5%	2.90 ± 0.16	-2.43 ± 0.12	0.91	4.20 ± 0.14
1.0%	3.72 ± 0.54	-2.45 ± 0.39	0.97	3.44 ± 0.72
2.0%	2.65 ± 0.16	-2.26 ± 0.10	0.94	4.54 ± 0.31
3.0%	2.42 ± 0.14	-2.77 ± 0.00	0.95	5.40 ± 0.26
		Johannisberg Riesling		
0.0%	3.05 ± 1.09	-2.61 ± 0.38	0.92	4.22 ± 0.98
0.5%	3.92 ± 0.93	-2.63 ± 0.01	0.96	3.38 ± 0.83
1.0%	3.65 ± 0.48	-2.89 ± 0.70	0.97	3.74 ± 0.11
2.0%	2.61 ± 0.12	-2.87 ± 0.17	0.95	5.13 ± 0.16
3.0%	2.54 ± 0.37	-2.55 ± 0.04	0.96	5.05 ± 0.63
		Melody		
0.0%	3.97 ± 0.34	-3.27 ± 0.98	0.97	3.79 ± 0.29
0.5%	4.52 ± 0.11	-3.66 ± 1.12	0.98	3.38 ± 0.48
1.0%	4.94 ± 1.35	-2.99 ± 1.28	0.98	3.06 ± 1.27
2.0%	3.21 ± 0.69	-3.39 ± 0.46	0.95	4.59 ± 0.59
3.0%	2.79 ± 0.91	-2.47 ± 0.05	0.96	4.77 ± 1.35
		Cabernet Sauvignon		
0.0%	2.61 ± 0.35	-2.67 ± 0.69	0.91	4.71 ± 0.06
0.5%	3.26 ± 0.27	-2.36 ± 0.27	0.95	3.76 ± 0.46
1.0%	3.01 ± 0.37	-2.81 ± 0.18	0.94	4.39 ± 0.35
2.0%	3.02 ± 0.41	-3.33 ± 0.76	0.98	4.81 ± 0.01
3.0%	2.09 ± 0.13	-2.00 ± 0.05	0.93	5.48 ± 0.20

^a Slope, intercept, and R² are from linear regression of Ln(Stress) vs Distance; Interval (min) is time of compression; Press aid is paper.

