Inverse Method to Estimate Microbial Inactivation Kinetic Parameters in Conduction-Heated Canned Foods

Kirk Dolan, Biosystems & Ag Engineering, MSU
Lijian Yang, Statistics & Probability, MSU
Chris Trampel, Electrical & Computer Engineering, Iowa State University

MICHIGAN STATE UNIVERSITY

Microbial Inactivation Kinetic Parameters

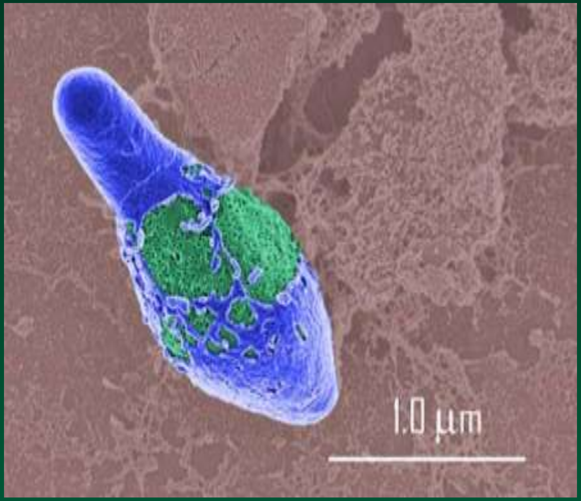
Needed to determine the time and temperature treatment for canned foods.

- Food containers used annually:
  - 22 billion steel cans
  - 75 billion glass jars
- Low-acid canned foods need ~121°C for 20-60 min. to inactivate *Clostridium botulinum.*

Handbook of Food Preservation, 2nd ed. M. S. Rahman, editor, p. 933, CRC Press

http://spoonfeedin.blogspot.com/2009/02/business-hidden-commodity-in-canned.html

Clostridium botulinum



- Most potent natural toxin known
- One-millionth of a gram of toxin can kill an adult
- Spores cannot grow and cannot produce toxin when pH < = 4.6
- Canning regulations are based on C. bot

How microbial kinetic parameters are usually estimated

- For high-moisture foods at constant temperature and constant moisture
  - Typical methods: capillary tubes for liquids
  - Assumes minimal lag time, no temp. gradients
  - Easy set-up and straightforward math

Example for high-moisture foods

- First-order model with Arrhenius rate constant. Temp. constant.

N: # of m.o.'s at time t

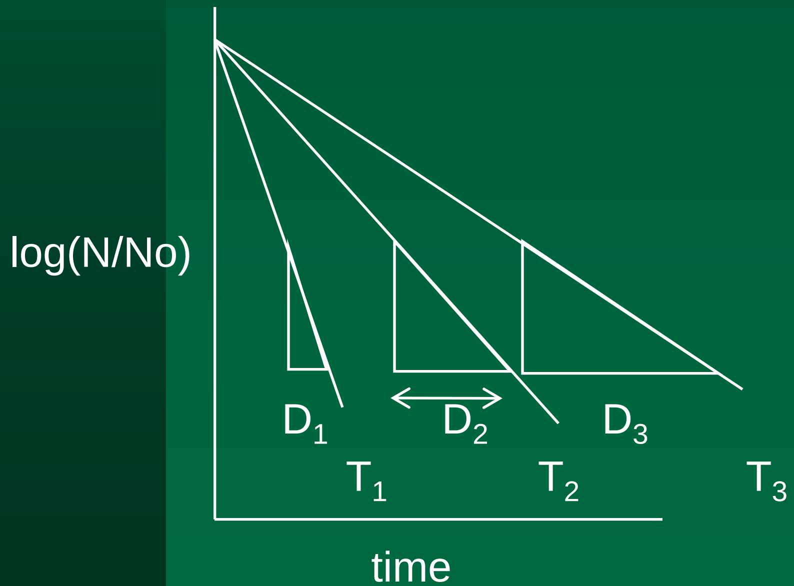
N_o: initial number m.o.'s

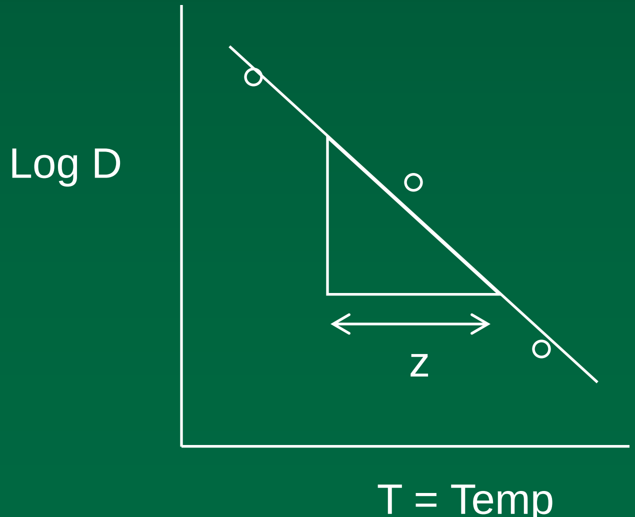
D_r = inverse rate, min

z = temp change to cause 10-fold increase in D

$$\log_{10}(N/N_0) = -t/D_r$$

$$D = D_r 10^{\frac{-(T-T_r)}{z}}$$





Disadvantages of isothermal methods

- Limited temperature range gives lower statistical confidence
- More experiments required
- Unavoidable thermal lags
- Small samples may not be large enough for high concentration of m.o.'s

Advantages of dynamic nonisothermal expts

- Can estimate parameters with one experiment
- Covers entire temp range
- No thermal lag, can handle solid foods
- Sample can be any size, as long as temperature gradient is known
- <u>Represents most true food processes</u>
- Disadvantage: Math is more complex

For Conduction-heated foods

- First-order model with Arrhenius rate constant. Temp. and moisture changing with time.
- First-order $\frac{dN}{dt} = -N / D$
- Reciprocal Rate: $D = D_r 10^{\frac{-(T - T_r)}{z}}$
- Integrate:

$$\log(N / N_0) = -\left(\frac{1}{D_r}\right)\int_0^t 10^{\left(\frac{T(t) - T_r}{z}\right)} dt$$

- This is the microbial survival ratio at any <u>one point</u> in the can.
- Nonlinear regression with Excel Solver or Matlab to estimate D_r, z

