Nonlinear Multi-parameter Estimation and Bootstrap Confidence Intervals for Degradation of Nutraceuticals in Foods Processed at High Temperature

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Introduction & Objectives

Degradation of nutraceuticals in low- and intermediate-moisture foods heated at high temperature (greater than 100°C) is difficult to model because of the temperature gradients and long heating time. Isothermal experiments above 100°C are difficult to design because they require high pressure and sophisticated instrumentation for temperature measurement. Therefore the objective of this study was to develop a non-isothermal method to estimate the thermal degradation kinetics of nutraceuticals like anthocyanins. Thermal and kinetic parameters were estimated for degradation of anthocyanins in grape pomace, a process that is representative of processing of low-moisture foods at higher temperatures.

Confidence intervals can help design food processes and improve food quality and safety. Bootstrap confidence interval is a more realistic estimate of the true confidence interval than the commonly used asymptotic confidence interval. Hence, the Asymptotic and Bootstrap confidence intervals were also estimated.

Methods

Grape pomace at moisture of 42% (wb) was heated in sealed 202 x 214 cans in a steam retort at 126.7 ^oC for over 60 minutes. Can center temperature was measured by thermocouple and predicted using COMSOL® software. Thermal conductivity (k) and specific heat (C_p) were estimated as quadratic functions of temperature using COMSOL® & nonlinear regression in MATLAB®. The k and C_p functions were then used to predict temperature inside the grape pomace during retorting. Similar heating experiments were run at different time-temperature treatments from 8 to 25 minutes for kinetic parameter estimation. Anthocyanin concentration in the grape pomace was measured using HPLC. Rate constant (k_{110^oC}) and activation energy (E_a) were estimated using nonlinear regression in MATLAB®. Confidence band and the prediction band were computed for the predicted Y (anthocyanin degradation). Inference regions and joint confidence regions for k_{110^oC} and E_a were estimated using the iterative methods for all the models. Bootstrap confidence intervals were calculated and compared to the 95% asymptotic confidence intervals.

Results

The degradation values were used to estimate the parameters, which were k_{110^oC} =0.06 min^{-1} and E_a=65.3 kJ/mol. The 95% confidence intervals for the parameters, and the confidence bands and prediction bands for anthocyanin retention were plotted. Asymptotic Confidence interval for k_{110^oC} and E_a were (0.055, 0.067) and (32.6,97.9) respectively. Bootstrap 95% confidence interval for k_{110^oC} and E_a were (0.055, 0.066) and (50.9, 100.82) respectively. Bootstrap confidence band and prediction bands for anthocyanin retention were smaller than asymptotic confidence and prediction bands, respectively. The smaller width of the bootstrap bands, which are considered more accurate than asymptotic bands, allows more accurate process design and cost-savings, leading to higher-quality nutraceutical products.

The methods described above are useful for process design for nutraceutical products. For researchers without access to MATLAB®, all calculations can also be done in MS Excel®. The availability of the software and the statistical packages makes nonlinear regression accessible for all researchers. Other investigators can consider reporting confidence intervals and confidence regions using the methods presented, for maximizing use of limited data sets and improving quality of food.

References

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