TABLE 4.
COMPARISON OF LINEAR VS POLYNOMIAL EXPRESSIONS
FOR RELAXATION CURVES^a

Concord					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	1.23 ± 0.24	-0.73 ± 0.00	-	0.96	1.02 ± 0.30
0.5%	1.53 ± 0.32	-0.70 ± 0.09	-	0.95	1.44 ± 0.49
1.0%	2.00 ± 0.04	-0.56 ± 0.05	-	0.96	3.07 ± 0.71
2.0%	2.35 ± 0.01	-0.49 ± 0.01	-	0.96	4.39 ± 0.13
3.0%	2.70 ± 0.01	-0.40 ± 0.01	-	0.96	7.32 ± 0.51
Polynomial					
0.0%	1.29 ± 0.26	-0.79 ± 0.03	-0.03 ± 0.01	0.99	1.02 ± 0.30
0.5%	1.60 ± 0.35	-0.76 ± 0.07	-0.03 ± 0.01	1.00	1.44 ± 0.49
1.0%	1.99 ± 0.02	-0.57 ± 0.09	-0.02 ± 0.00	1.00	3.07 ± 0.71
2.0%	2.40 ± 0.01	-0.55 ± 0.01	-0.03 ± 0.00	1.00	4.39 ± 0.13
3.0%	2.74 ± 0.02	-0.45 ± 0.02	-0.03 ± 0.01	0.99	7.32 ± 0.51
Cayuga White					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	0.89 ± 0.11	-0.70 ± 0.03	-	0.94	0.90 ± 0.18
0.5%	2.13 ± 0.33	-0.52 ± 0.08	-	0.96	3.85 ± 1.55
1.0%	2.57 ± 0.14	-0.40 ± 0.04	-	0.96	6.63 ± 0.97
2.0%	3.28 ± 0.07	-0.27 ± 0.04	-	0.96	16.51 ± 1.96
3.0%	3.41 ± 0.05	-0.24 ± 0.01	-	0.96	19.60 ± 1.43
Polynomial					
0.0%	0.82 ± 0.06	-0.67 ± 0.01	0.03 ± 0.02	0.99	0.90 ± 0.18
0.5%	2.16 ± 0.32	-0.55 ± 0.08	-0.02 ± 0.01	0.99	3.85 ± 1.55
1.0%	2.60 ± 0.17	-0.43 ± 0.01	-0.02 ± 0.01	1.00	6.63 ± 0.97
2.0%	3.31 ± 0.08	-0.30 ± 0.03	-0.02 ± 0.01	1.00	16.51 ± 1.96
3.0%	3.45 ± 0.05	-0.27 ± 0.01	-0.02 ± 0.00	1.00	19.60 ± 1.43
Johannisberg Riesling					
Linear					
Press Aid	a	b	c	R ²	End Stress (Pa. x 10 ⁻⁴)
0.0%	1.60 ± 0.36	-0.68 ± 0.03	-	0.96	1.68 ± 0.71
0.5%	2.13 ± 0.13	-0.55 ± 0.08	-	0.96	3.25 ± 0.87
1.0%	2.76 ± 0.17	-0.41 ± 0.05	-	0.96	7.57 ± 2.14
2.0%	3.10 ± 0.00	-0.32 ± 0.03	-	0.95	11.90 ± 0.76
3.0%	3.33 ± 0.02	-0.29 ± 0.02	-	0.95	16.35 ± 0.95
Polynomial					
0.0%	1.67 ± 0.33	-0.75 ± 0.01	-0.03 ± 0.01	1.00	1.68 ± 0.71
0.5%	2.19 ± 0.10	-0.62 ± 0.11	-0.03 ± 0.01	1.00	3.25 ± 0.87
1.0%	2.82 ± 0.16	-0.46 ± 0.06	-0.03 ± 0.01	1.00	7.57 ± 2.14
2.0%	3.16 ± 0.01	-0.37 ± 0.04	-0.03 ± 0.01	1.00	11.90 ± 0.76
3.0%	3.37 ± 0.01	-0.33 ± 0.03	-0.03 ± 0.01	1.00	16.35 ± 0.95

^a Linear expression: $\ln(\text{Stress}) = a + b(\ln \text{time})$

Polynomial expression: $\ln(\text{Stress}) = a + b(\ln \text{time}) + c(\ln \text{time})^2$

Analysis of Relaxation Portion of Curves

Better correlations were obtained for relaxation stress-time data for a power law relationship than with an exponential relationship. The use of a second order polynomial equation resulted in small values of the second order term. This is seen in Table 4 with data on Concord, Cayuga White, and Johannisberg Riesling grapes. Typical fit of data to power relationships is shown in Fig. 3 for Concord grapes with 1% and 3% paper press aid. The magnitudes of slopes, intercepts, R^2 , and stresses after 5 min for all grape cultivars are given in Table 5. The magnitudes of R^2 for \ln relaxation stress- \ln time correlations were high ranging from 0.94 to 0.96; the only exception was one set of data with Melody grapes containing 2% press aid with $R^2 = 0.80$. The slopes were also significantly dependent ($P < 0.001$) on the grape cultivar, but the differences in magnitudes were not as large as those of the \ln stress-ram distance. In general, the magnitudes of the slopes increased with the addition of 1-3% paper press aid. Further, in comparison to compression data (Table 3), the relaxation data (Table 5) indicated better the effect of added press aid.