Objectives

To develop a method to:

1. Estimate *kinetic parameters* for microbial inactivation in conduction-heated foods;
2. Compute confidence contours for the two kinetic parameters, D_r and z

Materials and Methods

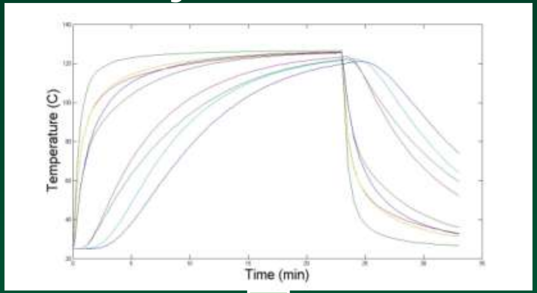
Overview of the process

Inoculation of canned pea puree with *B. Stearo-thermophilus* spores

$N_0 = 9 \times 10^5$ CFU/mL (Welt, et al. 1997 J. Food Sci)

Retorting at different times at 104, 112, 120C

Temperatures inside can predicted by analytical solns



Plating of spores to determine N

Kinetic parameter estimation

Materials

- Can size = 6.02 cm dia x 3.48 cm height (squat “tuna” can)
- Finite cylinder with boundary conditions of the 3rd kind: h ≈ 5500 $W/m^2°C$ (steam)
- Uniform initial temp = 0°C
- k = 0.68 W/m°C, Cp = 4100J/g°C

Analytical Solution to heating finite cylinder

- Analytic solution is product of infinite slab and cylinder for heating

$$\frac{T(r,\xi,t)-T_s}{T_i-T_s}=\sum_{m=1}^{\infty}\frac{2\left(\frac{hl}{k}\right)\cos\left(\lambda_m\xi\right)\sec(\lambda_m)\exp\left[-\lambda_m^2\left(\frac{\alpha t}{l^2}\right)\right]}{\left(\frac{hl}{k}\right)\left(\frac{hl}{k}+1\right)+\lambda_m^2}$$

Slab

$$\times\sum_{n=1}^{\infty}\frac{2\left(\frac{hR}{k}\right)J_0\left(\lambda_n r\right)\exp\left[-\lambda_n^2\left(\frac{\alpha t}{R^2}\right)\right]}{\left[\left(\frac{hR}{k}\right)^2+\lambda_n^2\right]J_0\left(\lambda_n\right)}$$

Cylinder

Analytical Solution to <u>cooling</u> finite cylinder

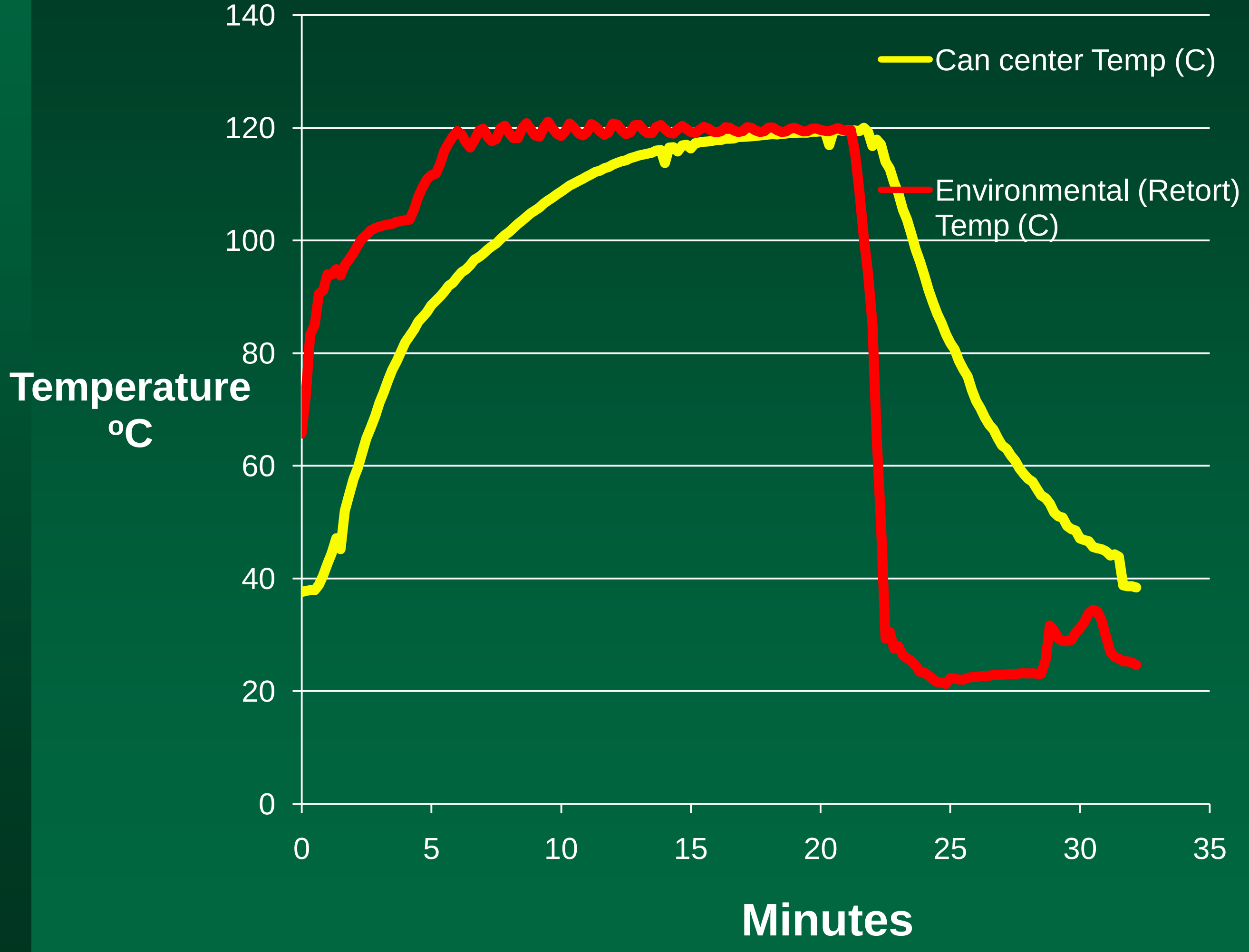
- Analytic solution when heating stops (non-uniform temp initial condition) and cooling begins . Assumes h = ∞ at boundary