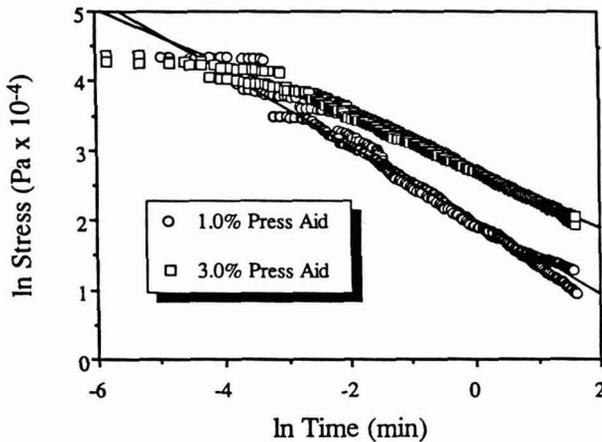


FIG. 3. \ln (STRESS) VS \ln (TIME) PLOTS OF REPLICATED RELAXATION DATA FOR CONCORD GRAPES WITH 1% and 3% PAPER PRESS AID

Magnitudes of stresses noted after five minutes of relaxation (σ_{T5}) were significantly affected ($P < 0.001$) by both the grape cultivar and by the press aid. Although the 5 min interval was arbitrary, it was selected in an attempt to standardize the time allowed for the stress to relax and to provide a common time for comparison. In particular, the addition of paper press aid resulted in 10 to 20-fold increase in σ_{T5} .

Juice Yields

Magnitudes of free run and total juice yields with and without paper press aids, and volume change during compression, for all grape cultivars are in Table 6. As expected, juice yields (wt %) were significantly dependent ($P < 0.001$) on grape cultivars and in decreasing order they were: Chardonnay, Baco Noir, Melody, Seyval Blanc, Johannisberg Riesling, Delaware, Cabernet Sauvignon, Cayuga White, Catawba, Niagara, and Concord. The yields also were affected significantly ($P < 0.001$) by the addition of paper press aid, with maximum yields being obtained when 1–2 wt% paper press aid was added; the increase in juice yields due to the addition of 1% paper press aid ranged between about 9 and 49%. Interaction effects of grape cultivar and amount of press aid were found to be significant ($P < 0.001$). However, further examination of the tables of mean values and the error mean square values revealed that the main effects of cultivar and of amount of press aid were significant.

Rice Hulls and Paper Press Aids

Juice yields were significantly affected, as stated earlier, by the amount of press aid (0.5 – 3.0), but were not affected by the type of press aid; i.e., there were no significant differences in the yields with paper and with rice hulls as press aids. In this context, it should be noted that when rice hulls are used the extracted juices tend to have more suspended solids (Rao *et al.* 1986). Also as stated earlier, magnitudes of σ_{T5} were significantly affected by the amount of press aid used; the magnitudes of σ_{T5} increased with increase in press aid aid with both rice hulls and paper.

Maturity of Grapes

The maturity of harvest date on Concord grapes was not a significant factor in juice yields or in the aforementioned pressing characteristics. Not surprisingly, the amount of press aid had a significant affect ($P < 0.001$) on the juice yield, on the slopes during compression and relaxation, and on σ_5 .

TABLE 5.
CALCULATED VALUES OF SLOPES AND INTERCEPTS OF RELAXATION DATA^a

Press Aid	Slope	Intercept	R ²	End Stress (Pa x 10 ⁻⁴)
		Baco Noir		
0.0%	-0.75 ± 0.09	1.37 ± 0.19	0.96	1.10 ± 0.45
0.5%	-0.42 ± 0.04	2.69 ± 0.13	0.97	7.32 ± 1.18
1.0%	-0.35 ± 0.10	3.02 ± 0.20	0.97	11.69 ± 3.73
2.0%	-0.23 ± 0.00	3.44 ± 0.07	0.96	20.98 ± 2.61
3.0%	-0.20 ± 0.02	3.54 ± 0.04	0.96	24.12 ± 2.23
		Seyval Blanc		
0.0%	-0.65 ± 0.02	1.41 ± 0.02	0.96	1.40 ± 0.07
0.5%	-0.46 ± 0.01	2.53 ± 0.09	0.96	5.62 ± 0.58
1.0%	-0.37 ± 0.01	2.84 ± 0.07	0.95	8.80 ± 0.61
2.0%	-0.33 ± 0.03	3.12 ± 0.04	0.96	12.55 ± 1.12
3.0%	-0.26 ± 0.01	3.33 ± 0.03	0.94	16.77 ± 0.93
		Niagara		
0.0%	-0.73 ± 0.09	0.95 ± 0.33	0.96	0.77 ± 0.37
0.5%	-0.66 ± 0.02	1.47 ± 0.05	0.96	1.60 ± 0.00
1.0%	-0.51 ± 0.04	2.10 ± 0.02	0.96	3.53 ± 0.15
2.0%	-0.33 ± 0.03	2.88 ± 0.09	0.96	10.37 ± 1.67
3.0%	-0.29 ± 0.04	3.18 ± 0.12	0.96	15.25 ± 2.75
		Delaware		
0.0%	-0.63 ± 0.04	1.82 ± 0.15	0.96	2.14 ± 0.35
0.5%	-0.54 ± 0.00	2.14 ± 0.02	0.96	3.24 ± 0.13
1.0%	-0.35 ± 0.00	2.83 ± 0.10	0.96	9.18 ± 1.30
2.0%	-0.26 ± 0.02	3.27 ± 0.17	0.96	16.88 ± 3.58
3.0%	-0.25 ± 0.01	3.32 ± 0.02	0.96	17.53 ± 0.07
		Concord		
0.0%	-0.73 ± 0.00	1.23 ± 0.24	0.96	1.02 ± 0.30
0.5%	-0.70 ± 0.09	1.53 ± 0.32	0.95	1.44 ± 0.49
1.0%	-0.56 ± 0.05	2.00 ± 0.04	0.96	3.07 ± 0.71
2.0%	-0.49 ± 0.01	2.35 ± 0.01	0.96	4.39 ± 0.13
3.0%	-0.40 ± 0.01	2.70 ± 0.01	0.96	7.32 ± 0.51
		Catawba		
0.0%	-0.72 ± 0.04	1.44 ± 0.20	0.96	1.34 ± 0.24
0.5%	-0.49 ± 0.04	2.29 ± 0.03	0.95	4.23 ± 0.18
1.0%	-0.37 ± 0.01	2.79 ± 0.13	0.96	8.97 ± 0.88
2.0%	-0.29 ± 0.01	3.10 ± 0.01	0.96	13.27 ± 0.33
3.0%	-0.31 ± 0.03	3.19 ± 0.06	0.97	14.19 ± 1.72
		Cayuga White		
0.0%	-0.70 ± 0.03	0.89 ± 0.11	0.94	0.90 ± 0.18
0.5%	-0.52 ± 0.08	2.13 ± 0.33	0.96	3.85 ± 1.55
1.0%	-0.40 ± 0.04	2.57 ± 0.14	0.96	6.63 ± 0.97
2.0%	-0.27 ± 0.04	3.28 ± 0.07	0.96	16.51 ± 1.96
3.0%	-0.24 ± 0.01	3.41 ± 0.05	0.96	19.60 ± 1.43
		Chardonnay		
0.0%	-0.64 ± 0.07	1.71 ± 0.46	0.96	2.06 ± 1.30
0.5%	-0.47 ± 0.00	2.39 ± 0.11	0.96	5.12 ± 0.48
1.0%	-0.35 ± 0.04	2.97 ± 0.17	0.96	10.63 ± 2.46
2.0%	-0.27 ± 0.02	3.27 ± 0.09	0.95	16.19 ± 1.94
3.0%	-0.26 ± 0.01	3.42 ± 0.01	0.96	19.39 ± 0.34
		Johannisberg Riesling		
0.0%	-0.68 ± 0.03	1.60 ± 0.36	0.96	1.68 ± 0.71
0.5%	-0.55 ± 0.08	2.13 ± 0.13	0.96	3.25 ± 0.87
1.0%	-0.41 ± 0.05	2.76 ± 0.17	0.96	7.57 ± 2.14
2.0%	-0.32 ± 0.03	3.10 ± 0.00	0.95	11.90 0.76
3.0%	-0.29 ± 0.02	3.33 ± 0.02	0.95	16.35 0.95
		Melody		
0.0%	-0.77 ± 0.00	1.36 ± 0.12	0.96	1.13 ± 0.02
0.5%	-0.49 ± 0.06	2.31 ± 0.18	0.96	4.19 ± 1.45
1.0%	-0.35 ± 0.02	2.94 ± 0.01	0.96	10.01 ± 0.56
2.0%	-0.27 ± 0.11	3.19 ± 0.34	0.80	23.17 ± 18.53
3.0%	-0.24 ± 0.01	3.39 ± 0.04	0.95	18.85 ± 1.56
		Cabernet Sauvignon		
0.0%	-0.68 ± 0.09	1.35 ± 0.27	0.95	1.43 ± 0.65
1.0%	-0.42 ± 0.02	2.83 ± 0.08	0.96	7.75 ± 1.24
2.0%	-0.36 ± 0.04	3.07 ± 0.04	0.94	10.78 ± 1.26
3.0%	-0.33 ± 0.00	3.22 ± 0.08	0.96	13.26 ± 2.10
0.5%	-0.51 ± 0.04	2.41 ± 0.04	0.96	4.49 ± 0.42