$$\frac{T(r,\xi,\tau)-T_w}{T_s-T_i} = 4\sum_{j=0}^{\infty}\sum_{i=0}^{\infty}\frac{(-1)^i \exp\left[-\left(\gamma^2\left(i+\frac{1}{2}\right)^2\pi^2+\omega_j^2\right)\tau_w\right]\mathrm{J}_\mathrm{o}\left(\omega_j r\right)}{\left(i+\frac{1}{2}\right)\pi\omega_j\,\mathrm{J}_1\left(\omega_j\right)}$$

$$\times\cos\left[\left(i+\tfrac{1}{2}\right)\pi\xi\right]\left\{\left(\frac{T_s-T_w}{T_s-T_i}\right)-\exp\left[-\left(\omega_j^2+\left(i+\tfrac{1}{2}\right)^2\pi^2\gamma^2\right)\tau\right]\right\}$$

Lenz, M. K., & Lund, D. B. (1977a). The lethality-Fourier number method: experimental verification of a model for calculating temperature profiles and lethality in conduction-heating canned foods. Journal of Food Science, 42(4), 989–996, 1001.

Typical measured temperature during steam retort processing



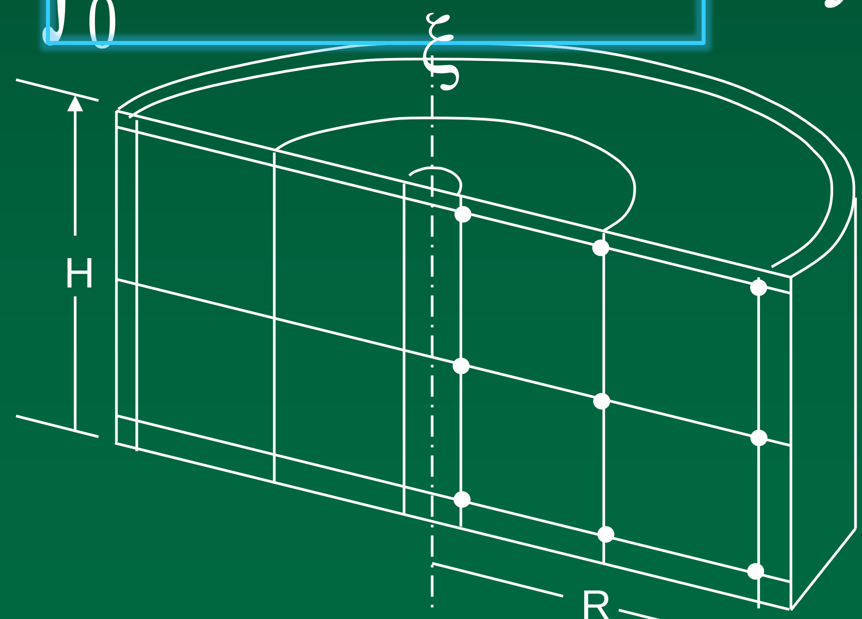
Model Formulation

Inverse Problem: Kinetic Parameters of Microbial inactivation

Calculated mass-average retention =

$$(\bar{N}/N_0)_{\text{model}} = 2\int_0^H \int_0^R 10^{-\left(\frac{1}{D_r}\right)\beta(r,\xi,t)} \, r\,dr\,d\xi$$

- 3-point Gauss integration



Methods: Different Heating Treatments

- Heated canned pea puree in retort at three constant retort temperatures (T_∞): 104, 112, 120°C
- Heating times: 0-305 min
- Standard microbial techniques to measure mass-average microbial retention in each can =

$$(\bar{N} / N_0)_{observed}$$

Estimation of Kinetic Parameters

- Minimize Sum of Squares of errors

$$SSQ = \sum_{i=1}^{n}\left[\log\left(\bar{N}/N_o\right)_{obs,\, i} - \log\left(\bar{N}/N_o\right)_{model,\, i}\right]^2$$

Scaled Sensitivity Coefficients

- Model: $\log(N/N_0) = -\left(\frac{1}{D_r}\right)\int_0^t 10^{\left(\frac{T(t)-T_r}{z}\right)}dt = -\left(\frac{1}{D_r}\right)\beta$

$$D_r \frac{\partial \log(N/N_0)}{\partial D_r} = D_r\left(\frac{\beta}{D_r^{\,2}}\right) = \boxed{-\log(N/N_0)}$$

$$z\frac{\partial \log(N/N_0)}{z} = z\left(\frac{1}{D_r z^2}\right)\int_0^t \left(T(r,z,t)-T_r\right)10^{\left(\frac{T(t)-T_r}{z}\right)}dt$$

$$= \boxed{\frac{\beta'}{D_r z}}$$

Parameter Joint Confidence Region

- Motulsky iteration method

$$SS_{all-fixed} = SS_{best-fit}\left(\frac{p}{n-p}F(p, n-p)+1\right)$$

- n: number of data
- p: number of parameters
- F: statistical F distribution
- Orders of magnitude more computationally intensive than elliptical approximation