^a Slope, intercept, and R² are from linear regression of Ln(Stress) vs Ln(time); End Stress is stress after 5 min relaxation.

TABLE 6.
JUICE YIELDS FOR ALL GRAPE CULTIVARS^a

Press Aid	Free Run Juice	Total Juice Yield	Solids Loss	Delta Volume
		Baco Noir		
0.0%	49.0 ± 1.9	71.1 ± 0.7	2.80 ± 0.11	25.2 ± 0.2
0.5%	49.1 ± 4.2	77.3 ± 0.5	2.76 ± 0.28	33.9 ± 0.2
1.0%	45.7 ± 2.7	77.8 ± 1.6	2.07 ± 0.00	37.4 ± 1.5
2.0%	38.3 ± 0.1	77.0 ± 0.2	1.95 ± 0.06	39.1 ± 0.2
3.0%	27.1 ± 1.9	75.2 ± 0.0	2.00 ± 0.11	46.1 ± 6.5
		Seyval Blanc		
0.0%	41.4 ± 4.1	69.0 ± 0.9	1.52 ± 0.45	28.3 ± 0.5
0.5%	41.6 ± 2.4	73.2 ± 1.0	1.59 ± 0.33	40.0 ± 0.0
1.0%	39.7 ± 0.1	74.9 ± 0.1	1.72 ± 0.28	36.0 ± 8.2
2.0%	32.6 ± 1.9	74.6 ± 1.2	1.71 ± 0.17	51.6 ± 1.6
3.0%	23.6 ± 4.1	71.6 ± 1.0	1.43 ± 0.68	68.9 ± 2.2
		Niagara		
0.0%	14.8 ± 1.1	50.5 ± 1.1	2.96 ± 0.56	42.4 ± 5.6
0.5%	25.3 ± 3.9	63.1 ± 3.0	2.27 ± 0.73	51.0 ± 0.4
1.0%	27.2 ± 0.6	69.4 ± 0.9	2.36 ± 0.17	72.0 ± 17.0
2.0%	20.8 ± 0.4	73.1 ± 2.0	1.75 ± 0.68	71.1 ± 2.0
3.0%	16.6 ± 1.4	70.5 ± 0.2	1.83 ± 0.90	72.9 ± 6.0
		Delaware		
0.0%	27.4 ± 7.6	65.3 ± 2.9	2.08 ± 0.34	53.8 ± 4.7
0.5%	31.6 ± 0.6	69.6 ± 0.3	1.83 ± 0.00	56.2 ± 2.7
1.0%	31.1 ± 0.2	73.4 ± 1.6	1.68 ± 0.22	66.3 ± 3.3
2.0%	25.1 ± 0.5	72.4 ± 0.5	1.12 ± 0.11	73.5 ± 7.6
3.0%	19.1 ± 0.3	72.0 ± 1.2	1.24 ± 0.40	76.1 ± 0.7
		Concord		
0.0%	10.12 ± 1.57	43.92 ± 0.02	2.75 ± 0.28	40.1 ± 3.4
0.5%	24.76 ± 1.64	61.00 ± 2.71	2.10 ± 0.52	46.7 ± 2.7
1.0%	23.84 ± 1.64	65.50 ± 1.69	1.68 ± 0.34	55.3 ± 2.2
2.0%	13.75 ± 0.95	63.89 ± 0.99	1.51 ± 0.34	68.0 ± 3.4
3.0%	9.60 ± 0.05	67.78 ± 0.94	1.35 ± 0.45	75.4 ± 3.4
		Catawba		
0.0%	24.9 ± 6.7	58.6 ± 3.3	2.75 ± 0.28	47.4 ± 0.7
0.5%	29.7 ± 1.6	69.3 ± 1.3	2.75 ± 0.52	56.6 ± 1.8
1.0%	28.9 ± 2.4	71.7 ± 1.1	1.91 ± 0.12	61.7 ± 0.4
2.0%	20.1 ± 1.1	69.4 ± 0.7	1.64 ± 0.40	64.5 ± 3.3
3.0%	15.9 ± 0.3	68.1 ± 0.2	1.95 ± 0.28	69.1 ± 2.5
		Cayuga White		
0.0%	32.4 ± 3.6	63.6 ± 0.1	2.60 ± 0.06	34.3 ± 1.1
0.5%	38.9 ± 0.4	74.0 ± 0.4	2.44 ± 0.17	50.4 ± 0.0
1.0%	39.2 ± 1.1	76.6 ± 0.6	1.96 ± 0.17	54.8 ± 2.5
2.0%	31.2 ± 0.4	75.7 ± 0.2	2.04 ± 0.62	67.1 ± 0.7
3.0%	25.6 ± 2.3	75.4 ± 0.7	1.96 ± 0.05	70.3 ± 2.7
		Chardonnay		
0.0%	45.6 ± 1.7	72.3 ± 2.3	2.24 ± 0.56	45.6 ± 7.6
0.5%	43.4 ± 2.9	75.8 ± 1.4	2.68 ± 0.29	54.2 ± 1.6
1.0%	40.5 ± 1.5	77.3 ± 1.3	1.95 ± 0.62	68.4 ± 15.6
2.0%	32.1 ± 1.0	76.3 ± 0.7	2.31 ± 0.23	63.8 ± 5.1
3.0%	22.6 ± 0.3	74.2 ± 1.5	2.28 ± 0.62	73.0 ± 2.9
		Johannisberg Riesling		
0.0%	25.2 ± 10.7	67.5 ± 1.0	2.99 ± 0.17	59.3 ± 2.0
0.5%	36.0 ± 4.6	71.4 ± 2.0	2.91 ± 1.07	58.9 ± 2.5
1.0%	37.7 ± 0.3	74.8 ± 1.1	1.96 ± 0.62	61.0 ± 2.5
2.0%	26.9 ± 0.3	73.4 ± 0.5	2.15 ± 0.11	71.4 ± 0.7
3.0%	21.4 ± 1.3	72.9 ± 1.6	1.95 ± 0.96	71.6 ± 2.4
		Melody		
0.0%	30.9 ± 2.0	69.4 ± 0.5	2.59 ± 0.06	53.8 ± 2.2
0.5%	41.9 ± 0.6	74.8 ± 0.5	2.59 ± 0.62	52.8 ± 4.4
1.0%	40.1 ± 3.5	76.2 ± 1.1	2.19 ± 0.40	68.0 ± 0.5
2.0%	35.3 ± 2.1	75.0 ± 0.3	2.90 ± 0.49	64.8 ± 3.3
3.0%	23.3 ± 2.4	72.8 ± 0.1	1.99 ± 0.56	74.0 ± 0.0
		Cabernet Sauvignon		
0.0%	18.0 ± 5.7	64.5 ± 1.2	3.03 ± 0.11	61.5 ± 1.1
0.5%	35.0 ± 2.8	70.6 ± 2.5	3.47 ± 0.39	57.0 ± 5.3
1.0%	36.3 ± 2.3	73.7 ± 0.9	2.63 ± 0.45	62.7 ± 4.0
2.0%	26.4 ± 2.1	71.4 ± 0.6	2.31 ± 0.45	69.5 ± 6.7
3.0%	19.5 ± 0.5	70.4 ± 1.3	2.07 ± 0.00	78.0 ± 0.5