Asymptotic Confidence Bands

- Supplied by nlinfit in Matlab®

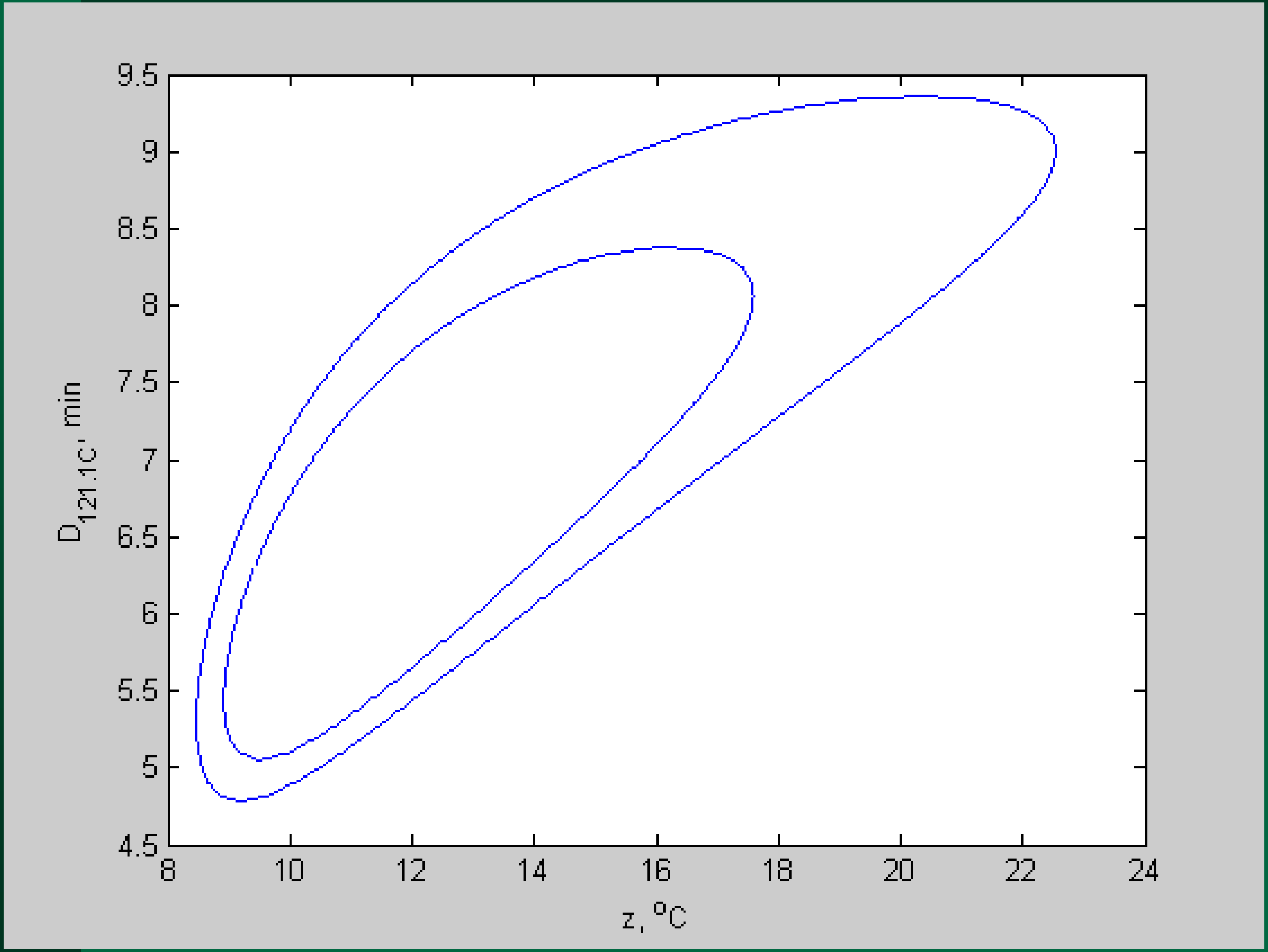
Results

Results for 2 parameters estimation

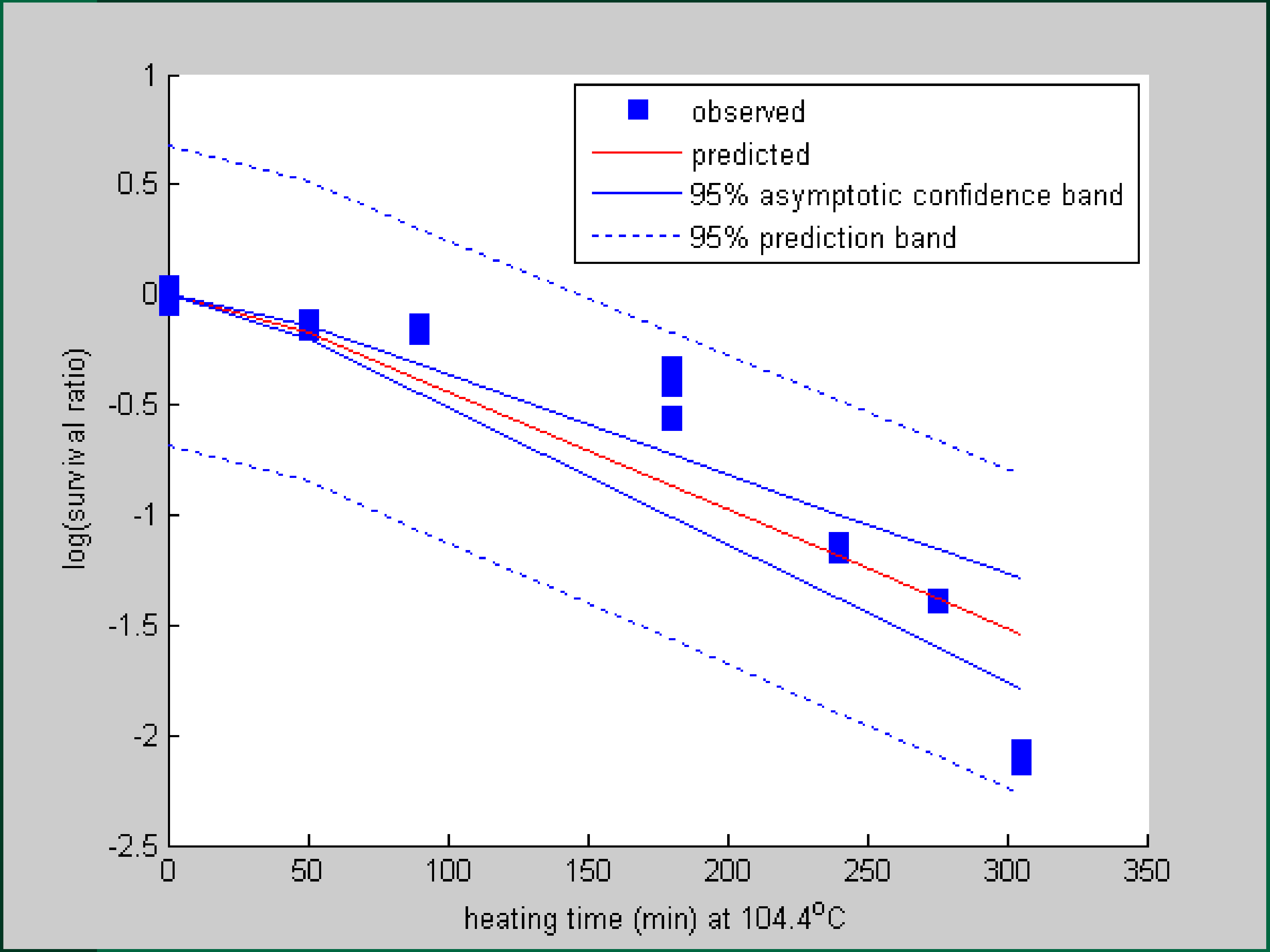
$$\left(\overline{N}/N_o\right)_{pred} = 2\iint 10^{-\frac{1}{D_r}\int_0^t 10^{\left(\frac{T(r,z,t)-T_r}{z}\right)} dt}\, r\,dr\,d\xi$$

root mean square error	Number of data	parameter estimate	standard error	correlation coefficient $\rho_{D_r z}$	95% asymptotic confidence interval
0.225	24	$D_{121.1^oC}$ = 6.67 min z = 11.74ºC	0.421 min^{-1} 0.900 ºC	0.529 (T_r = 381 K)	(5.80, 7.54) (9.88, 13.61)

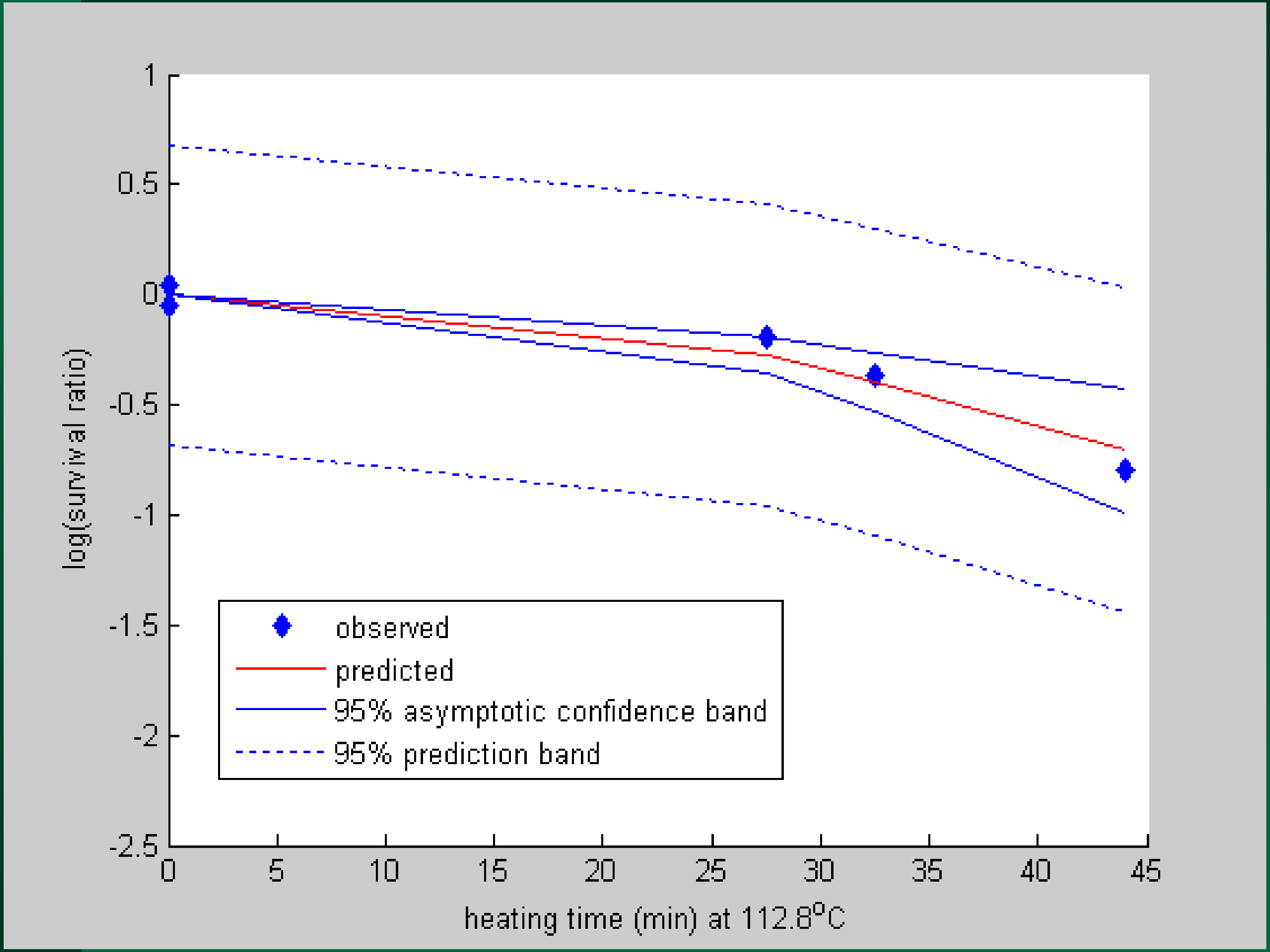
95%(inner) and 99% (outer) joint confidence region