^a Free Run, Yield, and Loss are expressed as % total weight; Press aid was ground paper; Delta Volume is difference in initial and final volumes in press expressed as cm³.

CONCLUSIONS

The amount of press aid was the most important variable that affected pressing characteristic as well as juice yields of the studied grape cultivars. There was no significant difference in the effect of rice hulls or paper press aids on pressing characteristics. The results emphasize the important role of press aids on pressing characteristics and juice yields, and these must be considered along with the previously reported effects of press aids on the aroma characteristics of unfermented must and wine (Kinzer and Schreier 1980). As expected, the pressing characteristics of the different grape cultivars studied: Baco Noir, Cabernet Sauvignon, Catawba, Cayuga White, Chardonnay, Concord, Delaware, Melody, Niagara, Johannisberg Riesling, and Seyval Blanc were significantly different.

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DEWALD, B., DULANEY, J.T., and TOUSTER, O. 1974. Solubilization and polyacrylamide gel electrophoresis of membrane enzymes with detergents. In *Methods in Enzymology*, Vol. xxxii, (S. Fleischer and L. Packer, eds.) pp. 82-91, Academic Press, New York.

HASSON, E.P. and LATIES, G.G. 1976. Separation and characterization of potato lipid acylhydrolases. *Plant Physiol.* 57,142-147.

ZABORSKY, O. 1973. *Immobilized Enzymes*, pp. 28-46, CRC Press, Cleveland, Ohio.

Journal abbreviations should follow those used in *Chemical Abstracts*. Responsibility for the accuracy of citations rests entirely with the author(s). References to papers in press should indicate the name of the journal and should only be used for papers that have been accepted for publication. Submitted papers should be referred to by such terms as "unpublished observations" or "private communication." However, these last should be used only when absolutely necessary.

Tables should be numbered consecutively with Arabic numerals. The title of the table should appear as below:

Table 1. Activity of potato acyl-hydrolases on neutral lipids, galactolipids, and phospholipids

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Figures should be listed in order in the text using Arabic numbers. Figure legends should be typed on a separate page. Figures and tables should be intelligible without reference to the text. Authors should indicate where the tables and figures should be placed in the text. Photographs must be supplied as glossy black and white prints. Line diagrams should be drawn with black waterproof ink on white paper or board. The lettering should be of such a size that it is easily legible after reduction. Each diagram and photograph should be clearly labeled on the reverse side with the name(s) of author(s), and title of paper. When not obvious, each photograph and diagram should be labeled on the back to show the top of the photograph or diagram.

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