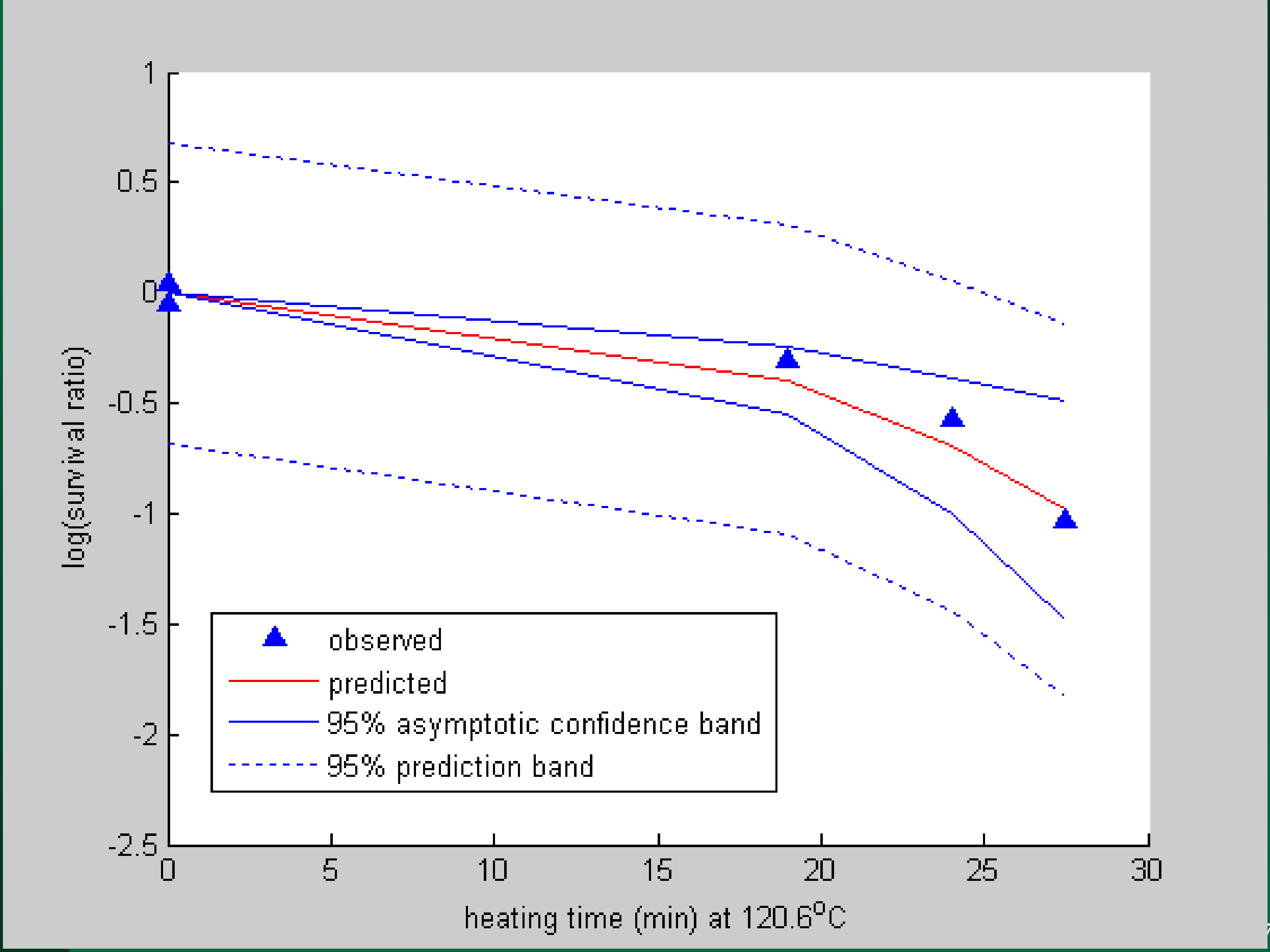
Results @ Retort Temp = 104.4°C



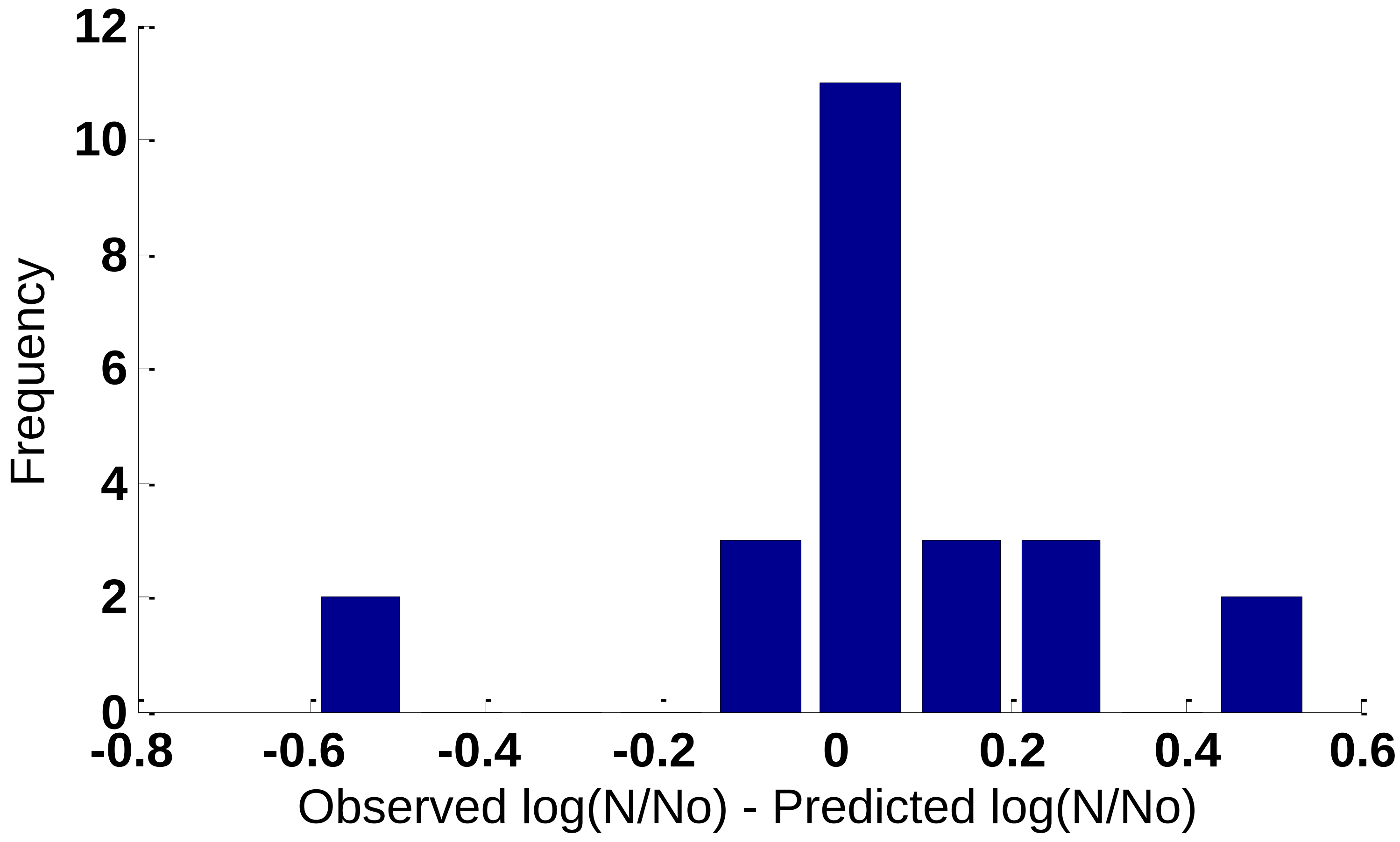
Results @ Retort Temp = 112.8°C



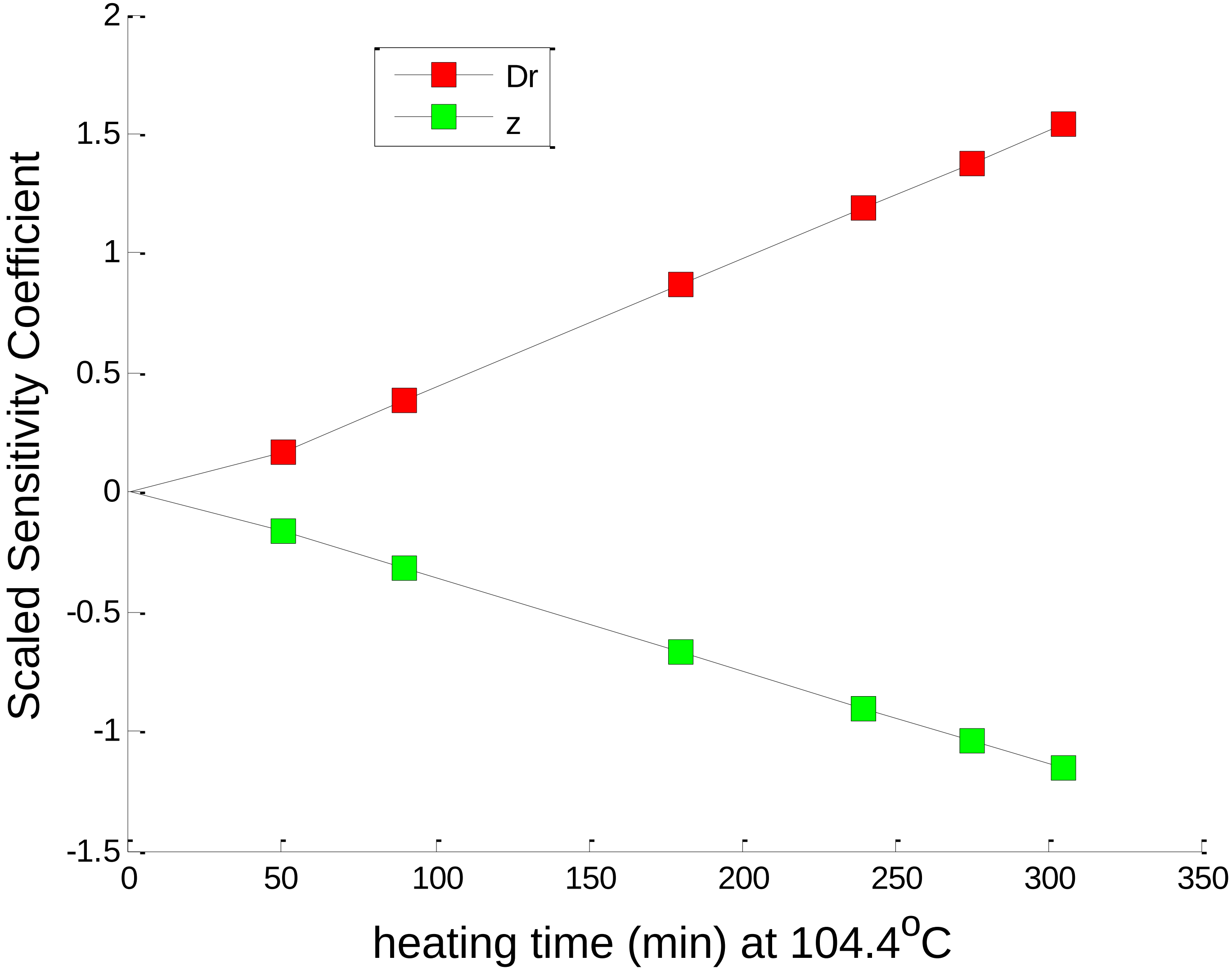
Results @ Retort Temp = 120.6°C



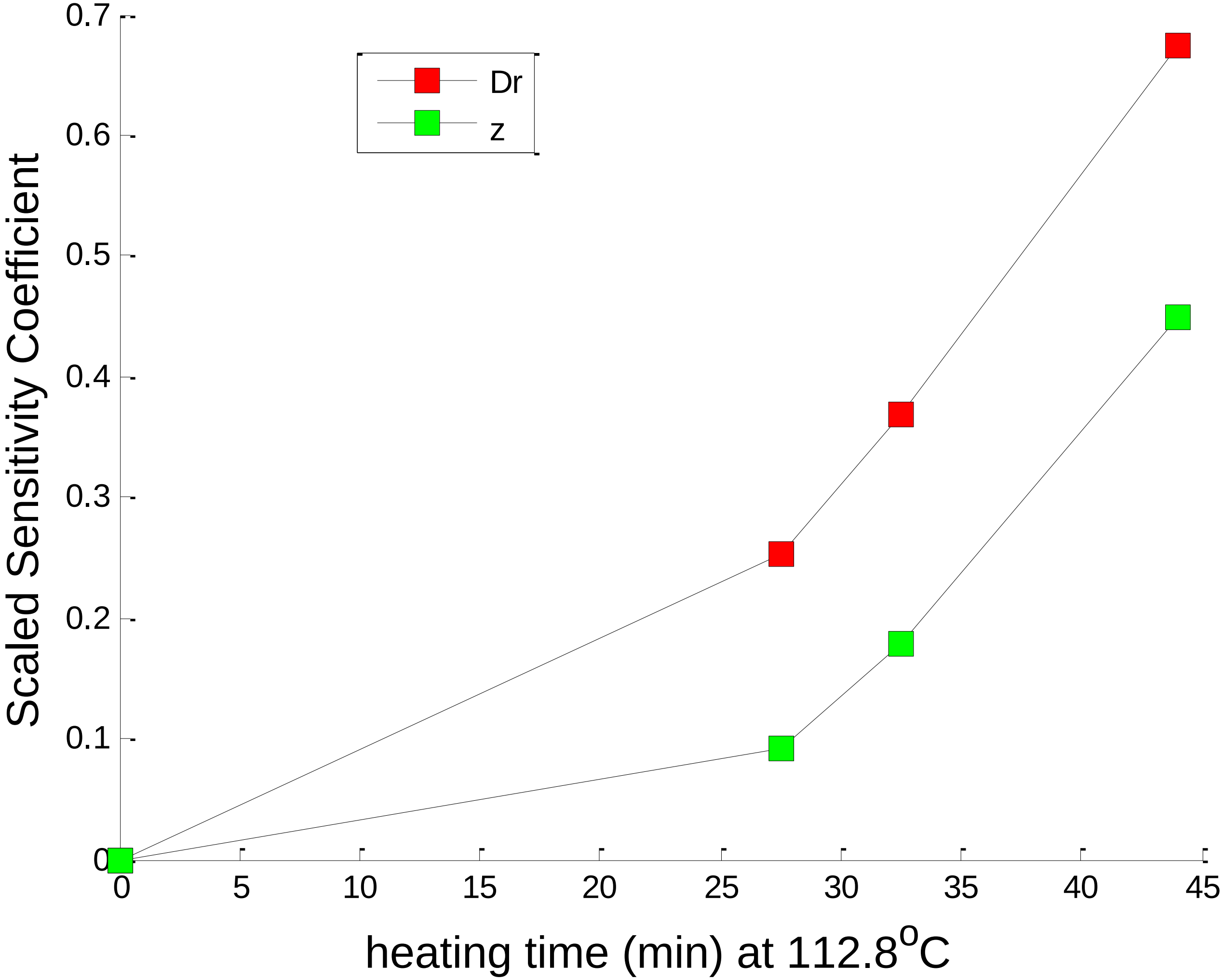
Residual Histogram



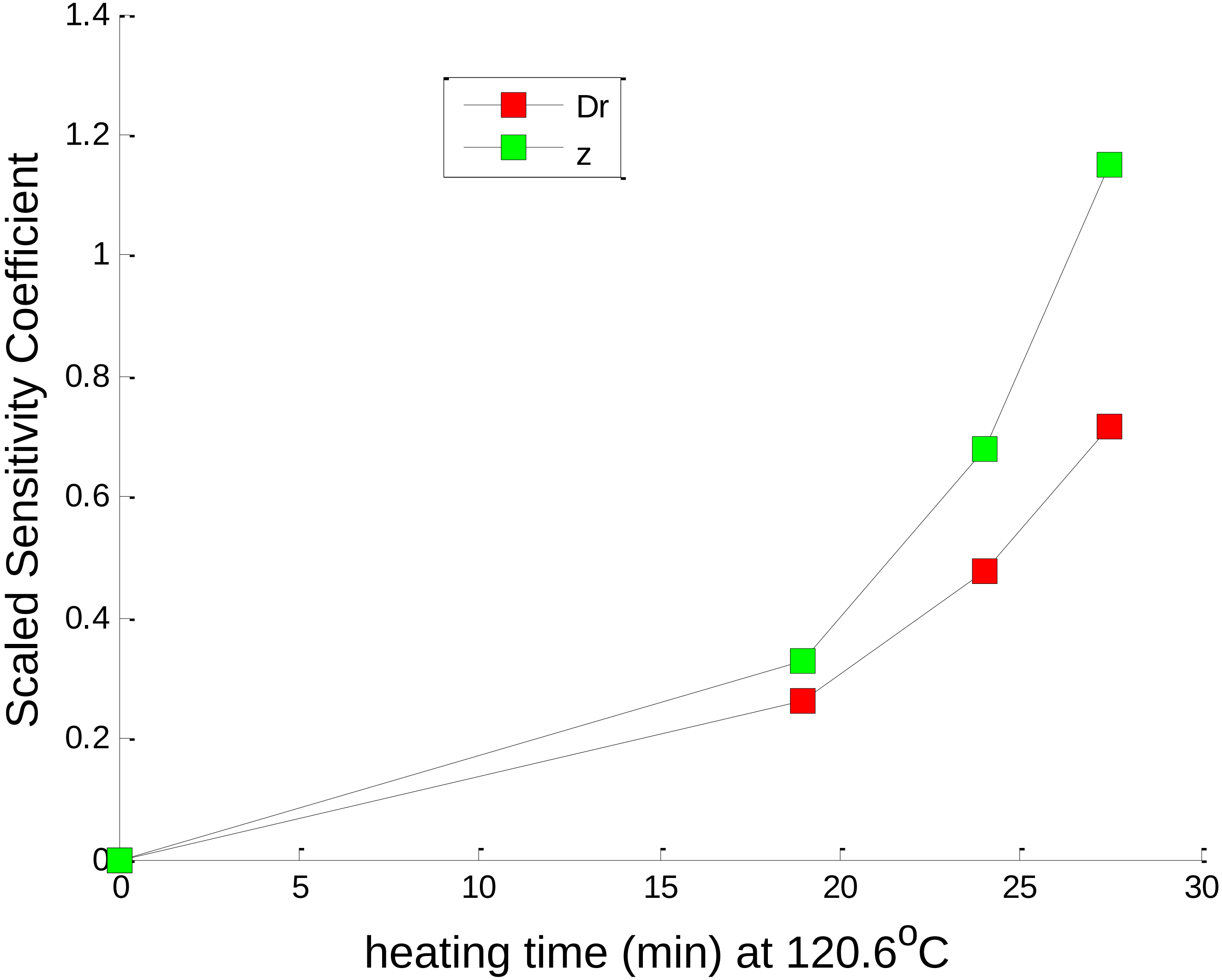
Sensitivity Coefficients at 104.4°C



Sensitivity Coefficients at 112.8°C



Sensitivity Coefficients at 120.6°C



New Information Provided by This Study

- Microbial kinetic parameters can be estimated simultaneously in conduction-heated foods
- Confidence bands for the dependent variable were computed
- Sensitivity coefficients computed
- Can save experimental $$ and effort

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